IDM UID
2ELUQH

VERSION CREATED ON / VERSION / STATUS
02 Sep 2009 / 1.2 / Approved

EXTERNAL REFERENCE / VERSION

Baseline Report (not under Configuration Control)

Appendix 13 Cleaning and Cleanliness

This Appendix specifies typical processes which conform to the requirements of the ITER Vacuum Handbook for the cleaning of vacuum vessels, components and assemblies which are required for the ITER Project. This covers vacuum vessels and any item which will be in a vacuum environment, whether individually or made up into assemblies containing a number of such items.

Approval Process			
	Name	*Action*	*Affiliation*
Author	**Worth L.**	**02 Sep 2009:signed**	**IO/DG/COO/PED/FCED/VS**
Co-Authors			
Reviewers			
Approver	**Pearce R.**	**09 Sep 2009:approved**	**IO/DG/COO/PED/FCED/VS**
Document Security: Internal Use *RO: Chiocchio Stefano*			
Read Access	**GG: MAC Members and Experts, GG: STAC Members & Experts, AD: ITER, AD: External Collaborators, AD: IO_Director-General, AD: EMAB, AD: Auditors, AD: ITER Management Assessor, project administrator, RO, LG: [CCS] CCS-All for Ext AM, LG: [CCS] CCS-Section Leaders, LG: [CCS] JACOBS, LG: [CCS] CCS-Doc Co...**		

Change Log			
Appendix 13 Cleaning and Cleanliness (2ELUQH)			
Version	***Latest Status***	***Issue Date***	***Description of Change***
v1.0	In Work	27 Aug 2008	
v1.1	In Work	12 Jan 2009	
v1.2	Approved	02 Sep 2009	Minor textual changes for consistency with Vacuum Handbook

ITER Vacuum Handbook
Appendix 13

Guide to Cleaning and Cleanliness for the ITER Project

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13 Guide for Cleaning and the Cleanliness of ITER Vacuum Components

13.1 Scope

As specified in the ITER Vacuum Handbook all vacuum components to be supplied to ITER are subject to the provision of a "clean work plan" and cleaning procedures. This requirement is waived for proprietary components which are compliant with the mandatory requirements of the ITER Vacuum Handbook and are supplied to ITER with Certification of Conformity.

This Appendix specifies typical processes which conform to the requirements of the ITER Vacuum Handbook for the cleaning of vacuum vessels, components and assemblies which are required for the ITER Project. This covers vacuum vessels and any item which will be in a vacuum environment, whether individually or made up into assemblies containing a number of such items.

This guide is intended to assist the *supplier* of vacuum components to ITER in the preparation of a clean work plan and cleaning procedures for submission to ITER for *acceptance.* Following the guidance in this Appendix should help *suppliers* to achieve the requirements of the ITER Vacuum Handbook.

The *supplier* is at liberty to utilise other techniques not described in this Appendix provided that the components manufactured comply with the requirements of the ITER Vacuum Handbook.

13.2 General Cleaning Requirements

In general, all components classified as VQC1 will need cleaning to Ultra High Vacuum standards. Those components classified as VQC2, VCQ3 and VCQ4 will generally be operated in less stringent vacuum environments and will therefore not require cleaning to such rigorous standards.

However, it is the responsibility of the *supplier* to satisfy themselves that they understand fully the implications of cleaning to the requisite standard.

Any proposed deviation from the procedures and processes described in this Appendix need to be *accepted* in writing by ITER. This is particularly important where the use of any chemical product (solvent, etchant, detergent, etc.) other than those specified is proposed.

13.3 Health and Safety

Some of the chemicals or equipment used in cleaning processes may be classified as hazardous.

It is the responsibility of the *supplier* to satisfy themselves that any cleaning procedure complies fully with local legislative and regulatory standards regarding health and safety of any or all processes used and that all operatives have received the necessary training.

The *supplier* shall have the responsibility of ensuring that all staff fully understand all health and safety information issued by the manufacturer or *supplier* of any chemical or equipment to be used. Neither ITER nor any of its agents shall be held responsible for any consequences arising from the application of any cleaning process described in this handbook unless it is under their direct control.

13.4 Proprietary Items and Trademarks

Where propriety items from particular manufacturers or *supplier*s are mentioned in this specification any or all trademarks are duly acknowledged. Manufacturers or contractors are free to suggest alternative items from other manufacturers or *supplier*s provided that they are chemically identical. Any such substitutions need to be *accepted* in writing by ITER.

13.5 Design Rules for Cleanability

At the design stage for a vacuum item, careful consideration should be given as to how the item is to be cleaned. In particular, crevices, blind holes, cracks, trapped volumes, etc., should be avoided as these will act as dirt and liquid traps and it can be very difficult to remove both dirt and cleaning materials such as solvents from such areas. Fortunately, good vacuum practice regarding trapped volumes will also result in a component which is cleanable.

13.6 Initial Inspection and Preparation

Prior to cleaning any item, the following inspection should take place:

1. All vacuum flanges or covers should be removed and the item stripped down as much as is permissible, ideally to single components.
2. All items should be clearly identified by scribing a suitable identification mark on an external surface (never a vacuum surface). This identifier will often be a drawing number with component identifier or some such which is carefully recorded. Alternatively, for items which are either small and are to be exposed to a vacuum, a suitable metal label, preferably of the same material as the component and bearing a scribed identifier may be tied with clean bare wire to the component. If none of this is possible, the items should be stored in a suitable container which is marked with an identifier before and after the cleaning process. After cleaning, these items should be packed in such a way that they will not be re-contaminated by the container.
3. The item should be inspected visually to identify any possible traps, etc. (see 13.5 above) which could affect the vacuum performance of the item, taking into account the specified cleaning process and vacuum regime in which the item is to be used.
4. All vacuum sealing faces should be inspected to ensure that there is no damage to the seal area such as scratches, pitting or other defects. If the seal is of the knife edge type, the knife edge should be carefully examined for damage which could affect the sealing properties.

5. Any adhesive tape attached to surfaces of the item whether or not they are to be exposed to vacuum must be removed and any adhesive residue carefully removed with the solvent isopropyl alcohol or ethanol.
6. Any marker pen or paint or similar on any surfaces of the item whether or not they are to be exposed to vacuum should be carefully removed by scraping if necessary followed by washing with the solvent isopropyl alcohol or ethanol and rinsing in demineralised water.
7. Any threaded holes, etc., whether or not they are to be exposed to vacuum, should be examined to see if there are traces of lubricants, cutting fluids or swarf left inside. Any such should be removed carefully using brushing or blowing out with clean compressed air or nitrogen and/or washing with a suitable solvent followed by rinsing with demineralised water, taking care that no residue is transferred to a vacuum surface.

13.7 Mechanical Processes on Vacuum Surfaces

Abrasive techniques to clean or to attempt to improve the appearance of the surfaces of vacuum components should 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 certainly without prior *acceptance* by ITER.

Accepted 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).

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.

Any other such operations may be carried out only with prior *acceptance*.

13.8 Use of acids

Acid treatment of any sort is to be avoided wherever possible and may only be carried out with specific prior *acceptance* by the ITER Vacuum RO. Most acid treatments are for cosmetic purposes only and may result in degradation of vacuum performance.

Where the use of acids is *accepted*, then exposure of the component must be kept to a minimum and must be followed by copious washing in hot demineralised water.

13.9 Treatment of Weld Burn

One particular use of acid pastes is in the removal of weld burn. In general such burns do not affect vacuum performance and are best left alone. Any scaling (i.e. loose oxides) should be removed using the techniques of Section 13.7.

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 *acceptable*.

13.10 Electropolishing for VQC1 Applications

Electropolishing should only be carried out where it is necessary to produce a smooth surface for reasons of electrical discharge or field emission minimisation, emissivity or similar purposes. It is usually unnecessary from a pure vacuum point of view and indeed can be detrimental to vacuum performance.

Electropolishing should be carried out in clean polishing tanks, using fresh electrolyte.

Local electropolishing can be carried out with tampons. Fresh clean pads dipped in clean electrolyte should be used and excessive pressure should be avoided.

After electropolishing, the item should be washed with copious quantities of hot demineralised water.

If required, vacuum Items for use in Class VQC 1 may be baked to 450 ^{o}C for at least 24 hours to remove the residual hydrogen and other contaminants introduced into the surface layers by the electropolishing process.

13.11 Handling and Packing

Handling and packaging of components should be in accordance with the requirements specified in the ITER Vacuum Handbook .

Specifically:

1. Once components have completed initial rough cleaning care should be taken that vacuum surfaces are never touched by bare skin. Powder free latex or nitrile gloves (over cotton or linen if desirable) should always be used when handling components. Coloured gloves are not *acceptable*.
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 protected as below. There must never be a break between any chemical cleaning stage and a subsequent water washing stage.
3. After the component has been cleaned and is completely dry, it should be packed carefully to ensure that it remains clean and free from damage. All vacuum sealing faces should be protected with a clean metal plate or a hardboard or similar fibre free board covered with clean aluminium foil held in place by a number of bolts through the fastener holes. Knife edges should be protected with clean metal gaskets (which may have been used previously, but they should be completely free from loose oxide scale). All ports should be covered 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.

Clean conditions for the handling of vacuum components are also defined in the ITER Vacuum Handbook.

13.12 Spray washing

Where an item is cleaned by spray washing, it should be ensured that all hoses, lances, spray heads, etc are thoroughly cleaned out with clean hot water before the cleaning process starts.

Washing should start at the top of the item and the spray should be worked down to the bottom, ensuring good run-off.

13.13 Standard Cleaning Procedure for Stainless Steel Components

13.13.1 Preclean

All debris, such as swarf, should be removed by physical means such as blowing out with a high pressure air line, observing normal safety precautions. Gross contamination, e.g. greases or cutting oils, etc., should be removed by washing, swabbing and rinsing with any non halogenated general purpose solvent. Scrubbing, wire brushing, grinding, filing or other mechanically abrasive methods may not be used (see 13.7 above).

13.13.2 Wash

1. The item should be washed down using a high pressure jet of hot town water (at approx. 80°C), using a simple mild alkaline detergent. The detergent should then be switched off and the item rinsed thoroughly with hot water until all visible traces of detergent have been eliminated.
2. If necessary, any scaling or deposited surface films should be removed by stripping with alumina or glass beads in a water jet in a slurry blaster.
3. The item should be washed down with a high pressure hot demineralised water jet (at approx. 80°C), with no detergent, ensuring that any residual beads are washed away. Particular attention should be paid to any trapped areas or crevices.
4. The item should be dried using an air blower with clean dry air, hot if possible.

13.14 Chemical Clean for Stainless Steel, or similar Items, for VQC 1 application.

With the addition of the relevant safety precautions, the cleaning process below can also be applied to beryllium,

1. Where possible, the item should be immersed completely in an ultrasonically agitated bath of hot clean liquid solvent for at least 15 minutes, or until the item has reached the temperature of the bath, whichever is longer. The temperature should be the maximum specified by the *supplier* of the solvent.
2. Halogenated solvents are not permitted.

3. Suitable solvents need to be *accepted* by ITER before use. Isopropyl Alcohol, Ethyl Alcohol, Acetone, Axarel 9100™, Citrinox™, P3 Almeco™ P36 or T5161 are *accepted* for this purpose.
4. Where technically feasible, after the liquid immersion stage, the item should be immersed in the vapour of the solvent used for at least 15 minutes, or until the item has reached the temperature of the hot vapour, whichever is longer.
5. It must be ensured that all liquid residues have been drained off, paying particular attention to any trapped areas, blind holes etc.
6. The item is then be washed down with a high pressure hot (approx. 80°C) water jet, using clean demineralised water. Detergent must not be used at this stage.
7. The item is dried in an air oven at approx 100°C or with an air blower using clean, dry, hot air.
8. If the item is too large to be cleaned by immersion the item may be cleaned by washing it down with a high pressure jet of P3 Almeco™ P36 or T5161.
9. The item is cooled to room temperature in a dry, dust free area conforming clean conditions as defined in ITER Vacuum Handbook .
10. The item is inspected for signs of contamination, faulty cleaning or damage.
11. The item is baked to a temperature of 300°C or whatever other temperature has been specified for a minimum period of 24 hours at temperature in accordance with the ITER Vacuum Handbook Appendix 15
12. The item is packed and protected as in 13.11 above.

13.15 Chemical Clean for Stainless Steel or similar Items for use on VQC 2, 3 & 4 components

All items may be cleaned to the specification for items in Class VQC 1

It is also be permissible to use halogenated hydrocarbon solvents for cleaning items in these classes by analogy with 13.13 and 13.14.

For items for Class VQC 2, 3 and 4, baking will not normally be necessary with the exception of items specifically listed in the Vacuum Handbook.

13.16 Chemical Clean for Copper and Copper Alloys

Items manufactured from copper or copper alloys may be cleaned using the procedures for stainless steel, except that in this case Almeco P3-36™ is not acceptable.

Copper surfaces may alternatively be cleaned using a light chromic acid or citric acid etch, followed by thorough washing in hot, clean demineralised water.

13.17 Cleaning Ceramics

Ceramics such as alumina and beryllium oxide may be cleaned using the process described here. Other ceramics may not be able to withstand the high temperature air bake, so manufacturers specifications' must be checked.

Beryllium oxide must in no circumstances be ground or scraped except in specialist facilities.

1. Any surface contamination is removed by wet slurry blasting with alumina powder, or by hand polishing with fine-mesh alumina or diamond powder in an acetone, ethanol or isopropyl alcohol carrier.
2. Components are baked at 1000°C in atmosphere for 24 hours in accordance with Appendix 15. The maximum baking temperature may be limited by the system component materials.
3. Items are wrapped in clean aluminium foil and sealed under dry nitrogen in a sealed polyethylene bag

13.18 Cleaning of Aluminium

1. Components are sprayed with high pressure jets at 60 °C with a 2% solution of Almeco 29™ (an alkaline detergent).
2. This is be repeated with a 2 % solution of Amklene D Forte™.
3. Components are rinsed thoroughly with a jet of hot demineralised water.
4. Components are dried with hot air at 80 °C.

Alternatively,

5. Components are immersed in Sodium Hydroxide (45 g l^{-1} of solution) at 45 °C for 1 - 2 minutes.
6. Components are rinsed thoroughly in hot demineralised water.
7. Components are immersed in an acid bath containing Nitric acid (50% v/v) and Hydrofluoric acid (3% v/v).
8. Components are rinsed thoroughly in hot demineralised water.
9. Components are dried in warm air.

13.19 Air Baking

Items manufactured from stainless steel and the like may be air baked to provide a low hydrogen outgassing surface.

Note that this procedure is not suitable for materials that form a loose oxide, e.g. copper.

Items should be chemically cleaned using the procedures of 13.13 above

Items should then be heated in air at a temperature of 450 °C for a period of 24 hours in accordance with Appendix 15.

13.20 "Snow" Cleaning

A final clean after assembly of components into a large vacuum system may be achieved by the use of "snow" cleaning.

Snow cleaning uses a high velocity stream of soft microscopic particles of solid CO_2 to wash the surface and is effective for removing particulates and some organic contamination from surfaces.

Operatives undertaking this procedure must wear suitable protective clothing and personal safety equipment

The procedures used will be as specified by the *supplier*s of the equipment.

Snow cleaning will normally only be used for items to Class VQC 1, but may be used on all vacuum components.

13.21 Cleaning Procedures for Vacuum Bellows

13.21.1 General

Great care has to be exercised when cleaning thin walled metal bellows, particularly those of edge-welded, nested construction. If any cleaning 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 such as Almeco are prone to particulate precipitation and therefore must not be used for bellows assemblies.

13.21.2 Procedure for Bellows for Class VQC 1 use

The bellows must be fixed in an extended position if possible.

1. Any traces of visible, loose contamination are removed with a gentle jet of clean, dry air or nitrogen.
2. The bellows are immersed in an ultrasonically agitated bath of isopropyl alcohol (IPA) or ethyl alcohol (ethanol).
3. The bellows should be vapour washed immediately in isopropyl alcohol or ethanol vapour.
4. The bellows, including the interspace where appropriate, must be thoroughly dried inside and out using a gentle jet of clean, dry, particulate free air or nitrogen.
5. The bellows should be placed in a dry air oven at 100 °C for at least 1 hour.
6. The bellows should be baked in a vacuum oven, for 24 hours at 250 °C with the bellows interspace pumped.
7. The bellows should be sealed under dry nitrogen in a polyethylene bag.

This procedure can be used for bellows used on VQC 2, 3 & 4 systems with the vacuum bake requirement waived.

13.22 Cleanliness

13.22.1 Wipe Test for Cleanliness

Gross contamination of a vacuum component may be assessed by means of a wipe test. This may be carried out “dry” or “wet”.

Gross contamination may also manifest itself as an “oily” or “solvent-like” smell.

Note that these tests are of a somewhat subjective nature and may not be conclusive and therefore should only be used as a guide to cleanliness and as a marker for subsequent cleaning operations should the tests result in a failure of cleanliness.

13.22.1.1 Dry test

The surface of the component is wiped gently with a clean lint free cloth.

If there is any evidence of a deposit on the cloth (i.e. a stain or a change in colour) then the item should be regarded as unclean.

Similarly if the surface of the component which has been wiped shows any evidence of a change in colour or reflectivity of light, then the item should be regarded as unclean.

13.22.1.2 “Wet” test

This uses a clean lint free cloth dipped in a solvent which evaporates at room temperature, such as isopropanol, ethanol or acetone.

Appropriate safety precautions against fire hazard, breathing in of solvent fumes, eye and skin protection must be taken.

1. The cloth is dipped in the solvent which is then be allowed to evaporate in a safe manner. There should be no change in the appearance of the surface of the dry cloth.
2. The cloth is dipped in the solvent and the surface of the component is wiped gently while the cloth is still wet.
3. The solvent is allowed to evaporate from the cloth and the surface of the component until they are dry.
4. If there is any evidence of a deposit on the cloth (i.e. a stain or a change in colour) then the item should be regarded as unclean.
5. Similarly if the surface of the component which has been wiped shows any evidence of a change in colour or reflectivity of light, then the item should be regarded as unclean.

If required, the deposit on the cloth may be analysed by a suitable means to determine the chemical nature of the contamination.

13.22.2 General Test for Cleanliness

An item shall be deemed to be clean for the purposes of this Appendix provided that it meets the following criteria.

Cleanliness is defined to mean that the concentrations of "contaminants" (i.e. unwanted gas species) in the residual gas spectrum of the item are less than the specified values.

The concentration of a species is defined as the fractional intensity of its measured partial pressure components related to that species defined in a particular way to the total pressure in the system expressed as a percentage.

The partial pressures of species in the vacuum system or related to the component being measured should be obtained using the equipment and procedures defined in Appendix 17 of the Vacuum Handbook.

The residual gas spectrum will have been recorded over 1 –200 amu

The spectrum will have been corrected for sampling error, mass discrimination and species relative sensitivities.

The definition of "general contaminants" is the sum of the partial pressures of all peaks present in the residual gas spectrum of mass to charge ratio (amu) equal to 39, 41-43 and 45 and above (*excluding* any above 45 specifically listed in the table below). Also to be excluded from this summation are any peaks related to the rare gases xenon (i.e. 132, 129, 131) and krypton (i.e. 84, 86, 83)

Table 13-1 Allowed concentrations of contaminants pertaining to VQC

Vacuum Class	General Contaminants	Perfluoropolyphenylethers Sum of (peak at 69 and 77 amu)	Chorinated species (Sum of peaks at 35 and 37 amu)	Comment
VQC 4	5	1	1	Excluding water (sum of 17 and 18 amu) from the total pressure
VQC 3	2	0.5	0.5	
VQC 2	1	0.1	0.1	If unbaked, excluding water as above
VQC 1	0.1	0.01	0.01	After bake

This general test for cleanliness can be carried out as part of the verification of component outgassing in accordance with Appendix 17

13.23 Definition of Terms

For the purposes of this specification, the words or terms listed in Table 13-2 below are taken to have the stated meanings.

Table 13-2 Definitions of terms used

Term	Definition
Contaminant	Any unwanted substance present on a surface
Brushing	Using a fibre glass or wire brush to gently remove loosely adhered matter (e.g. dust) from a surface
Swabbing	Vigorous rubbing with a lint free cloth or rag
Wiping	Gentle rubbing with a lint free cloth or rag, either dry or soaked in a liquid
Washing	Cleaning an item by total immersion in a liquid or by pouring or spraying a liquid over it
Dipping	Immersing an item in a liquid and removing it relatively quickly
Rinsing	Using copious quantities of a liquid to remove traces of a contaminant or other material from an item, usually by repeated dipping or pouring the liquid over the item
Scraping	Using a hand tool of a material harder that the item being scraped to gently remove a thin layer from a surface
Grinding	Using a wheel or stone to remove a substantial amount of material from a surface
Scribing	Marking a surface with a clean metal point, vibrating engraver or laser engraving device, usually for identification or marking out purposes
Sand or shot blasting	Using a stream of abrasive particles e.g. silica or alumina to remove a surface layer. The medium may be a gas or a liquid.
Polishing or burnishing	Using a paste of fine particles, e.g. diamond or alumina, or a dry tool to produce a smooth surface
Solvent	A material which removes a contaminant from an item by dissolving it to form a solution
Detergent	A material which removes a contaminant from an item by acting as a surfactant i.e. by hydrophobic or hydrophilic action. Often used interchangeably (but incorrectly) with the term soap.
Etching	Removing a surface layer by chemical action
Pickling	Stripping of the oxide layer from a surface by use of acids
Passivation	Modifying a surface so that it is left in an inactive state, usually by leaving a uniform oxide film on the surface
Electropolishing	Removal of the surface layers of a metal by immersing the surface in a buffered acid solution and applying an electrical potential.
Ultrasonic cleaning	Immersion of a component in a bath of liquid with ultrasonic agitation

Vapour washing	Immersion of a component in a hot vapour such that the vapour condenses on the item and runs off by gravitation, carrying any contaminant in solution or suspension
Glow discharge	An electrical discharge set up in a low pressure gas. Discharges may use dc or radio frequency potential (voltage) sources
Clean surface	A surface with the desired properties e.g. outgassing.