IDM UID
2EXDST
VERSION CREATED ON / VERSION / STATUS
08 Sep 2009 / 2.2 / Approved
EXTERNAL REFERENCE / VERSION

Guideline (not under Configuration Control)

Appendix 17 Guide to Outgassing Rates and their Measurment

Approval Process			
	Name	*Action*	*Affiliation*
Author	**Worth L.**	**08 Sep 2009:signed**	**IO/DG/COO/PED/FCED/VS**
Co-Authors			
Reviewers			
Approver	**Pearce R.**	**08 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 17 Guide to Outgassing Rates and their Measurment (2EXDST)			
Version	***Latest Status***	***Issue Date***	***Description of Change***
v1.0	In Work	27 Aug 2008	
v1.1	In Work	15 Jan 2009	
v1.2	In Work	15 Jan 2009	
v2.0	Signed	17 Feb 2009	
v2.1	Signed	02 Sep 2009	Minor textual changes for consistency with Vacuum Handbook. Refference added. Corrections to outgassing rate tables
v2.2	Approved	08 Sep 2009	Correction to cryostat outgassing figures

ITER Vacuum Handbook

Appendix 17

Guide to Outgassing Rates and their Measurement

	Name	Affiliation
Author/Editor	Liam Worth	Vacuum Group - CEP
Vacuum Responsible Officer	Robert Pearce	Vacuum Group - CEP

17.1 Scope

This Appendix is intended as a guide to the measurement of the total and partial thermal outgassing rates of materials, vacuum vessels, components and assemblies for the requirements of the ITER Project. It is intended that the guide be used to assist *suppliers* in producing outgassing test procedures to comply with the mandatory requirements of the ITER Vacuum Handbook. It also gives details on how the outgassing requirements for ITER systems have been derived.

It is envisaged that outgassing tests will normally be performed on components, parts of the component or "coupons" which have been subjected to the complete manufacturing process. Manufacturing operations which have been applied, including baking and cleaning operations, should be recorded and traceable to the coupon (where used) or to the manufactured component.

17.2 Limitations

This Appendix describes a set of procedures for the measurement of thermal outgassing from a vacuum item when used as part of the vacuum quality assurance procedures for the ITER Project. This Appendix describes the recommended procedures of the most widely used methods of measuring the outgassing rates; it does not consider all available methods. Despite this limitation, the techniques are more widely applicable and form a basis for more general good practice.

The supplier is at liberty to propose other methods of thermal outgassing measurement not described in this Appendix.

When this set of procedures is used to measure the outgassing from a component or coupon placed within a vacuum chamber, the outgassing of the chamber walls cannot usually be neglected and must be subtracted from the measured value to obtain that from the coupon. For this, an independent measurement of the wall outgassing from the empty chamber will be required – often referred to as a *blank run*.

Unless otherwise specifically indicated, outgassing measurements using these procedures will be carried out with the component under test at 100 °C

In these procedures the term *outgassing* shall be taken to mean *thermal outgassing* unless otherwise indicated.

The methods of measuring outgassing rates described in these procedures yield an average value of the outgassing rate for each surface exposed to the vacuum measurement system.

17.3 Specific Outgassing Rate

Outgassing is described in terms of the rate of desorption of gas from a vacuum surface.

The measured (or net) outgassing rate is the difference between the intrinsic outgassing rate (of the component) and the rate of re-adsorption on the surfaces of the test chamber.

The specific outgassing rate defined as the total gas load generated per unit time due to gas desorbing from a vacuum surface due to the temperature of the surface per unit area of desorbing surface. It is represented here by q_{th}. Units are $Pam^3s^{-1}m^{-2}$

Clearly,

$$Q_{th} = q_{th} \cdot A$$

Where:

Q_{th} is the total outgassing rate (Pa.m^3.s^{-1})

A is the area of the desorbing surface (m^2)

17.4 Generic Methods of Measuring Outgassing Rates

17.4.1 Rate of Rise of Pressure Method

This method of measuring outgassing rates is in principle very simple, but there are a number of considerations that need to be taken into account if the measurements are to be meaningful.

The principle of the method is that if one has a volume evacuated to a given pressure p_0 and then isolated from the vacuum pump, the specific thermal outgassing rate q_{th} is given by

$$q_{th} = \frac{V}{A} \cdot \frac{(p_t - p_0)}{t}$$

where V is the containing vessel volume

A is the total internal surface area of the desorbing surface

p_t is the pressure after a time interval t

provided that the outgassing rate is reasonably constant with both pressure (over the range $p_t \rightarrow p_0$) and the time interval t and that the temperature of the outgassing surfaces is constant.

Partial (i.e. species dependent) outgassing rates may be determined by using a calibrated gas analyser to measure the rate of rise of the partial pressure of a particular species.

What is actually measured using the rate of rise technique when the pressure remains in the high vacuum region or below, is the increase in number density of gas molecules entering the measurement volume of the “pressure” sensor. This increase can be affected by various processes, which can be classified as being either gas sources or gas sinks. A gas source is something which releases gas molecules into the interior of the vessel, and hence eventually into the measurement volume. A gas sink is something which adsorbs or absorbs a gas molecule which strikes it, i.e. it acts as a pump. This is further discussed later.

This method is quite simple to implement and requires the minimum of equipment. Since, during the measurement time the vacuum pump is valved off, there is no need to know the pumping speed (especially where the speed may be species dependent). Only one vacuum gauge is required. For absolute measurements, the gauge needs to be calibrated

for the outgassing species. Where only *relative* measurements of outgassing are required (e.g. before and after a process such as baking), provided the gauge is known to be reasonably stable in sensitivity, calibration may not be required.

No vacuum parameters of the system need to be calculated or measured, apart from the pressure.

This method works best for relatively low outgassing rates, where measurements can be take over a long time period. For high outgassing rates, the rise in pressure can be quite rapid, making time and/or pressure dependent measurements difficult.

The volume of the vessel (and all appendages) needs to be measured or calculated to a reasonable degree of accuracy.

This method is more suitable for the measurement of outgassing from vessels or assemblies rather than coupon samples, unless either the intrinsic outgassing rate of the coupon is very much higher than that of the containing vessel or the surface area of the sample is much higher than that of the vessel or both.

17.4.2 Dynamic Flow (Conductance) Method

In this method, the item being measured is pumped through a known conductance and the pressure difference across this conductance is measured. The specific thermal outgassing rate q_{th} is then given by

$$q_{th} = C \cdot \frac{\Delta p}{A}$$

where:

C is the conductance

Δp is the pressure difference across the conductance

A is the area of the desorbing surface

Partial (i.e. species dependent) outgassing rates may be determined by using calibrated gas analysers to measure the differences in partial pressure of the particular species.

The method is suitable for all but the lowest values of outgassing, since the value of the conductance can be chosen to give a sensible pressure difference. Variation in outgassing rate with time can readily be measured even when the outgassing rate is quite high or is varying relatively rapidly and the volume of the vessel is not required.

The method requires two vacuum gauges which must both be calibrated for the desorbing species for the most accurate results. Both must remain stable across the full range of measurement for the duration of the test. If partial outgassing rates are required, then two calibrated residual gas analysers (RGAs) should be fitted.

It requires the use of a pump whose speed is much larger than the conductance for all gas species.

The conductance, which is gas species dependent, must be measured or calculated to a reasonable degree of accuracy.

When measuring outgassing from coupon samples, the outgassing rate of the containment vessel must remain sufficiently stable for a blank run to yield a meaningful correction.

17.4.3 Variant Dynamic Flow Methods

One variant of this method assumes that the pressure on the pump side of the conductance is very much less than that on the sample side and so can be ignored. In this case only one calibrated gauge and one RGA is required, both situated upstream of the conductance. Good practice would require a total pressure gauge also to be fitted downstream of the conductance to ensure that the pressure conditions were being met, but this gauge need not be calibrated.

A second variant uses an arrangement of valves so that a single calibrated gauge can alternatively be exposed to either side of the conductance. This requires good linearity in the gauge and an outgassing rate which is stable over the time of measurement.

17.4.4 Weight Loss Method

The method of weight loss measurement can be used to measure outgassing rates from materials with high outgassing rates, for example organic materials.

The test consists of measuring the weight loss of a sample which has been subject to a defined thermal cycle under vacuum. The sample is placed in an effusion cell and heated. The outgassing flux is condensed on temperature controlled collectors which are placed in front of the sample. From the mass deposit on the collector the total mass loss (TML) and hence outgassing rate are derived, as function of time, and is usually expressed as %TML

The setup and procedure are described in the ASTM E595-93 standard and are widely used in characterisation of materials for use in space applications.

17.5 Sources of Errors in Measuring Outgassing

All methods of measuring outgassing are susceptible to errors which may yield misleading results. Detailed consideration should always be given to this.

17.5.1 System Effects

17.5.1.1 Vacuum Vessels and Conductance's

Either the internal volume of the outgassing measurement chamber, or the conductance between this and the pump, must be known to a reasonable degree of accuracy, dependent on the technique employed. Volumes are notoriously difficult to measure or calculate to high accuracies and are temperature dependent. In some cases (e.g. where bellows are present) they may also be dependent on the atmospheric pressure in the laboratory. Volumes will change if there are movable items present, e.g. vacuum valves.

The value of a conductance element is also temperature dependent and, more importantly, dependent on the mass of the gas species traversing the conductance. To some extent the transmission probability of gas molecules through a conductance is dependent on the size and shape of the vacuum chamber at each end.

It is usually assumed that in measuring outgassing, free molecular flow conditions prevail. This may or may not be the case and needs to be checked.

In the dynamic flow method, it is assumed that outgassing of the vacuum system downstream of the conductance does not influence what is happening in the measurement chamber upstream of the conductance.

17.5.1.2 Vacuum Gauges

The calibration of both total pressure and partial pressure gauges is non trivial and the stability of many gauges is not good. Clearly this may introduce significant measurement errors, especially in the two-gauge dynamic flow method.

17.5.1.3 Vacuum Pumps

Pumping speeds of vacuum pumps vary with the species being pumped, so for the dynamic flow method it is important to ensure that a sufficiently high pumping speed (i.e. compared to the conductance for the particular gas species) is maintained at all times.

17.5.1.4 Temperature

Some of the effects of temperature have been discussed above. However, outgassing is itself strongly dependent on temperature, so it is important that for the most accurate measurements, the entire apparatus is maintained at a constant temperature during the period in which measurements are being taken.

17.5.2 Gas Sources and Sinks

Errors in measured outgassing rates may be affected by sources of gas other than true outgassing entering the measuring volume of the gauge or gauges used. In this case an enhanced value will be measured. Likewise any pumping in the vessel for which outgassing is being measured will lead to an apparent value being measured which is lower than the true value. In extreme cases, negative values of apparent outgassing may be measured.

17.5.2.1 General Types of Gas Source or Sink

Possible sources of gas include:

- any surfaces exposed to the vacuum which release molecules by desorption other than thermal desorption or by permeation
- all joints, which tend to be areas of increased permeation
- leaks, real or virtual
- any gauge
- gas bursts from items moving in the vacuum system

Possible sinks for gas include:

- any surfaces exposed to the vacuum which can exhibit wall pumping, particularly "active" surfaces found in capture pumps even when switched off
- any gauge which can act as a pump

17.5.2.2 Surfaces as Sources

The point of the measurement is to measure thermal desorption from the surface of interest, i.e. gas molecules released by the absorption of phonons, so it is important that extraneous forms of desorption are minimised. Details are not discussed here, but it should be noted that the surfaces under investigation should not be exposed to significant fluxes of photons of wavelengths shorter than the short-wavelength end of visible or to electrons of energy greater than a few eV. It is also important that the temperature of the surfaces under test is kept constant as thermal desorption is an exponential function of temperature.

For metals at room temperature, permeation is only significant for hydrogen and even that would normally be very low unless very thin walls are present or when measuring very low outgassing rates. However it should be remembered that hydrogen is by far the dominant species in such cases and there is some debate as to whether hydrogen permeation is in fact the rate limiting step in outgassing from metals. The source of the hydrogen may be either dissolution from the bulk metal or passing from atmosphere on one side of the wall to the other. In practice, both will happen.

Glasses, plastics and elastomers may have quite large permeabilities for hydrogen, helium or water. Care must therefore be exercised when these are exposed to both atmosphere and vacuum.

A special case of thermal outgassing is evaporation or sublimation of the wall material (vapour pressure). For most normal vacuum materials, this is only a problem when measuring extremely low outgassing rates.

17.5.2.3 Surfaces as Sinks

When gas molecules strike a surface, in general they stick. They may stick for a short time before being re-emitted or they may stick for a long time. Here, the former process is ignored although it is important for the thermodynamics of the system. However, the latter process gives rise to the phenomenon known as wall pumping. In some cases this process can be enhanced by preparing a surface which is chemically active and deliberately used as a pump in, for example, a Titanium Sublimation Pump (TSP) or a Non-Evaporable Getter (NEG). A similar effect is seen when a surface is cooled to cryogenic temperatures. In normal circumstances the walls of a vacuum system are sufficiently inert that wall pumping is insignificant. However there are circumstances where this may not be the case. A surface which has been glow discharged will have had its chemistry altered somewhat and until a passivation film, usually an oxide, is formed may exhibit wall pumping. Similarly a surface where the gas concentration has been reduced by photon desorption, electron or ion desorption or high temperature thermal desorption may be sufficiently far from equilibrium to exhibit wall pumping.

It is very difficult to estimate what wall pumping speeds might be in such circumstances.

17.5.2.4 Joints

Vacuum joints can be regions of enhanced permeability, especially demountable joints using elastomer gaskets. However, welds and brazes may also be suspect. If components have been hydrogen brazed, then enhanced hydrogen outgassing may be experienced from all surfaces. Joints which have been welded using the Tungsten Inert Gas (TIG) process may exhibit enhanced outgassing of (usually) argon.

17.5.2.5 Leaks

Naturally, the presence of leaks can vitiate any sensible measurement of outgassing and so thorough leak checking of the test system is a necessity.

17.5.2.6 Moving items

When items move in a vacuum, gas molecules can be desorbed. The most common moving item in an outgassing measurement system will be a vacuum valve. These can generate significant gas bursts when moving. This can be minimised by operating them slowly and by thorough outgassing.

In practice, this is not usually very important in measuring outgassing rates. In the case of rate-of-rise measurements, the system is sealed and static. Any gas generated when the valve is closed at the start of measurement forms part of the base pressure. In the two gauge dynamic flow technique, valve states do not change during the measurement. In the variant of this technique where a single gauge is exposed successively to either side of the conductance to eliminate gauge errors, some care has to be exercised to minimise any such effects.

17.5.2.7 Gauges as Sources

Hot filament gauges are clearly potentially major sources of error in measurements of this type, since they not only run at high temperatures but will also cause local heating of the vacuum system. Enhanced outgassing will be experienced from the gauge and walls.

Cold cathode gauges are better than hot filament gauges in this respect since they operate at room temperature.

Ionisation gauges, hot or cold cathode, are also sources of x-rays, ions and electrons of sufficient energy to cause desorption when they strike surfaces. Cold cathode gauges may also generate energetic neutrals which may themselves cause desorption.

17.5.2.8 Gauges as Pumps

All ionisation gauges will act as pumps. Hot filament Bayard-Alpert Gauges typically exhibit pumping speeds of around 0.1 $l.sec^{-1}$ but this will normally be swamped by the outgassing.

Cold cathode gauges of the Penning or magnetron (whether inverted or not) type may well exhibit (net) pumping speeds of up to 1 $l.sec^{-1}$.

17.5.3 Some Practical Considerations

17.5.3.1 Minimising Errors

Clearly if one wishes to measure an outgassing rate, all of the above effects may play a part in introducing errors. Good vacuum practice will help in many cases to vitiate the worst of these. Leak testing should be carried out with a sensitivity of at least an order of magnitude better than the measured total outgassing rate. Permeation (but not of course bulk dissolution) can be reduced by surrounding the measurement chamber with a guard vacuum. Wall pumping may be reduced by waiting or by saturating the surface with an inert gas. This may of course make nonsense of what one is trying to do!

In practice, the bulk of the errors will come from the gauge. A hot cathode gauge should be mounted on a water-cooled side arm, preferably with a cooled baffle in the gauge throat. It should be well-degassed, and any pressure difference between the gauge and the measurement chamber carefully evaluated. If possible a cold cathode gauge should be used or a gauge specially designed to minimise outgassing.

The gauge head must be mounted out of line of sight of the surfaces being tested and tubulation to the gauge head should have as large a conductance as possible. As is so often the case, such requirements are to some extent contradictory so some compromise is necessary. There is not a lot one can do to eliminate the effects of gauge pumping. Recent developments using stable field ion emitters as the electron source for a Bayard-Alpert gauge may offer a good compromise for measuring low outgassing rates. The temperature effect is eliminated and gauge pumping is relatively low. Energetic electrons and X-rays are still produced however.

The most troublesome effect is gauge pumping. In many cases it is relatively easy to guess what the minimum outgassing rate to be expected from a sample might be. The surface area of the sample should then be such that the expected gas load generated is significantly greater than the gas load pumped by the measuring gauge. If this is not the case, then the measurement is not meaningful.

In some cases, where the measured pressures are within it's operating range, a suitable gauge is the spinning rotor gauge. Outgassing form this type of gauge is simply that of its rather small internal surface area and there is no pumping effect. It is best suited to rate of rise measurements.

Because not all sources of error can be eliminated, rate-of-rise measurements, for example, can only set a lower bound for the outgassing rate. It may be possible to estimate an upper bound by guessing the gauge pumping speed. If these two values are reasonably close, then the result may be meaningful. This assessment cannot be done unless a real effect, i.e. a measurable pressure rise, is obtained.

It will be apparent that in the rate of rise method, sufficient time must be allowed for the pressure to rise significantly. Initially after isolating the main pump, there will be a period when the system is not in a steady state as the various gas sources and sinks settle down, but in a well-behaved and well designed experiment, this should be relatively short and for a constant outgassing rate a log-log plot of pressure against time should yield a straight line of positive slope. For outgassing rates close to the pumping speed of the system sensible measurement times may well be of the order of hours, not minutes.

17.5.3.2 Time Zero for outgassing

As noted earlier, the outgassing rate measured is a function of the time that a surface has been exposed to vacuum (i.e. has been pumped), and an idealised characteristic is shown in Figure 17.5.3-1 (Note that no great significance should be attached to the actual values of outgassing rate shown in the figure.) It is clear that the measured value of outgassing will depend on when the measurement is made.

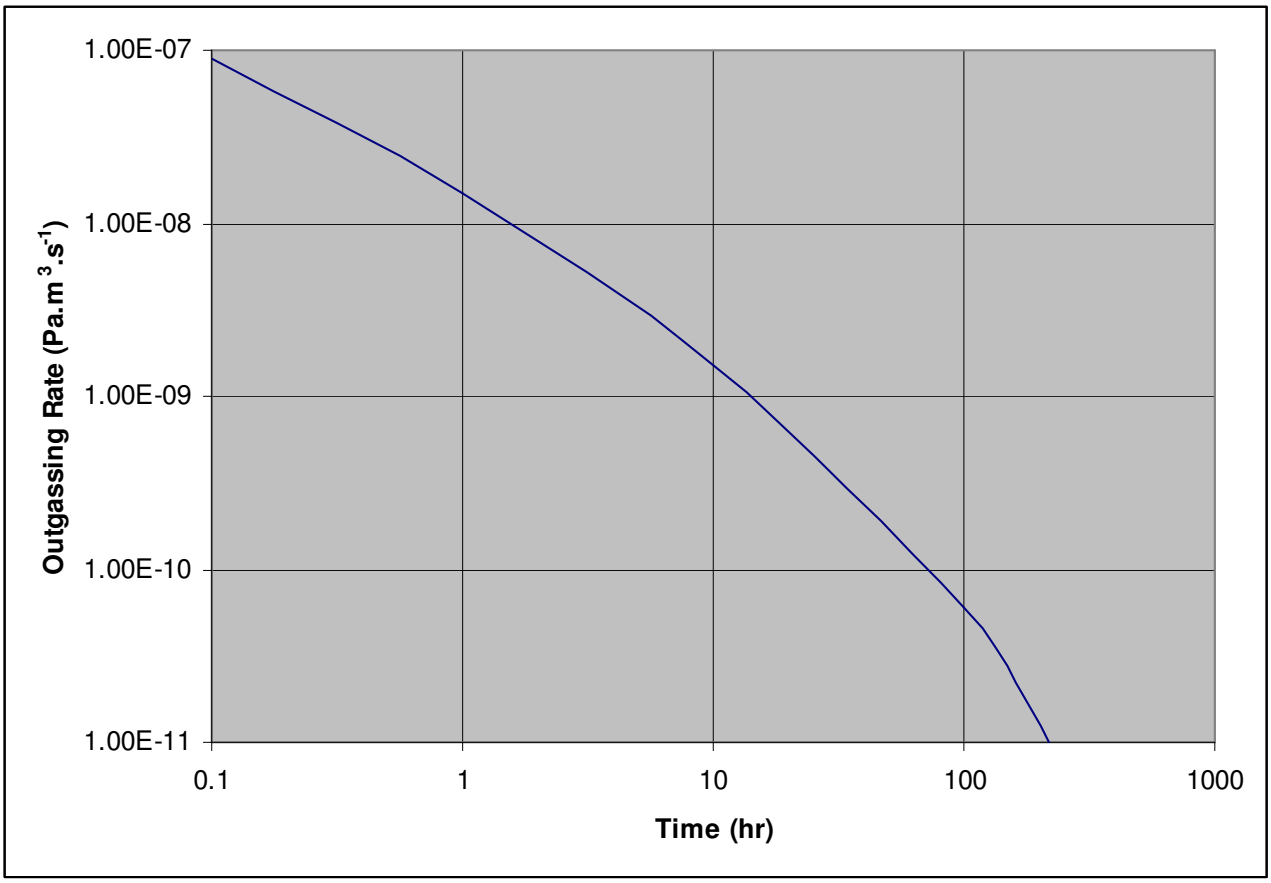

Figure 17.5.3-1 Idealised outgassing rate of a surface as a function of exposure time to vacuum

Because of the above, in order to achieve some sort of comparability, outgassing rates are often quoted as either 1 hour, 10 hour, 100 hour or “long term” rates. These are rates measured at these time intervals after time = 0. One matter of particular difficulty is determining just when time = 0 actually is. In a pump down, for example, when is the pressure determined by outgassing rather than removal of gas from the volume?

Since this set of procedures is intended for use in a quality assurance environment, this difficulty can be circumvented by careful specification of what should be done in individual cases.

17.5.4 Stating Outgassing Requirements

17.5.4.1 Vessel or Component Acceptance Tests as normally used in a Vacuum Quality Assurance Series of Procedures

In the specification for the vacuum item, if an outgassing test is required, then the specification should state the requirement in one of three alternative forms. These are as follows:

"*x* hours after the end of the procedure *y*, the specific outgassing rate shall be less than a value of *z* $Pa.m^3.sec^{-1}.m^{-2}$ using the measurement techniques described in the ITER Vacuum Handbook Appendix 17."

or

"m hours after the end of the procedure *n*, the total outgassing rate shall be less than a value of *r* $Pa.m^3.sec^{-1}$ using the measurement techniques described in the ITER Vacuum Handbook Appendix 17."

or

"k hours after the end of procedure g, the steady state specific outgassing rate shall be less than a value of *s* $Pa.m^3.sec^{-1}.m^{-2}$ using the measurement techniques described in the ITER Vacuum Handbook Appendix 17".

The steady state outgassing rate is defined at as the outgassing rate at the time when the rate of change of measured outgassing rate is less that 5 % over an elapsed time of 120 minutes.

That is to say:

$$\frac{q_t - q_{(t+120)}}{q_{(t+120)}} \leq 0.05$$

Where $q_{(t)}$ = specific outgassing rate at time *t* (minutes).

Procedures y, n and g will have been defined earlier in the specification and, unless there are good reasons otherwise, x and m will normally be 10 hours.

17.5.4.2 Testing items, materials or procedures for acceptability for more general use

Such tests are of a more generic nature and so some standardisation of results is necessary. There are two particular cases to be considered (a) where there is no form of processing and (b) where there is a processing stage included e.g. a bake.

Where no processing is involved outgassing measurements should be taken at intervals of 1 hour, 10 hours and (optionally) 100 hours after the start of pump down of the vacuum item. It should be noted that such results may be influenced by the pumping speed applied, so this should always be quoted.

Following a process stage, outgassing measurements should be taken at intervals of 1 hour, 10 hours and (optionally) 100 hours after the end of the process. In the case of a

bakeout, the end of the process may be defined as when the system returns to room temperature, unless a particular specification states otherwise. It should be noted that results may be influenced by the pumping speed applied, so this should always be quoted.

17.6 Procedures

17.6.1 General

17.6.1.1 Start Time

In the following procedures, it is assumed that the appropriate starting time for measurements has been set according to the considerations discussed earlier. This is referred to below simply as the start time.

17.6.1.2 Pump Set Conditioning

Before the start time, all pump sets will have been conditioned and proved to be leak tight and clean.

17.6.1.3 Vacuum Vessel Outgassing Measurements

The vessel should be assembled into the appropriate apparatus using flanges and gaskets appropriate to the vacuum regime for which the vessel is designed.

In the case of the measurement of outgassing of a vacuum chamber whilst being pumped from atmospheric pressure, a preliminary pump down should be made and the vessel and its appendages proved leak tight. Following this leak test, the vessel should be vented to either clean dry nitrogen (dew point < -50^{o}C) or normal atmosphere as specified in the test documentation. If nothing is so specified, then clean dry nitrogen is recommended.

In the (usually rare) circumstances of an outgassing measurement being required for a vessel in “as received” condition, then leak tests should be carried prior to the completion of the outgassing measurements to ensure that the results are not dominated by any leak being present. Clearly, great care must be taken during assembly to minimise the possibility of such leaks. If such a leak is detected, the originator of the request for test must be consulted before any further work is carried out.

17.6.1.4 Vacuum Component or Sample Outgassing Measurements

The component or sample should be inserted into a vacuum chamber for which the outgassing characteristics have been established in a blank run immediately prior to the tests.

For a meaningful measurement of outgassing, the expected outgassing load of the component or sample must be at least 10 times greater than that of the empty chamber.

The procedure to be followed will be the same as that for a vessel as specified in the request for test.

17.6.2 Rate of Pressure Rise Method

17.6.2.1 Equipment

The equipment used will typically take the form shown in Figure 17.6.2-1.

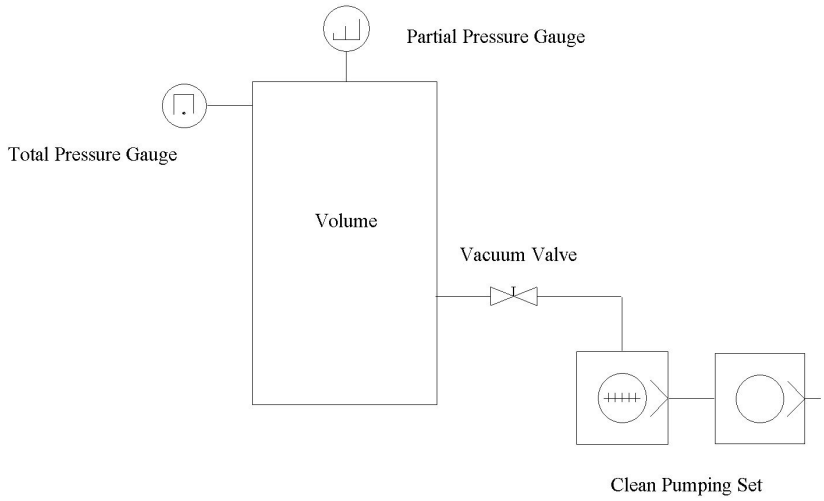

Figure 17.6.2-1 of outgassing - pressure rise technique.

The choice of pumping set and the type of total pressure gauge to be used will depend on the maximum total pressure expected during the measurements. The gauge is shown as a cold cathode device, but need not be. There are distinct advantages to using a Spinning Rotor Gauge if the pressures measured lie within its range of operation.

The use of a partial pressure gauge will normally mean that the total pressure should not normally rise above about 10^{-3} Pa during measurements unless some sort of sampling stage is used. The pump set should be chosen so that the volume may be evacuated through the valve to a reasonable pressure in a reasonable time. What “reasonable” means must be assessed on a case-by-case basis, but must be short compared to the time at which the first outgassing result is required.

17.6.2.2 Procedure

With the pump set under vacuum at or close to its ultimate, the vacuum valve is opened carefully and the volume evacuated to its base pressure or for the time at which an outgassing measurement is required, whichever is less.

Any processes specified (e.g. a bake cycle) are completed.

If the pressure achieved is below about 10^{-6} Pa, then any hot filament measuring devices should be thoroughly outgassed and the outgassing products pumped away.

The vacuum valve is closed and the pressure or partial pressure of the species of interest recorded at frequent intervals until a pressure rise of at least one decade is obtained. The times of recording each pressure should be noted. The use of a continuous record as on a chart recorder or a data logger is to be preferred.

If outgassing measurements are required at a number of values of pumping time, then the valve should be opened and the process repeated at the appropriate time.

The outgassing rate(s) are then calculated using the above formula.

17.6.3 Dynamic Flow Method

Note that only the two-gauge method is described here.

17.6.3.1 Equipment

The equipment used will typically take either of the forms shown in Figure 17.6.3-1. That on the left is more suited to measurements on vessels or assemblies, that on the right to coupon samples.

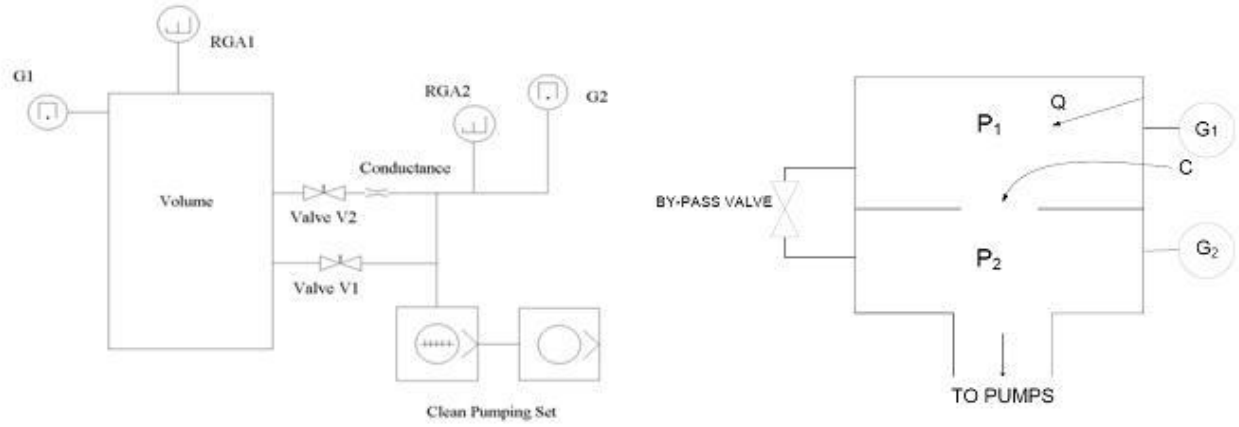

Figure 17.6.3-1 Equipment for the measurement of outgassing - dynamic flow technique.

The choice of pumping set and the types of total pressure gauges to be used will depend on the maximum total pressure expected during the measurements. The gauges shown are cold cathode devices, but need not be. The use of partial pressure gauges will normally mean that the total pressure should not normally rise above about 10^{-3} Pa at the gauge during measurements, unless some sort of sampling stage is used. The pump set should be chosen so that the volume may be evacuated to a reasonable pressure in a reasonable time. What “reasonable” means must be assessed on a case-by-case basis, but must be short compared to the time at which the first outgassing result is required.

The value of the conductance should be chosen so that a reasonable pressure differential is obtained.

17.6.3.2 Procedures

17.6.3.2.1 Outgassing measurements on a vessel

Here, the equipment shown on the left of Figure 17.6.3-1is the more suitable.

With the pump set under vacuum at or near its ultimate, vacuum valves V1 and V2 are opened carefully and the volume evacuated to its base pressure or for the time at which an outgassing measurement is required, whichever is less.

Any processes specified (e.g. a bake cycle) are completed.

If the pressure achieved is below about 10^{-6} Pa, then any hot filament measuring devices should be thoroughly outgassed and the outgassing products pumped away.

The vacuum valve V1 is closed and the pressures on either side of the conductance monitored until the values have stabilised over a period of about 15 minutes.

If quasi-continuous measurements of outgassing as a function of time are required, then sets of readings shall be taken at appropriate time intervals.

If outgassing measurements are required at a number of discrete values of pumping time, then the valve V1 is opened after a set of readings is complete and closed shortly before the next set is due, allowing sufficient time for the system to stabilise before each set of readings.

The outgassing rate(s) are then be calculated using the formula above.

17.6.3.2.2 Outgassing measurements on coupon samples

In this case, the equipment shown on the right of Figure 17.6.3-1 is the more suitable.

To be meaningful, the following procedure should be carried out first with the upper (test) chamber empty, then vented to clean, dry (dew point <-50^{o}C) nitrogen and the sample inserted. The sequence is then repeated, the sample removed and, ideally, a final sequence carried out on the empty system. The two blank (i.e. empty chamber) runs should give consistent results. The measured pressure in the upper chamber with the sample inserted must be significantly higher than the blank runs if a meaningful value of outgassing is to be calculated.

With the pump set under vacuum at or near its ultimate, the valve to the pumping set (not shown) and the by-pass valve are opened and the volume evacuated to its base pressure or for the time at which an outgassing measurement is required, whichever is less. The by-pass valve should be of sufficient size that adequate pumping speed is achieved above the conductance.

Any processes specified (e.g. a bake cycle) are completed.

If the pressure achieved is below about 10^{-6} Pa, then any hot filament measuring devices should be thoroughly outgassed and the outgassing products pumped away.

The by-pass valve should be closed and the pressures on either side of the conductance monitored until the values have stabilised over a period of about 15 minutes.

If quasi-continuous measurements of outgassing as a function of time are required, then sets of readings should be taken at appropriate time intervals.

If outgassing measurements are required at a number of discrete values of pumping time, then the by-pass valve should be opened after a set of readings is complete and closed shortly before the next set is due, allowing sufficient time for the system to stabilise before each set of readings.

The outgassing rate(s) are then calculated using the formula above.

17.7 Presentation of Results

On completion of outgassing tests a report should be issued recording:

- full details of the apparatus used (including volumes where appropriate)
- copies of calibration certificates for all gauges used
- details of the calculation of the value of the conductance (where appropriate)
- results of system leak tests
- proof of cleanliness of the pump set
- tabulated measurements of pressure with times at which readings were taken or copies of recorder traces as appropriate
- tabulated values of calculated total and partial outgassing rates as appropriate

17.8 Derivation of the ITER Outgassing Rate Requirements

The limits of outgassing rates for materials for use in ITER vacuum systems are given Table 17.8-1, which is Table 5-1 of the ITER Vacuum Handbook and the values are therefore mandatory.

These limits have been produced by taking into account the total surface area expected, available pumping speed, the desired pressure, and post assembly conditioning time, with due consideration of what is reasonably achievable.

		Maximum steady state Outgassing rate $Pa.m^3.s^{-1}.m^{-2}$		
VQC+	**Outgas temperature °C**	**Hydrogen isotopes**	**Impurities**	**Testing Guidelines**
1	100‡	1×10^{-7}	1×10^{-9}	Appendix 17
2	20	$1 \times 10^{-7*}$		Appendix 17
3	20	1×10^{-8}		Appendix 17
4	20	1×10^{-7}		Published data and conformity to clean work plan.

For VQC 2, 3 and 4, the outgassing rate excludes the partial outgassing rate for water and hydrogen.

‡ The outgassing test temperature can be reduced to 20 °C for components which normally operate at cryogenic temperatures.

+ For CFC, refer to the ITER Vacuum Handbook Section 26.7

* In the case of resins for magnets, it is considered that this target outgassing rate will be achievable. However a factor 10 increase will be permitted as an acceptance criterion.

Table 17.8-1 – Outgassing rates pertaining to VQC

17.8.1 Vacuum Vessel

In calculating the maximum outgassing rates specified for the Vacuum Vessel (VQC 1) the following assumptions and calculations have been used.

The approximate total surface area of vacuum vessel is 20000 m^2 and is calculated as the sum of the following:

- vacuum vessel+ports ≈ 3000 m^2
- port plugs ≈ 4000 m^2
- blankets ≈ 5000 m^2
- divertor ≈ 2000 m^2
- piping ≈ 1000 m^2
- in-vessel cabling ≈ 2500 m^2
- fixtures and fittings ≈ 2500 m^2

The ITER Project Integration Document (PID) specifies the vacuum vessel base pressure to be < 10^{-5} Pa for hydrogen and <10^{-7} Pa for impurities prior to ITER operations at the operating temperature of 100 °C.

Using a conservative estimate of the vacuum vessel pumping speed of 20 $m^3.s^{-1}$ yields a derived maximum hydrogen throughput of 2 x 10^{-4} $Pa.m^3.s^{-1}$

Thus, the maximum allowable outgassing rate of hydrogen prior to pulsing is calculated as,

$$q = \frac{Q}{A} = \frac{2.0x10^{-4}}{20000} = 1x10^{-8}\,\mathrm{Pa.m^3.s^{-1}.m^{-2}}$$

It is expected that a factor 10 decrease in the outgassing rate for hydrogen can be achieved by baking the vessel to 200 °C and hence the maximum outgassing rates for VQC 1 components has been defined in Table 17.8-1 as:

- 1x10^{-7} $Pa.m^3.s^{-1}.m^{-2}$ for hydrogen at 100 °C
- 1x10^{-9} $Pa.m^3.s^{-1}.m^{-2}$ for impurities at 100 °C

17.8.2 Cryostat

The outgassing requirement for VQC2 is derived from the need to manage three areas:-

1) To be able to pump down the cryostat initially in a reasonable time with limited pumping and conditioning capacity and to achieve a level of vacuum suitable for an insulation vacuum.
2) To avoid poisoning of the activated charcoal in the reference cryostat cryo-pumps with heavy hydrocarbons.
3) To ensure that over time, the build up impurities on the cold thermal shields does not adversely affect their emissivity and hence the heat load on the superconducting coils and the cryo-plant.

The specified outgasing limit for VQC 2 excludes water because it is considered that it will not be possible during the cryostat construction to avoid surfaces becoming water contaminated.

It is the case that for item 3 above water ice is likely to be the dominant issue. However other gasses which are condensable at 80K can also present a similar problem and these can be more difficult to condition once the cryostat is complete. To quantify an acceptable outgassing rate, water is used below, as there is a better database available for the relevant emissivity change.

In calculating the maximum outgassing rates specified for the ITER cryostat (VQC 2) the following assumptions and calculations have been used.

Source[27]	A (m^2)[27]	qH_2O($Pa.m^3.s^{-1}.m^{-2}$)+	QH_2O_{tot} ($Pa.m^3.s^{-1}$)	Pressure in cryostat (H_20,Pa)‡
Metallic surface	2.5×10^4	1×10^{-7}	2.5×10^{-3}	5.0×10^{-5}
Vacuum facing epoxy	1.3×10^3	1×10^{-5}	1.3×10^{-2}	2.6×10^{-4}

‡Assumes 50 m^3s^{-1} H_2O cryostat pumping speed.[27]

+Values from Table 17.8-1 & equation Section 17.8.1 after 100 hours.

Table 17.8-2- Assumed cryostat areas and calculated H_2O outgassing rates

Using the figures from Table 17.8-1 the calculated partial pressure of water vapour in the cryostat prior to the cool down of the magnets is approximately 2.6×10^{-4} Pa.

The 2007 ITER PID value for partial pressure of H_2O before cool-down is quoted as ≤ 2×10^{-7} Pa. This figure is considered to be unachievable and the basis can not be found.

Assuming the cryostat thermal shield does not cool uniformly, residual water will initially condense on cold spots covering an estimated 10% of the thermal shield surface area with an equivalent thickness of 0.02 monolayers.

After baking the vacuum vessel and cooling the magnet structures and thermal shields, the remaining source of condensable water will be from the cryostat walls and internal components which are at ambient (or elevated) temperature, having an estimated total surface area of 3000 m^2.

Assuming a steady state outgassing rate of 1×10^{-7} (H_2O) $Pa.m^3.s^{-1}.m^{-2}$, the load to the thermal shield remains unchanged for 3 years. Over approximately 8 years a coverage of H_2O of 2000 monolayers (1μ thickness) will form on the cryostat-facing thermal shield. The change in emissivity of the thermal shield due to formation of this water layer results in a calculated increase in heat load to the cryo-plant of approximately 50% [28].

The ice crystal size significantly affects the infra-red absorption and consequently the emissivity of a panel: the larger the crystals, the higher is the emissivity; therefore the morphology of the ice formation significantly effects the change in emissivity.

In this estimation, it is assumed that the water forms a uniform layer of ice over the thermal shield with the coverage rate constant over the time period considered. If the coverage rate is not constant, and it is assumed water condenses on the thermal shield in batches as “snow”, the time taken for a similar change in emissivity decreases to approximately 3 years.

The effect on emissivity due to the build up of ice can be seen in Figure 17.8.2-1[28] and the effect on the additional load to the cryo-plant due to water condensing on the cryostat-facing thermal shield is shown in Figure 17.8.2-2[28].

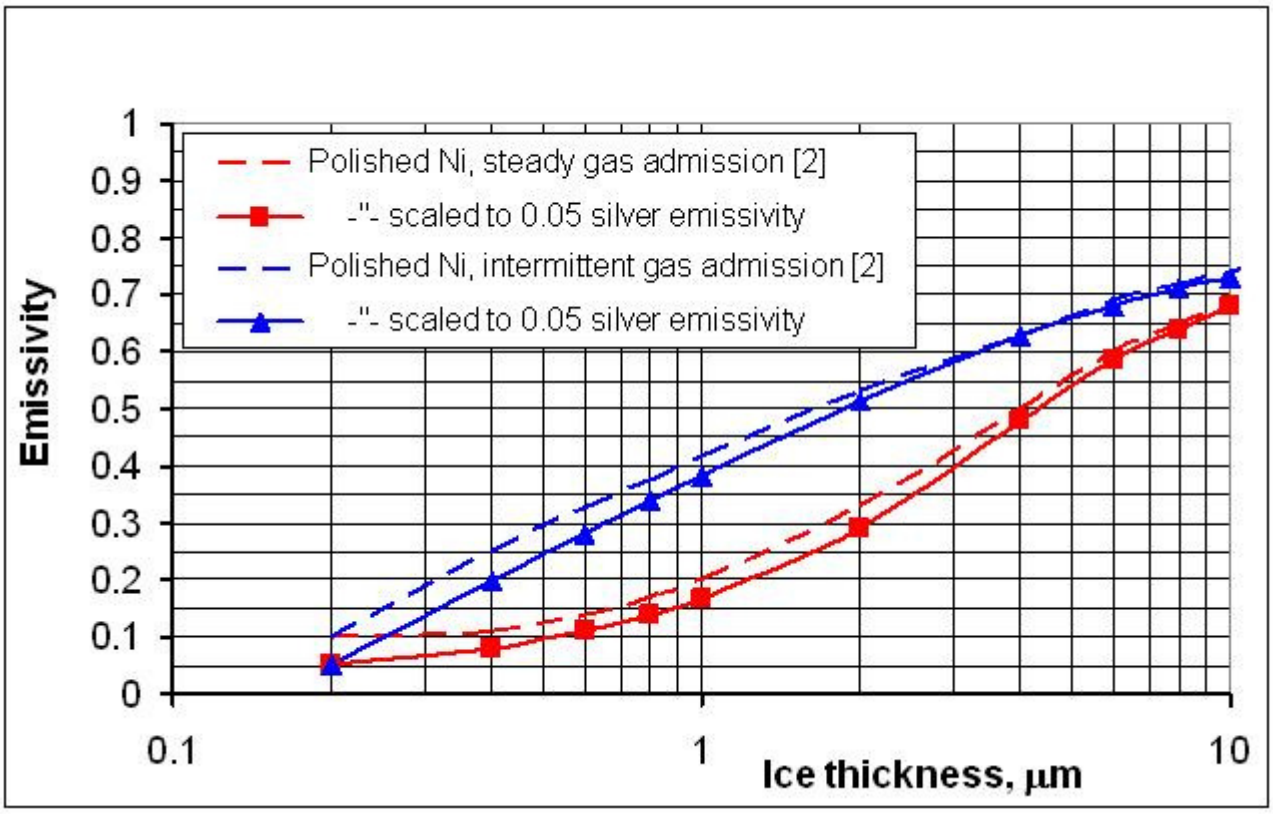

Figure 17.8.2-1 Effect on emissivity due to ice layer formation

It is considered that the effect on the emissivity of the cryostat thermal shields will be greater due the condensation of hydrocarbons outgassing from the cryostat internal components. Hence the maximum outgassing rate from cryostat vacuum-facing surfaces is defined in Table 17.8-1 to be1 x 10^{-7} $Pa.m^3.s^{-1}.m^{-2}$ (excluding water and hydrogen) at ambient temperature

In order to reduce the steady state outgassing rate of water from the cryostat internal surfaces, a method of purging the cryostat with dry nitrogen prior to cool down of the magnet structures and thermal shields is being studied. The order in which the cryostat cryogenic surfaces are cooled, and the resulting effect on the emissivity of the cryostat cold surfaces due to condensed gas, is also to be studied. (See [28] for further recommendations)

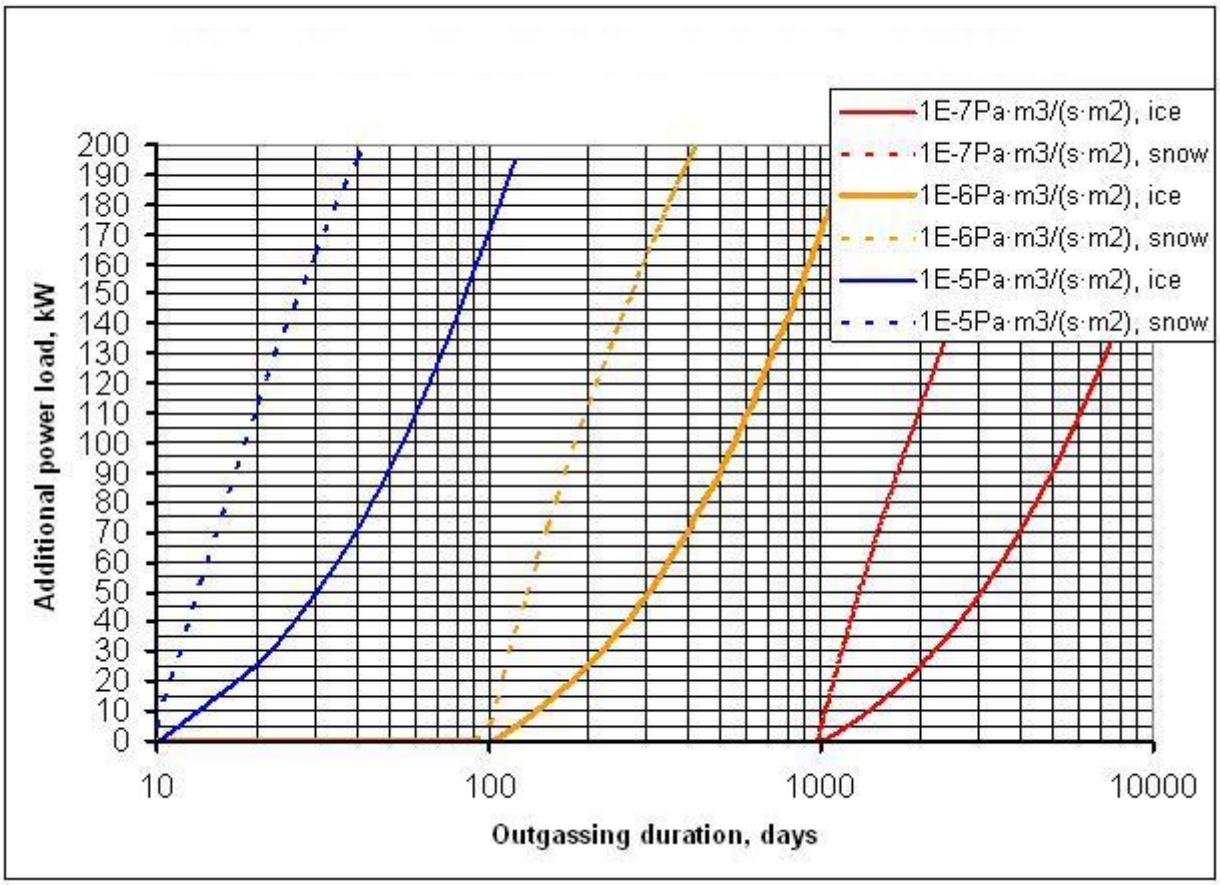

Figure 17.8.2-2 Additional power load on thermal shield coolant due to H_2O Outgassing

17.9 Outgassing Rates Review

The purpose of this section of the Appendix is to outline the methodology used in the assessment of outgassing rates from published data and to establish the relationship between common parameters which influence material outgassing rates

17.9.1 Material thermal outgassing

Thermal outgassing from material surfaces is time and temperature dependant and it can be shown that the measured outgassing rate from a metallic surface will increase by factor of about 10 by increasing the sample temperature from ambient to 100 ºC, and increases by a further decade by raising the sample temperature from100 to 250 ºC.[5]

The medium term (1 to 100h) outgassing from a surface can be described by a power law of the form:

$$Q = Q_0 . t^{-\alpha}$$

Where, α (the outgassing decay index) is typically near unity for metallic surfaces and 0.5 for epoxies and *t* is the time in hours [21].

The outgassing rate of a surface is also dependant on the surface condition. Factors affecting the outgassing rate include:

- chemical composition
- the presence of oxide layer's
- surface finishing
- cleaning and other processes

References to published data, listing outgassing rates for materials after varying surface treatments, are to be found in Section 17.10.

While a large record of outgassing rates can be found in literature for vacuum compatible materials comparisons of the reported data are difficult as, in many cases, for the same material differing surface treatments and measurement techniques are reported, some important factors may not be reported at all.

17.9.2 Unbaked Stainless Steel

The rate of outgassing from unbaked stainless steel is dependant of the process to which the stainless steel surface has been subjected. Outgassing rates gathered from literature (see Section 17.10) for Stainless steel after surface treatments are summarised in Table 17.9-1.

SST treatment	**q_{tot} ($Pa.m^3.s^{-1}.m^{-2}$) at 1h, 20ºC**
As received/fresh	$1x10^{-4}$
Degreased	$2x10^{-6}$
Surface finished (machined)	$2x10^{-7}$

Table 17.9-1 Outgassing rates of stainless steel after surface processing

Generally water is the dominant species outgassed from unbaked stainless steel and will evolve at a rate dependant on the elapsed pumping time of the surface. Generally, for unbaked stainless steel surfaces, water will remain the dominant outgassing species at pumping times in excess of 100 h.

17.9.3 Baked Stainless Steel

Baking at 150 ºC for a minimum of 24 h can reduce the total outgassing rate by a factor of 100 as water is desorbed from the metal surface. After this time the predominant outgassing species from clean stainless steel is hydrogen [5]. A reduction in the hydrogen outgassing rate can be achieved by vacuum firing or air baking the material.

After baking, stainless steel will generally exhibit outgassing rates between 10^{-9} and 10^{-10} $Pa.m^3.s^{-1}.m^{-2}$ (see Section 17.10.1)

17.9.4 Organic Material

For organic materials (epoxies etc), the method of weight loss measurement is usually used for the determination of outgassing rates with the outgassing rate quoted as a percentage of total weight loss, or gram/s.

Using the formula below the outgassing rate can be calculated from the total mass loss measurement

$$q = \frac{dM}{dt} \cdot \frac{RT}{M} \cdot 10^3$$

where:

q is the outgassing rate in $Pa.m^3.s^{-1}.m^{-2}$

R is the universal gas constant (83.14 $mbar.l.mol^{-1}.K^{-1}$)

dM/dt is the mass loss per unit time ($g.s^{-1}$)

T is the sample temperature (K)

M is the molecular mass of the outgassing species

Using the above formula it can be shown that for water outgassing from a surface at a rate of $1\mu g.s^{-1}$ the specific outgassing rate near room temperature will be approximately 1 $Pa.m^3.s^{-1}.m^{-2}$.

The outgassing rate of organic materials is also dependent on the fabrication process (curing temperature, chemical hardener, vacuum, inert gas process, etc.). There is a lack of published data on outgassing rates for material of the same composition which has undergone different fabrication processes, making comparisons difficult. Hence qualification of new organic materials for use on ITER will have to be performed using experimental data.

An analysis of weight loss measurements on epoxies shows that the ratio of water outgassing to impurity outgassing is approximately 100 to 1, so, assuming a well controlled fabrication process, a low outgassing epoxy should outgas at a rate in the range of 10^{-7} $Pa.m^3.s^{-1}.m^{-2}$ (excluding water) after 100 h baking (see Section 17.10.2).

17.10 Outgassing Rates - Published Data

Outgassing rates quoted in referenced publications are summarized in the tables below.

17.10.1 Stainless Steel

Published data on the outgassing rates of stainless steel following various surface treatments is given in Table 17.10-1.

Treatment	Total outgassing rate ($Pa.m^3.s^{-1}.m^{-2}$)	Time meas. (hours)	Reference
None	$2x10^{-4}$	1h	2
None	$2x10^{-5}$	10h	2
Polished & vapor degreased	$1.4x10^{-6}$	10h	2
None	$1.1x10^{-7}$	100h	2
Degrease + water rinse	$4.0x10^{-8}$	40h	2
Degrease + water rinse, baked in vacuum 150ºC for 12h	$4.0x10^{-9}$	5h after bakeout	2
Baked 24h @ 200°C	$9.3x10^{-10}$	100h	2
Unbaked	$2x10^{-7}$	10h	1
Baked (150º C,24h)	$2x10^{-9}$		1
Std cleaning	10^{-6}	1h	3
Baked	10^{-8}	1h	3
Untreated	$7x10^{-5}$		4
Degreased	$1x10^{-6}$		4
Baked	$3x10^{-10}$		4
unbaked	$9x10^{-7}$	20h	5
Electrochemical buffing	$5x10^{-8}$	50h	8
Electrochemical buffing followed by baking(215 °C,23h) and air (10days)	$1x10^{-8}$	50h	8
Electropolished, baked, air oxidation	$1x10^{-11}$		9
Air exposure/baking cycles	$1x10^{-10}$		10
UT cleaning + bake 250C,24h	$3x10^{-10}$		12
Various treatments	$2x10^{-6}$	100h	13
Annealing+bake	$2x10^{-11}$		14
Air firing	$3x10^{-11}$		15
Pre-baking+baking	$4x10^{-10}$		16
Chemical cleaning	$4x10^{-9}$		17
	$1x10^{-6}$	1h	18
Cleaned With bakeout	$8x10^{-7}$ $2x10^{-9}$		19
	$2x10^{-6}$	4h	20

Table 17.10-1 Outgassing rates for stainless steel – published data

17.10.2 Epoxies

Published data on the outgassing rates of various epoxies and resins is given in Table 17.10-2

Material	Outgassing rate (Pa.m3.s-1.m-2)	Outgassing rate % Total Mass Loss (TML)	Reference

Material	Outgassing rate (Pa.m3.s-1.m-2)	Outgassing rate % Total Mass Loss (TML)	Reference
RF4000 EV Roberts (baked)	$5x10^{-6}$ (10h)		22
ERL4221 union carbide (baked)	$1x10^{-5}$(10h)		
CY179 Ciba Geigy (baked)	$3x10^{-6}$(10h)		
1138 Ciba-Geigy (baked)	$2x10^{-6}$(10h)		
828 Shell chemical (baked)	$1x10^{-5}$(10h)		
DGEBA, + ≠ materials	10^{-3} -10^{-4}(10h)		23
Stycast	$4x10^{-5}$ (72h)	0.87	24
Redux 312UL	$7x10^{-6}$(72h)	0.40	25
Ablebond Ablestik		0.2	ESA database
Araldite resin	10^{-3}-10^{-4}(10h)		1
Polymers	10^{-5}(10h)		26

Table 17.10-2 Outgassing rates for epoxies and resins – published data

17.11 References

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