



Canadian Light Source Inc.

Vacuum Component

Cleaning Technical Procedure

8.7.33.1, Rev. 2

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1.0 INTRODUCTION

The Canadian Light Source (CLS) is a national facility on the University of Saskatchewan Campus in Saskatoon, Saskatchewan, Canada, which is operated by the Canadian Light Source Inc. (CLSI). This facility is a 3rd generation synchrotron light source with electron beam energy of 2.9 GeV, which operates within high and ultra-high vacuum systems and produces a high intensity source of infrared, visible, ultraviolet and x-ray radiation.

This procedure will outline preparation, procedural concerns and requirements for the cleaning of the CLSI vacuum components.

Complete compliance with this procedure is expected. However, any desired deviations from the procedures outlined in this document will be reviewed and must be approved by the CLSI in writing prior to use.

2.0 PERFORMANCE REQUIREMENTS

Unless otherwise specified, completely cleaned components shall demonstrate the **outgassing rate** from in-vacuum surfaces for the major vacuum materials as following:

2.1 For the CLSI **ultra-high vacuum** (UHV) system:

Stainless Steel 304, 304L, 304LN, 316L or 316LN	- 3.0×10^{-12} Torr·l/s·cm ² or lower,
Aluminum 6061 or 6063	- 6.0×10^{-12} Torr·l/s·cm ² or lower,
Copper Oxygen-Free High Conductivity (OFHC)	- 1.0×10^{-11} Torr·l/s·cm ² or lower.

NOTE: The outgassing rate of the particular material strictly depends on selected detergents and processes used in the cleaning procedure^[6].

2.2 For the CLSI **high vacuum** (HV) system:

Stainless Steel 304, 304L, 304LN, 316L or 316LN	- 3.0×10^{-10} Torr·l/s·cm ² or lower,
Aluminum 6061 or 6063	- 6.0×10^{-10} Torr·l/s·cm ² or lower,
Copper Oxygen-Free High Conductivity (OFHC)	- 1.0×10^{-9} Torr·l/s·cm ² or lower

*Rationale: The outgassing rate, consequently, as a part of the aggregate gas load will affect the operating pressure in a vacuum volume. The requirements for the **total gas load** are given in Section 4.7 and 4.8 of the CLSI Vacuum Component Fabrication Specification 8.8.33.4.*

3.0 GENERAL REQUIREMENTS

- 3.1 The cleaning procedure must be conducted in a designated "clean area". In this area the air shall be Class 10000 of purity or better. That area must be remote from other operations, free from oils or any contaminants exhausted by machine tools, pumps, engines or any other mechanical or chemical device or process so as to limit the potential for airborne contaminants.

3.2 Personnel shall be dressed so as to prevent dust or hair shedding or other contamination of any sort and follow the Handling Requirements in Section 9 of the CLSI Vacuum Component Fabrication Specification 8.8.33.4.

3.3 Prior to cleaning, all components shall be thoroughly examined for damage. All vacuum surfaces shall be free of cracks, scale, pilling or delamination, indentations or defects of any kind. Extreme care shall be taken to protect the knife sealing edges and sealing surfaces. Any evidences of scratches, pits, crosses rolling, inclusions or other defects are not acceptable.

NOTE: It is a good practice to perform a preliminary leak test before the cleaning procedure to avoid wasted efforts on cleaning in case of defecting parts.

3.4 All components to be cleaned shall be thoroughly inspected to identify areas which may trap contamination. Small and/or blind holes (~ 3.2 mm or less), re-entrant volumes, crevices and cracks are examples of areas that must be thoroughly examined and cleaned.

Rationale: During the cleaning procedure a chemical solutions may be trapped in these small areas and cause poor vacuum performance.

3.5 Cleaning of vacuum components as an assembly is prohibited. The exception is welded assemblies, which shall be stripped down far enough to clean all surfaces properly.

3.6 If mechanical operations are required to improve the vacuum surfaces, they must comply with Section 3 of the CLSI Vacuum Component Fabrication Specification 8.8.33.4.

3.7 Marking and labeling as per Section 8 of the CLSI Vacuum Component Fabrication Specification 8.8.33.4 is required.

3.8 The vendor shall provide a cleaning certificate that identifies all implemented steps in the cleaning procedure, signed by the responsible technician. A cleaning certificate is not necessary for components where an acceptable Residual Gas Analyzer (RGA) test certificate is present in accordance with requirements in Section 4.9 of CLSI Leak Test Technical Procedure 8.7.33.3.

NOTE: The required pressure during RGA test is specified in the contract specification. If not, then the vendor shall comply with Section 2 of the CLSI Vacuum Component Fabrication Specification 8.8.33.4.

3.9 Packing, Storing and Shipping shall follow instructions in Section 10 of CLSI Vacuum Component Fabrication Technical Specification 8.8.33.4

4.0 STANDARD CLEANING PROCEDURES

Below are cleaning procedures with a number of steps to achieve the required performance in Section 2 of this procedure. If any issues arise, written clarification should be obtained from the CLSI prior to cleaning. In addition, cleaning procedures for unusually shaped parts should be discussed with CLSI prior to cleaning.

Many of the processes stipulated in this document may be subject to regulation by local health and safety legislation. The vendor or responsible individual must ensure that the

procedures are compliant with any such legislation. If such legislation prohibits the use of certain chemicals, alternative procedures or chemicals may be substituted. However, compliance with Section 2 of this procedure and Section 4.9 of CLSI Leak Test Technical Procedure 8.7.33.3 is still required.

IMPORTANT! Once the cleaning process has started, the entire process should be completed. However, if the process is prematurely terminated, parts must be thoroughly washed with water to remove any chemical residue. The parts must then be dried before covering the vacuum seal faces.

4.1 Stainless Steel Vacuum Components (304L, 304LN, 316L or 316LN)

- 4.1.1 Remove all debris such as swarf by physical means such as high pressure air blast while observing normal safety precautions. Remove gross contamination by washing, swabbing or rinsing with any general purpose solvent. If necessary, remove any scaling or deposited surface films by stripping with alumina or glass beads in a water jet in a slurry blaster.
- 4.1.2 Wash with a high pressure hot water jet (~ 80°C), using a mild alkaline non-etch sodium hydroxide detergent with a pH = 9.5 ... 11 (see Section 4.2.2 below).
- 4.1.3 Wash thoroughly with a high pressure hot water jet (~ 80°C, without detergent), to remove traces of detergent or residual beads. Pay particular attention to any trapped areas or crevices.
- 4.1.4 Dry with clean dry hot air (~ 100°C).
- 4.1.5 Immerse completely in an ultrasonically agitated bath of clean hot stabilized solvent (see Section 4.2.1 below) for at least 15 minutes, or until the item has reached the temperature of the bath, whichever is longer.

Rationale: Solvent such as trichloroethylene or perchloroethylene (where use of these substances is permitted) often is best to reduce hydrocarbon contamination to a minimum, but must be removed completely by heating.
- 4.1.6 Vapour wash in solvent vapour for at least 15 minutes, or until the item has reached the temperature of the hot vapour, whichever is longer.
- 4.1.7 Ensure that all solvent residues have been drained off, paying particular attention to any trapped areas, blind holes etc.
- 4.1.8 Wash with a high pressure hot water jet (~ 80°C), using clean demineralised water (min. res. = 1000000 Ohm/cm). Detergent must not be used at this stage.
- 4.1.9 Immerse in a bath of hot alkaline degreaser (~60°C) with ultrasonic agitation for 5 minutes. After removal from the bath proceed with the next step immediately.

4.1.10 Wash with a high pressure hot water jet ($\sim 80^{\circ}\text{C}$), using clean demineralised water (min. res. = 1000000 Ohm/cm). Detergent must not be used at this stage anymore. Ensure that any particulate deposits from the alkaline bath are washed away.

4.1.11 Dry using an air blower with clean dry hot air ($\sim 100^{\circ}\text{C}$).

4.1.12 Allow component to cool in a dry, dust free area. Inspect the item for signs of contamination, faulty cleaning or damage.

4.1.13 Vacuum bake, preferably totally enclosed in a clean vacuum oven suitable for UHV service, for a minimum period of 24 hours at 250°C .

NOTE:

1. *Prior to the bakeout cycle the vacuum shall reach a pressure of 2.7×10^{-3} Pa (or $\sim 2.0 \times 10^{-5}$ Torr) or lower.*
2. *The pumping system shall comply with Section 3.2 and 3.3 of the CLSI Vacuum Component Leak Test Technical Procedure 8.7.33.2*
3. *The bake out temperature shall be ramped up to 250°C for a period of 4 hours or more, and after bake out ramped down to room temperature for a period of 4 hours or more.*
4. *In some circumstances, the bake out temperature at 150°C is acceptable with the extended bake out period of 48 hours or more, but that is less efficient.*

4.1.14 Pack and protect as per Section 10 of CLSI Vacuum Component Fabrication Technical Specification 8.8.33.4.

4.2 Alternative Solution in Cleaning Processes of the Stainless Steel

4.2.1 Recommended ^{[1], [5]} below solvents for use in Sections 4.1.5 and 4.1.6 will be deemed acceptable:

- Trichloroethane (Triklone N™), where local regulations permit its use
- Perchloroethylene, where local regulations permit its use
- Isoproyl Alcohol or Ethyl Alcohol
- Acetone
- Axarel 9100™ (but not for 4.1.6).
- In exceptional circumstances a light chromic or citric acids (e.g. Citranox ® 10-20 ml per liter, see <http://www.2spi.com/catalog/supp/citranox-liquid-acid-cleaner.shtml>) may be carried out with the prior written agreement of the CLSI. In this case the bake out temperature in Section 4.1.13 will be required minimum at 400°C

Rationale: Using of the acid solvents (or electropolishing as well) results a significant quantities of Hydrogen diffused in the surface layers. A good bake out is required to brig surface to the normal vacuum condition.

4.2.2 Recommended ^{[1], [5]} alkaline detergents based on sodium hydroxide will be deemed acceptable for using in the cleaning procedure of the Stainless Steel:

- P3-Almeco 36™, (12 g/liter - pH = 9.7),
<http://www.hub-chemie.de/c04/sida/de/31753.pdf>
- 0.2% (by weight) Alconox® (pH = 9.5),
<http://www.2spi.com/catalog/supp/alconox-powder.shtml>
- 2.0% DeContam® (pH = 10),
<http://www.espimetals.com/metals/catdecontam.htm>
- 6.0% Oakite HD-126

4.3 Stainless Steel 304, 304L and 316L Bellows

4.3.1 Great care must be exercised when cleaning thin walled metal bellows, particularly those of edge-welded, nested construction. The trapping of solvent residue between inner or outer convolutions can result in corrosion and eventual leaking. Similarly, if any particulates are deposited in the convolutions then puncturing of the thin wall can occur.

NOTE: Alkaline degreasing solutions are prone to particulate deposition and therefore must not be used in the cleaning procedure for bellows.

4.3.2 The bellows assembly should be fixed in the extended position during cleaning if possible.

4.3.3 Remove any traces of visible, loose contamination with a jet of clean, dry air or nitrogen.

4.3.4 Immerse in an ultrasonically agitated bath of isopropyl alcohol (IPA) or ethyl alcohol (ethanol).

4.3.5 Vapor wash immediately in isopropyl alcohol or ethanol vapor.

4.3.6 Thoroughly dry the bellows inside and out using a jet of clean dry air or nitrogen.

4.3.7 Dry in an air oven at 100°C for at least 1 hour.

4.3.8 Vacuum bake, preferably totally enclosed in a clean vacuum oven suitable for UHV service, for a minimum period of 24 hours at 250°C

4.3.9 Seal under dry nitrogen in a polyethylene bag.

4.4 Copper OFHC Vacuum Components

The cleaning procedure for copper components shall follow the cleaning procedure for stainless steel with the following exceptions:

- P3 Almeco™ 36 is not acceptable

4.5 Aluminum (6061 or 6063) Vacuum Components

The cleaning procedure for Aluminum shall follow the cleaning procedure for Stainless Steel in Section 4.1 above, with the following additional precautions:

- Prolonged contact of aluminum with trichloroethylene may cause a reaction with gas generation.
- Very thorough washing with a jet of hot demineralised water is required after exposure of aluminum to trichloroethylene followed by very thorough drying.

4.5.1 The CERN specification ^[1] for LEP aluminum chambers is as follows and may be used:

4.5.1.1 Spray with high pressure jet at 60 °C with a 2% solution of Almeco 29™(the alkaline detergent).

4.5.1.2 Repeat with a 2% solution of Amklene D Forte™.

4.5.1.3 Rinse thoroughly with a jet of hot demineralised water.

4.5.1.4 Dry with hot air at 80 °C.

4.5.2 An alternative procedure known to give good results and therefore acceptable is as follows:

4.5.2.1 Immerse in Sodium Hydroxide (45 gm-1 of solution) at 45 °C for 1 - 2 min.

4.5.2.2 Rinse in hot demineralised water.

4.5.2.3 Immerse in an acid bath containing Nitric acid (50% v/v) and Hydrofluoric acid (3% v/v).

4.5.2.4 Rinse in hot demineralised water.

4.5.2.5 Dry in warm air.

4.5.2.6 Aluminum shall be baked in a clean vacuum oven suitable for UHV service for 24 hours at a temperature no higher than 150 °C.

4.6 Ceramic Vacuum Components

4.6.1 Remove any surface contamination by wet slurry blasting with alumina powder, or by hand polishing with fine-mesh alumina powder in an acetone, ethanol or isopropyl alcohol carrier.

4.6.2 Air bake at 1000°C for 24 hours.

4.6.3 Wrap in new aluminum foil and seal under dry nitrogen in a polyethylene bag.

4.7 Beryllium

Beryllium is a hazardous material. Appropriate safety precautions should be in place when working with Beryllium. Care shall be taken to ensure that no particulates are generated. No stripping, cutting, machining or abrasive operations may be carried out on beryllium except in facility designed for that purpose.

Beryllium may be cleaned in accordance with the procedures for Stainless Steel. The requirement of a baking procedure shall be discussed with and approved by the CLSI prior to commencement.

4.8 Viton[®] O-rings

Cleaning O-rings with solvents shall be scrupulously avoided. If an O-ring is suspected to be contaminated with hydrocarbons, then bake it in a vacuum oven at the temperature 180-200 °C [7]. Upon completion of the bake out process all O-rings shall be packed in the sealed plastic bag.

4.9 Assemblies and Sub-assemblies

Many assemblies will contain parts and/or sub-assemblies for which, as individual items, more than one of the above cleaning procedures would be applicable. Individual items should be cleaned according to the appropriate procedure and assembly should then take place under clean conditions. Where this is not possible, the assembly (will often be a bellows) must be cleaned and baked according to the procedure most delicate.

5.0 APPLICABLE STANDARD AND PROCEDURE

The cleaning procedure of vacuum components shall meet the following specification and procedure below. Any issue of the documents below shall be the issue in effect as of the date of request for tender. Any conflicts between these documents shall be brought to the attention of CLSI in writing for resolution before any related action is to be taken by the vendor.

Canadian Light Source Inc. Vacuum Component Fabrication Technical Specification
8.8.33.4 Rev.1

Canadian Light Source Inc. Vacuum Component Leak Test Technical Procedure
8.7.33.2 Rev.2

6.0 REFERENCES

1. "CS/05/95: Specifications for the Cleaning of Components for Clean Vacuum Service in the Synchrotron Radiation Facility, R.J. Reid (Central Laboratory of the Research Councils Synchrotron Radiation Source at Daresbury Laboratory, Warrington, Cheshire, U.K., 1995).
2. "Technical Specification for Vacuum Systems", J. Khaw, editor (Stanford Linear Accelerator Center, Stanford University, Stanford California, USA, 1987)
3. "An Introduction to the Fundamentals of Vacuum Technology", H.G. Tompkins, (American Vacuum Society Monograph, American Institute of Physics, New York, 1984).

4. "A User's Guide to Vacuum Technology", John F. O'Hanlon, John Wiley & Sons, New York, (1989).
5. "A survey of vacuum material cleaning procedures: A subcommittee report of the American Vacuum Society Recommended Practices Committee", Y. Tito Sasaki, (Quantum Mechanics Corp., 1990)
6. "Review of papers regarding vacuum system and materials", M. Wong, 21-Mar-02, http://home.fnal.gov/~mlwong/outgas_rev.htm#clean
7. Phil Danielson: "Gas Loading and O-Rings", "Gas Loading from Elastomers and Metal Seals" <http://www.vacuumb.com/articles.htm>