CCLRC Daresbury Laboratory

Vacuum Science Group

Vacuum Systems

Leak Testing of Vacuum Vessels and Assemblies for ASTeC

R J Reid

CCLRC Daresbury Laboratory
Warrington WA4 4AD

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1. Introduction

This specification outlines the leak test procedure required for ultra high vacuum components to be used on ASTeC.

The phrase “test piece” as used in this document shall be taken to mean a single vacuum vessel or assembly of vessels and components as appropriate.

The phrase “clean vacuum” shall be taken to mean one in which the levels of contaminant species, for example hydrocarbons, perfluoropolyphenylethers, etc., are less than some specified set of criteria. Unless otherwise stated the criteria shall be taken to be those stated in ASTeC specification ASTEC-VAC-QCD-spc-005 *Acceptance Tests for Vacuum Vessels, Components and Assemblies*.

2. Definitions

Please refer to ASTEC-VAC-QCD-spc-001 *Definitions relevant to Quality Control Documentation* for definitions of various terms used in this document

3. Cleaning

Before carrying out any of these procedures, all items under test shall have been cleaned to ASTeC Specification ASTEC-VAC-QCD-003 *Procedures for the Cleaning of Vacuum Items* or equivalent as agreed.

4. Precautions

4.1 Dye Penetrant

Dye penetrant techniques and solvent ingress techniques are strictly prohibited.

4.2 Oils and Greases

Oil or grease shall not be used on any vacuum surface or seal. Care shall be taken that external surfaces are also free from oils, greases and solvent residues that could block porosity in wall surfaces.

4.3 Marking and Labelling

4.3.1 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.

4.3.2 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.

4.3.3 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.

4.4 Handling

The wearing of nylon or other lint-, powder- and lubricant-free fabric gloves is imperative when handling vacuum surfaces, gaskets, vacuum weld regions, etc.

4.5 Joints

4.5.1 Any internal vacuum joints shall be made off using clean, vacuum approved fasteners and clean tools.

4.5.2 Unless specifically agreed in writing prior to the tests, all flanges on the test piece shall be fitted with an appropriate metal gasket and all fasteners shall be tightened in such a manner that the flanges do not distort. Opposite flange faces in a flange pair should touch uniformly after tightening.

5. Equipment

5.1 General Vacuum Equipment

5.1.1 All items exposed to the test piece or to the high vacuum side of any pump set shall have been cleaned to ASTeC Specification ASTEC-VAC-QCD-003 *Procedures for the Cleaning of Vacuum Items* (or equivalent as agreed) and vacuum baked to at least 200^{o}C before use.

5.1.2 All connections between the test piece and any test equipment shall utilise metal pipework. Any flexible connections shall be made using metal bellows, either rolled, hydroformed or edge welded. All demountable joints shall be sealed with metal gaskets.

5.1.3 All valves used to isolate pump sets and/or measuring stations from the item under test shall be all-metal.

5.1.4 An all-metal vent valve, connected to a dry nitrogen supply (Dew point < -70^{o}C), shall be installed, either directly on the test piece or on the pipework connecting the high vacuum side of the pumping station to the test piece and close to that item.

5.2 Rough Pumps

5.2.1 No oil sealed pumps shall be permitted to be exposed directly to the item under test.

5.2.2 A rough pumping system comprising clean pumps or combinations of clean pumps approved for the purpose by ASTeC and capable of evacuating the item under test from atmospheric pressure to a pressure better than 10^{-3} mbar in a reasonable time shall be used.

5.2.3 Clean pumps which may be used include, among others, –

5.2.3.1 Cryosorption pumps cooled to liquid nitrogen temperature or lower

5.2.3.2 Diaphragm pumps utilising Viton™ or PTFE diaphragms

5.2.3.3 Scroll pumps with hermetically sealed bearings

5.2.3.4 Piston pumps with hermetically sealed bearings

5.2.3.5 Magnetically levitated turbomolecular and drag pumps with dry crash bearings.

5.2.4 Section 5.2.3 above is not an exhaustive list of acceptable pumps and simply comprises the main technologies available at the time this specification was last revised. Other types of pump may be acceptable for use, subject to the prior agreement of ASTeC.

5.2.5 In all cases, a residual gas spectrum of the rough pump set shall be recorded before opening the valve to the test piece. The rga shall sample (either directly or through a sampling stage) the residual gas in the connection line between roughing pump and test piece. This rga scan shall satisfy the definition of cleanliness to be found in ASTeC specification ASTEC-VAC-QCD-spc-005 *Acceptance Tests for Vacuum Vessels, Components and Assemblies*.

5.3 High and Ultra High Vacuum Pumping Equipment

5.3.1 Such equipment shall comprise clean pumps connected to the test piece by tubulation of suitable dimensions to provide adequate pumping speed into the test piece.

5.3.2 Where displacement pumps, e.g. turbomolecular pumps, are used, an all-metal valve of suitable size shall be provided close to the test piece to isolate the pump set from the test piece as required. Such an isolation valve shall not be required if capture pumps, e.g. sputter ion pumps are used, but may be fitted at the discretion of the manufacturer or other person carrying out these tests.

5.3.3 Suitable pumps which may be used include, among others, -

5.3.3.1 Magnetically levitated turbomolecular pumps with dry crash bearings backed by a clean pump (such as those described in 5.2.3 above)

5.3.3.2 Magnetically levitated wide range turbomolecular pumps with dry crash bearings backed by a clean pump (such as those described in 5.2.3 above)

5.3.3.3 Sputter ion pump, with or without NEG or TSP. In this case, care should be taken to ensure that there is sufficient pumping speed available for the test gas so that it can be "cleared" from the system in a reasonable time.

5.3.4 Section 5.3.3 above is not an exhaustive list of acceptable pumps and simply comprises the main technologies available at the time this specification was last revised. Other types of pump may be acceptable for use, subject to the prior agreement of ASTeC.

5.3.5 In the case where displacement pumps are used (or of a capture pump with an isolation valve), a residual gas spectrum of the pump set shall be recorded before opening the valve to the test piece. The rga shall sample the residual gas between the high vacuum side of the pump set and up to the isolation valve to the test piece. When capture pumps are used without an isolation valve, this spectrum shall be taken of the gas in the test piece with the pumps operating. This rga scan shall satisfy the definition of cleanliness to be found in ASTeC specification ASTEC-VAC-QCD-spc-005 *Acceptance Tests for Vacuum Vessels, Components and Assemblies*.

5.4 Leak Detection Instrumentation

5.4.1 Two methods of leak rate determination are permitted under this specification and a third may exceptionally be used, but only where specifically accepted by ASTeC for an individual contract.

5.4.2 The first approved method (Method 1) uses a "dry" cabinet or portable mass spectrometer leak detector unit ("leak detector"). Such units are commercially available from a number of manufacturers. Both direct and contra-flow types of instrument are permissible. The key item is that all the pumps in the unit (and any ancillary pumps to be used) meet the criteria described in Sections 5.2 and 5.3 above. The manufacturer's instructions for the operation and use of such a leak detector shall be adhered to rigidly during these tests. It is strongly advised, however, that the auto vent valve fitted to many such units is not used and the vent valve described in Section 5.1.4 above is used to vent the test piece after completion of the test.

5.4.3 The second approved method (Method 2) uses a residual gas analyser ("rga") to monitor the partial pressure of a test gas in the test piece. The rga head shall be fitted either directly to the test piece or to the high vacuum pump connection either above, or just below the isolation valve. (This may be the same rga which is used to monitor pump set cleanliness.) In either case, provision must be made for the rga head and the adjacent pipework to be baked to a temperature of at least 200^{o}C.

5.4.4 The third (non approved) method (Method 3) uses a conventional mass spectrometer leak detector fitted with standard vacuum pumps. Such leak detectors carry a risk of contaminating the test piece. To minimise this risk, the leak detector shall be connected into the fore-vacuum region of a turbomolecular pumping set (i.e. between turbomolecular pump and backing pump) using a suitable isolation valve. The auto vent valve fitted to many such leak detectors must not be used, and should preferably be disabled. The vent valve described in Section 5.1.4 above must be used to vent the test piece after completion of the test.

In this case, the turbomolecular pump may be one of those described in Section 5.3 above, or it may use greased bearings. **It shall <u>not</u> be oil lubricated**. A fully automatic isolation valve shall be fitted at the high vacuum side of the pump. This valve shall close in the event of any one of

5.4.4.1 failure of the turbomolecular pump or of the backing pump (whether this pump is a separate pump or one included in the leak detector)

5.4.4.2 mains electrical failure

5.4.4.3 pressure rise in the backing line to a value close to the critical backing pressure of the turbomolecular pump

5.4.5 All such incidents occurring during any test carried out under this specification for ASTeC must be recorded and reported in writing to ASTeC.

5.5 Reference Leak

5.5.1 A suitable standard leak or calibrated source of the test gas to be used ("reference leak") shall be provided so that the sensitivity of the leak detection system can be determined for the gas in question.

5.5.2 The reference leak shall have a leak rate commensurate with the specified maximum permissible leak for the item or system under test. (For example, if the maximum permissible leak rate specified is 1.10^{-9} mbar l sec^{-1}, then the reference leak should have a value between 1.10^{-10} mbar l sec^{-1} and 1.10^{-8} mbar l sec^{-1}.) The calibration of this reference leak shall be traceable to national standards and copies of such certification shall be provided to ASTeC.

5.5.3 If a mass spectrometer leak detector is used and this has a pre-fitted reference leak incorporated, this may be used provided traceability and certification is available.

5.5.4 The reference leak shall be fitted directly to the input flange of the leak detector by means of a tee-piece with all metal seals. The other branch of the tee-piece shall lead, via a suitable all-metal valve and all-metal tubulation, flexible or otherwise, to the test piece, again using all-metal seals.

5.5.5 Otherwise the reference leak shall be fitted to the test piece through an all-metal isolation valve using metal seals on the test piece side of the valve.

6. Procedures

6.1 General

6.1.1 Unless otherwise agreed in writing by ASTeC, the test piece shall have been baked under vacuum to a temperature of at least 250°C for a minimum of 24 hours before final leak test. The pumping system used for evacuation during such bakeout shall be of the same type as that to be utilised for this test procedure. Preliminary leak tests may be carried out before bakeout.

6.1.2 The item under test shall be evacuated to a pressure better than 10^{-3} mbar using the pumping system specified in 5.2 above.

6.1.3 The high or ultra high vacuum pumping system, having met any requirements specified in section 5.3 above, shall be used to evacuate the item under test to a pressure suitable for operation of the particular instrument being used for leak detection.

6.2 Probe Gas

6.2.1 The probe gas to be used for leak testing shall preferably be helium, but other gases detectable by the leak detector may be used, subject to the prior agreement of ASTeC in writing, and provided a reference leak for that gas is available.

6.3 Calibration of a Leak Detector

6.3.1 The sensitivity of the leak detection system shall be checked with the reference leak as follows.

6.3.1.1 Where a mass spectrometer leak detector is used and is calibrated in terms of a leak rate (e.g. mbar l sec^{-1}) then the indicated leak rate on the leak detector and the specified leak rate of the reference leak should differ by no more than ± 10% (making due allowance for the temperature and age of the reference leak) after at least a 5 minutes exposure of the reference leak to the leak detector input or until such time as the reading on the leak detector is stable as in 6.3.1.3 below.

6.3.1.2 If the leak detector has a pre-fitted reference leak then the procedure to be used shall be that determined by the manufacturer of the equipment

6.3.1.3 If a separate reference leak attached to the test piece is used, then the test piece shall be pumped to a suitable pressure as in 6.1.3 above and the valve to the reference leak opened. Once the indicated leak rate on the leak detector is stable

to within ± 1 division on the meter or ± 1 least significant digit on the display, then the reading may be compared with the reference leak value.

6.3.1.4 If the value of the indicated leak rate differs from the value of the reference leak by more than ± 10% (taking into account the temperature and age of the reference leak), then the leak detector must be adjusted following the manufacturer's procedure until this criterion is satisfied.

6.4 Calibration of a Residual Gas Analyser as a Leak Detector

6.4.1 Where a residual gas analyser is used, then the following procedure shall be used to calibrate the leak rate.

6.4.2 The rga shall be baked to at least 200^{o}C while the test piece is being baked.

6.4.3 While the test piece is cooling down at the end of the bakeout, once the temperature is between 150^{o}C and 100^{o}C and the pressure in the rga is less than 1 x 10^{-6} mbar, the rga shall be thoroughly degassed according to the manufacturer's instructions. After degas it shall remain operational until the end of the test sequence.

6.4.4 When the test piece and all the adjacent pipework have cooled to room temperature, the isolation valve to the standard leak shall be opened and the rga tuned to the standard leak gas.

6.4.5 After a minimum of 5 minutes pumping, the isolation valve to the standard leak shall be closed and the test piece pumped until the indicated partial pressure of the test gas is at least one decade below the value when the standard leak was open to the system.

6.4.6 Note that it might be necessary in some circumstances to reduce the pumping speed applied to the test piece to achieve this condition (e.g. when the value of the reference leak is small). Such a reduction shall be achieved by partially closing the isolation valve to the pump set where fitted. In this case, the position of this valve must not be altered until the test sequence is complete.

6.4.7 If the pumping system described in Section 5.3.3.3 is used and there is no isolation valve, then the pumps may need to be switched off. This will be satisfactory only for small leaks where the rate of rise of the test gas due to its presence in the atmosphere surrounding the test piece is sufficiently low that a reasonably stable base partial pressure of the test gas may be obtained and where the outgassing of the test piece is sufficiently low that the pressure does not rise above the safe operating pressure of the rga.

6.4.8 Whilst recording the partial pressure of the test gas, open the valve to the reference leak. When the recorded value is stable to within ± 2% over a period of 20 minutes, then record this partial pressure as the reference pressure (pr).

6.4.9 Close the valve to the reference leak and wait until the partial pressure of the test gas has fallen by at least 1 decade and is stable to with ± 2% over a period of 20 minutes. Record this partial pressure as the base pressure (p_0).

6.5 Carrying out the Leak Test

6.5.1 The test shall be carried out by spraying all joints, welds and surfaces with the probe gas, ensuring sufficient dwell time of the gas around the surface for porosity to be detected. This may be most conveniently achieved by enclosing the item under test in a polythene bag or tent and pressurising this with the probe gas.

6.5.2 If a pressure rise of the test gas is detected, then the relevant leak rate shall be recorded.

6.5.2.1 If a leak detector is used, this shall be the indicated reading on the meter or display.

6.5.2.2 If the rga method is used, then record the partial pressure of the test gas, p_l. The leak rate Q_l is then given by

$$Q_l = Q_r * \frac{(p_l - p_0)}{(p_r - p_0)}$$

where Q_r is the value of the reference leak.

6.5.3 If any leaks greater than the specified maximum leak rate are detected, approval for any remedial work necessary shall be obtained in writing from ASTeC prior to its being carried out. Such permission will not be required if simple tightening or remaking without rework of demountable joints cures the leak.

6.6 Post Test Checks

6.6.1 Following a final test in which the leak rate is found to be within specification, the procedure described in Section 6.3 above shall be repeated, to ensure that the equipment is still functioning satisfactorily. Repeatability to within ± 5% shall be deemed to be satisfactory. If this limit is exceeded, the entire test procedure shall be repeated.

6.6.2 Residual gas analysis shall be used as a general monitor of final system cleanliness and the item under test shall meet the requirements of ASTeC specification ASTEC-VAC-QCD-spc-005 *Acceptance Tests for Vacuum Vessels, Components and Assemblies*.

6.7 Venting

The item or system under test shall be vented to atmospheric pressure with dry nitrogen as described in Section 5.1.4 above.

7. Special procedures for “Hot” leak tests.

Where a specification calls for a leak test to be carried out at an elevated temperature, then the necessary modifications to the test procedures described in this specification shall be agreed between ASTeC and the manufacturer prior to tests being carried out.

8. Test Certificate

A suitable test certificate shall be completed for each item. Unless otherwise agreed this shall take the form shown in Vac/LT/Cert/001.