

# **CARS Environment, Safety and Health Plan**

**Version  
April 2020**

**Mark Rivers, CARS Director**



09/01/2013

---

[Signature]

[Date]

# **CARS Environment, Safety and Health Plan Table of Contents**

<b>CARS ENVIRONMENT, SAFETY AND HEALTH PLAN .....</b>	<b>2</b>
<b>SECTION 1 CARS SAFETY POLICIES.....</b>	<b>3</b>
<b>1.0 PURPOSE.....</b>	<b>3</b>
<b>1.1 SCOPE .....</b>	<b>3</b>
<b>1.2 REFERENCES .....</b>	<b>3</b>
<b>1.3 GENERAL POLICIES .....</b>	<b>3</b>
<b>SECTION 2 - CARS SAFETY ORGANIZATION &amp; RESPONSIBILITIES.....</b>	<b>4</b>
<b>APPENDIX A - SAFETY ASSIGNMENTS &amp; ESAF APPROVERS.....</b>	<b>5</b>
<b>APPENDIX A.1 CARS SAFETY ASSIGNMENTS (UPDATED 5/11/11) .....</b>	<b>5</b>
<b>APPENDIX A.2 CARS PERSONNEL WITH EXPERIMENT SAFETY APPROVAL AUTHORITY .....</b>	<b>6</b>
<b>APPENDIX A.3 CARS QUALIFIED ELECTRICAL WORKERS .....</b>	<b>7</b>
<b>APPENDIX B APS STANDARD PROCEDURES .....</b>	<b>8</b>
<b>APPENDIX C CARS SPECIFIC STANDARD OPERATING PROCEDURES .....</b>	<b>9</b>

## **Section 1 CARS Safety Policies**

### **1.0 Purpose**

CARS is committed to ensuring that all CARS activities are conducted in a safe and environmentally sound manner. This plan describes the CARS safety program which is implemented to fulfill this commitment.

### **1.1 Scope**

To ensure that all CARS activities are conducted in a safe and environmentally sound manner, this plan defines 1) the standards to be followed by CARS, and 2) the responsibilities within the CARS organization.

### **1.2 References**

All activities at Argonne National Laboratory - East (ANL-E) will conform to the requirements of the documents listed below, except as provided for by variances or APS procedures. All of the following are available through the CARS Safety Coordinators.

1. ANL-E Environment, Safety and Health Manual
2. APS User Policies and Procedures
3. ANL-E Hoisting and Rigging Manual
4. ANL-E Transportation Safety Manual
5. ANL-E Waste Handling Procedures Manual

### **1.3 General Policies**

- 1) Failing to conform with this plan may result in sanctions and/or the loss of access to the APS and CARS facilities.
- 2) Any person has the authority to stop activities that are unsafe or environmentally unsound.
- 3) CARS will comply with current version of the APS Policy and Procedure for configuration control of shielding systems. No safety system under configuration control is to be modified without CARS and APS approval. (Refer to the APS User Policies and Procedures for the complete policy and procedure).

4) CARS will cooperate with the APS to facilitate the oversight responsibilities of the APS, ANL and the DOE.

5) Experimenters shall identify to CARS the potential hazards associated with their activities and hazardous materials to be used in experiments at the APS, and no experiment shall proceed without CARS approved and posted APS Experiment Safety Approval Form (ESAF).

6) New or modified equipment and unreviewed activities must be approved by the CARS Director, or designee, prior to energizing the equipment or the start of work. Before any change in CARS operations that might reasonably be thought to increase the risk of significant adverse impact on the APS facilities, the environment or any person, is begun, CARS will obtain the written approval of the APS Operations Division Director, or designee.

7) CARS will maintain a list of current safety assignments (Appendix A) and will update this plan to keep it consistent with scope of CARS activities. The assignment list will be reviewed at least annually and the plan biannually with updates provided to the APS User Program Director.

## **Section 2 - CARS Safety Organization & Responsibilities**

The CARS Director has line responsibility for safety for all CARS activities at ANL and for ensuring that this plan is implemented. The Director is also responsible for evaluating and responding in a graded manner to non conformances with this plan.

The CARS Safety Officer communicates safety issues between the CARS Safety Coordinators and the CARS Director. The CARS Safety Officer ensures that the safety directives of the CARS Director are implemented.

The CARS Safety Coordinators report to the CARS Director and are responsible for implementing and overseeing conformance with this safety plan. The CARS Safety Coordinators ensure that CARS has access to the ANL-E ESH Manual and the other identified standards and to assist CARS members and users in meeting the requirements of these standards.

## Appendix A - Safety Assignments & ESAF Approvers

### Appendix A.1 CARS Safety Assignments (updated 5/11/11)

Duty	Assigned to:	Telephone	e-mail
<b>Director</b> (line responsibility for safety for CARS)	Mark Rivers (Sector 13)	630-252-0464	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
	Mark Rivers (Sector 14)	630-252-0464	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
	Mark Rivers (Sector 15)	630-252-0464	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
<b>CARS Safety Officer</b> (communicates safety issues between the CARS Director and the CARS Safety Coordinators)	Guy Macha (Sector 13)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 14)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 15)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
<b>CARS Safety Coordinators</b> (day to day responsibility for maintaining safe conditions in spaces occupied by the (CAT))	Mark Rivers (Sector 13)	630-252-0422	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
	Vukica Srajer (Sector 14)	630-252-0445	<a href="mailto:srajer@cars.uchicago.edu">srajer@cars.uchicago.edu</a>
	Binhua Lin (Sector 15)	630-252-0464	<a href="mailto:lin@cars.uchicago.edu">lin@cars.uchicago.edu</a>
<b>Experimental Safety Review Coordinator</b> (responsible for ensuring that safety controls are in place for experiments)	Mark Rivers (Sector 13)	630-252-0422	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
	Robert Henning (Sector 14)	630-252-0451	<a href="mailto:henning@cars.uchicago.edu">henning@cars.uchicago.edu</a>
	Binhua Lin (Sector 15)	630-252-0464	<a href="mailto:lin@cars.uchicago.edu">lin@cars.uchicago.edu</a>
<b>Chemical Safety Coordinator</b> (responsible for assisting in identifying and implementing proper controls for the hazards associated with chemicals , gases, and nonbiological samples)	Joanne Stubbs (Sector 13)	630-252-0427	<a href="mailto:stubbs@cars.uchicago.edu">stubbs@cars.uchicago.edu</a>
	Vukica Srajer (Sector 14)	630-252-0463	<a href="mailto:v-srajer@cars.uchicago.edu">v-srajer@cars.uchicago.edu</a>
	Wei Bu (Sector 15)	630-252-0470	<a href="mailto:bu@cars.uchicago.edu">bu@cars.uchicago.edu</a>
<b>Electrical Safety Coordinator</b> (responsible for ensuring that all CAT personal adhere to safe electrical design criteria and work practices)	Pasquale DiDonna(Sector 13)	630-252-0428	<a href="mailto:pdidonna@cars.uchicago.edu">pdidonna@cars.uchicago.edu</a>
	Guy Macha (Sector 14)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 15)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
<b>Hoisting and Rigging Coordinator</b> (performs and documents monthly inspections of nylon slings and assists chain fall hoist operator when necessary)	Mike Proskey (Sector 13)	630-252-0428	<a href="mailto:proskey@cars.uchicago.edu">proskey@cars.uchicago.edu</a>
	Guy Macha (Sector 14)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Wei Bu(Sector 15)	630-252-0402	<a href="mailto:bu@cars.uchicago.edu">bu@cars.uchicago.edu</a>
<b>Sealed Radioactive Source Custodian</b> (responsible for knowing the location of all sources at all times and will ensure that all necessary records are maintained)	Guy Macha (Sector 13)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 14)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 15)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
<b>Biosafety Coordinator</b> (responsible for assisting in identifying and implementing proper controls for the hazards associated with biological samples)	Vukica Srajer (Sector 13)	630-252-0445	<a href="mailto:srajer@cars.uchicago.edu">srajer@cars.uchicago.edu</a>
	Vukica Srajer (Sector 14)	630-252-0445	<a href="mailto:srajer@cars.uchicago.edu">srajer@cars.uchicago.edu</a>
	Vukica Srajer (Sector 15)	630-252-0445	<a href="mailto:srajer@cars.uchicago.edu">srajer@cars.uchicago.edu</a>
<b>Laser Control Area (LCA) Supervisor</b> (responsible for ensuring that devices using Lasers are operated safely)	Mark Rivers (Sector 13)	630-252-0422	<a href="mailto:rivers@cars.uchicago.edu">rivers@cars.uchicago.edu</a>
	Vukica Srajer (Sector 14)	630-252-0455	<a href="mailto:v-srajer@uchicago.edu">v-srajer@uchicago.edu</a>
	Yu-Sheng Chen (Sector 15)	630-252-0471	<a href="mailto:yschen@cars.uchicago.edu">yschen@cars.uchicago.edu</a>
<b>Hazardous Shipping Coordinator</b> (responsible for ensuring that hazardous materials are properly shipped)	Guy Macha (Sector 13)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 14)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>
	Guy Macha (Sector 15)	630-252-0448	<a href="mailto:macha@cars.uchicago.edu">macha@cars.uchicago.edu</a>



**Appendix A.3          CARS Qualified Electrical Workers**

**Approved by Mark Rivers, CARS Director**

**April, 27, 2020**

**Pasquale Di Donna          GSE CARS          [didonna@cars.uchicago.edu](mailto:didonna@cars.uchicago.edu)          (630) 252-0428**

## **Appendix B APS Standard Procedures**

Whenever possible CARS will follow APS Standard Procedures. The current version of these guidelines can be found on the APS User Safety web page <https://www.aps.anl.gov/Safety-and-Training> .



## **Appendix C CARS Specific Standard Operating Procedures**

CARS has also evaluated the unique hazards that will be encountered in its operations, and, to mitigate these hazards the has developed the Standard Operating Procedures listed and documented below:

- APPENDIX C.1 GSECARS SOP for Lasers at 13 ID-D (April 2016)
- APPENDIX C.2 GSECARS SOP for Lasers in Building 434A, Room A020 (February 2018)
- APPENDIX C.3 GSECARS SOP for Lasers at 13 BM-D (January 2014)
- APPENDIX C.4 GSECARS Safety Analysis and Standard Operating Procedures DDIA-30 Apparatus (March 2011)
- APPENDIX C.5 GSECARS SOP for Preparation of Beryllium Gaskets In Laboratory A030, Bldg 434A
- APPENDIX C.6 GSECARS Safety Procedures for 13-BM-D LVP Experiments (July 2008)
- APPENDIX C.7 GSECARS Safety Procedures for LVP Experiments at 13-ID-D (June 2008)
- APPENDIX C.8 GSECARS SOP for Gas Loading System Laser (October 2007)
- APPENDIX C.9 GSECARS SOP X-ray generators, Double-axis x-ray diffractometer and Laue camera in HGS-057 (April 2020)
- APPENDIX C.10 GSECARS - Safety Analysis and SOP COMPRES/GSECARS Gas Loading Apparatus (2/11/10)
- APPENDIX C.11 BioCARS SOP for Laser Controlled Areas at Bldg 400, Sector 14 (January, 2020)
- APPENDIX C.12 BioCARS Heavy Metal Soaking Solution Use, CARS Sector 14 (March 1999)
- APPENDIX C.13 BioCARS SOP for CO use at Sector 14
- APPENDIX C.14 BioCARS - SOP for Biosafety Level 2 Experiments at BioCARS Facility (November, 2014)
- APPENDIX C.15 BioCARS - SOP for Biosafety Level 3 Experiments at BioCARS Facility (November, 2014)

- APPENDIX C.16 ChemMatCARS - SOP for Lasers Controlled Area 15-ID-B, ChemMatCARS, sector 15 (December 2016)
- APPENDIX C.17 ChemMatCARS - SOP for Lasers Controlled Area 15-ID-C, ChemMatCARS, sector 15 (December 2016)
- APPENDIX C.18 ChemMatCARS - SOP for Lasers Controlled Area 15-ID-D, ChemMatCARS, sector 15 (July, 2019)
- APPENDIX C.19 GSECARS SOP for Lasers in Building 434B, Room 434 B-020 (March 2014)
- APPENDIX C.20 GSECARS SOP for Lasers at 13-BM-C (February 2018)
- APPENDIX C.21 CARS SOP for Handling Carcinogens

Argonne National Laboratory  
Advanced Photon Source  
GeoSoilEnviroCARS

**GSECARS SOP for Lasers at 13 ID-D**

(Modified April 22, 2016)

---

**Mark Rivers**

Name Printed

Signature

Date

Approved by:

ANL-E Laser Safety Officer or Deputy Laser Safety Officer:

**Bryan Broocks**

Name Printed

Signature

Date

ESH/QA Coordinator:

**Edmund Elroy Chang**

Name Printed

Signature

Date

Approved by:

Division Director:

**Bill Ruzicka**

Name Printed

Signature

Date

# GSECARS SOP for Lasers at 13 ID-D

## 1. Introduction

This is a Standard Operation Procedure to operate the lasers listed in Table 1 at the 13 ID-D station. The LCA includes the entire interior of the 13 ID-D station. It does not extend outside the station or into the 13 ID-C station.

Name of LCA supervisor: Mark Rivers

Principal laser operator: Vitali Prakapenka

**Table 1: Laser Specifications**

	Two Fiber lasers	CO2 Laser	He-Ne Laser	Green Diode Laser
Brand	IPG Photonics	Synrad	Melles Griot	Laser Quantum
Model	YLR-100-1064-WC	f201	05 LHR 991	Excel Laser
Serial number(s)	PL0703320 PL0703321	f201299040022	2933CL	810028
IHID numbers(s)	10703,10704	10636	10311	10919
Quantity	2	1	1	1
Wavelength	1064 nm	10600 nm	632 nm	532 nm
Diameter	7mm	4.5 mm	0.8 mm	1.5 mm
Divergence	0.3 mrad	4.0 mrad	1.2 mrad	<0.5 mrad
Power	100 W	200 W	30 mW	2 W
Mode	CW	CW/pulsed	CW	CW
Class	IV	IV	IIIb	IV

In addition to the lasers listed above, Class II red He-Ne or Class II green diode lasers will be used for alignment purposes.

## 2. User Training

There are two classes of users for these laser systems, Authorized Users and General Users.

The following requirements must be met in order to be added to the list of Authorized Users for these laser systems:

- Completion of the Argonne laser safety training course
- Completion of an Argonne laser eye exam
- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.
- "Hands-on" on-the-job alignment training (OJAT) by the Principal Laser Operator, who serves as the LCA trainer.

The following requirements must be met in order to be added to the list of General Users for these laser systems:

- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.

## 3. Operation modes

There are 11 laser shielding panels: panels 1-9 are in place for all operation modes and additional panels #10-11 are used only for setup mode when major optical configuration changes are required

The Class III and IV lasers can be operated in 3 modes:

1. **User mode:** The 13 ID-D station door is closed and no one is inside the LCA. The laser remote control units are outside the LCA and the lasers are operated remotely from the 13 ID-D control area. User mode operation can be performed by General Users and Authorized Users. All of the laser shielding panels #1-9 will be in their proper positions.
2. **Setup mode:** Authorized Users only are present inside the LCA. Such users will wear the appropriate eye protection for the laser in use. The laser remote control units will be inside the LCA. All of the laser shielding panels (#1-9) and additional panels #10-11 will be in their proper positions. One of the Panels #1, #2 or #9 may be open for *initial* alignment of the laser optics with class II red or green lasers and fine adjustment with class III and IV lasers.
3. **Alignment mode:** Authorized Users only are present inside the LCA. Such users will wear the appropriate eye protection for the laser in use. The laser remote control units will be inside the LCA. All of the laser shielding Panels #1-9 will be in their proper positions. One of the Panels #1, #2 or #9 may be open for *fine adjustment* of the laser optics with class II red or green lasers and fine adjustment with class III and IV lasers.

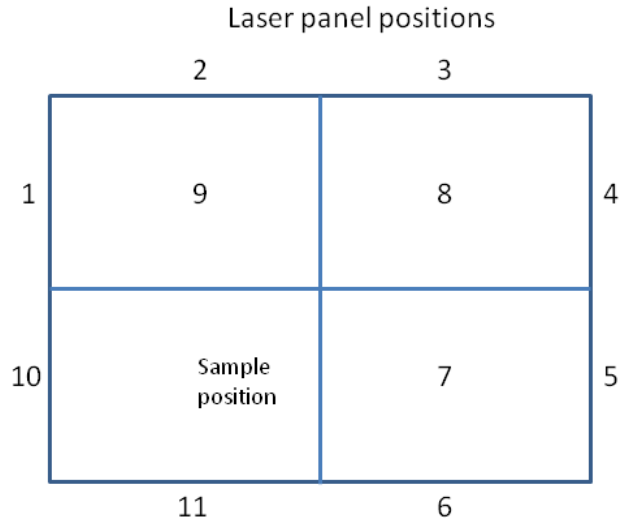
#### 4. Hazards

The IR beams from the fiber lasers and CO<sub>2</sub> laser are the main hazards because of their high power and the fact that the laser beams are invisible. There are no high-voltage hazards, because there will be no work performed on the power supplies or electrical leads with the power on.

#### 5. Controls

The following controls will be implemented in this LCA.

- For Setup Mode and Alignment Mode a laser partition from Kentek will be positioned just inside the door. It will be fastened to the upstream wall so that it cannot be moved. The downstream end of the partition will be kept close to the wall near the door, with just enough room for personnel to enter. The proper position for the partition will be marked with yellow tape on the floor. Appendix 1 is an analysis of the laser barrier protection afforded by this partition.
- A valence will be installed above the inside of the door to cover the gap above the partition.
- The circular hole between the 13 ID-C and 13 ID-D stations will be covered to prevent laser light from entering the 13 ID-C station. This cover will form a light tight seal around the beam pipe x-ray transport.
- A laser interlock system will be used to prevent unauthorized personnel from entering the LCA when the lasers are on. This interlock system is described in detail in Section 6.
- A sign will be posted on the 13 ID-D door which alerts people that they are about to enter an LCA and points out the laser warning light above.
- The keys to the laser power supplies will be kept under administrative control. When the lasers are not in use by qualified personnel the keys will be kept in a locked drawer in the LCA supervisor office.
- Appropriate eye protection will be worn by all personnel in the LCA whenever any Class IV or Class IIIb laser is operating. There are four pairs of goggles with OD of 5+ at 950-1600 nm and 10600 nm (for fiber and CO<sub>2</sub> lasers), two pair goggles with OD>6 at 450 - 550 nm (for Ar laser and for the Green Diode laser) and 4 pairs of goggles with OD 2 for operation with the He-Ne laser.
- The Class IV beam paths are almost completely enclosed. This enclosure consists of main solid panels ##1-9 around most of the perimeter of the beam path, and additional solid panels #10-11 around the regions of the beam path near the sample. . Panels #1, #2 and #9 may be open according to the alignment procedure described in section 9. The following picture shows the numbering of the panels.



## 6. Interlock System Description

The interlock system consists of the following hardware components:

- Laser Safety Panel is mounted on the upstream wall inside the LCA, and contains a Koyo PLC to implement the logic.
- A “panic” shutoff switch located in the LCA and clearly marked with a sign. This switch will turn off all class IV and IIIb lasers immediately when it is pressed. This switch will be in a fixed location so that operators can find it quickly in an emergency.
- A laser warning light installed above the hutch door outside the LCA. This light contains 3 indicators:
  - “No Hazard/Laser Off” (green)
  - “Caution/Laser Energized” (yellow)
  - “Danger/Laser On” (red)
- Laser control boxes for the CO<sub>2</sub> laser. This control box can be either inside or outside of the LCA. This box contains a number of controls, but the only two which are important for the interlock system are
  - A momentary Enable button. This line goes directly to the laser.
  - A momentary Shutter Open/Close button. This line passes through the PLC interlock system.
- Laser control boxes for the fiber lasers. These control boxes can be either inside or outside of the LCA. These boxes contain a number of controls, but the only two which are important for the interlock system are
  - A keyswitch for laser power
  - A momentary Emission Enable button. This line passes through the PLC interlock system.
- A red Danger light mounted above the door inside the LCA.
- A User Mode Enable/Disable switch and light mounted to the left of the door outside the hutch.

The interlock system has two different modes, Expert Mode, which is restricted to Authorized Users only, and User Mode, which can be used by General Users and Authorized Users.

Expert mode operates as follows:

- Pressing the Interlock button on the Laser Safety Panel does the following:
  - Lights the yellow interlock light on the Laser Safety Panel
  - Lights the yellow Caution light on the sign outside the LCA
  - Activates the photocell sensor which monitors access to the LCA through the door
  - Allows the individual lasers to be enabled

Once the interlock is turned on, the Enable buttons on the laser control boxes can be pressed to enable each laser. Pressing these buttons does the following:

- Lights the red “Laser On” light on the Laser Safety Panel
- Lights the red “Laser On” light on the sign outside the LCA
- Lights the red “Danger” sign above the door inside the LCA
- Allows the emission control or shutter control button on the laser control boxes to be activated

Pressing these buttons a second time disables the laser,.

The door photo-sensor works as follows when the beam is broken:

- System is not interlocked
  - Nothing happens
- System is interlocked and override button is NOT pressed
  - Alarm sounds
  - Interlock is tripped, and must be re-enabled at the Laser Safety Panel
  - If any laser was enabled, then the fault light illuminates and must be cleared with the button on the Laser Safety Panel
- System is interlocked and override button is pressed
  - Alarm sounds alerting operators that someone has entered LCA
  - Interlock remains active

User mode operates as follows:

- Hutch door must be closed. This is interlocked with a switch.
- Pressing User Mode Enable button does the following
  - Lights the yellow interlock light on the Laser Safety Panel (not visible to users)
  - Lights the yellow Caution light on the sign outside the LCA
  - Lights the red light above the User Mode Enable button
  - Enables all lasers in the PLC
  - Lights the red “Laser On” light on the Laser Safety Panel
  - Lights the red “Laser On” light on the sign outside the LCA
  - Lights the red “Danger” sign above the door inside the LCA
  - Allows the emission control or shutter control button on the laser control boxes to be used

To re-enter the hutch in User Mode the following should be done:

1. Press the emission control or shutter control buttons to turn off the lasers,
2. Press the User Mode Enable/Disable button again to disable the interlock. This turns off the red Danger and yellow Caution lights on the sign outside the LCA and disables the interlock.
3. Open the hutch door.

If the hutch door is opened without performing step 2) above then the interlock will be disabled, the alarm sounds, and the Fault Light illuminates. The fault must be reset on the Laser Safety Panel.

The panic button operates in all modes as follows:

- Disables the interlock, turning off all lasers
- Generates a fault condition. The fault which must be cleared at the Laser Safety Panel, and can only be cleared after resetting the panic button

## **7. Expert Mode operation procedures**

Expert Mode:

Only Authorized Users are allowed in the LCA when the interlock is enabled or when any Class III or Class IV laser is on. Only Authorized Users are permitted to operate the photocell override button.

The following checklist will be posted near the laser control units to remind operators of procedures to follow.

Before turning on any Class IIIb or Class IV laser:

- Check that the curtain is properly positioned.
- Check that the hole into 13 ID-C is covered
- Wear proper safety goggles
- Make sure that people inside the LCA are on the list of Authorized Users and are wearing proper safety goggles before activating the interlock system. (see list posted on hutch Safety Information Board)
- Only the Class II He-Ne or green lasers are to be used for initial optics alignment. The Class IIIb He-Ne laser is to be used for viewing samples in the diamond cell, not for optics alignment.

In addition, when using the Class IV lasers:

- The beam path enclosure panels are in place.

#### 8. User Mode operation procedures:

Before turning on any Class IIIb or Class IV laser:

- Check that the hole into 13 ID-C is covered
- The beam path enclosure panels are in place.

#### 9. Alignment procedures

**Panels #10 and #11 must be in place for initial alignment of laser and optics in setup mode. For fine adjustment of the co-alignment of the Class II and class III or IV lasers panels #10 and #11 may be removed.**

The following procedures are to be followed for routine alignment of the Class III or IV lasers with panels #1-#9 in place, and panels #10 and #11 in place if required per above.

- These alignment procedures are only to be done with the explicit approval of the LCA Supervisor or the Principal Laser Operator. Only Authorized Users who have also received On the Job Alignment Training (OJAT) from Vitali Prakash, who is the authorized alignment trainer for this LCA.
- Wear proper safety goggles.
- Verify the alignment of all optical components with guide laser (class-II).
- Verify that the beamstop/power meter is in the proper position after the first mirror.
- Verify that the power level is less than 5 watts.
- With the laser table shutter closed check alignment of the class-II guide lasers with class-III-IV lasers
- Use green or red class-II guide lasers co-aligned with Class III or IV lasers to check optics alignment
- Panels #1 or #2 may be open to permit alignment of the first two mirrors for the fiber lasers. When doing this the operator is to stand downstream and inboard of the mirror. The laser control unit must be positioned so that the laser can be turned on and off from this position. The alignment is performed by placing the fluorescent screen in the beam path to view the IR laser beam
- The top black cover panel (Panel #9) of the CO<sub>2</sub> laser optic box may be open to permit alignment of the two first laser mirrors. The alignment is performed by placing the special thermal image plates in the beam path to view the IR laser beam. When doing this the following conditions must be met:
  - Only one person may work above the level of the top of the CO<sub>2</sub> optics box.
  - A second person will assist operating the laser shutter. The second person will position themselves so that they are out of direct sight of the front of the first mirror.
  - They will open the laser shutter only when instructed to do so by the first operator.
  - The shutter will not be opened until the first operator has climbed the ladder and is in position above the level of the laser optics box.
  - The shutter will be closed before the first operator descends the ladder.

#### 10. Inspections and testing (forms to follow)

- The interlock system will be tested quarterly
- The laser eyewear will be inspected annually



### **11. Completion of Laser safety forms (forms to follow)**

Before laser keys are issued to an operator and the lasers are turned on, the Laser Safety Checkoff and Laser Safety Checklist forms must be completed and posted on the 13 ID-D Safety Information Board.

### **12. ANL Laser training (forms to follow)**

Two lists of laser users will be maintained and posted on the hallway door

- 1) Authorized Users, with ANL laser safety training and “On-the’Job” alignment training.
- 2) General Users, without ANL laser safety training





Laser Setup Safety Checklist *(must be completed for laser power supply key access)*  
**Sector 13 ID-D Laser Control Area (LCA)**

---

**Experimental Safety Approval Form** # \_\_\_\_\_

**Authorized Laser Users** *(list ALL individuals who will operate the laser)*

---



---

**Laser(s) to be used** (circle) :

Fiber Laser ANL 10703,10704	CO2 Laser ANL 10636	He-Ne Laser ANL 10311	Ar Laser ANL 10312	Green Diode Laser ANL 10919
--------------------------------	------------------------	--------------------------	-----------------------	--------------------------------

- Appropriate goggles for each laser to be used, are available for each laser user.
- Laser valence is installed above the inside of the hutch doors (see Photo #1).
- Laser beamhole cover over circular hole between ID-C and ID-D is in place (see Photo #2).
- Laser partition is positioned just inside the 13 ID-D hutch door (see Photo #3).
- Laser interlock system has been tested within 3 months – see Interlock Log.
- A sign is posted on the hutch door of 13 ID-D which alerts people that they are about to enter an LCA and points out the laser warning light above.
- All panels for enclosing beam path are in-place:  
 Panels #2 thru #9 will be in place when the YLF lasers are on.  
 Panels #1, #10 and #11 will be in place when the YLF laser is on *except during alignment.*

**Signatures**

Principal Laser Operator : \_\_\_\_\_ Date : \_\_\_\_\_

LCA Supervisor : \_\_\_\_\_ Date : \_\_\_\_\_





**Argonne National Laboratory  
Advanced Photon Source  
GeoSoilEnviroCARS**

**Standard Operating Procedures for  
Lasers in Building 434A, Room A020  
February 19, 2018**

---

Prepared by:  
LCA Supervisor

Mark Rivers \_\_\_\_\_  
630 252 0422 Date  
rivers@cars.uchicago.edu

Approved By:  
ANL-E Laser Safety Officer

Bryan Broocks \_\_\_\_\_  
Date

Approved By:  
APS AES Division ESH Coordinator

Edmund Elroy Chang \_\_\_\_\_  
Date

Approved by:  
APS AES Division Director

John Connolly \_\_\_\_\_  
Date

# GSECARS

## SOP for Lasers in Laboratory A020, Bldg 434A

### 1. Introduction

This is a Standard Operation Procedure to operate the lasers listed in Tables 1 and 2 in lab A020. There is a laser curtain from Kentek installed to partition the lab into an LCA on one half, and an electronics setup space on the other half. The partition curtain is Velcro'ed at top and bottom and a safety interlocked switch connects the two sides of the curtain in the middle. The door to the hallway and the rollup door to the experimental floor are interlocked, and the window of the door is covered. Both laser setups are enclosed in laser enclosures, enclosure #1 for the Raman system lasers and enclosure #2 for the laser ultrasonics probe, pump and fiber lasers.

Name of LCA supervisor: Mark Rivers

Principal laser operator: Vitali Prakapenka

**Table 1: Laser Specifications for Raman system (enclosure #1)**

Brand	Coherent	Coherent	Laser Quantum	Laser Quantum	Crysta Laser	IPG Photonics	Leukos
Model	MDB-266	Verdi V2	Ventus 473	Ventus 660	CL-946-200-S0	YLR-SM-100-1064-LP	CARS-SM-30
Serial number	GDP. 1220878. 0109	4151814 10 FNB	6548016	6547920	0012440	PL1520974	201605-279
ANL-E IHID	11085	11086	11087	11088	11090	11089	11142
Wavelength	266 nm	532 nm	473 nm	660 nm	946 nm	1064 nm	410 to 2400 nm
Diameter	0.8 mm	2.25 mm	1.50 mm	1.50 mm	2.0 mm	5 mm	1.6 mm
Divergence	0.55 mrad	0.5 mrad	0.6 mrad	0.6 mrad	0.6 mrad	0.3 mrad	0.8 mrad
Power	200 mW	2.0W	350 mW	350 mW	200 mW	100 W	100 mW
Mode	CW	CW	CW	CW	CW	CW	Pulsed
Class	IV	IV	IIIb	IIIb	IIIb	IV	IIIb



**Table 2: Laser Specifications for Laser Ultrasonics system (enclosure #2)**

	<b>Probe laser</b>	<b>Pump laser</b>	<b>Fiber Laser</b>
Brand	Coherent	Teem Photonics	IPG Photonics
Model	Compass 315M	PNP-M08010-100	YLR-SM-100-AC-Y11
Serial number(s)	LDP1100441138041	GR1102206	PL1110932
ANL-E IHID	11006	11007	11008
Wavelength	532 nm	1064 nm	1070 nm
Diameter	0.32 mm	0.3 mm	5 mm
Divergence	2.2 mrad	2 mrad	0.3 mrad
Power	150 mW	80 microJ, 1 kHz	100 W
Mode	CW	pulsed, 500 ps	CW
Class	IIIb	IV	IV

In addition to the lasers listed above, Class II He-Ne or solid state lasers will be used for alignment purposes.

## 2. Hazards

The laser beams are the main hazard. The fiber and pump lasers are particular hazards because of their high power and the fact that they are invisible. There is no high-voltage hazard, because there will be no work performed on the power supply or electrical leads with the power on.

## 3. Controls

The following controls are implemented in this LCA.

- A laser interlock system is used to prevent operation of the lasers in an unsafe condition. This interlock system is described in detail in Section 4.
- Interlocked enclosure #1 completely encloses the Raman system laser beam paths during normal operation. The table top enclosure consists of black sliding doors and top over an aluminum frame. A lighted sign above the enclosure indicates the presence of laser light inside. When the laser shutters are open, all doors must remain closed, the only exception being for alignment of the optics. Such alignment can only be performed by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. **Users without ANL Laser Safety Training, eye exam, and On-the Job Alignment Training may not operate the lasers with the enclosure open.**
- Interlocked enclosure #2 completely encloses laser ultrasonics system laser beam

paths during normal operation. The table top enclosure consists of black sliding doors and top over an aluminum frame. A lighted sign above the enclosure indicates the presence of laser light inside. When the laser light is present, all doors must remain closed, the only exception being for alignment of the optics. Such alignment can only be performed by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. **Users without ANL Laser Safety Training, eye exam, and On-the Job Alignment Training may not operate the probe, pump, or fiber lasers with the enclosure open.**

- A laser curtain from Kentek is installed to make a LCA on one side of the room. A lighted laser warning sign is installed on the non-LCA side of the curtain. The curtain has an interlock to indicate closed.
- A lighted laser warning sign is installed above enclosure #1 at eye level. A lighted laser warning sign is installed near enclosure #2. Lighted laser warning signs are also mounted to the left of the door to the hallway and to the left of the rollup door to the experimental floor. The meaning of these signs is explained in Section 4.
- The keys to the laser power supplies are kept under administrative control. When the lasers are not in use by qualified personnel the keys are kept in a locked drawer in the LCA Supervisor’s office.
- Appropriate eye protection will be worn by all personnel in the LCA whenever the lasers are operating in the Expert Mode. There are at least two pairs of goggles with the following specifications:

Kentek KGG-303F	Wavelength	200-535	536-540	541-550		
	Optical density	6	5+	2-4		
Kentek KGG-017F	Wavelength	200-280	850-5200	945-2300	1011-1500	2800-10600
	Optical density	7+	3+	5+	7+	5+
Kentek KSGG-6108G	Wavelength	190-400	625-647	630-644		
	Optical density	5+	5+	6+		
Kentek KSGG-8804G	Wavelength	190-400	645-670nm			
	Optical density	5+	6+			

#### 4. Interlock System Description

##### Hardware components

The interlock system consists of the following hardware components:

- Laser Safety Interlock Panel mounted on the wall to the right of the hallway door as entering the LCA. The interlock system is based on a programmable logic controller from PLC Direct. There is a second control panel to the left of the door. The

interlock system can also be operated from the computer with the EPICS control software.

- Laser warning lights installed to the left of the hallway door, to the left of the rollup door outside the LCA, above each interlocked enclosure, and on the outside the laser curtain in the electronics lab.
- Interlock switches on the hallway door, roll-up door and laser curtain.
- Interlocked enclosure #1 that completely encloses the Raman system lasers in this SOP.
- Interlocked enclosure #2 that completely encloses the pump, probe, and fiber lasers in this SOP.
- Emergency Stop button placed close to the laser enclosures.

**Laser interlocks**

The interlock system is designed to handle up to 10 lasers. The following table lists these laser and the interlock mechanism used for each.

Laser	Enclosure	Interlock mechanism
Coherent MDB-266	#1	shutter
Coherent Verdi V2	#1	shutter
Laser Quantum Ventus 473	#1	shutter
Laser Quantum Ventus 660	#1	shutter
Crysta Laser, CL-946-200-S0	#1	shutter
IPG Photonics, YLR-SM-100-1064-LP	#1	Laser emission
Leukos CARS-SM-30	#1	Laser emission
Coherent, Compass 315M	#2	Laser emission
Teem Photonics, PNP-M08010-100	#2	Laser emission
<b>Fiber Laser, IPG Photonics</b>	#2	Laser emission

Note: All of the lasers in Enclosure #1 are collectively referred to as “Laser 1” because they have a common emission and shutter interlock signal. These lasers cannot be individually enabled and disabled.

**Green “safe” lamps**

- When all doors (rollup door, hallway door, and laser curtain) are closed, the green Doors Closed lamp is lit
- When enclosure #1 is closed the green Enclosure Closed #1 lamp is lit
- When enclosure #2 is closed the green Enclosure Closed #2 lamp is lit
- When the laser 1 shutter is closed the green Laser 1 Shutter Closed lamp is lit

**“Expert mode” switch**

This is a keyed switch to enable Expert Mode. In Expert Mode exposed beams may be present in the LCA, and operation is only permitted by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. This key will be under the control of the principal laser user.

### **Enable request buttons**

- For the Raman table the Laser 1 Enable button enables the laser emission on the IPG IR laser and the Leukos continuum laser. This can only be enabled if enclosure #1 is closed, or the system is in Expert Mode.
- For the Raman table the Shutter 1 Enable request button enables the shutters for all of the other lasers (Coherent, Laser Quantum, Crysta Laser). This can only be enabled if enclosure #1 is closed, or the system is in Expert Mode. If it is safe to enable the shutter, then it will be enabled and the corresponding yellow Laser 1 Shutter Enabled lamp will be lit.
- The Lasers 2&3 Enable request button enables the pump and probe lasers. These share a common Laser Enable request button and Laser Enable lamp because they are always used together and there is a shortage of I/O on the PLC. These can only be enabled if enclosure #2 is closed, or the system is in Expert Mode. If it is safe to enable the lasers, then they will be enabled and the corresponding yellow Laser 2&3 Enabled lamp will be lit.
- The Laser 4 Enable request button enables the fiber laser on the ultrasonics table. This can only be enabled if enclosure #2 is closed, or the system is in Expert Mode. If it is safe to enable the laser, then it will be enabled and the corresponding yellow Laser 4 Enabled lamp will be lit.

The Enable buttons have a toggle action, i.e. pressing the enable button a second time will disable the corresponding laser or shutter.

### **Exposed Beam**

The following conditions can result in exposed beams in the LCA  
Laser 1 enabled, and laser 1 shutter enabled, and enclosure #1 open

OR

Lasers 2&3 enabled, and enclosure #2 open

OR

Laser 4 enabled, and enclosure #2 open

If any of these conditions are satisfied then the red Exposed Beam lamp on the interlock panel will be lit, and the red Danger – Beams Accessible Lamp on the signs outside the LCA will be illuminated. Exposed beam mode is only permitted by the interlock system when the Expert Mode key switch is enabled.

### **Fault Condition**

The following will result in a fault condition:

- Exposed Beam and key switch not in Expert Mode
- Exposed Beam and any doors or curtain open

A fault condition results in:

- An audible alarm
- The red Fault lamp will be lit
- Disabling of laser 1 emission
- Disabling of laser 1 shutter
- Disabling of laser 2 emission

- Disabling of laser 3 emission
- Disabling of laser 4 emission

To clear the Fault Condition it is necessary to press the Reset Fault button.

### **Panic Buttons**

There are two panic buttons available to immediately disable all lasers and shutters. The first button is on the Laser Interlock Panel. The second button is a red remote Emergency Stop button placed close to the laser enclosure. Pressing either button will immediately remove all laser and shutter enables and light the red Panic Stop lamp on the interlock panel. To remove the Panic Stop condition it is necessary to press the Panic Stop button on the interlock panel, toggling the Panic Stop state.

### **Laser Warning Signs**

- If no laser is enabled then the green No Hazard signs outside the LCA will be illuminated.
- If any laser is enabled then the yellow Caution signs outside the LCA will be illuminated.
- If there is an exposed beam condition inside the LCA then the red Danger – Beams Accessible Lamp signs outside the LCA will be illuminated.
- If there is laser light present inside an enclosure then the warning sign above that enclosure will be illuminated. This is defined as laser 1 emission enabled or shutter open, or laser 2-4 enabled.

### **Personnel Entrance**

- If the green No Hazard sign is illuminated then anyone may enter the LCA
- If the yellow Caution sign is illuminated then anyone may enter the LCA, after knocking to make sure that any personnel inside the LCA are not about to make a change to Expert Mode.
- If the red Danger – Beams Accessible sign is illuminated then no one may enter the LCA. Opening the door under this condition will generate a Fault Condition.

## **5. Operation procedures**

There are 2 allowed modes of operation for all lasers. GSECARS safety training is required for both modes. ANL laser safety training and eye exam is required for Expert Mode. Safety goggles are required for operating in Expert Mode.

### **1) Normal operations (User Mode)**

- Enclosure is closed.
- The laser may be operated at full power.

### **2) Optics alignment (Expert Mode)**

- The enclosure doors may be opened.
- The lasers will be run at minimum power (<5mW)
- Enclosure doors will be closed immediately after the optics alignment is complete.

Only authorized users who have received GSECARS training, read and signed this SOP are permitted to operate any of the lasers in this LCA in either mode. If such personnel have not completed ANL laser safety training, an eye exam and On-the Job Alignment Training then they are not permitted to operate the lasers in the Expert Mode.

The following checklist will be posted near the laser control units to remind operators of procedures to follow.

### **User Mode**

- Check that the curtain is properly positioned and safety interlock is closed.
- Check that the door to the hallway and the roll-up door to the experiment hall floor are closed and the green Doors Closed lamp on the laser interlock panel is lit.
- Make sure that people inside the LCA are on one of the two lists of authorized personnel (with or without ANL laser safety training) before enabling any lasers (see list posted in the hallway)
- Check that the enclosure is closed and the green Enclosure Closed lamp on the laser interlock panel is lit.

### **Expert Mode**

- Check that the curtain is properly positioned and safety interlock is closed.
- Check that the door to the hallway and the roll-up door to the experiment hall floor are closed and the green Doors Closed lamp on the laser interlock panel is lit.
- Wear proper safety goggles.
- Make sure that people inside the LCA are on the list of authorized personnel with ANL laser safety training and are wearing proper safety goggles before activating the interlock system. (see list posted in the hallway)
- For initial optics alignment, make sure the laser is run at low power (<20 mW).

All laser control units must be positioned on the optical table, outside the enclosure, so the laser can be turned on and off from the operator's position.

All viewing of the sample when it is illuminated with the laser beam will be done with the video camera system, not with direct observation.

## **6. Alignment Procedures**

- These alignment procedures are only to be done with the explicit approval of the LCA Supervisor or the Principal Laser Operator.
- Wear proper safety goggles.
- A Class II He:Ne or solid state alignment lasers will be used for preliminary alignment of most of the optical path. Coalignment of the He:Ne and class III-VI

lasers will be performed using only minimum power (<5 mW) of lasers. The path of the IR pump or fiber laser is to be determined with the use of the near-IR laser alignment sheets. After coalignment of the He:Ne and IR lasers, the He:Ne alignment laser must be used to align the rest of the beam path.

- Following optical alignment using the He:Ne laser, it is expected that small (~0.1 mm) adjustments of the pump or fiber laser beam at the sample position will be necessary due to dispersion in the focusing elements of the optical system. These adjustments are to be made using the minimum laser power necessary (< 5 mW). For this adjustment the minimum number of panels possible will be opened on the enclosure.
- The optical design for all lasers includes no vertical beam paths.

### **7. Inspections and testing (forms to follow)**

- The interlock system will be tested quarterly
- The laser eyewear will be inspected annually

The interlock system tests will consist of the following. This test will be performed with laser 1 power off, since the interlock is on the shutter not the power, and this will minimize the risk to testing personnel. Set power on lasers 2-4 to minimum possible value.

- With all lasers disabled verify that laser sign in hallway is green.
- Check that opening each enclosure panel results in Enclosure Closed light turning off.
- Disable Expert Mode with key switch
- Close both laser enclosures. Enable laser 1, enable laser 1 shutter. Open laser 1 shutter.
- Verify that laser light above enclosure #1 is lit. Verify that laser sign in hallway is yellow.
- Open enclosure #1 door slightly. Verify that a Fault Condition occurs, that laser 1 shutter enable is removed, and that the shutter is physically closed.
- Enable lasers 2-4. Open enclosure #2 door slightly. Verify that a Fault Condition occurs, that laser 2-4 enable is removed, and that the emission of 2-4 lasers is off.
- Re-enable laser 1, press panic button, verify that shutter enable is removed.
- Enable Expert Mode with key switch.
- Verify that Expert Mode light on interlock panel is lit.
- Close enclosure #1, reset fault, enable laser 1 emission, enable laser 1 shutters. Open laser 1 shutter.
- Open enclosure #1 door slightly.
- Verify that Exposed Beam light on interlock panel is lit.
- Have a second person verify that red and yellow lights on laser sign in hallway are lit.
- Open door to LCA, verify that this results in a Fault Condition.
- Reset fault, enable laser 2-4.
- Open enclosure #2 door slightly.

- Verify that Exposed Beam light on interlock panel is lit.
- Have a second person verify that red and yellow lights on laser sign in hallway are lit.
- Open door to LCA, verify that this results in a Fault Condition.
- Re-enable laser and repeat above step while opening roll-up door and laser curtain to verify that opening them also results in a fault condition.

### **8. Laser training (forms to follow)**

Two lists of laser users will be maintained and posted on the hallway door

- 1) Users With ANL Laser Safety Training and On-the Job Alignment Training.
- 2) Users Without ANL Laser Safety Training





**Laser eyewear inspection**

**(To be completed annually)**

<b>Date</b>	<b>Name</b>	<b>Signature</b>





## Appendix C.3 GSECARS SOP for Lasers at 13 BM-D (1/31/14)

### GSECARS SOP for Lasers at 13 BM-D (1/31/14)

#### 1. Introduction

This is a Standard Operation Procedure to operate the lasers listed in Table 1 at the 13 BM-D station. The laser controlled area (LCA) includes the entire interior of the 13 BM-D station. It does not extend outside the station or into the 13 ID-B station.

Name of LCA supervisor: Mark Rivers

Principal laser operator: Vitali Prakapenka

**Table 1: Laser Specifications**

	Coherent diode laser
Brand	Coherent
Model	Verdi – 2W
Serial number(s)	19848407
IHID#	10635
Quantity	1
Wavelength	532 nm
Diameter	2.2 mm
Divergence	0.5 mrad
Max. Power	2 W
Mode	CW
Class	IV

In addition to the laser listed above, Class II lasers will be used for alignment purposes.

In the description of the interlock system in this document we refer to the laser listed in Table 1 as laser 1. The interlock system has been built to handle two additional lasers (laser 2 and laser 3) in future. Lasers 2 and 3 do not currently exist.

#### 2. User Training

There are two classes of users for the laser system, Authorized Users and General Users.

The following requirements must be met in order to be added to the list of Authorized Users for the laser system:

- Completion of the Argonne laser safety training course
- Completion of an Argonne laser eye exam
- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.
- On-the-Job Laser Alignment training by the Principal Laser Operator.

The following requirements must be met in order to be added to the list of General Users for these laser systems:

- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.

#### 3. Operation modes

The class IV laser can be operated in 2 modes:

1. **User mode:** The 13 BM-D station door is closed and no one is inside the LCA. The laser shutter is remotely controlled by a computer interface from the 13 BM-D control area. User mode operation can be performed by General Users and Authorized Users,.
2. **Alignment mode:** Authorized Users only are present inside the LCA. Such users will wear the appropriate eye protection for the laser in use.. Laser shielding panels may be removed for alignment of the laser optics.

#### 4. Hazards

The green laser beam is the main hazard because of its high power. There is no high-voltage hazard, because there will be no work performed on the power supply or electrical leads with the power on.

#### 5. Controls

The following controls will be implemented in this LCA.

- For Alignment Mode a laser curtain from Kentek will be positioned just inside the door.
- A laser interlock system will be used to prevent personnel exposure to the laser beam. This interlock system is described in detail in Section 6.
- A laser warning sign, as supplied by the ANL LSO, will be posted on the 13 BM-D door which alerts people that they are about to enter an LCA and points out the laser warning light above.
- The keys to the laser power supplies and for Expert mode on the interlock system will be kept under administrative control. When the lasers are not in use by qualified personnel the keys will be kept in a locked drawer in the LCA supervisor office.
- Appropriate eye protection will be worn by all personnel in the LCA whenever exposed beams could be present. There are two pairs of laser protective eyewear with OD>4 at 450 - 550 nm (for Ar laser).
- The laser beam paths are almost completely enclosed. This enclosure consists of solid panels around most of the perimeter of the beam path. These solid panels are numbered 1, 3, 5, 7, 9, 10, 13, 14, 15, 16..(Note: panels 2, 4, 6, 8, 11 and 12 are no longer needed since an earlier SetupMode is no longer used). The panels are interlocked to ensure they are in place with an interlock cable. All panels will always be in place when the laser is on *except during alignment*. During alignment, the panels may be removed to allow the operator access to the optics which steers the laser beam

#### 6. Interlock System Description

The interlock system has two different modes, Expert Mode, which is restricted to Authorized Users only, and User Mode, which can be used by General Users and Authorized Users.

##### Interlock System Components

The interlock system consists of the following hardware components:

- Laser Safety Interlock Panel mounted on the upstream wall inside the LCA. The interlock system is based on a programmable logic controller from PLC Direct.
- A “panic” shutoff switch located in the LCA and clearly marked with a sign. This switch will be in a fixed location so that operators can find it quickly in an emergency. This switch functions as follows: if the panels are interlocked in place, it closes the laser shutter, since that is sufficient to guarantee that there are no exposed beams. If the panels are not interlocked in place (alignment mode) then the laser power is disabled.
- A laser warning light installed above the hutch door outside the LCA. This light contains 3 indicators:
  - “No Hazard/Laser Off” (green)
  - “Caution/Laser Eenergized” (yellow)
  - “Danger/Beams Accessible” (red)
- A red Danger light mounted above the door inside the LCA. The light is On when the laser is energized.

- The door photo-sensor works as follows when the beam is broken:
  - System is not interlocked
    - Nothing happens
  - System is interlocked and override button is NOT pressed
    - Alarm sounds
    - A Fault condition is set and must be reset at the Laser Safety Panel
    - If any laser was enabled, then the fault light illuminates and must be cleared with the button on the Laser Safety Panel
  - System is interlocked and override button is pressed
    - Alarm sounds alerting operators that someone has entered LCA
    - The Fault condition is not set.
- A User Mode Enable/Disable press button and light mounted to the left of the door outside the hutch.
- Enclosures panels that are interlocked to ensure they are in place.

#### Enable States:

There are 4 enable buttons and indicators.

- Laser 1 enable. This is a push button and yellow light on the laser interlock panel. Pressing the button once enables the laser 1 power (if the interlock conditions allow it). Pressing it a second time disables the laser power.
- Laser 1 shutter enable. This is a push button and yellow light on the laser interlock panel. Pressing the button once enables the laser 1 shutter (if the interlock state allows it). Pressing it a second time disables the laser shutter.
- Laser 2 enable. This button is for future use, when a second laser may be added to the system.
- Laser 3 enable. This button is for future use, when a third laser may be added to the system.

#### Exposed Beam:

The following conditions can lead to exposed beam in the LCA.

- Panels not interlocked in place
- or
- Laser shutter open

Exposed beams are only permitted by the interlock system if:

- The key is in Expert mode
- or
- The hutch door is closed, ensuring that no personnel are inside the LCA.

“Expert mode” switch

There is a keyed switch to enable “expert mode”. In expert mode exposed beams may be present with personnel in the LCA, and operation is only permitted by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. This key will be under the control of the principal laser user. This mode is to be used for alignment only.

#### Fault Condition

The following will result in a fault condition:

- Exposed Beam in LCA, hutch door open, and key switch not in Expert Mode
- Exposed Beam in LCA and the door photo sensor blocked
- Laser shutter commanded to close, but not closed within 0.5 second.

A fault condition results in:

- An audible alarm
- The red Fault lamp will be lit
- Disabling of the laser power and laser shutter.

To clear the Fault Condition it is necessary to press the Reset Fault button.

#### Panic Button

The panic button operates in both User and Expert modes as follows:

- Disables the interlock, turning off laser

- Generates a fault condition. The fault which must be cleared at the Laser Safety Panel, and can only be cleared after resetting the panic button

## 7. Operation procedures

### Expert Mode:

Important! Only Authorized Users are allowed in the LCA when the interlock is enabled. Only Authorized Users are permitted to operate the photocell override button.

The following checklist will be posted near the laser control unit to remind operators of procedures to follow. Before turning on the laser:

- Check that the access door between BM-D and ID-B is closed and padlocked with the APS Radiation Safety Tag 13-BM-D-X-01 is in place.
- Check that the curtain is properly positioned.
- Wear the specified laser protective eyewear
- Make sure that people inside the LCA are on the list of Authorized Users and are wearing proper safety goggles before activating the interlock system. (see the list posted on hutch Safety Information Board)
- Only the Class II lasers are to be used for initial optics alignment.

Expert mode operates as follows:

- Turning the “expert mode” key switch On allows expert mode operation
- The Laser 1 Enable button on the control interface can be pressed to enable the laser. Pressing the enable button does the following:
  - Lights the red “Exposed Beams” light on the Laser Safety Panel
  - Lights the yellow “Caution/Laser Energized” light on the sign outside the LCA
  - Lights the red “Danger/Exposed Beams” light on the sign outside the LCA
  - Lights the red “Danger” sign above the door inside the LCA
  - Enables the laser (L2 or L3) or laser shutter (L1)
- The Laser 1 Shutter Enable button on the control interface can be pressed to enable the laser shutter. Pressing the enable button does the following:
  - Lights Enables the laser 1 shutter
- To finish the “Expert Mode” operation,
  - press the enable button a second time disabling the laser
  - Turn the expert mode key to the Off position.

### User Mode:

Important! Only Qualified Users (General Users or Authorized Users, check section 2 for User Training requirements) are allowed to operate the laser system.

Before turning on the laser, check

- Check that the access door between BM-D and ID-B is closed and padlocked with the APS Radiation Safety Tag 13-BM-D-X-01 is in place.
- The beam path enclosure panels are in place and interlocked with the cable.

User mode operates as follows:

- Enable laser 1 on the interlock panel.
- Exit the hutch and close the door. This is interlocked with a switch.
- Pressing User Mode Enable button outside the hutch does the following
  - Enables the laser 1 shutter
  - Lights the yellow laser shutter enable light on the Laser Safety Panel (not visible to users)
  - Lights the orange light above the User Mode Enable button
- Once the laser 1 shutter is enabled, the EPICS control system is used to open the laser 1 shutter. Opening the shutter does the following:



- Lights the red “Exposed Beams” light on the sign outside the LCA
- Lights the red “Danger” sign above the door inside the LCA
- To re-enter the hutch in User Mode the following should be done:
  - Use the EPICS control system to close the laser 1 shutter. This turns off the red “Exposed beams” light on the sign outside the LCA
  - Press the User Mode Enable/Disable button again to disable the interlock. This turns off the orange light above the button and disables the laser 1 shutter.
  - Open the hutch door.
  - If the hutch door is opened without pressing the User Mode Enable/Disable button, then the laser shutter will be disabled, closing the laser shutter. This does not generate a fault, because although it is not the recommended procedure it is safe, and we do not want to shut off the laser power unnecessarily.

## **8. Alignment procedures**

The following procedures are to be followed when aligning the laser with panels removed.

- These alignment procedures are only to be done with the explicit approval of the LCA Supervisor or the Principal Laser Operator.
- Wear proper safety goggles all the time.
- Verify that a beamstop or power meter is in the proper position after the first mirror.
- Verify that the power level is less than 1 miliwatts.
- Panels may be removed to permit alignment of the first mirror. The laser control unit must be positioned so that the laser can be turned on and off from this position.
  - The rest optics after the first mirror are aligned with the help of a Class-II green diode laser.

## **9. Inspections and testing (forms to follow)**

- The interlock system will be tested quarterly
- The laser eyewear will be inspected annually

## **10. Completion of Laser safety forms (forms to follow)**

Before laser keys are issued to an operator and the lasers are turned on, the Laser Safety Checkoff and Laser Safety Checklist forms must be completed and posted on the 13 BM-D Safety Information Board.

## **11. ANL Laser training (forms to follow)**

Two lists of laser users will be maintained and posted on the Safety Information Board

- Authorized Users, with ANL laser safety training, eye exam, and GSECARS operation training. In addition, Authorized Users must have completed On-the-Job laser alignment training for this LCA, as certified by the required ANL-962 form with all required signatures.
- General Users, without ANL laser safety training, but with GSECARS operation training







Laser Setup Safety Checklist *(must be completed for laser power supply key access)*

**Sector 13 BM-D Laser Control Area (LCA)**

---

---

Experimental Safety Approval Form # \_\_\_\_\_

**Authorized Laser Users** *(list ALL individuals who will operate the laser)*

\_\_\_\_\_

\_\_\_\_\_

Laser(s) to be used *(circle)* :

Class IV Verdi-Diode

ANL 10635

- Appropriate protective eyewear for each laser to be used, are available for each laser user.
- Laser curtain is positioned just inside the 13 BM-D hutch door.
- Laser interlock system has been tested within 3 months – see Interlock Log.
- A sign is posted on the hutch door of 13 BM-D which alerts people that they are about to enter an LCA and points out the laser warning light above.
- All panels for enclosing beam path are in-place:  
Panels 1, 3, 5, 7, 9, 10, 13, 14, 15, 16 will be in place when the laser is on *except during alignment*.

**Signatures**

Principal Laser Operator : \_\_\_\_\_ Date : \_\_\_\_\_

LCA Supervisor : \_\_\_\_\_ Date : \_\_\_\_\_





**Safety Analysis and  
Standard Operating Procedures**

**DDIA-30 Apparatus**

**Mar., 2011**

**Yanbin Wang**



# Contents

<b>A</b>	<b>SYSTEM OVERVIEW .....</b>	<b>3</b>
A.1	PURPOSE	3
A.2	OTHER SYSTEMS	3
A.3	SYSTEM COMPONENTS	3
A.3.1	<i>The DDIA-30 module .....</i>	<i>3</i>
A.3.2	<i>The 1000 ton hydraulic press and main ram control .....</i>	<i>3</i>
A.3.3	<i>Module transport table.....</i>	<i>4</i>
A.3.4	<i>Differential ram hydraulic controllers.....</i>	<i>4</i>
A.3.5	<i>Hydraulic lines from roof-top to DDIA-30 .....</i>	<i>4</i>
A.3.6	<i>Pressure gauges.....</i>	<i>4</i>
A.3.7	<i>Displacement limit switches.....</i>	<i>4</i>
A.3.8	<i>Pressure switches.....</i>	<i>4</i>
A.3.9	<i>Burst disks.....</i>	<i>4</i>
A.3.10	<i>Pressure transducers.....</i>	<i>5</i>
A.3.11	<i>Spill-capturing plates .....</i>	<i>5</i>
<b>B</b>	<b>SAFETY FEATURES .....</b>	<b>10</b>
B.1	THE DDIA-30 MODULE	10
B.1.1	<i>Burst disk to prevent overload .....</i>	<i>10</i>
B.1.2	<i>Front shield .....</i>	<i>10</i>
B.2	THE TRANSPORT TABLE	10
B.2.1	<i>Safety stopping pins.....</i>	<i>10</i>
B.2.2	<i>Locking sliders.....</i>	<i>10</i>
B.3	THE DIFFERENTIAL RAMS	10
B.3.1	<i>Stress analysis and safety factor.....</i>	<i>10</i>
B.3.2	<i>Minimized physical access .....</i>	<i>11</i>
B.3.3	<i>Over-pressure prevention – pressure switch and displacement limitation .....</i>	<i>11</i>
B.3.4	<i>Spillage prevention – fluid containers under the controllers .....</i>	<i>11</i>
B.3.5	<i>Pinch points.....</i>	<i>11</i>
<b>C</b>	<b>STORED ENERGY ANALYSIS.....</b>	<b>12</b>
C.1	STORED ENERGY – MAIN RAM WITH DDIA-30	12
C.2	STORED ENERGY – DIFFERENTIAL PUMPS	12
<b>D</b>	<b>STANDARD OPERATING PROCEDURES.....</b>	<b>13</b>
D.1	AUTHORIZED PERSONNEL	13
D.2	PERSONNEL PROTECTIVE EQUIPMENT	13
D.3	RESTRICTING ACCESS AND POSTING	13
D.4	EMERGENCY PROCEDURES	13
D.5	LOADING A SAMPLE IN THE DDIA-30	13
D.6	DDIA-30 TRANSPORT	13
<b>E</b>	<b>INSPECTION AND MAINTENANCE .....</b>	<b>14</b>
<b>F</b>	<b>APPENDICES.....</b>	<b>15</b>
F.1	LOAD TEST RESULTS FOR DDIA-30	15
F.2	OH!SAWA DIFFERENTIAL RAM CONTROL SYSTEM DRAWINGS	16
F.3	OH!SAWA HYDRAULIC CONTROL SYSTEM COMPONENT SAFETY DOCUMENT	17
F.4	STRESS CALCULATION FOR THE 200 MPa DIFFERENTIAL PUMPS	26
F.5	AUTHORIZED DDIA-30 OPERATORS FOR APRIL 2010 TEST	27

## **A System Overview**

### **A.1 Purpose**

The DDIA-30 module is a new multi-anvil device for two general purposes: (1) to conduct high pressure experiments using the double-stage configuration with sintered diamond as second-stage (inner) anvils to generate pressures approach 100 GPa and (2) to conduct deformation experiments as a single stage for large sample volumes approaching 1 cm<sup>3</sup>. The module consists of two guide blocks, each with a built-in differential hydraulic ram to generate differential stress fields on samples within a cube-shaped pressure medium. The DDIA-30 is a community sponsored project. Funding was provided jointly by COMPRES (Consortium for Materials Properties Research in Earth Sciences) and GSECARS.

The DDIA-30 module was built by Rockland Research (West Nyack, New York), the same company that manufactured the 1000 ton press in 13-ID-D and the T-25 high pressure module (the entire system has been operational since 1999). Pressure control systems for the two differential rams were manufactured by a company called Oh!sawa Systems in Japan. The DDIA-30 system will be installed and operated at 13-ID-D in the existing 1000 ton hydraulic press. All of the high-pressure components are commercially purchased.

### **A.2 Other systems**

There are two similar modules installed in Japan, one at the Magma Factory of Tokyo Institute of Technology, the other at Geodynamics Research Center of Ehime University. In both cases the control systems for the differential systems were provided by Oh!sawa Systems. This is the reason we imported identical systems from Oh!sawa Systems. The DDIA-30 is the only such system in the US that can compete with the Japanese high pressure communities for ~100 GPa pressure generation on 0.5 mm<sup>3</sup> samples. This is also the only such system currently operating at a synchrotron source.

### **A.3 System components**

The system layout is shown in Figure 1. This design incorporates the following features.

#### **A.3.1 The DDIA-30 module**

Two guide blocks with six first-stage anvils. Each guide block is made of a single piece of high-toughness hardened steel, with four 45 degree flat surfaces (polished) and a built-in hydraulic ram. The upper and lower anvils are fastened direction on the guide blocks and facing each other. The four side anvils slide on 45 degree surfaces when the two guide blocks are compressed (see Fig. 1 for details).

The DDIA-30 was tested at Rockland Research Corp., after manufacture. Throughout the tests, Yanbin Wang was present and participated in the tests. The module was compressed to 9 MN in a 1000 ton press that is identical to the one we use at 13-ID-D, held overnight, with dial indicators monitoring the outer dimensions of the module. All displacements recorded by the indicators showed linear relation with applied load, and all measured displacements returned to zero when the load was removed. No plastic deformation was detected at the full load. Similar tests were repeated 4 times and the results were consistent. Then the module was loaded to 11 MN, ~22% over the design load, the module behaved the same. Then at a main ram load of 0.6 MN, pressures in the differential rams were then increase to 0.4 MN. Displacement measurements showed similar behavior. Appendix F.1 shows one set of the test results.

#### **A.3.2 The 1000 ton hydraulic press and main ram control**

At 13-ID-D, the DDIA-30 module will be compressed in the existing press frame in 13-ID-D, by the 1000 ton hydraulic ram (hereafter referred to as the main ram) acting upwards. This system has been in operation since 1999 (Fig. 2).

### **A.3.3 Module transport table**

A new transport table has been installed and tested to handle heavy modules like DDIA-30. This table consists of three heavy-duty rotary stages, connected with rails, safety stopping pins, and locks. Two rotary stages are located at the ends of the table, the third is in the middle of the table and can be connected to the rails in the press frame (Fig. 3). All locks and safety pins are inter-locked, so that only when a lock is unlocked can the module be allowed to move in the unlocked direction. Once the module is transported to the middle rotary stage, it is rotated 90 degrees and locked to the rails in the press frame. This allows a safe and ergonomic way to transport the module into the press.

### **A.3.4 Differential ram hydraulic controllers**

The differential hydraulic rams (D-rams) inside the DDIA-30 guide blocks are controlled by two independent pressure controllers mounted on the roof top of 13-ID-D (Fig. 4). Each controller consists of a 200 MPa hydraulic cylinder, mounted in a four-post press frame, with a motorized screw jack to control the speed of cylinder. The two complete systems are manufactured by Oh!sawa Systems. Engineering drawings and company provided safety documents are given in Appendices F.12 and F.3, respectively. Each hydraulic ram contains a maximum of 220 cc hydraulic fluid. At 200 MPa, the maximum volume reduction is 20%. The design and manufacturing processes were following the Japanese Industrial Standards (JIS), which are compatible with ISO9000 and ISO14000. See Japanese Industrial Standard Committee website at <http://www.jisc.go.jp/eng/jis-act/index.html>.

### **A.3.5 Hydraulic lines from roof-top to DDIA-30**

Standard high pressure hydraulic lines (tubing inner diameter of 1/16") have been installed to connect the differential ram controllers on the roof top to the DDIA-30 module inside 13-ID-D. These lines are commercial components from High Pressure Equipment Company, rated at 45000 psi (or approximately 300 MPa).

The differential pumps are capable of reaching 200 MPa (29000 psi), but we have set the Oh!sawa pressure switches to a maximum working oil pressure of 10,000 psi, and if the pressure switches fail, the burst disks, which are installed for each of the two differential hydraulic lines and are set at 10,000 psi, will open. This will ensure safety of the hydraulic lines.

### **A.3.6 Pressure gauges**

Each differential hydraulic controller is equipped with Riken pressure gauge (rated to 250 MPa) and one pressure transducer (digital pressure gauge rated at 250 MPa). The main hydraulic ram (maximum operating pressure 10000 psi) is also equipped with one analog pressure gauge (rated at 15000 psi), and one pressure transducer (rated at 10000 psi).

### **A.3.7 Displacement limit switches**

Over travel of each cylinder is limited by two displacement limit switches (upper and lower limits).

### **A.3.8 Pressure switches**

Each controller has a Riken pressure switch, which will automatically stop the motor when the pressure reaches a preset point (currently at 70 MPa or 10000 psi and adjustable). When pressure reaches to the setpoint, the system will automatically stop.

### **A.3.9 Burst disks**

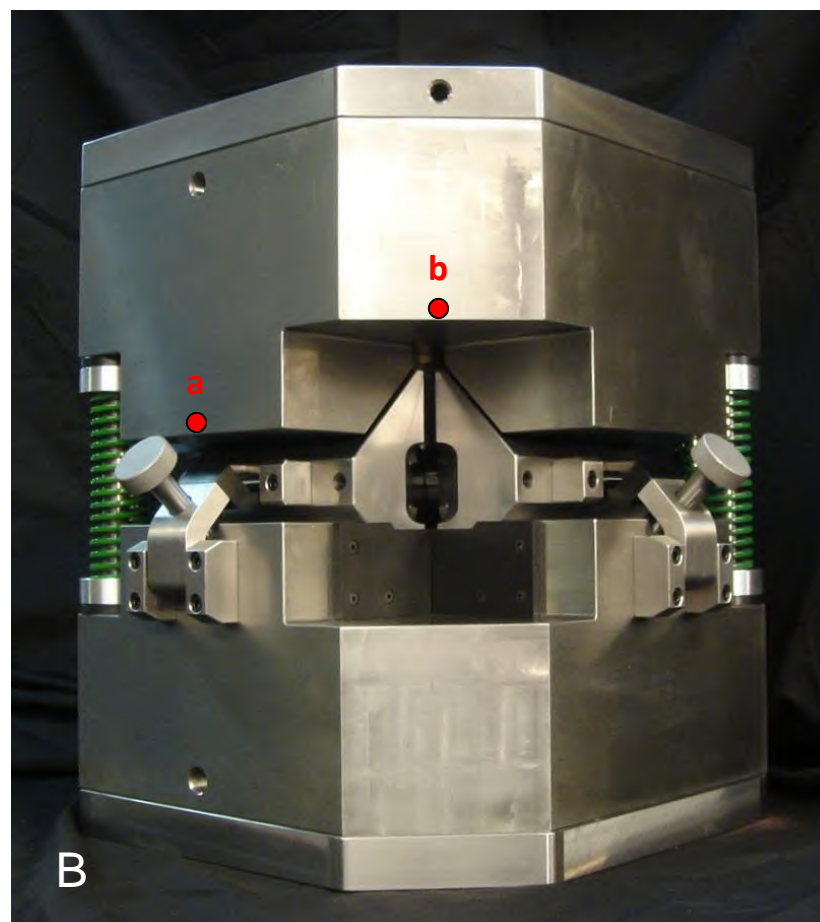
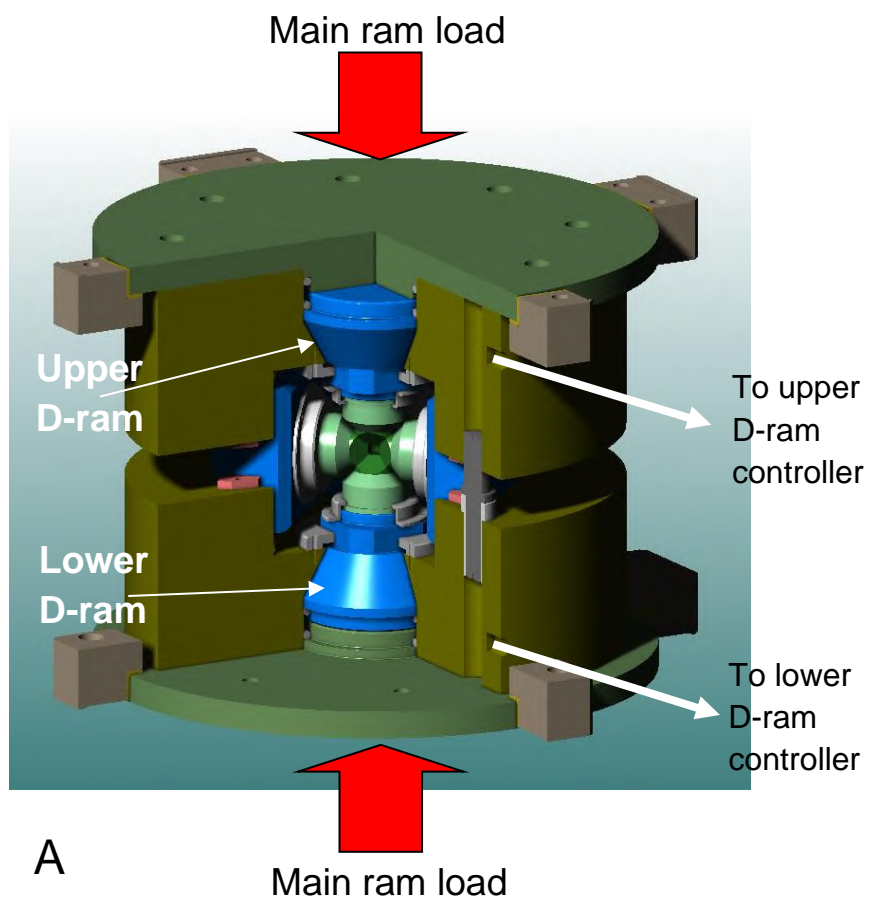
There are two burst disks (one for each hydraulic line) in the differential ram control systems which prevent over-pressurizing the low pressure system and high pressure system. They are rated at 10000 psi. The main hydraulic line (maximum pressure 10000 psi) also has a burst disk set at 8800 psi.

### **A.3.10 Pressure transducers**

There are two pressure transducers in the system to measure the pressure in the differential ram controllers (rated at 300 MPa). The main hydraulic line (to 1000 ton press, which operates at a maximum oil pressure of 10000 psi) has one pressure transducer rated at 10000 psi.

### **A.3.11 Spill-capturing plates**

To prevent spillage of hydraulic fluid (e.g., in the event of rupture O-ring), plastic plates are installed under the hydraulic cylinders. Small plastic containers (about 500 cc capacity) are also mounted under each rupture disk.



**Figure 1.** A: A cut-away view of the DDIA-30 module with schematic layout. B: Actual DDIA-30 module, showing the upper and lower guide blocks and wedges that hold the horizontal anvils. Horizontal dimension is roughly 0.6 m. Four points were monitored during load tests: points a and b and two point equivalent to a and b on the opposite side of the module (referred to as a1 and b1).

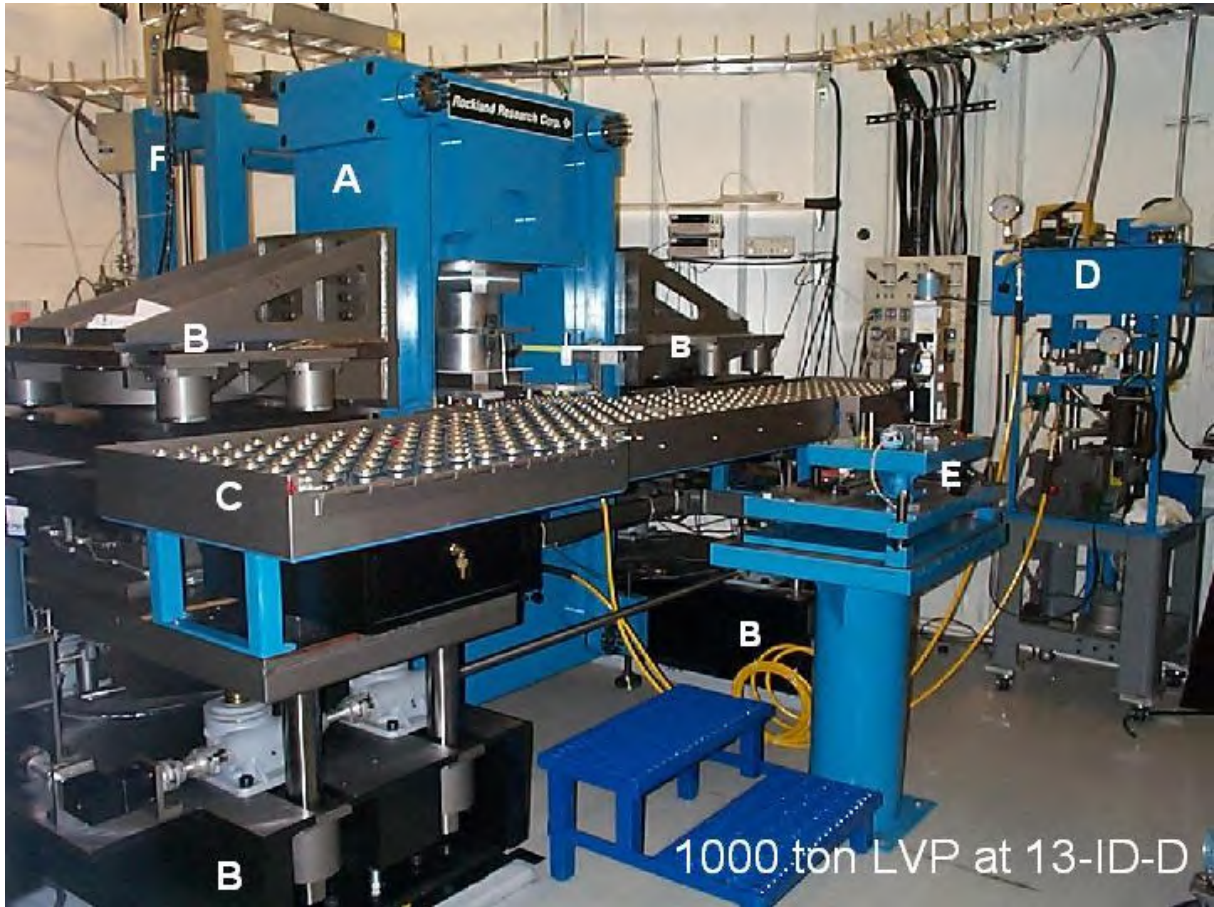


Figure 2. 1000 ton press in 13-ID-D. A: press frame with hydraulic ram. B: support table. C: module transport table (to be replaced by rail systems for DDIA-30). D: hydraulic controller. E: entrance slit

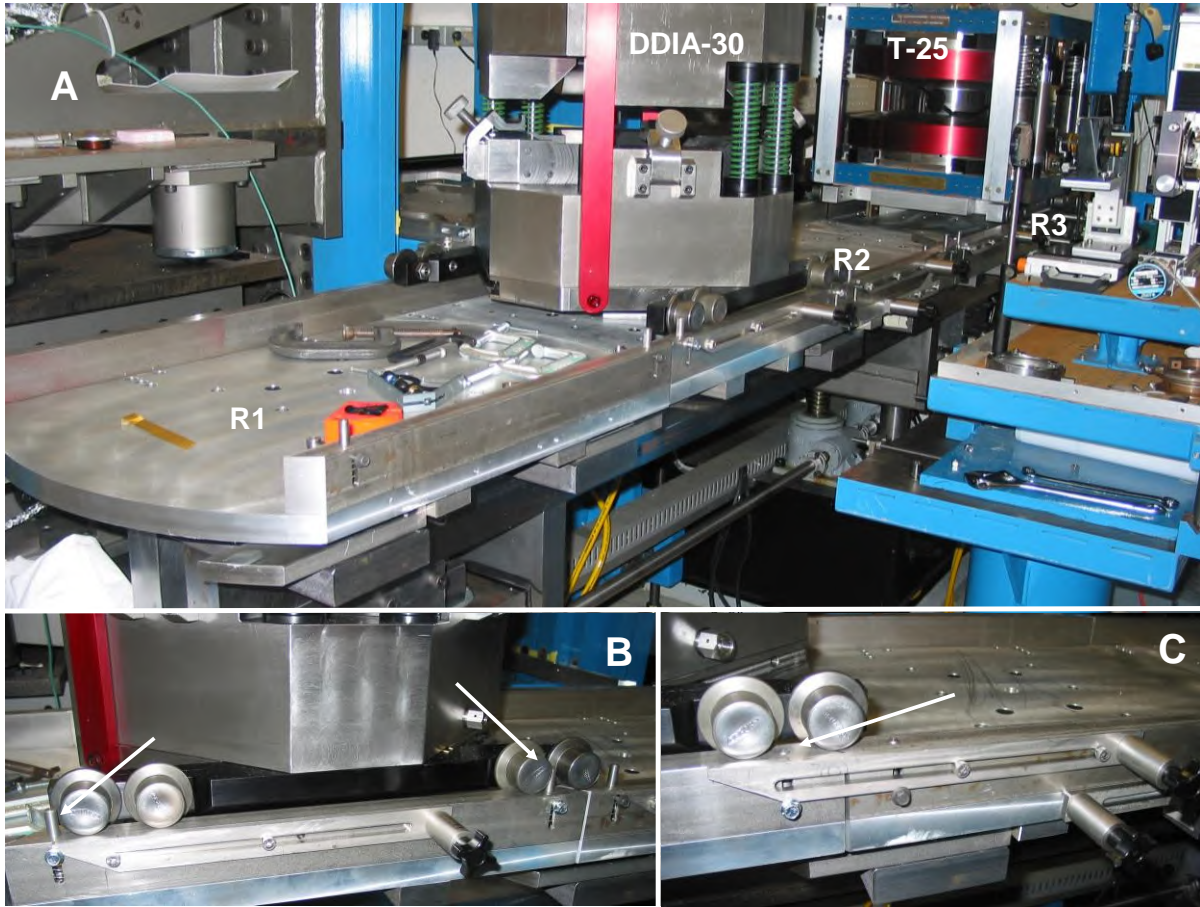


Figure 3. Transport table for the DDIA-30 and T-25 high pressure modules. A: Overall view. R1, R2, and R3 are the rotary stages for rotating the modules. B: Detailed view of safety stopping pins (white arrows). Locking positions. C: Unlocking position. Only when the pins are pushed down by the sliding locking bars, can the module be moved to the desired direction.



Figure 4. Two 200 MPa hydraulic pumps for controlling the differential rams in DDIA-30. These pumps have been mounted on the roof top of 13-ID-D. Hand rails, steps, and warning signs have been installed. Hydraulic lines into the hutch will be installed in May, 2010. Note that this location is restricted to authorized personnel only.



## **B Safety features**

### ***B.1 The DDIA-30 module***

#### **B.1.1 Burst disk to prevent overload**

The module design took a conservative approach, by using a large horizontal base dimension of 425 mm. The estimated normal stress acting on the 45 degree sliding surfaces is 200 MPa, one order of magnitude lower than the yield strength of the material used for the guide blocks. At a 9 MN axial load, the maximum radial displacement detected during the tests was 0.15 mm, corresponding to an elastic strain of  $7 \times 10^{-4}$ . In the 1000 ton press hydraulic line, there is a burst disk rated at 8800 psi (corresponding to a ram load of 9 NM), to ensure that no load greater than 9 MN can be applied. The two differential rams also have burst disks that will limit the maximum operating oil pressure to 70 MPa or 10000 psi.

#### **B.1.2 Front shield**

Occasionally anvils may fail inside the high pressure module. Although the anvils are largely contained inside the module, there is a small possibility for the debris to fly out through gaps between the guide block. A plexiglass will be placed in front of the 1000 ton press frame.

### ***B.2 The transport table***

#### **B.2.1 Safety stopping pins**

There are at least two stopping pins to keep the module in place at all times, so that the module is not free to move. Pin-up is the default position – all pins are spring loaded from the bottom up (Figs. 3A and B). In order to move the module to certain direction, pins in that direction must first be pushed down. This is accomplished by sliding the locking sliders (see below).

#### **B.2.2 Locking sliders**

By pulling the spring-loaded locking sliders and sliding them towards the safety pins, the sliders lock the rails on the rotary stage to the stationary portion of the transport table. Transporting a module from R1 to R2 requires operating on two locking sliders, which lock R1 and R2 to the stationary portion that connects the two rotary stages. These sliders push the safety pins down, allowing the module to be rolled from R1 to R2 (Fig. 3C).

In order to rotate R2, locking sliders must be disengaged from the stationary rails. Safety pins on R2 pop up when the sliders are disengaged, assuring that the module will not roll over R2. Only when sliders are disengaged can R2 be rotated.

After rotating R2 by 90 degrees, the same locking sliders are used to engage rails on R2 to the rails inside the press and push down the pins in the press, allowing the module to be rolled into the press. By disengage the sliders, pins in the press pop up, keeping the module in place.

### ***B.3 The differential rams***

Two potential hazards with differential controllers are over pressure and liquid spillage.

#### **B.3.1 Stress analysis and safety factor**

Appendix F.4 is the stress analysis for the 200 MP pumps and the calculation for safety-to-failure factor. The analysis indicates that stress levels are well below the nominal strength of the materials used. Stresses on the major components are 3.75 – 5.33 times lower than the yield strengths of the steels used.

### **B.3.2 Minimized physical access**

There is no need for any user to access physically the differential pumps on the roof top. Cameras will be installed so that pressures can be ready off the gages. Pressure transducers will also be installed in the hydraulic lines and pressure values will be logged and displayed on computer screens. A hand rails system has been built around the pumps, but access to the pumps are given to authorized personnel only.

### **B.3.3 Over-pressure prevention – pressure switch and displacement limitation**

Each of the two differential controllers has two limit switches to prevent the hydraulic cylinder from over travel. In addition, a pressure switch is installed in the hydraulic line for each controller, to ensure that no pressure greater than 70 MPa (10000 psi) can be built up.

### **B.3.4 Spillage prevention – fluid containers under the controllers**

All hydraulic lines are rated 2 times the operating pressure. Potential hydraulic fluid leakage would occur when O-rings are worn. Plastic containers are located under the hydraulic cylinder and will collect hydraulic liquid. For each cylinder, the maximum amount of hydraulic fluid is 220 cc, smaller than a coffee mug.

### **B.3.5 Pinch points**

There are no pinch points in the system. The hydraulic cylinder moves at extremely low speeds, on the order of 0.1 – 1 mm/s.

## C Stored Energy Analysis

Below is our analysis on stored energy in the systems. Independent analysis by Dejan Ristic concluded that “due to virtual incompressibility of liquids and small volumes, stored energy is low despite high pressures.”

### *C.1 Stored energy – Main ram with DDIA-30*

The main ram cylinder has a diameter of 17”. To compress the DDIA module, the ram advances upwards by a maximum amount of ~0.5”, at zero pressure in order for the ram top to touch the DDIA-30 module. This will require a total amount of 113.43 cubic inches of hydraulic fluid, or 0.001859m<sup>3</sup>. The zero pressure bulk modulus of the hydraulic fluid is  $K = 1700$  MPa. With a hydraulic fluid pressure of 8800 psi (= 60.67 MPa), volumetric fraction reduction is  $60.67/1700 = 0.03569$ , or 3.569%.

Note that this is an overestimate, as compression behavior is expected to decrease with increasing pressure. For the maximum volume of 1859 cc (0.001859m<sup>3</sup>), we obtain an actual volume reduction of  $1859 * 0.03569 = 66.3$  cc. This is the amount of extra fluid volume pumped into the ram at 1000 ton.

Assuming we lose all this volume in an incident (failure of one anvil inside the DDIA-30 module, for example), so the pressure in the cylinder goes back to zero. The (overestimated) total energy release is then  $\frac{1}{2} 60.67$  MPa \* 66.3 cc = 2210 N m (Joule).

This amount of energy release is roughly equivalent to two persons, each weighing ~120 lbs, jumping off a 1 meter (or 40 inch) high step.

### *C.2 Stored energy – Differential pumps*

Similarly for the differential pumps, with a maximum pressure of 200 MPa, total volumetric fraction reduction is  $200/1700 = 0.11765$ , or 11.765%.

Again this is an overestimate, as compression behavior is expected to decrease with increasing pressure. For the maximum volume of 228 cc, we obtain an actual volume reduction of  $228 * 0.11765 = 26.8$  cc. This is the amount of extra fluid volume pumped into the cylinder at 200 MPa.

Assuming we lose all this volume in an incident (burst of an O-ring, for example), so the pressure in the cylinder goes back to zero. The (overestimated) total energy release is then  $\frac{1}{2} 200$  MPa \* 26.8 cc = 2680 N m (Joule).

This amount of energy release is roughly equivalent to two persons, each weighing ~150 lbs, jumping off a 1 meter (or 40 inch) high step.

Also, note that the actual operating oil pressure is limited to 70 MPa (10000 psi) by burst discs and pressure switches. So the actual energy to be stored is much less than the above calculation, which is based on the designed full capacity.

## **D Standard Operating Procedures**

The following are the rules and procedures to be followed when operating the gas loading system.

### ***D.1 Authorized Personnel***

Only persons who have been trained in the use of the multi-anvil system, and whose names are on the list of Authorized Users may use the high pressure system. Users must agree that they understand this document, and will following these standard operating procedures. Appendix F.5 lists current authorized operators. Authorized users should read and understand ESH Manual 13.1 (Pressure Systems Safety) and complete ESH 119 (Pressure Safety Orientation).

### ***D.2 Personnel Protective Equipment***

Users must wear safety goggles entering the station 13-ID-D, when the system is under pressure.

### ***D.3 Restricting access and posting***

Warning signs must be posted on the hutch door, alerting unauthorized personnel to stay away from the hutch when the system is under pressure.

### ***D.4 Emergency Procedures***

The Emergency stop button can be pressed at any time. This will stop all the motors (including hydraulic controllers) and shut off power supply for heating. If there is a problem then contact Yanbin Wang or the APS Floor Coordinator.

### ***D.5 Loading a sample in the DDIA-30***

To load a sample assembly into the DDIA-30 module, the top guide block should be lifted. A mechanical lifting device has been installed for general users. The operators will all have proper training for operating the crane to qualify as authorized operator (Appendix F.5 lists all the authorized operators for this beam time).

### ***D.6 DDIA-30 transport***

After the sample assembly is loaded in the DDIA-30, the entire module is translated to the middle of the transport table, rotated by 90 degrees, and rolled into the hydraulic press. The operation principles are described in Section B.2. All operators should have proper training as part of the requirement to become authorized operators. A list of authorized persons for the April tests is given in Appendix F.5.

## **E Inspection and Maintenance**

All the hydraulic components will be inspected regularly by Guy Macha of CARS. Guy is a very experienced mechanical and vacuum technician with many years of experience in vacuum systems. It has been verified that the 1000 ton high pressure systems did not leak when pressurized to its maximum design pressure. Inspection of the hydraulic lines for the differential rams will be performed when that component is ready.

All burst disks will be replaced every 5 years, even if not used for a certain period. All pressure gauges (analog and digital) will be calibrated once a year.

Guy Macha is the only personnel who have been specifically authorized to perform mechanical work on the high pressure components (tubing, valves, gages, O-rings).

## F Appendices

### F.1 Load test results for DDIA-30

The DDIA-30 was tested at Rockland Research Corp. The module was compressed to 9 MN in a 1000 ton press, essentially identical to the one we use at 13-ID-D, held overnight, with dial indicators monitoring the outer dimensions of the module. No plastic deformation was detected. All displacements recorded by the indicators showed linear relation with applied load, and all measured displacements returned to zero when the load was removed. Similar tests were repeated 4 times and the results were consistent. Then the module was loaded to 11 MN, ~22% over the design load, the module behaved the same. Then at a main ram load of 0.6 MN, pressures in the differential rams were then increase to 0.4 MN. Displacement measurements showed similar behavior. I was present at these tests to witness the results. Figure A-1 shows one set of the test results.

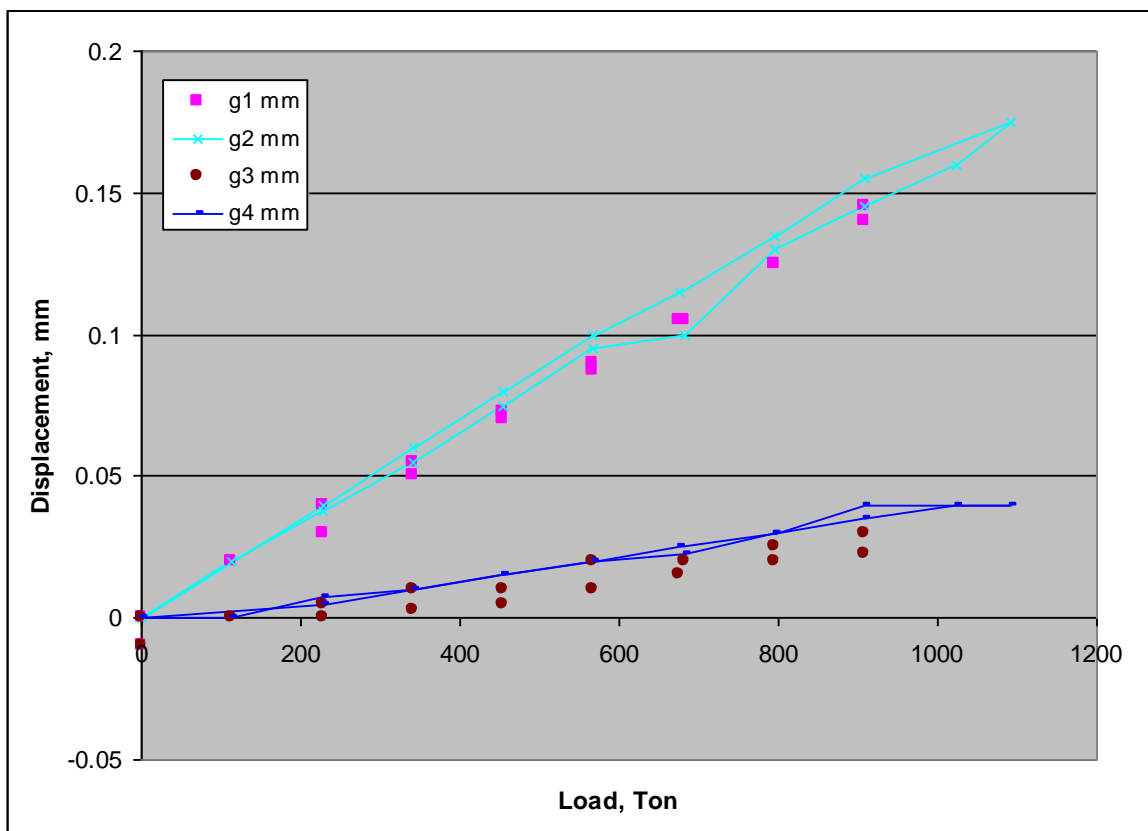


Figure A-1. Displacements on the DDIA-30 module measured during load test. Gage readings g1 and g2 correspond to point a and its equivalent in Fig. 1B, and gage readings g3 and g4 correspond to point b and its equivalent in Fig. 1B. Maximum radial displacement is 0.15 mm at the maximum design load (900 ton). Tests at loads 22% above the maximum design load show that the



### F.3 *Oh!sawa hydraulic control system component safety document*

#### 製造規格・検査試験圧力証明書

Production specification and inspection pressure certificate

The University of Chicago Geo Soil Enviro CARS 殿

#### 記

- 1) 200MPa プランジャーポンプ (GMP200-220B) は一部のネジを除き JIS (Japanese Industrial Standards) 規格によって製造された。  
200MPa plunger pump (GMP200-220B) was manufactured from the JIS (Japanese Industrial Standards) standard excluding a part of screw.
- 2) 試験、検査圧力は本機の最高使用圧力の 1.25 倍 (250MPa) で行なわれた。  
1.25 times (250MPa) the maximum allowable working pressure of this machine were cost by the examination and the inspection pressure.

以上、相違の無いことを証明いたします。

I prove there is no difference above.

2009年9月25日

184-0011 東京都小金井市東町4丁目41-23  
有限会社 オーサワシステム

代 表

大澤 昭夫 



TO.

J0G~J3G  
J31/2A~J7A  
SCREW JACK  
INSTRUCTION AND  
SERVICE MANUAL

SAFETY INSTRUCTION

1. INTRODUCTION.....1  
 2. CONSTRUCTION.....1  
 3. INSPECTION.....1  
 4. INSTALLATION.....1  
 5. LUBRICATION.....2  
 6. OPERATION AND MAINTENANCE.....4  
 7. DISASSEMBLING.....4  
 8. INFORMATION ABOUT INQUIRIES...5  
 9. WARRANTY.....5

改訂 REV. NO.	日付 DATE	改正内容 DESCRIPTION			承認 APPRV	担当 PREP.	
	配布先 DISTRIBUTION	作成日 DATE	MAR. 15. '2005	書類番号 DOC NO.	JIM-2-1-STD-E		
		発行元 ISSUED BY	ジャッキ事業部 技術課 JACK. DIVISION	承認 APPRV	照査 CHKD.	担当 PREP.	
		日本ギア工業株式会社 NIPPON GEAR CO., LTD.					

## SAFETY INSTRUCTIONS

### FOR MAINTENANCE MANAGER

These cautionary notes contain important matters concerning safety during handling of the gear unit.

Please read these instructions carefully before using the unit, and ensure that it is handled correctly.

Please ensure that the gear unit is handled by professional who has received specialized training.

#### Maintenance and Servicing, etc.

**⚠ WARNING** ...BEWARE OF ELECTROCUTION (electric types)!!

- (1) When wiring the unit, ensure that insulation has not been reduced by water or dampness.
- (2) Ensure that the unit is earthed securely.

Disregard of these warnings can lead to electrocution.

**⚠ WARNING** ...BEWARE OF ENTANGLEMENT IN ROTATING PARTS !!

- (1) Wait until all rotating parts have stopped completely before working on the unit.
- (2) When working on the unit, keep in touch with the power switch operator and let him know what you are doing.
- (3) Do not go near or touch the rotating shaft while the unit is in operation.

Disregard of these warnings can lead to bodily injury.

Keep this leaflet carefully where it can be seen at all times.

Proper maintenance, servicing and after-care is important to ensure correct and safe usage of the gear unit.

MANUFACTURER: NIPPON GEAR CO., LTD.  
(JAPAN)

 NIPPON GEAR CO., LTD.

1. Introduction

This manual contains handling instructions of the equipment described in the title and is intended for personnel directly responsible to the operation and maintenance of the equipment. Before starting up or inspecting the equipment, it should be read thoroughly.

To manufacturers: An effort should be made to furnish this manual to end-user personnel directly responsible to the management of the equipment.

2. Construction

The screw jack is for lifting and lowering heavy objects, and its construction is shown in drawing.

3. Inspection

Before installing the delivered equipment, carry out the following checks:

- (1) Check frame number, reduction ratio and stroke shown on the nameplate to verify that they conform to the order specifications.
- (2) Inspect the equipment for damage or evidence of rust during shipping or storage.
- (3) If damage or rust is present, notify both the Company and the Carrier immediately (within seven (7) days).

4. Installation

Whether the equipment is adequately installed or not affects the performance and lifetime of the screw jack. When installing the equipment, the following instructions should be carefully observed:

- (1) The screw jack must be installed to a rigid baseplate.
- (2) The screw shaft must be installed so that any lateral and unbalanced loads will not be placed to it over the entire length.
- (3) When the equipment is to be installed on a machine or device generating vibrations, care should be taken not to give the vibrations directly to the equipment.
- (4) A flexible coupling is recommended for connecting the input shaft and electric motor. In this case, take good care so as to have precise alignment, i.e. centering, between the two.
- (5) If the need arises for its installation different from the original, please contact Nippon Gear Co. or otherwise, the screw jack or its machine may be damaged by the installation or operation other than in the approved drawing.
- (6) Since end plate is not prepared in a screw shaft end, be careful of a setup of a position enough.
- (7) When screw shaft speed used it by 1500mm/min or more, please make the  $\phi 5 \sim \phi 10$  hole for air extraction in a screw cover.
- (8) When the pipe etc. is attached in the equipment with which the jack is attached, the noise may come out owing to the resonance accompanying operation.

- (9) When load acts on the installation bolts, please use the bolts of on-the-strength classification 10.9.

#### 5. Lubrication

- (1) The jack is by grease lubrication. If not specially required, NIPPECO S No.2. grease of Nippon Koyu Co., LTD. is used. It is suitable both under an extreme pressure and a non-extreme pressure.  
(The range of temperature is  $-15^{\circ}\text{C} \sim 130^{\circ}\text{C}$ )
- (2) For replenishment or replacement, use the NIPPECO S No.2. Don't use other brand. If this is mixed into the original one, the desired grease quality, such as consistency, dropping point, etc., is altered, thus lowering the lubrication capacity.
- (3) Exchange for other brand of grease should be made after complete removal of the original one. This grease must be an extreme pressure one, with excellent lubrication, having the consistency equivalent to NLGI standard No.2.(265~295). (table 1)

Table 1

Manufacturer	Trade Name	Grease base(soap)
※Nippon Koyu	NIPPECO S No.2	Li
Idemitsu Oil	DAPHNE CORONEX EP2	Li
Japan energy	LISONIX EP2	Li
Cosmo Oil	DYNAMAX EP2	Li
Mobil Oil	MOBILUX EP2	Li
Nippon Oil	EPNOC AP2	Li-Pb
Showa-shell Oil	ALVANIA EP2	Li-Pb

※ Nippon Gear standard

- (4) The endurance of grease is determined by operating conditions of the jack (load, frequency of the usage, and temperature) and introduction of foreign matter into the grease, as well as by the grease quality itself. So, it is not possible to state its life any definitely.

To have proper lubrication of the jack, however, replace the grease with a fresh one every one to two years. Exposure of the lifting shaft to a high temperature or non-covering of the lifting shaft to introduce foreign matter may accelerate deterioration of the grease quality. So check well when the machine (or other device) is to be inspected, as to whether there has occurred "bleeding" phenomenon or not; which is separation of grease and hence attachment of soap substance to the worm gear, bearing, etc.

If this happens, replace the grease before termination of the specified period.

Replacement with fresh is done by dismantling the ball screw jack.

The amount of grease required per jack is indicated in table 2.

- (5) In normal operation of jack, there is no need for the grease replenishment, until the designated period is terminated. If, however, the lifting shaft is uncovered, check the shaft each day for attachment of any foreign matter. Such is to be removed thoroughly with the grease; and apply fresh grease.
- (6) Deterioration of the grease occurs even during non-operation of the jack. After an extended period of its repose, therefore, check in this respect before usage of the jack.

Table 2. Quantity of Grease, per jack

JACK SIZE	Quantity
J0G	0.1 kg
J1G	0.4 kg
J2G	0.6 kg
J3G	1 kg
J3 1/2 A	1.5 kg
J4A	2.0 kg
J5A	3.0 kg
J6A	4.0 kg
J6 1/2 A	6.0 kg
J7A	8.0 kg

- (7) Since the screw shaft part of the jack does not have seal structure, grease may leak.

#### 6. Operation and maintenance

- (1) Before load operation of the jack, drive it under no or slight load for a few hours, to have the "running-in", i.e. fitting.
- (2) If there occurs any abnormality in sound or temperature during operation, suspend the operation, and dismantle the jack to check for the cause.
- (3) Please do not use it out of the predetermined stroke range.
- (4) Be sure to use a limit switch to control the length of stroke. Before operation of the jack, confirm whether the limit switch acts properly, or not.
- (5) The "standard" jacks are usable in an ambient temperature up to 100°C.  
(J0G~J3<sup>1</sup>/<sub>2</sub>A are up to 80°C)
- (6) Too high or too low indications on the ammeter (for the electric motor) or the manometer (for the hydraulic or air motor) in the operation panel imply abnormality in the jack, excessive load, abnormal values of the voltage or pressure, or failure of the power source. In this case, stop operation of the jack immediately, and check the cause.
- (7) Stops are to be taken to prevent any rusting during extended periods of non-operation, storage or shipment.

#### 7. Disassembling

Do not disassemble the screw jack.

If necessary, it is suggested the screw jack be returned to the factory.

8. Information about Inquiries

For inquiries or orders for renewal parts, please contact our sales representatives.

9. Warranty

1. A warranty period shall be effective for one year after the date of shipment from the Company.

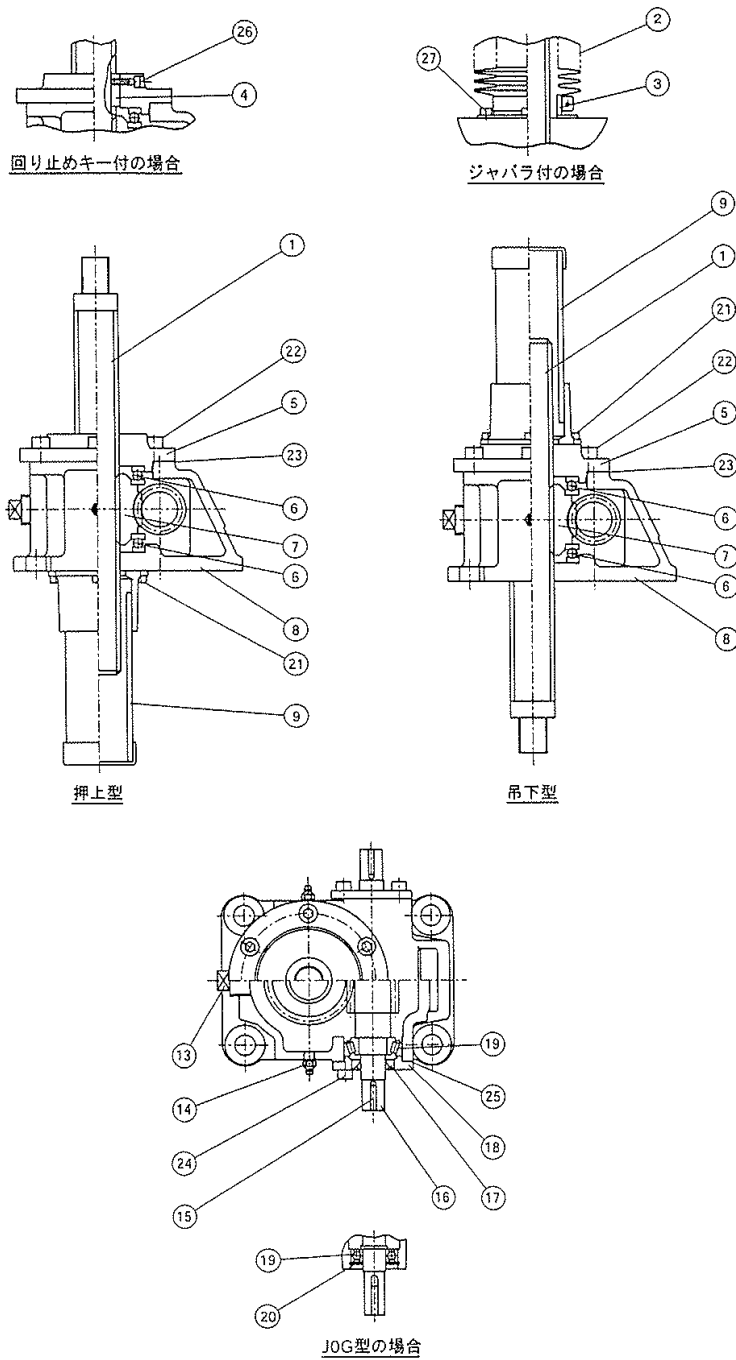
2. The Company's warranty applies for a period specified by the agreement at the time of purchase insofar as the product is operated within the rating and service conditions for which it was specifically sold. However, the warranty does not apply in the cases mentioned hereinafter even if the warranty period is effective.

- 1) Defects or failure of the product to perform resulting from operations beyond the rating and service conditions for which it was specifically sold
- 2) Loss or damage to the equipment due to natural disasters, including without limitation thereof fires, typhoons, floods, earthquakes, and any other circumstances over which the Company has no control.
- 3) Defects due to alterations or repair performed by others outside the Company or the Company authorized service facilities
- 4) Defects in materials, such as painting, natural discoloration of metal plating, rust, and separation of oil in grease, due to age-based change
- 5) Defects due to failure to follow handling instructions, or maintenance and inspection prescribed in the operation manual
- 6) Defects due to abuse or mishandling
- 7) Sensory phenomenon (sounds, vibrations and so on) that is considered to exert no influence on the quality and performance of the product
- 8) Consumables listed on the Parts List and other materials

3. When the defect on material and manufacture is discovered within the abovementioned term of a warranty, we should supply a substitute at the expense of our company. (A warranty range area is restricted in Japan)

The warranty does not cover any expenses for removal of allegedly defective equipment or parts or for installation costs of repaired or replaced equipment or parts. The Company shall not be liable for any losses or expenses resulting from out of use due to defective equipment. When compensation will be paid in value, the maximum of the amount of money does not exceed the selling price of the product for a claim.

7. スクリュージャッキ及びボールスクリュー  
ジャッキの構造を図1～5に示します。



符号	部品名称
1	ねじ軸
2	ジャバラ
3	フランジ
4	キー
5	ハウジングカバー
6	ベアリング
7	ウォームホイール
8	ハウジング
9	ねじ軸カバー
10	
11	
12	
13	プラグ
14	グリースニップル
15	キー
16	ウォームシャフト
17	オイルシール
18	サイドカバー
19	ベアリング
20	スナップリング
21	ボルト
22	ボルト
23	シム
24	ボルト
25	シム
26	ボルト
27	ボルト

図1 軸昇降型スクリュージャッキ



## F.4 Stress calculation for the 200 MPa differential pumps

### 200MPa プランジャーポンプ応力計算書

有限会社 オーサワシステム

1. ベース板(770x580x16t)は各部品を固定する役目のため、殆ど応力は発生しない。
2. 主な応力は油圧力を発生する①シリンダー、シリンダーが発生した力を受け止める②反力受け、その反力受けが受けた力をジャッキ本体に伝える③シャフトに発生する。
3. 計算式

- ① シリンダー (チューブ応力)

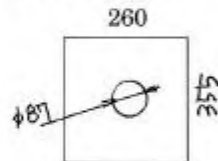
$$\sigma = \frac{\text{圧力(P)} \times \text{内径(D)}}{200 \times \text{シリンダー壁厚さ(t)}} = \frac{2000 \times 44}{200 \times 22} = 20(\text{kgf/mm}^2)$$

シリンダー材質 SCM435  $\sigma=95(\text{kgf/mm}^2)$ 以上

よって安全率 :  $95/20=4.75$  以上

- ② 反力受け (近似計算 反力受け中央部に集中応力を受けた場合)

シリンダー出力 :  $\phi 44 \times 2041(\text{kgf/cm}^2) \approx 31034(\text{kgf})$



$$\sigma^1 = \frac{M}{Z} = \frac{1/4 \times 31034 \times 280}{1/6 \times 260 \times 50^2} = 20.05$$

$$\sigma^2 = \frac{M}{Z} = \frac{1/4 \times 31034 \times 190}{1/6 \times 355 \times 50^2} = 9.96$$

反力受け材質 S45C  $\sigma=75(\text{kgf/mm}^2)$ 以上

よって安全率 :  $75/20=3.75$  以上

- ③ シャフトの引っ張り応力

最も細い部分 :  $\phi 26.5 \leftarrow$  M30 ネジ谷径

シリンダー出力 :  $\phi 44 \times 2041(\text{kgf/cm}^2) \approx 31034(\text{kgf})$

$$\sigma = \frac{31034}{4 \text{本} \times \frac{\pi}{4} \times 26.5^2} \approx 14.07$$

シャフト材質 S45C  $\sigma=75(\text{kgf/mm}^2)$ 以上

よって安全率 :  $75/14.07=5.33$  以上

### ***F.5 Authorized DDIA-30 operators for April 2010 test***

Requirements for user authorization:

1. Crane training (before lifting device is installed)
2. Transport table operation
3. Hydraulic operation
4. Familiarity with this SOP

Name	Badge number	Institution	Date authorized
Wang, Yanbin	85201	GSECARS	3/2010
Hilairret, Nadege		CNRS, Univ. Lille, France	3/2010
Yu, Tony		GSECARS	3/2010
Jing, Zhicheng		GSECARS	3/2010

## **Appendix C.5 GSECARS SOP for Preparation of Beryllium Gaskets In Laboratory A030, Bldg. 434A**

### **1. Introduction**

This is a Standard Operation Procedure to prepare Be gaskets for diamond cell experiments in room A030. The Be gasket dimensions are 0.5 mm in thickness and 3 mm in diameter. After pre-indentation, the thickness is less than 50 micrometers. The main preparation is to drill a hole about 10 – 100 microns in diameter at the center of the indented area, which involves machining on the order of  $10^{-5}$  g of Be.

### **2. Hazards**

Beryllium is highly toxic, inhalation of the dust resulting in berylliosis.

### **3. Controls**

The drill bits will be clearly marked “FOR BERYLLIUM”, and will be only used for drilling Be gaskets. A clearly marked container with a removable cover will be made for holding Be gaskets in liquid. The drilling operation will be done while the Be gasket in oil medium (e.g., glycerin) inside the container described above. After drilling, the waste oil will be poured to a clearly marked waste container in the airflow hood and the drilling container will be rinsed with alcohol. The waste container will be disposed of properly when it is full.

### **4. Authorized Personnel**

Authorized personnel should have training by the Principal Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.

The following is a list of personnel authorized. An updated list of authorized personnel will be posted besides the micro drill machine.

Name	Affiliation	Badge Number
Vitali Prakapenka	University of Chicago	85331

## **Appendix C.6    GSECARS Safety Procedures for 13-BM-D LVP Experiments (July, 2008)**

GSECARS, last updated July 6, 2008

1. Major high pressure components are certified by manufacturer to pressure specifications
2. Emergency shut-off switches for pressure generator and power supply are clearly labeled
3. Location of pressure generator clearly marked; do not turn any valves while system is under pressure
4. Location of pressure gauges are clearly marked
5. Make sure that hose from pressure relief valve on pressure generator is fastened to the generator unit and the open end is placed in a container
6. Only authorized and trained persons may access hutch while system is pressurized  
    Authorized operators:  
    See separate list
7. Safety goggles must be worn when near the press to examine tooling position/orientation and thermocouple contact etc., under pressure. Most visual examination can be done with in-hutch TV camera.
8. Warning signs will be placed on door when system is pressurized
9. Used anvils must be placed in proper containers; always wear safety goggles when handling anvils

## **Appendix C.7 GSECARS Safety Procedures for LVP Experiments at 13-ID-D (June, 2008)**

GSECARS Last updated: June 9, 2008

1. Major high pressure components are certified to pressure specification. All components have rated safety factors of 3 or above. System has been tested up to 110% of its designed capacity. Pressure relief valves set at 90% of the designed capacity so that if load exceeds 90%, hydraulic oil will flow back to the reservoir.
2. Emergency shut-off switches for the pressure generator and power supply clearly labeled.
3. Location of pressure generator clearly marked; do not turn any valves while system is under pressure.
4. Location of pressure gauges clearly marked.
5. Power supply has all connectors insulated.
6. Only authorized and trained persons may access hutch while system is pressurized  
Authorized operators:  
See separate list
7. Safety Goggles must be worn when near the press to examine tooling position/orientation and thermocouple contact etc., under pressure. Most visual examination can be done with in-hutch TV camera.
8. Warning signs will be placed on door when system is pressurized.
9. Used anvils must be placed in proper containers; always wear safety goggles when handling anvils.

Argonne National Laboratory  
Advanced Photon Source  
GeoSoilEnviroCARS

GSECARS SOP for Gas Loading System  
Laser

(Created October 15, 2007)

---

**Mark Rivers**

Name Printed

Signature

Date

Approved by:

ANL-E Laser Safety Officer or Deputy Laser Safety Officer:

**Bruce Murdoch**

Name Printed

Signature

Date

ESH/QA Coordinator:

**Edmund Elroy Chang**

Name Printed

Signature

Date

Approved by:

Division Director:

**Bill Ruzicka**

Name Printed

Signature

Date

## 1. Introduction

This is a Standard Operation Procedure to operate the laser listed in Table 1. This laser is part of the GSECARS gas-loading system which is currently located just downstream of the 13 ID-D station on the APS Experiment Hall floor. The LCA is a small enclosure that contains the high-pressure vessel for the gas loading system. No personnel can fit inside the LCA.

Name of LCA supervisor: Mark Rivers  
Principal laser operator: Vitali Prakapenka

**Table 1: Laser Specifications**

	<b>Diode pumped laser</b>
Brand	LaserMate
Model	GML532100FKAS
Serial number(s)	7065232
IHID numbers(s)	10727
Quantity	1
Wavelength	532 nm
Diameter	~2mm
Divergence	<1.5 mrad
Power	100 mW
Mode	CW
Class	IIIb

## 2. User Training

There are two classes of users for this laser system, Authorized Users and General Users.

The following requirements must be met in order to be added to the list of Authorized Users for these laser systems:

- Completion of the Argonne laser safety training course
- Completion of an Argonne laser eye exam
- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.
- "Hands-on" on-the-job alignment training (OJAT) by the Principal Laser Operator, who serves as the LCA trainer.

The following requirements must be met in order to be added to the list of General Users for these laser systems:

- Training by the Principal Laser Operator or his/her designee, which includes signing a statement that the user understands and agrees to abide by this SOP document.

## 3. Operation modes

The laser can be operated in 3 modes:

1. **User mode:** The enclosure doors are closed. The laser remote control units are outside the LCA and the lasers are operated remotely. User mode operation can be performed by General Users and Authorized Users,.
2. **Expert mode:** This mode is used for alignment and can only be used by Authorized Users. The enclosure doors are open. A laser screen will be used to shield the area from scattered laser light. Users will wear the appropriate eye protection

## 4. Hazards

The green laser light is the only laser hazard. There are no high-voltage hazards, because there will be no work performed on the power supplies or electrical leads with the power on.

## 5. Controls

The following controls will be implemented in this LCA.

- For Expert a laser partition from Kentek will be positioned near the enclosure to prevent scattered light from escaping.
- The keys to the laser power supply will be kept under administrative control. When the laser is not in use by qualified personnel the key will be kept in a locked drawer in the LCA supervisor office.
- Appropriate eye protection will be worn by all personnel when it is running in Expert Mode. There two pair goggles with OD>6 at 450 - 550 nm available.
- In User Mode the laser beam is completely enclosed in the LCA and there is no possibility of light escaping.

## 6. Interlock System Description

The interlock system consists of the following hardware components:

- Door switches to verify that the LCA doors are close.
- A key switch to change between User Mode and Expert Mode.
- A PLC to interlock the laser. The laser can be enabled if the enclosure doors are shut, or if the key switch is in User Mode.
- A push button that toggles the laser enable, and a red light to indicate that the laser is enabled.
- A laser power supply from the vendor with a power control knob and a key-switch to turn the laser off and on.
  - Interlock is tripped, and must be re-enabled at the Laser Safety Panel
  - If any laser was enabled, then the fault light illuminates and must be cleared with the button on the Laser Safety Panel
- System is interlocked and override button is pressed
  - Alarm sounds alerting operators that someone has entered LCA
  - Interlock remains active

## 7. Expert Mode operation procedures for alignment

Expert Mode:

Only Authorized Users are allowed to operate the system in Expert Mode.

The following checklist will be posted near the laser control units to remind operators of procedures to follow.

Before turning on the laser:

- Check that the curtain is properly positioned.
- Wear proper safety goggles
- The laser power must be decreased to <1mW for initial optics alignment.
- Enable the laser with the PLC keypad.

When alignment is complete:

- Return the keyswitch to User Mode
- Disable the laser with the PLC keypad.



## **8. User Mode operation procedures:**

Before turning on the laser:

- Check that the key switch is in User Mode.
- Check that the enclosure doors are shut. The interlock system will not enable the lasers if the doors are not shut.
- Enable the laser with the PLC keypad.

Before opening the enclosure doors:

- Disable the laser with the PLC keypad.

## **9. Inspections and testing (forms to follow)**

- The interlock system will be tested quarterly
- The laser eyewear will be inspected annually

## **10. ANL Laser training (forms to follow)**

Two lists of laser users will be maintained and posted on the hallway door

- 1) Authorized Users, with ANL laser safety training and “On-the’Job” alignment training.
- 2) General Users, without ANL laser safety training







**Appendix C.9 GSECARS SOP X-ray Generators, Double-axis X-ray  
Diffractometer and Laue camera in HGS-057 (April, 2020)**

**Standard Operating Procedure  
X-ray generators  
Double-axis x-ray diffractometer and Laue camera in HGS-057**

**Author:** Joanne Stubbs

**Date of Creation/Revision:** 12/15/2011; 11/6/2019; 4/16/2020

**Location of Work:** University of Chicago Campus, Hinds Laboratory for the Geophysical Sciences, Room 057

**Principal Investigator (PI):** Peter Eng

**Description:**

X-ray tube sources (copper, molybdenum, tungsten, etc.) will be used for x-ray diffraction measurements using a double-axis diffractometer and a Laue camera.

**Hazards:**

1. Potential for exposure to ionizing radiation from x-ray tube sources
2. Potential for injury resulting from improper use of high-voltage electrical equipment.

**Required Training:**

1. General radiation safety training given by the University of Chicago Radiation Safety Office
2. Lab-specific training given by either Peter Eng or Joanne Stubbs

**Safety Controls:**

1. Radiation levels in areas where personnel will be working will be as low as reasonably achievable (ALARA).
2. Sufficient lead and leaded-Plexiglas shielding to reduce radiation levels in work areas to background will be in place before x-ray generators are energized.
3. Shutters will be closed whenever x-ray measurements are not actively being made.
4. Work areas will be verified with a Geiger-Mueller (GM) counter to have radiation levels at background when shutters are open. The person operating the diffractometer or Laue camera will alert other personnel before opening shutters.
5. All personnel will wear assigned radiation badges whenever x-ray generators are energized in order to monitor for inadvertent exposure.

6. Personnel will not perform electrical work or service high-voltage equipment without appropriate training and prior authorization from the laboratory PI.

**Step-by-Step Operating Procedure:**

1. Confirm that all shielding is in place
2. Chiller power on
3. X-ray generator power on
4. High voltage on
5. Water fault test (turn off water flow, confirm that x-ray generator high voltage switches off, then reset and turn HV back on)
6. Warm up slowly (15 min at 10 kV/10 mA; 15 min at 20 kV/20 mA)
  - a. Double axis diffractometer – max 36 kV, 20 mA
  - b. Laue camera – max 40 kV, 20 mA
7. Shutter controller on (key)
8. Open shutter and verify with Geiger-Mueller counter that the work area for personnel is not receiving radiation above background
9. To shut down, reverse the above, leaving chiller on for at least 15 minutes after powering down x-ray generator to ensure that tube fully cools
10. Remove key(s) from shutter controller(s)

**Appendix C.10 GSECARS - Safety Analysis and SOP COMPRES/GSECARS Gas Loading Apparatus (2/11/10)**

**Contents**

**A SYSTEM OVERVIEW .....3**

A.1 PURPOSE..... 3

A.2 OTHER SYSTEMS..... 3

A.3 VOLUME UNITS.....3

A.4 PRESSURE UNITS..... 3

A.5 SYSTEM DESIGN GOALS.....4

A.6 SYSTEM COMPONENTS..... 4

**B SAFETY FEATURES.....10**

B.1 LECTURE BOTTLE..... 10

B.2 BURST DISKS..... 10

B.3 INTERLOCKS..... 10

B.4 OTHER HAZARDS..... 12

**C STORED ENERGY ANALYSIS.....13**

C.1 STORED ENERGY..... 13

C.2 FAILURE ANALYSIS .....14

    C.2.1 *Velocity and energy of worst-case projectile*.....14

    C.2.2 *Penetration depth of projectile in aluminum*.....15

    C.2.3 *Catastrophic venting*.....15

    C.2.4 *Projectile impact into door*.....16

**D STANDARD OPERATING PROCEDURES.....18**

D.1 AUTHORIZED PERSONNEL..... 18

D.2 PERSONNEL PROTECTIVE EQUIPMENT .....18

D.3 RESTRICTING ACCESS AND POSTING..... 18

D.4 ALLOWED GASES..... 18

D.5 INSTALLING OR PRESSURIZING A LECTURE BOTTLE..... 18

D.6 LOADING THE CELL INTO THE PRESSURE VESSEL..... 19

D.7 PRESSURIZING THE PRESSURE VESSEL .....19

D.8 CLOSING THE CELL .....19

D.9 VENTING THE PRESSURE VESSEL..... 19

D.10 REMOVING THE CELL..... 19

D.11 EMERGENCY PROCEDURES..... 20

**E MAINTENANCE, CALIBRATION AND INSPECTION .....21**

E.1 CALIBRATION CHECKS.....21

E.2 INSPECTION..... 21

E.3 MAINTENANCE..... 21

E.4 INSPECTION AND MAINTENANCE LOG .....22

**F APPENDICES .....23**

F.1 LADDER LOGIC PROGRAM.....23

F.2 ANL FORM 722 PRESSURE SYSTEM EVALUATION FORM .....23

F.3 MANUFACTURERS CERTIFICATION DOCUMENTS .....23

    F3.1 *Pressure vessel*.....23

        F3.1.1 Pressure testing certification .....23

        F3.1.2 E-mail regarding cloverleaf reactor calculations..... 23

F3.1.3 Cloverleaf reactor calculations.....	23
F3,1,4 Window design calculations.....	23
F3.1.5 Material test report for cloverleaf reactor 4340 steel .....	23
F3.1.6 E-mail certifying that no additional heat treatment was done on 4340 material.....	23
F3.1.7 Mechanical drawings of cloverleaf reactor .....	23
<i>F3.2 Pressure transducers.....</i>	<i>23</i>



## **A System Overview**

### **A.1 Purpose**

This system is designed to load gases at high pressure into diamond anvil cells. The gases are used either as a quasi-hydrostatic pressure medium to surround the sample, or as the sample itself. The gases can be loaded at pressures up to 29000 PSI, which is high enough so that the densities become comparable to the densities of the liquid phase. The gases to be loaded are all non-toxic and inert, and will be primarily He and Ne.

It is very important to have access to a gas loading system when conducting diffraction experiments at the APS. Being able to have samples under hydrostatic stress (very small strain) is critical for both powder and single crystal diffraction experiments.

The system operates by loading a diamond anvil cell into the pressure vessel, pressurizing the vessel, and then remotely sealing the cell at pressure, trapping the high-pressure gas with the sample inside the gasket hole between the two diamonds.

The system will be located at Sector 13, but it will be made available to anyone who wants to use it. This includes scientists running experiments on other APS sectors, or registered APS users who want to load cells to use in their home laboratories or elsewhere.

The system was largely funded by the COMPRES high-pressure consortium which is managed by SUNY Stony Brook. The design and assembly were done at GSECARS. All of the high-pressure components are commercially purchased.

### **A.2 Other systems**

There are a number of other gas loading systems installed in both the United States and other countries. In this country systems are installed at the Geophysical Laboratory at the Carnegie Institution of Washington, the CHESS synchrotron at Cornell University, and the Lawrence Livermore National Laboratory. Abroad there are gas loading systems at both ESRF and SPring-8.

The system we have constructed builds on the experience from these previous systems. Our system is designed to be very easy to safely use, because a PLC prevents incorrect operation of all valves. In addition the system provides optical access for a ruby fluorescence pressure measurement system, so that the user can accurately determine when the cell has sealed and reached the desired pressure. Our system also accommodates a wide range of diamond anvil cell designs, rather than being restricted to a single type of diamond cell.

We have a copy of the safety documents from Livermore and CHESS and can make these available on request.

### **A.3 Volume units**

The following are the conversion factors from volume units used in this document

$$1 \text{ liter} = 1000 \text{ cm}^3$$

$$1 \text{ scf} = 28.317 \text{ liters}$$

### **A.4 Pressure units**

There are several units of pressure that are in common use, and which we will use in this document.

The SI unit of pressure is the Pascal (Pa). It is defined as a force of 1 Newton per square meter, where 1 Newton is the force required to accelerate a mass of 1 kg by  $1 \text{ m/s}^2 = 1 \text{ kg ms}^{-2}$ . The units of Pa are thus  $\text{kg m}^{-1} \text{ s}^{-2}$ .

The unit we will use most in this document is the bar.

The following are the conversion factors from bar to other common units.

$$1 \text{ bar} = 10^5 \text{ Pa}$$

1 bar = 14.504 pounds/in<sup>2</sup> (PSI)

1 bar = 0.987 atmospheres

1 bar = 10<sup>5</sup> Newton/m<sup>2</sup> = 10 Newton/cm<sup>2</sup> ~ (1 kgf)/cm<sup>2</sup>, where 1kgf is the force due to gravity of 1 kg mass. kg/cm<sup>2</sup> is a pressure unit used in older literature, which about 2% less than 1 bar

1 atmosphere = 101325 Pa.

The Omega pressure gauges in the system display pressure in PSI. The computer user interface displays the Omega pressure gauge readings in both PSI and bars.

## A.5 System design goals

In order to incorporate input from the community in the design of the system a workshop was held at the APS on April 28, 2006. The following was the agenda for the workshop.

- **12:00 Lunch**
- **1:00 Introduction** **Mark Rivers**
- **1:15 Experience at CIW and suggestions for APS** **Dave Mao**
- **1:30 Experience at LLNL and suggestions for APS** **Choong-Shik Yoo**
- **1:45 Experience at CHESS and suggestions for APS** **Chang-Sheng Zha**
- **2:00 Experience at Bayreuth** **Alexei Kuznetsov**
- **2:15 Preliminary plans for APS system** **Clayton Pullins**
- **2:30 Discussion**
- **5:00 End**

The following 16 people attended the workshop.

Name		Affiliation
Bass	Jay	UIUC
Dera	Przemek	CIW
Frank	Mark	NIU
Heinz	Dion	Univ. of Chicago
Kunz	Martin	LBL
Mao	Dave	CIW
Nicol	Malcolm	UNLV
Prakapenka	Vitali	Univ. of Chicago
Pullins	Clayton	Univ. of Chicago
Eng	Peter	Univ. of Chicago
Rivers	Mark	Univ. of Chicago
Shen	Guoyin	CIW, HP-CAT
Sinogeikin	Stas	CIW
Sturhahn	Wolfgang	APS
Yoo	Choong-Shik	LLNL
Zha	Chang-Sheng	CIW

Based on the workshop discussions, the following key system design goals were identified:

- Able to load many kinds of cells
- Closure mechanism (motor driven screws) will close a clamping device, not the cell itself. Easy to add new cell designs, just a different clamp or different spacers
- Optical access to view the cell while loading.
- Vacuum pump to clean system before loading
- Ability to have no electrical parts except pressure transducers in high-pressure enclosure
  - Allows flammable gas operation in future
- Easy to safely operate

## A.6 System components

Based on these design goals a system design was developed. The system layout is shown in Figure 1. This design incorporates the following features.

**Low-pressure system:** The low pressure system includes valves 1, 2, and 4, the lecture bottle, gauge 1, and burst disk 2K. This part of the system operates at a maximum pressure of 1500 PSI. It is isolated from the high-pressure system by valve 3.

**High-pressure system:** The high pressure system includes valves 5, 6 and 7, the high-pressure vessel, gauge 2, and burst disk 30K. This part of the system operates at a maximum pressure of 30,000 PSI. It is isolated from the low-pressure system by valve 3.

**High pressure vessel:** The component is labeled “Cell” in the system layout. The vessel has a cloverleaf closure mechanism at each end. The upper end has an optical window to allow viewing the diamond cell while it is being loaded. The lower end has two rotary feedthroughs to close the cell holder. The vessel is 6.5” OD, 3”ID, and made of 4340 alloy. A detailed design of the vessel is shown in Appendix 0. This vessel is not designed to load hydrogen, but a future more expensive system could use a hydrogen-safe alloy.

**Compressor:** The low and high pressure systems are pressurized with an air-driven compressor (Newport Scientific 46-14021-2) that can achieve 30,000 PSI output pressure.

**Valves:** There are 7 high pressure valves in the system. All are air-actuated, and have switches to indicate their open or closed status. A mix of normally open (NO) and normally closed (NC) valves are used, so that the system fails into a completely depressurized state on loss of air pressure.

**Gas cylinders:** There are two gas cylinders in the system. The large gas cylinder is normally used as the supply of the pressurizing gas. The second is a small, lecture bottle. The large gas cylinder is used only to fill the lecture bottle, either directly if the pressure in the large cylinder is >1500 PSI, or with the aid of the Newport compressor if the pressure is <1500 PSI. The high-pressure vessel is only loaded from the small lecture cylinder, to limit the total volume of gas that can be pressurized to 30,000 PSI. This protects against excess stored energy in the system if users forget to add the required filler blocks to the pressure vessel.

**Burst disks:** There are two burst disks in the system which prevent over-pressurizing the low pressure system and high pressure system. The low pressure system burst disk is rated at 1500 PSI. The high pressure system burst disk is rated at 30,000 PSI.

**Pressure transducers:** There are two pressure transducers in the system to measure the pressure in the low pressure system and the high pressure system. The transducers produce a 0-5V output over their full scale pressure range. The low pressure transducer (GP:50 Model 211-C-RT-7-FM) has a full scale pressure range of 3000 PSI. The high-pressure transducer (GP:50 Model 212-C-UC-AA) has a full scale pressure range of 2000 bar (~30,000 PSI).

**Pressure gauges:** Each of the pressure transducers is connected to a digital panel meter (Omega DP41-S-S2AR). Each meter provides a digital panel display in PSI, 3 setpoint outputs that are connected to the PLC control system, and an RS-232 output that is connected to the control computer.

**Vacuum pump:** The entire system can be evacuated with a combination dry-roughing and turbomolecular vacuum pump (Alcatel Drytel 1025). This pump can achieve  $10^{-6}$  torr.

**Vacuum gauges:** The Drytel 1025 is equipped with a combination Pirani and cold-cathode vacuum gauge (Alcatel ACC 1009) that can read from 1 atmosphere to  $10^{-9}$  torr. The gauge has an RS-232 interface to the control computer.

**Optical system:** There is a long-working distance objective, coaxial illuminator, and video camera system for observing the diamond cell through the optical window in the upper clover leaf.

**PLC:** The operation of the air supply to the compressor and the air supply to all 7 valves is controlled by a Programmable Logic Controller (PLC, Automation Direct Model DL205). The PLC ensures that valves are opened and closed in the correct sequence, and ensures basic system safety. For example, it will open the vent valves before the burst disks fail, and will turn off the compressor if the output pressure exceeds a maximum allowed value. The PLC inputs include the status of each valve and the cabinet door switches. It accepts requests over Ethernet from the control computer to operate valves or the compressor. The PLC outputs control air cylinders to operate the valves and the compressor. It sends the status of each of the valves over Ethernet to the control computer. The PLC also controls the pneumatic cylinders that raise and lower the bottom plug of the pressure vessel. It also controls the laser interlock for the ruby fluorescence system. The operation of the PLC is discussed in detail in the Interlocks section below.

**Control computer:** There is a Windows control computer that is the primary user interface to the gas loading system. It displays the pressure in the low and high-pressure systems, the status of the compressor, and the status of all valves. Users change modes (e.g. vacuum pump down, vent, pressurize vessel) through this computer, which sends requests to the PLC. The PLC is responsible for executing the operations if it is safe to do so. The control computer also operates the stepper motors to drive the cell closure mechanism. The control software is based on the EPICS control system.

**Motor controller and driver:** The diamond cell is closed via stepper motor driven rotary feedthroughs into the pressure vessel. A 4-channel stepper motor controller (ACS MCB-4B) is used to control and drive the stepper motors. The controller is run by the control computer over an RS-232 connection.

**Ethernet to serial converter:** There are 4 RS-232 serial devices in the system: Omega pressure meters (2); vacuum gauge; and motor controller. A 4-port Ethernet to serial server (Moxa NPort 5410) is used to provide these serial ports.

**Ruby fluorescence system:** A online ruby fluorescence system provides pressure measurement through the optical window in the top of the pressure vessel. The system consists of a Class IIIb green laser, a visible light spectrometer, lenses and video camera, and positioning system. A separate safety document for the laser system has been prepared and approved by the Argonne Laser Safety Officer.

**Cabinet:** The entire system except the control computer is mounted in a sturdy cabinet (from Item North America). The cabinet is shown in the photograph in Figure 2. The bottom level of the cabinet houses the compressor, vacuum pump, and valves. The upper left houses the lecture bottle, pressure vessel and optical viewing system. The upper right side contains the controls and displays for the pressure gauges, vacuum gauge, and PLC. In the current design there is electrical equipment in the same cabinet as the pressure vessel. In a future design for hydrogen operation the vacuum pump and all electronics could be mounted in separate cabinet. The only connections to the gas-loading cabinet would be compressed air, vacuum, and the pressure transducer excitation and readout voltages.

The parts of the cabinet that contain high-pressure components (vessel, compressor, tubing, valves) are lined with 3/4" plywood panels. The part of the cabinet containing the pressure vessel has an additional shielding layer of 5/16" aluminum plate. This shielding is sufficient to protect the operators from plausible accidents, such as failure of a pressure fitting, breakage of the sapphire window, or ejection of a rotary feedthrough. The upper and lower cabinet doors have a sturdy latching mechanism, and the cabinet itself is of a sturdy aluminum frame construction. There are switches to sense that the enclosure doors are closed, and the safety system will not allow the high-pressure system to be pressurized when the doors are open. The upper left is also a laser enclosure for the ruby fluorescence system, and the doors must normally be closed to operate the laser.





**Figure 2. Annotated photograph of gas loading system**

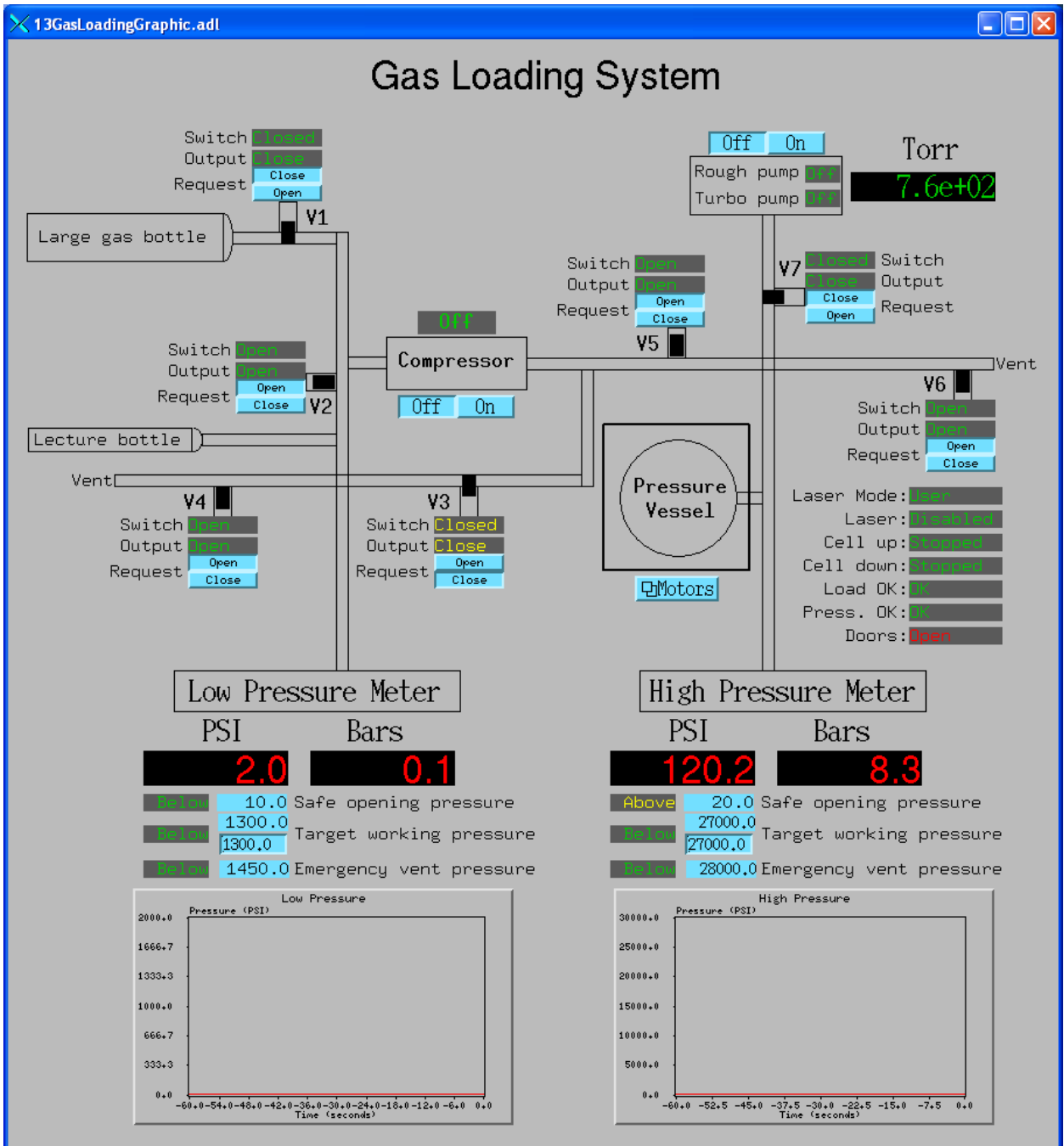


Figure 3. MEDM control screen for gas loading system

## B Safety features

The primary hazard of the system is the stored energy from the high pressure gas. This hazard is minimized by keeping the total volume of the high pressure gas as small as possible. The hazard is also mitigated through a set of engineered controls.

### B.1 Lecture bottle

The stored energy in the system is controlled by the amount of gas that is compressed. The stored energy in the system is reduced by minimizing the empty volume (void spaces) in the high-pressure system. We have designed our diamond cell clamping device to minimize the amount of empty space, thus minimizing the stored energy in the system when it is pressurized.

However, it is possible that a user could forget to install one or more components in the pressure vessel (e.g. the diamond anvil cell, or the clamping device), resulting in a much larger empty volume than normal. If the system was pressurized from a large gas cylinder the total amount of stored energy in the system would be much larger than desired. In order to protect against this, we have designed our system so that the high-pressure system can only be filled using a small “lecture bottle” gas cylinder. With this protection the total amount of gas that can be compressed to 2kbar is small. If the user forgets to install space-filling components then the compressor will simply empty the lecture bottle before the system reaches high pressure.

The amount of gas in a standard size gas cylinder (9”x55”) at 2000 PSI is about 250 scf (standard cubic feet, i.e. cubic feet at 1 atmosphere pressure), or 7075 liters. The amount of gas in a lecture bottle at 1800 PSI (Air Products size LB) is 2 scf or 56.6 liters. We will operate the lecture bottle at 1500 PSI, so the gas volume is  $1500/1800 \times 56.6 = 47.2$  liters.

We estimate the empty space in the system (pressure vessel, tubing and valves) to be  $20\text{cm}^3$ . Assuming the ideal gas law is obeyed, then the volume of gas required to reach 2 kbar from 1 bar is  $20\text{cm}^3 \times 2000 = 40000\text{cm}^3 = 40$  liters. Thus, the lecture bottle with 47.2 liters contains just over the amount of gas required to pressurize the system to 2 kbar. It is not possible to run the compressor with the lecture bottle pressure much less than 500 PSI, so this reserve capacity is needed. The gas is actually less compressible at high pressure than predicted by the ideal gas law, so the lecture bottle has more of a reserve capacity than the above calculation suggests. We will measure the minimum working pressure in the lecture bottle to allow the high-pressure system to reach 2kbar, and will limit the maximum pressure on the low-pressure system to this value.

### B.2 Burst disks

The maximum allowed pressure in the low-pressure system is limited by the rating of the lecture bottle, which is 1800 PSI. The system contains a 1500 PSI burst disk on the low pressure system. This burst disk is connected to the system vent line, and will vent the low pressure system in the case of a malfunction that results in more than 1500 PSI. The burst disk itself is a backup system. The primary protection is the low-pressure transducer, which is connected an Omega meter, which has a low pressure setpoint at 1450 PSI. If the pressure exceeds this setpoint, then the PLC will open the low pressure vent valve, V4.

The maximum allowed pressure in the high-pressure system is limited by the rating of the pressure vessel, which is 30,000 PSI. The system contains a 30,000 PSI burst disk on the high pressure system. This burst disk is connected to the system vent line, and will vent the high pressure system in the case of a malfunction that results in more than 30,000 PSI. The burst disk itself is a backup system. The primary protection is the high-pressure transducer, which is connected an Omega meter, which has a high pressure setpoint at 29,000 PSI. If the pressure exceeds this setpoint, then the PLC will open the high pressure vent valve, V6.

### B.3 Interlocks

The operation of the system is controlled via the PLC. All of the valves are air-operated, as is the system that raises and lowers the bottom plug of the pressure vessel and the diamond anvil cell. The PLC also implements the interlocks for the Class IIIb laser.

Requests to the PLC to operate the valves and other components are made via the Optimate panel buttons mounted on the front of the cabinet, or via the EPICS MEDM display on the control computer. The control computer



communicates with the PLC via the Modbus protocol over Ethernet. The PLC will perform the requested action only if it is safe to do so. The actual state of the valves and other components is displayed on the Optimate panel and on the EPICS MEDM display.

The system will fail into a safe state, with the low pressure and high pressure systems vented and the compressor turned off, on failure of either electrical power or air pressure. This is achieved by using the correct combination of normally open and normally closed high pressure valves.

The ladder logic for the PLC is listed in Appendix 0. The following are the interlock rules that this ladder logic implements:

1. If the Emergency Stop button is pressed, then put the system in a safe state, venting both the low pressure and high pressure systems. Close V1, open V2, close V3, open V4, open V5, open V6, close V7, turn off the compressor, and disable the laser.
2. If the low pressure system is above setpoint 3 on the low pressure meter (1450 PSI) then vent the low pressure system. Open V4, close V1, close V3, and turn off the compressor. This is an emergency vent that should activate before the low pressure burst disk fails.
3. If the high pressure system is above setpoint 3 on the high pressure meter (29000 PSI) then vent the high pressure system, open V6. This is an emergency vent that should activate before the high pressure burst disk fails.
4. If the enclosure doors are not shut then vent the high pressure system (open V6). This protects personnel from being exposed to any components that are at pressures greater than 1500 PSI.
5. If the lower plug of the pressure vessel is not in the closed position then vent the high pressure system. This prevents pressurizing the pressure vessel when the plug is not in the correct position.
6. If both V3 and V5 are closed then turn off the compressor. This prevents pressurizing the tubing between these valves, which is would not be protected by a burst disk if both valves were closed.
7. If the high pressure system is above setpoint 2 on the high pressure meter (normally 28000 PSI) then turn off the compressor. This prevents over-pressurizing the high pressure system without venting it.
8. If the low pressure system is above setpoint 2 on the low pressure meter (normally 1450 PSI) and V3 is open (so we are pressurizing the low pressure system) then turn off the compressor. This prevents over-pressurizing the low pressure system without venting it.
9. Close V1 if V6 is closed. This rule has two effects:
  - a. Prevent pressurizing gas from the large gas cylinder directly into the pressure vessel. Only the lecture bottle can be the gas source for the pressure vessel.
  - b. Prevent refilling the lecture bottle if the high pressure system is pressurized.
10. If the enclosure doors are open and the laser switch is not in Expert Mode then disable the laser. This prevents laser light from escaping from the enclosure.
11. If the high pressure system is above setpoint 1 of the high pressure meter (normally 30 PSI) then close V7. This protects the vacuum pump from damaging high pressure.

## **B.4 Other hazards**

There are two additional hazards in the system.

- 1) Laser light. There is a class IIIb laser used for the ruby fluorescence system. A separate safety document for the laser system has been prepared and approved by the Argonne Laser Safety Officer.
- 2) Pinch hazard. The pneumatic actuator that raises and lowers the bottom plug of the pressure vessel has the potential to harm fingers if they were placed near the top or bottom of the travel range of this device. This hazard is mitigated as follows: the only way to raise or lower the plug is via pressing the buttons on the Optimate panel. This panel is on the right-hand side of the cabinet, far enough away from the pressure vessel that it is not possible to place ones fingers in harms way. This requires a “one-man” rule for raising and lowering the plug, which is included in the standard operating procedures.

## C Stored Energy Analysis

### C.1 Stored energy

The Ideal Gas Law can be written as

$$PV = nRT$$

Where P is pressure, V is volume, n is the number of moles of gas, R is the gas constant ( $8.314 \text{ m}^3 \cdot \text{Pa K}^{-1} \text{ mol}^{-1} = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ ), and T is absolute temperature.

At 1 atmosphere pressure and 298 K the volume of a mol of an ideal gas is thus

$$\begin{aligned} V &= nRT/P = (8.314 \text{ m}^3 \cdot \text{Pa K}^{-1} \text{ mol}^{-1} * 298 \text{ K} * 1 \text{ mol}) / (101325 \text{ Pa}) \\ &= 0.024451 \text{ m}^3 \\ &= 24451 \text{ cm}^3 \\ &= 24.451 \text{ liters} \\ &= 0.856 \text{ ft}^3 \end{aligned}$$

The ideal gas law predicts that pressure and volume are inversely proportional. At low pressures nearly all gases follow the ideal gas law. At high pressures real gases deviate from the ideal gas law because the size occupied by the molecules themselves becomes comparable to the total volume occupied by the gases.

Bridgeman (1923) reported the volume of He at  $65^\circ$  and 3000 kg/cm<sup>2</sup> (2940 bar) as 22.16 cm<sup>3</sup>/mol. The ideal gas law predicts

$$24451/2940 = 8.32 \text{ cm}^3.$$

So He is compressed 2.66 times less at 2940 bar than would be predicted by the ideal gas law. The ideal gas law provides an upper limit on the compressibility of the gases that will be used in our apparatus. Because of this, the ideal gas law also provides an upper limit on the stored energy contained in a gas at a specific volume and pressure.

The energy of an material as pressure is increased at constant temperature is:

$$E_2 = E_1 + \int_{p_1}^{p_2} V dp$$

For an ideal gas this becomes:

$$E_2 = E_1 + nRT \ln(p_2/p_1)$$

The increase in energy as the pressure of 1 mol of gas is increased from 1 bar to 2000 bar at 298K is thus:

$$E_2 - E_1 = 1 * 8.314 * 298 * \ln(2000/1) = 18832 \text{ J} = 18.8 \text{ kJ}.$$

The volume of the lecture bottle is 47.2 liters, which is thus  $47.2/24.45 = 1.93$  mol. The stored energy in our system when it is pressurized to its maximum pressure is thus  $18.8 \text{ kJ} * 1.93 = 36.3 \text{ kJ}$ .

This energy can be compared to the muzzle energy of a 7.6mm rifle bullet, which is about 3.5kJ. The stored energy in our system is thus equivalent to  $36.3/3.5 = 10.4$ . So the stored energy about 10 times greater than that of a rifle bullet.

The energy can also be compared to a stick of dynamite which has about 2500kJ. The stored energy in our system is thus equivalent to  $36.3/2500 = 0.015$ . So the stored energy is less than that in 1/50 of a stick of dynamite.

Another comparison is to the stored energy in a standard gas cylinder (9"x55") at 2000 PSI (138 bar). The content of the cylinder is 250 scf = 7075 liters. This is thus 282 mols of an ideal gas.

The energy content of the cylinder is thus:

$$E_2 - E_1 = 282 * 8.314 * 298 * \ln(138/1) = 3.44 \text{ MJ}.$$

The stored energy of a standard gas cylinder is thus 95 times larger than the stored energy in our high pressure system.

The conclusion is that the stored energy is large enough to propel fragments with high velocity if there were to be a failure of a high pressure fitting. We have placed the apparatus in a cabinet to shield against such hazards in the unlikely event of a failure. However, the energy is small compared to standard compressed gas cylinders that are in routine use at the APS.

## C.2 Failure Analysis

### C.2.1 Velocity and energy of worst-case projectile

If a part of the pressure vessel or other high-pressure component were to fail it would cause one or more projectiles to be propelled at high speed. Here we consider what we believe to be a worst-case scenario for such a failure.

The velocity that a component would obtain is proportional to the force acting on it, and the length of time that the force is applied. Components that are “constrained” by being inside a tube (like the barrel of a gun) are accelerated for longer before the gas can escape past them. In our system the rotary feedthrough rods on the bottom plug of the vessel are the pieces which are most constrained in this manner, and hence are the “worst-case” failure points.

These feedthrough rods have the following properties:

Material: Stainless steel

Length: 8.375” (213 mm, 0.213m)

Diameter: 3/16” (4.76mm, 0.00476 m)

Cross-section area:  $\pi*(4.76/2)^2 \text{ mm}^2 = 17.8 \text{ mm}^2 = 1.78 * 10^{-5} \text{ m}^2$

Volume:  $213*\pi*17.8 \text{ mm}^3 = 3790 \text{ mm}^3 = 3.79 \text{ cm}^3 = 3.79*10^{-6} \text{ m}^3$

Density: 8000 g/m<sup>3</sup>

Mass:  $8000 \text{ g/m}^3 * 3.79*10^{-6} \text{ m}^3 = 30.3 \text{ g} = 0.0303 \text{ kg}$

The length of the hole in the pressure vessel plug is 6.81” = 173mm = 0.173m.

If the rod were suddenly to lose all of the confining force which holds it in place, and have zero friction against being expelled from the vessel, it would accelerate for the time it takes it to travel out of the pressure vessel plug, a distance of 0.173m.

The force acting on the rod is the pressure times the area. The maximum pressure is 2000 bar, or  $2*10^8 \text{ Pa} = 2*10^8 \text{ kg m}^{-1} \text{ sec}^{-2}$ . Multiplying by the cross-sectional area of the rod yields the force:  
 $2*10^8 \text{ kg m}^{-1} \text{ sec}^{-2} * 1.78 * 10^{-5} \text{ m}^2 = 3.56 * 10^3 \text{ kg m}^1 \text{ sec}^{-2}$

We assume that the force is constant during this acceleration, because increase in volume of the gas (about 3.8 cm<sup>3</sup>) as it ejects the rod is small compared to the total gas volume estimated previously (20cm<sup>3</sup>).

The acceleration will be the force divided by the mass:

$$3.56 * 10^3 \text{ kg m}^1 \text{ sec}^{-2} / 0.0303 \text{ kg} = 1.17 * 10^5 \text{ m sec}^{-2}$$

The distance traveled in a given time at constant acceleration is:

$$d = a*t^2/2.$$

The velocity as a function of time is

$$v = a*t$$

Eliminating time (t), and solving for v yields:

$$v = \text{sqrt}(2*d*a) = \text{sqrt}(2 * 0.173\text{m} * 1.17 * 10^5 \text{ m sec}^{-2}) = 201 \text{ m/s}$$

So the velocity of 201 m/s. This is about 3-5 times less than the velocity of a rifle bullet.

The kinetic energy is:

$$\frac{1}{2} * m * v^2 = \frac{1}{2} * 0.0303 \text{ kg} * (201 \text{ m/s})^2 = 612 \text{ J}.$$

This is about 6-8 times less than a rifle bullet.

### C.2.2 Penetration depth of projectile in aluminum

Given this analysis of the velocity and kinetic energy it is possible to estimate the penetration depth in the aluminum shielding plates for the system. The thickness of the aluminum is 5/16", or 8mm.

Tanaka et al.<sup>1</sup> measured the penetration depths and crater volumes for steel projectiles impacting aluminum plates at projectile velocities from 500 to 1800 m/s. This is 2.5 to 9 times greater than the velocity estimated above.

They give their results in terms of the normalized properties:

Equivalent projectile diameter

d = diameter of equivalent sphere for same mass as the projectile

Normalized penetration depth:

$P_n = P/d$  (P= actual penetration depth)

Normalized impact velocity

$V_n = V/c_L$  (V= actual velocity;  $c_L$  = longitudinal sound speed of aluminum target = 6100 m/s)

For our system we determine the normalized projectile diameter (diameter of sphere of same mass) from the known volume of the rod (3.79 cm<sup>3</sup>). The equivalent radius is thus:

$$3.79 = 4/3 * \pi * r^3$$

$$r = (3.79 / (4/3 * \pi))^{1/3} = 0.97 \text{ cm.}$$

The equivalent diameter is thus  $0.97 * 2 = 1.94 \text{ cm.}$

The normalized impact velocity is

$$V_n = (201 \text{ m/s}) / 6100 \text{ m/s} = 0.033$$

Using equation 4 from Tanaka et al.

$$P_n = 27.3 * V_n^{1.58} = 27.3 * (.033)^{1.58} = 0.125$$

Thus, their results predict a normalized penetration depth of 0.125. Multiplying by the equivalent projectile diameter (1.94 cm) yields 0.24cm predicted penetration depth, or 2.4 mm. Since the actual thickness of the aluminum shielding on our enclosure is 8mm, we conclude that the shielding is sufficient to stop the worst-case projectile.

Tanaka also plotted a relationship between the volume of the impact crater and the kinetic energy of the projectile (from his Figure 13). The equation of the room-temperature data in this plot can be well-fitted by the equation:

$$V \text{ (cm}^3\text{)} = 0.5 * \text{energy (kJ)}$$

Since the kinetic energy was calculated above as 612 J (0.612 kJ) we can compute the expected crater diameter:

$$V \text{ (cm}^3\text{)} = 0.5 * 0.612 = 0.306 \text{ cm}^3.$$

The equivalent radius of the rod is 0.97cm. The depth of the crater (P) can thus be calculated as:

$$\text{Volume} = 0.306 \text{ cm}^3 = P * \pi * r^2 = P * \pi * 0.97^2$$

$$P = 0.306 / (\pi * 0.97^2) = 0.103 \text{ cm} = 1.03 \text{ mm.}$$

This again leads to the conclusion that the 8 mm aluminum panels are sufficiently thick.

### C.2.3 Catastrophic venting

---

<sup>1</sup> Tanaka, K. M. Mishida, N. Takada, "High-speed penetration of a projectile into aluminum alloys at low temperatures", International Journal of Impact Engineering, **33** (2006) 788-798.

Another accident scenario that must be considered is the near instantaneous venting of the pressure vessel inside the enclosure. If the volume of gas is too large then this could result in an overpressure inside the enclosure before the gas can escape, which could blow out the doors or panels.

The dimensions of the part of the enclosure holding the pressure vessel are  $1.07\text{m} * 0.86\text{m} * 0.37\text{m} = 0.34\text{m}^3 = 340$  liters. The volume of the gas in the lecture bottle is 47 liters at 1 atmosphere. Thus, all of the gas were to be instantaneously vented there would be an extra 47 liters of gas in the 340 liters of the enclosure. This would raise the pressure by  $(340+47)/340 = 1.138 = 13.8\%$  above normal pressure. The force per is thus

$$10^5 \text{ Pa} * 0.138 = 1.38 * 10^4 \text{ N/m}^2$$

The dimensions of the doors are  $1.07\text{m} * .43\text{m} = 0.46\text{m}^2$ . The worst-case force on the door is thus

$$0.46\text{m}^2 * 1.38 * 10^4 \text{ N/m}^2 = 6348 \text{ N} = 1427 \text{ lb.}$$

The door closure is comprised of two 8mm steel pins on the top and bottom of the door. We assume an ultimate tensile strength for the steel of 500 MPa and taking the shear strength as 75% of the tensile strength, then the shear strength for an 8mm diameter rod (4mm radius) is

$$0.75 * 500 * 10^6 \text{N/m}^2 * \pi * (.004\text{m})^2 = 18849 \text{ N.}$$

The force on each rod is less than 3200 N, which is much less than the shear strength of the rods.

The door hinges are held in place by six ¼-20” screws. Using a similar calculation to that above the tensile strength for a 6.3 mm diameter screw (3.1mm radius) is:

$$500 * 10^6 \text{N/m}^2 * \pi * (.0031\text{m})^2 = 15095 \text{ N.}$$

The force on each screw is  $6348/6 = 1058 \text{ N}$ . The force is thus much less than the tensile strength of the screws.

#### **C.2.4 Projectile impact into door**

We can calculate the force on the door if the worst-case projectile above were to impact the door. In order to compute the force we need to compute the time it would take for the projectile to decelerate from its initial velocity to 0. We make the simple assumption of constant deceleration from the initial velocity to 0 over the distance that the projectile penetrates the aluminum plate. The penetration depth in the aluminum plate was estimated at between 1 and 2.4 mm above. The largest force (maximum deceleration) occurs for the shortest penetration (1mm), so we will assume this value.

The initial velocity is 201 m/s. The average velocity as the projectile penetrates the aluminum (assuming constant deceleration) is thus 100 m/s. Since the distance traveled at this average velocity is 1mm, we can calculate the time to decelerate :

$$\text{time} = 10^{-3}\text{m} / (100 \text{ m/s}) = 10^{-5} \text{ s}$$

$$\text{The acceleration is thus } 200\text{m/s} / (10^{-5}\text{s}) = 2 * 10^7 \text{ m/s}^2$$

Using  $F=M*a$ , and knowing that the mass of the projectile is (0.0303 kg) the force can be computed as

$$F = 0.0303 \text{ kg} * 2 * 10^7 \text{ m/s}^2 = 606000 \text{ N}$$

This calculation would seem to indicate that the force is extremely high, large enough to break the rods and screws whose strength is 30-40 times less than this value. However this assumes that the door panels are infinitely rigid and can transmit the force to the hinges and rods. In fact what happens is that the door will deform, which extends the time of the impulse by the time it reaches the hinges and rods, so that the force is much less. This is very difficult to compute.

However, a simple computation is based on conservation of momentum. If all of the momentum of the projectile is transferred to the door (worst case assumption) and if the door was completely unconstrained (as if the latch were not locked) how fast would the door open?

The mass of the door can be estimated from the mass of the aluminum and plywood panels. The area of the door is  $0.46 \text{ m}^2$ . The thickness of the aluminum is 8mm, so the volume of the aluminum is thus  $0.46\text{m}^2 * .008 \text{ m} = 3.68 * 10^{-3}\text{m}^3$ . The density of aluminum is  $2750 \text{ kg/m}^3$  so the mass of the aluminum panel is 10.1 kg. The plywood is about 17mm, so the volume of plywood is  $7.82*10^{-3}\text{m}^3$ . The density of birch plywood is about  $750 \text{ kg/m}^3$ , so the mass of the plywood panel is 5.9 kg. The total mass of the door is thus  $10.1 + 5.9 = 16.0 \text{ kg}$ .

Conservation of momentum dictates that the velocity of the projectile times its mass is the same as the velocity of the door (V) times its mass.

$$201 \text{ m/s} * 0.0303 \text{ kg} = V * 16.0 \text{ kg}.$$

$$V = 201 \text{ m/s} * .0303 / 16.0 \text{ kg} = 0.38 \text{ m/s}.$$

The velocity of the door is thus only 0.38 m/s. This is so slow that it would not cause any personnel injury if someone were to be struck by a door opening at this speed. Thus, even under the worst-case assumption that the door latch was not fastened or broke from the impact of the projectile (extremely unlikely), the mass of the door is so large compared to the projectile that its velocity is harmlessly low.

## D Standard Operating Procedures

The following are the rules and procedures to be followed when operating the gas loading system.

### D.1 Authorized Personnel

Only persons who have been trained in the use of the gas loading system, and whose names are on the list of Authorized Users may use the gas loading system. Users must agree that they understand this document, and will following these standard operating procedures.

In order to use the laser on the ruby fluorescence system users must also read the standard operating procedures for the laser, and have their names on the separate list of authorized users for the laser system.

### D.2 Personnel Protective Equipment

Users must wear safety goggles when any component of the gas loading system is pressurized above 1500 PSI.

### D.3 Restricting access and posting

Before undertaking any operation which involves turning on the compressor the following must be done:

- Position temporary barriers to restrict access to the area around the gas loading system to authorized personnel only. These barriers must include signs warning that high-pressure gas loading operations are in progress and that access is restricted to authorized personnel only.

- 

### D.4 Allowed gases

The only gases that may be used in the system are the following noble gases:

- He
- Ne
- Ar
- Kr
- Xe
- N<sub>2</sub>
- CO<sub>2</sub>

Only non-radioactive isotopes of these gases may be loaded.

Other gases may be allowed in the future, and these will be added to this list. It is forbidden to load any gas that is not listed in this section.

### D.5 Installing or pressurizing a lecture bottle

The pressure vessel is pressurized from the lecture bottle with the compressor. The lecture bottle may be filled by any of the following 3 methods.

- 1) Install a new lecture bottle. Remove the existing lecture bottle and install a new one. The lecture bottle cannot contain more than 1500 PSI because this is the rating of the burst disk on the low pressure system. If it is a new bottle from a vendor it may contain up to 1800 PSI, and so will need to be connected to a regulator and some gas removed before installing it in the system.
- 2) Fill the lecture bottle directly from the large gas cylinder. If the large gas cylinder contains sufficient pressure (typically 1500 PSI or more) then the lecture bottle can be filled as follows. Ensure that the regulator on the laser gas cylinder is set to 1500PSI or less; install the lecture bottle (if not already installed); close the low pressure vent (V4), close V3, open V2, open V6, and finally open V1. This will fill the lecture bottle to the pressure controlled by the regulator on the large gas cylinder. When the lecture bottle has finish filling (low pressure gauge has stopped increasing) then close V1.
- 3) Fill the lecture bottle from the large gas cylinder with the compressor. If the large gas cylinder does not contain sufficient pressure to fill the lecture bottle directly (typically less than 1500 PSI) then the lecture bottle can be filled



using the compressor as follows. Ensure that the regulator on the laser gas cylinder is set to 1500PSI or less; install the lecture bottle (if not already installed); close the low pressure vent (V4), close V5, close V2, open V3, close V6, and open V1. Set the target working pressure on the low pressure system to the desired value (typically 1450 PSI). Turn on the compressor. This will fill the lecture bottle to the pressure controlled by the target working pressure. When the lecture bottle has finished filling turn off the compressor, close V1, and close V3.

## D.6 Loading the cell into the pressure vessel

To load a cell into the pressure vessel follow these steps.

- Lower the bottom plug of the pressure vessel if necessary. This is done by rotating the bottom plug to the left so that the “Load OK” switch indicates closed on the MEDM screen. Then press the Cell Down button on the Optimate panel, lowering the bottom plug with the air cylinders. **Only the person pressing the Cell Down button is allowed to be near the apparatus when lowering the plug, to avoid pinched fingers.**
- Place the cell into the cell holder. Be sure to install the springs and any filler pieces.
- Place the cell holder on top of the bottom plug, aligning the hex socket screws to the rotary feedthroughs.
- Raise the bottom plug and cell holder into the pressure vessel. Make sure the bottom plug is still rotated to the left so that the “Load OK” switch indicates closed on the MEDM screen. Then press the Cell Up button on the Optimate panel, raising the bottom plug with the air cylinders. **Only the person pressing the Cell Up button is allowed to be near the apparatus when raising the plug, to avoid pinched fingers.**
- Once the bottom plug is fully up, release the Cell Up button on the Optimate, and quickly rotate the bottom plug to the right so that the Press. OK switch indicates closed on the MEDM screen. This indicates that the plug is correctly installed, and allows the pressure vessel to be pressurized.

## D.7 Pressurizing the pressure vessel

Once the cell and bottom plug are correctly installed the pressure vessel can be pressurized as follows:

- Close the enclosure doors and verify that the Doors Closed light on the Optimate panel is illuminated.
- Put on safety goggles.
- Close the high pressure vent valve, V6. Close the vacuum pump valve, V7. Open V2 and V5.
- Set the target working pressure for the high pressure system to the desired value, up to a maximum of 28,500 PSI.
- Turn on the compressor. When the pressure reaches 5000 PSI, turn off the compressor and wait for one minute, checking for leaks (high pressure gauge decreasing).
- If leaks are found vent the system (open V6) and fix the leaks.
- If no leaks are found then start the compressor again and pressurize to the desired value. Leave the compressor on so that it keeps the pressure at the desired value, even if there are small leaks.

## D.8 Closing the cell

Once the cell is pressurized it can be closed and sealed as follows:

- Open the motor control screen.
- Rotate the motors together (Together virtual motor) in the positive direction in small increments. 1 unit is 1 full revolution of the closing screws.
- Use the video system and ruby fluorescence system to determine when the cell has sealed and reached the desired pressure.

## D.9 Venting the pressure vessel

Once the cell has been closed and sealed the pressure vessel can be vented as follows:

- Turn off the compressor
- Close V2.
- Open V6.

## D.10 Removing the cell

When the system is fully vented the cell can be removed as follows:

- Lower the bottom plug of the pressure vessel. This is done by rotating the bottom plug to the left so that the “Load OK” switch indicates closed on the MEDM screen. Then press the Cell Down button on the

Optimate panel, lowering the bottom plug with the air cylinders. **Only the person pressing the Cell Down button is allowed to be near the apparatus when lowering the plug, to avoid pinched fingers.**

### **D.11 Emergency Procedures**

The Emergency Stop button can be pressed at any time. This will fully vent the system (opening V4 and V6), turn off the compressor, and disable the laser. If there is a problem then contact Mark Rivers or the APS Floor Coordinator.

## **E Maintenance, Calibration and Inspection**

### **E.1 Calibration checks**

The calibration of the pressure transducers shall be checked every six months. This check can be performed by seeing whether the pressure read by the transducers agrees with that on the gas regulator. For this measurement the pressure on the gas regulator must be at least 1000 PSI, and valves 1, 2, 3 and 5 must be open, and valves 4, 6 and 7 must be closed. The agreement must be within 5%, and if it is outside this range then the pressure transducers and/or gauges must be sent to the manufacturer for recalibration.

The calibration of the gas regulator needs to be checked every 12 months. This check can be performed by connecting the regulator in parallel with another regulator and checking for agreement, or by sending it back to the manufacturer for re-certification. If the check is done with another regulator then the agreement must be within 5%.

### **E.2 Inspection**

The initial assembly and pressure and leak testing of the system was performed by Guy Macha of CARS. Guy is a very experienced mechanical and vacuum technician with many years of experience in vacuum systems. It was verified that the high pressure and low pressure systems did not leak when pressurized to their maximum design pressure. This was measured with a He leak detector when the system was pressurized with He.

Only personnel who have been specifically authorized by Mark Rivers may perform mechanical work on the high pressure components (tubing, valves, compressor). These personnel currently include Guy Macha and Clayton Pullins. Clayton has extensive experience in mechanical design and assembly.

The operation of the interlock system must be checked every 12 months. This check consists of ensuring correct operation of all 11 items in the Interlocks description in section 0.

### **E.3 Maintenance**

The compressor must be maintained according to the instructions in the user manual. This consists primarily of lubrication at scheduled intervals.

No other components have required maintenance, except for the calibration checks described above. The vacuum pump will probably need to be rebuilt periodically when its performance begins to degrade, and this will be performed by qualified personnel, such as Guy Macha.

## E.4 Inspection and Maintenance Log

Item	Frequency	Date	Performed by
<b>Pressure transducer calibration check</b>	<b>6 months</b>		
<b>Regulator calibration check</b>	<b>12 months</b>		
<b>Interlock check</b>	<b>12 months</b>		
<b>Compressor maintenance</b>	<b>6 months</b>		

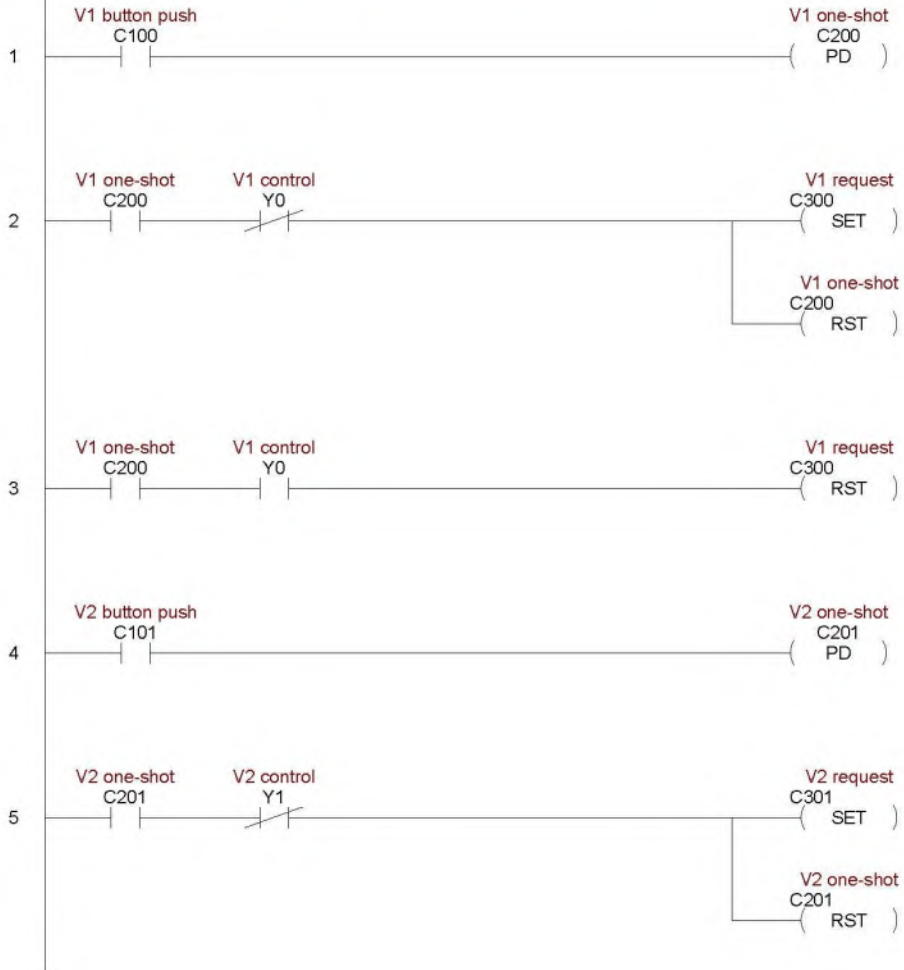
## **F Appendices**

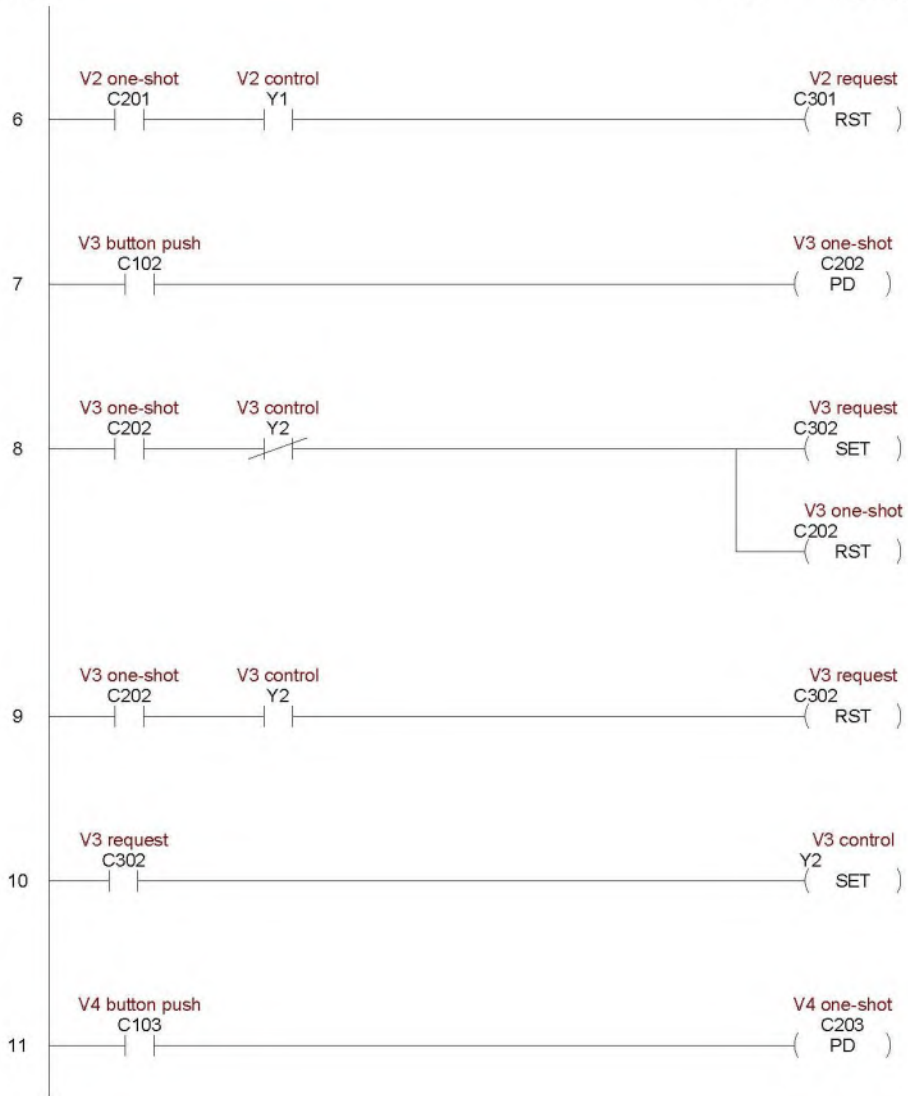
<b>F.1 Ladder logic program</b>	<b>249</b>
<b>F.2 ANL Form 722 Pressure System Evaluation Form</b>	<b>263</b>
<b>F.3 Manufacturers certification documents</b>	
<b>F3.1 Pressure vessel</b>	
<b>F3.1.1 Pressure testing certification</b>	<b>267</b>
<b>F3.1.2 E-mail regarding cloverleaf reactor calculations</b>	<b>268</b>
<b>F3.1.3 Cloverleaf reactor calculations</b>	<b>269</b>
<b>F3.1.4 Window design calculations</b>	<b>274</b>
<b>F3.1.5 Material test report for cloverleaf reactor 4340 steel</b>	<b>276</b>
<b>F3.1.6 E-mail certifying that no additional heat treatment was done on 4340 material</b>	<b>277</b>
<b>F3.1.7 Mechanical drawings of cloverleaf reactor</b>	<b>278</b>
<b>F3.2 Pressure transducers</b>	<b>279</b>

10/17/2007

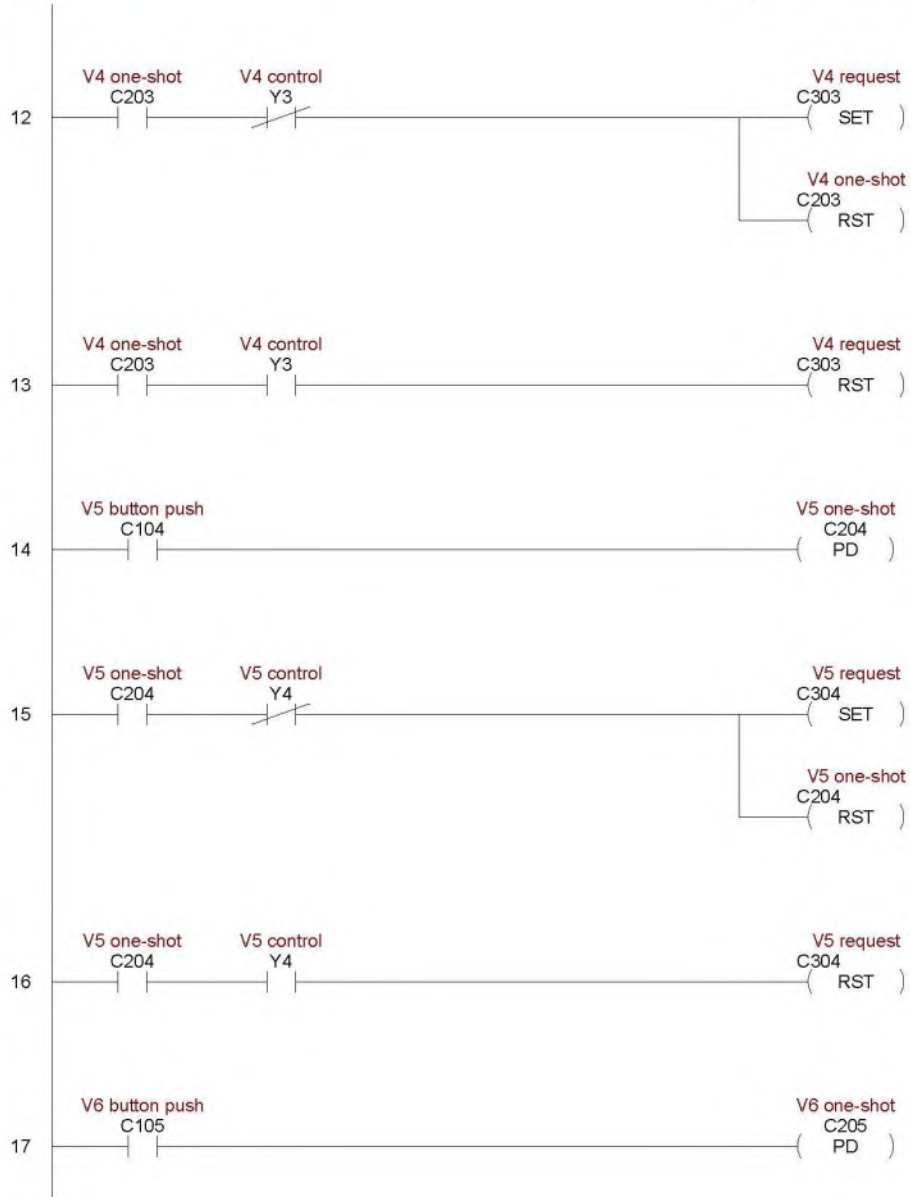
Path: p:\epics\dirctsft (gse laser interlock plc)\13-gas-loading\13\_gas\_loading.prj  
Save Date: 10/17/07 12:25:54  
Creation Date: 04/28/07 23:52:58  
PLC Type: 240  
Class ID: DirectLogic 205 Series  
Link Name: Ethernet Gas  
Description: Gas loading system PLC

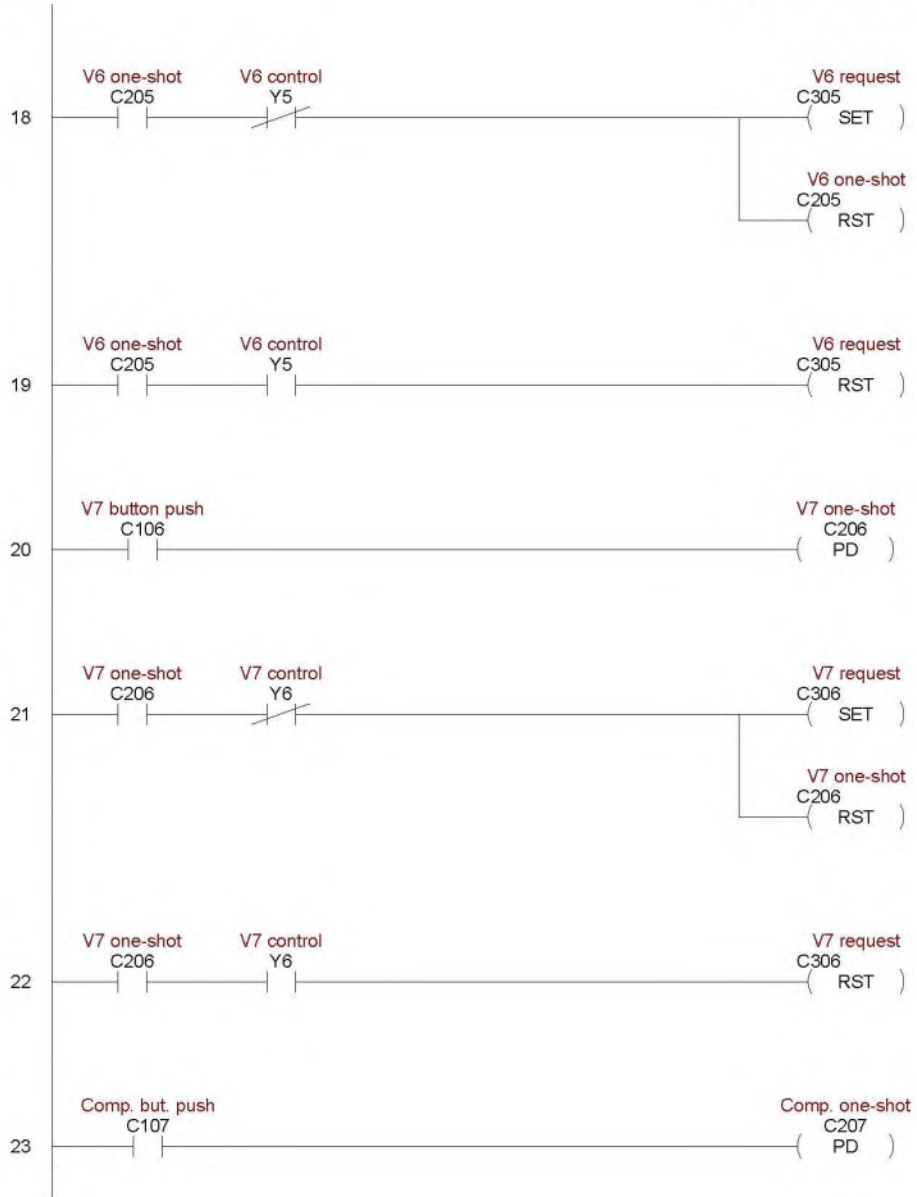
C300-C306 are used for the requested state of valves V1-V7 (outputs Y0-Y7). C307 is the requested state of the compressor (Y7). C310-C311 are the requested state of the mechanical and turbo pumps.  
 Before the ladder logic program is scanned the requested states may have changed from Modbus (EPICS).  
 The ladder logic first implements any requested changes from the Optimate Panel.  
 The ladder logic then changes any requested states to obey the interlock rules.  
 Finally the ladder logic copies the requested state to the actual Y outputs  
 The 0 state is requests the "normal" operation state of the valve. This is normally closed (V1, V7) or normally open (V2, V3, V4, V5, V6).  
 The 0 state turns off the compressor, mechanical pump, and turbo pump.

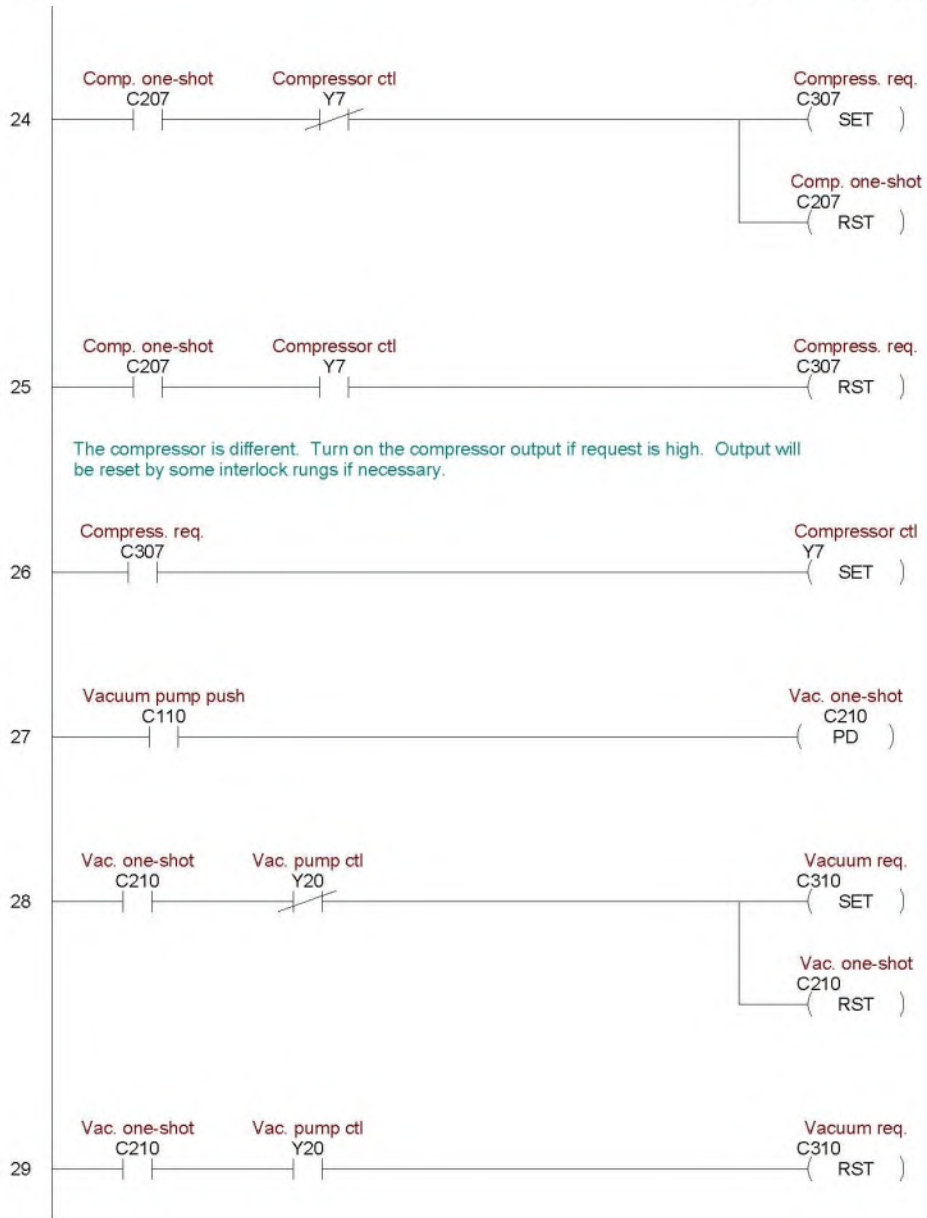


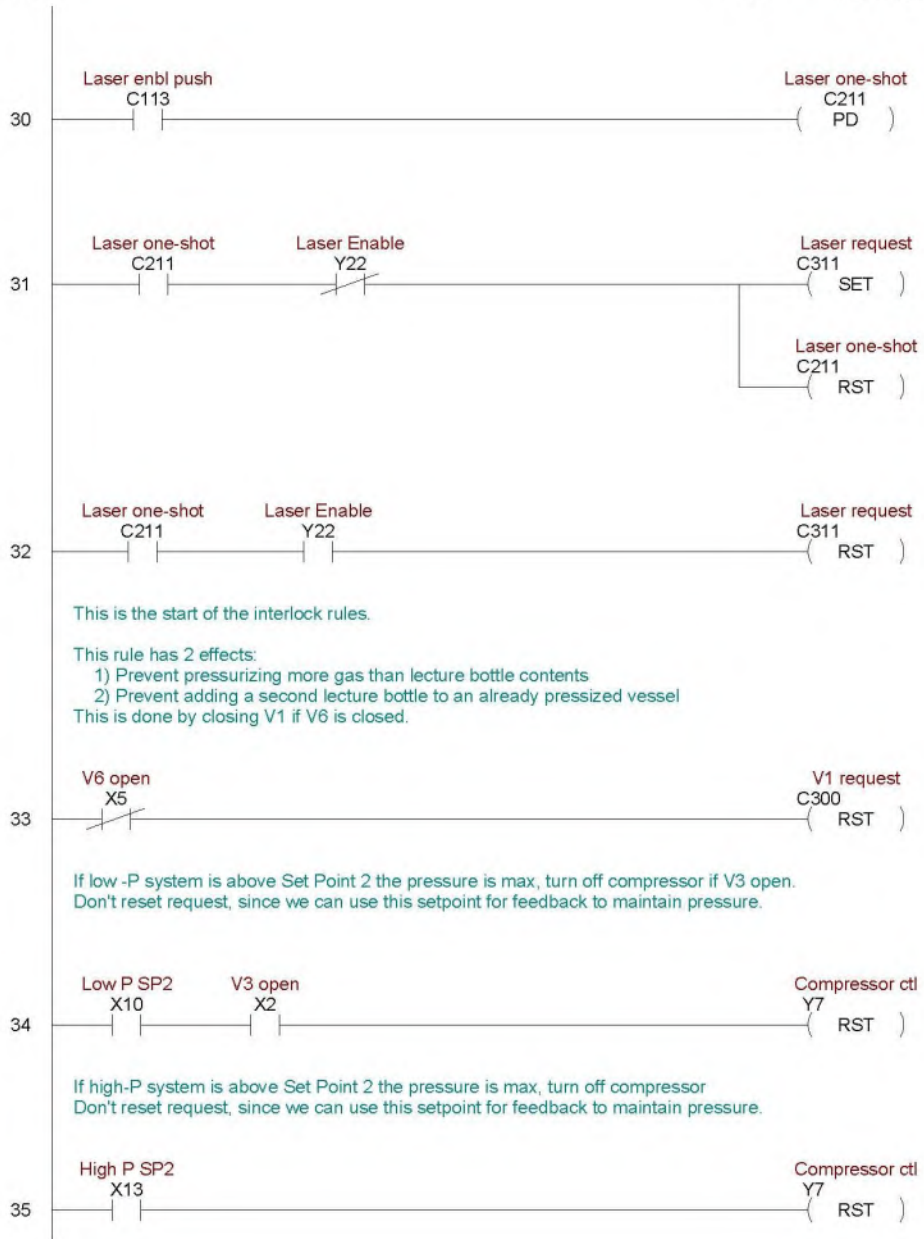


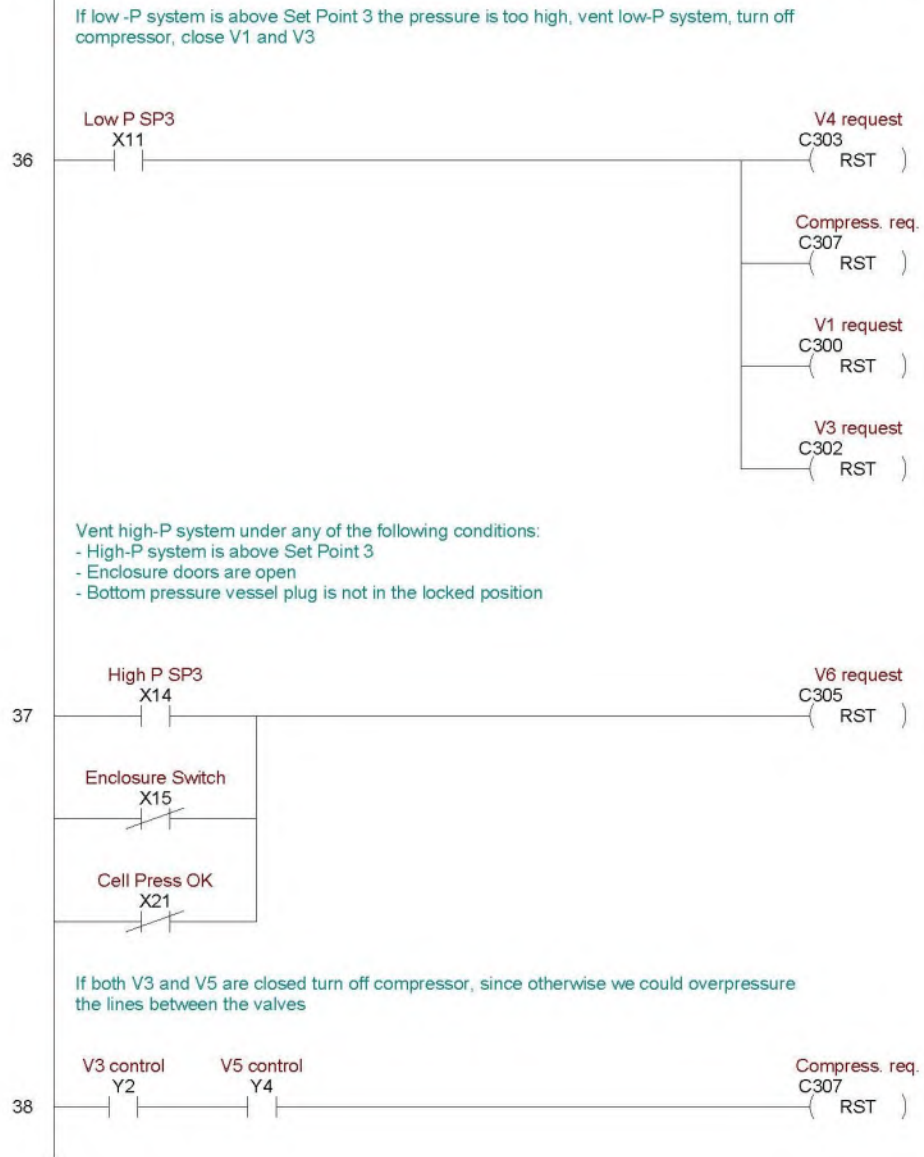


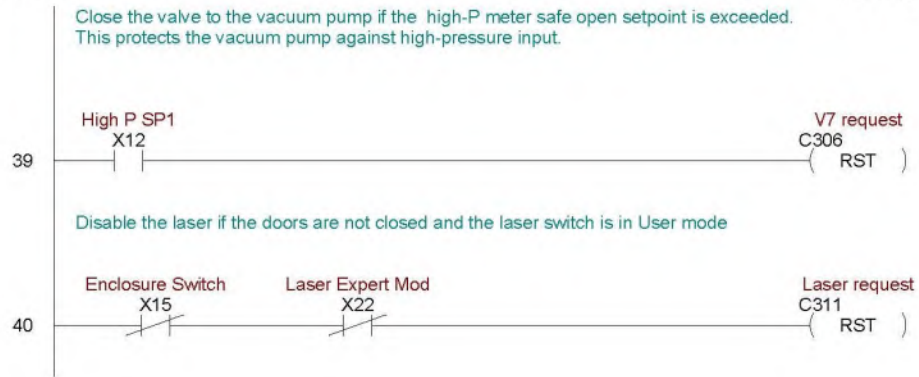




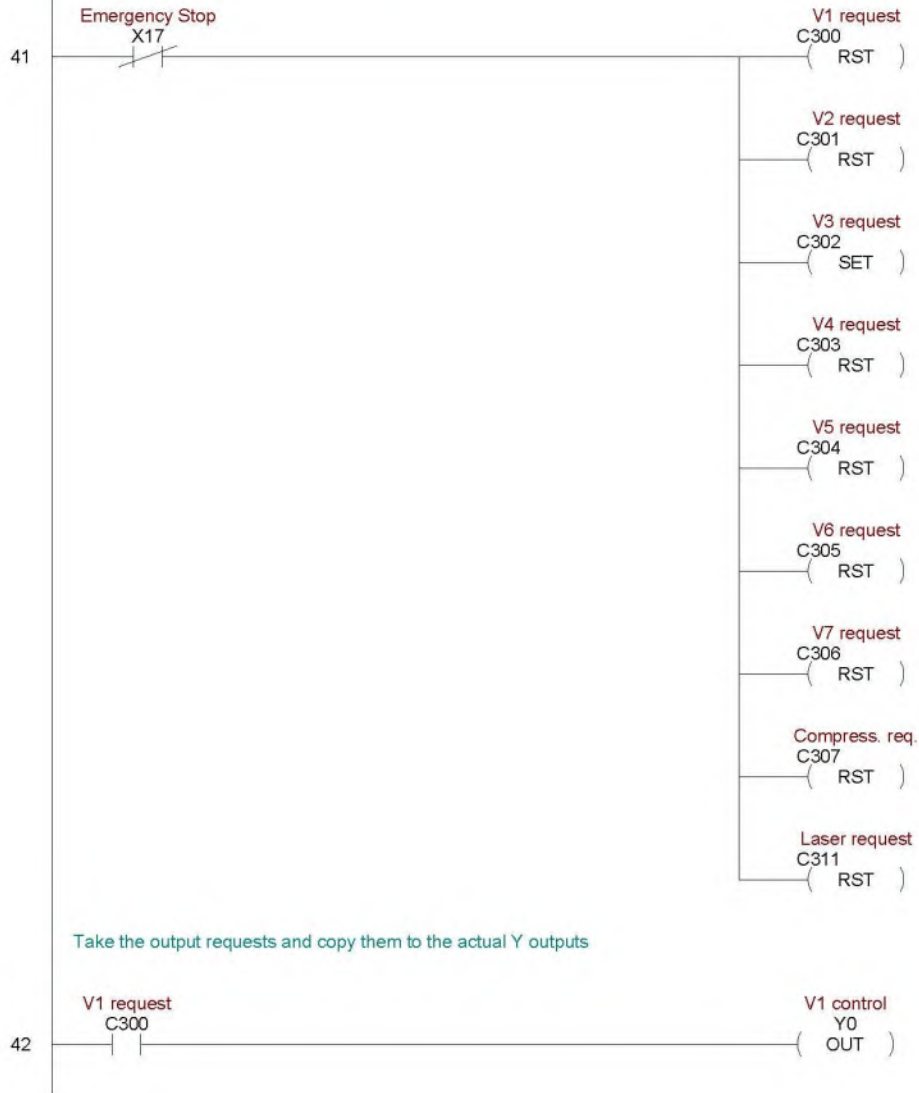


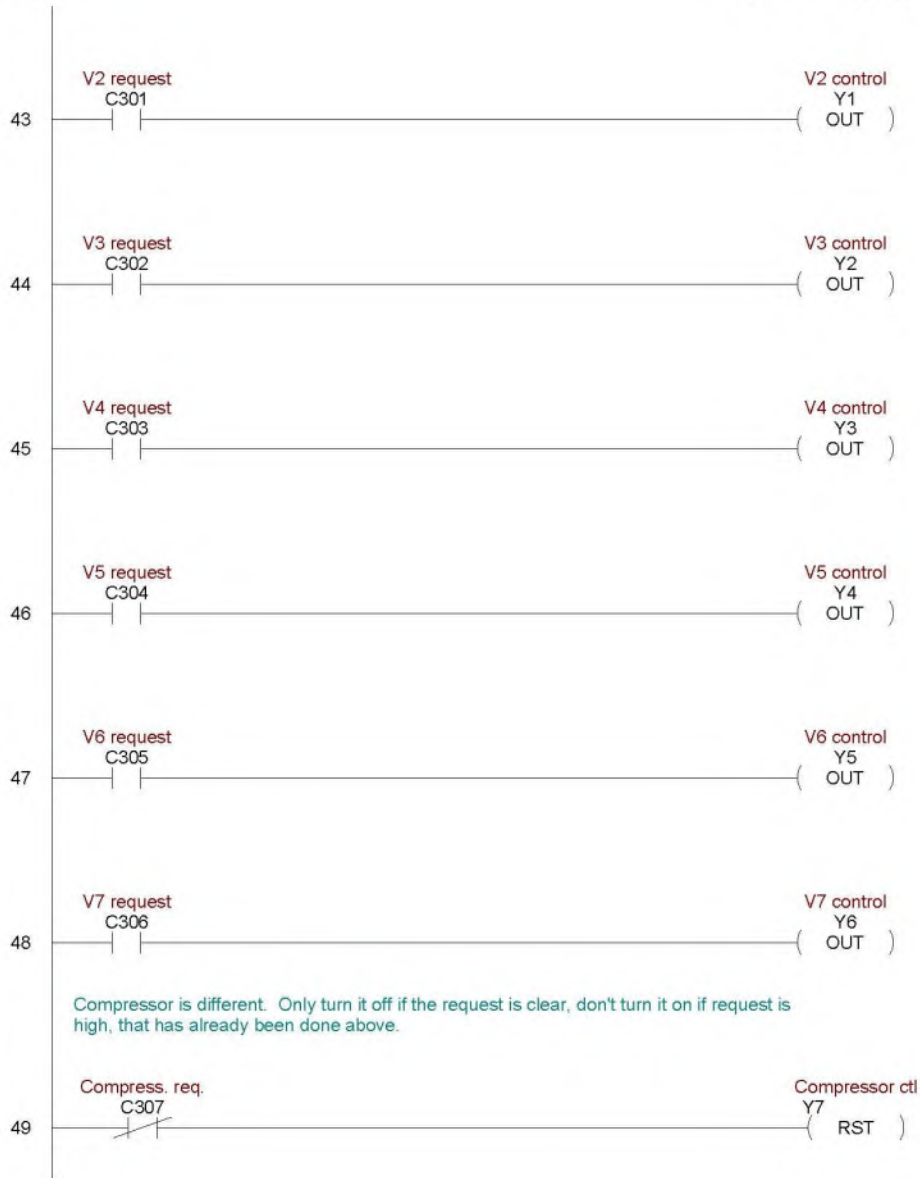




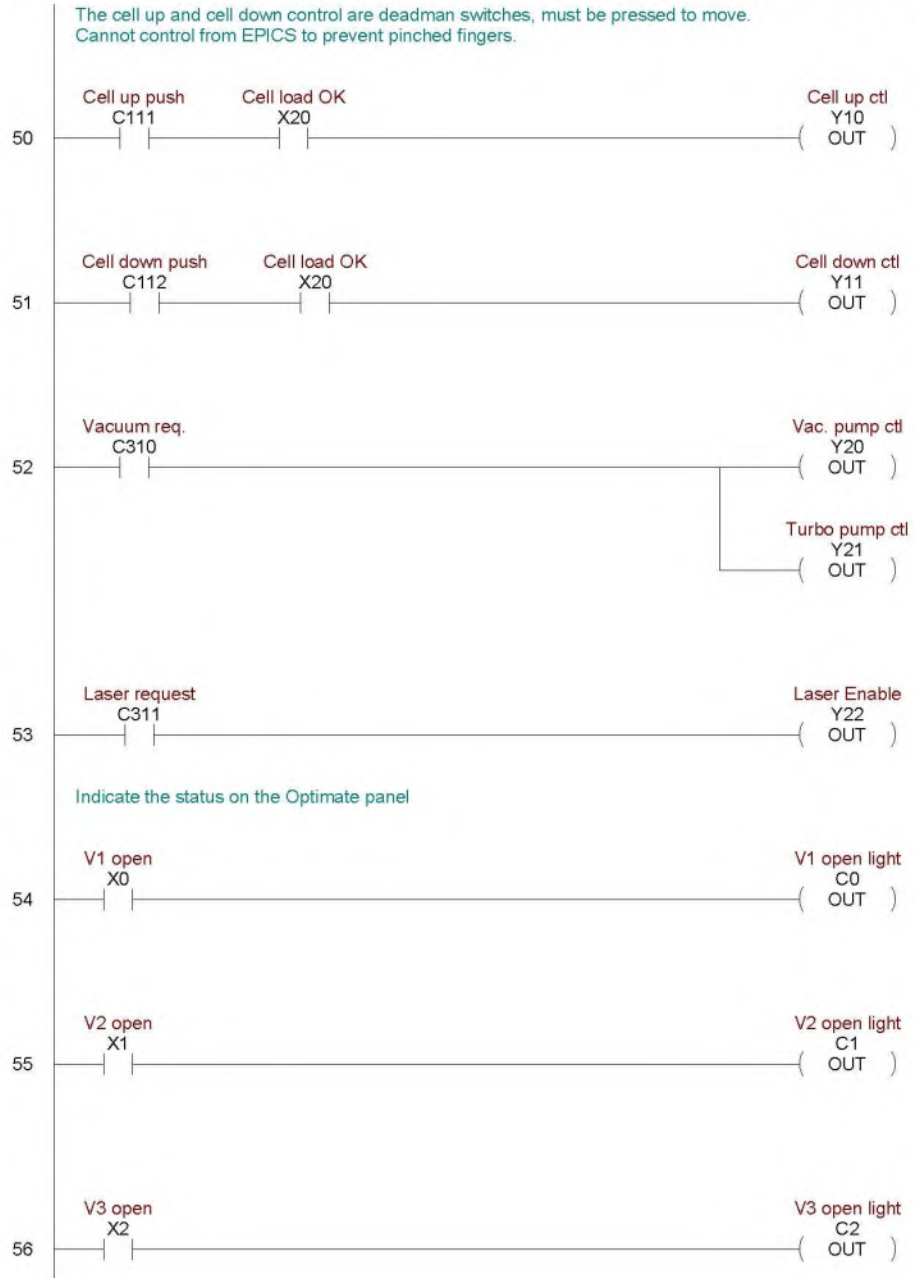


If the emergency stop is pressed then put the system in a safe state. V1 closed, V2 open, V3 closed, V4 open, V5 open, V6 open, V7 closed, compressor off

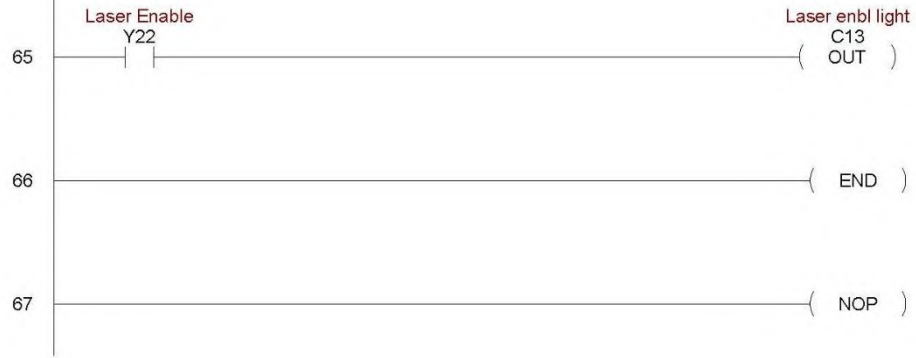














## PRESSURE SYSTEM EVALUATION FORM

Prepared by: Mark Rivers, Joseph Pluth & Clayton Pullins

Date: 10/16/07

System Identification: GSECARS/COMPRES Gas Loading System

System Location: Sector 13

Responsible Division: GSECARS

Responsible Person: Mark Rivers

System Inspection Interval:

Last System Inspection:

<b>System Description*</b>	Primary: Gas Loading System for Diamond Anvil Cell: Compressor, Air-Actuated Valves, Pressure Lines, Programmable Logic Controller			
	Auxiliary(s):			
<b>System Material(s)</b>	Material Certification: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No Describe Material: Pressure vessel is 4340 steel with vendor certification			
<b>Size (Volume):</b>	~ 30 ml of void space			
<b>Will the system contain radioactive material?:</b>	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No			
<b>Description of Contents</b> <i>(Examples: Nitrogen, hydraulic fluid, air, radioactive corrosive liquid, etc.)</i>	Inert gases (He, Ne, Ar, Kr, Xe)			
<b>Pressure and Temperature Ratings</b>	MAWP (psig)	30,000	Maximum (°F/°C)	70-100 F
	MOP (psig)	30,000	Minimum (°F/°C)	70-100 F
<b>Applicable Code, Standard, etc.</b>	ASME BPV Code Section VIII Division 3 2007			
<b>National Board No:</b>				
<b>Manufacturer</b>	<input checked="" type="checkbox"/> Commercial <input type="checkbox"/> Custom High-pressure components are all commercial.  Manufacturer Names: Compressor: Super Pressure Newport Scientific Pressure vessel, air actuated valves, pressure lines and burst disks: High Pressure Equipment Company Pressure Transducers: GP:50  Manufacturer Date: All components were manufactured in 2007. Manufacturer Serial Numbers: Pressure vessel: 0268324-1 Pressure Transducers: 55784, 55785 Compressor: 52584:			

<b>Pressure Relief Device:</b>	<input type="checkbox"/> Pressure Relief Valve Is it lock-wired? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No	Pressure Relief Setting (psig): PLC vents LP system at 1400 PSI, HP system at 29,000 PSI Rupture disks: LP=1500 PSI, HP=30,000 PSI
	<input checked="" type="checkbox"/> Rupture Disk	
	<input checked="" type="checkbox"/> Other: PLC monitors pressure transducers, opens vent valves on over-pressure	PR Inspection Interval (Years): Last PR Inspection Date:
<b>Available Documentation*</b>	System Design Description: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Commercial manuals for compressor. GSECARS manual for system description	
	Operating & Maintenance Manuals: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Commercial manuals for compressor. GSECARS manual for system description	
	Drawings: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Commercial drawings for pressure vessel and compressor. GSECARS manual for system layout.	
	Other: <input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Describe: Document(s) No.:	
<b>Reviews*</b> <i>(Historical Reviews)</i>	Design: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Document(s) No.: ANL ICMS# APS_1235346	
	Safety: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Document(s) No.: ANL ICMS# APS_1235346	
	Analyses &/or Calculations: <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> Not Verified Document(s) No.: ANL ICMS# APS_1235346	
<b>Safety Evaluation for Operation*</b> <i>(Review Team to perform)</i>	Visual Inspection: Reviewed by Jeff T. Collins (APS/AES/MED) William F. Toter (TSD-CS)  11/14/07	
	Technical Evaluation:	

<b>Failure Risk Analysis*</b>	<p>Other Observation:</p> <hr/> <p>To People:  Consequence: <input checked="" type="checkbox"/> High <input type="checkbox"/> Moderate <input type="checkbox"/> Low <input type="checkbox"/> Negligible  Frequency: <input checked="" type="checkbox"/> Beyond Extremely Unlikely <input type="checkbox"/> Extremely Unlikely  <input type="checkbox"/> Unlikely <input type="checkbox"/> Anticipated</p> <p>Describe: The stored energy at 30,000 PSI is about 30kJ which is sufficient to propel a dangerous projectile. Failure is extremely unlikely, and there is shielding to protect personnel in the case of plausible failure.</p> <hr/> <p>To Equipment:  Consequence: <input type="checkbox"/> High <input checked="" type="checkbox"/> Moderate <input type="checkbox"/> Low <input type="checkbox"/> Negligible  Frequency: <input type="checkbox"/> Beyond Extremely Unlikely <input checked="" type="checkbox"/> Extremely Unlikely  <input type="checkbox"/> Unlikely <input type="checkbox"/> Anticipated</p> <p>Describe: Plausible damage to equipment would be confined to the pressure system itself.</p> <hr/> <p>To the Environment:  Consequence: <input type="checkbox"/> High <input type="checkbox"/> Moderate <input type="checkbox"/> Low <input checked="" type="checkbox"/> Negligible  Frequency: <input type="checkbox"/> Beyond Extremely Unlikely <input checked="" type="checkbox"/> Extremely Unlikely  <input type="checkbox"/> Unlikely <input type="checkbox"/> Anticipated</p> <p>Describe: Small amounts of inert gas would be released to the room, posing no risk.</p>

\*Attach additional document, if needed.

Recommendation: <input type="checkbox"/> Remove from Service <input type="checkbox"/> Continue to Operate
Conclusion Basis:

--

Approved: \_\_\_\_\_ Date: \_\_\_\_\_  
Review Team Leader Signature

Resulting Action:  Remove from Service  Continue to Operate

Explain if Different from Recommendation:          
---

Approved: \_\_\_\_\_ Date: \_\_\_\_\_  
Division Director Signature

Approved: \_\_\_\_\_ Date: \_\_\_\_\_  
Associate Laboratory Director Signature

**High Pressure Equipment Company**  
**Pressure Test Report**

Order No. 0268324                      Date 3/30/07  
Part No. 801930 CL reactor              Design Pressure 30 000 psi  
Test Pressure 35 000 psi              Test Duration 5 minutes  
Medium WATER                      Test Temperature Ambient

Conducted By   
QC 

Notes: University of Chicago PO W065710  
S/N 0268324-1



**From:** John Metalonis **Sent:** Saturday, November 17, 2007 10:43 AM **To:** Tony Pepicello **Subject:** RE: Reactor

Tony,

This vessel is a modification of our standard CL1 reactor that was designed in the mid 60s. There has been no need for modifications to the design since then. The only thing we did was add a closure to both ends. There was no third party review of the design or witness of the hydrotest. The only thing we could offer would be a set of calculations. A sample of the CL1 calculation is attached for reference. The only difference would be that there would be no body blind end analysis since there are closures at both ends.

The only caveat with this particular vessel is that the rods must be secured somehow from flying out due to forces from pressure.

John A Metalonis Manager - Design

High Pressure Equipment Company 1222 Linden  
Avenue Erie PA 16505 USA

814 838 2028 814 838 6075 (fax)

CONFIDENTIALITY NOTICE: The contents of this message (including any attachments) are confidential and may be privileged. If you are not the intended recipient of this message, you are not authorized to read, print, retain, copy, disseminate, distribute or use this message or any part thereof. If you received this message in error, please reply to the sender and delete this message from your system. Thank you

**HIGH PRESSURE EQUIPMENT COMPANY**  
Erie, Pennsylvania USA

Cloverleaf reactor calculations  
Models CL-1, CL-2, CL-3

Design pressure 30 000 psi

Calculations based on ASME BPV Code  
Section VIII Division 3 2007

11/17/2007

Materials of construction

4340 alloy steel with the following minimum room temperature properties

Ultimate tensile strength  $S_t := 135000 \cdot psi$

Yield strength  $S_y := 105000 \cdot psi$

## Design constants

$D_{TC} := 3.105 \cdot in$	Cover shear diameter
$D_I := 3.000 \cdot in$	Inside diameter
$D_O := 6.500 \cdot in$	Outside diameter
$D_{seal} := 3.000 \cdot in$	Seal diameter
$D_U := 4.525 \cdot in$	Body undercut diameter
$L_{TC} := 2.250 \cdot in$	Cover shear length
$P := 30000 \cdot psi$	Design pressure
$S := 45000 \cdot psi$	Allowable stress from pressure
$t_B := 1.625 \cdot in$	Body bottom thickness
$t_W := 0.5 \cdot (D_O - D_I)$	
$t_W = 1.750 \cdot in$	Body wall thickness
$Y := \frac{D_O}{D_I}$	
$Y = 2.167$	OD/ID wall ratio

Maximum operating pressure [KD-221.1]

$$P_{dmax} := \frac{2}{3} \cdot S_y \cdot \ln(Y)$$

$$P_{dmax} = 54123 \text{ psi}$$

Minimum wall thickness [KD-221.1]

Given

$$P = \frac{2}{3} \cdot S_y \cdot \ln\left(\frac{D_I + 2 \cdot t_w}{D_I}\right)$$

$$t_{min} := \text{find}(t_w)$$

$$t_{min} = 0.803 \text{ in} \quad t_w = 1.750 \text{ in}$$

Cover shear stress

$$\tau_c := \frac{P \cdot D_{seal}^2 \cdot \frac{\pi}{4}}{0.5 \cdot D_{TC} \cdot \pi \cdot L_{TC}}$$

$$\tau_c = 19324 \text{ psi}$$

Longitudinal stress at body undercut

$$\sigma_L := \frac{P \cdot D_{seal}^2 \cdot \frac{\pi}{4}}{(D_O^2 - D_U^2) \cdot \frac{\pi}{4}}$$

$$\sigma_L = 12400 \text{ psi}$$

Minimum body bottom thickness [KD-242]

$$t_{bmin} := D_I \cdot \sqrt{\frac{0.33 \cdot P}{\frac{S_y}{1.5}}}$$

$$t_{bmin} = 1.128 \text{ in} \quad t_B = 1.625 \text{ in}$$

Minimum hydrostatic test pressure [KT-311]

$$P_{tmin} := 1.25 \cdot P$$

$$P_{tmin} = 37500 \text{ psi}$$

Maximum hydrostatic test pressure [KT-312.1]

$$P_{tmax} := S_y \cdot \ln(Y)$$

$$P_{tmax} = 81185 \text{ psi}$$

Burst pressure [KD-230(a)]

$$P_b := \frac{2}{\sqrt{3}} \cdot S_y \cdot \left( 2 - \frac{S_y}{S_t} \right) \cdot \ln(Y)$$

$$P_b = 114576 \text{ psi}$$

$$\frac{P_b}{P} = 3.819$$

**WINDOW DESIGN**

Specify:

$$d_p := 0.500 \cdot \text{in} \quad \text{view port diameter}$$

$$D_w := d_p \cdot 3 \quad \text{window OD} \quad D_w = 1.500 \text{ in}$$

$$P := 30000 \cdot \text{psi} \quad \text{design pressure}$$

Design constants:

(Sapphire)

$$\sigma_s := 5000 \cdot \text{psi} \quad (\text{Window material bending stress})$$

$$\nu_s := 0.02 \quad (\text{Window material Poisson ratio})$$

$$E_s := 50 \cdot 10^6 \cdot \text{psi} \quad (\text{Window material elastic modulus})$$

$$k := 4 \cdot 10^{-6} \cdot \frac{\text{in}}{\text{in}} \quad (\text{max deflection/diameter } (y/d) \text{ ratio})$$

$$y := k \cdot d_p$$

$$y = 2.000 \times 10^{-6} \text{ in} \quad (\text{Allowable deflection})$$

Minimum window thickness based on bending stress

$$t_{\text{mins1}} := \text{if} \left( \frac{D_w}{d_p} < 3, \frac{D_w + d_p}{2} \cdot \sqrt{\frac{0.3 \cdot P}{\sigma_s}}, d_p \cdot \sqrt{\frac{0.3 \cdot P}{\sigma_s}} \right)$$

$$t_{\text{mins1}} = 0.671 \text{ in}$$

$$t_{\text{mins1}} = 17.04 \text{ mm}$$

Minimum window thickness based on deflection (From Roark *Formulas for Stress and Strain* 5th ed table 24 cases 10a & 10b.)

$$t_{\text{mins2}} := \text{if} \left[ \frac{D_w}{d_p} < 3, \left[ \frac{3 \cdot P \cdot (D_w + d_p)^4 \cdot (5 - 4 \cdot \nu_s - \nu_s^2)}{4096 \cdot E_s \cdot y} \right]^{\frac{1}{3}}, \left[ \frac{3 \cdot P \cdot d_p^4 \cdot (1 - \nu_s^2)}{256 \cdot E_s \cdot y} \right]^{\frac{1}{3}} \right]$$

$$t_{\text{mins2}} = 0.603 \text{ in}$$

$$t_{\text{mins2}} = 15.33 \text{ mm}$$

Minimum thickness

$$t_{\text{mins}} := \max(t_{\text{mins1}}, t_{\text{mins2}}) \quad t_{\text{mins}} = 0.671 \text{ in}$$

$$\text{Used } t := 1.000 \cdot \text{in}$$



**CERTIFICATE OF MATERIAL TEST REPORTS**



**H-A Industries**  
A Division of A. M. Castle & Co.  
4627 Columbia Avenue  
Hammond, IN 46327

CASTLE METALS FRANKLIN PARK  
3400 N. WOLF ROAD

FRANKLIN PARK IL 60131-1319 USA

H-A INDUSTRIES CERTIFIES THAT APPROPRIATE INFORMATION IS TAKEN FROM TEST REPORTS FURNISHED TO US BY OUR SUPPLIER AND WHICH ARE ON FILE IN OUR OFFICE.

WE CERTIFY THAT WE HAVE NO KNOWLEDGE OF MERCURY OR RADIOACTIVE MATERIAL USED IN THE MELTING OR PROCESSING OF STEEL SOLD BY OUR COMPANY.

MANUFACTURER (MIL)		CUST. ORDER NO.		CUST. REQUISITION NO.	
TIMKEN COMPANY		STOCK-03909			
IAC	HEAT NO.	ORDER NO.	LINE NO.	SHIP QTY.	SHIP DATE
13964	Y8268 G9845	267312	1	6,000.0	

**DESCRIPTION**

6.7500 RD A4340 HR QT SF/SR 285/341 HB SURFAC 18/24

\* \* \* CHEMICAL ELEMENTS \* \* \*

C	MN	P	S	SI	NI	CR	MO	AL
.41	.75	.008	.031	.25	1.81	.79	.27	.038
CU	.25							

\* \* \* MECHANICAL PROPERTIES \* \* \*

TENSILE 143000 PSI, YIELD 123000 PSI, ELONG 20.5, R.A. 59.8  
 HARDNS HB 311/ 312, GRAIN = FINE, GRAIN R.R. 22.0  
 CAST = INGOT, MERCURY FREE, MEETS NAPT

\* \* \* INDUSTRY SPECIFICATIONS \* \* \*

J O M I N Y

1	2	3	4	5	6	7	8	12	16	20	24	28	32
59	58	58	57	57	56	57	56	55	55	55	54	53	51

ASTM-A29-03, ASTM-A322-91 (REAPPROVED 2001), ASTM-A434-04(BC/BD)  
 UNS#-G43400 (OR LATEST REVISIONS)  
 HOT ROLL VACUUM DEGAS RD# 4812 REC'D 2-23-05 QUENCHED & TEMPERED STRESS FREE  
 AUST: 1650 F 79 MIN / QUENCH: WATER / TEMPER: 1270 F 75 MIN / HB @ MID-RAD = 307/311 FROM IAC 22792 04/21/05

H-A INDUSTRIES  
A DIVISION OF A. M. CASTLE & CO.  
 Name: JAN L. CARUSO  
 NATIONAL TECH. SERVICE MGR.  
*Jan L. Caruso*  
 Signature

0012746  
050284 JM 2007/02/05

---

**From:** John Metalonis [mailto:john@highpressure.com]  
**Sent:** Thu 12/13/2007 2:01 PM  
**To:** Clayton Pullins  
**Cc:** Tony Pepicello  
**Subject:** RE: Reactor and Window

1. There was no additional heat treatment of the 4340 material. The final product has the same unaltered properties as those shown on the material test report.

2. Window calculation attached.

John A Metalonis  
Manager - Design

High Pressure Equipment Company  
1222 Linden Avenue  
Erie PA 16505 USA

814 838 2028  
814 838 6075 (fax)

CONFIDENTIALITY NOTICE: The contents of this message (including any attachments) are confidential and may be privileged. If you are not the intended recipient of this message, you are not authorized to read, print, retain, copy, disseminate, distribute or use this message or any part thereof. If you received this message in error, please reply to the sender and delete this message from your system. Thank you



**Calibration Record**

Model Number : 212-C-UC/FM  
 Serial Number : 55784  
 Pressure Range : 0 BARG to 2000 BARG

Work Order Number : 226454  
 Customer : THE UNIVERSITY OF CHICAGO

*1 barg = 14.5 psi*

**Final Run**

Pressure (BARG)	Output (Vdc)
0	-0.023
2000	4.938
FSO	4.964

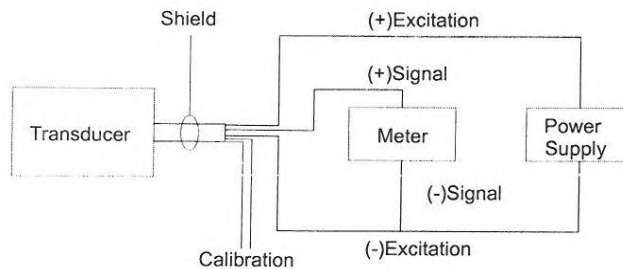
**Wiring Connections**

A / 1 (Red)	(+)Excitation
B / 2 (Green)	(+)Signal
C / 3 (White)	No Connection
D / 4 (Black)	(-)Excitation/Signal
E / 5 (Blue)	Optional Cal
F / 6 (Brown)	Optional Cal
Shield	No Connection

Excitation Voltage: 10.2 V to 42.0 Vdc  
 Test Excitation: 24.0 V

Calibrated by: KK

**200 Series Wiring Schematic**



2770 Long Road \* Grand Island, NY 14072 \* USA \* Phone (716)773-9300  
 Fax (716)773-5019 \* email: sales@gp50.com \* http://www.gp50.com

Calibration Record

Model Number : 211-C-RT-7/FM  
Serial Number : 55785  
Pressure Range : 0.0 PSIG to 3000 PSIG

Work Order Number : 226454  
Customer : THE UNIVERSITY OF CHICAGO

Final Run

Pressure (PSIG)	Output (Vdc)
0.0	.009
3000	5.030
FSO	5.021

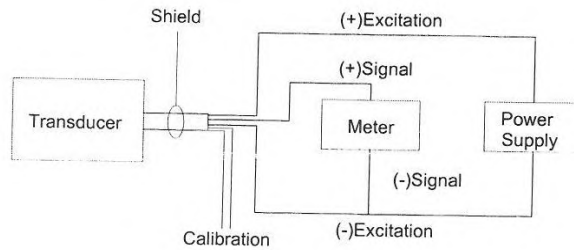
Wiring Connections

A / 1 (Red)	(+)Excitation
B / 2 (Green)	(+)Signal
C / 3 (White)	No Connection
D / 4 (Black)	(-)Excitation/Signal
E / 5 (Blue)	Optional Cal
F / 6 (Brown)	Optional Cal
Shield	No Connection

Excitation Voltage: 9.0 V to 40.0 Vdc  
Test Excitation: 24.0 Vdc

Calibrated by: KK

200 Series Wiring Schematic



2770 Long Road \* Grand Island, NY 14072 \* USA \* Phone (716)773-9300  
Fax (716)773-5019 \* email: sales@gp50.com \* http://www.gp50.com

**Appendix C.11 BioCARS SOP for Laser Controlled Areas at Bldg 400, Sector 14  
(January, 2020)**

**Standard Operating Procedures for  
Laser Controlled Areas  
Building 400, Sector 14**

Version 10  
January 2, 2020

Prepared by: Vukica Srajer, Sector 14 LCA Supervisor

Same as version 9, January 2019, except:

- List of authorized laser users updated (Appendix 2)
- List of lasers updated (Appendix 1; ChemMatCARS ns laser and two high power LEDs; four NIH/Anfinrud diode lasers)

**Approved by:**

---

Vukica Srajer                      Date  
LCA Supervisor

---

Edmund Chang                      Date  
APS/ASD ESH Coordinator

---

Bryan Brooks                      Date  
ANL-E Laser Safety Officer

---

John Connolly                      Date  
APS/AES Division Director

## **Introduction**

This Standard Operating Procedure (SOP) describes the use of Class IV and Class III lasers, listed in Appendix 1, in the Sector 14 LCAs: Laser Lab (Rm. 14-ID-6) and the experimental stations 14-ID-B, 14-BM-D, and 14-BM-C at Sector 14. Class IV lasers represent a hazard for eye damage from direct beam, specular reflections, and possibly diffuse reflections. They also have the potential to cause burns on the skin or ignite flammable material.

Any laser from the list can be used in the Laser Lab or the experimental stations provided the appropriate interlocks and warning signs are implemented, as approved by the APS/ANL laser safety personnel. Any changes in the optical set-up from previously approved configurations, will be documented and require approval by the APS/ANL laser safety personnel.

## **Laser Safety Personnel**

LCA Supervisor:

Vukica Srajer (630) 252 – 0455 [v-srajer@uchicago.edu](mailto:v-srajer@uchicago.edu)

APS/AES-ESH Coordinator

Edmund Chang (630) 252 – 6714 [change@aps.anl.gov](mailto:change@aps.anl.gov)

ANL-E Laser Safety Officer

Bryan Broocks (630) 252 – 3396 [bbroocks@anl.gov](mailto:bbroocks@anl.gov)

## **Authorized Users**

No person is allowed to operate any of the listed laser systems unless all four of the following requirements are met:

1. Completion of all APS user training.
  2. Completion of the ANL Laser Safety Training (ESH-120) and provided evidence of laser-specific medical eye examination.
  3. Familiarity with the content of this SOP.
  4. Approval by the LCA Supervisor who will add the name to the list of authorized laser users.
- Currently authorized users are listed in Appendix 2 of this document.

## **On-the-job Alignment Training**

Following a mandate of the DOE Office of Sciences and as part of the policy for safe laser work, the Advanced Photon Source Laser Safety Committee has developed the policy that all laser control area supervisors (LCAS) or their designated trainers are required to provide on-the-job training for laser alignments (Class 3b and 4) within their LCA. Laser users must complete on-the-job training and have specific authorization from the LCAS before performing any laser alignments without direct supervision.

## **Scientific Collaborators & Spectators**

Scientific collaborators have access to the LCA for scientific work if they meet the requirements for authorized users. They must follow the laser set-up and operating procedures described in this SOP and its appendices.

Spectators are only permitted to enter the LCA in presence of authorized personnel with all laser beams enclosed and not accessible or all laser shutters are closed (laser light is not accessible).

If no laser hazards exist for the LCA (i.e the laser power supplies are shut off and power supply keys are in the lock box), the access to these stations is not restricted.

## **General Setup and Laser Operation**

### ***Laser Controlled Areas (LCA)***

Both the Laser Lab (Room 14-ID-6) and the experimental stations will be used as an LCA (see Appendix 3, Figure 1 for the layout of Sector 14).

As a safety measure, the Laser Lab doors are fitted with locks that **cannot** be set to an unlocked state; personnel are forced to use a key to enter. Distribution of Laser Lab keys is limited to personnel approved by the LCA Supervisor.

The experimental stations are designed for use as an LCA. The 14-ID-B station door is automatic and in this case a curtain interlocked with a magnetic switch is used to contain scattered laser light. The station doors for 14-BM-C and 14-BM-D are operated manually and have a position switches used for the laser interlock controls. The laser interlock switches are magnetic switches for the manual doors and operate independently of the PSS door interlocks. Although the manual doors can be locked from the outside, they can be unlocked from the inside (in case of power outage the doors are unlocked). If the switch inside the station is placed in the “unlock” position the door cannot be locked from the outside. Therefore we foresee no situation of personnel being accidentally locked inside the station and not being able to exit.

### ***Standard Optical Configuration***

Laser light paths are placed, wherever possible, below or above the eye level. In cases where this is not possible, appropriate shields are in place to prevent viewing in the plane of laser beams. All beam paths will be enclosed whenever possible and as close to the sample as possible.

All Class IV lasers (Appendix 1) are operated with external shutters positioned as close as possible to the exit port of the laser beam.

All Class IV lasers in use have a light shield (referred to as the “External Optics Box”, or EOB) that encloses all optical components on the optical table outside of the main laser system, including the external laser shutters. This enclosure is kept closed except during an alignment process (with appropriately attenuated beam) or during remote laser use, with no personnel present in the LCA. In addition, the laser can only be operated with an open cover or open EOB box cover if the LCA curtain/door is closed and the laser is interlocked.



## ***Normal Operation***

We distinguish two operating modes:

- Local: Laser light is used in the same room where the laser is located (Laser Lab or experimental stations). In this case this room is the LCA. The 14-BM-C and 14-BM-D stations and Laser Lab only operate in local mode, while 14-ID-B station is used in both local and remote mode.
- Remote: The laser is located inside the Laser Lab and the laser light is passed via mirror system or optical fiber to the experimental station when the external laser shutter is opened.

If the laser is located in the Laser Lab and its cover and EOB are closed, only the room receiving the laser light (14-ID-B) is defined as an LCA. This status is established by an interlock system, which for the experimental station requires the external laser shutter opening to be enabled from the station, so that the laser light can be passed from the Laser Lab to the experimental station.

Both Laser Lab and the receiving experimental station are considered to be an LCA jointly if the laser cover or any of the EOB are open. The joint LCA function is established by the interlock system.

## **Eyewear**

Standard protective eyewear will be provided and is kept in the Laser Lab. A list of the available eyewear is posted in the Laser Lab in the form of a poster and used as a laser eyewear selection guide (Appendix 4). Appendix 1 provides minimum OD requirements for the eyewear to be used for each specific laser.

All laser protective eyewear will be inspected at least annually and the results recorded in a log (template in Appendix 6). This inspection log will be posted at the entrance of the Laser Lab.

## **Alignment Hazard Control**

Only authorized users can be present in the LCA during alignment. During alignment, the laser shutter is controlled locally. The path between the output of the focusing lens (or optical fiber) and the sample cannot be enclosed due to the spatial constraints of the experimental conditions.

All optics alignments are initially done using either laser 26/IHID 11034 in the low power mode or one of the Class II HeNe lasers (see Appendix 1). If necessary, additional alignments that involve Class III and Class IV laser beams are done at the minimum laser power possible. In addition to the alignment of external optics, alignment of the laser systems is necessary. This requires in some cases the laser covers to be open. The alignments are done at the minimum laser power necessary.

All personnel will wear appropriate protective eyewear providing sufficient optical density to reduce the output of each laser beam wavelength below the maximum permissible exposure level as specified by ANSI 136.2 (2000) (see Appendix 1 for minimum OD requirement for specific lasers). When the laser output power is reduced for alignment and it is necessary to

view the laser beam, the operating personnel will use eyewear appropriate for the reduced power levels. For more detailed alignment instructions see the Special Alignment Procedures section of this SOP.

**Laser Hazard Control** Laser hazard warning signs are posted at the entrance to the LCA. An interlock system is implemented. The fundamental rule for the interlock system is: the LCA doors cannot be open when laser beams are accessible within the LCA. Opening of the LCA doors will initiate appropriate measures to turn off the light, either by closing the laser shutter or turning off the laser power.

### **14-ID-6 (Laser lab) and 14-ID-B LCAs**

In addition, the 14-ID-B LCA has lighted three-color warning signs placed inside and outside the LCA. Green light (“NO HAZARD – Laser off”) indicates safe entry to the room, yellow light (“CAUTION – Laser energized”) indicates laser power on but all laser light is enclosed, and red light (“DANGER – Beams accessible”) indicates unshielded laser light present in the room. The warning lights reflect these conditions:

- In case of the Laser Lab, the yellow light will be ON if the ps laser located permanently in the laser lab is powered. The red light is ON if any unshielded laser light is present in the room (e.g. laser cover is open during laser alignment).
- In the case of the 14-ID station, light indicators refer to the laser status and presence of laser light from local and remote sources. For example, if laser light is delivered from the laser lab (Rm. 14-ID-6) to 14-ID-B, the red DANGER light outside 14-ID-B will be lit.

The safety status of the LCA and the laser systems is controlled by a complex PLC-controlled interlock system. Its main controller is located in the Laser Lab and a slave controller is located in the 14-ID-B experimental station. The interlock system includes the following components:

- Each room containing a laser or able to receive laser light from a remote location has non-defeatable door interlock switches.
- Laser covers and the covers of the EOB are equipped with interlock switches.
- The external laser shutters are integrated in the interlock system.
- Panic buttons are also integrated in the interlock system.

In particular, the following logic rules have been implemented:

Case 1. Laser source and experiment are located in the same LCA.

- When a laser is powered its external laser shutter can only be opened when the LCA door/curtain is closed. If the door/curtain is opened while the external laser shutter is open, the shutter will close.
- When a laser is powered, its cover can be removed if the LCA door/curtain is closed. If the door is open, opening of laser cover will close the appropriate shutter. If the door opens while the laser cover is off, the laser shutter will be closed. Each laser shutter has position switches. If the interlock system senses a malfunctioning shutter (i.e. shutter fails to close when commanded), power to the laser will be cut off extinguishing the laser light.
- If any of the LCA doors/curtains open while the EOB is opened, the appropriate shutter will be closed.

- Panic buttons will close laser shutters at any time.

Case 2. Laser light is passed from the Laser Lab to the 14-ID-B experimental station.

- Same rules as above apply to 14-ID-B. In addition, the following rules are also implemented:
- Transfer of laser light from the laser lab into the 14-ID-B experimental station must be enabled from this station. The external laser shutter can then be opened only if the station door/curtain is closed. Actual opening of the external laser shutter is delayed by 10 seconds, preceded by acoustic and optical warning signals (interval beep and blinking LED).
- If remote laser light is present, opening of the station door will close the laser shutter.
- Panic buttons in the station will close all shutters at any time.
- The following table summarizes the various conditions and status of the colored warning signs when a laser is powered.

Three-way illuminated laser warning sign key displayed at the inside and outside of the LCA:

Laser Energized	Laser Shutter Open	Laser Cover or EOB Open	Warning Light
No	-	-	Green
Yes	No	No	Yellow
Yes	Yes	No	Yellow
Yes	No	Yes	Yellow
Yes	Yes	Yes	Red

Green: No Hazard Laser Off  
 Yellow: Caution Laser Energized  
 Red: Danger Beams accessible

Administrative control of the laser power supply keys is used to prevent unauthorized operation of the equipment. The laser lab is always locked and laser keys are removed from the units when not in use. The laser keys are stored in a locked key box.

Every laser station is equipped with several panic buttons. In an emergency these panic buttons close all laser shutters. One panic button is integrated in the Laser Status Box and another one in the remote control box for each laser. When the laser light is transferred from the laser lab into an experimental station, an additional remote box with panic button is located in this station.

**Additional Hazards**

High Voltage:

- 110 VAC, 20 A
- 110 VAC, 20 A
- 110 VAC, 15 A
- 110 VAC, 20 A
- 110 VAC, 20 A
- 110 VAC, 20 A

- power for laser #1
- power for laser #2
- power for laser #3
- power for laser #4
- power for laser #5
- power for laser #6

110 VAC, 23 A	power for laser #7
110 VAC, 23 A	power for laser #8
110 VAC, 20 A	power for laser #9
110 VAC, 20 A	power for laser #10
110 VAC, 20 A	power for laser #11
110 VAC, 20 A	power for laser #12
250 VAC, 20 A 3Ø	power for laser #13
110 VAC, 20 A	power for laser #14
110 VAC, 20 A	power for laser #15
110 VAC, 20 A	power for laser #16
110 VAC, 20 A	power for laser #17
208 VAC, 40 A 3Ø	power for chillers

### **Control of Emergencies and Abnormal Situations**

In the events of

- Laser burns to eyes and/or skin: Call 911, shut down the laser system, report to the LCA supervisor and the APS floor coordinator.
- Fire: Call 911, quickly evacuate from the LCA and activate the nearest fire alarm.
- Severe Weather Warning: Evacuate immediately to the nearest tornado shelter.
- Breakdown of a high voltage: Call 911 if help is needed shut off power at the main circuit breaker, and report to the LCA supervisor.
- Laser coolant (water) accidentally discharged into the environment: Shut down the laser system, report to the LCA supervisor and the APS floor coordinator.

### **Other Control Measures**

- Any change in laser locations, optical and interlock configurations will be reported to the LCA Supervisor and ANL-E Laser Safety Officer, and approval will be obtained prior to their use.
- Laser interlocks will be tested for proper operation on a timely basis (annually) or whenever the interlock hardware/software configuration changes according to the procedures described in Appendix 5. The results are recorded in a log posted at the entrance of the Laser Lab (log template is in Appendix 6).

## **Special Alignment Procedures**

### **Alignment Protocols for Picosecond Laser System located in Laser Lab (Rm. 14-ID-6)**

(Procedures written by Philip Anfinrud/NIH)

#### **Safety Measures**

The following policies and procedures are implemented:

- Only authorized personnel are allowed in the LCA while the laser is in operation and beams are accessible.
- All beam alignment will be carried out by expert laser personnel.
- Personnel will wear appropriate protective laser goggles according to recommendation of the LSO.
- Both doors of the 14ID laser lab are locked. Keys to the lab are issued to expert laser users and trained technicians only.

#### **Operating Procedures**

- Verify that the laser interlock is active, especially if the interlock was inactive during the time period when laser was not used (check the laser log).
- Verify that the appropriate laser shields are in place.
- Verify that the laser warning lights are working.
- Make sure that all personnel are authorized (a list of authorized personnel is posted at the LCA).
- Wear proper safety goggles.
- Take off any metal hand jewelry and watches when operating lasers.

#### **Picosecond Laser Safety Interlock System**

- The following laser interlock mechanisms are implemented and active for the PLS operation
- The four pump lasers for the Spitfire (Figure 2), the two Empower lasers, the Tsunami, and the Millennia have electrically actuated shutters (1, 2, 3, and 4 in Figure 2) that are interlocked to the safety system. If the door to the LCA is opened and there are laser beams accessible, the shutters close.
- It may be necessary to remove the covers of the Spitfire, the Tsunami, and the Topas lasers to conduct tuning and alignment procedures. If the LCA door is opened, while the covers are off, all shutters will close.
- The optics box on the left end of the enclosure is also interlocked. If the LCA door is opened while this box is opened, shutters are closed.
- The external laser shutters can be opened only if the LCA doors are closed. Opening of any LCA door will close the shutters.
- When a laser is energized, the laser cover can be opened providing the room door is closed. If the door is open and a laser cover is opened, the shutters will close.

- Panic buttons will close all shutters.
- When the power supplies are turned on and laser light **IS NOT** accessible the yellow “Laser Energized” caution sign will be lit at the entrances to the LCA and in the interior of the LCA.
- When the power supplies are turned on and laser light **IS** accessible (covers off the laser enclosures) the red “Beams Accessible” caution sign will be lit at the entrances to the LCA and in the interior of the LCA.

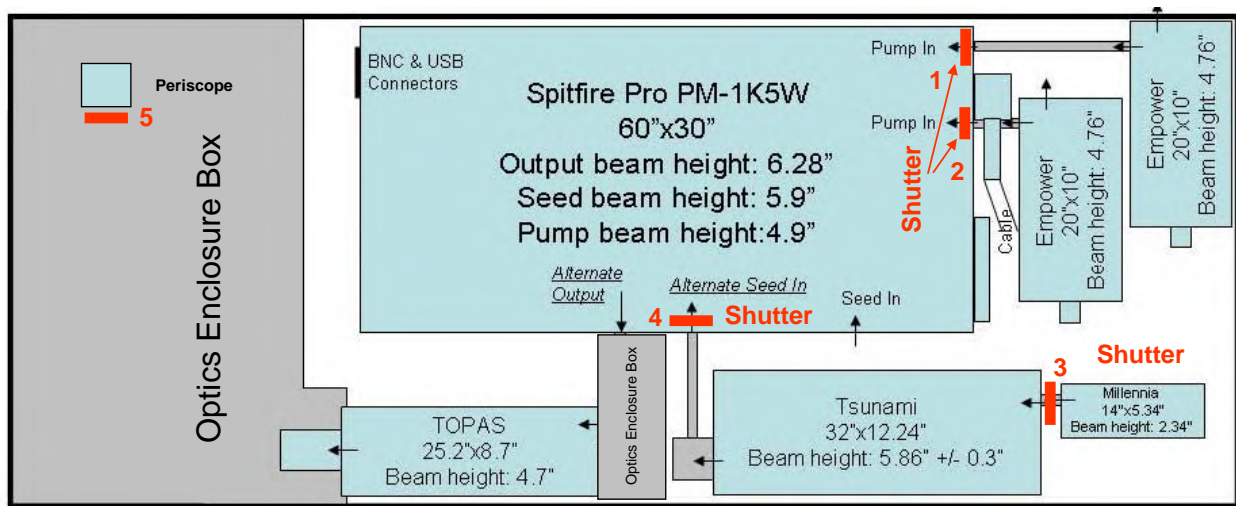


Figure 2: The layout of the picosecond laser system is shown. The two Empower pump lasers and the Millennia seed laser have Mechanical shutters (1, 2, and 3) that stop the beam from entering the Spitfire enclosure. If the cover of the Spitfire is open, and the LCA door opens, these shutters will close and no light will be present in the LCA. If the Cover of the Tsunami is open and the LCA door is opened the Millennia shutter (3) is closed. The 5<sup>th</sup> shutter in the Optics Enclosure Box is closed if the 14-ID-B station laser interlock is violated.

## Protocol for Exchanging Dichroic Wavelength Separator after TOPAS

First, refer to photos and a spreadsheet that specifies the optimal set of parameters for each dichroic separator; these files are located on the Desktop of the laser-control computer. Next, kill the TOPAS output by closing the Empower laser shutters. Remove the side cover on the optics enclosure at the end of the laser table. Identify the wavelength separator that needs to be exchanged (there are two orthogonally mounted separators). With a 1/4-20 Allen wrench, remove the screws that secure the home-built safety shield and the wavelength separator. Replace the separator with one optimized for the desired wavelength, and reattach the safety shield. Translate the appropriate dichroic separator into position (depends on the polarization of the desired wavelength). For uv operation, a broadband mirror needs to be moved from its temporary docking station and secured in the holder along the TOPAS beam path. Set the lens-translation stages according to a spreadsheet that specifies the optimal setting for each dichroic separator. Note that there is one translation stage in the laser hutch optics enclosure and three stages in the beam conditioning enclosure (see below). Don protective eyewear before opening the Empower laser shutters and thereby enabling TOPAS output. Partially close the first alignment iris (in front of the beam expansion telescope) to a dimension slightly smaller than the

beam size; fine tune the second mirror mount upstream of the first iris to center the beam on that iris. Fine tune the mirror mount immediately upstream of the first iris to center the beam on the (partially closed) downstream iris. After performing this coarse alignment, reopen the irises and replace the enclosure cover.

#### **Precautions taken:**

Laser beam characteristics after dichroic beam separation optics:

- Average power: < 15 mW at 27 Hz
- Maximum pulse energy: ~0.5 mJ/pulse
- Pulse duration: 1.2 ps
- Wavelength(s): tunable from UV to Near IR
- Beam size: 8 mm
- Maximum contamination: ~100  $\mu$ J at 780 nm

Protective eyewear will be worn to block invisible radiation (uv and IR) as well as visible radiation below 535nm. With our protective eyewear, the TOPAS output remains visible on the beam-alignment irises due to either nonlinear generation of visible light and/or parasitic parametric generation. Thus, we can safely and accurately align the beam to the irises in the enclosure. The dichroic separator used to isolate 535 nm light also functions up to about 610 nm. Consequently, alignment performed at 535 nm also applies to much of the visible spectrum.

The optics enclosure is interlocked to the laser hutch doors; if a door is opened with the enclosure in an open state, shutters close and terminate the TOPAS output.

#### **Protocol for Coupling Laser light into Optical Fiber**

Close the laser shutters mounted in the Spitfire Pro enclosure before removing the top cover. Insert the mirror used to intercept the Empower beam and direct it toward the fiber optic. With the beam stop positioned between the collimating lens and the fiber, the pump Empower shutter is opened and the Empower current is raised to a level just above lasing threshold. The mirror used to intercept the Empower output is fine tuned to center the beam on a pair of irises that defines the path to the fiber optic. The opening of the last iris is reduced to ~5 mm to ensure that the focused beam is entirely contained within the 100-um core size of the multi-mode fiber. Remove the beam stop in front of the fiber and inset a pellicle beam splitter into the beam path at ~45 degrees. View the reflection from the fiber tip on a distant white card and ensure that the spot is centered on the fiber tip. Replace the top cover of the Spitfire enclosure. Using a joule meter to measure the pulse energy in the experimental hutch, dial up the laser current to achieve ~0.25 mJ pulse energy, the maximum needed for planned experiments.

### Precautions taken:

Laser beam characteristics during alignment into the fiber:

Average power: < 15 mW at 27 Hz

Maximum pulse energy: < 0.5 mJ/pulse in Spitfire enclosure

Pulse duration: ~200 ns

Wavelength: 527 nm

Beam size: ~12 mm

- Removal of the beam stop in front of the fiber optic activates an interlock that ties the Empower laser shutter to the door of the experimental hutch. Consequently, the hutch must be interlocked before light can be transmitted to the experimental hutch.
- With these precautions, no protective eyewear will be required for this alignment procedure.

### Protocol for Beam Conditioning Optics Alignment (14-ID-B Procedure)

The pulse repetition frequency is set to 27 Hz and the attenuator in the laser hutch is set to maximum attenuation. An alignment jig with a 2-mm aperture is inserted into the beam path immediately after the first receiving mirror. Before opening the remote laser shutter (located in the laser hutch), only laser-certified users are allowed to remain in the hutch, and the laser safety interlock requires that the door to the X-ray hutch be closed before the shutter can be opened. Under these conditions, the pulse energy received by the beam conditioning optics is < 1  $\mu$ J, and the average power is < 27  $\mu$ W. The energy transmitted through the beam conditioning optics is monitored by a calibrated internal photodiode. The pulse energy transmitted through the 2-mm aperture on the first alignment jig is estimated to be < 20 nJ, and its average power is estimated to be less 0.6  $\mu$ W. At this average power level, the brightness of the beam is more than 3 orders of magnitude weaker than a laser pointer, but remains sufficiently bright (when tuned to visible wavelengths) to align the beam into the downstream optics. The beam is first centered on the aperture of the first alignment jig using motorized beam steering in the laser hutch. If necessary, tweak the beam steering controls on the first receiving mirror mount to center the transmitted beam on the aperture of an identical alignment jig positioned downstream of the two echelon assemblies. Once this alignment is verified, the jigs are removed and the beam alignment through a prism-slit assembly is assessed by viewing the transmitted spatial distribution on a white card. The cylindrical focusing optics are translated to a position that achieves optimum transmission through the prism-slit assembly. The optimum lens position is a function of wavelength. The divergence of the beam after the prism-slit assembly is > 200 mrad in one dimension, owing to the 100-mm focal length cylindrical focusing optic located upstream of the prism-slit assembly. After collimation with a 40-mm focal length optic, the beam is directed downward toward the sample, and passes through a tilt-tunable waveplate. To set the waveplate to quarter-wave rotation, which produces circularly polarized light, we insert a Glan-laser prism and a retro-reflecting mirror into the beam path. The reflected beam retraces its path, and the intensity transmitted through the Glan-laser prism is monitored visually while adjusting the tilt angle and rotation of the waveplate. When the intensity is minimized, the waveplate is properly



aligned. For these operations, the beam is contained within the laser enclosure with no stray reflections emerging from it.

### **Protocol for Laser-Sample Alignment**

A motorized attenuator located between the beam conditioning optics and the sample position is set to maximum attenuation, which reduces the pulse energy to  $<1$  nJ. A pinhole is mounted at the sample position and centered on the cross hairs of cameras used to position the sample at the intersection of the laser and X-ray beams. A manual micrometer is adjusted to optimize the focus in the plane of the sample, and motorized translation is used to center the focused spot on the camera cross hairs. For these operations, the focused spot is monitored on a computer screen.

#### **Precautions taken:**

Laser beam characteristics (inside beam conditioning optics enclosure):

Average power:  $< 12$  mW at 27 Hz without and  $<24$   $\mu$ W with attenuation.

Maximum pulse energy:  $< 0.4$  mJ/pulse without and  $<0.8$  uJ with attenuation.

Pulse duration:  $\sim 25$  ps (FWHM) after echelon pulse stretcher

Wavelength(s): user selectable from 350 - 1000 nm

Beam size:  $\sim 20$  mm at input of echelon pulse stretcher

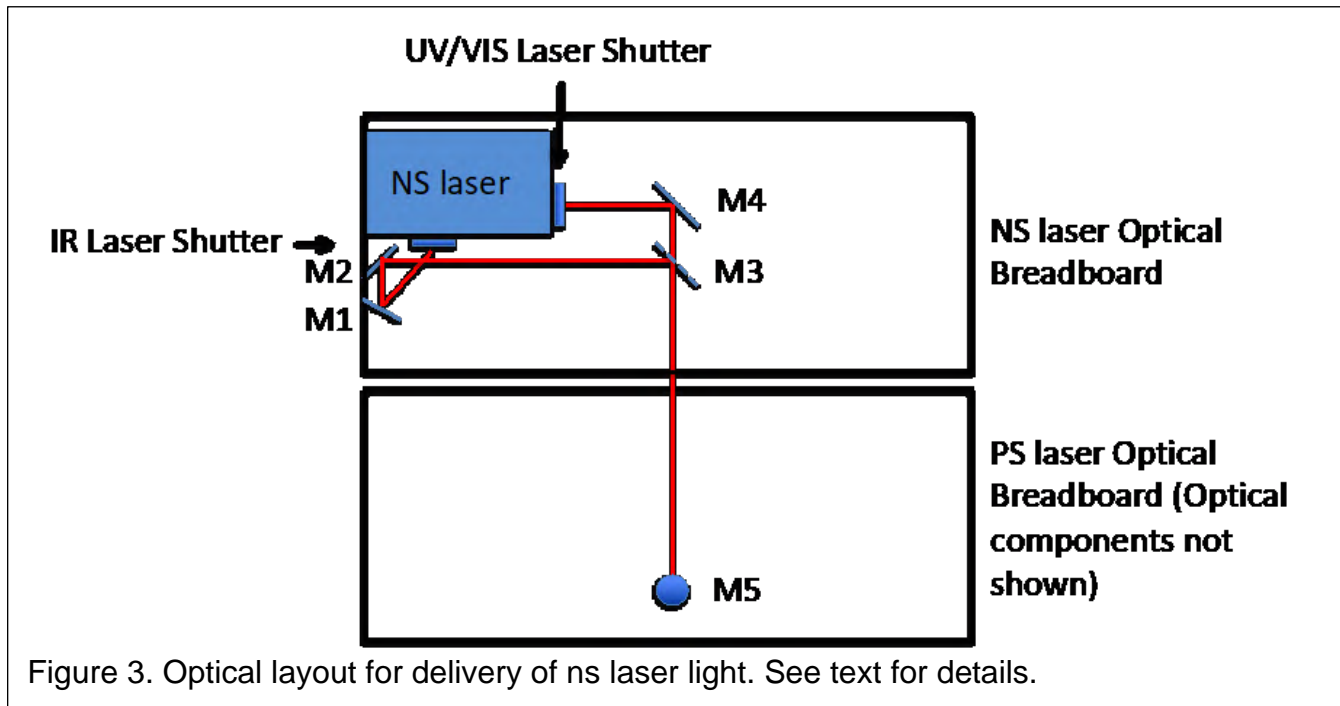
- The alignment jigs must be in place and the laser-hutch attenuator must be set to maximum attenuation before first opening the laser shutter.
- The degree of attenuation is to be verified by monitoring the photodiode signal in the beam conditioning optics enclosure.
- With these precautions, no protective eyewear will be required for visible radiation.

**Optical Configuration and Alignment Procedures:**  
**Pulsed ns lasers (class IV)**

**Optical configuration 1 (14-ID-B LCA)**

This description covers the installation of the BioCARS ns laser (Opotek Opolette, IHID 10882) in the 14-ID hutch, where the laser beam is routed via free space delivery either to the sample location directly or is coupled into an optical fiber and delivered to the sample location.

As shown in Figure 3, the ns laser is located on an optical board above the main optical table. The board is an extension of the board that carries ps laser optics. This extension board is located upstream of the ps laser optics board. Both UV/VIS and IR output ports of the laser have an interlocked laser safety shutter placed immediately downstream of the port. Only one shutter can be opened at a time. A series of mirrors are used to direct the light beams to the sample position. For the IR beam, the path M1-M2-M3-M5 is used. The first three mirrors keep the beam in the horizontal plane while M5 directs the beam down toward the sample. The UV/VIS beam path uses M4 and M5. M3 is a removable mirror that is only used for the IR beam. With this configuration, either IR or UV/VIS light is delivered directly in a vertical direction, pointing down via M5 mirror, to the sample position. For some experiments, laser light needs to be delivered to the sample in the horizontal plane, semi-coaxial with the X-ray beam. In those cases, the



down-going laser beam is focused into an optical fiber and delivered to the sample position from the fiber output, mounted upstream and outboard of the sample location.

## **Safety and Interlocks**

The laser shutters will be interlocked to the LCA door so that they can open only if the LCA door is closed. Also, when the ns laser is in operation, a light warning sign outside the LCA will be lit to alert personnel to the laser light hazard.

## **Alignment (local) mode and procedure**

The initial alignment will be done with minimal laser power. The mirrors will be put in place and used to deliver the beam along the intended path. Once the preliminary beam path has been established, the laser will be operated at full power but an attenuator will be inserted to minimize the beam intensity to finish the final focusing at the sample position. Only authorized personnel will be present during the alignment.

Laser goggles with an OD > 2 @ laser wavelength will be used during the alignment mode.

1. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard
2. Verify proper laser attenuation
3. Make sure the door to LCA is closed
4. Operate the laser shutter from inside the LCA

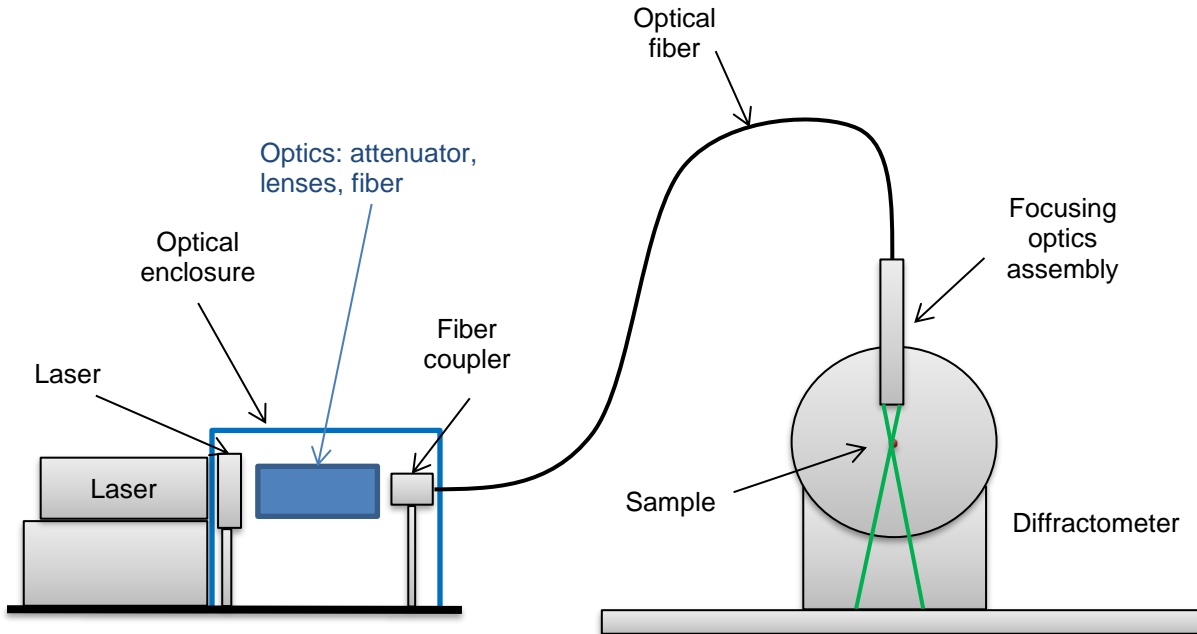
## **User (remote) mode**

User experiment will be conducted in this mode. No users will be present in the LCA while laser shutter is open.

1. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard.
2. Make sure the door to LCA is closed

## **Optical configuration 2 (any LCA)**

The Opotek Opolette ns lasers (ID 10882 and ID 10853) can be setup on a portable cart and used in any of our LCAs (laser lab, 14-ID-B, 14-BM-C and 14-BM-D hutches). A light-tight box (optical enclosure) is placed directly in front of the laser exit port, enclosing a laser shutter as the first element (see figure below), necessary optical elements (attenuator, collimating/focusing lenses, fiber) and an output fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering while coupling laser light into an output optical fiber. Laser light is delivered by the fiber to the sample mounted on a diffractometer. The light-delivery end of the fiber is connected to an enclosed focusing optics assembly. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent (>10°) beyond the focal spot. The assembly is mounted pointing down.



### **Safety and interlocks**

The laser shutter will be interlocked to the LCA door so that it can open only if the LCA door is closed. Also, when the ns laser is in operation, a light warning sign outside the LCA will be lit to alert personnel to the laser light hazard.

### **Alignment (local) mode**

In this mode, laser light will be coupled into the optical fiber and the laser light output from the fiber will be aligned at the sample position. Minimum laser power will be used in this mode ( $<10\mu\text{J}$  at the sample). Only authorized personnel will be present during the alignment.

Laser goggles with an OD  $> 2$  @ laser wavelength will be used during the alignment mode.

5. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard
6. Verify proper laser attenuation
7. Make sure the door to LCA is closed
8. Operate the laser shutter from inside the LCA

### **User (remote) mode**

User experiment will be conducted in this mode. No users will be present in the LCA while laser shutter is open.

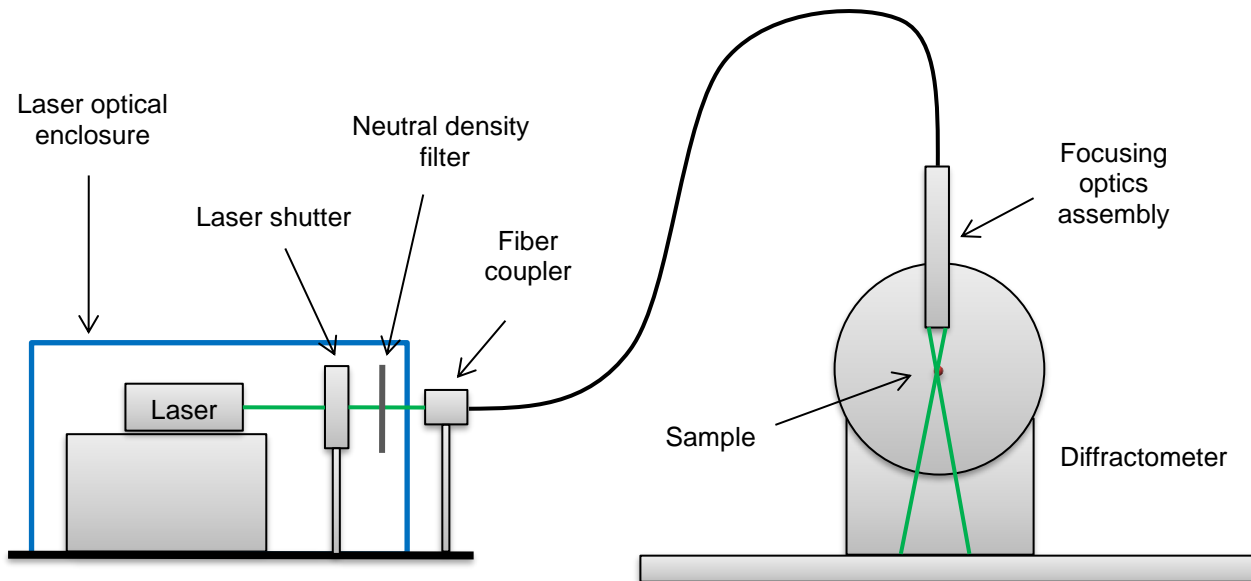
3. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard.
4. Make sure the door to LCA is closed

5. Operate the laser shutter remotely, from the LCA control area

**Optical Configuration and Alignment Procedures:  
CW lasers or high power LEDs (Class IIIB and Class IV)**

**Optical configuration (any LCA)**

The laser is enclosed in a light-tight box. A laser shutter is placed directly in front of the light exit port, followed by an optical fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering while coupling laser light into an optical fiber. Laser light is delivered to the sample mounted on a diffractometer by an optical fiber. The light-delivery end of the fiber is connected to an enclosed focusing optics assembly. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent ( $>10^\circ$ ) beyond the focal spot. The assembly is mounted pointing down.



**Safety and interlocks**

The class IIIB laser shutter will be interlocked to the LCA door so that it can open only if the hutch door is closed. Also, when the IIIB laser is in operation, a light warning sign outside the LCA will be lit to alert personnel to the laser light hazard.

**Alignment (local) mode**

In this mode, laser light will be coupled into the optical fiber and the laser light output from the fiber will be aligned at the sample position. Minimum laser power will be used in this mode (1-5mW). Only authorized personnel will be present during the alignment.

Laser goggles with an OD > 2 @ laser wavelength will be used during the alignment mode.

1. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard
2. Verify an 1.5-2 OD filter is placed at the exit of the laser shutter
3. Make sure the door to LCA is closed
4. Operate the laser shutter from inside the LCA

### **User (remote) mode**

User experiment will be conducted in this mode. No users will be present in the LCA while laser shutter is open.

1. Verify that the laser warning light outside the LCA is ON to alert personnel to the laser light hazard.
2. Make sure the door to LCA station is closed
3. Operate the laser shutter remotely, from the LCA control area

## Appendix 1

## Picosecond Laser System

ANL IHID #	10695	10690	10691	10692	10693	10694
<b>Brand</b>	Spectra-Physics	Spectra-Physics	Spectra-Physics	Spectra-Physics	Spectra-Physics	Light Conversion
<b>Model</b>	Millennia Pro 5s	Tsunami	Empower 30	Empower 30	Spitfire Pro PM	TOPAS-p
<b>Serial #</b>	E2002	2665	193-30	107-30	1073	05347
<b>Type</b>	Green Diode	Ti:Sapphire	Nd:YLF	Nd:YLF	Ti:Sapphire	Optical Parametric Amplifier (OPA)
<b>Beam Characteristics</b>						
<b>Wavelength</b>	532 nm	710-980 nm	527 nm	527 nm	750-840 nm	tunable
<b>Diameter</b>	~2.3 mm	<2 mm	3 mm	3 mm	7 mm	~6 mm
<b>Divergence</b>	<0.5 mrad	<1 mrad				< 1 mrad
<b>Mode</b>	CW	Pulsed	Pulsed	Pulsed	Pulsed	Pulsed
<b>Pulse Width</b>		100 fsec	100-300 nsec	100-300 nsec	1.2 psec	1.2 psec
<b>Rep. Rate</b>	CW	80 MHz	1 kHz	1 kHz	1 kHz	10-1000 Hz
<b>Power/ Pulse Energy</b>	>5 W -NA-	720nm-400mW 790nm - 700mW 850nm-550mW 950nm -200mW 720nm-5.0 nJ 790nm-8.8 nJ 850nm-6.9 nJ	20 W  20 mJ	20 W  20 mJ	5 mW  >5 mJ	240-2600nm ~100 mW 1150-2600nm - >1.250 mJ 580 - 800nm - > 250 µJ 533 - 600nm - > 300 µJ 475 - 533nm - > 450 µJ 400 - 475nm - > 30 µJ 290 - 400nm - > 75 µJ 300 - 355nm - > 100 µJ 240 - 300nm - > 50 µJ
<b>Class</b>	<b>IV</b>	<b>IV</b>	<b>IV</b>	<b>IV</b>	<b>IV</b>	<b>IV</b>
<b>Laser Safety Glasses</b>						
<b>Wavelength</b>	532 nm	710-980 nm	527 nm	527nm	750-840 nm	240 – 2600 nm
<b>OD (min)</b>	5	4	7+	7+	7	5



## OPOTEK BioCARS Nanosecond Laser System

<b>ANL IHID #</b>	This laser is a component of 10882	10882
<b>Brand</b>	Quantel USA	OPOTEK HE 355LD+UV2M
<b>Model</b>	Ultra 100	Opolette
<b>Serial #</b>	1103280209	2466
<b>Type</b>	Nd:YAG	OPO

### Beam Characteristics

<b>Wavelength</b>	1064 nm	225-2200 nm
<b>Diameter</b>	~3 mm	~4 mm
<b>Divergence</b>	<10 mrad	<2 mrad
<b>Mode</b>	pulsed	pulsed
<b>Pulse Width</b>	5 ns	5 ns
<b>Rep. Rate</b>	20Hz	20Hz
<b>Power/ Pulse Energy</b>	4W (max) 400 mJ (max)	0.4W (max) 5-7 mJ

<b>Class</b>	<b>IV</b>	<b>IV</b>
--------------	-----------	-----------

### Laser Safety Glasses

<b>Wavelength</b>	1064nm	225-2200 nm
<b>OD (min)</b>	6	5

## OPOTEK Marius Schmidt Nanosecond Laser System (University of Wisconsin at Milwaukee)

This laser is owned by and resides at the University of Wisconsin at Milwaukee.  
It is only occasionally used at BioCARS.

<b>ANL IHID #</b>	This laser is a component of 10853	10853
<b>Brand</b>	Quantel	OPOTEK
<b>Model</b>	Brio	Opolette HE 355 II
<b>Serial #</b>	1008180309	2272
<b>Type</b>	Nd:YAG	OPO
<b>Beam Characteristics</b>		
<b>Wavelength</b>	355 nm	410-2200 nm
<b>Diameter</b>	~3 mm	~3 mm
<b>Divergence</b>	<10 mrad	<1 mrad
<b>Mode</b>	pulsed	pulsed
<b>Pulse Width</b>	5ns	5ns
<b>Rep. Rate</b>	20Hz	20Hz
<b>Power/ Pulse Energy</b>	35 mJ	4-10 mJ
<b>Class</b>	<b>IV</b>	<b>IV</b>
<b>Laser Safety Glasses</b>		
<b>Wavelength</b>	355 nm	410-2200 nm
<b>OD (min)</b>	5	5

## OPOTEK ChemMatCARS Nanosecond Laser System

This laser is owned by and resides at ChemMatCARS.  
It is only occasionally used at BioCARS.

<b>ANL IHID #</b>	This laser is a component of 11299	11299
<b>Brand</b>		OPOTEK
<b>Model</b>		Opolette HE355-LD
<b>Serial #</b>		3827
<b>Type</b>	Nd:YAG	OPO
<b>Beam Characteristics</b>		
<b>Wavelength</b>	355 nm	225-2200 nm
<b>Diameter</b>	~4 mm	~4 mm
<b>Divergence</b>		<1.5 mrad
<b>Mode</b>	pulsed	pulsed
<b>Pulse Width</b>	5 ns	5 ns
<b>Rep. Rate</b>	20Hz	20Hz
<b>Power/ Pulse Energy</b>		4-10 mJ
<b>Class</b>	<b>IV</b>	<b>IV</b>
<b>Laser Safety Glasses</b>		
<b>Wavelength</b>	355 nm	410-2200 nm
<b>OD (min)</b>	5	5

## ChemMatCARS 532nm CW laser

This laser is owned by and resides at sector 15 (Yu-Sheng Chen). It is only occasionally used at BioCARS.  
The output beam of this laser is typically coupled to an optical fiber for light transport to the sample.

<b>ANL IHID #</b>	10800
<b>Brand</b>	Crystal Laser
<b>Model</b>	CL532-025-L
<b>Serial #</b>	2912006-4079-26702
<b>Type</b>	Diode laser
<b>Beam Characteristics</b>	
<b>Wavelength</b>	532 nm
<b>Diameter</b>	0.32 mm
<b>Divergence</b>	2 mrad
<b>Mode</b>	CW
<b>Pulse Width</b>	
<b>Power/ Pulse Energy</b>	<30mW@ 532nm
<b>Class</b>	<b>IIIB</b>
<b>Laser Safety Glasses</b>	
<b>Wavelength</b>	532 nm
<b>OD (min)</b>	2-3

## ChemMatCARS 532nm CW laser

This laser is owned by and resides at sector 15 (Yu-Sheng Chen). It is only occasionally used at BioCARS. The output beam of this laser is typically coupled to an optical fiber.

<b>ANL IHID #</b>	10860
<b>Brand</b>	Coherent
<b>Model</b>	315M-100
<b>Serial #</b>	H030326263
<b>Type</b>	Diode pumped solid state

### Beam Characteristics

<b>Wavelength</b>	532 nm
<b>Diameter</b>	0.32 mm
<b>Divergence</b>	<2.2 mrad
<b>Mode</b>	CW
<b>Pulse Width</b>	
<b>Power/ Pulse Energy</b>	100mW@ 532nm

<b>Class</b>	<b>IIIB</b>
--------------	-------------

### Laser Safety Glasses

<b>Wavelength</b>	532 nm
<b>OD (min)</b>	2-3

## BioCARS HeNe lasers

<b>ANL IHID #</b>	10467	10468	10469
<b>Brand</b>	Res. Elec.-Opt	Res. Elec.-Opt	Res. Elec.-Opt
<b>Model</b>	LHGR-0100M	LHGR-0035M	LHRR-0100
<b>Serial #</b>		3203-1291-1737	3203-1291-29
<b>Type</b>	He-Ne	He-Ne	He-Ne

### Beam Characteristics

<b>Wavelength</b>	543 nm	543 nm	633 nm
<b>Diameter</b>	0.9 mm	0.9 mm	0.84 mm
<b>Divergence</b>	1.2 mrad	1.2 mrad	0.9 mrad
<b>Mode</b>	CW	CW	CW
<b>Pulse Width</b>			
<b>Rep. Rate</b>			
<b>Power/ Pulse Energy</b>	<5mW	<5mW	1 mW

<b>Class</b>	IIIa	IIIa	IIIa
--------------	------	------	------

### Laser Safety Glasses

OD (Wavelength)

## NIH/NIDDK 473nm CW laser

This laser is owned by and resides at NIDDK/NIH (Philip Anfinrud). It is only occasionally used at BioCARS.

<b>ANL IHID #</b>	10836
<b>Brand</b>	CNI
<b>Model</b>	MLL-III-483
<b>Serial #</b>	
<b>Type</b>	LD pumped all-solid-state

### Beam Characteristics

<b>Wavelength</b>	473nm
<b>Diameter</b>	2mm
<b>Divergence</b>	1.5mrad
<b>Mode</b>	CW
<b>Pulse Width</b>	
<b>Power/ Pulse Energy</b>	100mW

<b>Class</b>	<b>IIIB</b>
--------------	-------------

### Laser Safety Glasses

<b>Wavelength</b>	473nm
<b>OD (min)</b>	5

## ChemMatCARS CW Laser Diodes

These lasers are owned by and reside at sector 15. They are occasionally used at BioCARS.  
For these disposable lasers, ANL IHID #s are assigned to a particular class of lasers, not each laser head  
(multiple heads are available for each class).

<b>ANL IHID #</b>	10912 (4 heads)	10996 (2 heads)	10995 (1 head)
<b>Brand</b>			
<b>Model</b>	M650D150-3-1670	M808D150-3-1670	M405D150-3-1670
<b>Serial #</b>	AK1894BA	AK1894CO	AK1894EM
<b>Type</b>	Laser diode	Laser diode	Laser diode
<b>Beam Characteristics</b>			
<b>Wavelength</b>	650nm	808nm	405nm
<b>Diameter</b>	0.5mm	0.5mm	0.5mm
<b>Divergence</b>	0.1mrad	0.1mrad	0.1mrad
<b>Mode</b>	CW		
<b>Pulse Width</b>			
<b>Power/ Pulse Energy</b>	150mW	150mW	150mW
<b>Class</b>	<b>IIIB</b>	<b>IIIB</b>	<b>IIIB</b>
<b>Laser Safety Glasses</b>			
<b>Wavelength</b>	650nm	808nm	405nm
<b>OD (min)</b>	5	5	5



## BioCARS and ChemMatCARS High-Power LEDs

These LEDs are incoherent, broad-band optical light sources. They are not included and classified in laser safety standard but we assigned laser Class based on output laser power. The output beam of these lasers is typically coupled to an optical fiber. Yu Sheng Chen is custodian for these LEDs.

<b>ANL IHID #</b>	11002	11003	11004	11001	L0004	L0005
<b>Brand</b>	ThorLabs	ThorLabs	ThorLabs	ThorLabs	ThorLabs	ThorLabs
<b>Model</b>	M385L2	M505L3	M660L3	M365L2	M362LP1	M450LP1
<b>Serial #</b>	M00289724	M00290739	M00289182		M00520466	M00531119
<b>Type</b>	High-power LED	High-power LED	High-power LED	High-power LED	High-power LED	High-poer LED
<b>Beam Characteristics</b>						
<b>Wavelength</b>	385 nm	505 nm	660 nm	365 nm	365 nm	445 nm
<b>Diameter</b>					1.4 mm	1.5 mm
<b>Divergence</b>					4188 mrad	4880 mrad
<b>Mode</b>	CW	CW	CW	CW	CW	CW
<b>Pulse Width</b>						
<b>Power/ Pulse Energy</b>	270mW	400mW	640mW	190mW	1.4W	2.1W
<b>Class</b>	<b>IIIB</b>	<b>IIIB</b>	<b>IV</b>	<b>IIIB</b>	<b>IIIA (3R)</b>	<b>IIIB (3R)</b>
<b>Laser Safety Glasses</b>						
<b>Wavelength</b>	385 nm	505nm	660nm	365nm	365nm	445nm
<b>OD (min)</b>	5	4-5	4-5	5		

## NIH/NIDDK CW laser diodes

These laser diodes are owned by NIDDK/NIH (Philip Anfinrud). They are typically fiber-coupled.

<b>ANL IHID #</b>	11034	11205	11036	11204	11261	11226	11227
<b>Brand</b>	ThorLabs	ThorLabs	ThorLabs	Thorlabs	Thorlabs	ThorLabs	CNI
<b>Model</b>	CPS532	LP520-SF15	LP520-SF15	LP462-MF1W	L462P1400MM	L462P1400MM	SLD-488-60
<b>Serial #</b>	C140331-231			17102451			
<b>Type</b>	Laser Diode	SM-Fiber-Pigtailed Laser Diode	SM-Fiber-Pigtailed Laser Diode	Laser Diode	Laser Diode	Laser Diode	Laser diode
<b>Beam Characteristics</b>							
<b>Wavelength</b>	532.2nm	520nm	520nm	462nm	462nm	462nm	488nm
<b>Diameter</b>				1mm	1mm		1.7mm
<b>Divergence</b>				1mrad	192mrad		0.367mrad
<b>Mode</b>	CW	CW	CW	CW	CW	CW	CW
<b>Pulse Width</b>							
<b>Power/ Pulse Energy</b>	4.4mW	15mW	15mW	1.1W (1W nominal)	1.4W (0.5W nominal)	1.4W (0.14W nominal)	60mW
<b>Class</b>	<b>IIIA</b>	<b>IIIB</b>	<b>IIIB</b>	<b>IV</b>	<b>IV</b>	<b>IV</b>	<b>IIIB</b>
<b>Laser Safety Glasses</b>							
<b>Wavelength</b>	532nm	520nm	520nm	462nm	462nm	462nm	488nm
<b>OD (min)</b>	4-5	4-5	4-5	5	5	5	4-5

## Appendix 2

## List of authorized personnel

I have read and understood the SOP for laser use at Sectors 14

<b>Person</b>	<b>Badge Number</b>	<b>Signature/Date</b>	<b>Eye Exam Completed (y/n &amp; date)</b>
Vukica Srajer (LCA Supervisor and Principal Laser Operator)	85120		Y, 3/13/1998
Robert Henning (Principal Laser Operator)	50901		Y, 5/13/2008
Irina Kosheleva (Principal Laser Operator)	48119		Y, 8/17/2009
Philip Anfinrud (Expert Laser Operator)	68187		Y, 1/4/2007
Hyun Sun Cho	80917		Y, 1/4/2007
Friedrich Schotte	69453		Y, 2/12/2006
Tim Graber	85342		Y, 1/7/2002
Stephen M. Durbin	85217		Y, 8/3/2010
Jason B. Benedict	82618		Y, 3/9/2012
Jan O. Davidsson	225898		Y/ 10/3/2012
Anne Marie March	58856		Y, 2009
Gilles Doumy	59634		Y, 2010
Janne Ihalainen	231573		Y, 5/16/2013
Heli Lehtivuori	232258		Y, 5/17/2013

**List of authorized personnel (Continuation)**

**I have read and understood the SOP for laser use at Sectors 14**

<b>Person</b>	<b>Badge Number</b>	<b>Signature/Date</b>	<b>Eye Exam Completed (y/n &amp; date)</b>
Heikki Takala	231711		Y, 5/17/2013
Anthony DiChiara	230980		Y, 6/22/2013
Radoslaw Kaminski	205182		Y, 10/17/2014
Oscar Berntsson	239693		Y, 2/8/2016
Matthys Panman	284154		Y, 3/15/2016
Julian Bergtholdt	286394		Y, 7/6/2016
Dennis Dierksmeyer	286040		Y, 6/28/2016
Max Wiedorn	284230		Y, 4/13/2017
Valentyn Stadnytsky	285723		Y, 9/22/2017
Zhong Ren	85140		N/A
Mateo Levantino	305414		N/A
Yu Sheng Chen	88772		N/A

Appendix 3

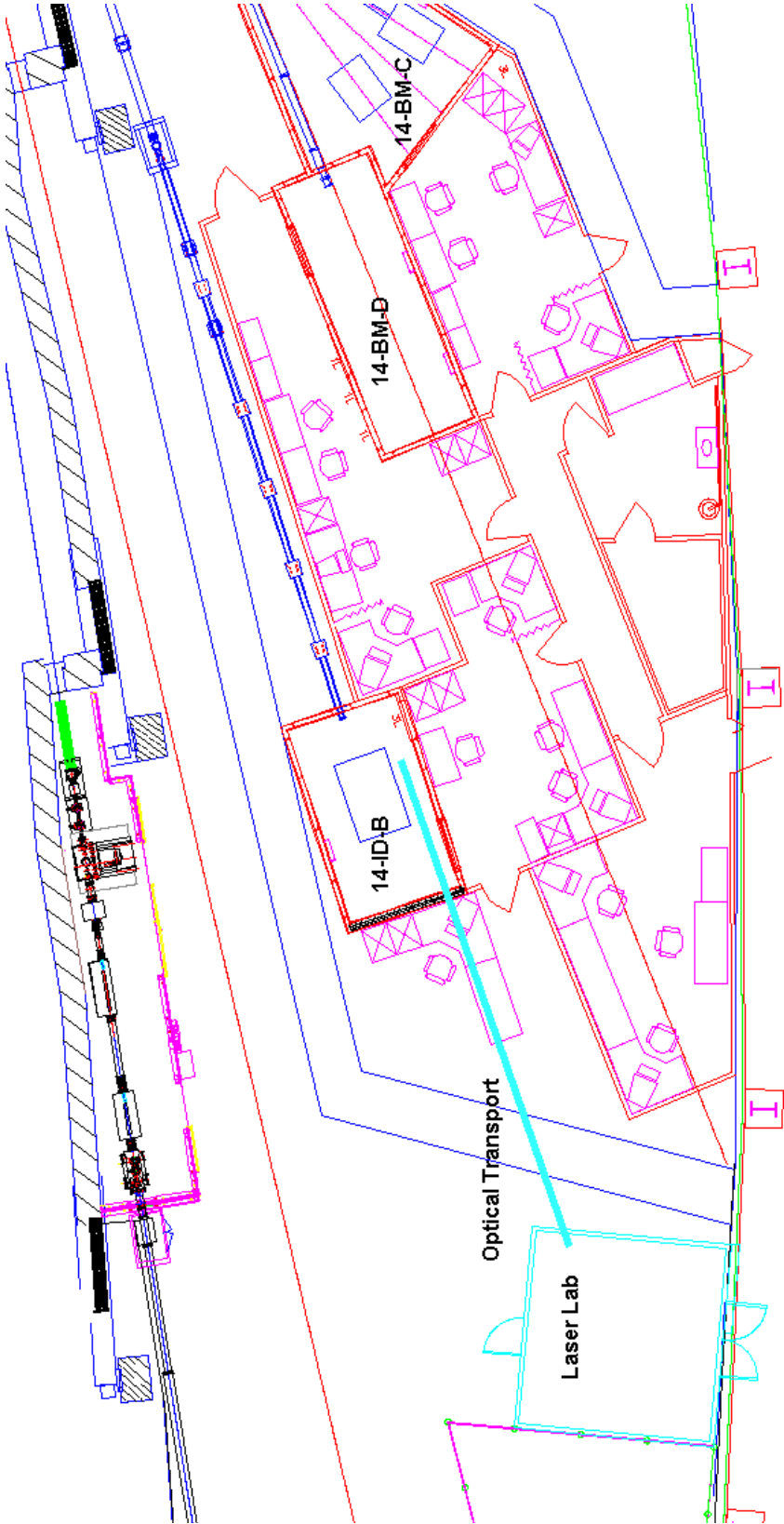


Figure 1

Sector-14

## Appendix 4

### Laser Eyewear located at Sectors 14

#### **Glendale LSR-MED Argon LGW LRP12 (1)**

488 – 515 nm	OD > 4.5
190 – 488 nm	OD > 7

#### **Glendale ARG/Nd:YAG LGW (1)**

850 – 1080 nm	OD > 7
750 – 850 nm	OD > 5
710 – 750 nm	OD > 3
520 – 532 nm	OD > 7
190 – 520 nm	OD > 9

#### **Laser Vision L614 LGT (1)**

680 – 900 nm	OD > 5
580 – 680 nm	OD > 6
570 – 580 nm	OD > 5

#### **Uvex LOTG-YAG/KTP (2)**

1064 nm	OD > 7
865 – 1063 nm	OD > 5
840 – 864 nm	OD > 4
800 – 839 nm	OD > 3
190 – 532 nm	OD > 7

#### **Uvex LOTG-ARGON ALNG (1)**

488 – 515 nm	OD 2 – 3
190 – 380 nm	OD 7

#### **Uvex LOTG-Broadband A (2)**

755 – 1064 nm	OD 6
731 – 754 nm	OD 4 – 5
710 – 730 nm	OD 3 – 4
190 – 532 nm	OD 7

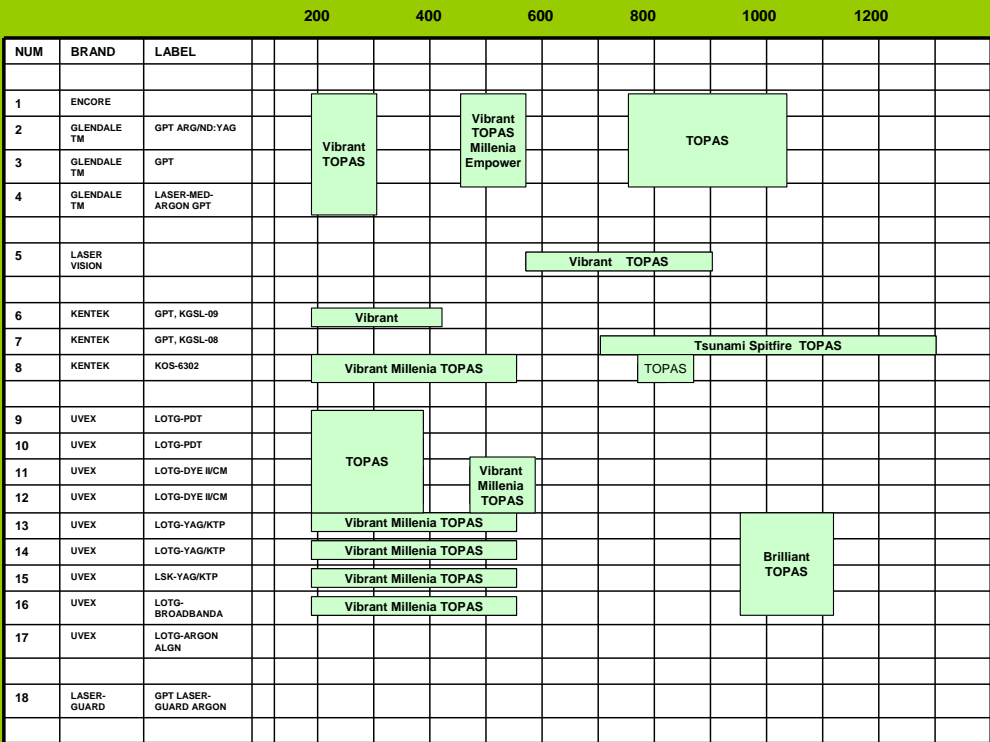
#### **Uvex LOTG-PTD (1)**

610 – 695 nm	OD 4 – 5
585 – 605 nm	OD 2 – 3
190 – 380 nm	OD 7

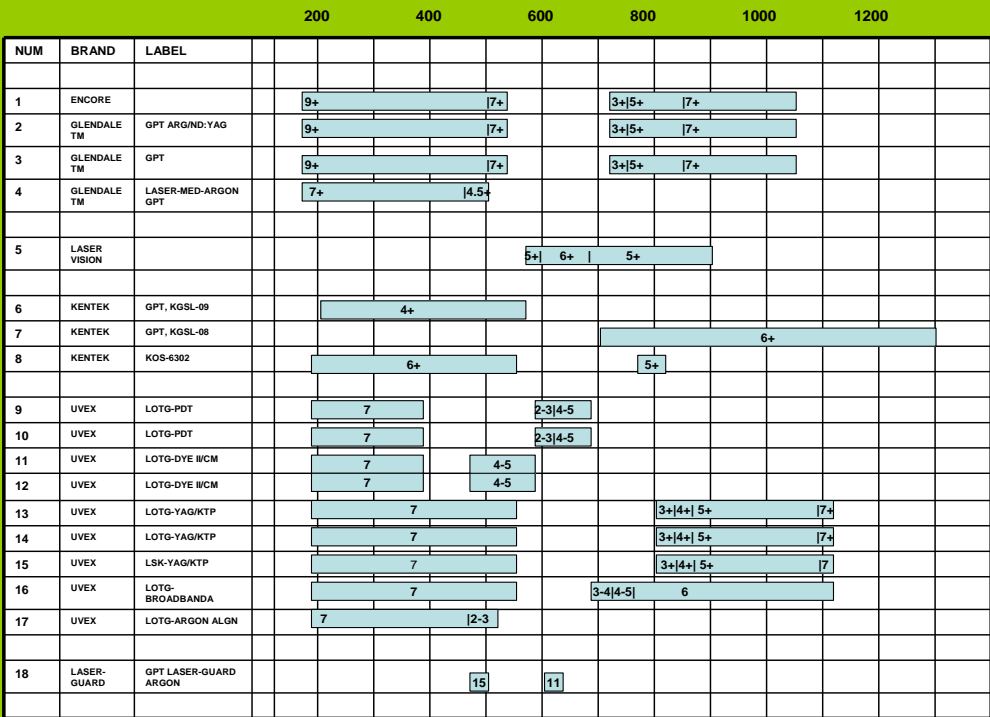
#### **Kentek LOTG-PTD (1)**

800 – 815 nm	OD 6+
785 – 820 nm	OD 5+
190 – 532 nm	OD 6+

**SUGGESTED CHOICE OF LASER FOR GOGGLES AS FUNCTION OF WAVELENGTH**  
Wavelength, nm



**OPTICAL DENSITY OF GOGGLES AS FUNCTION OF WAVELENGTH**  
Wavelength, nm



Optical density is a measure of the transmittance of an optical element for a given length at a given wavelength  $\lambda$  [1]:  
 $OD_{\lambda} = -\log_{10} T = -\log_{10}(I_t/I_0)$

$T$  = the per-unit transmittance  
 $I_0$  = the intensity of the incident light beam     $I_t$  = the intensity of the transmitted light beam  
 1.R.Henderson and K. Schulmeister, 2004, Laser Safety, p.409-410

Poster located in LCA to aid in the selection of safety goggles. All goggles are numbered for easy identification.

Appendix 5

**Laser Interlock System Test**

Verify the functionality of the interlock system as described in the SOP.

- Check the status of the LCA warning lights according table below
- Confirm the proper operation of the external laser shutter for the active laser system
- Test emergency shut-down (test does not require accessible beam in the LCA).

Laser Energized	Laser Shutter Open	Laser Cover or EOB Open	Warning Light
No	-	-	Green
Yes	No	No	Yellow
Yes	Yes	No	Yellow
Yes	No	Yes	Yellow
Yes	Yes	Yes	Red

Green: No Hazard Laser Off  
Yellow: Caution Laser Energized  
Red: Danger Beams accessible





# On-the-Job Laser Systems Alignment Safety Training

Each of the users listed in this table have passed the OJAT course

<b>Name</b>	<b>Badge</b>	<b>Room</b>	<b>*ANL Laser Registration ID</b>	<b>Scope of alignment covered</b>	<b>Date Completed</b>
Timothy Graber	85342	14ID-6	10498 10499 10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	4/14/2010
Philip Anfinrud	68187	14ID-6	10498 10499 10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	4/15/2010
Hyun Sun Cho	80917	14ID-6	10498 10499 10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	4/15/2010
Robert Henning	50901	14ID-6	10498 10499 10690 10691 10692 10693 10694 10695	Picosecond laser wavelength change	4/14/2010
Irina Kosheleva	48119	14ID-6	10498 10499 10690 10691 10692 10693 10694 10695	Picosecond laser wavelength change	3/14/2010
Robert Henning	50901	14ID-6 14ID-B	10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	10/19/2012
Irina Kosheleva	48119	14ID-6 14ID-B	10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	10/19/2012
Robert Henning	50901	14ID-6 14ID-B	10882 10853 10800 10860	Nanosecond and IIIB cw laser alignment	9/27/2012
Irina Kosheleva	48119	14ID-6 14ID-B	10882 10853 10800 10860	Nanosecond and IIIB cw laser alignment	9/27/2012
Robert Henning	50901	14ID-6	10691 10692	Coupling ns laser pulses	1/30/2013

				from Empower laser into optical fiber	
Irina Kosheleva	48119	14ID-6	10691 10692	Coupling ns laser pulses from Empower laser into optical fiber	1/30/2013
Anthony DiChiara	230980	14ID-6 14ID-B	10690 10691 10692 10693 10694 10695	Picosecond laser alignment and wavelength change	6/28/2013

\* 10498 and 10499 IDs refer to the obsolete Opotek Brilliant Nd:YAG and Vibrant OPO ns laser systems. These are replaced by similar ns laser systems: Quantel Nd:YAG and Opotek Opolette OPO, number 7 and 8 in Appendix 1, ID 10882.

## **Appendix C.12 BioCARS Heavy Metal Soaking Solution Use, CARS Sector 14 (March 1999)**

BioCARS - Sector 14

Heavy Metal Soaking Solution Use

(Formally known as "Protocol for Handling Heavy Metal Soaking Solutions for Isomorphous Replacement in Protein Crystals at CARS Sector 14")

1. Post the "Caution: Heavy Metal Solutions" sign in the working area prior to usage.
2. Ensure that containers of solutions containing heavy atoms are clearly labeled, identifying contents, owner's name, contact telephone number and date.

Heavy metal solutions can only be made up in the 434B030 Chemistry Lab. Safety glasses and appropriate hole-free liquid resistant gloves must be worn. Containers of volatile heavy metal solutions shall be opened under an operating hood in this lab. The area in the hood or the bench top where heavy metals solutions are used must be lined with absorbent material. You MUST NOT allow heavy metal solution to enter the lab sinks. Work in the containment trays provided.

Quantities of less than 1 ml of solution, containing less than 0.1 M of heavy metals, may be removed from the 434B030 laboratory in labeled and tightly sealed containers and transported to the sample preparation area at the beamline for soaking crystals.

3. Soaking crystals at the beamline shall be performed in a Heavy Metal containment tray; absolutely no exceptions. The tray must be lined with absorbent material.
4. Spills outside the containment tray, of even a few drops, shall be wiped up immediately with absorbent material. (Again, safety glasses and gloves must be worn.) Wipes must NOT be disposed of in the general waste stream but must be bagged and logged into our hazardous waste system. Notify BioCARS staff immediately of any heavy metal spill that escapes the containment tray.
5. Any liquid or solid waste, including gloves, absorbent material and other contaminated material should be bagged, clearly labeled and disposed of as hazardous waste. Contact the BioCARS staff to insure proper disposal.
6. Disposal of heavy atom solutions at CARS must be indicated on the ESA form. It is preferred that heavy atom solutions be returned to the user's home laboratory at the finish of the experiment.

Navrotski

Updated 06 Mar 1999

[Based on SSRL model: <https://biosgl.slac.stanford.edu/hardware/heavymetal.html>]

## **Appendix C.13 BioCARS SOP for CO use at Sector 14**

Carbon monoxide will be used in small quantities (of the order of 1ml/min over an 1h period) for preparation of biological samples. Same procedure will be used for other toxic gases (such as H<sub>2</sub>S, (CN)<sub>2</sub>) if need arises for their use at Sector 14.

### **Hazards**

Toxic gas with no warning properties; decreases the ability of the blood to carry oxygen to the tissues. It is also flammable. (See attached MSDS from Matheson Gas Products and Howard Hughes Medical Institute).

### **Control measures**

- CO will be used only in the continuously vented chemical hood of the chemistry lab at Sector 14. The hood needs to be free of strong oxidizers, strong reducing agents, interhalogen compounds (such as ClF<sub>3</sub> and BrF<sub>3</sub>), sodium and potassium or Cl<sub>2</sub>.
- A carbon monoxide monitor will be installed in the chemistry lab, next to the chemical hood.
- The CO cylinder size to be used should be no larger than 15ft<sup>3</sup> and no more than 1 cylinder should be present on site.
- The cylinder will be placed upright, properly secured (with appropriate stands) and tagged with “full/in-use/empty” labels.
- The cylinder in use will be properly connected to a pressure regulator to prevent gas leakage.
- Main cylinder valve will be tested for functionality at least every six months.
- When not in use, CO cylinder will be stored in the outdoor gas cage “Sector 14” in the central storage area of LOM 434

### **Operating procedure**

- Make sure the chemical hood is working before use of the CO gas.
- Make sure to close main cylinder and the regulator valves when finished with use.
- Post a sign at the hood when CO gas is in use.

**Appendix C.14 BioCARS - SOP for Biosafety Level 2 Experiments at BioCARS Facility (November, 2014)**

**NOTE: Blue text highlights differences for BSL2 and BSL3 SOPs**

---

**Standard Operating Procedures for  
Biosafety Level 2 Experiments at BioCARS Facility**

---

July, 2008  
Revised February, 2014  
Revised November, 2014

Prepared by:  
Vukica Srajer

Approved by:

	<i>Signature</i>	<i>Date</i>
Dan Schabacker, ANL IBC Chair	_____	_____
Bruce Glagola, APS User Safety Officer	_____	_____
Nena Moonier, APS Biosafety Officer	_____	_____
Keith Moffat, BioCARS PI	_____	_____
Guy Macha, CARS Safety Officer	_____	_____
Vukica Srajer, BioCARS Safety and Biosafety Coordinator	_____	_____

BioCARS maintains as-built drawings, specifications, and control system logic descriptions for the ventilation system as well as the manufacturer's instruction manuals for all units that comprise the HVAC system. BioCARS also maintains documentation that demonstrates performance of the HVAC system, malfunctions and their resolution.

Annual re-certification of the BioCARS facility for the BSL-2/3 operation is initiated by the APS Biosafety Officer. Re-certification is based on procedures approved by the ANL IBC and will be performed by the appropriate APS/ANL personnel. The ANL IBC maintains the records and results of the BioCARS re-certification. A copy of the results is provided to BioCARS.

BioCARS maintains completed forms included in this document, and evidence of medical surveillance and respirator training for BioCARS staff.

BioCARS Biosafety Coordinator will review this SOP as needed and make revisions based on experience and changing facility conditions (the log of reviews is kept in the BioCARS BSL-2/3 Maintenance Logbook). BioCARS Biosafety Coordinator will also revise this SOP if there are major changes in the engineering or administrative controls. Any major revisions of the engineering or administrative controls are subject to approval of the SOP by the ANL IBC.

**Blue text in this document represents differences between BSL-2 and BSL-3 SOPs.**

**Abbreviations:**

AHU:	Air Handling Unit
ANL:	Argonne National Laboratory
APS:	Advanced Photon Source
BSC:	Biosafety Cabinet
BSL-2:	Biosafety Level 2
BSL-3:	Biosafety Level 3
EM:	Emergency Management, ANL Security and Counterintelligence Division
EQO-IH:	ANL Industrial Hygiene
FMS:	Facility and Management Services Division for Argonne National Laboratory
HVAC:	Heating, Ventilation and Air Conditioning
IBC:	Institutional Biosafety Committee
JC:	Johnson Controls
MSDS:	Material Safety Data Sheet
PAPR:	Powered Air Purifying Respirators
VHP:	Vapor-phase Hydrogen Peroxide



## Standard Operating Procedures for Biosafety Level 2 Experiments at BioCARS Facility

### Overview of the Facility and BSL-2 Procedures

#### 1. BioCARS Facility

- 1.1 BioCARS Facility is a National User Facility for Macromolecular Crystallography where users from universities and other research institutions conduct X-ray diffraction experiments on crystals of macromolecules. It is located at Sector 14 at the Advanced Photon Source (APS), Argonne National Laboratory (ANL).
- 1.2 Majority of BioCARS users conduct crystallographic experiments that do not require biocontainment. Typical experiments require 1-3 days.
- 1.3 A sub-group of BioCARS users conducts experiments involving samples that require BSL-2 or BSL-3 biocontainment. Agents classified as BSL-2 involve a broad range of indigenous moderate-risk agents that are present in the community and associated with human disease of varying severity resulting from exposure via contact with mucous membranes, contact with non-intact skin, percutaneous injury, or ingestion. Agents classified as BSL-3 are indigenous or exotic agents associated with serious and potentially lethal human diseases as a result of exposure by the inhalation route. Other agents may include toxins, in which the primary risk is to the experimenter.
- 1.4 Because BioCARS is a facility where both standard and BSL-2/3 experiments are conducted, particular care is taken to properly prepare the facility for the BSL-2/3 operation and to properly return the facility to standard, non-BSL-2/3 operation upon completion of a BSL-2/3 experiment.
- 1.5 The facility consists of one bending magnet experimental station (14-BM-C) and one insertion device station (14-ID-B). Second bending magnet station, 14-BM-D, is no longer in use.
- 1.6 All experimental stations (hutches) are qualified for BSL-2/3 experiments.
- 1.7 Only a single BSL-2/3 experiment can be conducted at the facility at any time, in one of the BioCARS experimental stations. Other, non-BSL2/3 experiments may be conducted in other two experimental stations at the same time if approved by the ANL IBC for a particular BSL-3 agent.
- 1.8 Cultures of BSL-2/3 microorganisms are prohibited at BioCARS.
- 1.9 There is no long term storage of BSL-2/3 materials at the facility. BSL-2/3 material is shipped by users before the experiment and disposed or shipped back to home institution upon the completion of the experiment (**SOP 103**).
- 1.10 Use of BSL-4 agents and select agents (the latter as defined in, **42 CFR Part 73, 7 CFR Part 331 and 9 CFR Part 121**) is prohibited at BioCARS.

- 1.11 The BioCARS users working with the BSL-2/3 agents have to be qualified and trained at their home institution for working with such materials (**SOP 102, SOP 104**).
- 1.12 BioCARS staff will not be involved in direct work with BSL-2/3 agents. Staff will, however, assist users with the use of BioCARS equipment as well as address hardware and software problems during a BSL-2/3 experiment.
- 1.13 The facility is equipped with a Class II, Type B2 Biosafety Cabinet (BSC). The schematics of the BSC and the BSC practices and procedures are in the **Appendix B**. The BSC is recertified by a certified vendor annually and after any maintenance and ventilation system modifications.
- 1.14 All unpacked BSL-2/3 material will be stored in the BSC room upon arrival. All manipulations of open (uncontained) BSL-2 samples will be conducted in the BSC to the greatest extent possible. [Safety glasses with side shields are required for manipulation of BSL-2 material outside of the BSC within the biocontainment area \(see 1.2.2\).](#) [NIOSH approved face masks are also required is samples are not contained.](#)
- 1.15 BioCARS Chemistry Lab and Cold room will not be used for storage or work with BSL-2/3 agents. Cold room cannot be used while BSL-2/3 samples or waste are present in the BSC room.
- 1.16 Only a single crystal of BSL-2 material will be transferred from the BSC or from a dewar with contained, frozen crystals in the BSC or control room to the experimental station where the X-ray diffraction experiment is conducted. A two-person rule is in effect during the crystal transfer. A detailed description of sample containment and transfer is given in **SOP 107**.
- 1.17 [24h attendance is not routinely required for BSL-2 experiments but BioCARS Biosafety Coordinator and user spokesperson will be on-call in case of an incident or emergency. For some BSL-2 experiments, 24h attendance might be warranted by the hazard level of the particular BSL-2 agent as determined by ANL IBC \(SOP 107\). In those cases, one user and one BioCARS staff member have to be present in the BioCARS facility or LOM.](#)

## 2. Facility Layout and Air Handling System

- 2.1 The layout of the facility is shown in the **Appendix A**.
- 2.2 BSL-2 biocontainment area during a BSL-2 experiment consists of:
  - a) one experimental station (hutch)
  - b) adjacent control room
  - c) the facility corridor (14-BM-7)
  - d) the BSC room (14-BM-9)
- 2.3 The shower, sink and eye wash station are located in the BSC room.
- 2.4 The only entrance into the BSL-2 biocontainment area is via computer room (14-BM-10) during the BSL-2 experiment. The door between the BSL-2 control area and the APS experimental hall is locked to prevent

entry from the experimental hall, but allows emergency exit from the control room.

- 2.5 The BioCARS HVAC system consists of 5 Air Handling Units (AHU) connected to the main APS conditioned air supply.
- 2.6 The HVAC system controls are pre-set for two operating modes: normal mode (non-BSL-2/3 operation) and biocontainment mode (BSL-2/3 operation).
- 2.7 Routine BSL-2 experiments are conducted using the normal HVAC mode. If determined necessary by the ANL IBC, biocontainment mode will be implemented. In those cases, prior to a BSL-2 experiment, one BioCARS experimental station is declared a BSL-2 area and the HVAC is switched from standard to biocontainment mode. Both tasks are responsibility of BioCARS staff (**SOP 101**). The specific steps of preparing the facility for the BSL-2 operation in biocontainment mode are described in **SOP 106**.
- 2.8 The control unit for changing HVAC configuration (JC-Control Box) is located on the roof of the facility. Access to the roof is limited to BioCARS staff and APS Floor Coordinators by locking the door for the duration of the BSL-2 experiments.
- 2.9 In the biocontainment mode, the selected BSL-2 containment area (2.2) is under negative pressure with respect to the APS experimental hall.
- 2.10 In the biocontainment mode, directional airflow is established in the facility as shown in **Appendix A**. In this mode, the non-BSL-2 control rooms and stations are sealed (**SOP 6**) to prevent unwanted air-flow from the BSL-2 containment area.
- 2.11 Exhaust air from the BSL-2 containment area is HEPA filtered and directed out of the APS building via either the BSC exhaust or a bag-in/bag-out HEPA filter located downstream of the exhaust port from each experimental station. The BioCARS exhaust system is independent of any other APS exhaust system.
- 2.12 All doors within the facility must remain closed when not in use for the duration of the BSL-2 experiment.
- 2.13 The status of the HVAC system can be monitored via Epics Status Screen on computers in the control and computer rooms (14-BM-5, 14-BM-6, 14-ID-3, 14-BM-10). The status can also be checked on the Metasys Terminal at the JC-Control Box, located on the roof of the BioCARS Facility (**Checklist 0**).
- 2.14 An audible alarm will alert experimenters in the facility and BioCARS staff in the office area (LOM) about an HVAC fault condition (**SOP 109**).
- 2.15 The main exhaust fan (EF104) for the facility and the Biosafety Cabinet are connected to the APS emergency power supply (**SOP 109**).

### 3. Access to the Facility

- 3.1 The entrance to and the exit from the facility during the BSL-2 experiments are through the computer room (14-BM-10). The access to the containment area is restricted to authorized personnel during the BSL-2 experiments. The list of authorized personnel for each BSL-2

experiment must be posted at the facility entrance door (from the APS experimental hall) during the experiment (**SOP 106**). Same list must also be posted at the entrance to the containment area (on the door to the BioCARS corridor). A removable chain must also be placed in front of the entrance to the containment area (See **Checklist 2**).

- 3.2** User authorization for each BSL-2 experiment requires:
- a) signing that they have read and understood this SOP (**Checklist 1**)
  - b) signing the Risk Factors Form (**Appendix D**)
  - c) letter by the user home institution IBC authorizing work with the specified BSL-2 agent (**BioCARS questionnaire, Appendix C, 4.1**)
- 3.3** BioCARS staff authorization for each BSL-2 experiment (as staff contact or technical support) requires:
- a) signing that they have read and understood this SOP (**list at the end of the SOP, before Appendix A**)
  - b) signing the Risk Factors Form (**Appendix D**)
  - c) [current medical fitness evaluation if mandated by the BioCARS PI \(arranged through University of Chicago Occupational Medicine\)](#)
  - d) completion of the ANL ESH560 training “Biosafety Awareness Training”
- 3.4** Vaccination to the BSL-2 agent (when a vaccine is available) may be required for access by discretion of the BioCARS PI. When a vaccine is available but vaccination is not declared mandatory, BioCARS personnel must sign a vaccine declination form if they choose to not be vaccinated. All personnel that are offered a vaccine will be trained on the vaccine’s benefits and risks before they are asked if they want to receive it.

#### **4. Protective clothing and other PPE**

Protective clothing, gowning and de-gowning procedures are explained in **SOP 105**.

#### **5. Responsibilities**

Responsibilities of users, their home institutions, BioCARS staff, BioCARS Safety Committee and ANL/APS safety personnel are listed in **SOP 101**.

#### **6. Incident Response**

An incident is defined as an event that creates overt or potential exposure of users or staff to a BSL-2 agent, such as loss of a crystal outside of the biosafety cabinet (this is considered to be a breach of primary containment of the sample), loss of BioCARS biocontainment where biocontainment area is defined in 2.2.2 above (this is considered to be a breach of secondary containment) or failure of critical equipment (BSC, crystal cryo-cooler). Every incident will be reported by the BioCARS Biosafety Coordinator to the CARS Safety Committee, ANL IBC and APS.

## Standard Operating Procedures and Checklists:

### Standard Operating Procedures:

<b>SOP 101</b> (p. 8):	Responsibilities	(BioCARS users and staff)
<b>SOP 102</b> (p. 11):	Approval of BSL-2 Experiments	(BioCARS users and staff)
<b>SOP 103</b> (p. 12):	Shipping and Receiving of BSL-2 Samples	(BioCARS users and staff)
<b>SOP 104</b> (p. 15):	User Training and Certification	(BioCARS users and staff)
<b>SOP 105</b> (p. 17):	Protective Clothing and Other Personal Protective Equipment	(BioCARS users and staff)
<b>SOP 106</b> (p. 19):	Preparation of the Facility for BSL-2 Operation	(BioCARS staff and APS floor coordinator)
<b>SOP 107</b> (p. 20):	BSL-2 Practices and Procedures	(BioCARS users and staff)
<b>SOP 108</b> (p. 25):	Response to Overt or Potential Personnel Exposure to BSL-2 Agent	(BioCARS users, staff and APS Floor Coordinator)
<b>SOP 109</b> (p. 27):	Response to the Failure of Facility HVAC and Other Equipment	(Users, BioCARS staff and APS Floor Coordinator)
<b>SOP 110</b> (p. 29):	Medical and Other Emergencies During the BSL-2 Experiments	(Users, BioCARS staff, APS floor coordinator and APS/ANL emergency response team)
<b>SOP 111</b> (p. 30):	BSL-2 Waste Disposal	(Users, BioCARS staff and APS Floor Coordinator)
<b>SOP 112</b> (p. 31):	End of Experiment Procedures	(BioCARS users and staff)
<b>SOP 113</b> (p. 32):	HVAC and other Safety Equipment Maintenance and Testing	(BioCARS staff and PSF personnel)

### Appendices:

<b>Appendix A</b> (p. 45):	BioCARS Facility Layout
<b>Appendix B</b> (p. 46):	BioCARS Biosafety Cabinet
<b>Appendix C</b> (p. 50):	Questionnaire for BioCARS Proposals Involving Viable BSL-2 and BSL-3 Viruses
<b>Appendix D</b> (p. 58):	Risk Factors Form
<b>Appendix E</b> (p. 59):	BSL-2 Sample Log-in/Log-out Form
<b>Appendix F</b> (p. 61):	Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS

### Checklists:

<b>Checklist 0</b> (p. 34):	Preparation of the HVAC and BSC
<b>Checklist 1</b> (p. 36):	User Training for BSL-2 Experiments at BioCARS
<b>Checklist 2</b> (p. 38):	Preparation of the Facility for a BSL-2 Experiment
<b>Checklist 3</b> (p. 41):	Shift Duties of the BioCARS Operating Staff
<b>Checklist 4</b> (p. 42):	Response by BioCARS staff on Duty to Failure of the Facility HVAC or Emergency Outside of the BSL-2 Facility
<b>Checklist 5</b> (p. 43):	Facility Cleanup after the BSL-2 Experiment

## **SOP 101: Responsibilities**

Date: July 2008

Purpose: Defines responsibilities of users, BioCARS staff and APS/ANL safety personnel for conducting safely BSL-2 experiments at the BioCARS facility.

For: BioCARS users and staff

### **1. Responsibilities of users and their home institution**

- 1.1** Identify the hazardous material and provide the MSDS and other relevant information related to the material.
- 1.2** Provide pertinent regulations used by the home institution, including evidence that each user has been authorized to work with the biohazardous agent at the user's home institution.
- 1.3** Provide certification of proper training and authorization for work with the BSL-2 agent at the home institution for all experimenters.
- 1.4** Identify transportation permit and shipping requirements and ensure proper shipping/transportation of the material to and from the BioCARS facility. Users are advised to contact the Hazardous Materials transportation specialists and Biosafety officer at their home institution regarding the compliance with transportation requirements. This includes USDA permits for interstate transport of infectious agents that affect agriculturally important animals and crops.
- 1.5** Comply with all regulations including this SOP and follow instructions given by BioCARS and APS/ANL staff.
- 1.6** Identify 2 users from the group as emergency contacts.
- 1.7** All users working with the BSL-2 agents must affirm that they are experienced and skilled in techniques and procedures used for work with such agents by providing an authorization by the home institution to work with the agent (see SOP 104). Any observation of improper handling of BSL-2 samples has to be reported to the spokesperson for the user group as well as to the BioCARS Biosafety Coordinator and it may result in denial of sample handling by the user in question based on the ANL stop work policy.

### **2. Responsibilities of the BioCARS Biosafety Coordinator**

- 2.1** Accept responsibility for an experiment identified as BSL-2.
- 2.2** Inform ANL IBC chairman, APS Biosafety Officer, APS User Safety Officer and APS floor Coordinator on duty. Floor coordinator will notify APS/ANL staff and prepare call-lists for incident response teams (**SOP 106, Appendix F**).

- 2.3 Obtain all relevant information from the PI of the user group and from user home institution. Initiate and coordinate the experiment approval process with the ANL IBC and the APS.
- 2.4 Gain sufficient knowledge about the nature of the hazard to make a judgment whether this SOP can be used to mitigate the hazard.
- 2.5 Send the BioCARS BSL-2 SOP to users.
- 2.6 Collect certifications of training/authorization from users and pass them to the ANL IBC.
- 2.7 Designate a particular BioCARS station for the BSL-2 experiment and supervise the appropriate set-up of the HVAC system if biocontainment mode is required (**SOP 106**).
- 2.8 Supervise the preparation of the facility for the BSL-2 experiment and return of the facility to normal operation (**SOP 106, SOP 112**). No VHP decontamination of the facility is required in case of an incident with BSL-2 samples.

### 3. Responsibilities of the BioCARS staff contact for the experiment

- 3.1 Coordinate communication with users regarding their preparation for the experiment after experiment is approved by the IBC.
- 3.2 Inform in advance any non-BSL-2 user groups approved to conduct experiments in the BioCARS facility during the BSL-2 experiment about the BSL-2 experiment and its associated hazards.
- 3.3 With users, IBC and APS representatives, participate in receiving user samples, inspecting of the packaging and logging of samples (**SOP 103**).
- 3.4 Ensure that all BioCARS staff who are on the list of authorized personnel for the access to the facility during the BSL-2 experiment have read and signed this SOP before the experiment (template signature sheet is at the end of the SOP, before Appendix A).
- 3.5 Provide BioCARS-specific BSL-2 training to all visiting scientists participating in the BSL-2 experiment (**SOP 104**). All visiting scientists have to sign the **Checklist 1** each time they visit.
- 3.6 Prepare the facility for the BSL-2 experiment (**SOP 106**) according to the **Checklist 2**.
- 3.7 Return the facility to regular operation (**SOP 112**) by using **Checklist 5**.
- 3.8 BioCARS staff contact is listed as an emergency contact. In case of personnel exposure to a BSL-2 agent, staff contact will supervise the response together with the user designated as an on-site emergency contact and the APS Floor Coordinator (**SOP 108**).

### 4. Responsibilities of the BioCARS operating staff

- 4.1 Ensure that the facility is working as designed and certified per **Checklist 3** daily (3 times per day, if 24h attendance is required).
- 4.2 If other, non-BSL-2 user groups are approved to conduct experiments in the BioCARS facility during the BSL-2 experiment, inform the users

about the nature of the BSL-2 biohazard, about entry and exit rules, and about emergency and evacuation procedures.

- 4.3 Participate in the response to overt or potential personnel exposure to BSL-2 agent (**SOP 108**) and in the response to the failure of facility HVAC and other equipment (**SOP 109**).
- 4.4 Evacuate the facility according to **Checklist 4** during an emergency (**SOP 110**).

## 5. Responsibilities of the APS Floor Coordinator on duty

- 5.1 Notify APS/ANL staff about the upcoming BSL-2 experiment. Prepare call-lists for appropriate incident response teams (**SOP 106, Appendix F**).
- 5.2 Participate in verification of the preparation of the facility for the BSL-2 experiment (**SOP 106**).
- 5.3 Participate in the response to overt or potential personnel exposure to BSL-2 agent (**SOP 108**) and in the response to the failure of facility HVAC and other equipment (**SOP 109**).
- 5.4 In case of emergency evacuate the facility according to **Checklist 4**.

## 6. Responsibilities of the BioCARS PI

- 6.1 Approve the BioCARS BSL-2 SOP.
- 6.2 Decide on Medical Surveillance Plan for BioCARS staff.
- 6.3 Decide on mandatory vaccination for BioCARS staff when vaccine is available.
- 6.4 Exclude BioCARS personnel from the BSL-2 area if medical requirements are not met.

## 7. Responsibilities of the ANL IBC

- 7.1 Approve the BioCARS BSL-2 SOP.
- 7.2 Approve a specific BSL-2 agent for use in the BioCARS facility based on information provided by users via the ANL IBC registration form and BioCARS BSL-2 questionnaire (in case of virus samples), as well as additional documentation provided by users.
- 7.3 Approve specific experimental procedures for work with a particular BSL-2 agent.

## 8. Responsibilities of the APS

- 8.1 Approve the BioCARS BSL-2 SOP.
- 8.2 Approve a specific BSL-2 agent for use in the BioCARS facility based on user response to the BSL-2 questionnaire and provided documentation.

## 9. Responsibilities of the CARS Safety Committee

Approve the BioCARS BSL-2 SOP.



## **SOP 102: Approval of BSL-2 Experiments**

Date: July 2008

Purpose: Establishes the process of approval for a particular BSL-2 experiment to be conducted at the BioCARS facility.

For: BioCARS users and staff

### **1. Application for beamtime**

- 1.1 Application for beamtime (proposal) is submitted via regular APS proposal submission route. Classification of the sample as BSL-2 has to be stated.
- 1.2 At least 2 months advanced notification to BioCARS/APS/ANL regarding the upcoming BSL-2 experiment is essential to assure proper preparation.
- 1.3 The BioCARS staff in charge of scheduling will make the initial contact with users. Basic information will be obtained regarding the sample, experiment and desired time for the experiment. This basic information is passed to the BioCARS Biosafety Coordinator.
- 1.4 The BioCARS Biosafety Coordinator informs APS Biosafety Officer about the upcoming BSL-2 experiment. APS Biosafety Officer informs all necessary APS/ANL personnel.

### **2. Approval process**

- 2.1 BSL-2 SOP (which includes a BioCARS Questionnaire in the Appendix C) and ANL Registration Form For Pathogens, Cells, Tissues and OPIM are sent to users by the BioCARS Biosafety Coordinator. Both the BioCARS Questionnaire (for virus samples) and ANL Registration Form must be filled out as soon as possible.
- 2.2 Each user is required to be trained and authorized for work with the BSL-2 agent by the IBC at their home institution.
- 2.3 Each user needs to sign the Risk Factors Form (Appendix D).
- 2.4 Based on sample and authorization questionnaires, the ANL IBC either requests additional information or approves the experiment and makes decisions regarding:
  - a) authorized personnel
  - b) PPE for the experiment
  - c) need for 24h attendance and use of HVAC in biocontainment mode

## **SOP 103: Shipping and Receiving of BSL-2 Samples**

Date: May 2007

Purpose: Summarizes ANL, APS and BioCARS acceptable procedures for shipping BSL-2 user samples from home institution to BioCARS, for receiving samples at BioCARS, for sample logging in/out, and, if necessary, for shipping samples back from BioCARS to user home institution.

For: BioCARS users and staff

### **1. Shipping user BSL-2 samples from home institution to BioCARS**

- 1.1** Users are responsible for determining if possession and transportation permits are required for the specific BSL-2 agent. Users are responsible for obtaining the required permits.
- 1.2** The home institution must provide to BioCARS/ANL approved and university-signed transfer permit if required at least ten business days prior to delivery of the material to BioCARS.
- 1.3** For all purposes, including obtaining appropriate permits when necessary, the users are considered in possession of the BSL-2 samples throughout the transportation and during the BSL-2 experiments at BioCARS.
- 1.4** Users shall be listed as **both the shippers and receivers** of the samples. For both permit application (when necessary) and shipping of the samples, the following address shall be used for **the receiver** when shipping samples from home institution to BioCARS facility:

User Name  
Building 434B, Sector 14  
9700 South Cass Avenue  
Argonne, IL 60439

- 1.5** Identification of the user home institution as both the shipper and receiver is acceptable under the following conditions: (a) the home institution identifies the BioCARS facility at the APS/ANL as a new/additional location for research on any applicable permits, (b) the home institution is the sole user of the material while it is on the ANL site, (c) the home institution is legally responsible for control and possession of the material while it is at the ANL site, and (d) the home institution initiates and is legally responsible for transport of the material in accordance with all applicable regulations and requirements.
- 1.6** Since users are shippers, they are directly responsible for the correct and compliant transport by air or ground both (a) from the home institution to BioCARS facility and (b) from BioCARS facility to home institution.
- 1.7** If users are transporting the BSL-2 samples themselves, they must provide an estimated time of arrival and the names of the person(s) who will be the

custodian of the package. Upon entering the ANL, users have to drive directly to the BioCARS facility (no intermediate stops on the ANL campus).

- 1.8 Upon arrival of the user's package at the BioCARS facility and before the package is transferred from the vehicle that delivered it, the APS and BioCARS representatives, together with users, will visually inspect the exterior of the packages that contain infectious material. If there is any concern about the integrity of the package, package will be left in the vehicle and 911 will be dialed.
- 1.9 The primary purpose of the inspection is to assure that the package has no readily visible cause for concern (e.g., damaged, partially open, leaking, etc.) The inspection is NOT intended to verify compliance with federal regulations with regard to documentation, external labeling, and internal packaging. The users are solely responsible for conformance with applicable federal regulations. The user representative must be prepared to provide the following to the APS representative:
  - a) an MSDS or equivalent
  - b) letter from the institution's hazardous materials transportation specialist that assures packaging conforms with applicable state and federal requirements and was performed or supervised by a qualified person
  - c) copies of any transportation permits required by the USDA or other agencies
  - d) copy of the shipper's declaration

## 2. Receiving user BSL-2 samples

- 2.1 Prior to opening, the package with the BSL-2 samples is locked inside the BSC room for storage. Emergency access to the BioCARS facility key is available through the APS Floor Coordinator at LOM 434 (the second facility key is kept by the BioCARS Biosafety Coordinator for the duration of the experiment).
- 2.2 While the material is at BioCARS, on the ANL site, the material remains property of the user home institution.
- 2.3 The package will be opened in the BSC and inspected by users in the presence of the BioCARS staff contact. If there are any concerns about the integrity of the samples (broken capillaries, for example), leave the containment area and inform the BioCARS Biosafety Coordinator and the APS Floor Coordinator. Incident response team will be consulted regarding the further action (**Appendix F**).
- 2.4 A log-in/log-out process is established for all infectious materials that users receive at BioCARS. The log-in information includes the number of containers/wells/crystals. The log-in/log-out form is in the **Appendix E**. BioCARS staff contact for the experiment makes sure that the log-in form is filled out by users and the form included into the BSL-2 Procedure Logbook.
- 2.5 Unpacked samples must be stored in the Biosafety Cabinet.

**3. Shipping user BSL-2 samples from BioCARS to home institution**

- 3.1** As a part of the log-out process, BioCARS staff contact makes sure that users fill out the Log-out form (**Appendix E**) and certify that all BSL-2 material has been rendered non-viable or has been packaged for transport/shipping to home institution.
- 3.2** For the BSL-2 samples where a special permit requires that the owner remains in possession of the material at all times, the users will arrange for transport of the BSL-2 samples back to the home institution. In this case, the user-provided shipping papers will identify the users as both the shipper and receiver. The following address shall be used for the **shipper** when shipping such samples from BioCARS facility to home institution:

User Name  
Building 434B, Sector 14  
9700 South Cass Avenue  
Argonne, IL 60439

- 3.3** For all other BSL-2 samples, the shipping of samples from BioCARS to the home institution has to be done via ANL/HazMat Shipping/Receiving. ANL in this case is a shipper and, based on information from users regarding the samples, assures compliance with state and federal regulations for marking, package certification and labeling, and shipping paper documentation,

## **SOP 104: User Training and Certification**

Date: June 2005

Purpose: Establishes training requirements and procedures for BSL-2 users of BioCARS facility.

For: BioCARS users and staff

### **1. Training**

- 1.1** BioCARS staff members that are given access to the BSL-2 area during a BSL-2 experiment will be trained at the ANL on basic practices of work with BSL-2 agents (although they will not be involved in direct work with the BSL-2 agent).
- 1.2** BioCARS staff members that are given access to the BSL-2 area during a BSL-2 experiment will have walk-through training by the BioCARS Biosafety Coordinator before the BSL-2 experiment.
- 1.3** All users involved in the BSL-2 experiment at BioCARS must be authorized by the IBC at their home institution for work with the specified BSL-2 agents (**SOP 102**).
- 1.4** All users working with the BSL-2 agents must affirm that they are experienced and skilled in techniques and procedures used for work with such agents, including the use of Biosafety Cabinets. Any observation of improper handling of BSL-2 samples must be reported to the spokesperson for the user group as well as to the BioCARS Biosafety Coordinator. This may result in denial of sample handling by the user in question based on the ANL stop work policy.
- 1.5** This SOP is sent to users well ahead of the start of their experiment.
- 1.6** All users are also given a copy of this SOP by their BioCARS staff contact upon arrival. They are instructed to read it carefully to refresh their knowledge about the facility and procedures. The users are given the opportunity to ask any questions and discuss any issues related the SOP material.
- 1.7** The BioCARS staff contact will tour the facility with users and identify the location of the equipment, PPE and other safety items.
- 1.8** The BioCARS staff contact will also go through **Checklist 1** with users and explain:
  - a) gowning/de-gowning protocols
  - b) procedure for transferring samples to the experimental station
  - c) [when use of face mask and safety glasses are required](#)
  - d) proper waste disposal
  - e) response to autoinoculation
  - f) response to alarms due to HVAC problems
  - g) response to other emergencies

## 2. Certification

At the end of the training, each user will sign the **Checklist 1** to indicate that they have received the facility-specific training for handling the BSL-2 materials at BioCARS.

## **SOP 105: Protective Clothing and Other Personal Protective Equipment (PPE)**

Date: July 2008

Purpose: Establish protective clothing and respirator needs and procedures during BSL-2 experiments

For: BioCARS users and staff

### **1. Users**

- 1.1. Users will wear disposable lab coats while in the BSL-2 containment area. The lab coats must be worn closed and at all times while in the containment area. Users have to remove lab coats aseptically whenever exiting the containment area.
- 1.2. Upon first use, lab coats will be marked with person's name. When leaving the containment area the lab coats will be taken off in the BioCARS corridor (see BioCARS Facility Layout in Appendix A) and will be hung on hooks in the corridor to be re-used upon return. If, however, the lab coats are suspected to be contaminated during the experiment because of a known incident/event, they must be disposed promptly.
- 1.3. Sequence for taking off gloves and lab coats before leaving the facility: remove outer gloves aseptically, remove the lab coat, remove the inner gloves; use hand sanitizer before leaving the containment area.
- 1.4. Used lab coats will be disposed in the biohazard waste bags in the BioCARS corridor (see BioCARS Facility Layout in Appendix A) upon completion of the experiment.
- 1.5. Disposable gloves must be worn while handling the BSL-2 samples. When sharps are involved, two pairs of gloves will be worn. Wearing nitrile gloves as a second pair of gloves over powderless latex gloves is recommended for ease of use. Gloves must be worn over the sleeves of the lab coats/overalls. Jewelry (rings, bracelets) and watches will be removed during the BSL-2 experiments as they affect the integrity of the gloves. Users must check the integrity of gloves before use (e.g. by visual inspection and inflating with air).
- 1.6. Gloves have to be aseptically removed (with a special care not to contaminate the inner gloves) after any manipulation of BSL-2 material.
- 1.7. Safety glasses with side shields will be worn while working with samples outside of the biosafety cabinet. In addition, NIOSH approved face masks will be worn if samples are not contained.
- 1.8. When not in use, safety glasses and face masks will be left in the control room or in the corridor, labeled with user names.
- 1.9. Safety glasses and face masks will be disposed in the biohazard waste bags upon completion of the experiment.

- 1.10. Lab coats used in the biocontainment area must not be taken outside of the containment area, except when properly packed for disposal at the end of an experiment.
- 1.11. Contact lenses may be worn in the biocontainment area only if safety glasses are worn.

## 2. BioCARS staff

- 2.1. While helping users with the BioCARS equipment during the BSL-2 experiments, BioCARS staff must wear lab coats and one pair of gloves.
- 2.2. Contact lenses are allowed.
- 2.3. If present in the containment area during an activity that requires use of face masks by users, BioCARS staff must also wear NIOSH approved face masks.
- 2.4. BioCARS staff shall call users before entering the facility to inform themselves about user activities which will determine what PPE staff needs to wear.
- 2.5. Protective clothing will remain within containment area until the conclusion of the experiment.
- 2.6. Gloves will be removed aseptically.

## 3. Visitors and observers

No visitors and observers are allowed in the facility during a BSL-2 experiment.

## 4. Location of protective clothing and PPE

Supply of clean lab coats, gloves, eye wear and NIOSH approved face masks is located in the computer room.



## **SOP 106: Preparation of the Facility for BSL-2 Operation**

Date: May 2007

Purpose: Establishes procedures for changing the operation mode from standard to BSL-2 mode at the BioCARS facility and for preparation of the designated area for the BSL-2 work (**Checklist 2**). Both are responsibility of the BioCARS staff contact for the BSL-2 experiment, supervised by the Biosafety Coordinator (**SOP 101**) and observed by the APS Floor Coordinator.

For: BioCARS staff and APS Floor Coordinator

1. Inform APS Biosafety Officer (who will inform all other necessary APS/ANL personnel)
2. Designate members of incident response teams with assistance from the APS and ANL representatives (**Appendix F**) for:
  - a) medical and other emergencies
  - b) failure of HVAC or other equipment (**SOP 109**)
  - c) overt or potential personnel exposure to biohazard (**SOP 108**)

A list of incident response team members will be prepared by the APS Floor Coordinator, who will also inform the incident response teams of the BSL-2 experiment. Specific aspects of incident response will be discussed by the appropriate team.

### **3. Prepare HVAC system and BSC**

For experiments that require BSL-3 containment, arrange with FC the check of the HVAC system operation by FSM, per instruction provided by APS.

BioCARS staff contact (trained and supervised by the BioCARS Biosafety Coordinator) and the APS Floor Coordinator verify the proper operation of the HVAC and BSC: follow **Checklist 0**.

### **4. Prepare facility**

BioCARS staff will follow **Checklist 2** to prepare the facility for the BSL-2 experiment.

## **SOP 107: BSL-2 Practices and Procedures**

Date: July 2008

Purpose: Establish protocols for sample handling.

For: BioCARS users and staff

### **1. General**

- 1.1** 24h attendance is not required for routine BSL-2 experiments but the BioCARS Biosafety Coordinator and the user spokesperson will be on-call in case of an incident or emergency. Depending on the nature of the specific BSL-2 agent, the ANL IBC may require 24h attendance (**SOP 107**). In that case, one user and a BioCARS staff member would be required to remain at all times in the BioCARS area (containment area, computer room, kitchen area or BioCARS LOM). The user may get a permission from the BioCARS staff on duty to go to the APS Guest House and have rest under two conditions:
  - a) sample is in the hutch and an extended data collection has been set-up (several hours)
  - b) user is reachable at all times.
- 1.2** Store properly packed BSL-2 samples in the BSC room, next to the BSC. A dewar with frozen, contained BSL-2 samples can also be stored in the control room during the active experiment for the ease of transferring of frozen samples to the experimental hutch.
- 1.3** Store all unpacked samples in the Biosafety Cabinet for the duration of the experiment. The BioSafety cabinet is lined with the plastic-backed absorbent paper without obstructing the airflow in the cabinet.
- 1.4** For room temperature experiments, pre-mount crystals in quartz capillaries at the home institution whenever possible.
- 1.5** Use stainless steel dewars for work with frozen crystals whenever possible.
- 1.6** Safe microbiological practices must be applied when handling BSL-2 materials, as described in the CDC-NIH Biosafety in Microbiological and Biomedical Laboratories (a copy of the document is in the computer room) and in the **Checklist 1**.
- 1.7** **Biosafety Cabinet Work Practices and Procedures** must be followed (described in the **Appendix B**).
- 1.8** Protective clothing and other protective equipment must be worn as described in the **SOP 105**.
- 1.9** Gloves must be safely changed after any manipulation of the BSL-2 material, overt or suspected contact with the BSL-2 material or contact with contaminated items. To minimize the potential contamination of surfaces and equipment, do not touch any surfaces, door handles or the equipment with potentially contaminated gloves.
- 1.10** Replacing outer gloves (**when two pairs of gloves are required**):

- a) Take off the outer gloves aseptically (taking special care not to contaminate the inner gloves).
  - b) Dispose used outer gloves in a biohazard waste container.
  - c) Put on a clean pair of outer gloves.
- 1.11** Wash hands (in the BSC room) or use clinical grade hand sanitizer whenever gloves are changed or removed. Wash hands at the earliest opportunity after leaving the biocontainment area.
- 1.12** Do not remove from the Biosafety Cabinet items that are potentially contaminated until they have been surface decontaminated.
- 1.13** When exiting the containment area from the corridor to the computer room, do not open the door with gloved hands. Dispose gloves first and use the gel-based disinfectant (by the door) as a final hand cleaning step.

## **2. Sample handling:**

- 2.1** Room temperature crystals:
- 2.1.1** Crystal mounting must be done in the BSC.
  - 2.1.2** Use only quartz capillaries.
  - 2.1.3** No mouth pipetting is allowed.
  - 2.1.4** Use epoxy to seal the capillaries.
  - 2.1.5** Collect all waste from the crystal mounting process in the biohazard bags or biohazard sharp containers in the Biosafety Cabinet.
  - 2.1.6** Disinfect the capillary exterior. Use extreme caution not to break the capillary.
  - 2.1.7** Mount a capillary on a brass pin (use epoxy to glue it to the pin). Insert brass pin into a pin holder (Hampton Adjustable Crystal Mount).
  - 2.1.8** Prepare a capillary for transfer from the BSC to the diffractometer:
    - a) use the secondary container provided by BioCARS
    - b) place the pin holder with the capillary on the magnetic base in the secondary container
    - c) screw the cover of the secondary container over the capillary
  - 2.1.9** Disinfect the secondary container before taking it out of the Biosafety cabinet.
- 2.2** Pre-frozen crystals:
- 2.2.1** Prepare pre-frozen crystal for transport to the hutch (see 2.2.3 for alternative protocol):
    - a) place a small transfer dewar filled with liquid nitrogen in the BSC
    - b) pull out one cane from the user dewar; take out (using Hampton Vial Clamp) one vial with a crystal from the cane; put the vial into the transfer dewar in the BSC.
    - c) Hampton Cryotong can be used (instead of the vial) to enclose the crystal for transfer to the hutch.

**2.2.2** Transfer the small dewar with the vial or Cryotong to the hutch by hand or using a small BioCARS provided cart. Make sure all preparations for sample mounting in the hutch are done (see **4** below) before transferring the small dewar.

**2.2.3** To reduce the risks associated with the transfer of frozen crystals from the BSC room to the hutch, **2.2.1** can be done in the control room. The user dewar with frozen, contained crystals will be stored in that case in the control room (only during the active experiment period).

**2.3** Freezing crystals:

**2.3.1** Preparation will be done in the BSC.

**2.3.2** Using standard cryo-tools (Such as Hampton CrystalCap with CryoLoop, CrystalWand, VialClamp) a crystal is flash frozen by plunging it into liquid nitrogen in a short dewar. Place frozen crystal either into a vial or a Hampton Cryotong.

**2.3.3** Transport the dewar with the vial or Cryotong to the hutch by hand or using a small BioCARS provided cart. Make sure all preparations for sample mounting in the hutch are done – see **4** below – before transporting the dewar.

**2.4** Safety glasses must be worn while manipulating samples outside of the BSC. NIOSH approved face masks must also be worn when working with uncontained samples. Double gloves are required when working with sharps.

### **3. Handling spills in the Biosafety Cabinet**

**3.1** A loss of crystal in the Biosafety Cabinet is not regarded as an incident and is handled as a spill.

**3.2** Small spills within the Biosafety Cabinet are handled immediately by disinfecting the affected area, folding and removing the contaminated absorbent paper toweling and placing it into a biohazard bag. Make sure to minimize the aerosol formation while removing the paper. Any splatter onto items within the cabinet, as well as the cabinet interior, shall be wiped immediately with a towel dampened with decontaminating solution.

### **4. Transferring crystal from the Biosafety Cabinet room to the diffractometer:**

**4.1** Prepare diffractometer for sample mounting before the sample transfer:

a) move the beamstop and detector away to make enough space for maneuvers needed to place the capillary or frozen crystal on the magnetic base on the diffractometer

b) position the kappa angle of the diffractometer into appropriate position for crystal mounting

- c) place and secure a tray with absorbent paper under the sample position and soak the paper with disinfectant
- d) use trial loop without a crystal or trial capillary to adjust the goniometer z translation properly

- 4.2** Only a single crystal at the time is transferred from the Biosafety Cabinet or the control room to the diffractometer.
- 4.3** Crystal transfer is conducted under a two-person rule where one person transports the sample and another assists (opens doors, warns other users and prevents collisions and tripping, monitors sample mounting on the diffractometer).
- 4.4** Safety glasses, gloves and a lab coat are all required to be worn while handling and transferring BSL-2 crystals. NIOSH approved face masks must also be worn when working with uncontained samples outside of the BSC. Double gloves are required when working with sharps.

## **5. Mounting a crystal on the diffractometer:**

- 5.1** Make sure diffractometer is prepared as outlined above in **4.1**.
- 5.2** Mount a crystal on the diffractometer:
  - a) When the crystal is in a screw-cap vial, loosen the magnetic cap before mounting the vial. Mount the vial on the magnet and remove the vial.
  - b) Mount magnetic cap vials or capillary-mounted crystals on the diffractometer magnet.
- 5.3** Examine immediately on the monitor in the hutch the magnified image of the loop/capillary with the crystal. Make sure capillary is intact in case of room temperature experiment. Make sure the loop contains the crystal when frozen samples are used. In case of lost crystals, follow the procedure described in **SOP 108**.

## **6. After crystal mounting:**

- 6.1** Before touching any equipment, properly remove gloves and replace with clean gloves.
- 6.2** Remove the crystal transport tools and transport dewar from the hutch and return them to the BSC. Replace the gloves again.
- 6.3** Finish preparations inside the hutch (remove the tray, position the beam stop and the detector), search the hutch and close the hutch door. The rest of preparation that does not require access to the hutch shall be done with the hutch door closed.

## **7. Removing a crystal from the diffractometer:**

- 7.1** Confirm that crystal (loop or capillary) is not lost before entering the hutch, by checking the crystal-viewing monitor in the control room.
- 7.2** With the same PPE used for crystal mounting, enter the hutch and move back the detector and the beam stop.

- 7.3** Remove the crystal and submerge it into a non-breakable jar containing disinfectant whenever possible. **If necessary for scientific reasons, crystals can be saved for a later re-use. Return the dewar with the saved frozen crystal (enclosed by a vial or a Cryotong) or the capillary container with the saved crystal to the BSC room.**
- 7.4** Properly remove gloves and replace with clean gloves.

## **SOP 108: Response to Overt or Potential Personnel Exposure to BSL-2 Agent**

Date: July 2008

Purpose: Establish procedures for a response to: (a) loss of crystal; (b) autoinoculation (cuts/sticks with sharps, splashes to mucous membranes). See also summary in the Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS**. VHP decontamination of the facility is not required if a BSL-2 crystal is lost.

For: BioCARS users, staff and APS Floor Coordinator

### **1. Loss of crystal in the experimental station**

- 1.1** All PPE must be kept on.
- 1.2** Immediately soak the area of the spill with disinfectant.
- 1.3** Take off the gloves (dispose in a biohazard bag).
- 1.4** Exit the hutch and CLOSE the hutch door. Put on a clean pair of gloves. Wait for 60min before returning to the hutch.
- 1.5** Inform the BioCARS staff on duty. BioCARS staff will inform BioCARS Biosafety Coordinator and APS Floor Coordinator.
- 1.6** BioCARS staff will work together with the user to:
  - i. Dispose of the old absorbent paper and replace it with new absorbent paper if the crystal landed on the absorbent paper. Fold the contaminated paper carefully so that the potentially contaminated surface remains inside (do not crumple or wad the paper), place the paper in a biohazard bag and then place this bag in the large biohazard bag by the BSC.
  - ii. If crystal landed outside of the area covered by the absorbent paper, conduct another round of disinfection of the affected area.
- 1.7** Post a "Do not enter" sign at the entrance door of the containment area.
- 1.8** BioCARS Biosafety Coordinator will discuss the event with the APS Biosafety officer to decide if and under what conditions experiment can be continued.

### **2. Loss of crystal outside of the experimental station**

- 2.1** All PPE must be kept on.
- 2.2** Immediately cover the area of the spill with paper, and apply disinfectant solution to the paper, beginning at the periphery and then to the center. The disinfectant-soaked paper shall be left undisturbed for at least 30 minutes (exact time depends on the susceptibility of the BSL-2 agent to the disinfectant).

- 2.3 Take off the gloves and the lab coat (dispose in a biohazard bag).
- 2.4 Exit the area for 60min, leaving the hutch door OPEN.
- 2.5 Inform the BioCARS staff on duty. BioCARS staff will inform BioCARS Biosafety Coordinator and APS Floor Coordinator.
- 2.6 BioCARS staff will work together with the user to:
  - iii. Dispose of the old absorbent paper and replace it with new absorbent paper if the crystal landed on the absorbent paper. Fold the contaminated paper carefully so that the potentially contaminated surface remains inside (do not crumple or wad the paper), place the paper in a biohazard bag and then place this bag in the large biohazard bag by the BSC.
  - iv. If crystal landed outside of the area covered by the absorbent paper, conduct another round of disinfection of the affected area.
- 2.7 Post a “Do not enter” sign at the entrance door of the containment area.
- 2.8 BioCARS Biosafety Coordinator will discuss the event with the APS Biosafety officer to decide if and under what conditions experiment can be continued.

**3. Autoinoculation (cuts and sticks by sharps, splashes to mucous membranes)**

- 3.1 Accompanying person or BioCARS staff dials 911.
- 3.2 Remove contaminated gloves and squeeze vigorously if possible to allow the wound to bleed.
- 3.3 Wash the wound with soap and water for 5min and apply sterile bandage if necessary (stored in the BSC room).
- 3.4 For splashes to mucous membrane, rinse with large amounts of water at the sink in the BSC room. Eyes shall be irrigated for at least 5min using the eye wash station in the BSC room.
- 3.5 Take off protective clothing and dispose it.
- 3.6 Exit the BSL-2 area.
- 3.7 BioCARS staff on duty informs BioCARS Biosafety Coordinator and APS Floor Coordinator.



## **SOP 109: Response to the Failure of Facility HVAC and Other Equipment**

Date: July 2008

Purpose: Establish procedures in case of HVAC failure and possible loss of containment and failure of other critical equipment (such as BSC and crystal cryo-cooler).  
See also summary in the Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS**

For: BioCARS users, staff and APS Floor Coordinator

- 1. Loss of containment due to failure of the main facility exhaust fan EF104**
  - 1.1** An alarm will sound and BioCARS PA notification system will be activated (**announcement: “Attention BioCARS staff, HVAC problem”**). Pressure differential monitors will sound a local alarm. BSC alarm will sound to indicate loss of negative pressure above the BSC and BSC will turn off.
  - 1.2** Stop all work, but keep all PPE on.
  - 1.3** If working with open samples, place all open samples at hand into primary and secondary containers or dispose them into a closest sample waste container with disinfectant.
  - 1.4** Take off and dispose gloves and protective clothing. Wash or disinfect hands.
  - 1.5** If wearing face mask/safety glasses, take these off just before leaving the containment area.
  - 1.6** Exit the containment area and inform the BioCARS staff on duty about the incident.
  - 1.7** BioCARS staff on duty will follow steps listed in **Checklist 4**.
  - 1.8** A conference will be convened by BioCARS staff contact, user emergency contact, BioCARS Biosafety Coordinator and APS Floor Coordinator. A response procedure will be developed.
  
- 2. BSC failure: Improper down flow velocity**
  - 2.1** A local BSC alarm will sound. The exhaust from the BSC is maintained as long as the EF104 is operating.
  - 2.2** If working in the BSC, follow **1.2** to **1.6**.
  - 2.3** If anywhere else in the facility, exit the BSL-2 area.
  - 2.4** Inform the BioCARS staff on duty about the incident.
  - 2.5** BioCARS staff: follow **1.7-1.8**.

**3. Failure of the crystal cryo-cooler**

- 3.1** Failure of the crystal cryo-cooler and loss of the cold gas stream will cause warming up and drying out of the crystal that is mounted on the diffractometer. This will be evident by looking at the crystal-viewing monitor and checking the sample temperature (monitored outside of the hutch). If this occurs during the data collection, stop the data collection. Do not open the hutch door.
- 3.2** If this occurs in the process of mounting a crystal on the diffractometer, follow **1.2** to **1.6**.
- 3.3** BioCARS staff: follow **1.7-1.8**.

## **SOP 110: Medical and Other Emergencies During the BSL-2 Experiments**

Date: May 2007

Purpose: Establish safe procedures for Medical and Facility Emergencies during the BSL-2 experiments.  
See also summary in Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS.**

For: BioCARS users, staff, APS Floor Coordinator and APS/ANL emergency response team.

### **1. Medical Emergencies not related to biohazard exposure**

- 1.1. BioCARS staff or user dials 911 and informs the APS Floor Coordinator.
- 1.2. Follow instructions by the 911 personnel and wait for the medical emergency personnel to arrive.

### **2. Fire emergency**

- 2.1 Leave the facility.
- 2.2 Call 911 and contact APS Floor Coordinator.
- 2.3 Gather outside of the 434B LOM, at the parking lot.
- 2.4 Wait for the instructions from the APS Floor Coordinator before returning to the facility.

### **3. Other emergencies outside of the BSL-2 facility (e.g. Severe Weather Alert)**

- 3.1 Stop all work.
- 3.2 Place all open samples at hand into primary and secondary containers or dispose them into a closest sample waste container with disinfectant.
- 3.3 Take off gloves and wash or disinfect hands with hand sanitizer.
- 3.4 Take off and dispose protective clothing in the BSC room. Wash hands.
- 3.5 Users and BioCARS staff on duty will collect and double bag all biohazard waste and place the waste in the BSC room.
- 3.6 Leave the containment area.
- 3.7 BioCARS staff will lock BSC room. BioCARS staff will follow steps listed in **Checklist 4.**
- 3.8 **Steps 3.1 to 3.7 shall normally be completed. However, protection of human life and health has priority over full assurance that personal decontamination and removal of protective clothing has been thoroughly completed.**

## **SOP 111: BSL-2 Waste Disposal**

Date: July 2008

Purpose: Establish procedures for safe BSL-2 waste disposal.

For: BioCARS users, staff and APS Floor Coordinator

### **1. BSL-2 waste pickup by ANL Waste Management**

- 1.1** Disposable contaminated materials are collected in biohazard bags, which are collected at the end of the experiment, sealed and placed in the BSC room.
- 1.2** The bags are surface decontaminated and placed in secondary bags.
- 1.3** Double-bagged waste is placed into shipping boxes provided by ANL Waste Management.
- 1.4** BioCARS staff contact fills out the Waste Management form and schedules the pick-up. Advanced notification (two weeks prior to the experiment) of the ANL Waste Management regarding the approximate pick-up time is needed to assure timely waste pickup.
- 1.5** ANL Waste Management will pick up the shipping boxes with the doubly-bagged waste as soon as possible after the BSL-2 experiment, after the routine decontamination of the facility (**Checklist 5**, part 1).

### **2. Autoclaving**

- 2.1** A portable autoclave is available for autoclaving the non-disposable tools and materials.
- 2.2** The autoclave is located in the biosafety cabinet room.
- 2.3** Trained BioCARS staff will conduct autoclaving. Instructions for autoclaving will be posted by the autoclave. Autoclaving for 60min at 121-132°C is required.
- 2.4** BioCARS staff verifies the effectiveness of the autoclaving monthly (verification records are kept in the Maintenance Logbook).

**SOP 112: End of Experiment Procedures**

Date: March 2004

Purpose: Return the facility to standard (no biocontainment) operation.

For: BioCARS users and staff

1. Follow **Checklist 5**.

## **SOP 113: Safety equipment maintenance and testing**

Date: July 2008

Purpose: Establish procedures for safety equipment maintenance, testing and certification..

For: BioCARS staff

**Important:** Record all maintenance in the BioCARS BSL-2/3 Maintenance Logbook.

### **1. Five BioCARS AHUs located on the facility roof**

*Before each APS run (three times per year):*

- 1.1 Check AHUs for excessive vibrations or any other obvious problems.
- 1.2 Check the integrity of belts and replace if necessary.
- 1.3 Check chilled water pipes and water mixing valves for leakage.

*Every six months:*

- 1.4 Check **On** and **Off** indicator lights on the electrical box (mounted on the AHU) and replace any burnt-out bulbs.
- 1.5 Grease the two ball bearings for the fan shaft (Mobil Oil Corporation, Mobilith AW2 temp range -29°C to 163°C)
- 1.6 Replace fan drive belt (Part # Gates Truflex 5L530 rated for equipment of 3 to 17 HP use). Make sure that the belt is not adjusted too tightly and that the upper pulley is aligned with the lower pulley. (A slight squeak on the start up of an AHU is normal.)
- 1.7 Replace air filters, two for each AHU (McMaster-Carr Part # 2211K65 Antimicrobial Pleated Panel Filter). Pay strict attention to the air flow direction as air filters are directional.
- 1.8 On completion of the maintenance tasks, reinstall the fan belt and pulley covers.
- 1.9 Clean externally (vacuum up any belt dust etc.)

### **2. Differential pressure monitors in BioCARS corridor and BSC room**

- 2.1 Check pressure differentials before each BSL-2/3 experiment and compare to the list of values in the “BioCARS BSL-2/3 Maintenance Logbook”. Report any significant discrepancies to the BioCARS Biosafety Coordinator.

- 2.2 Do **NOT** adjust any switches or adjustments screws on the units before talking to the BioCARS Biosafety coordinator. The status switch shall be on “-“ for all units but computer room unit, which shall be “+”.
- 2.3 Do not attempt to re-calibrate the units. If you suspect the calibration problem (as evidenced by comparing the unit reading with the results of annual pressure differential measurements), send the unit to the manufacturer for re-calibration.

### 3. **Biohazard alarms**

Biohazard alarms have to be tested before each BSL-2/3 experiment (appropriated pressure monitors have to be on).

### 4. **Biosafety Cabinet**

Arrange for annual re-certification of the Biosafety Cabinet with Salus ([www.salustech.net](http://www.salustech.net)).

### 5. **HEPA filter and pre-filter**

- 5.1 Annual testing is routinely provided by the ANL IH personnel.
- 5.2 Before each BSL-2/3 experiment check the pressure differential for the pre-filter.
- 5.3 Request that ANL IH change the filter if necessary (clean filter pressure differential < 0.2” H<sub>2</sub>O, alarm goes off at 0.8” but this does not affect any hardware – just a reminder that pre-filter is getting clogged).

### 6. **PAPRs respirators**

- 6.1 Every month recharge batteries for all units.
- 6.2 Before each BSL-2/3 experiment run all PAPR units for 8h and recharge batteries.
- 6.3 Test for proper operation (instructions are in the BioCARS BSL-2/3 Maintenance Logbook).

### 7. **Autoclave**

- 7.1 BioCARS staff verifies the effectiveness of the autoclaving monthly (verification records are kept in the Maintenance Logbook).

**Checklist 0: Preparation of the HVAC and BSC for BSL-2 operation**

**(BioCARS staff contact and APS Floor Coordinator)**

- Check the status of the AHUs (check the integrity of belts and replace if necessary).

**Is biocontainment mode of the air handling system required by ANL IBC:**

- Yes  
 No

**If biocontainment mode is required, assure the biocontainment of the designated area:**

- Arrange with FC the check of the HVAC system operation by FSM, per instructions provided by APS.
- seal the doors to the non-BSL-2/3 control areas on both sides;  
 arrange for an ANL safety specialist (or APS personnel certified by the ANL safety specialist) to perform documented leak check (smoke test) across the sealed doors and for verification of the directional flows in the biocontainment area and across all doors at the perimeter of the facility.

**If biocontainment mode is required, in the presence of an APS floor coordinator:**

- Switch the HVAC mode from standard to biocontainment mode using the HVAC JC-Control Box (located on the BioCARS facility roof).
- Selects the proper experimental station for the BSL-2/3 experiment using the HVAC JC-Control Box.
- Make sure the door to the BioCARS facility roof (where HVAC JC-Control Box is located) is locked.

Verify proper HVAC operation for the biocontainment mode using the EPICS Status Screen and other indicators:

- EF104 running (status ON)
- The hutch door switch (modifies the exhaust flow rate from the hutch) and dampers of the AHU for the BSL-2/3 control room operate properly:
- a) Hutch door open (door switch deactivated):  
 AHU make up air damper fully open (no air recirculation in the control room); exhaust rate from the BSL-2/3 hutch is in excess of 950cfm
  - b) Hutch door closed (door switch activated):



AHU make up air damper fully closed (air recirculation is on in the control room); exhaust rate from the BSL-2/3 hutch is <400cfm

**Verify proper operation of BSC (if used during the experiment):**

- Verify that the Biosafety cabinet is on and operating properly: display on the BSC showing down flow rate ~ 60 fpm, exhaust rate ~730cfm.

**If biocontainment mode is required: Verify operation of pressure monitors**

- Pressure differential monitors in the corridor are turned on for the BSL-2/3 control room, computer room and the BSC room (otherwise the Biohazard Alarm buttons in the control rooms/BSC room will not work).
- Pressure monitors for non-BSL-2/3 control room are off (rooms are sealed so pressure differentials are 0; alarms will sound continuously if turned ON; DO NOT adjust alarm set points since any station may be used in the future for a BSL-2/3 experiment).

Signature of the BioCARS staff contact for the experiment and APS Floor Coordinator:

BioCARS staff: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

APS Floor Coordinator: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**Checklist 1: User Training for BSL-2 Experiments at BioCARS**

**Material :** \_\_\_\_\_

**Disinfectant:** \_\_\_\_\_

Does the material pose a Human Health Hazard (Yes / No)

Only Authorized Personnel allowed in the BSL-2 area (designated hutch, adjacent control room, corridor and Biosafety Cabinet room).

24h attendance is not required for routine BSL-2 experiments but the BioCARS Biosafety Coordinator and the user spokesperson will be on-call in case of an incident or emergency. The ANL IBC may require 24h attendance depending on the nature of the specific BSL-2 agent (**SOP 107**). In that case, one user and a BioCARS staff member will be required to remain at all times in the BioCARS area (containment area, computer room, kitchen area or BioCARS LOM). The user may get permission from the BioCARS staff on duty to go to the APS Guest House and have rest under two conditions:

- a) sample is in the hutch and an extended data collection has been set-up (several hours)
- b) user is reachable at all times.

Sample storage and manipulation has to be done in the BSC room. In case of frozen crystals, a dewar with contained, frozen crystals can be stored in the control room during the experiment for easier transfer of a crystal to the hutch.

Two-person rule is in effect for sample transfer from the BSC room to the diffractometer.

Absolutely no food or drinks are allowed. No application of cosmetics while in the biocontainment area.

Collect all waste in biohazard bags and all sharps in biohazard sharps containers.

Collect “dead” crystals in disinfectant (non-breakable) jar (with a lid). **Used crystals can be saved for later re-use.**

Apply safe microbiological practices as described in the CDC-NIH Biosafety in Microbiological and Biomedical Laboratories (a copy of the document is in the computer room).

**Wear a lab coat, safety glasses with side shields and gloves when handling BSL-2 material. Two pairs of gloves are required if working with sharps. If samples are not contained, face mask is also required when samples are outside of the BSC. Wash hands (next to the Biosafety Cabinet) or use hand sanitizer properly (at the hutch exit and control room exit) after handling the material, after removing gloves, and before leaving the facility.**

No mouth-pipetting is allowed.

Avoid using glass pipettes whenever possible. Do not handle broken glass/sharps by hand.

Use of needles is forbidden, unless a special permission is obtained from ANL IBC.

Keep workplaces clean and disinfect twice a day and immediately after a spill.

Store all items not needed for the experiment outside the containment BSL-2 area.

In case of a loss of crystal or spill, inoculation, facility HVAC/equipment failure, or other emergency follow the list of action listed in SOP 108, 109, 110.

When the biocontainment mode of the air handling is in effect, adjustment of the exhaust rate from the hutch is slow. To reduce the potential turbulence of the cold gas stream from the cryo-cooler, when closing the manual hutch door in 14-BM-C:

- close the door partially (to engage the door switch for exhaust adjustment)
- wait until the exhaust rate from the hutch drops to ~500cfm (monitor on the EPICS screen in the control room) before closing the door completely (few minutes).

14-ID-B door is automatic so just close the hutch door as usually.

**User home institution:**

**List of experimenters:**

---

---

---

---

**I have read and understood this SOP:**

Instructor:

Experimenters:

Name: \_\_\_\_\_ Date: \_\_\_\_\_

Signatures:

---

---

---

---

## Checklist 2: Preparation of the Facility for a BSL-2 Experiment

### 1. Biosafety Cabinet Room

Is BSC used? :  Yes  No

If Yes is checked:

- Biosafety Cabinet is running with the proper down flow rate (~60 lfpm) and exhaust rate (~720-750cfm) for at least 20 minutes.

Down flow rate:

Exhaust rate:

- Cabinet interior is clean. Plastic-backed absorbent paper is in place (not obstructing the air flow).

- Biohazard bags and sharp containers are in the cabinet (not obstructing the air flow).

**Other preparation:**

- A larger biohazard container for disposal of overalls and gloves is in place.

- BioCARS portable autoclave is in place.

- "NO USE" sign is posted on the Cold Room door. Cold Room lights/fan are turned OFF. Fan opening is sealed.

Post at the door:

- Copy of **Checklist 1**

- ESAF

- Biohazard sign

### 2. BSL-2 Hutch

- Floor is mopped and dust removed as much as possible. All unnecessary items are removed.

- User cart is in place if needed.

- Plastic-backed absorbent paper covering as much of the equipment as possible is in place.

- Biohazard bags and sharp containers are in place.

- A container with disinfecting solution is in place and labeled.

- A non-breakable container with a lid for disposing used crystals is in place and labeled.

- Tray with absorbent paper is placed under sample. Remind users to soak paper often with bleach.

- Hand sanitizer dispenser is in place at the exit of the hutch.

- Set up a video camera or an alternative means to monitor the temperature of the cryo-cooler from outside of the hutch.

- If ID-B hutch is used, seal the laser port on the ID-B hutch ceiling.

### 3. BSL-2 Control Room

- Floor is mopped and dust removed as much as possible. All unnecessary items are removed.
- Portable sink and eye wash from the APS are in place and functional.
- Control room emergency exit door is locked from outside. Biohazard sign is posted.
- Sign posted on emergency exit door (inside):  
“Stop! Emergency Exit Only!”
- All regular refuse and sharp containers are removed. Biohazard bags and biohazard sharp containers are labeled and in place
- A larger biohazard container for disposal of lab coats and gloves is in place.
- A container with disinfecting solution is in place and labeled.
- Hand sanitizer dispenser is in place at the exit of the control room.
- Computer keyboards are covered by a plastic protector or replaced by washable keyboards. Computer mice are covered by a plastic protector.
- Copy of ESAF is posted on the door.

### 4. Corridor

- Access to the corridor from other, non-BSL-2 control rooms blocked and sign posted not to enter the corridor
- Hand sanitizer dispenser in place at the exit of the corridor
- A larger biohazard container for disposal of lab coats and gloves in place

### 5. Computer room

#### Post at the door to the BioCARS corridor:

- ESAF
  - Biohazard sign
  - List of authorized personnel
  - List of user and BioCARS staff emergency contacts and phone numbers
- The door to the corridor is chained off (chain is far enough to permit safe opening of the door and exit from the corridor).
  - A supply of PPE is in place.
  - Copy of **Biosafety in Microbiological and Biomedical Laboratories and BioCARS SOP** is available.

### 6. Post on the main entrance of the facility (double door entrance from the APS experimental hall to the computer room)

- Biohazard sign
- List of all authorized personnel
- List of user and BioCARS staff emergency contacts and phone numbers

**7. AHU panel (by the ID-B station)**

- Lock the AHU panel cover and keep the key in the office for the duration of the experiment.
- Post a “No Access” sign on the cover.

**8. Administrative**

- Inform neighboring Sectors 13 and 15 (directly or via CARS Safety Officer) about the BSL-2 experiment at BioCARS.
- Inform the custodian (directly or via APS Floor Coordinator) not to enter the facility for the duration of the experiment.
- Place the BSL-2 Procedure Logbook at the BioCARS User Administrator’s desk. The Procedure Logbook contains BioCARS sign-up sheet, User Sample Questionnaire, ESAF, MSDS for the BSL-2 agent, Sample Log-in/Log-out form and all checklists from this SOP.
- Take the facility key from the key box and keep in the office for the duration of the experiment. Return it to the key box only after the BSL-2 waste is picked up.

Signature of the BioCARS staff contact for the experiment:

Name: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**Checklist 3: Shift Duties of the BioCARS Operating Staff**

If non-BSL-2 users are starting an experiment on a non-BSL-2 station, explain:  
1) emergency doors will be used to enter/exit their control room; the entrance/exit through corridor is not permitted  
2) evacuation procedure and route in case of an emergency  
3) Cold Room cannot be used

If biocontainment mode is required (see **Checklist 0**), check if the appropriate station is selected as BSL-3 (EPICS Status screen).

Biosafety cabinet is operating properly:  
down flow rate (~60fpm):  fpm  
exhaust rate (~750cfm):  cfm

Exhaust Fan 104 is working properly (check Metasys EPICS screen on any BioCARS linux computer).

If biocontainment mode is required, record pressure differentials (**compare to BioCARS BSL-2/3 Maintenance Logbook**)

BSL-2 control room:   
Computer room:   
BSC cabinet room:

BSL-2 station emergency door is locked from the outside.

Biohazard sign, list of authorized personnel and emergency contacts are posted at the BioCARS facility door and at the entrance to the corridor.

Signature of the BioCARS operating staff member:

Name: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

**Checklist 4:**      [Response by BioCARS staff on duty to failure of the facility HVAC \(SOP 109\) or emergency outside of the BSL-2 facility \(SOP 110\)](#)

- In case of medical or other emergency dial 911 and provide details.**
- If present in the facility, non-BSL-2 users are instructed to leave the facility immediately using the explained (see **Checklist 3**) exit path (emergency doors). BSL-2 users will follow **SOP 108, 109 or 110**.
- Emergency personnel – user and staff – contacted. BioCARS Biosafety coordinator contacted. APS Floor Coordinator contacted.
- Make sure all BSL-2 users are out by checking the BSL-2 control room through the glass window of the emergency exit from the APS experimental hall. Check if the control room emergency exit door is locked.
- Tape off the entrance to the BSL-2 area (from the computer room) and post a sign (“Warning! Do not enter! Biohazardous condition.”)
- Make sure all non-BSL-2 users are out by checking the non-BSL-2 control rooms using emergency exits.
- All users are accounted for.

Signature of the BioCARS operating staff member:

Name: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_



**Checklist 5:      Facility Cleanup after the BSL-2 experiment**

- Absorbent paper is removed from the BSC and the BSL-2 hutch. Fold paper carefully so that the potentially contaminated surface remains inside. Do not crumple/wad the paper.
- The BSC interior and all horizontal and vertical surfaces in the containment area are wiped with disinfectant solution
- All biohazard bags and sharp containers collected and placed in the BSC room for waste pickup (see SOP 111). Biohazard bags are double bagged and placed in shipping boxes when boxes are provided by the ANL Waste Management.
- Non-disposable contaminated tools and materials are disinfected and then either autoclaved or packed to be transported (as biohazards) to the home institution for autoclaving
- Standard refuse and sharps containers placed back
- Emergency exit door unlocked
- Biohazard signs removed
- All other BSL-2 related postings removed
- Unblock the hutch exhausts in non-BSL-2 hutches
- Unseal the Cold Room fan in the BSC room
- Facility key return to standard location (key box)
- Biohazard Folder with all forms removed from User Administrator's desk
- Waste autoclaved or picked up by the ANL Waste management.

Signature of the BioCARS staff contact for the experiment:

Name: \_\_\_\_\_ Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**BioCARS staff: I read, understood and will comply with this SOP.**

**Name (print)**

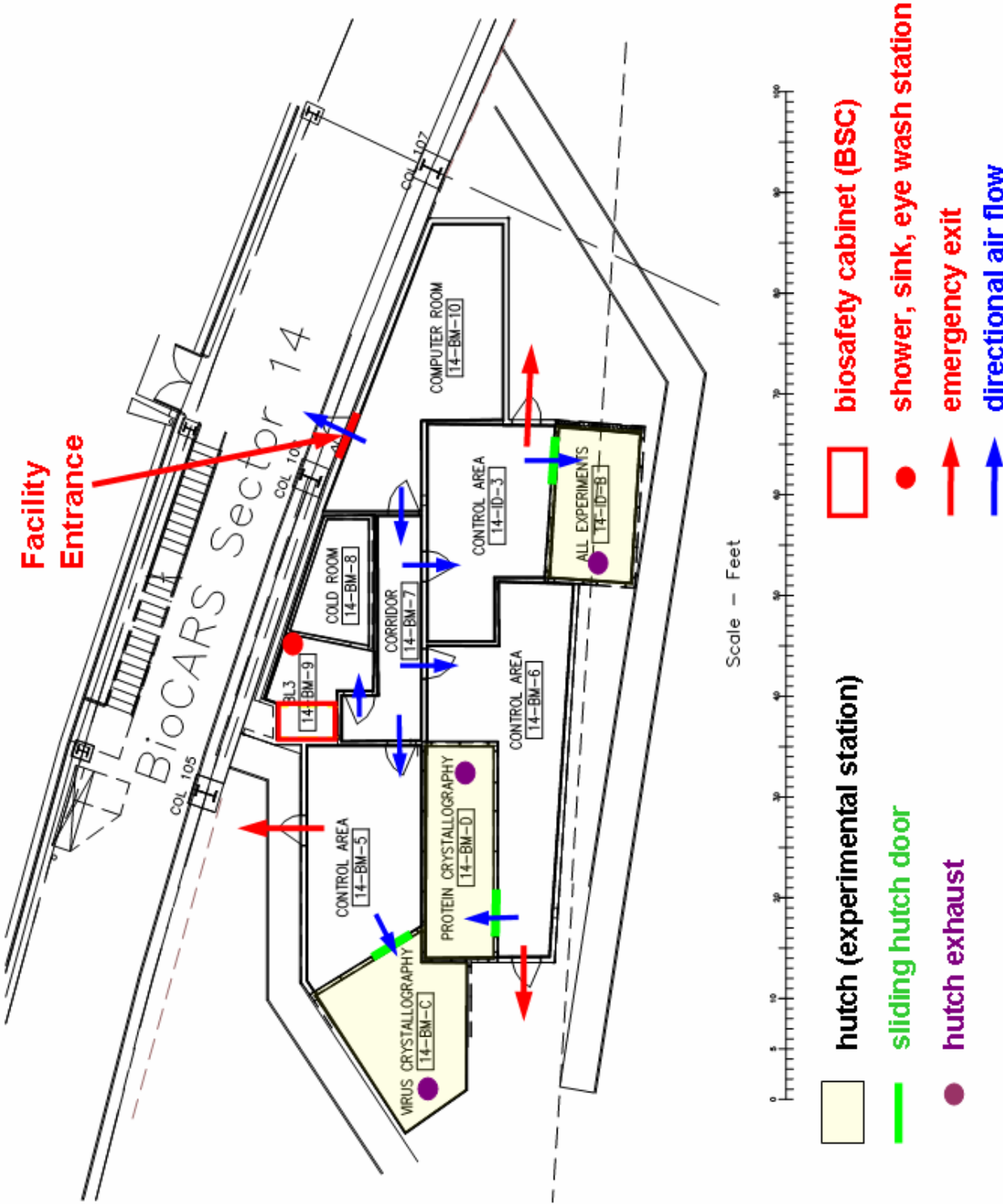
**Signature**

**Date**

_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

# Appendix A

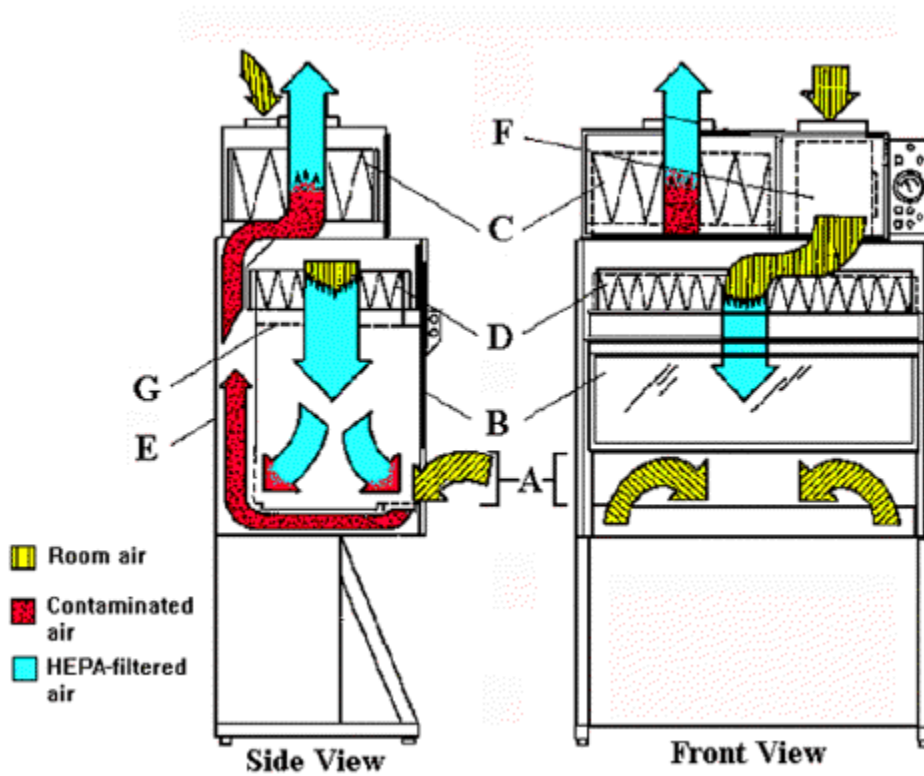
## BioCARS Facility Layout



## Appendix B

### BioCARS Biosafety Cabinet

#### Class II, Type B2 Biosafety Cabinet



- A. front opening
- B. sash
- C. exhaust HEPA filter
- D. supply HEPA filter
- E. negative pressure exhaust plenum
- F. supply blower
- G. filter screen

**Note:** The cabinet exhaust is connected to the facility exhaust fan EF104.

## Biosafety Cabinet (BSC) Work Practices and Procedures

Addapted from: Primary Containment for Biohazards:  
Selection, Installation and Use of Biological Safety Cabinets  
SECTION V  
BSC Use by the Investigator: Work Practices and Procedures  
U.S. Department of Health and Human Services  
Public Health Service  
Centers for Disease Control and Prevention *and* National Institutes of Health  
September 1995

### Preparing for Work Within a BSC

- BSC blowers shall be operated at least five minutes before beginning work to allow the cabinet to "purge".
- The work surface, the interior walls (not including the supply filter diffuser), and the interior surface of the window shall be wiped with 70% ethanol (EtOH), a 1:100 dilution of household bleach (i.e., 0.05% sodium hypochlorite), or other disinfectant as determined by the investigator to meet the requirements of the particular activity. When bleach is used, a second wiping with sterile water is needed to remove the residual chlorine, which may eventually corrode stainless steel surfaces.
- Similarly, the surfaces of all materials and containers placed into the cabinet shall be wiped with 70% ETOH to reduce the introduction of contaminants to the cabinet environment.
- Pacing all necessary materials in the BSC before beginning work will minimize the number of arm-movement disruptions across the air barrier of the cabinet.
- The rapid movement of arms in a sweeping motion into and out of the cabinet will disrupt the air curtain. Moving arms slowly, perpendicular to the face opening of the BSC, is recommended.
- Other personnel activities in the BSC room shall be minimized (e.g., rapid movement, open/closing room doors, etc.) as they also may disrupt the BSC air barrier.
- Protective clothing has to be worn (**SOP 106**). Two pairs of gloves are worn to provide hand protection. Gloves shall be pulled over the sleeves, rather than worn inside.
- Manipulation of materials shall be delayed for one minute after placing the hands/arms inside the cabinet. This allows the cabinet to stabilize and to "air sweep" the hands and arms to remove surface microbial contaminants.
- The front grille must not be blocked by anything, including user's arms. Raising the arms slightly will alleviate this problem.
- All operations shall be performed at least 4 inches from the front grille on the work surface.
- Materials or equipment placed inside the cabinet may cause disruption to the airflow. Only the materials and equipment required for the immediate work shall be placed in the BSC. All Extra supplies shall be stored outside the cabinet.

### Material Placement Inside the BSC

- Plastic-backed absorbent toweling shall be placed on the work surface (but not on the front or rear grille openings). This facilitates routine cleanup and reduces splatter and aerosol formation during an overt spill.
- All materials shall be placed as far back in the cabinet as practical. Similarly, aerosol-generating equipment (e.g., vortex mixers, tabletop centrifuges) shall be placed toward the rear of the cabinet.
- Bulky items such as biohazard bags shall be placed to one side of the interior of the cabinet.
- The autoclavable biohazard collection bag shall not be taped to the outside of the cabinet.

- Upright pipette collection containers shall not be used in BSCs nor placed on the floor outside the cabinet. The frequent inward/outward movement needed to place objects in these containers is disruptive to the integrity of the cabinet air barrier and can compromise both personnel and product protection. Only horizontal pipette discard trays containing an appropriate chemical disinfectant shall be used within the cabinet.
- Potentially contaminated materials shall not be brought out of the cabinet until they have been surface decontaminated. Alternatively, contaminated materials can be placed into a closable container for transfer to an autoclave or for other decontamination treatment.

### **Operations Within a BSC**

- Many common procedures conducted in BSCs may create splatter or aerosols. Good microbiological techniques shall always be used when working in a biological safety cabinet.
- The general work flow shall be from "clean to contaminated (dirty)". Materials and supplies shall be placed in such a way as to limit the movement of "dirty" items over "clean" ones.
- Open flames are not used in a biological safety cabinet. An open flame in a BSC would create turbulence which disrupts the pattern of air supplied to the work surface. When absolutely necessary, touch-plate microburners equipped with a pilot light to provide a flame on demand may be used. The burner must be turned off when work is completed.
- Users must determine the appropriate method of decontaminating materials that will be removed from the BSC at the conclusion of the work.

### **Decontamination**

#### *Surface Decontamination*

- All containers and equipment shall be surface decontaminated and removed from the cabinet when work is completed. At the end of the work, the final surface decontamination of the cabinet shall include a wipe-down of the work surface, the cabinet's sides and back, and the interior of the glass. Users shall remove their gloves and gowns and wash their hands as the final step.
- Small spills within the BSC can be handled immediately by removing the contaminated absorbent paper toweling and placing it into the biohazard bag. Any splatter onto items within the cabinet, as well as the cabinet interior, shall be immediately wiped with a towel dampened with decontaminating solution. Gloves shall be changed after the work surface is decontaminated and before placing clean absorbent toweling in the cabinet. Hands shall be washed whenever gloves are changed or removed.
- Spills large enough to result in liquids flowing through the front or rear grilles require more extensive decontamination. All items within the cabinet shall be surface decontaminated and removed. After ensuring that the drain valve is closed, decontaminating solution can be poured onto the work surface and through the grille(s) into the drain pan.
- Twenty to thirty minutes is generally considered an appropriate contact time for decontamination, but this varies with the disinfectant and the microbiological agent. Manufacturer's directions shall be followed. The spilled fluid and disinfectant solution on the work surface shall be absorbed with paper towels and discarded into a biohazard bag. The drain pan shall be emptied into a collection vessel containing disinfectant. A flexible tube shall be attached to the drain valve and be of sufficient length to allow the open end to be submerged in the disinfectant within the collection vessel. This procedure serves to minimize aerosol generation. The drain pan shall be flushed with water and the drain tube removed.

#### *Gas Decontamination*

BSCs that have been used for work involving infectious materials must be decontaminated before HEPA filters are changed or internal repair work is done. Before a BSC is relocated, a risk assessment which considers the agents manipulated within the BSC must be done to determine the need for decontamination. The most common decontamination method uses formaldehyde gas, although more recently hydrogen peroxide vapor has been used successfully.

## Appendix C

### Questionnaire for BioCARS Proposals Involving Viruses

#### OBJECTIVES

Objectives of the questionnaire are to determine and *validate*

- (a) whether the use of the material is permitted at BioCARS,
- (b) the appropriate virus-specific hazard controls, and
- (c) the applicable regulatory requirements for transportation

#### INSTRUCTIONS

Please submit a separate questionnaire for each viral species proposed. However, contact the BioCARS Biosafety Coordinator if you believe that responses for *multiple closely related viruses/serotypes* may be adequately described by completion of a *single* questionnaire.

Please provide additional information when requested as an attachment to the questionnaire (refer to the relevant question).

If you find that a YES or NO response is inadequate/ambiguous without explanation, please provide clarification in the attachment.

#### Scientist responsible for experiment

\_\_\_\_\_

**Address** \_\_\_\_\_

**Phone number** \_\_\_\_\_

**E-mail:** \_\_\_\_\_

**Signature:** \_\_\_\_\_

#### 1. Identify the virus

2.1. Formal name of virus, including (if applicable) serotype/variant, etc.

\_\_\_\_\_

2.2. Informal/common name of virus and/or disease

\_\_\_\_\_

2.3 Classification of the virus



BSL-2 BSL-3 **2. Regulatory requirements**

		YES	NO
<b>2.1</b>	The virus is a viable form of EITHER (a) an HHS select agent virus, or (b) a USDA high consequence virus. (See list in Appendix Q1) <b>If YES, STOP.</b> <b>The virus is NOT permitted at BioCARS.</b>		
<b>2.2</b>	The virus is a viable form of a viral etiologic agent for which interstate transport is subject to the regulations defined by 42 CFR Part 72. (See Appendix Q2 for list of regulated viruses.)		
<b>2.3</b>	A federal permit for importation or interstate transportation is required. (See Appendix Q3 for information about the regulations.)		

**3. Availability of information from common sources\***

		YES	NO
<b>3.1</b>	An Agent Summary Statement is in Section VII-F of the CDC “Biosafety in Microbiological and Biomedical Laboratories” <a href="http://www.cdc.gov/od/ohs/biosfty/bmb14/bmb14toc.htm">http://www.cdc.gov/od/ohs/biosfty/bmb14/bmb14toc.htm</a>		
<b>3.2</b>	An MSDS for the virus is published by Health Canada <a href="http://www.hc-sc.gc.ca/pphb-dgsp/psd/ftss/index.html">http://www.hc-sc.gc.ca/pphb-dgsp/psd/ftss/index.html</a>		
<b>3.3</b>	A Risk Group is identified in Appendix B of the NIH Guidelines for Research Involving Recombinant DNA Molecules <a href="http://www4.od.nih.gov/oba/rac/guidelines_02/NIH_Guidelines_Apr_02.htm">http://www4.od.nih.gov/oba/rac/guidelines_02/NIH_Guidelines_Apr_02.htm</a>		
<b>3.4</b>	A biosafety category appears in the matrix compiled by the American Biological Safety Association <a href="http://www.absa.org/resriskgroup.html">http://www.absa.org/resriskgroup.html</a>		
<b>3.5</b>	The pathogenic/epidemiologic characteristics of the virus is described in the CDC Infectious Diseases Information Index <a href="http://www.cdc.gov/ncidod/diseases/index.htm">http://www.cdc.gov/ncidod/diseases/index.htm</a>		
<b>3.6</b>	The characteristics of the virus/disease are described by the World Health Organization <a href="http://www.who.int/health-topics/idindex.htm">http://www.who.int/health-topics/idindex.htm</a> and <a href="http://www.who.int/vaccine_research/diseases/en/">http://www.who.int/vaccine_research/diseases/en/</a>		

\*Some of the queries in Sections 3 and 4 require citations of appropriate literature. For some responses, citation of the sources above (or similar resources) may be adequate.

#### 4. Biosafety at your institution

		YES	NO
4.1	<p>Has each user been trained and authorized for the work with the virus at your home institution by the home institution's IBC?</p> <p>If <b>YES</b>, provide a letter of authorization.</p> <p>If <b>NO</b>, <b>please obtain training and authorization. They are required for conducting experiments at BioCARS.</b></p>		
4.2	<p>Your organization's Institutional Biosafety Committee has approved a protocol/standard operating procedure for the work with the virus in your laboratory.</p> <p>If <b>YES</b>, provide</p> <ul style="list-style-type: none"> <li>• The minimum biosafety level authorized by the IBC</li> <li>• Evidence of IBC approval</li> <li>• The protocol approved by the IBC.</li> </ul> <p>If <b>NO</b>, explain why.</p> <p><b>If your IBC authorized laboratory manipulation of the virus under BSL-1 hazard controls, consult the BioCARS biosafety officer before completing the remainder of this table.</b></p>		
4.3	<p>Your institution requires screening by medical professionals to identify individuals who may be more susceptible to infection than the normal adult population.</p> <p><b>Each user must sign the Risk Factor Form (Appendix D of the SOP).</b></p>		
4.4	<p>Use of the virus in your laboratory or vivarium requires respiratory protection.</p> <p>If <b>YES</b>, describe the type(s) of devices used.</p> <p><b>As use of respirators is required for work with BSL-3 samples at BioCARS. BioCARS SOP requires evidence of respirator training and fit-testing in accordance with OSHA standards.</b></p>		
4.5	<p>An FDA-approved vaccine is available for the virus.</p> <p>If <b>YES</b>, explain the vaccination policy at home institution.</p>		
4.6	<p>FDA-approved antiviral therapeutic agent(s) are available for beneficial treatment of infected humans.</p> <p>If <b>YES</b>, identify the therapeutic agent(s) and whether your institution maintains a supply.</p>		
4.7	<p>Information about the stability of the virus on various types of surfaces, especially those relevant to a laboratory is available (from the literature and/or acquired in your laboratory).</p> <p>If <b>YES</b>, provide brief summary information and describe its source*.</p> <p><b>Data (from literature or home institution) on viability of the agent on typical lab surfaces have to be provided if work with frozen BSL-3 crystals is planned.</b></p>		

		YES	NO
<b>4.8</b>	Information about disinfectants to which the virus is particularly susceptible and/or relatively resistant is available (from the literature and/or acquired in your laboratory). If <b>YES</b> , provide brief summary information and describe its source*. <b>Effective disinfectant:</b>		

\* See Section 2 for typical sources of information.

**5. Additional information about the virus**

The source of the virus or its serotype/variant is (check all that apply):

- A repository
- Another research group
- A naturally infected host species obtained by your laboratory
- A host species experimentally infected in your laboratory
- Cultured host cells experimentally infected in your laboratory
- Other

Briefly explain each indicated source.

	YES	NO	
<b>5.1</b>			There is published information describing multiple naturally occurring serotypes/variants of the virus. If YES, describe the characteristics of the serotype(s)/variant(s) proposed for analysis, with emphasis on enhancement or diminution of pathogenicity, severity of disease, transmissibility, infectivity, susceptibility of certain populations, environmental stability, sensitivity to anti-viral drugs, effectiveness of vaccines, or other characteristics relevant to biohazard. Is the serotype/variant proposed for analysis indigenous to a particular region? Provide literature references if available.*
<b>5.2</b>			The virus has been intentionally genetically modified either in your laboratory or another investigator's laboratory. If YES, describe the genetic modification and its effect (as available) on the characteristics indicated in item ___ immediately above.
<b>5.3</b>			Inhalation is a primary route of transmission for the principal natural host(s).
<b>5.4</b>			Although inhalation is not a primary route of transmission, there is published evidence that inhalation is, or is likely to be, a route of infection under some conditions, e.g., experimental animal models, livestock workers, or those who work with infected tissues or laboratory cultures. If YES, provide brief, summary information and references.*
<b>5.5</b>			An arthropod is a natural vector for infection of higher animals or humans.
<b>5.6</b>			Humans are among the natural hosts.
<b>5.7</b>			Non-human primates are among the natural hosts.
<b>5.8</b>			There is published evidence that the virus is infectious for non-human primates, even if primates are not the principal natural host.
<b>5.9</b>			Although the principal host(s) is non-primate animals, there is published evidence for zoonotic infection of humans, e.g., for livestock workers, consumers of products from infected animals; or those who work with infected tissues or laboratory cultures.
<b>5.10</b>			Human to human transmission of the viral disease is known to occur. If YES, describe the prevalence and route(s) of such transmission; and provide definitive references.*

	YES	NO	
<b>5.11</b>			There is documented evidence that asymptomatic humans may serve as short- and/or long-term sources for transmission of disease. If YES, describe the prevalence of such transmission and provide definitive references.*
<b>5.12</b>			There is published evidence that specific human populations are more susceptible to infection or the consequences of infection (e.g., newborns, elderly, fetus, immunocompromised, other medical condition). If YES, provide brief summary information and references.*
<b>5.13</b>			Information is available, even if approximate, regarding the infectious dose in (as available) the principal host(s), in humans, or in an experimental animal model(s). If YES, provide information about dose, species, and route(s).*

\* See Section 2 for typical sources of information.

## Questionnaire: APPENDIX Q1

### Select Agents and High Consequence Agents Regulated by HHS and USDA

#### *HHS Select Agents and Overlap Select Agents<sup>1</sup>*

- Crimean-Congo haemorrhagic fever virus
- Ebola virus
- Cercopithecine herpesvirus 1 (Herpes B virus)
- Lassa fever virus
- Marburg virus
- Monkeypox virus
- South American Haemorrhagic Fever viruses (Jinni, Machupo, Sabia, Flexal, Guanarito)
- Tick-borne encephalitis complex (flavi) viruses (Central European Tick-borne encephalitis, Far Eastern Tick-borne encephalitis [Russian Spring and Summer encephalitis, Kyasanur Forest disease, Omsk Hemorrhagic Fever] )
- Variola major virus (Smallpox virus) and Variola minor virus (Alastrim)
- Eastern Equine Encephalitis virus
- Nipah and Hendra Complex viruses
- Rift Valley fever virus
- Venezuelan Equine Encephalitis virus

#### *USDA High Consequence Livestock or Plant Viruses<sup>2</sup>*

- Akabane virus
- African swine fever virus
- African horse sickness virus
- Avian influenza virus (highly pathogenic)
- Blue tongue virus (Exotic)
- Camel pox virus
- Classical swine fever virus
- Foot and mouth disease virus
- Goat pox virus
- Lumpy skin disease virus
- Japanese encephalitis virus
- Malignant catarrhal fever virus (Exotic)
- Menangle virus
- Newcastle disease virus (VVND)
- Peste Des Petitis Ruminants virus
- Sheep pox virus
- Swine vesicular disease virus
- Vesicular stomatitis virus (Exotic)
- Plum Pox Potyvirus
- Rinderpest virus

---

<sup>1</sup> Possession, Use, and Transfer of Select Agents, Interim Final Rule, 42 CFR Part 73 (December 13, 2002). Department of Health and Human Services, Office of the Inspector General <http://www.cdc.gov/od/sap/docs/btarule.pdf>

<sup>2</sup> Possession, Use and Transfer of Biological Agents and Toxins, 7 CFR 331 and 9 CFR 121 (December 13, 2002). Department of Agriculture, Animal and Plant Health Inspection Service (APHIS) [https://www.aphis.usda.gov/brs/fedregister/BRS\\_20021213a.pdf](https://www.aphis.usda.gov/brs/fedregister/BRS_20021213a.pdf)

## Questionnaire: APPENDIX Q2

### Viral Etiologic Agents for Which Interstate Transport is Regulated

#### by 42 CFR Part 72

(Appendix Q2 does not include those etiologic agents regulated by 42 CFR Part 73, and thus appear in Appendix Q1 for this BioCARS questionnaire).

- Adenoviruses—human—all types
- Arboviruses—all types
- Coxsackie A and B viruses—all types
- Cytomegaloviruses
- Dengue viruses—all types
- Echoviruses—all types
- Encephalomyocarditis virus
- Hemorrhagic fever agents including, but not limited to, Crimean hemorrhagic fever(Congo), Junin, Machupo viruses, and Korean hemorrhagic fever viruses
- Hepatitis associated materials (hepatitis A, hepatitis B, hepatitis nonA-nonB)
- Herpesvirus—all members
- Infectious bronchitis-like virus
- Influenza viruses—all types
- Lymphocytic choriomeningitis virus
- Measles virus
- Mumps virus
- Parainfluenza viruses—all types
- Polioviruses—all types
- Poxviruses—all members
- Rabies virus—all strains
- Reoviruses—all types
- Respiratory syncytial virus
- Rhinoviruses—all types
- Rotaviruses—all types
- Rubella virus
- Simian virus 40
- Tick-borne encephalitis virus complex, including Russian spring-summer encephalitis, Kyasanur forest disease, Omsk hemorrhagic fever, and Central European encephalitis viruses.
- Vaccinia virus.
- Varicella virus
- Vesicular stomatis viruses—all types
- White pox viruses
- Yellow fever virus

## **Questionnaire: APPENDIX Q3**

### **USDA/APHIS Permit for Interstate Transport**

#### Explanation

Federal regulations require a USDA-APHIS (Veterinary) permit for interstate transfer of

All cultures or collections of organisms or their derivatives, which may introduce or disseminate any contagious or infectious disease of animals (including poultry). [9 CFR 122.1(d)]

Information about the requirements is available at <http://www.aphis.usda.gov/vs/ncie/>.

The permit is available at <http://www.aphis.usda.gov/vs/ncie/pdf/vs16-3.pdf>

The instructions for permit application VS 16-3 include the following:

“Generally, a USDA veterinary permit is needed for materials derived from animals or exposed to animal-source materials. Materials which require a permit include animal tissues, blood, cells or cell lines of livestock or poultry origin, RNA/DNA extracts, hormones, enzymes, monoclonal antibodies for IN VIVO use in non-human species, certain polyclonal antibodies, antisera, bulk shipments of test kit reagents, and microorganisms including bacteria, viruses, protozoa, and fungi.”

See also <http://www.cdc.gov/od/ohs/biosfty/impertper.htm> and the application form and explanatory information at <http://www.cdc.gov/od/ohs/biosfty/impertper.htm>

### **USER RESPONSIBILITY FOR PERMITTING**

The USER and his/her institution are solely responsible for acquiring the required permit or permits. When completing a permit application, the user’s institution shall be both the shipper and the receiver, thus avoiding the identification of Argonne National Laboratory as either a recipient or shipper. Within BioCARS, the user is responsible for maintaining custody of materials requiring a transportation and/or import permit.

Argonne National Laboratory reserves the authority request evidence of permitting prior to final authorization for the experiment, and upon arrival of the material at Argonne National Laboratory. If the permitting requirements are not adequately met, Argonne National Laboratory reserves the authority to either prohibit or limit the use of the material.

### **PLAN AHEAD FOR PERMITTING**

Acquisition of a permit may require much longer than expected.

## Appendix D

### Risk Factors Form

#### Individual Notification of Risk Factors

Some medical conditions increase susceptibility to infection and/or increase the severity of the consequences of infection. For example, persons who are immunocompromised or immunosuppressed may be at increased risk of acquiring infections. Immunocompromised and/or immunosuppressed individuals at risk include (but are not limited to) those with HIV infection, cancer, or other chronic illnesses (e.g., diabetes, lupus and other autoimmune diseases, eczema, dermatitis, certain respiratory diseases/conditions); those undergoing drug therapy that causes immunosuppression; and those who have undergone certain surgical procedures (e.g., splenectomy, gastrectomy). Certain infectious agents may adversely affect a fetus during pregnancy if the mother is infected with the agent. Pregnancy is a predisposing risk factor for some infectious diseases. Alcoholism and drug abuse are also predisposing risk factors for some infectious diseases.

Name of individual \_\_\_\_\_

Institutional Affiliation \_\_\_\_\_

Identifying number (APS/ANL system) \_\_\_\_\_

Name of individual's supervisor, the principal investigator, or equivalent

\_\_\_\_\_

***Individual's acknowledgement:* By my signature below, I acknowledge that I have read and understood the risk factors information provided above.**

Signature \_\_\_\_\_ Date \_\_\_\_\_



## Appendix E

### BSL-2 Sample Log-in/Log-out Form

**PI:**  
**ESAF Pen #:**  
**Experiment title:**  
**Spokesperson:**

#### Log-in

##### Transportation info:

Transported by users

Shipped via:

##### Arrival date/time:

##### Samples transported/shipped:

Frozen

Number of dewars:

Number of crystals:

Room temperature/trays

Number of trays:

Number of drops/wells:

Estimated number of crystals:

Room temperatures/capillaries

Number of capillaries/crystals:

Spokesperson signature: \_\_\_\_\_

## Log-out

**Departure date/time:**

**Number of crystals used for data collection:**

**Any crystals lost during mounting/dismounting:**

Yes. How many:

No.

### **User Log-out Certification:**

I certify that all BSL-2 material transported/shipped to BioCARS has been either rendered non-viable or is being sent back to the home institution.

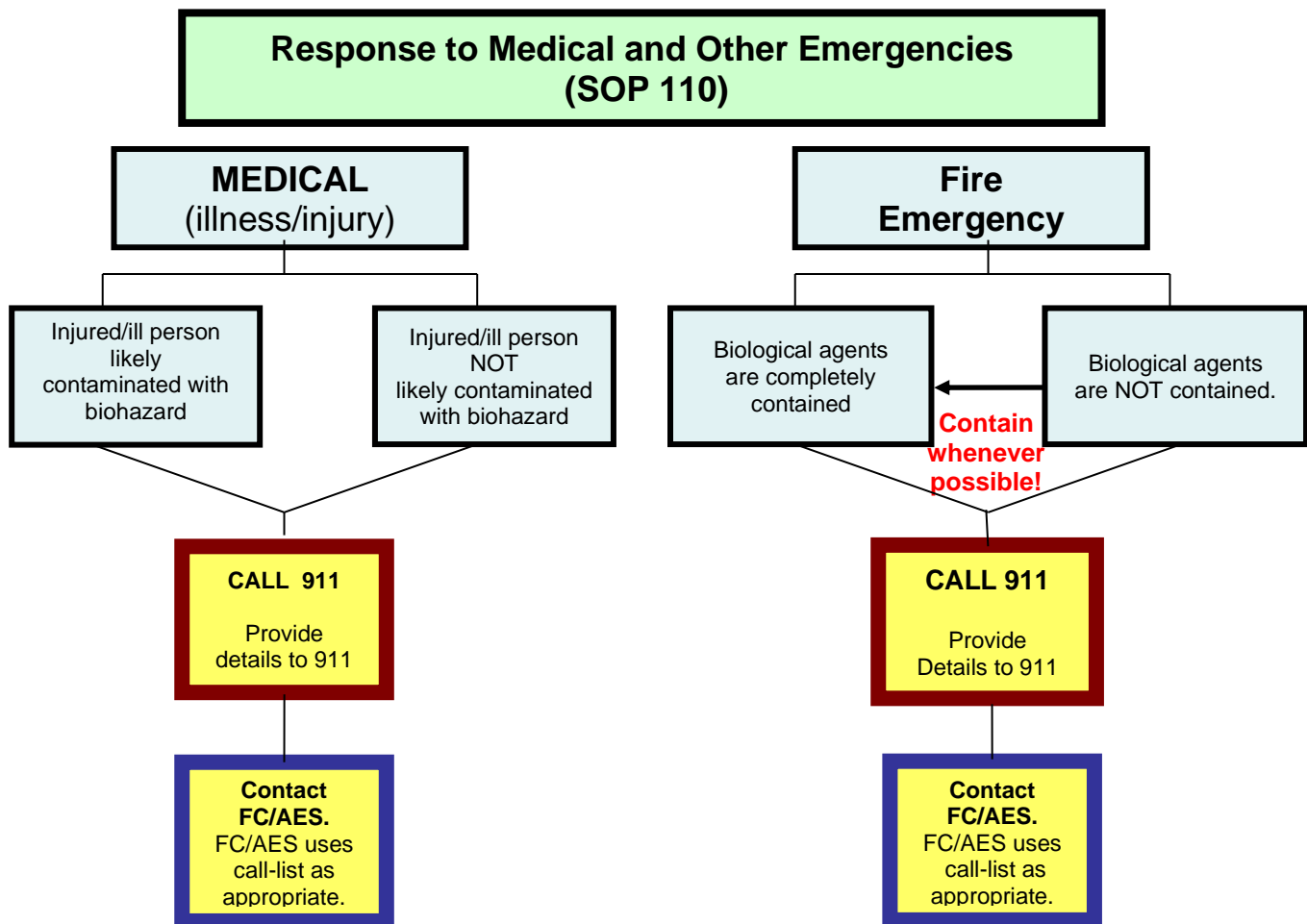
Spokesperson signature:

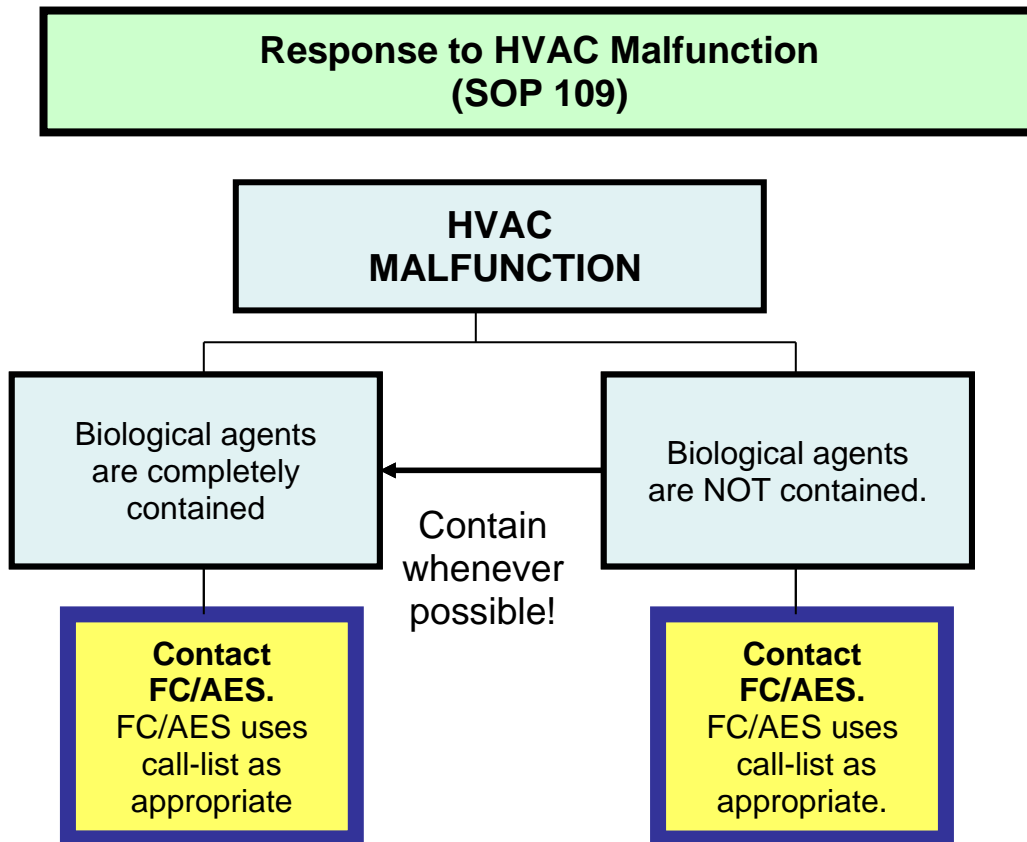
---

## Appendix F

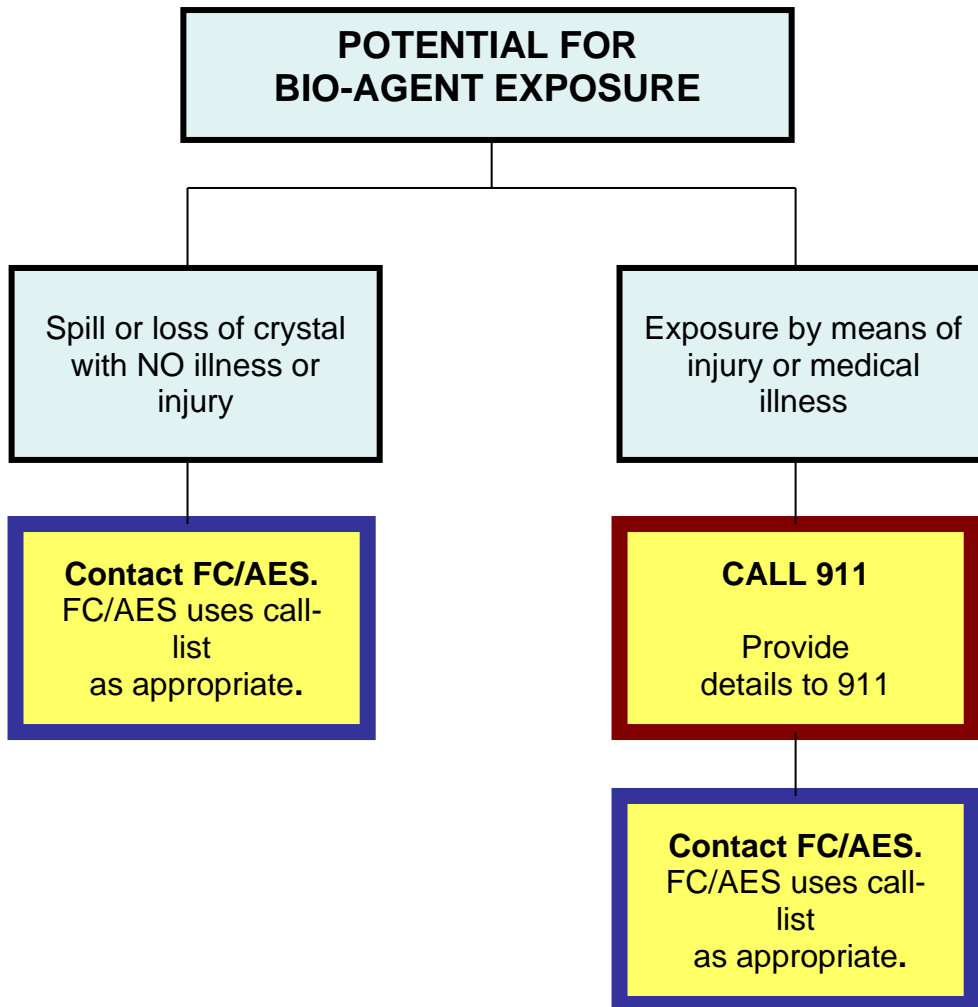
### Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS

- A. Medical and other emergencies during the presence of biohazardous materials
- B. Failure of HVAC or other system/equipment important for biosafety
- C. Response to overt or potential exposure of personnel to biohazardous material
- D. VHP (vapor phase hydrogen peroxide) leak detected and not contained

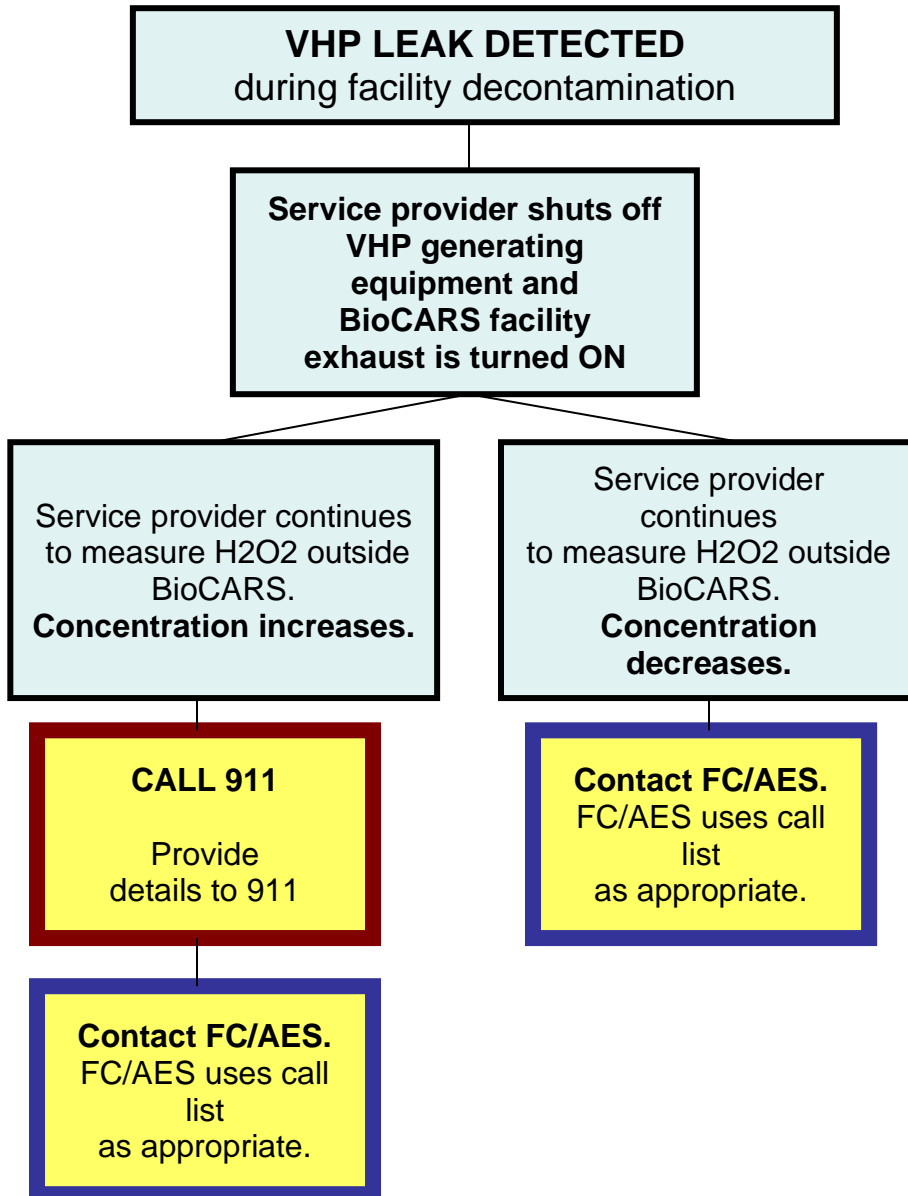




**Response to Potential Exposure to Biohazard  
(SOP 108)**



**Response to VHP leak to Experiment Hall  
(SOP 113)**



## Pressure Differentials in BioCARS Facility

**Pressure differentials in the BioCARS facility (from pressure monitors; units: inch H<sub>2</sub>O)**

**Status on April 3, 2006 (Rob Henning)**

		Computer room	ID-B	BM-C	BM-D	BSC
ID-B BSL-3 mode	Hutch door open	+0.021	-0.002	-0.010	-0.007	-0.006
	Hutch door closed	+0.022	-0.004	-0.007	-0.007	-0.006
	ID-B emergency door open	+0.020	-0.000	-0.010	-0.007	-0.006
BM-C in BSL-3 mode	Hutch door open	+0.020	-0.002	-0.030	-0.005	-0.006
	Hutch door closed	+0.021	-0.003	-0.009	-0.007	-0.007
Normal mode		+0.021	-0.003	-0.009	-0.007	-0.007

All light/alarm set points set to about 0 (setting is not precise).

It takes 5-10min for equilibration of the pressures/flows after the hutch door contact has been activated/deactivated while closing/opening hutch door (this contact activates flow rate adjustments).

When the hutch door is open (contact deactivated), the exhaust rate from the hutch is quite high (no air re-circulation). If the door is closed too quickly (before the flow rates have adjusted), turbulence inside the hutch will be created that will disturb the cryo gas stream.

**Set points set by Reinhard Pahl and Vukica Srajer (8/17/04)**

**Appendix C.15 BioCARS - SOP for Biosafety Level 3 Experiments at BioCARS Facility (November, 2014)**

**NOTE: Blue text highlights differences for BSL2 and BSL3 SOPs**

---

**Standard Operating Procedures for  
Biosafety Level 3 Experiments at BioCARS Facility**

---

July 2008  
Revised February, 2014  
Revised November, 2014

Prepared by:  
Vukica Srajer

Approved by:

	<i>Signature</i>	<i>Date</i>
Dan Schabacker, ANL IBC Chair	_____	_____
Bruce Glagola, APS User Safety Officer	_____	_____
Nena Moonier, APS Biosafety Officer	_____	_____
Keith Moffat, BioCARS PI	_____	_____
Guy Macha, CARS Safety Officer	_____	_____
Vukica Srajer, BioCARS Safety and Biosafety Coordinator	_____	_____



BioCARS maintains as-built drawings, specifications, and control system logic descriptions for the ventilation system as well as the manufacturer's instruction manuals for all units that comprise the HVAC system. BioCARS also maintains documentation that demonstrates performance of the HVAC system, malfunctions and their resolution.

Annual re-certification of the BioCARS facility for the BSL-2/3 operation is initiated by the APS Biosafety Officer. Re-certification is based on procedures approved by the ANL IBC and will be performed by the appropriate APS/ANL personnel. The ANL IBC maintains the records and results of the BioCARS re-certification. A copy of the results is provided to BioCARS.

BioCARS maintains completed forms included in this document, and evidence of medical surveillance and respirator training for BioCARS staff.

BioCARS Biosafety Coordinator will review this SOP as needed and make revisions based on experience and changing facility conditions (the log of reviews is kept in the BioCARS BSL-2/3 Maintenance Logbook). BioCARS Biosafety Coordinator will also revise this SOP if there are major changes in the engineering or administrative controls. Any major revisions of the engineering or administrative controls are subject to approval of the SOP by the ANL IBC.

**Blue marking in this document represents differences between BSL-2 and BSL-3 SOPs.**

**Abbreviations:**

AHU:	Air Handling Unit
ANL:	Argonne National Laboratory
APS:	Advanced Photon Source
BSC:	Biosafety Cabinet
BSL-2:	Biosafety Level 2
BSL-3:	Biosafety Level 3
EM:	Emergency Management, ANL Security and Counterintelligence Division
EQO-IH:	ANL Industrial Hygiene
FMS:	Facility and Management Services Division for Argonne National Laboratory
HVAC:	Heating, Ventilation and Air Conditioning
IBC:	Institutional Biosafety Committee for Argonne National Laboratory
JC:	Johnson Controls
MSDS:	Material Safety Data Sheet
PAPR:	Powered Air Purifying Respirators
VHP:	Vapor-phase Hydrogen Peroxide

## Standard Operating Procedures for Biosafety Level 3 Experiments at BioCARS Facility

### Overview of the Facility and BSL-3 Procedures

#### 1. BioCARS Facility

- 1.1 BioCARS Facility is a National User Facility for Macromolecular Crystallography where users from universities and other research institutions conduct X-ray diffraction experiments on crystals of macromolecules. It is located at Sector 14 at the Advanced Photon Source (APS), Argonne National Laboratory (ANL).
- 1.2 Majority of BioCARS users conduct crystallographic experiments that do not require biocontainment. Typical experiments require 1-3 days.
- 1.3 A sub-group of BioCARS users conducts experiments involving samples that require BSL-2 or BSL-3 biocontainment. Agents classified as BSL-2 involve a broad range of indigenous moderate-risk agents that are present in the community and associated with human disease of varying severity resulting from exposure via contact with mucous membranes, contact with non-intact skin, percutaneous injury, or ingestion. Agents classified as BSL-3 are indigenous or exotic agents associated with serious and potentially lethal human diseases as a result of exposure by the inhalation route. Other agents may include toxins, in which the primary risk is to the experimenter.
- 1.4 Because BioCARS is a facility where both standard and BSL-2/3 experiments are conducted, particular care is taken to properly prepare the facility for the BSL-2/3 operation and to properly return the facility to standard, non-BSL-2/3 operation upon completion of a BSL-2/3 experiment.
- 1.5 The facility consists of one bending magnet experimental station (14-BM-C) and one insertion device station (14-ID-B). Second bending magnet station, 14-BM-D, is no longer in use.
- 1.6 All three experimental stations (hutches) are qualified for BSL-2/3 experiments.
- 1.7 Only a single BSL-2/3 experiment can be conducted at the facility at any time, in one of the BioCARS experimental stations. Other, non-BSL2/3 experiments may be conducted in other two experimental stations at the same time if approved by the ANL IBC for a particular BSL-3 agent.
- 1.8 Cultures of BSL-2/3 microorganisms are prohibited at BioCARS.
- 1.9 There is no long term storage of BSL-2/3 materials at the facility. BSL-2/3 material is shipped by users before the experiment and disposed or shipped back to home institution upon the completion of the experiment (**SOP 103**).
- 1.10 Use of BSL-4 agents and select agents (the latter as defined in, **42 CFR Part 73, 7 CFR Part 331 and 9 CFR Part 121**) is prohibited at BioCARS.

- 1.11 The BioCARS users working with the BSL-2/3 agents have to be qualified and trained at their home institution for working with such materials (**SOP 102, SOP 104**).
- 1.12 BioCARS staff will not be involved in direct work with BSL-2/3 agents. Staff will, however, assist users with the use of BioCARS equipment as well as address hardware and software problems during a BSL-2/3 experiment.
- 1.13 The facility is equipped with a Class II, Type B2 Biosafety Cabinet (BSC). The schematics of the BSC and the BSC practices and procedures are in the **Appendix B**. The BSC is recertified by a certified vendor annually and after any maintenance and ventilation system modifications.
- 1.14 All unpacked BSL-2/3 material will be stored in the BSC room upon arrival. All manipulations of open (uncontained) BSL-3 samples will be conducted in the BSC to the greatest extent possible. [Respiratory protection is required for manipulation of uncontained BSL-3 material elsewhere outside of the BSC within the biocontainment area \(see 1.2.2\)](#).
- 1.15 BioCARS Chemistry Lab and Cold room will not be used for storage or work with BSL-2/3 agents. Cold room cannot be used while BSL-2/3 samples or waste are present in the BSC room.
- 1.16 Only a single crystal of BSL-3 material will be transferred from the BSC or from a dewar with contained, frozen crystals in the BSC or control room to the experimental station where the X-ray diffraction experiment is conducted. A two-person rule is in effect during the crystal transfer. A detailed description of sample containment and transfer is given in **SOP 107**.
- 1.17 [During BSL-3 experiments 24h attendance is required \(in the facility or LOM\) by one user and one BioCARS staff member \(SOP 107\)](#).

## 2. Facility Layout and Air Handling System

- 2.1 The layout of the facility is shown in the **Appendix A**.
- 2.2 A BSL-3 biocontainment area during a BSL-3 experiment consists of:
  - a) one experimental station (hutch)
  - b) adjacent control room
  - c) the facility corridor (14-BM-7)
  - d) the BSC room (14-BM-9).
- 2.3 The shower, sink and eye wash station are located in the BSC room.
- 2.4 The only entrance into the BSL-3 biocontainment area is via computer room (14-BM-10) during the BSL-3 experiments. The door between the BSL-3 control area and the APS experimental hall is locked to prevent entry from the experimental hall, but allows emergency exit from the control room.
- 2.5 The BioCARS HVAC system consists of 5 Air Handling Units (AHU) connected to the main APS conditioned air supply.
- 2.6 The HVAC system controls are pre-set for two operating modes: normal mode (non-BSL-2/3 operation) and biocontainment mode (BSL-2/3 operation).

- 2.7 Prior to a BSL-3 experiment, one BioCARS experimental station is declared a BSL-3 area and the HVAC is switched from standard to biocontainment mode. Both tasks are responsibility of BioCARS staff (**SOP 101**). The specific steps of preparing the facility for the BSL-3 operation are described in **SOP 106**.
  - 2.8 The control unit for changing HVAC configuration (JC-Control Box) is located on the roof of the facility. Access to the roof is limited to BioCARS staff and APS Floor Coordinators by locking the door for the duration of the BSL-3 experiments.
  - 2.9 The selected BSL-3 containment area (2.2) is under negative pressure with respect to the APS experimental hall.
  - 2.10 Directional airflow is established in the facility as shown in **Appendix A**. The non-BSL-3 control rooms and stations are sealed (**SOP 6**) to prevent unwanted air-flow from the BSL-3 containment area.
  - 2.11 Exhaust air from the BSL-3 containment area is HEPA filtered and directed out of the APS building via either the BSC exhaust or a bag-in/bag-out HEPA filter located downstream of the exhaust port from each experimental station. The BioCARS exhaust system is independent of any other APS exhaust system.
  - 2.12 All doors within the facility must remain closed when not in use for the duration of the BSL-3 experiment.
  - 2.13 The status of the HVAC system can be monitored via Epics Status Screen on computers in the control and computer rooms (14-BM-5, 14-BM-6, 14-ID-3, 14-BM-10). The status can also be checked on the Metasys Terminal at the JC-Control Box, located on the roof of the BioCARS Facility (**Checklist 0**).
  - 2.14 An audible alarm will alert experimenters in the facility and BioCARS staff in the office area (LOM) about an HVAC fault condition (**SOP 109**).
  - 2.15 The main exhaust fan (EF104) for the facility and the Biosafety Cabinet are connected to the APS emergency power supply (**SOP 109**).
3. **Access to the Facility**
- 3.1 The entrance to and the exit from the facility during the BSL-2/3 experiments are through the computer room (14-BM-10). The access to the containment area is restricted to authorized personnel only during the BSL-2/3 experiments. The list of authorized personnel for each BSL-2/3 experiment must be posted at the facility entrance door (from the APS experimental hall) during the experiment (**SOP 106**). Same list must also be posted at the entrance to the containment area (on the door to the BioCARS corridor). A removable chain must also be placed in front of the entrance to the containment area (See **Checklist 2**).
  - 3.2 User authorization for each BSL-3 experiment requires:
    - a) signing that they have read and understood this SOP (**Checklist 1**)
    - b) signing the Risk Factors Form (**Appendix D**)

- c) letter by the user home institution IBC authorizing work with the specified BSL-2 agent (**BioCARS questionnaire, Appendix C, 4.1**)
  - d) [certification from the user home institution regarding medical approval, fitting and training for respirator use.](#)
- 3.3** BioCARS staff authorization for each BSL-3 experiment (as staff contact or technical support) requires:
- a) signing that they have read and understood this SOP (**list at the end of the SOP, before Appendix A**)
  - b) signing the Risk Factors Form (**Appendix D**)
  - c) [current medical fitness evaluation \(arranged through University of Chicago Occupational Medicine\)](#)
  - d) completion of the ANL ESH560 training “Biosafety Awareness Training”
  - e) [current respirator training and fit testing from the ANL](#)
- 3.4** Vaccination to the BSL-2 agent (when a vaccine is available) may be required for access by discretion of the BioCARS PI. When a vaccine is available but vaccination is not declared mandatory, BioCARS personnel must sign a vaccine declination form if they choose to not be vaccinated. All personnel that are offered a vaccine will be trained on the vaccine’s benefits and risks before they are asked if they want to receive it.

#### **4. Protective clothing and other PPE**

Protective clothing, [respirator use](#), gowning and de-gowning procedures are explained in **SOP 105**.

#### **5. Responsibilities**

Responsibilities of users, their home institutions, BioCARS staff, BioCARS Safety Committee and ANL/APS safety personnel are listed in **SOP 101**.

#### **6. Incident Response**

An incident is defined as an event that creates overt or potential exposure of users or staff to a BSL-3 agent, such as loss of a crystal outside of the biosafety cabinet (this is considered to be a breach of primary containment of the sample), loss of BioCARS biocontainment where biocontainment area is defined in 2.2.2 above (this is considered to be a breach of secondary containment) or failure of critical equipment (BSC, crystal cryo-cooler). Every incident will be reported by the BioCARS Biosafety Coordinator to the CARS Safety Committee, ANL IBC and APS.

## 2. Standard Operating Procedures and Checklists:

### Standard Operating Procedures:

<b>SOP 101</b> (p. 8):	Responsibilities	(BioCARS users and staff)
<b>SOP 102</b> (p. 12):	Approval of BSL-3 Experiments	(BioCARS users and staff)
<b>SOP 103</b> (p. 13):	Shipping and Receiving of BSL-3 Samples	(BioCARS users and staff)
<b>SOP 104</b> (p. 16):	User Training and Certification	(BioCARS users and staff)
<b>SOP 105</b> (p. 18):	Protective Clothing and Other Personal Protective Equipment	(BioCARS users and staff)
<b>SOP 106</b> (p. 20):	Preparation of the Facility for BSL-3 Operation	(BioCARS staff and APS Floor Coordinator)
<b>SOP 107</b> (p. 21):	BSL-3 Practices and Procedures	(BioCARS users and staff)
<b>SOP 108</b> (p. 25):	Response to Overt or Potential Personnel Exposure to BSL-3 Agent	(BioCARS users, staff and APS Floor Coordinator)
<b>SOP 109</b> (p. 27):	Response to the Failure of Facility HVAC and Other Equipment	(Users, BioCARS staff and APS Floor Coordinator)
<b>SOP 110</b> (p. 29):	Medical and Other Emergencies During the BSL-3 Experiments	(Users, BioCARS staff, APS Floor Coordinator and APS/ANL emergency response team)
<b>SOP 111</b> (p. 30):	BSL-3 Waste Disposal	(Users, BioCARS staff and APS Floor Coordinator)
<b>SOP 112</b> (p. 31):	End of Experiment Procedures	(BioCARS users and staff)
<b>SOP 113</b> (p. 32):	Facility decontamination	(BioCARS staff and APS Floor Coordinator)
<b>SOP 114</b> (p. 33):	HVAC and other Safety Equipment Maintenance and Testing	(BioCARS staff and PSF personnel)

### Appendices:

<b>Appendix A</b> (p. 46):	BioCARS Facility Layout
<b>Appendix B</b> (p. 47):	BioCARS Biosafety Cabinet
<b>Appendix C</b> (p. 51):	Questionnaire for BioCARS Proposals Involving Viable BSL-2 and BSL-3 Viruses
<b>Appendix D</b> (p. 59):	Risk Factors Form
<b>Appendix E</b> (p. 60):	BSL-3 Sample Log-in/Log-out Form
<b>Appendix F</b> (p. 62):	Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS

### Checklists:

<b>Checklist 0</b> (p. 35):	Preparation of the HVAC and BSC
<b>Checklist 1</b> (p. 37):	User Training for BSL-3 Experiments at BioCARS
<b>Checklist 2</b> (p. 39):	Preparation of the Facility for a BSL-3 Experiment
<b>Checklist 3</b> (p. 42):	Daily Duties of the Operating Staff
<b>Checklist 4</b> (p. 43):	Response by BioCARS staff on Duty to Failure of the Facility HVAC or to Emergency
<b>Checklist 5</b> (p. 44):	Facility Cleanup after the BSL-3 Experiment

## **SOP 101: Responsibilities**

Date: July 2008

Purpose: Defines responsibilities of users, BioCARS staff and APS/ANL safety personnel for conducting safely BSL-3 experiments at the BioCARS facility.

For: BioCARS users and staff

### **1. Responsibilities of users and their home institution**

- 1.1** Identify the hazardous material and provide the MSDS and other relevant information related to the material.
- 1.2** Provide pertinent regulations used by the home institution, including evidence that each user has been authorized to work with the biohazardous agent at the user's home institution.
- 1.3** Provide certification of proper training and authorization for work with the BSL-2 agent at the home institution for all experimenters.
- 1.4** Identify transportation permit and shipping requirements and ensure proper shipping/transportation of the material to and from the BioCARS facility. Users are advised to contact the Hazardous Materials transportation specialists and Biosafety officer at their home institution regarding the compliance with transportation requirements. This includes USDA permits for interstate transport of infectious agents that affect agriculturally important animals and crops.
- 1.5** Comply with all regulations including this SOP and follow instructions given by BioCARS and APS/ANL staff.
- 1.6** Identify 2 users from the group as emergency contacts.
- 1.7** All users working with the BSL-3 agents must affirm that they are experienced and skilled in techniques and procedures used for work with such agents by providing an authorization by the home institution to work with the agent (see SOP 104). Any observation of improper handling of BSL-3 samples has to be reported to the spokesperson for the user group as well as to the BioCARS Biosafety Coordinator and it may result in denial of sample handling by the user in question based on the ANL stop work policy.

### **2. Responsibilities of the BioCARS Biosafety Coordinator**

- 2.1** Accept responsibility for an experiment identified as BSL-3.
- 2.2** Inform ANL IBC chairman, APS Biosafety Officer, APS User Safety Officer and APS floor Coordinator on duty. Floor coordinator will notify APS/ANL staff and prepare call-lists for incident response teams (**SOP 106, Appendix F**).



- 2.3 Obtain all relevant information from the PI of the user group and from user home institution. Initiate and coordinate the experiment approval process with the ANL IBC and the APS.
- 2.4 Gain sufficient knowledge about the nature of the hazard to make a judgment whether this SOP can be used to mitigate the hazard.
- 2.5 Send the BioCARS BSL-3 SOP to users.
- 2.6 Collect certifications of training/authorization from users and pass them to the ANL IBC.
- 2.7 Designate a particular BioCARS station for the BSL-3 experiment and supervise the appropriate set-up of the HVAC system (**SOP 106**).
- 2.8 Arrange for VHP decontamination of the facility (if decontamination is needed in case of an incident make a stand-by arrangement; ANL IBC might determine that an end-of-experiment decontamination is necessary even in the absence of an incident; **SOP 113**).
- 2.9 Supervise the preparation of the facility for the BSL-3 experiment and return of the facility to normal operation. Supervise VHP decontamination of the facility when required (**SOP 106, SOP 112, SOP 113**).

### 3. Responsibilities of the BioCARS staff contact for the experiment

- 3.1 Coordinate communication with users regarding their preparation for the experiment after experiment is approved by the IBC.
- 3.2 Inform in advance any non-BSL-3 user groups approved to conduct experiments in the BioCARS facility during the BSL-3 experiment about the BSL-3 experiment and its associated hazards.
- 3.3 With users, IBC and APS representatives, participate in receiving user samples, inspecting of the packaging and logging of samples (**SOP 103**).
- 3.4 Ensure that all BioCARS staff who are on the list of authorized personnel for the access to the facility during the BSL-3 experiment have read and signed this SOP before the experiment (template signature sheet is at the end of the SOP, before Appendix A) and are certified and trained for use of respirators.
- 3.5 Provide BioCARS-specific BSL-3 training to all visiting scientists participating in the BSL-3 experiment (**SOP 104**). All visiting scientists have to sign the **Checklist 1** each time they visit.
- 3.6 Prepare the facility for the BSL-3 experiment (**SOP 106**) according to the **Checklist 2**.
- 3.7 Return the facility to regular operation (**SOP 112**) by using **Checklist 5**.
- 3.8 BioCARS staff contact is listed as an emergency contact. In case of personnel exposure to a BSL-3 agent, staff contact will supervise the response together with the user designated as an on-site emergency contact and the APS Floor Coordinator (**SOP 108**).

### 4. Responsibilities of the BioCARS operating staff

- 4.1 Ensure that the facility is working as designed and certified per **Checklist 3** times per day.

- 4.2 If other, non-BSL-3 user groups are approved to conduct experiments in the BioCARS facility during the BSL-3 experiment, inform the users about the nature of the BSL-3 biohazard, about entry and exit rules, and about emergency and evacuation procedures.
  - 4.3 Participate in the response to overt or potential personnel exposure to BSL-3 agent (**SOP 108**) and in the response to the failure of facility HVAC and other equipment (**SOP 109**).
  - 4.4 Evacuate the facility according to **Checklist 4** during an emergency (**SOP 110**).
- 5. Responsibilities of the APS Floor Coordinator on duty**
- 5.1 Notify APS/ANL staff about the upcoming BSL-3 experiment. Prepare call-lists for appropriate incident response teams (**SOP 106, Appendix F**).
  - 5.2 Participate in verification of the preparation of the facility for the BSL-3 experiment (**SOP 106**).
  - 5.3 Participate in the response to overt or potential personnel exposure to BSL-3 agent (**SOP 108**) and in the response to the failure of facility HVAC and other equipment (**SOP 109**).
  - 5.4 In case of emergency evacuate the facility according to **Checklist 4**.
- 6. Responsibilities of the BioCARS PI**
- 6.1 Approve the BioCARS BSL-3 SOP.
  - 6.2 Decide on Medical Surveillance Plan for BioCARS staff.
  - 6.3 Decide on mandatory vaccination for BioCARS staff when vaccine is available.
  - 6.4 Exclude BioCARS personnel from the BSL-3 area if medical requirements are not met.
- 7. Responsibilities of the ANL IBC**
- 7.1 Approve the BioCARS BSL-3 SOP.
  - 7.2 Approve a specific BSL-3 agent for use in the BioCARS facility based on information provided by users via the ANL IBC registration form and BioCARS BSL-3 questionnaire (in case of virus samples), as well as additional documentation provided by users.
  - 7.3 Approve specific experimental procedures for work with a particular BSL-3 agent.
  - 7.4 Participate in risk-based determination of the need for VHP decontamination of the BioCARS facility.
- 8. Responsibilities of the APS**
- 8.1 Approve the BioCARS BSL-3 SOP.
  - 8.2 Approve a specific BSL-3 agent for use in the BioCARS facility based on user response to the BSL-3 questionnaire and provided documentation.

**8.3** Participate in risk-based determination of the need for VHP decontamination of the BioCARS facility.

**9. Responsibilities of the CARS Safety Committee**

Approve the BioCARS BSL-3 SOP.

## **SOP 102: Approval of BSL-3 Experiments**

Date: July 2008

Purpose: Establishes the process of approval for a particular BSL-3 experiment to be conducted at the BioCARS facility.

For: BioCARS users and staff

### **1. Application for beamtime**

- 1.1. Application for beamtime (proposal) is submitted via regular APS proposal submission route. Classification of the sample as BSL-3 has to be stated.
- 1.2. At least 2 months advanced notification of BioCARS/APS/ANL regarding the upcoming BSL-3 experiment is essential to assure proper preparation.
- 1.3. The BioCARS staff in charge of scheduling will make the initial contact with users. Basic information will be obtained regarding the sample, experiment and desired time for the experiment. This basic information is passed to the BioCARS Biosafety Coordinator.
- 1.4. The BioCARS Biosafety Coordinator informs APS Biosafety Officer about the upcoming BSL-3 experiment. APS Biosafety Officer informs all necessary APS/ANL personnel.

### **2. Approval process**

- 2.1 BSL-3 SOP (which includes a BioCARS Questionnaire in the Appendix C) and ANL Registration Form For Pathogens, Cells, Tissues and OPIM are sent to users by the BioCARS Biosafety Coordinator. Both the BioCARS Questionnaire (for virus samples) and ANL Registration Form must be filled out as soon as possible.
- 2.2 Each user is required to be trained and authorized for work with the BSL-3 agent by the IBC at their home institution.
- 2.3 Each user needs to sign the Risk Factors Form (Appendix D).
- 2.4 Based on sample and authorization questionnaires, the ANL IBC either requests additional information or approves the experiment and makes decisions regarding:
  - a) authorized personnel
  - b) PPE for the experiment
  - c) [post-experiment VHP decontamination of the BioCARS facility](#)

## **SOP 103: Shipping and Receiving of BSL-3 Samples**

Date: May 2007

Purpose: Summarizes ANL, APS and BioCARS acceptable procedures for shipping BSL-3 user samples from home institution to BioCARS, for receiving samples at BioCARS, for sample logging in/out, and, if necessary, for shipping samples back from BioCARS to user home institution.

For: BioCARS users and staff

### **1. Shipping user BSL-3 samples from home institution to BioCARS**

- 1.1** Users are responsible to determine if possession and transportation permits are required for the BSL-3 agent they are working with. Users are responsible to apply for and obtain the permits when required.
- 1.2** The home institution must provide to BioCARS/ANL approved and university-signed transfer permit if required at least ten business days prior to delivery of the material to BioCARS.
- 1.3** For all purposes, including obtaining appropriate permits when necessary, the users are considered in possession of the BSL-3 samples throughout the transportation and during the BSL-3 experiments at BioCARS.
- 1.4** Users shall be listed as **both the shippers and receivers** of the samples. For both permit application (when necessary) and shipping of the samples, the following address shall be used for **the receiver** when shipping samples from home institution to BioCARS facility:

User Name  
Building 434B, Sector 14  
9700 South Cass Avenue  
Argonne, IL 60439

- 1.5** Identification of the user home institution as both the shipper and receiver is acceptable under the following conditions: (a) the home institution identifies the BioCARS facility at the APS/ANL as a new/additional location for research on any applicable permits, (b) the home institution is the sole user of the material while it is on the ANL site, (c) the home institution is legally responsible for control and possession of the material while it is at the ANL site, and (d) the home institution initiates and is legally responsible for transport of the material in accordance with all applicable regulations and requirements.
- 1.6** Since users are shippers, they are directly responsible for the correct and compliant transport by air or ground both (a) from the home institution to BioCARS facility and (b) from BioCARS facility to home institution.
- 1.7** If users are transporting the BSL-3 samples themselves, they must provide an estimated time of arrival and the names of the person(s) who will be the

custodian of the package. Upon entering the ANL, users have to drive directly to the BioCARS facility (no intermediate stops on the ANL campus).

- 1.8 Upon arrival of the user's package at the BioCARS facility and before the package is transferred from the vehicle that delivered it, the APS and BioCARS representatives, together with users, will visually inspect the exterior of the packages that contain infectious material. If there is any concern about the integrity of the package, package will be left in the vehicle and 911 will be dialed.
- 1.9 The primary purpose of the inspection is to assure that the package has no readily visible cause for concern (e.g., damaged, partially open, leaking, etc.) The inspection is NOT intended to verify compliance with federal regulations with regard to documentation, external labeling, and internal packaging. The users are solely responsible for conformance with applicable federal regulations. A user representative will be prepared to provide to the APS representative for the APS file:
  - a) an MSDS or equivalent
  - b) letter from the institution's hazardous materials transportation specialist that assures packaging conforms with applicable state and federal requirements and was performed or supervised by a qualified person
  - c) copies of any transportation permits by the USDA or other agencies
  - d) copy of the shipper's declaration

## 2. Receiving user BSL-3 samples

- 2.1 Prior to opening, the package with the BSL-3 samples is stored in the BSC room. The room is locked before the 24h attendance goes into effect. Emergency access to the BioCARS facility key is available through the APS Floor Coordinator at LOM 434 (the second facility key is kept by the BioCARS Biosafety Coordinator for the duration of the experiment).
- 2.2 While the material is at BioCARS, on the ANL site, the material remains property of the user home institution.
- 2.3 The package will be opened in the BSC and inspected by users in the presence of the BioCARS staff contact for the experiment. If there are any concerns about the integrity of the samples (broken capillaries, for example), leave the containment area and inform the BioCARS Biosafety Coordinator and the APS Floor Coordinator. Incident response team will be consulted regarding the further action (**Appendix F**).
- 2.4 A log-in/log-out process is established for all infectious materials that users receive at BioCARS. The log-in information includes the number of containers/wells/crystals. The log-in/log-out form is in the **Appendix E**. BioCARS staff contact for the experiment makes sure that the log-in form is filled out by users and the form included into the BSL-3 Procedure Logbook.
- 2.5 Unpacked samples must be stored in the Biosafety Cabinet.

**3. Shipping user BSL-3 samples from BioCARS to home institution**

- 3.1** As a part of the log-out process, BioCARS staff contact makes sure that users fill out the Log-out form (**Appendix E**) and certify that all BSL-3 material has been rendered non-viable or has been packaged for transport/shipping to home institution.
- 3.2** For the BSL-3 samples where a special permit requires that the owner remains in possession of the material at all times, the users will arrange for transport of the BSL-3 samples back to the home institution. In this case, the user-provided shipping papers will identify the users as both the shipper and receiver. The following address shall be used for the **shipper** when shipping such samples from BioCARS facility to home institution:

User Name  
Building 434B, Sector 14  
9700 South Cass Avenue  
Argonne, IL 60439

- 3.3** For all other BSL-3 samples, the shipping of samples from BioCARS to the home institution has to be done via ANL/HazMat Shipping/Receiving. ANL in this case is a shipper and, based on information from users regarding the samples, assures compliance with state and federal regulations for marking, package certification and labeling, and shipping paper documentation,

## **SOP 104: User Training and Certification**

Date: June 2005

Purpose: Establishes training requirements and procedures for BSL-3 users of BioCARS facility.

For: BioCARS users and staff

### **1. Training**

- 1.1** BioCARS staff members that are given access to the BSL-3 area during a BSL-3 experiment will be trained at the ANL on basic practices of work with BSL-3 agents (although they will not be involved in direct work with the BSL-3 agent).
- 1.2** BioCARS staff members that are given access to the BSL-3 area during a BSL-3 experiment will have walk-through training by the BioCARS Biosafety Coordinator before the BSL-3 experiment.
- 1.3** All users involved in the BSL-3 experiment at BioCARS have to be authorized by the home institution IBC for work with the same agent at the home institution (**SOP 102**).
- 1.4** All users working with the BSL-3 agents must affirm that they are experienced and skilled in techniques and procedures used for work with such agents, including the use of Biosafety Cabinets. Any observation of improper handling of BSL-3 samples has to be reported to the spokesperson for the user group as well as to the BioCARS Biosafety Coordinator. This may result in denial of sample handling by the user in question based on the ANL stop work policy.
- 1.5** This SOP is sent to users well ahead of the start of their experiment.
- 1.6** All users are also given a copy of this SOP by their BioCARS staff contact upon arrival. They are instructed to read it carefully to refresh their knowledge about the facility and procedures. The users are given the opportunity to ask any questions and discuss any issues related the SOP material.
- 1.7** The BioCARS staff contact will tour the facility with users and identify the location of the equipment, PPE and other safety items.
- 1.8** The BioCARS staff contact will also go through **Checklist 1** with users and explain:
  - a) gowning/de-gowning protocols
  - b) procedure for transferring samples to the experimental station
  - c) [when respirator use is required](#)
  - d) proper waste disposal
  - e) response to autoinoculation
  - f) response to alarms due to HVAC problems
  - g) response to other emergencies



**2. Certification**

At the end of the training, each user will sign the **Checklist 1** to indicate that they have received the facility-specific training for handling the BSL-3 materials at BioCARS.

## **SOP 105: Protective Clothing and Other Personal Protective Equipment (PPE)**

Date: July 2008

Purpose: Establish protective clothing and respirator needs and procedures during BSL-3 experiments

For: BioCARS users and staff

### **1. Users**

- 1.1** Users are required to wear disposable tyvek overalls (with integral shoe covers) over street clothing while in the containment area, with the front completely closed. Overalls must be worn at all times while in the containment area. Users have to remove overalls aseptically whenever exiting the containment area.
- 1.2** Used tyvek overalls will be discarded every time prior to exiting the containment area (see 1.4). Clean tyvek overalls will be used when re-entering.
- 1.3** Sequence for taking off gloves and tyvek overalls before leaving the facility: remove outer gloves aseptically, remove the overalls, remove the inner gloves, use hand sanitizer before leaving the containment area.
- 1.4** Used tyvek overalls will be disposed in the biohazard waste bags in the BioCARS corridor (see BioCARS Facility Layout in Appendix A) every time when exiting containment area.
- 1.5** Two pairs of disposable gloves must be worn while handling the BSL-3 samples. Wearing nitrile gloves as a second pair of gloves over powderless latex gloves is recommended for ease of use. Gloves must be worn over the sleeves of the overalls. Jewelry (rings, bracelets) and watches will be removed during the BSL-3 experiments as they affect the integrity of the gloves. Users must check the integrity of gloves before use (e.g. by visual inspection and inflating with air).
- 1.6** Outer gloves have to be aseptically removed (with a special care not to contaminate the inner gloves) after any manipulation of BSL-3 material.
- 1.7** Half-face respirators and goggles or PAPR respirators shall be worn if determined necessary when working with samples outside of the biosafety cabinet. ANL IBC will specify the criteria for respirator use prior to a specific BSL-3 experiment, based on the risk assessment for a particular BSL-3 agent. Users can bring their own respirators for which they have been fitted and trained at the home institution.
- 1.8** When not in use, respirators and goggles will be left in the control room or in the corridor, labeled with user names.
- 1.9** Respirators and goggles will be disinfected upon completion of the experiment. Users will bring their respirators back to home institution.

**1.10** Tyvek overalls used in the biocontainment area must not be taken outside of the containment area, except when properly packed for disposal at the end of an experiment.

**1.11** Contact lenses may be worn in the biocontainment area only if goggles are worn.

**1. BioCARS staff**

**2.1** While helping users with the BioCARS equipment during the BSL-3 experiments, BioCARS staff must wear tyvek overalls (over the street clothing) and one pair of gloves.

**2.2** Contact lenses are allowed.

**2.3** If present in the containment area during an activity that requires use of respirators by users, BioCARS staff must also wear respirators.

**2.4** BioCARS staff shall call users before entering the facility to inform themselves about user activities which will determine what PPE staff needs to wear.

**2.5** Protective clothing will be discarded within containment every time prior to exiting. Clean protective clothing will be used for re-entry.

**2.6** Gloves will be removed aseptically.

**3. Visitors and observers**

No visitors and observers are allowed in the facility during a BSL-3 experiment.

**4. Location of protective clothing and PPE**

Supply of clean tyvek overalls, gloves, eye wear and PAPR respirators is located in the computer room.

## **SOP 106: Preparation of the Facility for BSL-3 Operation**

Date: May 2007

Purpose: Establishes procedures for changing the operation mode from standard to BSL-3 mode at the BioCARS facility and for preparation of the designated area for the BSL-3 work (**Checklist 2**). Both are responsibility of the BioCARS staff contact for the BSL-3 experiment, supervised by the Biosafety Coordinator (**SOP 101**) and observed by the APS Floor Coordinator.

For: BioCARS staff and APS Floor Coordinator

1. Inform APS Biosafety Officer (who will inform all other necessary APS/ANL personnel).
2. Designate members of incident response teams with assistance from the APS and ANL representatives (**Appendix F**) for:
  - a) medical and other emergencies
  - b) failure of HVAC or other equipment (**SOP 109**)
  - c) overt or potential personnel exposure to biohazard (**SOP 108**)
  - d) **VHP leak during facility decontamination.**

A list of incident response team members will be prepared by the APS Floor Coordinator. APS Floor Coordinator will inform the incident response teams about the BSL-3 experiment. Specific aspects of incident response will be discussed by the appropriate team.

3. **BioCARS Biosafety Coordinator will arrange in advance for decontamination (with a decon service provider like BioQuell). Decontamination will be arranged on a stand-by basis even if the end-of-experiment decontamination is not required by the IBC for a particular BSL-3 agent. This way rapid access by the decon crew will be assured in case of an incident.**
4. **Prepare HVAC system and BSC**

For experiments that require BSL-3 containment, arrange with FC the check of the HVAC system operation by FSM, per instruction provided by APS.

BioCARS staff contact (trained and supervised by the BioCARS Biosafety Coordinator) and the APS Floor Coordinator verify the proper operation of the HVAC and BSC: follow **Checklist 0**.

5. **Prepare facility**

BioCARS staff will follow **Checklist 2** to prepare the facility for the BSL-3 experiment.

## **SOP 107: BSL-3 Practices and Procedures**

Date: July 2008

Purpose: Establish protocols for sample handling.

For: BioCARS users and staff

### **1. General**

- 1.1** 24h attendance is required during the BSL-3 experiments. One user and one BioCARS staff member have to be in the BioCARS area (containment area, computer room, kitchen area or BioCARS LOM).
- 1.2** Store properly packed BSL-3 samples in the BSC room, next to the BSC. A dewar with frozen, contained BSL-3 samples can also be stored in the control room during the active experiment for the ease of transferring of frozen samples to the experimental hutch.
- 1.3** Store all unpacked samples in the Biosafety Cabinet for the duration of the experiment. The Biosafety cabinet is lined with the plastic-backed absorbent paper without obstructing the airflow in the cabinet.
- 1.4** For room temperature experiments, pre-mount crystals in quartz capillaries at the home institution whenever possible.
- 1.5** Use stainless steel dewars for work with frozen crystals whenever possible.
- 1.6** Safe microbiological practices must be applied when handling BSL-3 materials, as described in the CDC-NIH Biosafety in Microbiological and Biomedical Laboratories (a copy of the document is in the computer room) and in the **Checklist 1**.
- 1.7** **Biosafety Cabinet Work Practices and Procedures** must be followed (described in the **Appendix B**).
- 1.8** Protective clothing and other protective equipment must be worn as described in the **SOP 105**.
- 1.9** Outer gloves must be safely changed after any manipulation of the BSL-3 material, overt or suspected contact with the BSL-3 material or contact with contaminated items. To minimize the potential contamination of surfaces and equipment, do not touch any surfaces, door handles or the equipment with potentially contaminated gloves.
- 1.10** Replacing outer gloves:
  - a) Take off the outer gloves aseptically (taking special care not to contaminate the inner gloves).
  - b) Dispose used outer gloves in a biohazard waste container.
  - c) Put on a clean pair of outer gloves.
- 1.11** Wash hands (in the BSC room) or use clinical grade hand sanitizer whenever both pairs of gloves are changed or removed. Wash hands at the earliest opportunity after leaving the biocontainment area.
- 1.12** Do not remove from the Biosafety Cabinet items that are potentially contaminated until they have been surface decontaminated.

- 1.13** When exiting the containment area from the corridor to the computer room, do not open the door with gloved hands. Dispose gloves first and use the gel-based disinfectant (by the door) as a final hand cleaning step.

## **2. Sample handling:**

- 2.1** Room temperature crystals:
- 2.1.1** Crystal mounting must be done in the BSC.
  - 2.1.2** Use only quartz capillaries.
  - 2.1.3** No mouth pipetting is allowed.
  - 2.1.4** Use epoxy to seal the capillaries.
  - 2.1.5** Collect all waste from the crystal mounting process in the biohazard bags or biohazard sharp containers in the Biosafety Cabinet.
  - 2.1.6** Disinfect the capillary exterior. Use extreme caution not to break the capillary.
  - 2.1.7** Mount a capillary on a brass pin (use epoxy to glue it to the pin). Insert brass pin into a pin holder (Hampton Adjustable Crystal Mount).
  - 2.1.8** Prepare a capillary for transfer from the BSC to the diffractometer:
    - a) use the secondary container provided by BioCARS
    - b) place the pin holder with the capillary on the magnetic base in the secondary container
    - c) screw the cover of the secondary container over the capillary
  - 2.1.9** Disinfect the secondary container before taking it out of the Biosafety cabinet.
- 2.2** Pre-frozen crystals:
- 2.2.1** Prepare pre-frozen crystal for transport to the hutch (see 2.2.3 for alternative protocol):
    - a) place a small transfer dewar filled with liquid nitrogen in the BSC
    - b) pull out one cane from the user dewar; take out (using Hampton Vial Clamp) one vial with a crystal from the cane; put the vial into the transport dewar in the BSC.
    - c) Hampton Cryotong can be used (instead of the vial) to enclose the crystal for transport to the hutch.
  - 2.2.2** Transfer the small dewar with the vial or Cryotong to the hutch by hand or using a small BioCARS provided cart. Make sure all preparations for sample mounting in the hutch are done (see 4 below) before transferring the small dewar.
  - 2.2.3** To reduce the risks associated with the transfer of frozen crystals from the BSC room to the hutch, **2.2.1** can be done in the control room. The user dewar with frozen, contained crystals will be stored in that case in the control room (only during the active experiment period).

- 2.3 Freezing crystals:
    - 2.3.1 Preparation will be done in the BSC.
    - 2.3.2 Using standard cryo-tools (Such as Hampton CrystalCap with CryoLoop, CrystalWand, VialClamp) a crystal is flash frozen by plunging it into liquid nitrogen in a short dewar. Place frozen crystal either into a vial or a Hampton Cryotong.
    - 2.3.3 Transport the dewar with the vial or Cryotong to the hutch by hand or using a small BioCARS provided cart. Make sure all preparations for sample mounting in the hutch are done – see 4 below – before transporting the dewar.
  - 2.4 [Appropriate respirators and goggles must be worn while manipulating samples outside of the BSC.](#)
- 3. Handling spills in the Biosafety Cabinet**
- 3.1 A loss of crystal in the Biosafety Cabinet is not regarded as an incident and is handled as a spill.
  - 3.2 Small spills within the Biosafety Cabinet are handled immediately by disinfecting the affected area, folding and removing the contaminated absorbent paper toweling and placing it into a biohazard bag. Make sure to minimize the aerosol formation while removing the paper. Any splatter onto items within the cabinet, as well as the cabinet interior, shall be wiped immediately with a towel dampened with decontaminating solution.
- 4. Transferring a crystal from the Biosafety Cabinet room to the diffractometer:**
- 4.1 Prepare the diffractometer for sample mounting before the sample transfer:
    - a) move the beam stop and detector away to make enough space for maneuvers needed to place the capillary or frozen crystal on the magnetic base on the diffractometer
    - b) position the kappa angle of the diffractometer into appropriate position for crystal mounting
    - c) place and secure a tray with absorbent paper under the sample position and soak the paper with disinfectant
    - d) use trial loop without a crystal or trial capillary to adjust the goniometer z translation properly
  - 4.2 Only a single crystal at the time is transferred from the Biosafety Cabinet or the control room to the diffractometer.
  - 4.3 Crystal transfer is conducted under a two-person rule where one person transports the sample and another assists (opens doors, warns other users and prevents collisions and tripping, monitors sample mounting on the diffractometer).



- 4.4 Appropriate respirators and goggles, two pairs of gloves and tyvek overalls are all required to be worn while handling and transferring BSL-3 crystals.

**5. Mounting a crystal on the diffractometer:**

- 5.1 Make sure diffractometer is prepared as outlined above in 4.1.
- 5.2 Mount a crystal on the diffractometer:
  - a) When the crystal is in a screw-cap vial, loosen the magnetic cap before mounting the vial. Mount the vial on the magnet and remove the vial.
  - b) Mount magnetic cap vials or capillary-mounted crystals on the diffractometer magnet.
- 5.3 Examine immediately on the monitor in the hutch the magnified image of the loop/capillary with the crystal. Make sure capillary is intact in case of room temperature experiment. Make sure the loop contains the crystal when frozen samples are used. In case of lost crystals, follow the procedure described in **SOP 108**.

**6. After crystal mounting:**

- 6.1 Before touching any equipment, properly remove outer gloves and replace with clean gloves.
- 6.2 Remove the crystal transport tools and transport dewar from the hutch and return them to the BSC. Replace the outer gloves again.
- 6.3 Finish preparations inside the hutch (remove the tray, position the beam stop and the detector), search the hutch and close the hutch door. The rest of preparation that does not require access to the hutch shall be done with the hutch door closed.

**7. Removing a crystal from the diffractometer:**

- 7.1 Confirm that crystal (loop or capillary) is not lost before entering the hutch, by checking the crystal-viewing monitor in the control room.
- 7.2 With the same PPE used for crystal mounting, enter the hutch and move back the detector and the beam stop.
- 7.3 Remove the crystal and submerge it into a non-breakable jar containing disinfectant.
- 7.4 Properly remove outer gloves and replace with clean gloves.

## **SOP 108: Response to Overt or Potential Personnel Exposure to BSL-3 Agent**

Date: February 2007

Purpose: Establish procedures for a response to: (a) loss of crystal; (b) autoinoculation (cuts/sticks with sharps, splashes to mucous membranes). See also summary in the Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS**. VHP decontamination of the facility might be required if a BSL-3 crystal is lost, if determined necessary by the incident response team.

For: BioCARS users, staff and APS Floor Coordinator

### **1. Loss of crystal in the experimental station**

- 1.1 Respirators and all protective clothing must be kept on.
- 1.2 Immediately soak the absorbent paper with disinfectant.
- 1.3 Take off the outer gloves (dispose them in the biohazard bag).
- 1.4 Exit the hutch and CLOSE the hutch door.
- 1.5 Press the Biohazard Alarm button next to the control room entrance/exit door. This will activate the BioCARS PA system and inform the staff and other users in the facility about the biohazard condition (announcement: "Attention BioCARS staff, support requested").
- 1.6 Take off the tyvek suit (use the biohazard bag in the control room to dispose them).
- 1.7 Exit the control room and close the door.
- 1.8 Take the respirator and the gloves off just before exiting the BSL-3 area (before entering the computer room). While holding breath, exit the BSL-3 area.
- 1.9 Inform the BioCARS staff on duty about the incident.
- 1.10 BioCARS staff on duty will BioCARS staff will inform BioCARS Biosafety Coordinator and APS Floor Coordinator and follow steps listed in **Checklist 4**.
- 1.11 A conference will be convened by BioCARS staff contact, user emergency contact, BioCARS Biosafety coordinator, APS Floor Coordinator and others from APS/ANL on the incident response team. A clean-up procedure will be developed based on information provided by users involved in the loss of crystal.

### **2. Loss of crystal outside of the experimental station**

- 2.1 Respirator and all protective clothing must be kept on.
- 2.2 Immediately cover the area of the spill with absorbent paper, and apply disinfectant solution to the paper, beginning at the periphery and then to

the center. In general, the disinfectant-soaked paper shall be left undisturbed for 15 to 30 minutes, depending on the knowledge of susceptibility to the disinfectant.

- 2.3 Take off the outer gloves (dispose in a biohazard bag).
  - 2.4 OPEN the hutch door. Press the Biohazard Alarm button next to the control room entrance/exit door. This will activate the BioCARS PA system and inform the staff and other users in the facility about the biohazard condition (announcement: "Attention BioCARS staff, support requested").
  - 2.5 Take off the tyvek suit in the control room if the incident occurred in the corridor (use the biohazard bag in the control room to dispose them). Do the same in the corridor if the incident occurred in the control room (use the biohazard bag in the control room to dispose them)
  - 2.6 Follow steps 1.7 to 1.10 above. Exit via control room if incident occurred in the corridor. Exit via computer room if incident occurred in the control room.
3. **Autoinoculation** (cuts and sticks by sharps, splashes to mucous membranes):
- 3.1 Accompanying person or BioCARS staff dials 911.
  - 3.2 Remove contaminated gloves and squeeze vigorously if possible to allow the wound to bleed.
  - 3.3 Wash the wound with soap and water for 5min and apply sterile bandage if necessary (stored in the BSC room).
  - 3.4 For splashes to mucous membrane, rinse with large amounts of water at the sink in the BSC room. Eyes shall be irrigated for at least 5min using the eye wash station in the BSC room.
  - 3.5 Take off protective clothing and dispose it.
  - 3.6 Exit the BSL-3 area.
  - 3.7 BioCARS staff on duty informs BioCARS Biosafety Coordinator and the APS Floor Coordinator.

## **SOP 109: Response to the Failure of Facility HVAC and Other Equipment**

Date: June 2005

Purpose: Establish procedures in case of HVAC failure and possible loss of containment and failure of other critical equipment (such as BSC and crystal cryo-cooler).  
See also summary in the Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS**

For: BioCARS users, staff and APS Floor Coordinator

- 1. Loss of containment due to a failure of the facility main exhaust fan EF104**
  - 1.1** An alarm will sound and BioCARS PA notification system will be activated (**announcement: “Attention BioCARS staff, HVAC problem”**). BSC alarm will sound to indicate loss of negative pressure above the BSC and BSC will turn off.
  - 1.2** Stop all work. Keep all PPE on.
  - 1.3** If working with open samples, place all open samples at hand into primary and secondary containers or dispose them into a closest sample waste container with disinfectant.
  - 1.4** Take off and dispose outer gloves. Take off and dispose protective clothing. Take off inner gloves and wash or disinfect hands.
  - 1.5** If wearing respirator/goggles, take these off just before leaving the containment area.
  - 1.6** Exit the containment area and inform the BioCARS staff on duty about the incident.
  - 1.7** BioCARS staff on duty will follow steps listed in **Checklist 4**.
  - 1.8** A conference will be convened by BioCARS staff contact, user emergency contact, BioCARS Biosafety Coordinator and APS Floor Coordinator. A response procedure will be developed.
  
- 2. BSC failure: Improper down flow velocity**
  - 2.1** A local BSC alarm will sound. The exhaust from the BSC is maintained as long as the EF104 is operating.
  - 2.2** If working in the BSC, follow **1.2** to **1.6**.
  - 2.3** If anywhere else in the facility, exit the BSL-3 area.
  - 2.4** Inform the BioCARS staff on duty about the incident.
  - 2.5** BioCARS staff: follow **1.7-1.8**.

**3. Failure of the crystal cryo-cooler**

- 3.1** Failure of the crystal cryo-cooler and loss of the cold gas stream will cause warming up and drying out of the crystal that is mounted on the diffractometer. This will be evident by looking at the crystal-viewing monitor and checking the sample temperature (monitored outside of the hutch). If this occurs during the data collection, stop the data collection. Do not open the hutch door.
- 3.2** If this occurs in the process of mounting a crystal on the diffractometer, follow **1.2** to **1.6**.
- 3.3** BioCARS staff: follow **1.7-1.8**.

## **SOP 110: Medical and Other Emergencies During the BSL-3 Experiments**

Date: May 2007

Purpose: Establish safe procedures for Medical and Facility Emergencies during the BSL-3 experiments.  
See also summary in Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS.**

For: BioCARS users, staff, APS Floor Coordinator and APS/ANL emergency response team.

### **1. Medical Emergencies not related to biohazard exposure**

BioCARS staff or user dials 911 and informs APS Floor Coordinator.  
Follow instructions by the 911 personnel and wait for the medical emergency personnel to arrive.

### **2. Fire emergency**

- 2.1 Leave the facility.
- 2.2 Call 911 and contact APS Floor Coordinator.
- 2.3 Gather outside of the 434B LOM, at the parking lot.
- 2.4 Wait for the instructions from the APS Floor Coordinator before returning to the facility.

### **3. Other emergencies outside of the BSL-3 facility (e.g. Severe Weather Alert)**

- 3.1 Stop all work.
- 3.2 Place all open samples at hand into primary and secondary containers or dispose them into a closest sample waste container with disinfectant.
- 3.3 Take off gloves and wash or disinfect hands with hand sanitizer.
- 3.4 Take off and dispose protective clothing in the BSC room. Wash hands.
- 3.5 Users and BioCARS staff on duty will collect and double bag all biohazard waste and place the waste in the BSC room.
- 3.6 Leave the containment area.
- 3.7 BioCARS staff will lock BSC room. BioCARS staff will follow steps listed in **Checklist 4.**
- 3.8 **Steps 3.1 to 3.7 shall normally be completed. However, protection of human life and health has priority over full assurance that personal decontamination and removal of protective clothing has been thoroughly completed.**

## **SOP 111: BSL-3 Waste Disposal**

Date: July 2008

Purpose: Establish procedures for safe BSL-3 waste disposal.

For: BioCARS users, staff and APS Floor Coordinator

### **1. BSL-3 waste pickup by ANL Waste Management**

- 1.1** Disposable contaminated materials are collected in biohazard bags, which are collected at the end of the experiment, sealed and placed in the BSC room.
- 1.2** The bags are surface decontaminated and placed in secondary bags.
- 1.3** Double-bagged waste is placed into shipping boxes provided by ANL Waste Management.
- 1.4** BioCARS staff contact fills out the Waste Management form and schedules the pick-up. Advanced notification (two weeks prior to the experiment) of the ANL Waste Management regarding the approximate pick-up time is needed to assure timely waste pickup.
- 1.5** ANL Waste Management will pick up the shipping boxes with the doubly-bagged waste as soon as possible after the BSL-3 experiment, after the routine decontamination of the facility (**Checklist 5**, part 1) [and before the VHP decontamination](#).

### **2. Autoclaving**

- 2.1** A portable autoclave is available for autoclaving the non-disposable tools and materials.
- 2.2** The autoclave is located in the biosafety cabinet room.
- 2.3** Trained BioCARS staff will conduct autoclaving. Instructions for autoclaving will be posted by the autoclave. Autoclaving for 60min at 121-132°C is required.
- 2.4** BioCARS staff verifies the effectiveness of the autoclaving monthly (verification records are kept in the Maintenance Logbook).

## **SOP 112: End of Experiment Procedures**

Date: March 2005

Purpose: Return the facility to standard (no biocontainment) operation.

For: BioCARS users and staff

- 1.** Follow **Checklist 5, Part I**
- 2.** Follow **SOP 113** if facility VHP decontamination is declared by the ANL IBC necessary at the end of the experiment.
- 3.** Follow **Checklist 5, Part II.**



## **SOP 113: Facility VHP Decontamination**

Date: June 2005

Purpose: Establish procedures for the VHP decontamination of the facility.  
See also summary in Appendix F **Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS**

For: BioCARS staff and APS Floor Coordinator

### **1. VHP Decontamination**

- 1.1 VHP decontamination of the facility will be done after an incident (1.7 p.8) if determined necessary by the incident response team.
- 1.2 The end-of-experiment VHP decontamination of the facility will be done when decided to be necessary by the ANL IBC, after the review and assessment of hazards for a particular BSL-3 agent.

### **2. VHP Decontamination procedure**

- 2.1 Decontamination will be done as a Room Bio-decontamination Service (RBDS) by, for example BioQuell (<https://www.bioquell.com/>). Decontamination procedure has to be approved by the ANL EQO-IH.
- 2.2 RBDS utilizes low temperature, “residue-free” bio-decontamination technology, where free radicals generated from hydrogen peroxide vapor bio-deactivate micro-organisms. Once the bio-deactivation is accomplished, the hydrogen peroxide is catalytically converted to water and oxygen.
- 2.3 The decontamination service provider will supply needed equipment as well as highly trained RBDS engineers. The service will be optimized and customized to the conditions and topology of the rooms in the BioCARS facility as well as to the health and safety requirements.
- 2.4 The service provider will also conduct a verification of bio-decontamination using bio-indicators. Full verification typically requires 7 days.
- 2.5 BioCARS and APS/PFS personnel will assist to service engineers if needed with the use of the BioCARS HVAC and with sealing of the facility. The HVAC will be turned off.
- 2.6 Possible leaks of the hydrogen peroxide during the decontamination will be actively monitored by service engineers.
- 2.7 If a leak is detected: BioQuell personnel will shut off the VHP generating equipment and the BioCARS facility exhaust will be turned ON.

## **SOP 114: Safety equipment maintenance and testing**

Date: July 2008

Purpose: Establish procedures for safety equipment maintenance, testing and certification.

For: BioCARS staff

**Important:** Record all maintenance in the BioCARS BSL-2/3 Maintenance Logbook.

### **1. Five BioCARS AHUs located on the facility roof**

*Before each APS run (three times per year):*

- 1.1 Check AHUs for excessive vibrations or any other obvious problems.
- 1.2 Check the integrity of belts and replace if necessary.
- 1.3 Check chilled water pipes and water mixing valves for leakage.

*Every six months:*

- 1.4 Check **On** and **Off** indicator lights on the electrical box (mounted on the AHU) and replace any burnt-out bulbs.
- 1.5 Grease the two ball bearings for the fan shaft (Mobil Oil Corporation, Mobilith AW2 temp range -29°C to 163°C)
- 1.6 Replace fan drive belt (Part # Gates Truflex 5L530 rated for equipment of 3 to 17 HP use). Make sure that the belt is not adjusted too tightly and that the upper pulley is aligned with the lower pulley. (A slight squeak on the start up of an AHU is normal.)
- 1.7 Replace air filters, two for each AHU (McMaster-Carr Part # 2211K65 Antimicrobial Pleated Panel Filter). Pay strict attention to the air flow direction as air filters are directional.
- 1.8 On completion of the maintenance tasks, reinstall the fan belt and pulley covers.
- 1.9 Clean externally (vacuum up any belt dust etc.)

### **2. Differential pressure monitors in BioCARS corridor and BSC room**

- 2.1 Check pressure differentials before each BSL-2/3 experiment and compare with the list of values in the “BioCARS BSL-2/3 Maintenance Logbook”. Report any significant discrepancies to the BioCARS Biosafety Coordinator.

- 2.2 Do **NOT** adjust any switches or adjustments screws on the units before talking to the BioCARS Biosafety coordinator. The status switch shall be on “-“ for all units but computer room unit, which shall be “+”.
- 2.3 Do not attempt to re-calibrate the units. If you suspect the calibration problem (as evidenced by comparing the unit reading with the results of annual pressure differential measurements), send the unit to the manufacturer for re-calibration.

### 3. **Biohazard alarms**

Biohazard alarms have to be tested before each BSL-2/3 experiment (appropriated pressure monitors have to be on).

### 4. **Biosafety Cabinet**

Arrange for annual re-certification of the Biosafety Cabinet with Salus ([www.salustech.net](http://www.salustech.net)).

### 5. **HEPA filter and pre-filter**

- 5.1 Annual testing is routinely provided by the ANL IH personnel.
- 5.2 Before each BSL-2/3 experiment check the pressure differential for the pre-filter.
- 5.3 Request that ANL IH change the filter if necessary (clean filter pressure differential < 0.2” H<sub>2</sub>O, alarm goes off at 0.8” but this does not affect any hardware – just a reminder that pre-filter is getting clogged).

### 6. **PAPRs respirators**

- 6.1 Every month recharge batteries for all units.
- 6.2 Before each BSL-2/3 experiment run all PAPR units for 8h and recharge batteries.
- 6.3 Test for proper operation (instructions are in the BioCARS BSL-2/3 Maintenance Logbook).

### 7. **Autoclave**

- 7.1 BioCARS staff verifies the effectiveness of the autoclaving monthly (verification records are kept in the Maintenance Logbook).

**Checklist 0: Preparation of the HVAC and BSC for BSL-3 operation**

**(BioCARS staff contact and APS Floor Coordinator)**

- Check the status of the AHUs (check the integrity of belts and replace if necessary).
- Test the Biohazard Alarm in the designated BSL-3 control room and the Biosafety Cabinet room.

**To assure the biocontainment of the designated area:**

- Arrange with FC the check of the HVAC system operation by FSM, per instructions provided by APS.
- seal the doors to the non-BSL-2/3 control areas on both sides;
- arrange for an ANL safety specialist (or APS personnel certified by the ANL safety specialist) to perform documented leak check (smoke test) across the sealed doors and for verification of the directional flows in the biocontainment area and across all doors at the perimeter of the facility.

**In the presence of an APS floor coordinator:**

- Switch the HVAC mode from standard to biocontainment mode using the HVAC JC-Control Box (located on the BioCARS facility roof).
- Selects the proper experimental station for the BSL-2/3 experiment using the HVAC JC-Control Box.
- Make sure the door to the BioCARS facility roof (where HVAC JC-Control Box is located) is locked.

Verify proper HVAC operation for the biocontainment mode using the EPICS Status Screen and other indicators:

- EF104 running (status ON)
- The hutch door switch (modifies the exhaust flow rate from the hutch) and dampers of the AHU for the BSL-2/3 control room operate properly:
  - a) Hutch door open (door switch deactivated):  
AHU make up air damper fully open (no air recirculation in the control room); exhaust rate from the BSL-2/3 hutch is in excess of 950cfm
  - b) Hutch door closed (door switch activated):  
AHU make up air damper fully closed (air recirculation is on in the control room); exhaust rate from the BSL-2/3 hutch is <400cfm

**Verify proper operation of BSC (if used during the experiment):**

- Verify that the Biosafety cabinet is on and operating properly: display on the BSC showing down flow rate ~ 60 fpm, exhaust rate ~730cfm.

**Verify operation of pressure monitors:**

- Pressure differential monitors in the corridor are turned on for the BSL-2/3 control room, computer room and the BSC room (otherwise the Biohazard Alarm buttons in the control rooms/BSC room will not work).
- Pressure monitors for non-BSL-2/3 control room are off (rooms are sealed so pressure differentials are 0; alarms will sound continuously if turned ON; DO NOT adjust alarm set points since any station may be used in the future for a BSL-2/3 experiment).

Signature of the BioCARS staff contact for the experiment and APS Floor Coordinator:

BioCARS staff: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

APS Floor Coordinator: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**Checklist 1: User Training for BSL-3 Experiments at BioCARS**

**Material :** \_\_\_\_\_

**Disinfectant:** \_\_\_\_\_

Does the material pose a Human Health Hazard (Yes / No)

Only Authorized Personnel allowed in the BSL-3 area (designated hutch, adjacent control room, corridor and Biosafety Cabinet room).

The BSL-3 experiments have to be attended by users (and a BioCARS staff member) at all times (24h). Users do not have to be in the containment area all the time but have to be in the BioCARS area (computer room, BioCARS LOM, kitchen area).

Sample storage and manipulation has to be done in the BSC room. In case of frozen crystals, a dewar with contained, frozen crystals can be stored in the control room during the experiment for easier transfer of a crystal to the hutch.

Two-person rule is in effect for sample transfer from the BSC room to the diffractometer.

Absolutely no food or drinks are allowed. No application of cosmetics while in the biocontainment area.

Collect all waste in biohazard bags and all sharps in biohazard sharps containers.

Collect “dead” crystals in disinfectant (non-breakable) jar (with a lid).

Apply safe microbiological practices as described in the CDC-NIH Biosafety in Microbiological and Biomedical Laboratories (a copy of the document is in the computer room).

Wear a tyvek suit and gloves when handling BSL-3 material. In addition, wear respirators and goggles when working with samples outside of the BSC. Wash hands (next to the Biosafety Cabinet) or use hand sanitizer properly (at the hutch exit and control room exit) after handling the material, after removing gloves, and before leaving the facility.

No mouth-pipetting is allowed.

Avoid using glass pipettes whenever possible. Do not handle broken glass/sharps by hand.

Use of needles is forbidden, unless a special permission is obtained from ANL IBC.

Keep workplaces clean and disinfect twice a day and immediately after a spill.

Store all items not needed for the experiment outside the containment BSL-3 area.

In case of a loss of crystal or spill, inoculation, facility HVAC/equipment failure, or other emergency follow the list of action listed in SOP 108, 109, 110.

**Adjustment of the exhaust rate from the hutch is slow.** To reduce the potential turbulence of the cold gas stream from the cryo-cooler, when closing the manual hutch door in 14-BM-C:

- close the door partially (to engage the door switch for exhaust adjustment)
- wait until the exhaust rate from the hutch drops to ~500cfm (monitor on the EPICS screen in the control room) before closing the door completely (few minutes).

14-ID-B door is automatic so just close the hutch door as usually.

**User home institution:**

**List of experimenters:**

---

---

---

---

---

**I have read and understood this SOP:**

Instructor:

Experimenters:

Name: \_\_\_\_\_ Date: \_\_\_\_\_

Signatures:

---

---

---

---

---

## Checklist 2: Preparation of the Facility for a BSL-3 Experiment

### 1. Biosafety Cabinet Room

Is BSC used? :  Yes  No

If Yes is checked:

Biosafety Cabinet is running with the proper down flow rate (~60 lfpm) and exhaust rate (~720-750cfm) for at least 20 minutes.

Down flow rate:

Exhaust rate:

Cabinet interior is clean. Plastic-backed absorbent paper is in place (not obstructing the air flow).

Biohazard bags and sharp containers are in the cabinet (not obstructing the air flow).

**Other preparation:**

A larger biohazard container for disposal of overalls and gloves is in place.

BioCARS portable autoclave is in place.

"NO USE" sign is posted on the Cold Room door. Cold Room lights/fan are turned OFF. Fan opening is sealed.

Post at the door:

Copy of **Checklist 1**

ESAF

Biohazard sign

### 2. BSL-3 Hutch

Floor is mopped and dust removed as much as possible. All unnecessary items are removed.

User cart is in place if needed.

Plastic-backed absorbent paper covering as much of the equipment as possible is in place.

Biohazard bags and sharp containers are in place

A container with disinfecting solution is in place and labeled

A non-breakable container with a lid for disposing used crystals is in place and labeled.

Tray with absorbent paper is placed under sample. Remind users to soak paper often with bleach.

Hand sanitizer dispenser is in place at the exit of the hutch.

Set up a video camera or an alternative means to monitor the temperature of the cryo-cooler from outside of the hutch.

If ID-B hutch is used, seal the laser port on the ID-B hutch ceiling.



**3. BSL-3 Control Room**

- Floor is mopped and dust removed as much as possible. All unnecessary items are removed.
- Portable sink and eye wash from the APS are in place and functional.
- Control room emergency exit door is locked from outside. Biohazard sign is posted.
- Sign posted on emergency exit door (inside):  
“Stop! Emergency Exit Only!”
- All regular refuse and sharp containers are removed. Biohazard bags and biohazard sharp containers are labeled and in place
- A larger biohazard container for disposal of overalls and gloves is in place.
- A container with disinfecting solution is in place and labeled.
- Hand sanitizer dispenser is in place at the exit of the control room.
- Computer keyboards are covered by a plastic protector or replaced by washable keyboards. Computer mice are covered by a plastic protector.
- Copy of ESAF is posted on the door.

**4. Corridor**

- Access to the corridor from other, non-BSL-3 control rooms blocked and sign posted not to enter the corridor
- Hand sanitizer dispenser in place at the exit of the corridor
- A larger biohazard container for disposal of overalls and gloves in place

**5. Computer room****Post at the door to the BioCARS corridor:**

- ESAF
  - Biohazard sign
  - List of authorized personnel
  - List of user and BioCARS staff emergency contacts and phone numbers
- The door to the corridor is chained off (chain is far enough to permit safe opening of the door and exit from the corridor).
  - A supply of PPE is in place.
  - Copy of **Biosafety in Microbiological and Biomedical Laboratories** and BioCARS SOP is available.

**6. Post on the main entrance of the facility (double door entrance from the APS experimental hall to the computer room)**

- Biohazard sign
- List of all authorized personnel
- List of user and BioCARS staff emergency contacts and phone numbers

**7. AHU panel (by the ID-B station)**

- Lock the AHU panel cover and keep the key in the office for the duration of the experiment.
- Post a “No Access” sign on the cover.

**8. Administrative**

- Inform neighboring Sectors 13 and 15 (directly or via CARS Safety Officer) about the BSL-3 experiment at BioCARS.
- Inform the custodian (directly or via APS Floor Coordinator) not to enter the facility for the duration of the experiment.
- Place the BSL-3 Procedure Logbook at the BioCARS User Administrator’s desk. The Procedure Logbook contains BioCARS sign-up sheet, User Sample Questionnaire, ESAF, MSDS for the BSL-3 agent, Sample Log-in/Log-out form and all checklists from this SOP.
- Take the facility key from the key box and keep in the office for the duration of the experiment. Return it to the key box only after the BSL-3 waste is picked up [and VHP decontamination \(if required\) is finished.](#)

Signature of the BioCARS staff contact for the experiment:

Name: \_\_\_\_\_  
Signature: \_\_\_\_\_ Date: \_\_\_\_\_

**Checklist 3: Shift Duties of BioCARS Operating Staff**

- If non-BSL-3 users are starting an experiment on a non-BSL-3 station, explain:
  - 1) emergency doors will be used to enter/exit their control room; the entrance/exit through corridor is not permitted
  - 2) evacuation procedure and route in case of an emergency
  - 3) Cold Room cannot be used

Check if the appropriate station is in selected as BSL-3 (EPICS Status screen).

Biosafety cabinet is operating properly:  
down flow rate (~60fpm):  fpm  
exhaust rate (~750cfm):  cfm

Exhaust Fan 104 is working properly (check Metasys EPICS screen on any BioCARS linux computer).

Record pressure differentials (**compare to BioCARS BSL-2/3 Maintenance Logbook**)

BSL-3 control room:   
Computer room:   
BSC cabinet room:

BSL-3 station emergency door is locked from the outside.

Biohazard sign, list of authorized personnel and emergency contacts are posted at the BioCARS facility door and at the entrance to the corridor.

Signature of the BioCARS operating staff member:

Name: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

**Checklist 4:**      **Response by BioCARS staff on duty to an incident (SOP 108), failure of the facility HVAC (SOP 109) or emergency outside of the BSL-3 facility (SOP 110)**

- In case of medical or other emergency dial 911 and provide details.**
- If present in the facility, non-BSL-3 users instructed to leave the facility immediately using the explained (see **Checklist 3**) exit path (emergency doors). BSL-3 users will follow **SOP 108, 109 or 110**.
- Emergency personnel – user and staff – contacted. BioCARS Biosafety coordinator contacted. APS Floor Coordinator contacted.
- Make sure all BSL-3 users are out by checking the by checking the BSL-3 control room through the glass window of the emergency exit from the APS experimental hall. Make sure all users are out. Check if the control room emergency exit door is locked.
- Tape off the entrance to the BSL-3 area (from the computer room) and post a sign (“Warning! Do not enter! Biohazardous condition.”)
- Make sure all non-BSL-3 users are out by checking the non-BSL-3 control rooms using emergency exits.
- All users are accounted for.

Signature of the BioCARS operating staff member:

Name: \_\_\_\_\_

Signature: \_\_\_\_\_ Date: \_\_\_\_\_ Time: \_\_\_\_\_

**Checklist 5: Facility Cleanup after the BSL-3 experiment**

**Part I: Before VHP Decontamination (if decontamination required)**

- Absorbent paper is removed from the BSC and the BSL-3 hutch. Fold paper carefully so that the potentially contaminated surface remains inside. Do not crumple/wad the paper.
- The BSC interior and all horizontal and vertical surfaces in the containment area are wiped with disinfectant solution
- All biohazard bags and sharp containers collected and placed in the BSC room for waste pickup (see SOP 111). Biohazard bags are double bagged and placed in shipping boxes when boxes are provided by the ANL Waste Management.
- Non-disposable contaminated tools and materials are disinfected and then either autoclaved or packed to be transported (as biohazards) to the home institution for autoclaving

**Part II: After VHP decontamination (if decontamination required)**

- Standard refuse and sharps containers placed back
- Emergency exit door unlocked
- Biohazard signs removed
- All other BSL-3 related postings removed
- Unblock the hutch exhausts in non-BSL-3 hutches
- Unseal the Cold Room fan in the BSC room
- Facility key return to standard location (key box)
- Biohazard Folder with all forms removed from User Administrator's desk
- Waste autoclaved or picked up by the ANL Waste management.

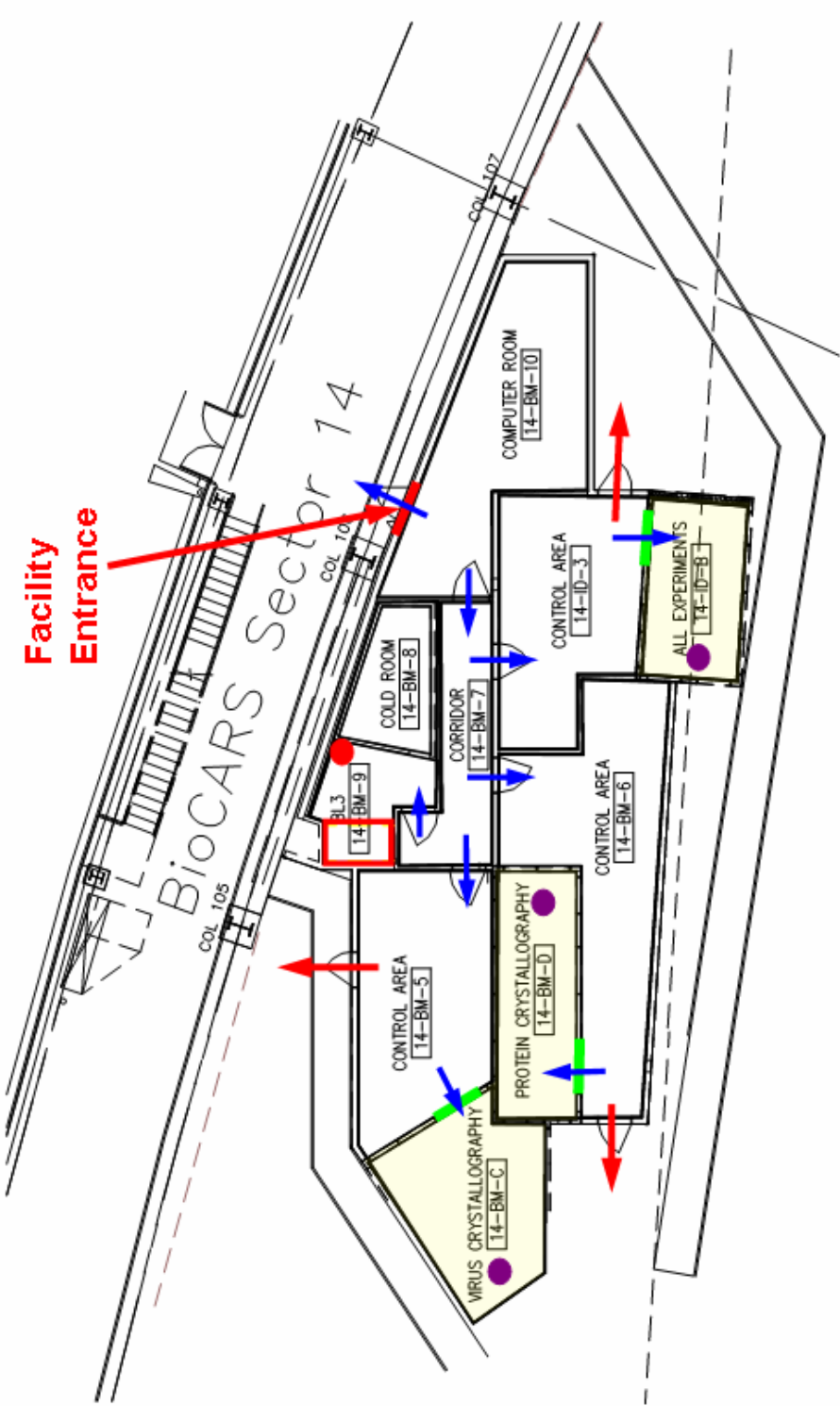
Signature of the BioCARS staff contact for the experiment:

Name: \_\_\_\_\_ Signature: \_\_\_\_\_ Date: \_\_\_\_\_








**BioCARS staff: I read, understood and will comply with this SOP.**

Name (print)	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

# Appendix A BioCARS Facility Layout



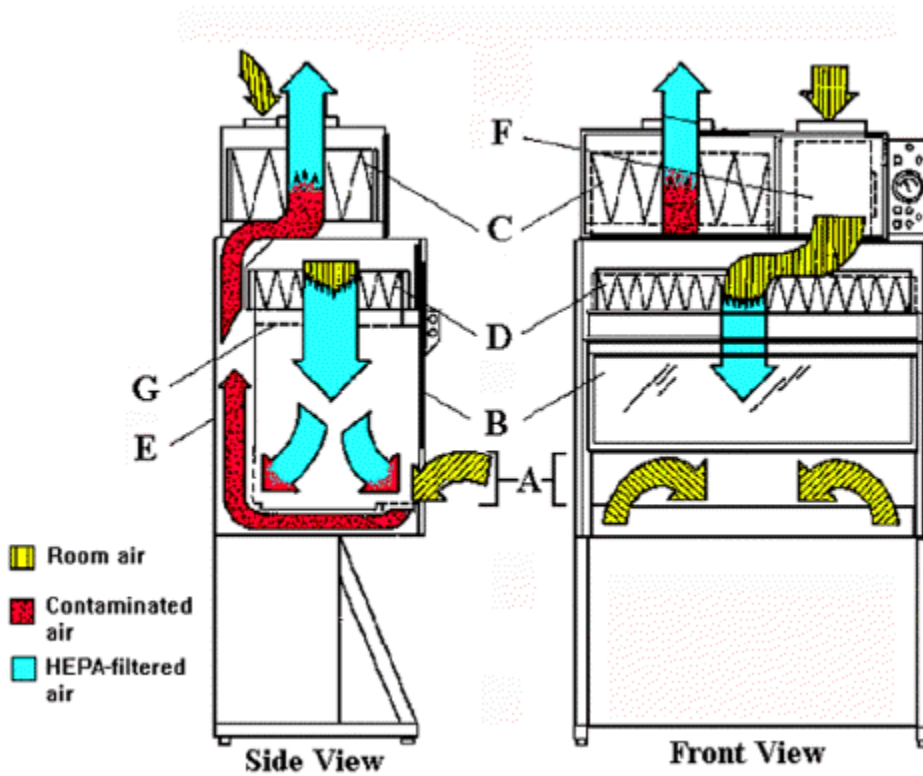
Scale - Feet

	<b>hutch (experimental station)</b>		<b>biosafety cabinet (BSC)</b>
	<b>sliding hutch door</b>		<b>shower, sink, eye wash station</b>
	<b>hutch exhaust</b>		<b>emergency exit</b>
			<b>directional air flow</b>

## Appendix B

### BioCARS Biosafety Cabinet

#### Class II, Type B2 Biosafety Cabinet



- A. front opening
- B. sash
- C. exhaust HEPA filter
- D. supply HEPA filter
- E. negative pressure exhaust plenum
- F. supply blower
- G. filter screen

**Note:** The cabinet exhaust is connected to the facility exhaust fan EF104.



## Biosafety Cabinet (BSC) Work Practices and Procedures

Adapted from: Primary Containment for Biohazards:  
Selection, Installation and Use of Biological Safety Cabinets  
SECTION V  
BSC Use by the Investigator: Work Practices and Procedures  
U.S. Department of Health and Human Services  
Public Health Service  
Centers for Disease Control and Prevention *and* National Institutes of Health  
September 1995

### Preparing for Work Within a BSC

- BSC blowers shall be operated at least five minutes before beginning work to allow the cabinet to "purge".
- The work surface, the interior walls (not including the supply filter diffuser), and the interior surface of the window shall be wiped with 70% ethanol (EtOH), a 1:100 dilution of household bleach (i.e., 0.05% sodium hypochlorite), or other disinfectant as determined by the investigator to meet the requirements of the particular activity. When bleach is used, a second wiping with sterile water is needed to remove the residual chlorine, which may eventually corrode stainless steel surfaces.
- Similarly, the surfaces of all materials and containers placed into the cabinet shall be wiped with 70% ETOH to reduce the introduction of contaminants to the cabinet environment.
- Pacing all necessary materials in the BSC before beginning work will minimize the number of arm-movement disruptions across the air barrier of the cabinet.
- The rapid movement of arms in a sweeping motion into and out of the cabinet will disrupt the air curtain. Moving arms slowly, perpendicular to the face opening of the BSC, is recommended.
- Other personnel activities in the BSC room shall be minimized (e.g., rapid movement, open/closing room doors, etc.) as they also may disrupt the BSC air barrier.
- Protective clothing has to be worn (**SOP 106**). Two pairs of gloves are worn to provide hand protection. Gloves shall be pulled over the sleeves, rather than worn inside.
- Manipulation of materials shall be delayed for one minute after placing the hands/arms inside the cabinet. This allows the cabinet to stabilize and to "air sweep" the hands and arms to remove surface microbial contaminants.
- The front grille must not be blocked by anything, including user's arms. Raising the arms slightly will alleviate this problem.
- All operations shall be performed at least 4 inches from the front grille on the work surface.
- Materials or equipment placed inside the cabinet may cause disruption to the airflow. Only the materials and equipment required for the immediate work shall be placed in the BSC. All Extra supplies shall be stored outside the cabinet.

### Material Placement Inside the BSC

- Plastic-backed absorbent toweling shall be placed on the work surface (but not on the front or rear grille openings). This facilitates routine cleanup and reduces splatter and aerosol formation during an overt spill.
- All materials shall be placed as far back in the cabinet as practical. Similarly, aerosol-generating equipment (e.g., vortex mixers, tabletop centrifuges) shall be placed toward the rear of the cabinet.
- Bulky items such as biohazard bags shall be placed to one side of the interior of the cabinet.
- The autoclavable biohazard collection bag shall not be taped to the outside of the cabinet.

- Upright pipette collection containers shall not be used in BSCs nor placed on the floor outside the cabinet. The frequent inward/outward movement needed to place objects in these containers is disruptive to the integrity of the cabinet air barrier and can compromise both personnel and product protection. Only horizontal pipette discard trays containing an appropriate chemical disinfectant shall be used within the cabinet.
- Potentially contaminated materials shall not be brought out of the cabinet until they have been surface decontaminated. Alternatively, contaminated materials can be placed into a closable container for transfer to an autoclave or for other decontamination treatment.

### **Operations Within a BSC**

- Many common procedures conducted in BSCs may create splatter or aerosols. Good microbiological techniques shall always be used when working in a biological safety cabinet.
- The general work flow shall be from "clean to contaminated (dirty)". Materials and supplies shall be placed in such a way as to limit the movement of "dirty" items over "clean" ones.
- Open flames are not used in a biological safety cabinet. An open flame in a BSC would create turbulence which disrupts the pattern of air supplied to the work surface. When absolutely necessary, touch-plate microburners equipped with a pilot light to provide a flame on demand may be used. The burner must be turned off when work is completed.
- Users must determine the appropriate method of decontaminating materials that will be removed from the BSC at the conclusion of the work.

### **Decontamination**

#### *Surface Decontamination*

- All containers and equipment shall be surface decontaminated and removed from the cabinet when work is completed. At the end of the work, the final surface decontamination of the cabinet shall include a wipe-down of the work surface, the cabinet's sides and back, and the interior of the glass. Users shall remove their gloves and gowns and wash their hands as the final step.
- Small spills within the BSC can be handled immediately by removing the contaminated absorbent paper toweling and placing it into the biohazard bag. Any splatter onto items within the cabinet, as well as the cabinet interior, shall be immediately wiped with a towel dampened with decontaminating solution. Gloves shall be changed after the work surface is decontaminated and before placing clean absorbent toweling in the cabinet. Hands shall be washed whenever gloves are changed or removed.
- Spills large enough to result in liquids flowing through the front or rear grilles require more extensive decontamination. All items within the cabinet shall be surface decontaminated and removed. After ensuring that the drain valve is closed, decontaminating solution can be poured onto the work surface and through the grille(s) into the drain pan.
- Twenty to thirty minutes is generally considered an appropriate contact time for decontamination, but this varies with the disinfectant and the microbiological agent. Manufacturer's directions shall be followed. The spilled fluid and disinfectant solution on the work surface shall be absorbed with paper towels and discarded into a biohazard bag. The drain pan shall be emptied into a collection vessel containing disinfectant. A flexible tube shall be attached to the drain valve and be of sufficient length to allow the open end to be submerged in the disinfectant within the collection vessel. This procedure serves to minimize aerosol generation. The drain pan shall be flushed with water and the drain tube removed.

#### *Gas Decontamination*

BSCs that have been used for work involving infectious materials must be decontaminated before HEPA filters are changed or internal repair work is done. Before a BSC is relocated, a risk assessment which considers the agents manipulated within the BSC must be done to determine the need for decontamination. The most common decontamination method uses formaldehyde gas, although more recently hydrogen peroxide vapor has been used successfully.

## Appendix C

### Questionnaire for BioCARS Proposals Involving Viruses

#### OBJECTIVES

Objectives of the questionnaire are to determine and *validate*

- (a) whether the use of the material is permitted at BioCARS,
- (b) the appropriate virus-specific hazard controls, and
- (c) the applicable regulatory requirements for transportation

#### INSTRUCTIONS

Please submit a separate questionnaire for each viral species proposed. However, contact the BioCARS Biosafety Coordinator if you believe that responses for *multiple closely related viruses/serotypes* may be adequately described by completion of a *single* questionnaire.

Please provide additional information when requested as an attachment to the questionnaire (refer to the relevant question).

If you find that a YES or NO response is inadequate/ambiguous without explanation, please provide clarification in the attachment.

#### Scientist responsible for experiment

\_\_\_\_\_

**Address** \_\_\_\_\_

**Phone number** \_\_\_\_\_

**E-mail:** \_\_\_\_\_

**Signature:** \_\_\_\_\_

#### 1. Identify the virus

2.1. Formal name of virus, including (if applicable) serotype/variant, etc.

\_\_\_\_\_

2.2. Informal/common name of virus and/or disease

\_\_\_\_\_

2.3 Classification of the virus

BSL-2 BSL-3 **2. Regulatory requirements**

		YES	NO
<b>2.1</b>	The virus is a viable form of EITHER (a) an HHS select agent virus, or (b) a USDA high consequence virus. (See list in Appendix Q1) <b>If YES, STOP.</b> <b>The virus is NOT permitted at BioCARS.</b>		
<b>2.2</b>	The virus is a viable form of a viral etiologic agent for which interstate transport is subject to the regulations defined by 42 CFR Part 72. (See Appendix Q2 for list of regulated viruses.)		
<b>2.3</b>	A federal permit for importation or interstate transportation is required. (See Appendix Q3 for information about the regulations.)		

**3. Availability of information from common sources\***

		YES	NO
<b>3.1</b>	An Agent Summary Statement is in Section VII-F of the CDC “Biosafety in Microbiological and Biomedical Laboratories” <a href="http://www.cdc.gov/od/ohs/biosfty/bmb14/bmb14toc.htm">http://www.cdc.gov/od/ohs/biosfty/bmb14/bmb14toc.htm</a>		
<b>3.2</b>	An MSDS for the virus is published by Health Canada <a href="http://www.hc-sc.gc.ca/pphb-dgsp/psd/ftss/index.html">http://www.hc-sc.gc.ca/pphb-dgsp/psd/ftss/index.html</a>		
<b>3.3</b>	A Risk Group is identified in Appendix B of the NIH Guidelines for Research Involving Recombinant DNA Molecules <a href="http://www4.od.nih.gov/oba/rac/guidelines_02/NIH_Guidelines_Apr_02.htm">http://www4.od.nih.gov/oba/rac/guidelines_02/NIH_Guidelines_Apr_02.htm</a>		
<b>3.4</b>	A biosafety category appears in the matrix compiled by the American Biological Safety Association <a href="http://www.absa.org/resriskgroup.html">http://www.absa.org/resriskgroup.html</a>		
<b>3.5</b>	The pathogenic/epidemiologic characteristics of the virus is described in the CDC Infectious Diseases Information Index <a href="http://www.cdc.gov/ncidod/diseases/index.htm">http://www.cdc.gov/ncidod/diseases/index.htm</a>		
<b>3.6</b>	The characteristics of the virus/disease are described by the World Health Organization <a href="http://www.who.int/health-topics/idindex.htm">http://www.who.int/health-topics/idindex.htm</a> and <a href="http://www.who.int/vaccine_research/diseases/en/">http://www.who.int/vaccine_research/diseases/en/</a>		

\*Some of the queries in Sections 3 and 4 require citations of appropriate literature. For some responses, citation of the sources above (or similar resources) may be adequate.

#### 4. Biosafety at your institution

		YES	NO
4.1	<p>Has each user been trained and authorized for the work with the virus at your home institution by the home institution's IBC?</p> <p>If <b>YES</b>, provide a letter of authorization.</p> <p>If <b>NO</b>, <b>please obtain training and authorization. They are required for conducting experiments at BioCARS.</b></p>		
4.2	<p>Your organization's Institutional Biosafety Committee has approved a protocol/standard operating procedure for the work with the virus in your laboratory.</p> <p>If <b>YES</b>, provide</p> <ul style="list-style-type: none"> <li>• The minimum biosafety level authorized by the IBC</li> <li>• Evidence of IBC approval</li> <li>• The protocol approved by the IBC.</li> </ul> <p>If <b>NO</b>, explain why.</p> <p><b>If your IBC authorized laboratory manipulation of the virus under BSL-1 hazard controls, consult the BioCARS biosafety officer before completing the remainder of this table.</b></p>		
4.3	<p>Your institution requires screening by medical professionals to identify individuals who may be more susceptible to infection than the normal adult population.</p> <p><b>Each user must sign the Risk Factor Form (Appendix D of the SOP).</b></p>		
4.4	<p>Use of the virus in your laboratory or vivarium requires respiratory protection.</p> <p>If <b>YES</b>, describe the type(s) of devices used.</p> <p><b>As use of respirators is required for work with BSL-3 samples at BioCARS. BioCARS SOP requires evidence of respirator training and fit-testing in accordance with OSHA standards.</b></p>		
4.5	<p>An FDA-approved vaccine is available for the virus.</p> <p>If <b>YES</b>, explain the vaccination policy at home institution.</p>		
4.6	<p>FDA-approved antiviral therapeutic agent(s) are available for beneficial treatment of infected humans.</p> <p>If <b>YES</b>, identify the therapeutic agent(s) and whether your institution maintains a supply.</p>		
4.7	<p>Information about the stability of the virus on various types of surfaces, especially those relevant to a laboratory is available (from the literature and/or acquired in your laboratory).</p> <p>If <b>YES</b>, provide brief summary information and describe its source*.</p> <p><b>Data (from literature or home institution) on viability of the agent on typical lab surfaces have to be provided if work with frozen BSL-3 crystals is planned.</b></p>		

		YES	NO
<b>4.8</b>	Information about disinfectants to which the virus is particularly susceptible and/or relatively resistant is available (from the literature and/or acquired in your laboratory). If <b>YES</b> , provide brief summary information and describe its source*. <b>Effective disinfectant:</b>		

\* See Section 2 for typical sources of information.

## 5. Additional information about the virus

The source of the virus or its serotype/variant is (check all that apply):

- A repository
- Another research group
- A naturally infected host species obtained by your laboratory
- A host species experimentally infected in your laboratory
- Cultured host cells experimentally infected in your laboratory
- Other

Briefly explain each indicated source.

	YES	NO	
<b>5.1</b>			There is published information describing multiple naturally occurring serotypes/variants of the virus. If YES, describe the characteristics of the serotype(s)/variant(s) proposed for analysis, with emphasis on enhancement or diminution of pathogenicity, severity of disease, transmissibility, infectivity, susceptibility of certain populations, environmental stability, sensitivity to anti-viral drugs, effectiveness of vaccines, or other characteristics relevant to biohazard. Is the serotype/variant proposed for analysis indigenous to a particular region? Provide literature references if available.*
<b>5.2</b>			The virus has been intentionally genetically modified either in your laboratory or another investigator's laboratory. If YES, describe the genetic modification and its effect (as available) on the characteristics indicated in item ___ immediately above.
<b>5.3</b>			Inhalation is a primary route of transmission for the principal natural host(s).
<b>5.4</b>			Although inhalation is not a primary route of transmission, there is published evidence that inhalation is, or is likely to be, a route of infection under some conditions, e.g., experimental animal models, livestock workers, or those who work with infected tissues or laboratory cultures. If YES, provide brief, summary information and references.*
<b>5.5</b>			An arthropod is a natural vector for infection of higher animals or humans.
<b>5.6</b>			Humans are among the natural hosts.
<b>5.7</b>			Non-human primates are among the natural hosts.
<b>5.8</b>			There is published evidence that the virus is infectious for non-human primates, even if primates are not the principal natural host.
<b>5.9</b>			Although the principal host(s) is non-primate animals, there is published evidence for zoonotic infection of humans, e.g., for livestock workers, consumers of products from infected animals; or those who work with infected tissues or laboratory cultures.
<b>5.10</b>			Human to human transmission of the viral disease is known to occur. If YES, describe the prevalence and route(s) of such transmission; and provide definitive references.*

	YES	NO	
<b>5.11</b>			There is documented evidence that asymptomatic humans may serve as short- and/or long-term sources for transmission of disease. If YES, describe the prevalence of such transmission and provide definitive references.*
<b>5.12</b>			There is published evidence that specific human populations are more susceptible to infection or the consequences of infection (e.g., newborns, elderly, fetus, immunocompromised, other medical condition). If YES, provide brief summary information and references.*
<b>5.13</b>			Information is available, even if approximate, regarding the infectious dose in (as available) the principal host(s), in humans, or in an experimental animal model(s). If YES, provide information about dose, species, and route(s).*

\* See Section 2 for typical sources of information.



## Questionnaire: APPENDIX Q1

### Select Agents and High Consequence Agents Regulated by HHS and USDA

#### *HHS Select Agents and Overlap Select Agents<sup>1</sup>*

- Crimean-Congo haemorrhagic fever virus
- Ebola virus
- Cercopithecine herpesvirus 1 (Herpes B virus)
- Lassa fever virus
- Marburg virus
- Monkeypox virus
- South American Haemorrhagic Fever viruses (Jinni, Machupo, Sabia, Flexal, Guanarito)
- Tick-borne encephalitis complex (flavi) viruses (Central European Tick-borne encephalitis, Far Eastern Tick-borne encephalitis [Russian Spring and Summer encephalitis, Kyasanur Forest disease, Omsk Hemorrhagic Fever] )
- Variola major virus (Smallpox virus) and Variola minor virus (Alastrim)
- Eastern Equine Encephalitis virus
- Nipah and Hendra Complex viruses
- Rift Valley fever virus
- Venezuelan Equine Encephalitis virus

#### *USDA High Consequence Livestock or Plant Viruses<sup>2</sup>*

- Akabane virus
- African swine fever virus
- African horse sickness virus
- Avian influenza virus (highly pathogenic)
- Blue tongue virus (Exotic)
- Camel pox virus
- Classical swine fever virus
- Foot and mouth disease virus
- Goat pox virus
- Lumpy skin disease virus
- Japanese encephalitis virus Malignant catarrhal fever virus (Exotic)
- Menangle virus
- Newcastle disease virus (VVND)
- Peste Des Petitis Ruminants virus
- Sheep pox virus
- Swine vesicular disease virus
- Vesicular stomatitis virus (Exotic)
- Plum Pox Potyvirus
- Rinderpest virus

---

<sup>1</sup> Possession, Use, and Transfer of Select Agents, Interim Final Rule, 42 CFR Part 73 (December 13, 2002). Department of Health and Human Services, Office of the Inspector General <http://www.cdc.gov/od/sap/docs/btarule.pdf>

<sup>2</sup> Possession, Use and Transfer of Biological Agents and Toxins, 7 CFR 331 and 9 CFR 121 (December 13, 2002). Department of Agriculture, Animal and Plant Health Inspection Service (APHIS) <http://www.aphis.usda.gov/vs/ncie/pdf/btarule.pdf>

## Questionnaire: Appendix Q2

### Viral Etiologic Agents for Which Interstate Transport is Regulated

#### by 42 CFR Part 72

(Appendix Q2 does not include those etiologic agents regulated by 42 CFR Part 73, and thus appear in Appendix Q1 for this BioCARS questionnaire).

- Adenoviruses—human—all types
- Arboviruses—all types
- Coxsackie A and B viruses—all types
- Cytomegaloviruses
- Dengue viruses—all types
- Echoviruses—all types
- Encephalomyocarditis virus
- Hemorrhagic fever agents including, but not limited to, Crimean hemorrhagic fever(Congo), Junin, Machupo viruses, and Korean hemorrhagic fever viruses
- Hepatitis associated materials (hepatitis A, hepatitis B, hepatitis nonA-nonB)
- Herpesvirus—all members
- Infectious bronchitis-like virus
- Influenza viruses—all types
- Lymphocytic choriomeningitis virus
- Measles virus
- Mumps virus
- Parainfluenza viruses—all types
- Polioviruses—all types
- Poxviruses—all members
- Rabies virus—all strains
- Reoviruses—all types
- Respiratory syncytial virus
- Rhinoviruses—all types
- Rotaviruses—all types
- Rubella virus
- Simian virus 40
- Tick-borne encephalitis virus complex, including Russian spring-summer encephalitis, Kyasanur forest disease, Omsk hemorrhagic fever, and Central European encephalitis viruses.
- Vaccinia virus.
- Varicella virus
- Vesicular stomatis viruses—all types
- White pox viruses
- Yellow fever virus

## **Questionnaire: APPENDIX Q3**

### **USDA/APHIS Permit for Interstate Transport**

#### Explanation

Federal regulations require a USDA-APHIS (Veterinary) permit for interstate transfer of

All cultures or collections of organisms or their derivatives, which may introduce or disseminate any contagious or infectious disease of animals (including poultry). [9 CFR 122.1(d)]

Information about the requirements is available at <http://www.aphis.usda.gov/vs/ncie/>.

The permit is available at <http://www.aphis.usda.gov/vs/ncie/pdf/vs16-3.pdf>

The instructions for permit application VS 16-3 include the following:

“Generally, a USDA veterinary permit is needed for materials derived from animals or exposed to animal-source materials. Materials which require a permit include animal tissues, blood, cells or cell lines of livestock or poultry origin, RNA/DNA extracts, hormones, enzymes, monoclonal antibodies for IN VIVO use in non-human species, certain polyclonal antibodies, antisera, bulk shipments of test kit reagents, and microorganisms including bacteria, viruses, protozoa, and fungi.”

See also <http://www.cdc.gov/od/ohs/biosfty/impptper.htm> and the application form and explanatory information at <http://www.cdc.gov/od/ohs/biosfty/impptper.htm>

### **USER RESPONSIBILITY FOR PERMITTING**

The USER and his/her institution are solely responsible for acquiring the required permit or permits. When completing a permit application, the user’s institution shall be both the shipper and the receiver, thus avoiding the identification of Argonne National Laboratory as either a recipient or shipper. Within BioCARS, the user is responsible for maintaining custody of materials requiring a transportation and/or import permit.

Argonne National Laboratory reserves the authority request evidence of permitting prior to final authorization for the experiment, and upon arrival of the material at Argonne National Laboratory. If the permitting requirements are not adequately met, Argonne National Laboratory reserves the authority to either prohibit or limit the use of the material.

### **PLAN AHEAD FOR PERMITTING**

Acquisition of a permit may require much longer than expected.

## Appendix D

### Risk Factors Form

#### Individual Notification of Risk Factors

Some medical conditions increase susceptibility to infection and/or increase the severity of the consequences of infection. For example, persons who are immunocompromised or immunosuppressed may be at increased risk of acquiring infections. Immunocompromised and/or immunosuppressed individuals at risk include (but are not limited to) those with HIV infection, cancer, or other chronic illnesses (e.g., diabetes, lupus and other autoimmune diseases, eczema, dermatitis, certain respiratory diseases/conditions); those undergoing drug therapy that causes immunosuppression; and those who have undergone certain surgical procedures (e.g., splenectomy, gastrectomy). Certain infectious agents may adversely affect a fetus during pregnancy if the mother is infected with the agent. Pregnancy is a predisposing risk factor for some infectious diseases. Alcoholism and drug abuse are also predisposing risk factors for some infectious diseases.

Name of individual \_\_\_\_\_

Institutional Affiliation \_\_\_\_\_

Identifying number (APS/ANL system) \_\_\_\_\_

Name of individual's supervisor, the principal investigator, or equivalent

\_\_\_\_\_

***Individual's acknowledgement:* By my signature below, I acknowledge that I have read and understood the risk factors information provided above.**

Signature \_\_\_\_\_ Date \_\_\_\_\_

## Appendix E

### BSL-3 Sample Log-in/Log-out Form

**PI:**  
**ESAF Pen #:**  
**Experiment title:**  
**Spokesperson:**

#### Log-in

##### Transportation info:

Transported by users

Shipped via:

##### Arrival date/time:

##### Samples transported/shipped:

Frozen

Number of dewars:

Number of crystals:

Room temperature/trays

Number of trays:

Number of drops/wells:

Estimated number of crystals:

Room temperatures/capillaries

Number of capillaries/crystals:

Spokesperson signature: \_\_\_\_\_

## Log-out

**Departure date/time:**

**Number of crystals used for data collection:**

**Any crystals lost during mounting/dismounting:**

Yes. How many:

No.

**User Log-out Certification:**

I certify that all BSL-3 material transported/shipped to BioCARS has been either rendered non-viable or is being sent back to the home institution.

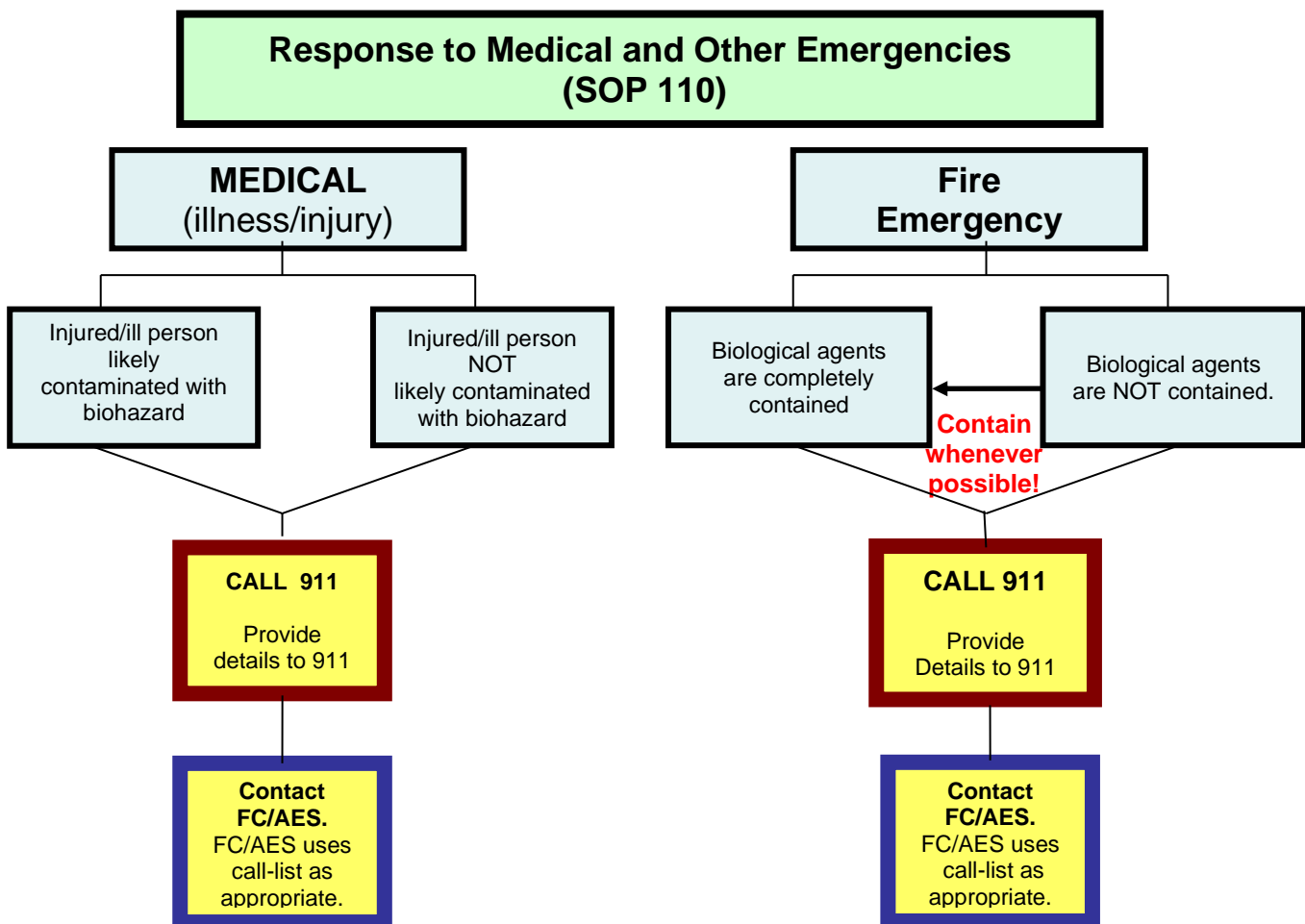
Spokesperson signature:

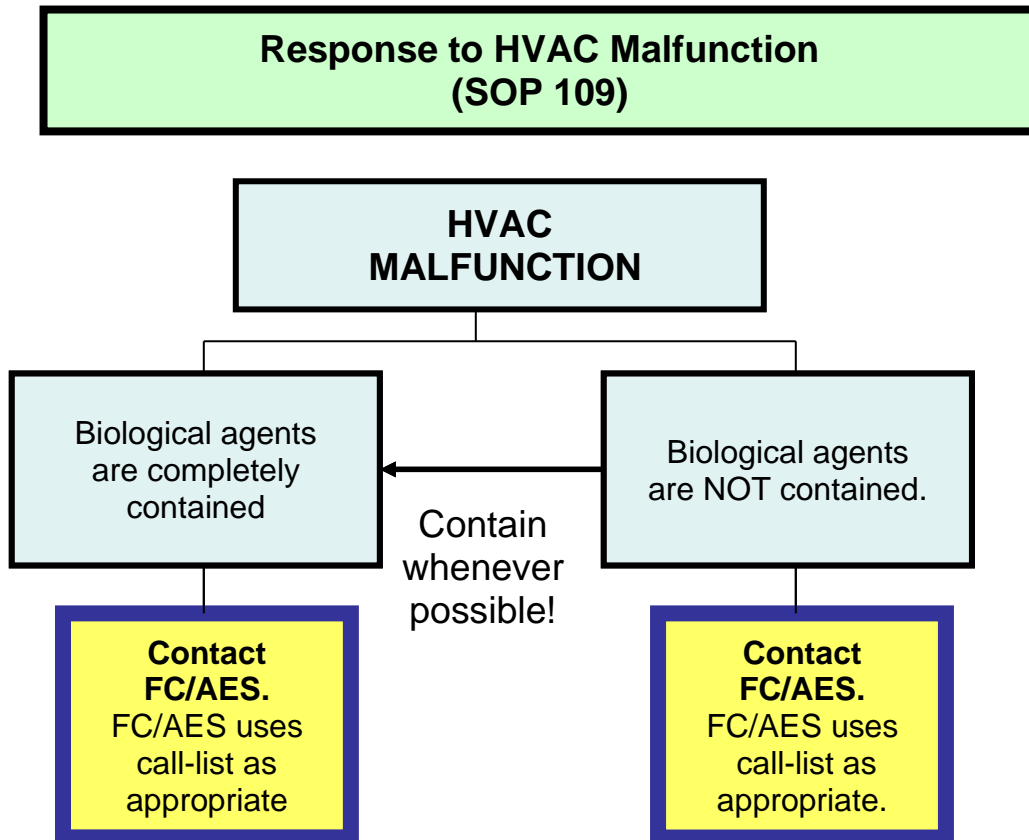
---

## Appendix F

### Flowchart on Decisions Regarding Responses During the Presence of Biohazardous Materials at BioCARS

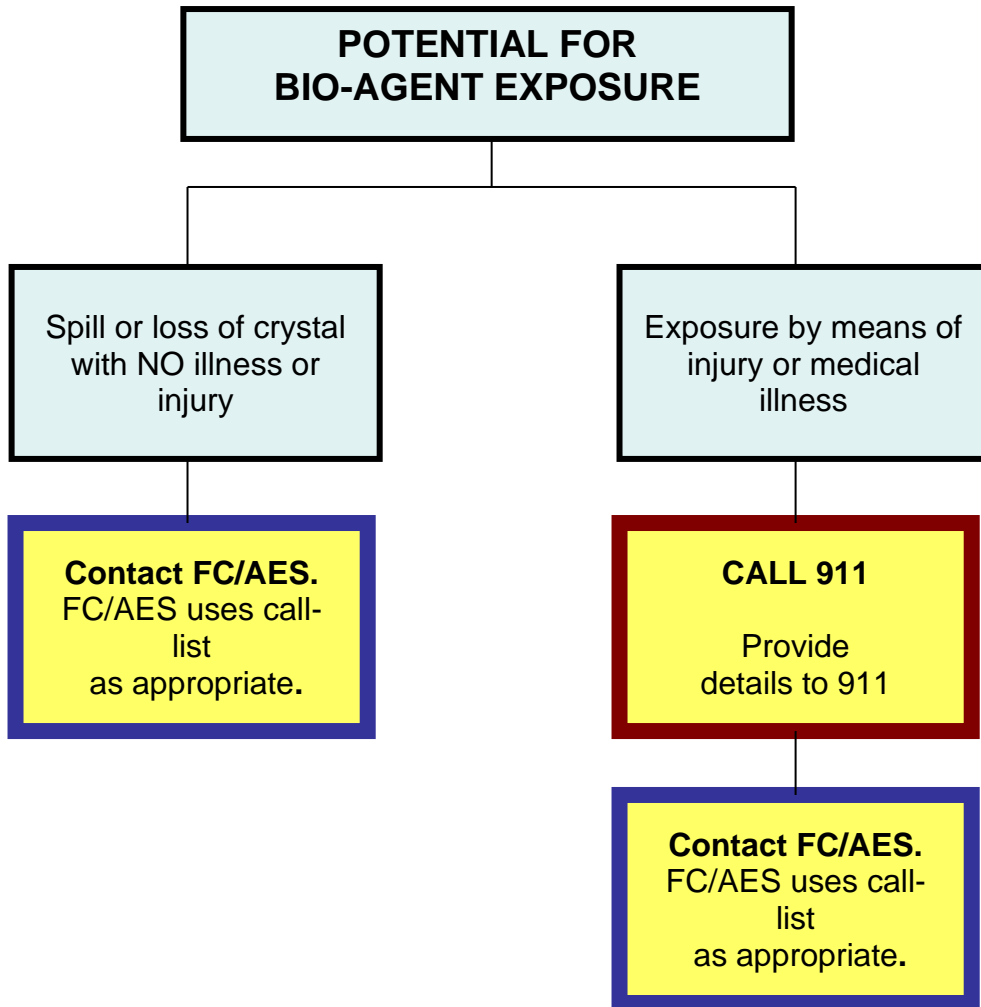
- A. Medical and other emergencies during the presence of biohazardous materials
- B. Failure of HVAC or other system/equipment important for biosafety
- C. Response to overt or potential exposure of personnel to biohazardous material
- D. VHP (vapor phase hydrogen peroxide) leak detected and not contained



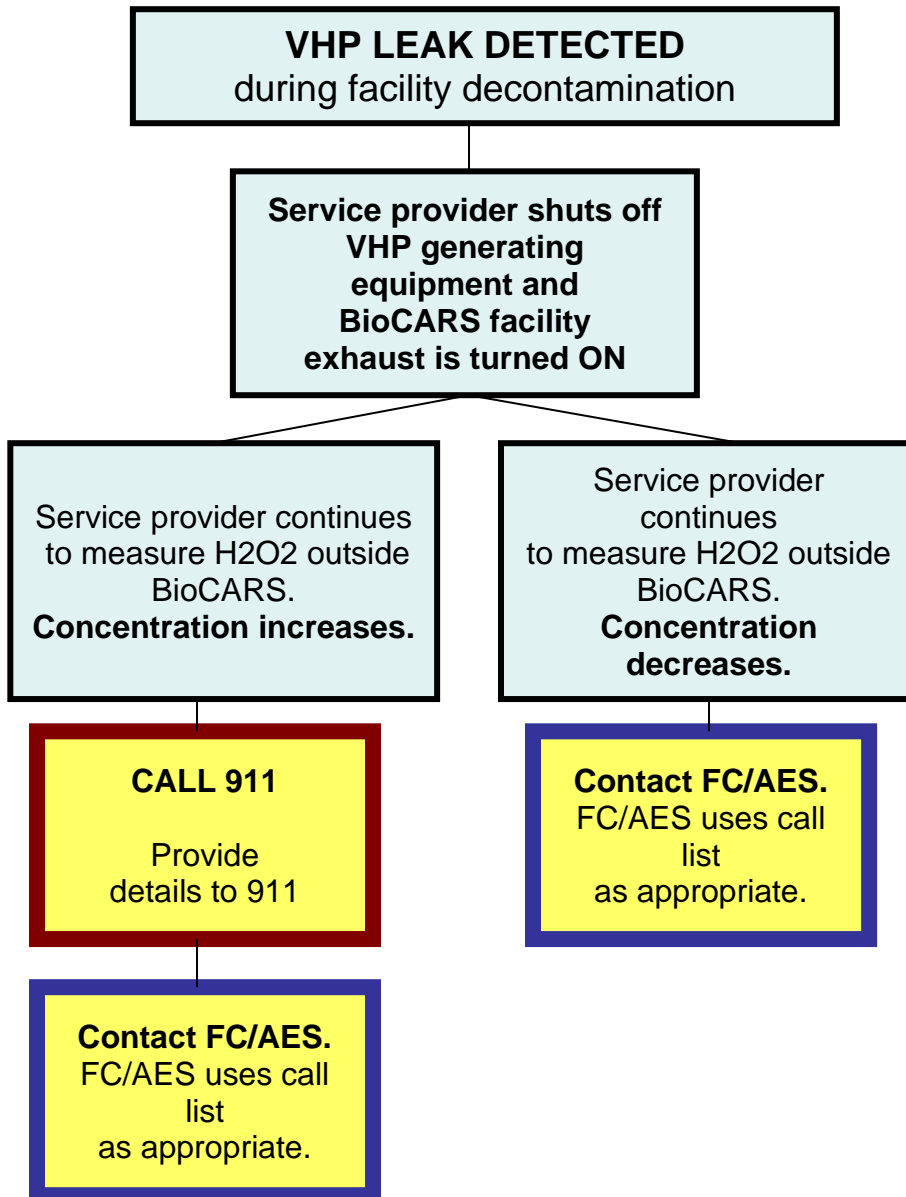




**Response to Potential Exposure to Biohazard  
(SOP 108)**



**Response to VHP leak to Experiment Hall  
(SOP 113)**



## Pressure Differentials in BioCARS Facility

**Pressure differentials in the BioCARS facility (from pressure monitors; units: inch H<sub>2</sub>O)**

**Status on April 3, 2006 (Rob Henning)**

		Computer room	ID-B	BM-C	BM-D	BSC
ID-B BSL-3 mode	Hutch door open	+0.021	-0.002	-0.010	-0.007	-0.006
	Hutch door closed	+0.022	-0.004	-0.007	-0.007	-0.006
	ID-B emergency door open	+0.020	-0.000	-0.010	-0.007	-0.006
BM-C in BSL-3 mode	Hutch door open	+0.020	-0.002	-0.030	-0.005	-0.006
	Hutch door closed	+0.021	-0.003	-0.009	-0.007	-0.007
Normal mode		+0.021	-0.003	-0.009	-0.007	-0.007

All light/alarm set points set to about 0 (setting is not precise).

It takes 5-10min for equilibration of the pressures/flows after the hutch door contact has been activated/deactivated while closing/opening hutch door (this contact activates flow rate adjustments).

When the hutch door is open (contact deactivated), the exhaust rate from the hutch is quite high (no air re-circulation). If the door is closed too quickly (before the flow rates have adjusted), turbulence inside the hutch will be created that will disturb the cryo gas stream.

**Set points set by Reinhard Pahl and Vukica Srajer (8/17/04)**

## Standard Operating Procedures for

Lasers Controlled Area 15-ID-B

ChemMatCARS, sector 15

December 02, 2016

Version 4

---

Prepared by:

Yu-Sheng Chen \_\_\_\_\_.

ChemMatCARS  
LCA Supervisor Date

Approved By:

Bryan Broocks \_\_\_\_\_.

ANL-E Laser Safety Officer Date

Edmund Elroy Chang \_\_\_\_\_.

APS/AES Division Coordinator Date

Bill Ruzicka \_\_\_\_\_.

APS/AES Division Director Date

## 1. Introduction

This is the Standard Operation Procedure to operate the lasers listed in Table 1 at the 15-ID-B station. The Laser Controlled Area (LCA) includes the entire 15-ID-B station and the B1 mini-station. The LCA does not include any area outside of the station.

**TABLE 1: Laser Specification and description**

**The Coherent LASER- COMPASS diode pumped laser system/315M-100 system**

---

Model	diode pumped laser/315M-100
ANL# Head	10860
Serial Number	H030326263
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	100
Beam Diameter at $1/e^2$ (mm)	0.32
Beam Divergence (mrad)	<2.2
Beam Asymmetry	< 100:1 vertical
Operation Mode	CW
Class	3B

---

**Crystalaser- Diode Pumped CrystaLaser/ CL532-025-L system**

---

Model	Diode Pumped laser/ CL532-025-L
ANL# Head	10800
Serial Number	2912006-4079-26702
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at $1/e^2$ (mm)	0.32
Beam Divergence (mrad)	2
Operation Mode	CW
Class	3B

---

---

**Crystalaser- Diode Pumped CrystaLaser/ CL532-025-L system**

---

Model	Diode Pumped laser/ CL532-025-L
ANL# Head	11083
Serial Number	0011632
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at 1/e <sup>2</sup> (mm)	0.45
Beam Divergence (mrad)	2
Operation Mode	CW
Class	3B

---

**M650D150-3-1670**

---

Model	M650D150-3-1670
ANL# Head	10912
Serial Number	AK1894BA
Quantity	4
Wavelength (nm)	650
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~0.6
Operation Mode	CW
Class	3B

---

**M808D150-5-1660**

---

Model	M808D150-3-1660
ANL# Head	10996
Serial Number	AK1894CO
Quantity	2
Wavelength (nm)	808
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~10
Operation Mode	CW
Class	3B

---

**M405D150-3-1670**

---

Model	M405D150-3-1670
ANL# Head	10995
Serial Number	AK1894EM
Quantity	1
Wavelength (nm)	405
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~0.6
Operation Mode	CW
Class	3B

---

**BioCARS OPOTEK Nanosecond Laser System**

This laser belongs to the sector 14. The output beam of this laser is typically coupled to a fiber optic for transport to the experiment. Below is a table listing the specifications of the laser.

---

Model	Quantel USA	OPOTEK
	/Ultra 100	/Opolette
ANL# Head	This laser is a component of 10882	10882
Serial #	1103280209	2466
Type	Nd:YAG	OPO
Quantity	1	1
Wavelength(nm)	1064	225-2200
Max Output Power(W)	4	0.4
Pulse Energy(mJ)	400	5-7
Beam Diameter at 1/e <sup>2</sup> (mm)	~3	~4
Beam Divergence (mrad)	<10	<2
Operation Mode	Pulsed	Pulsed
Pulse Width(nsec)	5	5
Rep. Rate	20Hz	20Hz
Class	4	4

---

**THORLABS Light Emitted Diodes(LED)**

**UV (365 nm) Mounted High-Power LED-M365L2**

---

Model	M365L2
ANL# Head	11001
Serial Number	M00291196
Quantity	1
Wavelength (nm)	365
Max Output Power (mW)	190
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	Beam Divergence (mrad)
Operation Mode	CW
Class	N/A

---

**UV (385 nm) Mounted High-Power LED-M385L2**

---

Model	M385L2
ANL# Head	11002
Serial Number	M00289724
Quantity	1
Wavelength (nm)	385
Max Output Power (mW)	270
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	N/A

---

**UV (505 nm) Mounted High-Power LED-M505L3**

---

Model	M505L3
ANL# Head	11003
Serial Number	M00290739
Quantity	1
Wavelength (nm)	505
Max Output Power (mW)	400
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	N/A

---



## UV (660 nm) Mounted High-Power LED-M6L3

---

Model	M660L3
ANL# Head	11004
Serial Number	M00289182
Quantity	1
Wavelength (nm)	660
Max Output Power (mW)	640
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	N/A

---

### 2. Laser Safety Personnel

LCA Supervisor for Sector 15

Yu-Sheng Chen (630) 252 – 0471

[yschen@cars.uchicago.edu](mailto:yschen@cars.uchicago.edu)

APS AES Division ESH Coordinator

Edmund Chang (630) 252 – 6714

[change@aps.anl.gov](mailto:change@aps.anl.gov)

ANL-E Laser Safety Officer

Bryan Brooks (630) 252 – 3396

[bbrooks@anl.gov](mailto:bbrooks@anl.gov)

### 3. Authorized Users

No person is allowed to operate the above laser systems unless all four of the following requirements are met:

- Completion of all APS user training requirements and authorization by the APS User Office for access to the experimental floor.
- Completion of the ANL Laser Safety Training (ESH-120) and laser-specific medical eye examination approved by ANL Medical Department.
- Familiarity with the contents of this SOP and practical training on using this laser system by the principal laser operator.
- Approval by the LCA Supervisor who will add the name to the list of authorized users.

Currently authorized users are listed in the Appendix B of this document.

### 4. Scientific Collaborators & Spectators

Scientific collaborators have access to the LCA for scientific work if they meet the requirements for authorized users. They must follow the laser set-up and operating procedures described in this SOP and its appendices.

Spectators are only permitted to enter the LCA in the presence of authorized personnel and when the lasers are turned off or laser shutters are closed (laser light is not accessible).

When no laser hazards exist in the LCA, the access to the LCA is not restricted.

## 5. General Setup and Laser Operation

### *Laser Controlled Areas (LCA)*

Laser is located in the LCA (station 15-ID-B). Station 15-ID-B is fully interlocked with a laser curtain to shield the entrance during laser operation. Warning signs will be posted on the station door. Warning lights are also located both on the inside wall of the station and at the entrance to the station.

### *Standard Optical Configuration*

The laser is enclosed in a light-blocking box. A laser shutter is placed directly at the light exit port of the laser, followed by an optical fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering. Light from the fiber is delivered to a sample position at the Diffractometer, also located in the LCA (see the layout of the configuration in Appendix A).

### **Laser Operation**

We distinguish two operating modes:

**Local mode:** Only authorized users are present inside the LCA. Users shall wear the appropriate eye protection for the laser in use. The laser remote control unit is located inside the LCA. Laser shielding panels shall be in their proper positions for operation.

**Remote mode:** The LCA door and laser curtain are closed and no one is inside the LCA. The laser remote control unit or computer is outside the LCA and the lasers are operated remotely from the 15-ID-B control area. There shall always be authorized users present in the control area when the laser is operating. Otherwise laser will be turned off.

## 6. Eyewear

Standard protective eyewear for the laser will be provided and kept in the LCA. The laser protective eyewear will be inspected at least annually and the results recorded in a log attached to the end of this SOP. This inspection sheet is also posted at the entrance of the LCA. All personnel in the LCA shall wear appropriate eye protection whenever the laser is operating. Three pairs of goggles are provided with following specifications:

<b>Laser ID/Wavelength</b>	<b>Optical Density</b>	<b>Wavelength(nm)</b>
<b>1076/377nm</b>	<b>7+</b>	<b>190-398 nm</b>
<b>10860/532nm</b>	<b>&gt;4.5</b>	<b>520-532nm</b>
<b>10800/532nm</b>	<b>&gt;9</b>	<b>190-520nm</b>
<b>10803/532nm</b>	<b>&gt;9</b>	<b>190-520nm</b>

10912/650nm	>4~5	628-690nm
10882/1064nm	>6	1064nm
10882/255~2200nm	>5	225-2200nm
10995/405nm	>9	190-520nm
10996/808nm	>5	225-2200nm
11001/365nm	7+	190-398 nm
11002/385nm	7+	190-398 nm
11003/505nm	>9	190-520nm
11004/660nm	>4~5	628-690nm

All laser protective eyewears will be inspected at least annually and the results recorded in a log attached to the end of this SOP(See Appendix D). This inspection sheet is also posted at entrance of the LCA.

## 7. Laser Hazard Control

Laser hazard warning signs are posted at the entrance to the LCA. In addition, the LCA has three-colored warning lights placed inside the area.

- Green light (“NO HAZARD – Laser off”) indicates safe entry to the room.
- Yellow light (“CAUTION – Laser energized”) indicates that laser is ready to be operated.
- Red light (“DANGER – Beams accessible”) indicates unshielded laser light present in the room.

The warning lights reflect these conditions:

The safety status of the LCA and the laser system is controlled by an interlock system. Its main controller is located in the LCA. The interlock includes the following components:

- The 15-ID-B hutch has non-defeatable curtain with interlock switches.
- The external laser shutters (below called laser shutters) are integrated in the interlock system.
- Emergency cut-off switch for the laser is also integrated in the interlock system.
- Internal laser interlock(power off).

The fundamental rules implemented in this interlock system are:

- In the local mode: the LCA curtain cannot be open while laser beam is accessible within the LCA. Opening of the LCA curtain will **close the laser shutter**.
- In the remote mode: the laser shutter is controlled remotely, from the area outside of the LCA. Opening of the LCA curtain will **close the laser shutter**.

Administrative control of the laser power supply key is implemented to prevent unauthorized operation of the laser. When the experiment is finished, the laser key is removed and stored in the key box in the BioCARS/ChemMatCARS Laser Lab. Only authorized personnel can access the Laser Lab.

The circular hole between the 15-ID-B and 15-ID-C stations shall be covered using the high-power X-ray beam stop to prevent laser light from entering the 15-ID-C station. In addition the laser shall not be operated when the 15-ID-B-1 mini hutch is opened.

Laser is surrounded by a light-blocking enclosure box. The laser shutter is placed at the exit port of the laser. Past the shutter, laser light is directly coupled to an optical fiber and delivered via fiber to a sample at the Diffractometer in the LCA. The laser light from the fiber is collimated and focused at the sample position by a small, enclosed lens assembly mounted at the exit end of the fiber. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent ( $>10^\circ$ ) beyond the focal spot. The lens assembly is mounted pointing down. Laser light alignment at the sample will be done with a minimum laser power (2mW). Following the alignment, the data collection will be held on remote mode. No one will be inside the hutch.

## **8. Additional Hazards**

None.

## **9. Control of Emergencies and Abnormal Situations**

In the cases of

- Laser burns to eyes and/or skin: Call 911, shut down the laser system, report to the LCA supervisor and the APS floor coordinator.
- Fire: Call 911, quickly evacuate from the LCA and activate the nearest fire alarm.
- Severe Weather Warning: Turn laser off and evacuate immediately to the nearest tornado shelter.
- Laser “Flash” or direct skin exposure or other laser “near miss”: shut down the laser system report to the LCA supervisor and APS floor coordinator.

## **10. Hazard**

Laser light/LED light.

## 11. Interlock System Description

**The interlock system described in this SOP is designed for an LCA equipped with a single laser.** It prevents unsafe operation which may cause the user injury due to laser light exposure. If the LCA door opens while laser light is accessible in the LCA (laser energized and laser shutter open), the interlock system either cuts off the laser power (in the local mode operation) or closes the laser shutter (in the remote mode operation), stopping the laser beam immediately. The laser power will not turn back on automatically after such event. There is a panic button inside the LCA. Pushing the button will turn off the laser power and light the FAULT indicator immediately, in the event of an emergency. An event is triggered when the shielding curtain and the EOB are both open while the laser is energized. Manually pressing a panic button can also cause this trip.

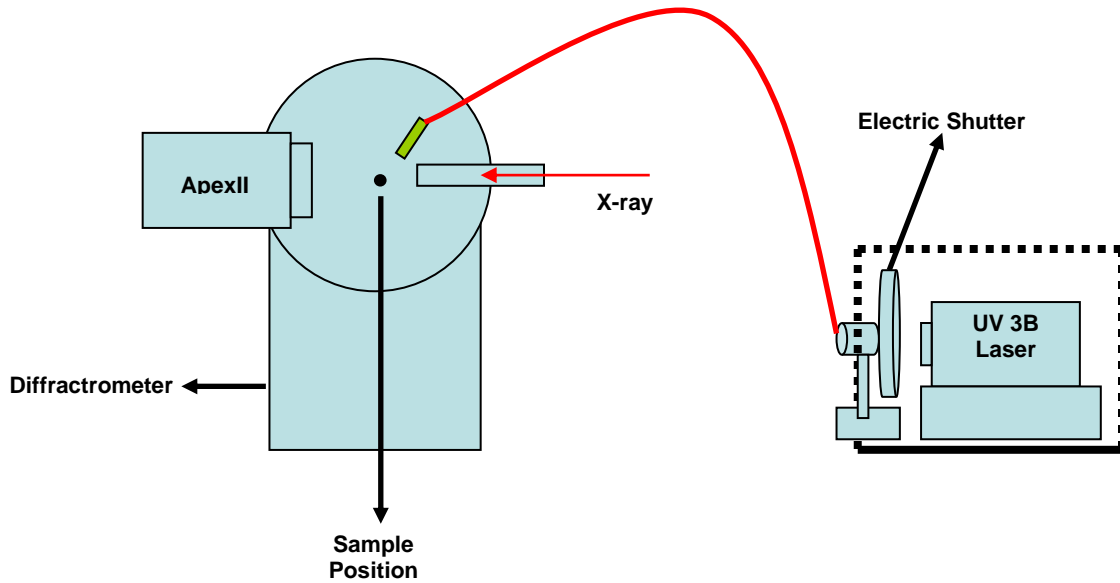
## 12. Operating procedures

The following operating procedure will be followed:

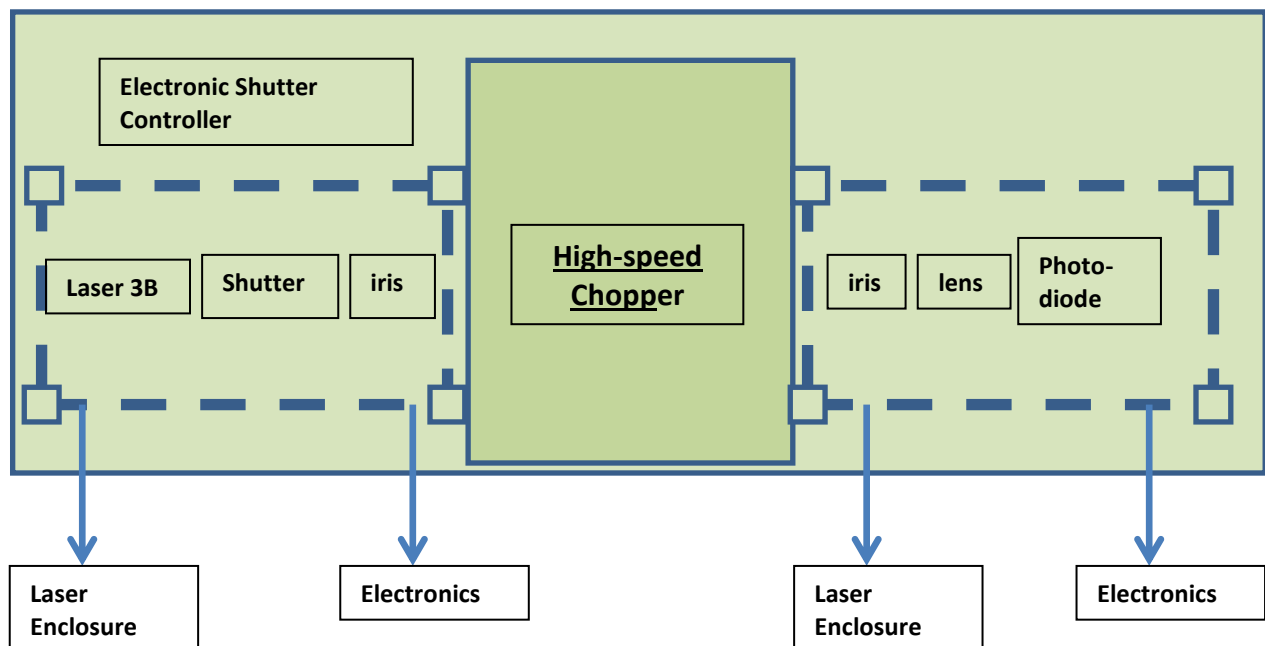
- Check that the door is properly positioned (make sure the magnetic switches on both doors overlap).
- Make sure safety goggles are available.
- Make sure that people inside the LCA are on the list of authorized personnel and are wearing proper safety goggles before activating the interlock system. (The authorized personnel list is posted on the Safety Information Board for 15-ID-B)
- Follow the alignment procedure listed in Appendix C.

## Appendix A

### Layout for the use of the 3B Laser (ID 10761/10800/10860/10995/10912/10996) and Thorlabs high power LED(ID?/?/?/?) in the 15-ID-B station For Photo-crystallography

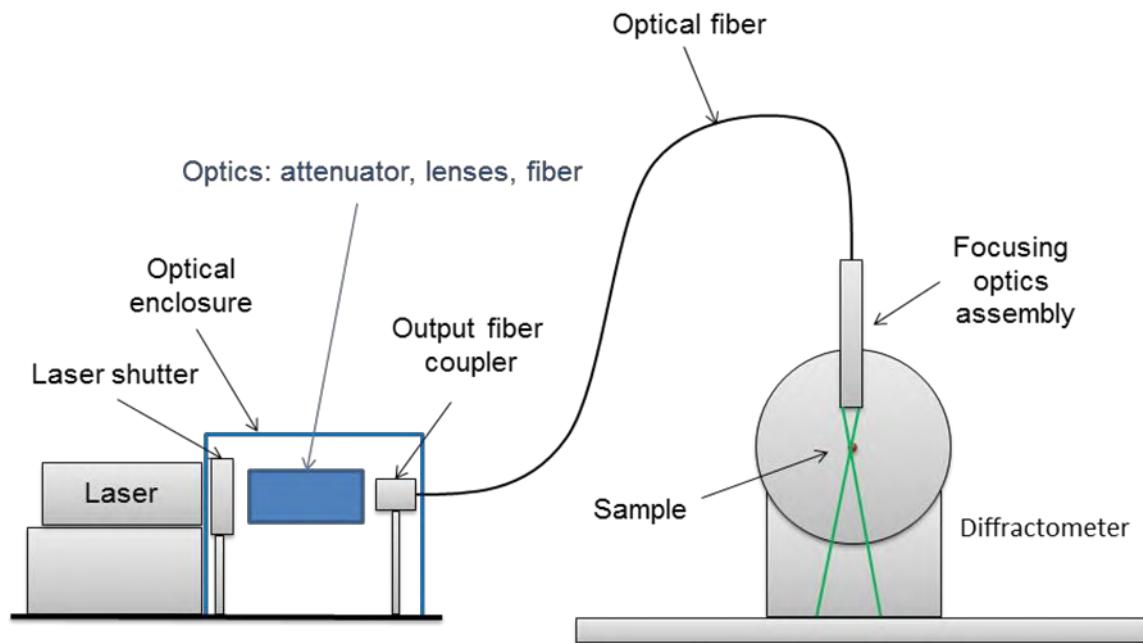


### Layout for the use of the 3B Laser(ID 10912) in the 15-ID-B station For Chopper Timing Determination



## Layout for the use of the Class4 Laser(ID 10882) in the 15-ID-B station

The Oportek Opolette ns laser (ID 10882) is setup on a portable cart and therefore can be used in any of our LCA. A light-tight box (optical enclosure) is placed directly in front of the laser exit port, enclosing a laser shutter as the first element (see figure below), necessary optical elements (attenuator, collimating/focusing lenses, fiber) and an output fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering while coupling laser light into an output optical fiber. Laser light is delivered by the fiber to the sample mounted on a diffractometer. The light-delivery end of the fiber is connected to an enclosed focusing optics assembly. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent ( $>10^\circ$ ) beyond the focal spot. The assembly is mounted pointing down.







## Appendix C

### Laser (UV/3B/4) alignment procedures

1. Make sure the laser is in the lowest power mode.
2. Make sure that there are **NO** obstructions in the beam that might scatter the laser light.
3. Check laser shutter and internal interlock and make sure they work properly.
4. Make sure the optical fiber is in place.
5. Make sure laser enclosure box is in place.
6. Turn on the laser at low power.
7. Align the optical fiber coupler while monitoring the laser power at the exit of the fiber with a power meter to maximize the transmitted light intensity (<10 $\mu$ J at the sample).
8. With laser shutter closed, place the output end of the fiber into its mount at the microspectrophotometer.
9. With the laser power set at the minimum, align the laser beam at the sample location.
10. With laser shutter closed, place the sample at the Diffractometer and align for collection of X-ray diffraction data.
11. Following the alignments, the data collection is carried remotely at 15-ID-B control area. There is no one in the 15-ID-B station.







## Standard Operating Procedures for

Lasers Controlled Area 15-IDC

ChemMatCARS, sector 15

December 2, 2016

Version 4

---

Prepared by:

Yu-Sheng Chen

LCA Supervisor Date  
ChemMatCARS

Approved By:

Bryan Broocks

ANL-E Laser Safety Officer Date

Edmund Elroy Chang

APS/AES Division Coordinator Date

Bill Ruzicka

APS/AES Division Director Date

## 1. Introduction

This is the Standard Operation Procedure to operate the lasers listed in Table 1 at the 15-ID station. The Laser Controlled Area (LCA) includes the entire 15-IDC station. The LCA does not include any area outside of the station.

**TABLE 1: Laser Specification and description**

<b>Crystallaser- Diode Pumped CrystaLaser/ CL532-025-L system</b>	
Model	Diode Pumped laser/ <b>CL532-025-L</b>
ANL# Head	10800
Serial Number	2912006-4079-26702
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at 1/e <sup>2</sup> (mm)	0.32
Beam Divergence (mrad)	2
Operation Mode	CW
Class	IIIB

<b>Crystallaser- Diode Pumped CrystaLaser/ CL532-025-L system</b>	
Model	Diode Pumped laser/ CL532-025-L
ANL# Head	11803
Serial Number	0011632
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at 1/e <sup>2</sup> (mm)	0.45
Beam Divergence (mrad)	2
Operation Mode	CW
Class	3B

## **The Coherent LASER-COMPASS diode pumped laser system/315M-100 system**

---

Model	diode pumped laser/315M-I 00
ANL# Head	10860
Serial Number	H030326263
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	100
Beam Diameter at I/e <sup>2</sup> (mm)	0.32
Beam Divergence (mrad)	<2.2
Beam Asymmetry	< 100:1 vertical
Operation Mode	CW
Class	IIIB

---

### **2. Laser Safety Personnel**

LCA Supervisor for Sector 15

Yu-Sheng Chen (630) 252 – 0471

[yschen@cars.uchicago.edu](mailto:yschen@cars.uchicago.edu)

APS AES Division ESH Coordinator

Edmund Chang (630) 252 – 6714

[change@aps.anl.gov](mailto:change@aps.anl.gov)

ANL-E Laser Safety Officer

Bruce Murdoch (630) 252 – 4905

[btmurdoch@anl.gov](mailto:btmurdoch@anl.gov)

### **3. Authorized Users**

No person is allowed to operate the above laser systems unless all four of the following requirements are met:

- a) Completion of all APS user training requirements and authorization by the APS User Office for access to experimental floor.
- b) Completion of the ANL Laser Safety Training (ESH-120) and laser-specific medical eye examination approved by ANL Medical Department.
- c) Familiarity with the content of this SOP and practical training on using this laser system by the principal laser operator.
- d) Approval by the LCA Supervisor who will add the name to the list of authorized users.

Currently authorized users are listed in the Appendix B of this document.

### **4. Scientific Collaborators & Spectators**

Scientific collaborators have access to the LCA for scientific work if they meet the requirements for authorized users. They must follow the laser set-up and operating procedures described in this SOP and its appendices.

Spectators are only permitted to enter the LCA in presence of authorized personnel and the lasers are turned off or laser shutters are closed (laser light is not accessible). No laser hazards exist in the LCA, the access to the LCA is not restricted.

## 5. General Setup and Laser Operation

### *Laser Controlled Areas (LCA)*

Laser is located in the LCA (station 15-IDC). Station 15-ID-C is fully interlocked with a laser curtain to shield the entrance during laser operation. All the windows will be covered by the laser safety curtains. Warning Sign is located at the entrance to the station.

### *Standard Optical Configuration*

The laser is enclosed in a light-blocking box. A laser shutter is placed directly at the light exit port of the laser, followed by an optical fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering. Light from the fiber is delivered to a sample cell sits on the station, also located in the LCA (see the layout of the configuration in Appendix A).

### **Laser Operation**

We distinguish two operating modes:

**Local mode:** Only authorized users are present inside the LCA. Users shall wear the appropriate eye protection for the laser in use. The laser local control unit is located inside the LCA. Laser shielding panels shall be in their proper positions for operation and the laser safety curtains shall be closed.

**Remote mode:** The LCA door and laser safety curtains are closed and no one is inside the LCA. The laser remote control unit is outside the LCA and the lasers are operated remotely from the 15-IDC control area. There shall always be authorized users present in the control area when the laser is operating.

## 6. Eyewear

Standard protective eyewear for the laser will be provided and kept in the LCA. The laser protective eyewear will be inspected at least annually and the results recorded in a log attached to the end of this SOP. This inspection sheet is also posted at the entrance of the LCA. All personnel in the LCA shall wear appropriate eye protection whenever the laser is operating. Two pairs of goggles are provided with following specifications:

Optical Density	Wavelength (nm)
>7	5000-11000 nm
>4.5	520-532nm
>9	190-520nm

All laser protective eyewears will be inspected at least annually and the results recorded in a log attached to the end of this SOP(See Appendix D). This inspection sheet is also



posted at entrance of the LCA

## 7. Laser Hazard Control

Laser hazard warning signs are posted at the entrance to the LCA. When the lights is on, it indicates the laser shutter is on and no one is allowed to enter the LCA, and when the lights is off, it indicates the laser shutter is closed.

All the windows of LCA are covered by laser safety curtains.

The safety status of the LCA and the laser system is controlled by an interlock system. Its main controller is located in the LCA. The interlock includes the following components:

- The 15-ID-C hutch has non-defeatable curtain with interlock switches.
- The external laser shutters (below called laser shutters) are integrated in the interlock system.
- Emergency cut-off switch for the laser is the switch of laser power controller. The laser power controller is out of the laser enclosure box, in case the power of laser needs to be adjusted or turned off.

Fundamental rules implemented in this interlock system are:

- a) In the local mode: The local mode is turned on after you turn on the local mode switch and plug the laser control box key in local mode controller, and the LCA curtain cannot be open while laser beam is The accessible within the LCA. Opening of the LCA curtain will **close the laser shutter**.
- b) In the remote mode: The remote mode is turned on after you turn on the remote mode switch and plug the laser control box key in the remote control box outside of the hutch, then the laser shutter is controlled remotely from the area outside of the LCA. Opening of the LCA curtain will **close the laser shutter**.

Administrative control of the laser power supply key and the laser control box key are implemented to prevent unauthorized operation of the laser. When the experiment is finished, the keys are removed and stored in the key box in the ChemMatCARS Laser Lab. Only authorized personnel can access the Laser Lab.

During the experiment is operating at 15IDC, the 15-ID-B stations' door is closed to prevent laser light from entering the 15-ID-B station. All the windows of 15-ID-C are covered by laser safety curtains.

Laser is surrounded by a light-blocking enclosure box. The laser shutter is placed at the exit port of the laser. Past the shutter, laser light is directly coupled to an optical fiber and delivered via fiber to a sample cell on the sample table. The laser light from the fiber is collimated and expanded to a 2mm parallel beam. The beam passes through the liquid-

liquid interface and diffraction grating, and is collected by a photodiode. Laser light alignment at the sample will be done with minimum laser power (0.5mW). Following the alignment, the data collection will be carried on remote mode. No one will be inside the hutch during remote mode operation.

## **8. Additional Hazards**

None.

## **9. Control of Emergencies and Abnormal Situations**

In the cases of

- Laser burns to eyes and/or skin: Call 911, shut down the laser system, report to the LCA supervisor and the APS floor coordinator.
- Fire: Call 911, quickly evacuate from the LCA and activate the nearest fire alarm.
- Severe Weather Warning: Turn laser off and evacuate immediately to the nearest tornado shelter.
- Breakdown of a high voltage system: Call 911 if help is needed shut off power at the main circuit breaker, and report to the LCA supervisor.

## **10. Hazard**

Laser light.

## **11. Interlock System Description**

The interlock system described in this SOP is designed for a LCA equipped with a single laser. It prevents unsafe operation which may cause the user injury due to laser light exposure. If the LCA laser safety curtains open while laser light is accessible in the LCA (laser energized and laser shutter open), the interlock system closes the laser shutter, stopping the laser beam immediately. The power supply of the laser is outside of the laser enclosure box, and can be turned off manually if emergency happens.

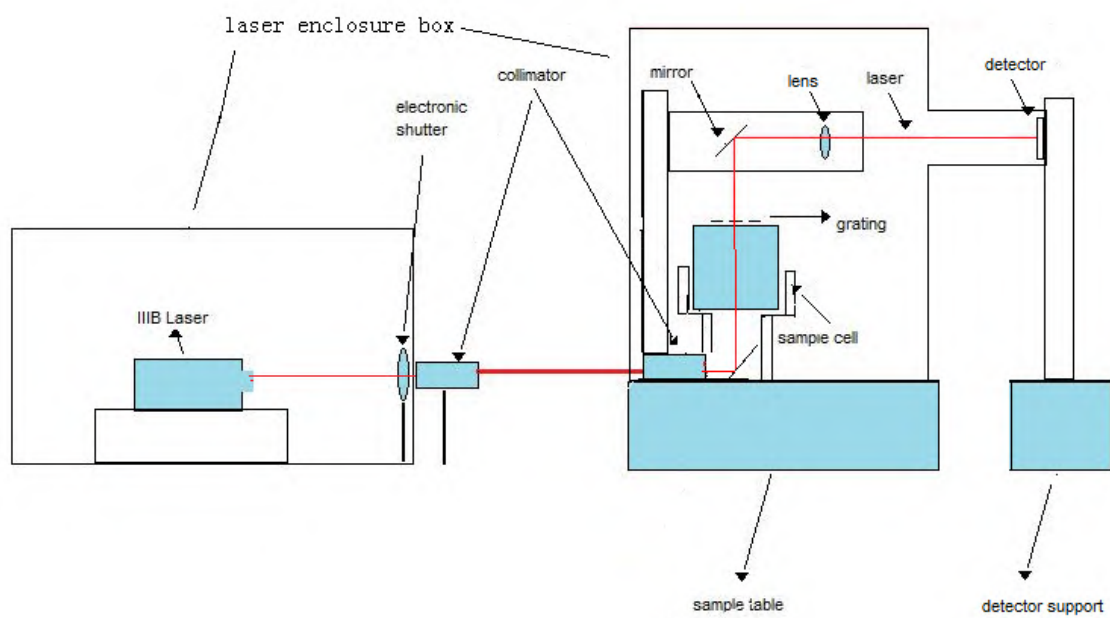
## **12. Operating procedures**

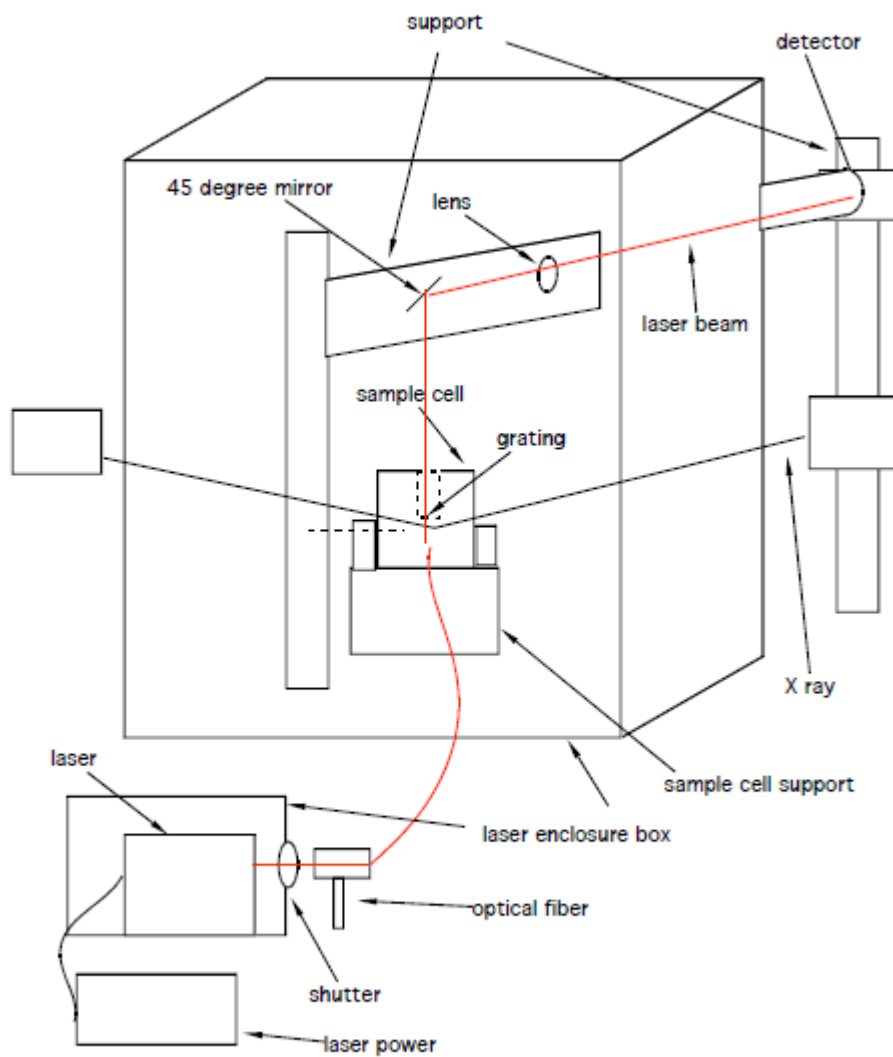
The following operating procedure will be followed:

- Check that the door is properly positioned (make sure the magnetic switches on both doors overlap).
- Make sure safety goggles are available.
- Make sure that people inside the LCA are on the list of authorized personnel and are wearing proper safety goggles before activating the interlock system. (The authorized personnel list is posted on the Safety Information Board for 15-IDC)
- Follow the alignment procedure listed in Appendix C.

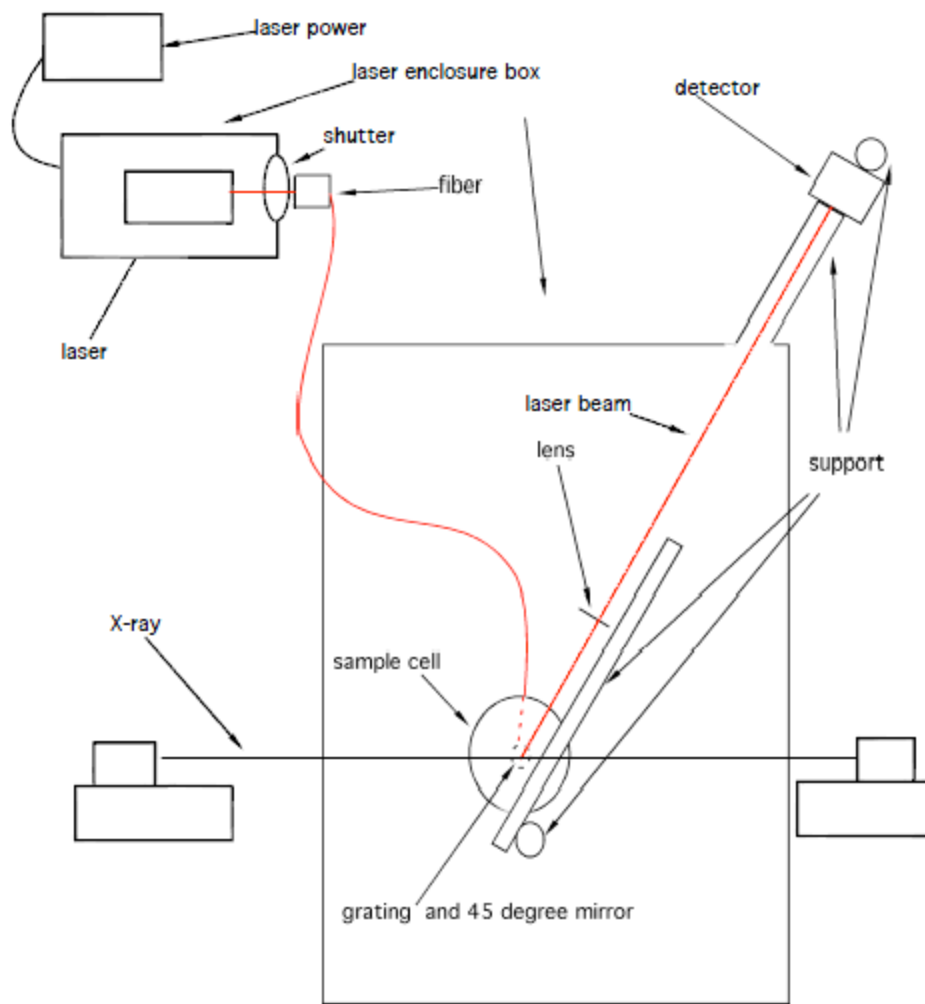
## **Appendix A**

**Layout for the use of the IIB Laser in the 15-IDC station**





Front view



**TOP VIEW**

## Appendix B

### List of authorized personnel

I have read and understood the SOP for laser use in the 15-IDC LCA.

<b>Person</b>	<b>Badge Number</b>	<b>Signature</b>
Yu-Sheng Chen	88772	
Hao Yu	81735	

## **Appendix C**

### **Laser (Green laser/IIIB) alignment procedures**

1. Make sure the laser is in the lowest power mode.
2. Make sure that there are **NO** obstructions in the beam that might scatter the laser light.
3. Check laser shutter and internal interlock and make sure they work properly.
4. Make sure the optical fiber is in place.
5. Make sure laser enclosure box is in place.
6. Turn on the laser at low power.
7. Align the optical fiber coupler while monitoring the laser power at the exit of the fiber with a power meter to maximize the transmitted light intensity.
8. With laser shutter closed, place the output end of the fiber into its mount at the sample lift table.
9. With the laser power set at the minimum, align the laser beam at the sample location and the position of photodiode.
10. With laser shutter closed, place the sample at the sample lift table and align for collection of laser scattering information.
11. Following the alignments, the data collection is carried remotely at 15-IDC control area. There are no people in the 15-IDC station.





## Standard Operating Procedures for

Lasers Controlled Area 15-ID-D

ChemMatCARS, sector 15

July 17, 2019

Version 2

---

Prepared by:

Yu-Sheng Chen \_\_\_\_\_.

ChemMatCARS  
LCA Supervisor Date

Approved By:

Bryan Broocks \_\_\_\_\_.

ANL-E Laser Safety Officer Date

Edmund Elroy Chang \_\_\_\_\_.

APS/AES Division Coordinator Date

John Connolly \_\_\_\_\_.

APS/AES Division Director Date

## 1. Introduction

This is the Standard Operation Procedure to operate the lasers listed in Table 1 at the 15-ID-D station. The Laser Controlled Area (LCA) includes the entire 15-ID-D station and the 15ID-C station. The LCA does not include any area outside of the station.

**TABLE 1: Laser Specification and description**

**The Coherent LASER- COMPASS diode pumped laser system/315M-100 system**

---

Model	diode pumped laser/315M-100
ANL# Head	10860
Serial Number	H030326263
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	100
Beam Diameter at $1/e^2$ (mm)	0.32
Beam Divergence (mrad)	<2.2
Beam Asymmetry	< 100:1 vertical
Operation Mode	CW
Class	3B

---

**Crystalaser- Diode Pumped CrystaLaser/ CL532-025-L system**

---

Model	Diode Pumped laser/ CL532-025-L
ANL# Head	10800
Serial Number	2912006-4079-26702
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at $1/e^2$ (mm)	0.32
Beam Divergence (mrad)	2
Operation Mode	CW
Class	3B

---

## Crystalaser- Diode Pumped CrystaLaser/ CL532-025-L system

---

Model	Diode Pumped laser/ CL532-025-L
ANL# Head	11083
Serial Number	0011632
Quantity	1
Wavelength (nm)	532
Max Output Power (mW)	25
Power Adjustment (mW)	0.5-27
Beam Diameter at 1/e <sup>2</sup> (mm)	0.45
Beam Divergence (mrad)	2
Operation Mode	CW
Class	3B

---

### M650D150-3-1670

---

Model	M650D150-3-1670
ANL# Head	10912
Serial Number	AK1894BA
Quantity	4
Wavelength (nm)	650
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~0.6
Operation Mode	CW
Class	3B

---

### M808D150-5-1660

---

Model	M808D150-3-1660
ANL# Head	10996
Serial Number	AK1894CO
Quantity	2
Wavelength (nm)	808
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~10
Operation Mode	CW
Class	3B

---

## M405D150-3-1670

---

Model	M405D150-3-1670
ANL# Head	10995
Serial Number	AK1894EM
Quantity	1
Wavelength (nm)	405
Max Output Power (mW)	150
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	0.5
Beam Divergence (mrad)	0.1~0.6
Operation Mode	CW
Class	3B

---

### BioCARS OPOTEK Nanosecond Laser System

This laser belongs to the sector 14. The output beam of this laser is typically coupled to a fiber optic for transport to the experiment. Below is a table listing the specifications of the laser.

---

Model	Quantel USA	OPOTEK
ANL# Head	/Ultra 100 This laser is a component of 10882	/Opolette 10882
Serial #	1103280209	2466
Type	Nd:YAG	OPO
Quantity	1	1
Wavelength(nm)	1064	225-2200
Max Output Power(W)	4	0.4
Pulse Energy(mJ)	400	5-7
Beam Diameter at 1/e <sup>2</sup> (mm)	~3	~4
Beam Divergence (mrad)	<10	<2
Operation Mode	Pulsed	Pulsed
Pulse Width(nsec)	5	5
Rep. Rate	20Hz	20Hz
Class	4	4

---

### ChemMatCARS OPOTEK Nanosecond Laser System

This laser belongs to the sector 15. The output beam of this laser is typically coupled to a fiber optic for transport to the experiment. Below is a table listing the specifications of the laser.

---

Model	Quantel USA	OPOTEK
	/Ultra 100	/Opolette
ANL# Head	This laser is a component of 11299	11299
Serial #		3287
Type	Nd:YAG	OPO
Quantity	1	1
Wavelength(nm)	1064	225-2200
Max Output Power(W)	4	0.4
Pulse Energy(mJ)	400	5-7
Beam Diameter at 1/e <sup>2</sup> (mm)	~3	~4
Beam Divergence (mrad)	<10	<2
Operation Mode	Pulsed	Pulsed
Pulse Width(nsec)	5	5
Rep. Rate	20Hz	20Hz
Class	4	4

---

### THORLABS Light Emitted Diodes(LED)

#### UV (365 nm) Mounted High-Power LED-M365LP1

---

Model	M365LP1
ANL# Head	L0004
Serial Number	M00520466
Quantity	1
Wavelength (nm)	365
Max Output Power (mW)	6800
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	1.4
Beam Divergence (mrad)	
Operation Mode	CW
Class	RG2

---

---

### UV (365 nm) Mounted High-Power LED-M365L2

---

Model	M365L2
ANL# Head	11001
Serial Number	M00291196
Quantity	1
Wavelength (nm)	365
Max Output Power (mW)	190
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	Beam Divergence (mrad)
Operation Mode	CW
Class	RG0

---

### UV (450 nm) Mounted High-Power LED-M450LP1

---

Model	M450LP1
ANL# Head	L0005
Serial Number	M00531119
Quantity	1
Wavelength (nm)	450
Max Output Power (mW)	7000
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	1.5
Beam Divergence (mrad)	
Operation Mode	CW
Class	RG3(High)

---

### UV (385 nm) Mounted High-Power LED-M385L2

---

Model	M385L2
ANL# Head	11002
Serial Number	M00289724
Quantity	1
Wavelength (nm)	385
Max Output Power (mW)	270
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	RG0

---

**(505 nm) Mounted High-Power LED-M505L3**

---

Model	M505L3
ANL# Head	11003
Serial Number	M00290739
Quantity	1
Wavelength (nm)	505
Max Output Power (mW)	400
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	RG1

---

**(660 nm) Mounted High-Power LED-M660L3**

---

Model	M660L3
ANL# Head	11004
Serial Number	M00289182
Quantity	1
Wavelength (nm)	660
Max Output Power (mW)	640
Power Adjustment (mW)	
Beam Diameter at 1/e <sup>2</sup> (mm)	
Beam Divergence (mrad)	
Operation Mode	CW
Class	RG1

---

**(355nm) Spectra-Physics EONE-355-1Y**

---

Model	EONE-355-1Y
ANL# Head	11140
Serial #	OY-3311-B81-ENG
Type	YAG
Quantity	1
Wavelength(nm)	355
Max Output Power(W)	1.25

---



Pulse Energy( $\mu$ J)	80
Beam Diameter at $1/e^2$ (mm)	$\sim 3$
Beam Divergence (mrad)	$< 2.8$
Operation Mode	Pulsed
Pulse Width(nsec)	5.0-15.0
Rep. Rate	10kHz
Class	4

---

## 2. Laser Safety Personnel

LCA Supervisor for Sector 15

Yu-Sheng Chen (630) 252 – 0471

[yschen@cars.uchicago.edu](mailto:yschen@cars.uchicago.edu)

APS AES Division ESH Coordinator

Edmund Chang (630) 252 – 6714

[change@aps.anl.gov](mailto:change@aps.anl.gov)

ANL-E Laser Safety Officer

Bryan Brooks (630) 252 – 3396

[bbrooks@anl.gov](mailto:bbrooks@anl.gov)

## 3. Authorized Users

No person is allowed to operate the above laser systems unless all four of the following requirements are met:

- Completion of all APS user training requirements and authorization by the APS User Office for access to the experimental floor.
- Completion of the ANL Laser Safety Training (ESH-120) and laser-specific medical eye examination approved by ANL Medical Department.
- Familiarity with the contents of this SOP and practical training on using this laser system by the principal laser operator.
- Approval by the LCA Supervisor who will add the name to the list of authorized users.

Currently authorized users are listed in the Appendix B of this document.

## 4. Scientific Collaborators & Spectators

**Scientific collaborators have access to the LCA for scientific work if they meet the requirements for authorized users. They must follow the laser set-up and operating procedures described in this SOP and its appendices.**

**Spectators are only permitted to enter the LCA in the presence of authorized personnel and when the lasers are turned off or laser shutters are closed (laser light is not accessible).**

**When no laser hazards exist in the LCA, the access to the LCA is not restricted.**

## 5. General Setup and Laser Operation

### *Laser Controlled Areas (LCA)*

Laser is located in the LCA (station 15-ID-D). Station 15-ID-D has two entrances. One is front entrance with two doors. The other one is back entrance with one door. Station 15-ID-D front entrance (door) is fully interlocked with a laser curtain to shield the entrance during laser operation. **The back entrance (door) is always closed and interlocked with a pair of magnetic switches to shield the back entrance during laser operation.** Warning signs will be posted on the station door. Warning lights are also located both on the inside wall of the station and at the entrance to the station.

### *Standard Optical Configuration*

The laser is enclosed in a light-blocking box. A laser shutter is placed directly at the light exit port of the laser, followed by an optical fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering. Light from the fiber is delivered to a sample position at the Diffractometer, also located in the LCA (see the layout of the configuration in Appendix A).

### **Laser Operation**

We distinguish two operating modes:

**Local mode:** Only authorized users are present inside the LCA. Users shall wear the appropriate eye protection for the laser in use. The laser remote control unit is located inside the LCA. Laser shielding panels shall be in their proper positions for operation.

**Remote mode:** The LCA door and laser curtain are closed and no one is inside the LCA. The laser remote control unit or computer is outside the LCA and the lasers are operated remotely from the 15-ID-D control area. There shall always be authorized users present in the control area when the laser is operating. Otherwise laser will be turned off.

## 6. Eyewear

Standard protective eyewear for the laser will be provided and kept in the LCA. The laser protective eyewear will be inspected at least annually and the results recorded in a log attached to the end of this SOP. This inspection sheet is also posted at the entrance of the LCA. All personnel in the LCA shall wear appropriate eye protection whenever the laser is operating. Three pairs of goggles are provided with following specifications:

Laser ID/Wavelength	Optical Density	Wavelength(nm)
1076/377nm	7+	190-398 nm
10860/532nm	>4.5	520-532nm
10800/532nm	>9	190-520nm
10803/532nm	>9	190-520nm
10912/650nm	>4~5	628-690nm
10882/1064nm	>6	1064nm
10882/255~2200nm	>5	225-2200nm
10995/405nm	>9	190-520nm
10996/808nm	>5	225-2200nm
11001/365nm	7+	190-398 nm
11002/385nm	7+	190-398 nm
11003/505nm	>9	190-520nm
11004/660nm	>4~5	628-690nm
<u>11140/355nm</u>	7+	190-398 nm

All laser protective eyewears will be inspected at least annually and the results recorded in a log attached to the end of this SOP (See Appendix D). This inspection sheet is also posted at entrance of the LCA.

## 7. Laser Hazard Control

Laser hazard warning signs are posted at the entrance to the LCA. In addition, the LCA has three-colored warning lights placed inside the area.

- Green light (“NO HAZARD – Laser off”) indicates safe entry to the room.
- Yellow light (“CAUTION – Laser energized”) indicates that laser is ready to be operated.
- Red light (“DANGER – Beams accessible”) indicates unshielded laser light present in the room.

The warning lights reflect these conditions:

The safety status of the LCA and the laser system is controlled by an interlock system. Its main controller is located in the LCA. The interlock includes the following components:

- The 15-ID-D hutch front entrance has non-defeatable curtain with interlock switches. The back entrance has magnetic door switches.
- The external laser shutters (below called laser shutters) are integrated in the interlock system.
- Emergency cut-off switch for the laser is also integrated in the interlock system.
- Internal laser interlock (power off).

The fundamental rules implemented in this interlock system are:

- In the local mode: the LCA front entrance door curtain cannot be open while laser beam is accessible within the LCA. Opening of the LCA front entrance door curtain will **close the laser power**.
- In the local mode: the LCA back entrance door cannot be open while laser beam is accessible within the LCA. Opening of the LCA back entrance door will **close the laser shutter**.
- In the remote mode: the laser shutter is controlled remotely, from the area outside of the LCA. Opening of the LCA either front entrance door curtain or back entrance door will **close the laser shutter**.

Administrative control of the laser power supply key is implemented to prevent unauthorized operation of the laser. When the experiment is finished, the laser key is removed and stored in the key box in the BioCARS/ChemMatCARS Laser Lab. Only authorized personnel can access the Laser Lab.

The circular hole between the 15-ID-C and 15-ID-D stations shall be covered using the high-power X-ray beam stop to prevent laser light from entering the 15-ID-D station.

Laser is surrounded by a light-blocking enclosure box. The laser shutter is placed at the exit port of the laser. Past the shutter, laser light is directly coupled to an optical fiber and delivered via fiber to a sample at the Diffractometer in the LCA. The laser light from the fiber is collimated and focused at the sample position by a small, enclosed lens assembly mounted at the exit end of the fiber. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent ( $>10^\circ$ ) beyond the focal spot. The lens assembly is mounted pointing down. Laser light alignment at the sample will be done with a minimum laser power (2mW). Following the alignment, the data collection will be held on remote mode. No one will be inside the hutch.

## 8. Additional Hazards

None.

## 9. Control of Emergencies and Abnormal Situations

In the cases of

- Laser burns to eyes and/or skin: Call 911, shut down the laser system, report to the LCA supervisor and the APS floor coordinator.
- Fire: Call 911, quickly evacuate from the LCA and activate the nearest fire alarm.
- Severe Weather Warning: Turn laser off and evacuate immediately to the nearest tornado shelter.
- Laser “Flash” or direct skin exposure or other laser “near miss”: shut down the laser system report to the LCA supervisor and APS floor coordinator.

## 10. Hazard

Laser light/LED light.

## 11. Interlock System Description

**The interlock system described in this SOP is designed for an LCA equipped with a single laser.** It prevents unsafe operation which may cause the user injury due to laser light exposure. If the LCA door opens while laser light is accessible in the LCA (laser energized and laser shutter open), the interlock system either cuts off the laser power (in the local mode operation) or closes the laser shutter (in the remote mode operation), stopping the laser beam immediately. The shutter will not turn back on automatically after such event until reset button is pressed. There is a panic button inside the LCA. Pushing the button will turn off the laser power in the event of an emergency. An event is triggered when the shielding curtain and the laser enclosure box(EOB) are both open while the laser is energized. Manually pressing a panic button can also cause this trip.

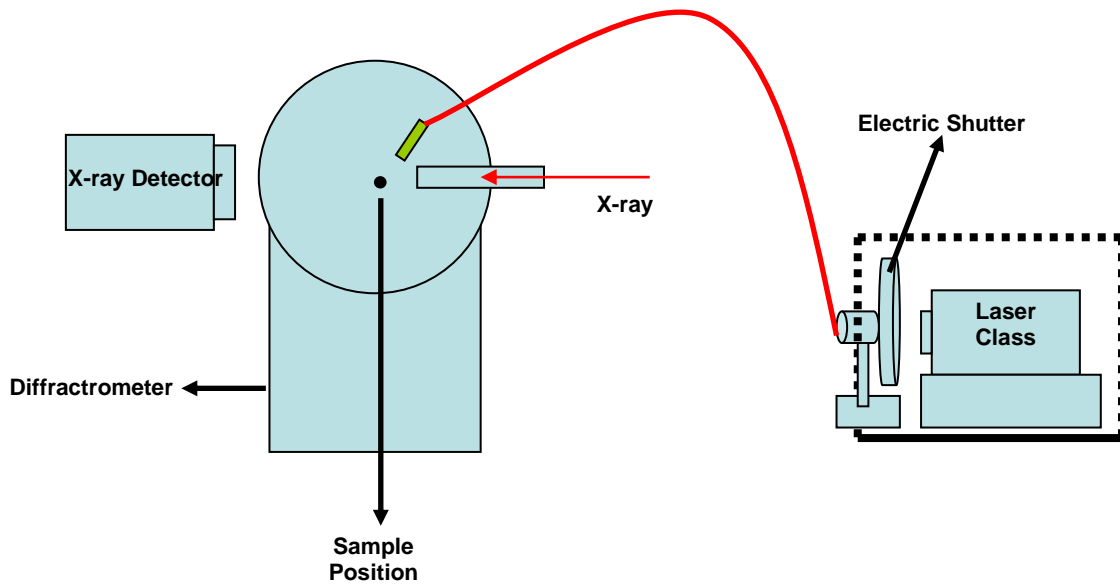
## 12. Operating procedures

The following operating procedure will be followed:

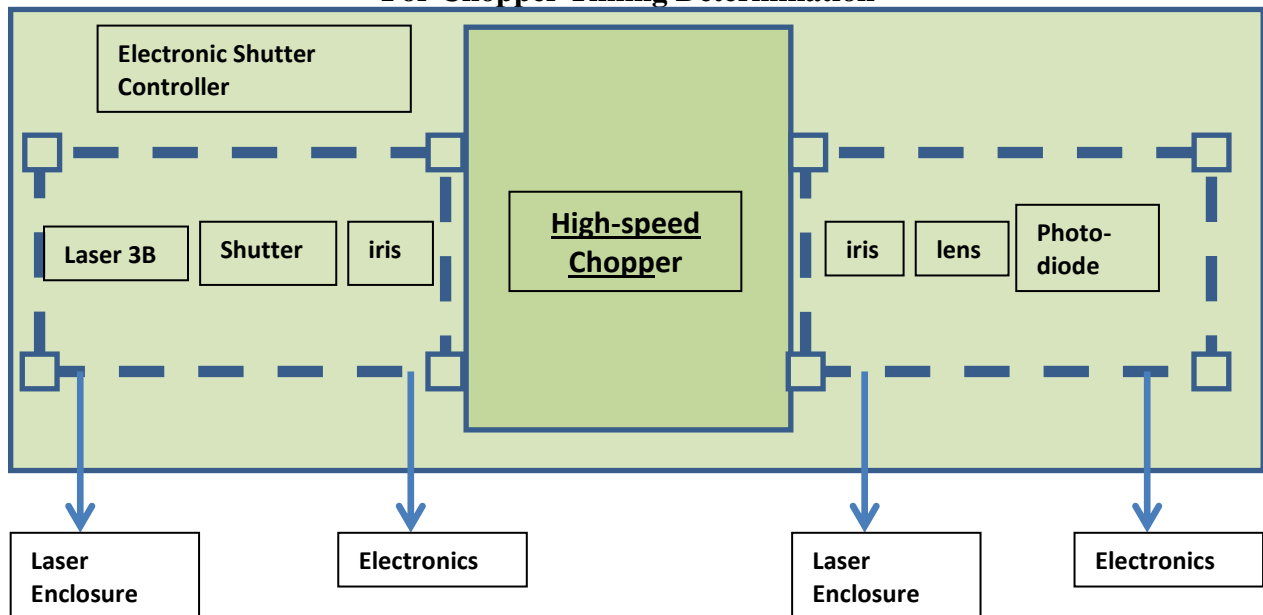
- Check that the door is properly positioned (make sure the magnetic switches on both doors overlap).
- Make sure safety goggles are available.
- Make sure that people inside the LCA are on the list of authorized personnel and are wearing proper safety goggles before activating the interlock system. (The authorized personnel list is posted on the Safety Information Board for 15-ID-D)
- Follow the alignment procedure listed in Appendix C.

## Appendix A

### Layout for the use of the 4/3B Laser and RG3 LED (IHIDs 10761/10800/10860/10995/10912/10996/L0005) in the 15-ID-D station for Photo-crystallography

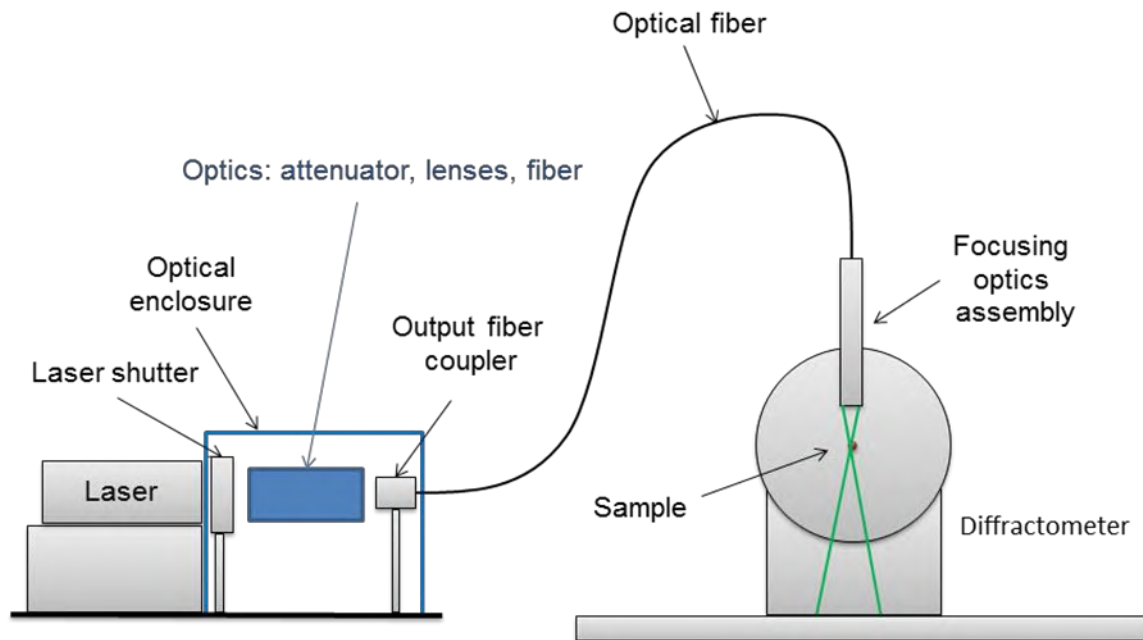


### Layout for the use of the 3B Laser (IHID 10912) in the 15-ID-D station For Chopper Timing Determination



## Layout for the use of the Class4 Laser (IHIDs 10882, 11140, 11299) New Laser in the 15-ID-D station

The Oportek Opolette ns lasers (IHIDs 10882 and 11299) and Spectra-Physics ns (IHID11140) laser are setup on a portable cart and therefore can be used in any of our LCA. A light-tight box (optical enclosure) is placed directly in front of the laser exit port, enclosing a laser shutter as the first element (see figure below), necessary optical elements (attenuator, collimating/focusing lenses, fiber) and an output fiber coupler. The set-up therefore minimizes possible specular reflections and diffuse scattering while coupling laser light into an output optical fiber. Laser light is delivered by the fiber to the sample mounted on a diffractometer. The light-delivery end of the fiber is connected to an enclosed focusing optics assembly. The light is focused at a distance of 20-30mm from the exit of the assembly and very divergent ( $>10^\circ$ ) beyond the focal spot. The assembly is mounted pointing down.



**Appendix B**

**List of authorized personnel**

I have read and understood the SOP for laser use in the 15-ID-D LCA.

<b>Person</b>	<b>Badge Number</b>	<b>Signature</b>
<b>Yu-Sheng Chen</b> (Principal Laser Operator)	<u>88772</u>	<hr/>
<hr/>	<hr/>	<hr/>
<hr/>	<hr/>	<hr/>
<hr/>	<hr/>	<hr/>
<hr/>	<hr/>	<hr/>
<hr/>	<hr/>	<hr/>
<hr/>	<hr/>	<hr/>



## Appendix C

### Laser (UV/3B/4) alignment procedures

1. Make sure the laser is in the lowest power mode.
2. Make sure that there are **NO** obstructions in the beam that might scatter the laser light.
3. Check laser shutter and internal interlock and make sure they work properly.
4. Make sure the optical fiber is in place.
5. Make sure laser enclosure box is in place.
6. Turn on the laser at low power.
7. Align the optical fiber coupler while monitoring the laser power at the exit of the fiber with a power meter to maximize the transmitted light intensity ( $<10\mu\text{J}$  at the sample).
8. With laser shutter closed, place the output end of the fiber into its mount at the microspectrophotometer.
9. With the laser power set at the minimum, align the laser beam at the sample location.
10. With laser shutter closed, place the sample at the Diffractometer and align for collection of X-ray diffraction data.
11. Following the alignments, the data collection is carried remotely at 15-ID-D control area. There is no one in the 15-ID-D station.





**Argonne National Laboratory  
Advanced Photon Source  
GeoSoilEnviroCARS**

**Standard Operating Procedures for  
Lasers in Building 434B, Room B020  
March 7, 2014**

---

Prepared by:  
LCA Supervisor

Mark Rivers \_\_\_\_\_  
630 252 0422 Date  
rivers@cars.uchicago.edu

Approved By:  
ANL-E Laser Safety Officer

Bryan Broocks \_\_\_\_\_  
Date

Approved By:  
APS AES Division ESH Coordinator

Edmund Elroy Chang \_\_\_\_\_  
Date

Approved by:  
APS AES Division Director

Bill Ruzicka \_\_\_\_\_  
Date

# GSECARS

## SOP for Lasers in Laboratory B020, Bldg 434b March 7, 2014

---

### 1. Introduction

This is a Standard Operation Procedure to operate the laser listed in Table 1 in lab B020. The laser setup is enclosed in laser enclosure interlocked according to the class IV laser requirements.

Name of LCA supervisor: Mark Rivers  
Principal laser operator: Vitali Prakapenka

**Table 1: Laser Specifications**

	<b>Fiber Laser</b>
Brand	IPG Photonics
Model	YLR-SM-100
Serial number(s)	PL0603210
ANL-E IHID	10702
Quantity	1
Wavelength	1064
Diameter	7 mm
Divergence	0.3 mrad
Power	100 W
Mode	CW
Class	IV

---

In addition to the lasers listed above, Class II solid-state laser (532 nm, <1mW) will be used for alignment purposes.

### 2. Hazards

When laser is in operation the IR laser beam is the main hazard because of its high power and the fact that it is invisible. There is no high-voltage hazard, because there will be no work performed on the power supply or electrical leads with the power on.

### 3. Controls

The following controls will be implemented in this LCA.

- A laser interlock system is used to prevent operation of the laser in an unsafe condition. This interlock system is described in detail in Section 4.
- For alignment in “Expert mode” a laser partition from Kentek will be positioned near the enclosure to prevent scattered light from escaping. The door will be roped off to prevent access of unauthorized persons during alignment.
- An interlocked enclosure completely encloses the fiber laser setup and laser beam path during normal operation. The table top enclosure consists of black sliding doors and top/side panels over an aluminum frame. When the laser light is present, all doors must remain closed, the only exception being for alignment of the optics. Such alignment can only be performed by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. **Users without ANL Laser Safety Training, eye exam, and On-the Job Alignment Training may not operate the laser with the enclosure open.**
- The keys to the laser power supplies are kept under administrative control. When the laser is not in use by qualified personnel the keys are kept in a locked drawer in the LCA Supervisor’s office.
- Appropriate eye protection will be worn by all personnel whenever the laser is operating in Expert Mode. There is a pair of goggles with OD of 6 at 1060 nm. It is not necessary to wear goggles if the enclosure is interlocked, i.e. if the system is not being used in Expert Mode.

### 3. Operation modes

The laser can be operated in 2 modes:

1. **User Mode:** The enclosure doors are closed. The laser remote control units are outside the LCA and the lasers are operated remotely.
2. **Expert mode:** This mode is used for alignment and can only be used by Authorized Users. The enclosure doors are open. A laser screen will be used to shield the area from scattered laser light. Authorized Users will wear the appropriate eye protection

### 4. Interlock System Description

#### Hardware components

The interlock system consists of the following hardware components:

- Laser Safety Interlock Panel mounted on the wall. The interlock system is based on a programmable logic controller from PLC Direct.
- Interlocked enclosure that completely encloses the laser and beam path.
- Emergency Stop button placed close to the laser enclosure.

### **Green “safe” lamps**

- When the enclosure doors are closed, the green Enclosure Closed lamp is lit
- When the laser is disabled the green Laser 1 Laser Off lamp is lit

### **“Expert mode” switch**

This is a keyed switch to enable Expert Mode. In Expert Mode exposed beams may be present in the room, and operation is only permitted by authorized personnel who have completed the ANL Laser Safety Training and completed an eye examination. This key will be under the control of the principal laser user.

### **Enable request button**

- The Laser Enable request button enables the fiber laser. This can only be enabled if the enclosure is closed, or the system is in Expert Mode. If it is safe to enable the laser, then it will be enabled and the corresponding red Laser On lamp will be lit.

The Enable button has a toggle action, i.e. pressing the enable button a second time will disable the laser or.

### **Fault Condition**

The following will result in a fault condition:

- Laser enabled, enclosure open, and key switch not in Expert Mode

A fault condition results in:

- An audible alarm
- The red Fault lamp will be lit
- Disabling of laser emission

To clear the Fault Condition it is necessary to press the Reset Fault button.

### **Panic Button**

There is red Emergency Stop button available to immediately the laser. Pressing the button will immediately remove the laser enable.

### **Operation procedures**

There are 2 allowed modes of operation for all lasers. GSECARS safety training is required for both modes. ANL laser safety training and eye exam is required for Expert Mode. Safety goggles are required for operating in Expert Mode.

#### **1) Normal operations (User Mode)**

- Enclosure is closed.
- The laser may be operated at full power.

## 2) Optics alignment (Expert Mode)

- The enclosure doors may be opened.
- The laser will be run at minimum power (<5W)
- Enclosure doors will be closed immediately after the optics alignment is complete.

Only authorized users who have received GSECARS training, read and signed this SOP are permitted to operate any of the lasers in this LCA in either mode. If such personnel have not completed ANL laser safety training, an eye exam and On-the Job Alignment Training then they are not permitted to operate the lasers in the Expert Mode.

The following checklist will be posted near the laser control units to remind operators of procedures to follow.

### User Mode

- Check that the enclosure is closed and the green Enclosure Closed lamp on the laser interlock panel is lit.

### Expert Mode

- Check that the door to the hallway is closed and the sign warning no one to enter because laser alignment is in progree.
- Wear proper safety goggles.
- Make sure that people inside the LCA are on the list of authorized personnel with ANL laser safety training and are wearing proper safety goggles before activating the interlock system. (see list posted in the hallway)
- For initial optics alignment, make sure the laser is run at low power (<20 mW).

The laser control unit must be positioned on the optical table, outside the enclosure, so the laser can be turned on and off from the operator's position.

All viewing of the sample when it is illuminated with the laser beam will be done with the video camera system, not with direct observation.

## 6. Alignment Procedures

- These alignment procedures are only to be done with the explicit approval of the LCA Supervisor or the Principal Laser Operator.
- Wear proper safety goggles.
- A Class II He:Ne or solid state alignment lasers will be used for preliminary alignment of most of the optical path. Coalignment of the He:Ne and class III-VI lasers will be performed using only minimum power (<5 W) of lasers. The path of the IR fiber laser is to be determined with the use of the near-IR laser alignment sheets. After coalignment of the He:Ne and IR lasers, the He:Ne alignment laser must be used to align the rest of the beam path.
- Following optical alignment using the He:Ne laser, it is expected that small (~0.1 mm) adjustments of the pump or fiber laser beam at the sample position will be



necessary due to dispersion in the focusing elements of the optical system. These adjustments are to be made using the minimum laser power necessary (< 5 W). For this adjustment the minimum number of panels possible will be opened on the enclosure.

- The optical design for all lasers includes no vertical beam paths.

### **7. Inspections and testing (forms to follow)**

- The interlock system will be tested annually
- The laser eyewear will be inspected annually

The interlock system tests will consist of the following.

- Set power on the laser to minimum possible value.
- Check that opening each enclosure panel results in Enclosure Closed light turning off.
- Disable Expert Mode with key switch
- Close laser enclosures. Enable laser.
- Open enclosure door slightly.
- Verify that a Fault Condition occurs, that laser enable is removed, and that the laser emission is off.
- Enable Expert Mode with key switch.
- Verify that Expert Mode light on interlock panel is lit.
- Enable laser.
- Open enclosure door slightly.
- Disable Expert Mode with key switch.
- Verify that a Fault Condition occurs, that laser enable is removed, and that the laser emission is off.

### **8. Laser training (forms to follow)**

Two lists of laser users will be maintained and posted on the hallway door

- 1) Users With ANL Laser Safety Training and On-the Job Alignment Training.
- 2) Users Without ANL Laser Safety Training



**Laser eyewear inspection**

**(To be completed annually)**

<b>Date</b>	<b>Name</b>	<b>Signature</b>



**GSECARS**  
Users Without ANL Laser Safety Training

**Signature Sheet for 434-B020 Laser Operations**

**Persons signing this form certify that :**

- 1. They have read, understood and will abide by the GSECARS Standard Operating Procedure for laser operations in laboratory 434-B020,**
- 2. They understand that they must not operate the laser in the Expert Mode**

<b>ANL Badge No.</b>	<b>Print Name</b>	<b>Affiliation</b>	<b>Signature</b>	<b>Date</b>

Argonne National Laboratory  
Advanced Photon Source  
GeoSoilEnviroCARS

GSECARS SOP for Lasers at 13 BM-C

(Modified February 5, 2018)

---

**Mark Rivers**

Name Printed

Signature

Date

Approved by:

ANL-E Laser Safety Officer or Deputy Laser Safety Officer:

**Bryan Broocks**

Name Printed

Signature

Date

ESH/QA Coordinator:

**Edmund Elroy Chang**

Name Printed

Signature

Date

Approved by:

Division Director:

**John Connolly**

Name Printed

Signature

Date

# GSECARS SOP for Lasers at 13 BM-C

## 1. Introduction

This is a Standard Operation Procedure to operate the lasers listed in Table 1 at the 13 BM-C station. The LCA includes the entire interior of the 13 BM-C station. It does not extend outside the station.

Name of LCA supervisor: Mark Rivers

Principal laser operator: Dongzhou Zhang

**Table 1: Laser Specifications**

	<b>Fiber laser</b>	<b>Green Diode Laser</b>
Brand	IPG Photonics	Laser Quantum
Model	YLR-200-AC-Y11	GEM 1000
Serial number(s)	PLMP1570413	6544099
IHID numbers(s)	11080	11110
Quantity	1	1
Wavelength	1064 nm	532 nm
Diameter	5mm	1 mm
Divergence	0.3 mrad	0.8 mrad
Power	200 W	1 W
Mode	CW	CW
Class	IV	IV

In addition to the lasers listed above, Class II green diode laser will be used for alignment purposes. The fiber laser also has a Class II red alignment laser built-in which can be operated without enabling the class IV IR laser. This red alignment laser may be used at any time, and is independent of the interlock system.

## 2. User Training

There are two classes of users for these laser systems, Authorized Users and General Users.

The following requirements must be met in order to be added to the list of Authorized Users for these laser systems:

- Completion of the Argonne laser safety training course
- Completion of an Argonne laser eye exam
- Training by the Principal Laser Operator or his/her designee, and agreement to abide by this SOP document.
- "Hands-on" on-the-job alignment training (OJAT) by the Principal Laser Operator, who serves as the LCA trainer.
- The list of authorized users is maintained by the ANL-E Laser Safety Officer.

The following requirements must be met in order to be added to the list of General Users for these laser systems:

- Training by the Principal Laser Operator or his/her designee, and agreement to abide by this SOP document.

## 3. Operation modes

The Class IV lasers can be operated in 2 modes:

1. **User mode:** The 13 BM-C station door is closed and no one is inside the LCA. The lasers are operated remotely from the 13 BM-C control area. User mode operation can be performed by General Users and Authorized Users.
2. **Alignment mode:** Authorized Users only are present inside the LCA. Such users will wear the appropriate eye protection for the laser in use. Only the green diode laser can be operated in this mode. The fiber laser can only be operated in user mode.

#### 4. Hazards

The IR beams from the fiber laser are the main hazards because of their high power and the fact that the laser beams are invisible. There are no high-voltage hazards, because there will be no work performed on the power supplies or electrical leads with the power on.

#### 5. Controls

The following controls will be implemented in this LCA.

- A laser curtain from Kentek is hung inside the door. This curtain has switches on both sides which must be engaged to enable Alignment Mode.
- A laser interlock system will be used to prevent unauthorized personnel from entering the LCA when the lasers are on. This interlock system is described in detail in Section 6.
- A sign will be posted on the 13 BM-C door which alerts people that they are about to enter an LCA and points out the laser warning light.
- The keys to the laser power supplies will be kept under administrative control. When the lasers are not in use by qualified personnel the keys will be kept in a locked drawer in the LCA supervisor office.
- Appropriate eye protection will be worn by all personnel in the LCA whenever any Class IV laser is operating. There are four pairs of goggles with OD of 5+ at 950-1600 nm and 10600 nm (for fiber laser), two pair goggles with OD>6 at 450 - 550 nm (for the Green Diode laser).

#### 6. Interlock System Description

The interlock system consists of the following hardware components:

- Laser Safety Panel is mounted on the wall inside the LCA, and connects to a PLC to implement the logic.
- A “panic” shutoff switch located in the LCA and clearly marked with a sign. This switch will turn off all class IV lasers immediately when it is pressed. This switch will be in a fixed location so that operators can find it quickly in an emergency.
- A laser warning light installed above the hutch door outside the LCA. This light contains 3 indicators:
  - “No Hazard/Laser Off” (green)
  - “Caution/Laser Energized” (yellow)
  - “Danger/Laser On” (red)
- EPICS control screens to control the operation of the lasers from inside or outside the hutch.
- A lighted Danger sign mounted on the wall inside the LCA.
- There are switches mounted on both station doors that detect when the doors are closed.
- There are switches mounted on both sides of the curtain that detect when the curtain is closed.

The interlock system has two different modes, Expert Mode, which is restricted to Authorized Users only, and User Mode, which can be used by General Users and Authorized Users. Expert mode is used only for Alignment mode described above.

Expert mode operates as follows:



- Expert mode can only be enabled if the curtain is closed and both switches engaged.
- Pressing the Laser Enable button on either the Laser Safety Panel or EPICS screen does the following:
  - Lights the appropriate Enable light on the Laser Safety Panel
  - Lights the yellow Caution light on the sign outside the LCA
  - Activates the photocell sensor which monitors access to the LCA through the door
  - Allows the green diode laser emission to be enabled

Once the interlock is turned on, the Enable buttons on the Laser Safety Panel or EPICS screen can be pressed to enable the emission of the green diode laser. Pressing these buttons does the following:

- Lights the red “Laser On” light on the Laser Safety Panel
- Lights the red “Laser On” light on the sign outside the LCA
- Lights the “Danger” sign on the wall inside the LCA
- Allows the laser emission and power to be controlled from EPICS screen

Pressing these buttons a second time disables the laser emission.

The door photo-sensor works as follows when the beam is broken:

- System is not interlocked
  - Nothing happens
- System is interlocked and override button is NOT pressed
  - Alarm sounds
  - Interlock is tripped, and must be re-enabled at the Laser Safety Panel or EPICS screen.
  - If any laser was enabled, then the fault light illuminates and must be cleared with the button on the Laser Safety Panel or EPICS screen
- System is interlocked and override button is pressed
  - Interlock remains active

User mode operates as follows:

- Hutch door must be closed. This is interlocked with a switch.
- Pressing the Laser Enable button on the EPICS screen does the following:
  - Lights the yellow interlock light on the Laser Safety Panel (not visible to users)
  - Lights the yellow Caution light on the sign outside the LCA
  - Enables the lasers, but not the laser emission
- Pressing the Emission Enable buttons on the EPICS screen enables the emission of each laser. Pressing these buttons does the following:
  - Lights the red “Laser On” light on the Laser Safety Panel
  - Lights the red “Laser On” light on the sign outside the LCA
  - Lights the “Danger” sign inside the LCA
  - Allows the laser emission and power to be controlled from EPICS screen

To re-enter the hutch in User Mode the following should be done:

1. Press the emission control buttons on the EPICS screen to turn off the laser emission
2. Open the hutch door.

If the hutch door is opened without performing step 1) above then the interlock will be disabled, the alarm sounds, and the Fault Light illuminates. The fault must be reset on the Laser Safety Panel or EPICS screen.

The panic button operates in all modes as follows:

- Disables the interlock, turning off all lasers
- Generates a fault condition. The fault which must be cleared at the Laser Safety Panel, and can only be cleared after resetting the panic button

## 7. Expert Mode operation procedures

Only Authorized Users are allowed in the LCA when the interlock is enabled or when any Class IV laser is on. Only Authorized Users are permitted to operate the photocell override button.

The following checklist will be posted near the laser control units to remind operators of procedures to follow.

Before turning on the green diode:

- Check that the curtain is closed and the switches are engaged.
- Wear proper safety goggles
- Make sure that people inside the LCA are on the list of Authorized Users and are wearing proper safety goggles before activating the interlock system. (see list posted on hutch Safety Information Board)
- Alignment of the green laser must be done at minimum laser power (3mW).

### **8. Alignment procedures**

- Alignment of IR laser
  - This laser is only to be aligned with the red guide laser. The red guide laser can be used at any time, and is independent of the interlock system.
- Alignment of green laser
  - Alignment of the green laser must be done with the laser at minimum power (3mW)
  - Wear proper safety goggles.

### **9. Inspections and testing (forms to follow)**

- The interlock system will be tested quarterly
- The laser eyewear will be inspected annually

### **11. ANL Laser training (forms to follow)**

Two lists of laser users will be maintained and posted

- 1) Authorized Users, with ANL laser safety training and “On-the Job” alignment training.
- 2) General Users, without ANL laser safety training

**Inspection Logs**

**Interlock system testing  
(To be completed annually)**

<b>Date</b>	<b>Name</b>	<b>Signature</b>

**Laser eyewear inspection  
(To be completed annually)**

<b>Date</b>	<b>Name</b>	<b>Signature</b>

## Appendix C.21 CARS SOP for Handling Carcinogens (October 2017)

### CARS Standard Operating Procedure for Handling Carcinogens

- 1. Designated Areas and Qualified Personnel**  
Unpacking and preparing the carcinogens can take place in any CARS chemistry laboratory by designated personnel listed on ESAF form.
- 2. Hazard Information Training**  
MSDSs will be posted in the designated area, and all chemical containers will bear appropriate labels. “Danger – Cancer Hazard” signs and copies of this SOP will be posted on the fume hood and all entrances to both the chem lab and the experimental hutch to provide notification of the hazards. The experimenters will understand the hazards and safety procedures prior to starting work.
- 3. Engineering Exposure Controls**  
All sample preparation will be done under the fume hood. Sample containers will remain closed when not in immediate use and will bear the required labeling. Containers will be stored on a spill tray under the fume hood. Any incidental waste will be disposed of in accordance with point 6.
- 4. Personal Protective Equipment**  
Impervious gloves, protective eyewear and facemasks will be worn for all preparation, and gloves will be worn for all additional handling (carrying to and from the experimental hutch, insertion into the beam, etc.). The fume hood will be wiped clean upon completion of the experiment.
- 5. Disposal**  
Gloves, task wipes and other miscellaneous waste will be temporarily stored in a sealed plastic bag in the fume hood, and will bear the appropriate “Cancer hazard” labels. At the conclusion of the experiment, all waste and excess sample materials will be disposed of according to ANL rules and guidelines

October 12/2017