

The Impact of Environmental Protection Legislation on Cleaning Procedures for Accelerator Vacuum Systems

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Abstract

Modern accelerators, and in particular storage rings, demand high standards of cleanliness in the preparation of vacuum system components in order that attained pressures do not have a detrimental effect on the performance specification of the machine. For stainless steel components the traditional cleaning agents have been the highly efficacious chlorinated hydrocarbons and chloro-fluoro carbons (CFCs). With the increasing realisation of the effect of such chemicals on the environment, many are being phased out and others will become increasingly restricted to use in essential processes. A programme of research into the efficacy of a range of alternative cleaning agents for stainless steel has been undertaken within our Laboratory, with particular regard to the requirements of the Synchrotron Radiation Source facilities. We define the efficacy of an agent in terms of the residual vacuum attained in a test system after cleaning of a deliberately contaminated sample in that agent. We have carried out measurements of total outgassing rates, of partial pressures of residual gas species and obtained preliminary results on electron stimulated desorption. These results will lead to an informed choice for a suitable and viable cleaning process for use within this Laboratory.

1. INTRODUCTION

Synchrotron light source vacuum systems such as the SRS at Daresbury have to operate at pressures in the low 10^{-9} mbar region to obtain extended beam lifetimes.¹ Not only is it necessary to achieve this pressure, but also the partial pressures of high-Z (atomic number) species (normally referred to as contaminants) needs to be several orders of magnitude lower than this. It is thus necessary to minimise photon and electron stimulated desorption of such high-Z species since desorption processes dominate the vacuum performance.

In order to meet such a stringent requirement, it is necessary to implement a rigorous cleaning process for all vacuum vessels and components. The process currently used at Daresbury utilises a multi-stage approach which has proven to be effective over a number of years.² It was first introduced when the SRS was under construction and was based upon the experience of other accelerator laboratories, notably CERN and DESY. The process remains much the same today, although some improvement and upgrading has taken place. However, due to current and proposed environmental protection legislation (under EC Regulation 3952/92), two of the main cleaning agents used at Daresbury (methyl chloroform and CFC 113) will have to be replaced. A new cleaning process will therefore have to be implemented. If this process is based on chemical or solvent cleaning, then any chemicals or solvents used will clearly have to be acceptable

for such use within the current legislative framework. A complication in making such a choice is that it is anticipated that legislation will be enacted within the next decade further controlling the use of organic solvents, in particular the chlorinated hydrocarbons. The nature and scale of such control is at present unclear.

This paper describes work carried out at Daresbury to find suitable replacement solvents and presents some of the results obtained. Early results and further discussion have already been reported.³

2. CRITERIA FOR SURFACE CLEANLINESS

It is generally accepted that there is no satisfactory, universal criterion which defines a clean surface. One method that could be employed is to use surface analytical techniques which can determine whether or not a surface is atomically clean. However such a definition is not necessarily appropriate to the technological surfaces that are considered here.⁴ Other techniques also exist, for example industrial water-break tests, but these only indicate the presence or absence of relatively gross contaminants.

Because the cleaning process has to achieve a result, namely low desorption in the environment of the electron storage ring, it is reasonable to adopt a practical criterion for defining surface cleanliness. We therefore define a satisfactory cleaning process as one which results in a vacuum vessel surface with sufficiently low room temperature thermal outgassing, particularly of high mass number species, together with sufficiently low photon stimulated desorption, again particularly of high mass number species. The term *sufficiently low* needs to be defined. It is known that the current cleaning process used at the laboratory produces a satisfactory result. Hence any acceptable cleaning process must achieve outgassing and desorption results no worse than those obtained by using the current process.

3. MEASUREMENT OF THERMAL OUTGASSING RATES

Outgassing rates are measured by the throughput method^{5,6} in the apparatus shown schematically in figure 1.

During all measurements, a hydrocarbon free turbo-molecular pump set was used to eliminate any spurious pumping speed effects dependant on the molecular species being pumped. The conductance (C) used was an all metal gate valve with an aperture drilled into the valve plate. An ion pump was used to pump the system during bakeout. Two nominally identical Bayard-Alpert ionisation gauges were used for pressure measurement. These two gauges were not calibrated absolutely, although great care was taken to ensure that they gave comparable readings while the conductance valve was open. Because the method relies upon differences in

gauge readings, comparisons of outgassing rates from samples cleaned in different cleaning agents can be obtained with a high degree of confidence.

The partial pressure gas analyser was used to determine the nature of the outgassing species, and then to compare the efficacy of various solvents in reducing high-mass species as opposed to low mass species.

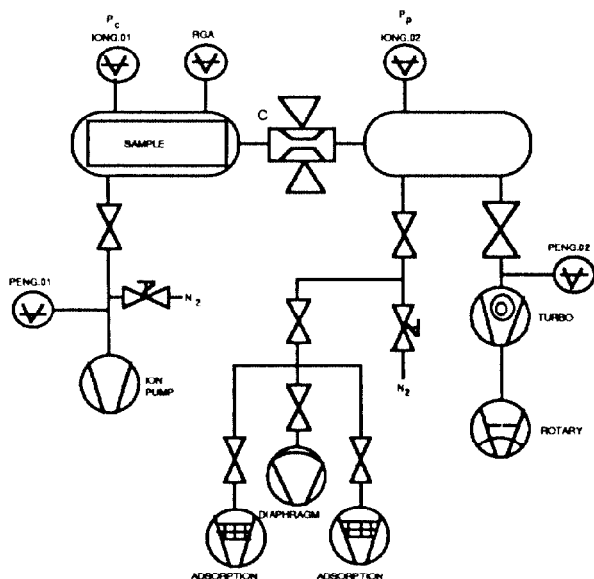


Figure 1. Schematic of the apparatus used for measuring outgassing rates.

The tests were done in a careful and routine manner and involved deliberately contaminating each sample before cleaning with the appropriate cleaning agent. The contaminants chosen for this purpose were typical of those which would most likely be encountered in the normal fabrication and processing of a vacuum vessel. The cleaning agents used were selected from those currently available in the UK market and chosen as representative of the major categories of cleaning agent. They are listed in table 1. More information about them, as well as full details of the procedures adopted, are given elsewhere⁷.

4. RESULTS OF OUTGASSING TESTS

Table 1 lists the measured outgassing rates and information from the partial pressure analysis. The results are presented in order of increasing outgassing and have been discussed more fully elsewhere,³ but the principle conclusions are that the current cleaning agents listed in table 1 as Genklene LV and Almeco-P3 perform the best of the those tested, and that the partial pressure analysis reveals that the performance of "traditional" solvents in removing high mass contaminants is superior to that of the alkali cleaners.

The term "traditional solvents" usually refers to chlorinated hydrocarbon or other hydrocarbon solvents which are not water based and may consist of one or more organic compounds (e.g. in table 1 Triklone, Axarel and DP112 are such).

Alkaline cleaning agents (e.g. Safewash and RBS (809)) are usually aqueous liquids consisting largely of water with a

mixture of inorganic compounds that increases the pH to alkaline. These cleaning agents are often, but not always, biodegradable.

Cleaning Agent	Outgassing Rate (mbar l/s)/cm ²	Ratio of Masses >40 to 28 (excluding 44)
Blank Run (Typ.)	3.2×10^{-13}	9.5×10^{-3}
Almeco-P3	5.0×10^{-13}	1.4×10^{-2}
Genklene LV	6.8×10^{-13}	1.2×10^{-2}
Safewash	8.3×10^{-13}	3.5×10^{-2}
Axarel-9100	8.9×10^{-13}	2.2×10^{-2}
RBS (809)	1.1×10^{-12}	3.4×10^{-2}
Isopropyl Alcohol	1.4×10^{-12}	3.7×10^{-3}
RBS (25)	1.5×10^{-12}	3.5×10^{-2}
Triklone	1.6×10^{-12}	3.2×10^{-3}
DP 112	1.6×10^{-12}	1.7×10^{-2}
Purasolv-ELS	2.7×10^{-12}	9.8×10^{-2}

Table 1. Total outgassing rates and the relative effect of each cleaning agent in removing high mass number species. Column 2 lists the ratio of the peak height of all peaks above mass 40 with the exception of mass 44 (CO₂) to the peak at mass 28 (CO + N₂). This is a measure of hydrocarbons and other contaminants.

5. MEASUREMENT OF STIMULATED DESORPTION

The photon stimulated desorption (PSD) and electron stimulated desorption (ESD) coefficients are defined respectively as the number of gas molecules released into the vacuum chamber for each photon or electron incident on a surface. The measurement of PSD coefficients is by no means straightforward, but the measurement of ESD coefficients is much more tractable. In order to use ESD as a guide to PSD it is necessary to make the assumption that a surface which exhibits low ESD will also exhibit low PSD. This assumption has not been proved and there is insufficient experimental data to validate it empirically. However, in view of the accepted mechanism that PSD is mediated by secondary electron emission from a surface,^{8,9} it is difficult to envisage how a surface which exhibits high ESD could simultaneously exhibit low PSD. The opposite is less obvious but assumed to be true.

Tests are underway to measure ESD from samples which have been deliberately contaminated as above and then cleaned in those solvents known to give acceptable thermal outgassing.

Tests are carried out in the apparatus used for the thermal desorption tests, but incorporating a heated filament assembly with associated drive power supplies and control electronics. The filament assembly consists of a high current feedthrough and a 1.0mm diameter tungsten filament wound around a ceramic former. The assembly fits into the sample chamber such that the cylindrical sample surrounds the filament. The sample is electrically isolated from the chamber. When the filament is heated, a bias voltage can be applied between

sample and filament so that the sample is bombarded with thermionically emitted electrons of well defined kinetic energy. Bias voltage, emission current, sample chamber pressure, partial pressures of species in the region of the sample and the power to drive the filament are all monitored.

The filament is slowly driven up to full power with the bias voltage off to allow the initial high thermal gas load to be pumped away, and the temperature and pressure of the system is allowed to stabilise before measurements are taken. The bias voltage is then turned on, the change in pressure is recorded and the residual gas analyser collects data on the changes in gas species due to the ESD. The bias voltage is turned off after sufficient data has been collected and before any thermal effects due to the electron bombardment of the sample become significant.

6. RESULTS OF ESD TESTS

ESD measurements are at an early stage so only preliminary results are available.

Figure 2 shows the change in total pressure and table 2 lists the corresponding changes in selected partial pressures due to ESD.

These results were obtained from a sample cleaned using the current standard cleaning agent, methyl chloroform, and establishes the baseline for comparison of the desorption from samples cleaned with other cleaning agents. Further results will be presented in due course.

From the graph of figure 2 the total quantity of gas desorbed from the sample due to ESD can be determined and the coefficient of desorption is calculated as 5.1×10^{-6} molecules per electron.

Table 2 shows that the principle gas species desorbed are hydrogen (mass 2), carbon-monoxide (mass 28) and methane (mass 16 and 15).

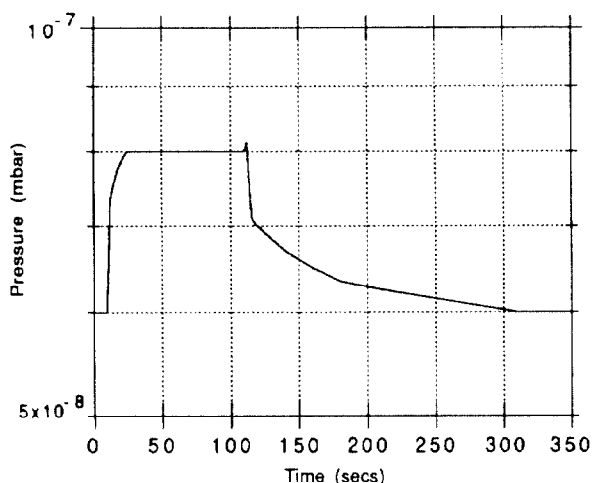


Figure 2. Change in total pressure in sample chamber due to electron stimulated desorption.

m/e	Gas Desorption Yields From ESD ($\times 10^{-7}$ mbar l/s)
2	10
28	5
16	1.8
15	1.1
18	0.4
12	0.3
44	0.3
14	0.3
32	0.05

Table 2. Partial pressure analysis of gas species desorbed during ESD. The species are listed in order of decreasing desorption, and are nitrogen equivalent.

7. CONCLUSIONS

The results of the outgassing tests show that the cleaning agents currently used at Daresbury perform the best of those tested. This is encouraging in that it confirms work done during the construction of the SRS in determining a cleaning process. However, the drawback is that it seems difficult to obtain such good results from cleaning agents which are environmentally acceptable.

The results of the partial pressure analysis done during the outgassing tests indicates that chlorinated hydrocarbon cleaning agents are more effective in reducing high mass contaminants than other hydrocarbons or the alkaline cleaners.

The preliminary ESD results indicate that the technique is of sufficient sensitivity to allow meaningful comparisons of results obtained through the use of different cleaning agents.

It now seems possible that, at Daresbury, methyl chloroform (Genklene) will be replaced by trichloroethylene (Triklone). It is well established as a cleaning agent for stainless steel for ultra high vacuum applications. The results show its performance to be comparable to that of methyl-chloroform. The technology available for using the solvent is well established and the cost of its use is acceptable. There are health and safety implications in its use, but these are well understood and can be managed in a satisfactory way. However it will be necessary to complete ESD tests before making a final choice. A possible uncertainty lies in the continuing acceptance of trichloroethylene under environmental protection legislation.

8. REFERENCES

- [1] R.J. Reid, *Vacuum*, **44**, 473 (1993)
- [2] R.J.Reid, *Ultra High Vacuum Guide*, UHV/02/90, available from the authors.
- [3] J.D.Herbert, A.E.Groome and R.J.Reid, *J. Vac Sci. Technol.*, *in press*
- [4] A.G. Mathewson et al, *J. Vac Sci. Technol.*, **A7**, 77 (1989)
- [5] R.M. Zabel, *Rev. Sci. Instrum.*, **4**, 233 (1933)
- [6] S.H. Cross, *Vacuum*, **10**, 86 (1960)
- [7] J.D.Herbert, *DL Internal Report* (unpublished)
- [8] G.E. Fischer and R.A. Mack, *J. Vac Sci. Technol.*, **2**, 123 (1965)
- [9] M. Bernardini and L. Malter, *J. Vac Sci. Technol.*, **2**, 130 (1965)