
**CCLRC Daresbury Laboratory**

# Vacuum Science Group

# Vacuum Systems

## Procedures for the Cleaning of Vacuum Items

**R J Reid**

CCLRC Daresbury Laboratory
Warrington WA4 4AD

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# 1. Introduction

This document is in large measure a reissue of the CLRC document DL/CS/05/95 (Issue 2) with minor changes to the context and introductory material. Some small updates have also been made. It is intended as an interim document, since the use of chlorinated hydrocarbon solvents as a primary cleaning agent is, at the time of writing (late 2003), a matter of debate on safety grounds. The possibility of substitutes for use in the demanding application of a synchrotron source is the subject of an active research programme at Daresbury Laboratory. When that programme is complete, this document will be revised and reissued to take account of its findings.



# 2. Definitions

Reference should be made to ASTEC-VAC-QCD-spc-001 *Definitions relevant to Quality Control Documentation* for the definitions of various terms used in this document.



# 3. Health and Safety

Many of the chemical agents and processes described in this document are subject to control or regulation under various parts of Health and Safety Legislation or Regulations. Any persons or Companies implementing all or any part of this Specification must satisfy themselves that they are conversant with and are adequately implementing any such Legislation or Regulations. They must ensure that they and their staff are fully conversant with all safety documentation issued by the suppliers of any such chemical agents or any equipment using such chemical agents and are fully conversant with the requisite safety precautions and personal protective equipment required. ASTeC cannot be held responsible for any consequences of the use of any chemical agent or process called in this document except where such use is under their direct control.

# 4. Comments on this Specification

Three overriding criteria have been applied in drawing up this specification.

The first is the considerable body of experience which CLRC Daresbury Laboratory (and other such laboratories world-wide) has accumulated regarding the cleanliness required for vacuum vessels particularly for the stringent requirements of Synchrotron Light Sources.

The second is the applicable parts of Environmental Protection Legislation. In recent years this legislation has greatly restricted the types of chemical solvents which are permitted for metal cleaning purposes. It has also greatly increased the precautions to be taken in working with many permitted solvents.

The third is that there is at present some variation in what is permitted in some countries and not in others.

The overall effect is that this specification is rather complex and demands careful reading to determine which parts are applicable in any given situation.

# 5. General Procedures

## 5.1 Inspection

5.1.1 All components which are received for cleaning must be examined for damage before starting any operations. Particular attention should be paid to knife edges and other sealing faces to ensure that there are no scratches, pits or other defects which could cause vacuum leaks. This should be repeated after each phase of cleaning.

5.1.2 Components should be carefully inspected so that any areas which might form potential contamination traps, e.g. blind holes, re-entrant volumes, crevices or cracks, are identified so that particular attention can be paid to ensuring that they are satisfactorily cleaned out.

## 5.2 Mechanical Operations on Vacuum Surfaces

5.2.1 Abrasive techniques to clean or to attempt to improve the appearance of the surfaces of vacuum components must be kept to an absolute minimum and are preferably avoided. The use of grinding wheels, wire brushes, files, harsh abrasives, sand, shot or dry bead

blasting, polishing pastes and the like is prohibited under normal circumstances and without the prior permission of ASTeC.

5.2.2 Permitted techniques are slurry blasting with alumina or glass beads in a water jet; gentle hand use of a dry fine stone or a fine stone lubricated with isopropyl alcohol or ethanol; hand polishing using fine mesh alumina in an isopropyl alcohol or ethanol carrier on a lint free cloth; hand polishing with ScotchBrite™ (Alumina loaded, Grade A).

5.2.3 If any such surface finish technique is employed, care must be taken that any powder or other residues are removed by copious washing in hot water.

5.2.4 Any other operations may be carried out only with the prior approval of ASTeC.

**5.3 Use of acids**

5.3.1 Acid treatment of any sort is normally prohibited and may only be carried out with the specific prior agreement of ASTeC. Most acid treatments are for cosmetic purposes only and may result in degradation of vacuum performance.

5.3.2 Components must not be “pickled” in acid baths.

5.3.3 If in exceptional circumstances acids are permitted, then exposure of the component must be kept to a minimum and must be followed by copious washing in hot demineralised water.

**5.4 Treatment of Weld Burn**

5.4.1 One particular use of acid pastes is in the removal of weld burns. In general such burns do not affect vacuum performance and are best left alone. Any scaling must be removed using the techniques of Section 5.2 above.

5.4.2 If it is desired to remove burns, then slurry blasting with alumina in water or hand burnishing with alumina powder is a satisfactory alternative. Heavy abrading, grinding or wire brushing is prohibited. Hand finishing with ScotchBrite™ or a dry stone is also permissible.

**5.5 Marking and Labelling**

5.5.1 Marking out or marking for identification should be carried out only with clean, dry scribers, vibrating engravers or laser engraving, never by acid etching or marker pen. Vacuum surfaces should only be marked if it is essential so to do.

5.5.2 Labels for identification purposes preferably should be tied to components or, in the case of small components, fixed to packing bags. Self-adhesive labels, tapes, etc. if essential, may only be fixed to non-vacuum surfaces of components and care should be taken to ensure that the adhesive used is soluble in acetone.

5.5.3 Marking for identification purposes should be laser engraved or dry-scribed at the position identified on the appropriate drawings. Where this is impractical, these should take the form of an engraved metal tag securely wired to the fixing point identified on the appropriate drawings.

**5.6 Handling and Packing**

5.6.1 Once components have completed the initial rough cleaning (e.g. as in 6.1 and 6.2 below) care should be taken that vacuum surfaces are never touched by bare skin. Gloves should always be used when handling components. Care should be taken in selecting such gloves. Polyethylene or natural vinyl is to be preferred. Coloured gloves should be tested to ensure that the dyes do not leach out when exposed to the solvents used. Gloves with talcum powder, chalk or other powders inside should be avoided since the powder can migrate into components. A good solution is to use lint-free fabric gloves inside polyethylene gloves.

5.6.2 Once components have started the cleaning process they should complete the cycle without a break. If it is unavoidable that a delay occurs between stages, then care must be exercised that the component is thoroughly dry before storage, and all seal faces and ports must be covered as in 5.6.3 below. There must never be a break between any chemical cleaning stage and a subsequent water washing stage.

5.6.3 After the component has been cleaned and is completely dry, it must be packed carefully to ensure that it remains clean and free from damage. Protect all seal faces and/or knife edges with clean used metal gaskets where possible; cover all ports with strong clean new aluminium foil and plastic covers. Small items should be wrapped in clean aluminium foil and sealed in a polyethylene bag, under dry nitrogen if possible.

## 6. Standard Cleaning Procedure for Stainless Steel Components

### 6.1 Preclean

Remove all debris such as swarf by physical means such as blowing out with a high pressure air line, observing normal safety precautions. Remove gross contamination by washing out, swabbing or rinsing with any general purpose solvent. Scrubbing, wire brushing, grinding, filing or other mechanically abrasive methods may not be used (see 5.2 above).

### 6.2 Wash

6.2.1 Wash in a high pressure hot water (approx. 80$^{o}$C) jet, using a simple mild alkaline detergent. Switch off detergent and continue to rinse thoroughly with water until all visible traces of detergent have been eliminated.

6.2.2 If necessary, remove any scaling or deposited surface films by stripping with alumina or glass beads in a water jet in a slurry blaster.

6.2.3 Wash down with a high pressure hot (approx. 80$^{o}$C) water jet, with no detergent, ensuring that any residual beads are washed away. Pay particular attention to any trapped areas or crevices.

6.2.4 Dry using an air blower with clean dry air, hot if possible.

### 6.3 Chemical Clean

6.3.1 Immerse completely in an ultrasonically agitated bath of clean hot stabilised trichloroethylene (Triklone N™) for at least 15 minutes, or until the item has reached the temperature of the bath, whichever is longer.

6.3.2 Vapour wash in trichloroethylene vapour for at least 15 min minutes, or until the item has reached the temperature of the hot vapour, whichever is longer.

6.3.3 Ensure that all solvent residues have been drained off, paying particular attention to any trapped areas, blind holes etc.

6.3.4 Wash down with a high pressure hot (approx. 80$^{o}$C) water jet, using clean demineralised water. Detergent must not be used at this stage.

6.3.5 Immerse in a bath of hot (60$^{o}$C) alkaline degreaser (P3 Almeco™ P36 or T5161) with ultrasonic agitation for 5 min. After removal from the bath carry out the next step of the procedure immediately.

6.3.6 Wash down with a high pressure hot (approx. 80$^{o}$C) water jet, using clean demineralised water. Detergent must not be used at this stage. Ensure that any particulate deposits from the alkaline bath are washed away.

6.3.7 Dry in an air oven at approx 100$^{o}$C or with an air blower using clean, dry, hot air.

### 6.4 Finishing

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

6.4.2 Pack and protect as in 5.6.3 above.

## 7. Alternative Cleaning Processes for Stainless Steel

It is recognised the many manufacturers will be unable to comply in detail with sections 6.3.1, 6.3.2 or 6.3.5 above of this specification. The following alternative chemicals will be deemed acceptable, but must be agreed in advance in writing by ASTeC. Other procedures may also be acceptable and the use of these must also be agreed in advance between the manufacturer and ASTeC.

### 7.1 Replacement solvents for sections 6.3 and 6.3.2 above:

7.1.1 Trichloroethane, where local regulations permit its use

7.1.2 Perchloroethylene, where local regulations permit its use

7.1.3 Isopropyl Alcohol

7.1.4 Ethyl Alcohol

7.1.5 Acetone

7.1.6 Axarel 9100™ (but not for 6.3.2 above)

In exceptional circumstances the use of only one or two of the procedures 6.3.1, 6.3.2 or 6.3.5 above might be deemed acceptable provided prior permission is granted by ASTeC.

## 8. Cleaning Procedures for Copper Components

In general, copper is cleaned in the same manner as stainless steel, except that Almeco P3-36™ is not acceptable in this case.

In exceptional circumstances a light chromic acid or citric acid etch may be carried out with the prior agreement of ASTeC. The provisions of section 5.3 above must be observed.

## 9. Cleaning Procedures for Vacuum Bellows

### 9.1 General

Great care has to be exercised when cleaning thin walled metal bellows, particularly those of edge-welded, nested construction. If any solvent residues are trapped between the convolutions, either inside or outside, these can result in corrosion which can rapidly cause leaks to develop. Similarly, if any particulates are deposited in the convolutions, mechanical puncturing can take place. Alkaline degreasing solutions are prone to particulate precipitation and therefore must not be used for bellows assemblies.

### 9.2 Procedure

9.2.1 The bellows should be fixed in an extended position if possible.

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

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

9.2.4 Vapour wash immediately in isopropyl alcohol or ethanol vapour.

9.2.5 Thoroughly dry the bellows inside and out using a jet of clean, dry, particulate free air or nitrogen.

9.2.6 Rinse in acetone or ethyl alcohol.

9.2.7 Thoroughly dry the bellows inside and out using a jet of clean, dry, particulate free air or nitrogen.

9.2.8 Dry in an air oven at 100$^{o}$C for at least 1 hour.

9.2.9 Bake, preferably by total immersion in a vacuum oven, for 24 hours at 250$^{o}$C.

9.2.10 Seal under dry nitrogen in a polyethylene bag.

## 10. Knitmesh™

### 10.1 General

'Knitmesh' is a loose woven copper braid used for RF screening purposes.

### 10.2 Procedure

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

10.2.2 Wash in any general purpose solvent.

10.2.3 Dry in a jet of clean dry air or nitrogen.

10.2.4 Immerse in an ultrasonically agitated bath of stabilised trichloroethylene.

10.2.5 Vapour wash immediately in trichloroethylene vapour.

10.2.6 Wash in hot demineralised water.

10.2.7 Wash in acetone or ethyl alcohol.

10.2.8 Thoroughly dry using a jet of clean dry air or nitrogen.

10.2.9 Bake by total immersion in a vacuum furnace, for 6 hours at 300$^{o}$C.

10.2.10 Wrap in clean new aluminium foil and seal under dry nitrogen in a polyethylene bag.

## 11. Cap Head Screws for RF Window Assembly

These are stainless steel screws which are silver plated to avoid sticking. They should be cleaned using the normal procedure for cleaning stainless steel (see Section 6 above) prior to plating by an electroless silver process.

## 12. Ceramics

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.

Air bake at 1000$^{o}$C for 24 hours.

Wrap in aluminium foil and seal under dry nitrogen in a polyethylene bag.

## 13. Aluminium Components

### 13.1 General

Aluminium may be cleaned by the process for stainless steel in Section 6 above, but care has to be taken. It should only be used for small components. Prolonged contact of aluminium with trichloroethylene may cause a reaction with gas generation. Very thorough washing with a jet of hot demineralised water is required after exposure of aluminium to trichloroethylene followed by very thorough drying.

### 13.2 CERN Procedure

The CERN specification for LEP aluminium chambers is as follows and this process is the preferred treatment for large vessels.

13.2.1 Spray with high pressure jets at 60$^{o}$C with a 2% solution of Almeco 29™ (an alkaline detergent).

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

13.2.3 Rinse thoroughly with a jet of hot demineralised water.

13.2.4 Dry with hot air at 80$^{o}$C.

### 13.3 Alternative Procedure

Another procedure known to give good results and which is acceptable is as follows.

13.3.1 Immerse in Sodium Hydroxide (45 g $l^{-1}$ of solution) at 45$^{o}$C for 1 - 2 min.

13.3.2 Rinse in hot demineralised water.

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

13.3.4 Rinse in hot demineralised water.

13.3.5 Dry in warm air.

## 14. Beryllium

Working with beryllium is subject to stringent safety requirements and the appropriate safety authorities must always be consulted before carrying out any such work. However, provided that care is taken to ensure that no particulates are generated and provided that suitable precautions are taken, components may be handled safely. No stripping, cutting, machining or abrasive operations may be carried out on beryllium except in purpose built facilities.

Beryllium may be cleaned in accordance with the procedures for stainless steel except that 5.2.2 and 5.2.3 above may not be used.

## 15. Assemblies and Sub-assemblies

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