Reprinted from

Journal of Vacuum Science & Technology A

JVST A

Second Series
Volume 18, Number 4, Part II
July/August 2000

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pp. 1782-1788

An official journal of the American Vacuum Society
Published for the Society by the American Institute of Physics

Dry vacuum pumps: A method for the evaluation of the degree of dry

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(Received 26 August 1999; accepted 8 March 2000)

The drive towards dry vacuum pumping has occurred across the spectrum of vacuum applications from semiconductor manufacture to industrial processing. This brings with it a need to systematically evaluate and quantify the degree of cleanliness characteristic of any particular pump; currently, there is no universally accepted method to perform this function. A methodology developed for repeatable measurements of pump cleanliness will be discussed. It utilizes residual gas analysis with carefully controlled pump conditions. This facilitates direct comparisons of the degree of cleanliness between pumps of the same and those of different design. Additionally, it allows for the assessment of methods (either in pump design or use) introduced to improve cleanliness. © *2000 American Vacuum Society.* [S0734-2101(00)11304-1]

I. INTRODUCTION

The drive towards contamination-free, dry vacuum environments in many manufacturing applications has led to the development of "*dry pumping systems*." These pumps contain no sealing or lubricating fluids in the swept volume. However, they do maintain a supply of oil and low vapor pressure grease for lubricating mechanical components such as gears and bearings (fluorocarbon oils and greases are commonly used lubricants in dry pumps due to their low vapor pressure). Although the gearing mechanism of a dry pump can be separated from the vacuum space by either static or dynamic shaft seals, lubricant exposure to the vacuum space may still be a possibility. In addition, some pumps will have bearings, lubricated with low vapor pressure grease, directly exposed to the vacuum space. At extremely low pressure it may be possible for some of the lubricant, in the vapor phase, to find a route via backmigration through the pump and appear as a contaminant in the residual atmosphere at the pump inlet.

Pump applications requiring a high degree of cleanliness may employ several techniques to counteract oil vapor backmigration. For vapor pumps and rotary vane pumps these can include installing liquid nitrogen traps, molecular sieves, or controlled gas bleeds to restrict the vapor progressing along the foreline. Conventional dry pumps employ high-performance dynamic shaft seals to reduce lubricant transfer and gas ballast to provide a barrier between the pumped gases and the gearbox to restrict backmigration at source. Previous work has also shown that a small flow of gas at either the pump inlet or ballast ports is sufficient to reduce backmigration and suppress fluorocarbon oil contamination of the residual atmosphere to below measurable limits.[1–3]

In order to gain a qualitative measurement of the degree of dry (or the level of contamination) a technique has been previously developed (at a preliminary level) to quantify the relative amount of fluorocarbon contamination, associated with dry pump lubricants, in the residual atmosphere at a dry-pump inlet under controlled conditions.[4] In this article we report on a recently developed extension to this method that now allows for direct comparisons of (residual atmosphere) contamination at the inlet of pumps of the same and different design. The application of this method to qualify the effectiveness of design modifications and other methods employed will be discussed and exemplified using results obtained from the analysis of the residual atmosphere at the inlet of a number of commercial pumps.

II. METHOD

A schematic of the experimental apparatus is given in Fig. 1. The analysis chamber is pumped by a BOC Edwards EXT70 turbomolecular pump backed by an alumina trapped two-stage rotary pump. Residual gas analysis is performed using a Hiden analytical HAL2/511 quadropole mass spectrometer with a range of up to 510 atomic mass units (amu). A hot cathode ionization gauge monitors the pressure in the analysis chamber, the ultimate pressure of which was consistently of the order of 10^{-9} Torr following a 24 h bakeout. The analysis chamber is connected to the pump under test via a 1-meter DN25NW flexible foreline. A fine leak valve is manually regulated to draw a sample from the foreline at a rate of approximately 7.0×10^{-5} Torr l s^{-1} thus maintaining a sampling pressure in the analysis chamber of 1.0×10^{-6} Torr. A capacitance manometer and a pirani gauge measure the pressure at the pump inlet. All vacuum system components are "*as new*" or vapor degreased prior to use. The vacuum gauges are cleaned prior to each use in order to remove possible traces of condensed oil vapor.

The fluorocarbon oil and grease employed for lubrication purposes in most vacuum pumps are the perfluoropolyether (PFPE) variety which are reported[5–7] to have the general structure as shown in Fig. 2. The mass spectra cracking pattern for these lubricants was found to have a main peak at mass 69(CF_3) and secondary mass peaks at 97(C_2F_3O) and 119(C_2F_5), which is consistent with data published elsewhere.[5,7] This technique provides a relative measurement of pump contamination by measuring the partial pressure

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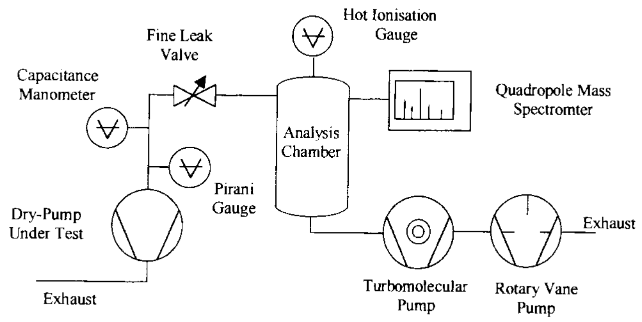

FIG. 1. Schematic of the experimental apparatus.

TABLE I. Capacitance manometer measurements of ultimate pressure and construction details for each of the scroll pumps evaluated for the degree of contamination in the residual atmosphere at the pump inlet.

Pump	Ultimate pressure (Torr) ±20%	Details
A	3.9×10^{-2}	Single stage
B	5.6×10^{-2}	Parallel stages
C	2.1×10^{-2}	Two stage with bearing purge and intrinsic ballast
D	1.4×10^{-2}	Two stage with bearing purge and no intrinsic ballast
E	4.1×10^{-3}	Parallel stages

corresponding to the main fluorocarbon mass spectral peak at molecular weight (M/e) 69 and that of a reference peak; this is selected as the hydrogen peak (H_2) at M/e 2. This is discussed in further detail below.

Hydrogen is a permanent gas in vacuum systems and is always present in finite quantities. The PFPE contains no hydrogen species and hence does not contribute to measurements of the hydrogen partial pressure. Furthermore, when inlet and ballast purges are disabled the hydrogen content in the residual atmosphere at the inlet of pumps, of similar construction and with comparable ultimate pressures, has been found to be relatively consistent. Therefore, the partial pressure measured at M/e 2 makes an ideal reference level with which to relate the partial pressure measured at M/e 69.

Previous work has shown that the quantity of contaminating gas species appearing in the residual atmosphere at the pump inlet is a function of the particular pump, and will gradually increase over time until an equilibrium level is eventually achieved.[4] The length of time required to reach the equilibrium level has been found to be dependent on the temperature and dimensions of the foreline connecting the analysis chamber to the pump. It has been found however, that the final equilibrium contamination level attained for a given pump is consistent regardless of the foreline temperature and dimensions (these effectively develop a system time constant). Hence, to improve on our previously reported method[4] the diameter of the foreline has been reduced and heating the foreline (previously to temperatures in excess of 80 °C) is no longer required. The residual atmosphere in the foreline is now sampled with the foreline at room temperature.

When the contamination levels in the residual atmosphere at a given pump inlet reaches an equilibrium state the ratio calculated from the partial pressure at M/e 69 to that at M/e 2 is used to give an indication of pump contamination. Calculating a contamination value in this manner compensates

$$\begin{array}{l}\qquad\quad CF_3 \\ \qquad\quad\ | \\ CF_3O-(-CF-CF_2-O)_m-(CF_2-O)_n-CF_3\end{array}$$

FIG. 2. General structure of PFPE oils typically used in vacuum applications.

for any signal drift that may occur during the sampling process. The resulting value is a useful indicator to the relative contamination of the residual atmosphere and can therefore be used to quantify production modifications or for cleanliness comparison with other pumps of similar construction.

The modified technique described above is exemplified in the next section by the analysis of the residual atmosphere at the inlet to scroll type vacuum pumps. In addition to bearing lubrication scroll pumps use polytetrafluoroehtylene (PTFE) based materials for the tip seal. PTFE has the structure $[C_2F_4]_n$ and under mass spectral analysis show cracking patterns similar to that of the PFPE oil/grease. However, if PTFE components are present in the residual atmosphere there would be additional peaks characteristic of the matrix additive also identifiable (e.g., $M/e=100$, 148, or 170).

III. RESULTS

The method outlined above was used to assess the degree of contamination in the residual atmosphere at the inlet of several production scroll pumps. The pumps were drawn from a selection of different manufacturers. Details of the pumps evaluated and the ultimate pressures attained after 24 h pumping are outlined in Table I. *Pumps* A, B, D and E were run without gas ballast. *Pump* C is the same pump as D, and was run with intrinsic gas ballast activated. Bearing purges, if present, were unmodified and run under manufacturers recommended standard settings.

The residual atmosphere at the inlet of each of the five pumps was analyzed continuously over a 12-h period and the partial pressure measurements at M/e 69 and M/e 2 logged. Figure 3 shows the partial pressure measurement at M/e 69 against time for each of the pumps. At the commencement of each run the background partial pressure at M/e 69 was typically of the order of 10^{-11} Torr. After 12 h a background pressure was observed with *pump* A whereas *pump* B showed an increase in the partial pressure measured at M/e 69 to 1.2×10^{-10} Torr. In contrast, *pumps* C, D, and E show a significant increase in the partial pressure measured at M/e 69 over the same time period. The partial pressure at M/e 69 rises to 6.3×10^{-9} Torr for *pump* C, 8.1×10^{-9} Torr for *pump* D, and 2.6×10^{-8} Torr for *pump* E.

Figure 4 shows the relative contamination value calculated from the ratio of the partial pressure measured at M/e 69 to that measured at M/e 2 over the same 12-h period. In

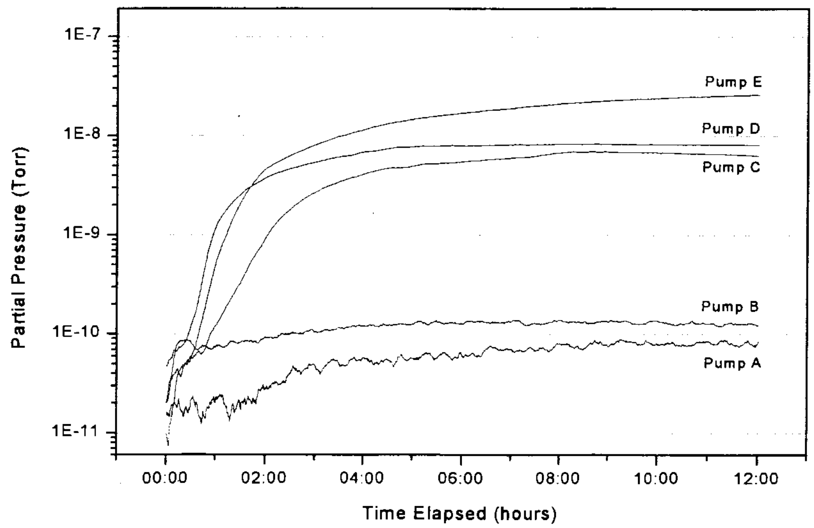

FIG. 3. Trend measurement of the partial pressure at $M/e = 69$ mass peak in the residual atmosphere at the inlet of scroll pumps with a 1.0 m DN25NW foreline at room temperature.

this analysis *pump* A exhibits improved cleanliness over *pump* B. *Pump* A has a value of less than 0.01 compared to 0.03 for *pump* B (the value for *pump* A is attributed to the background signal which is discussed below). The relative contamination values calculated for *pumps* C, D, and E are significantly larger in magnitude than those of *pumps* A and B. *Pump* C has a relative contamination value of approximately 1.2 compared to a value of 3.0 for *pump* D. The results show that the intrinsic gas ballast on *pump* C provides a slight improvement in its cleanliness characteristic (compared to the same pump without the intrinsic ballast, i.e., *pump* D). Although the magnitude of partial pressure measured at M/e 69 in the residual atmosphere of *pump* E was the largest of all the pumps, the calculated contamination value for *pump* E is only 0.36. This is because a larger magnitude partial pressure measured at 2 M/e causes the relative contamination value to be significantly lower than the values calculated for *pumps* C and D. The increased partial pressure measured at M/e 2 may be attributed to a number of factors and will be discussed in more detail below.

It is evident from the plots in Fig. 4 that the relative contamination values are continuing to increase slightly after the 12-h period. Hence, to ensure a stable equilibrium is attained the pumps are left to run at ultimate for a total of 24 h. Figures 5–9 show mass spectrographs of the residual atmosphere for each pump measured after 24 h running at ultimate. *Pump* A shows no measurable signal present at M/e 69, see Fig. 5. In the residual atmosphere of *pump* B a partial pressure of 1.0×10^{-10} Torr at M/e 69 is measured, see Fig. 6. In comparison, the partial pressures measured at M/e 69 for *Pumps* C, D, and E are significantly larger at 3.7×10^{-9} and 8.5×10^{-9} Torr, and 6.5×10^{-8} Torr, respectively, see Figs. 7, 8, and 9.

It is noted from the spectrographs of Figs. 5, 6, 7, and 8 that the hydrogen partial pressure at M/e 2 in the residual atmosphere at the inlet of *pumps* A, B, C, and D is relatively

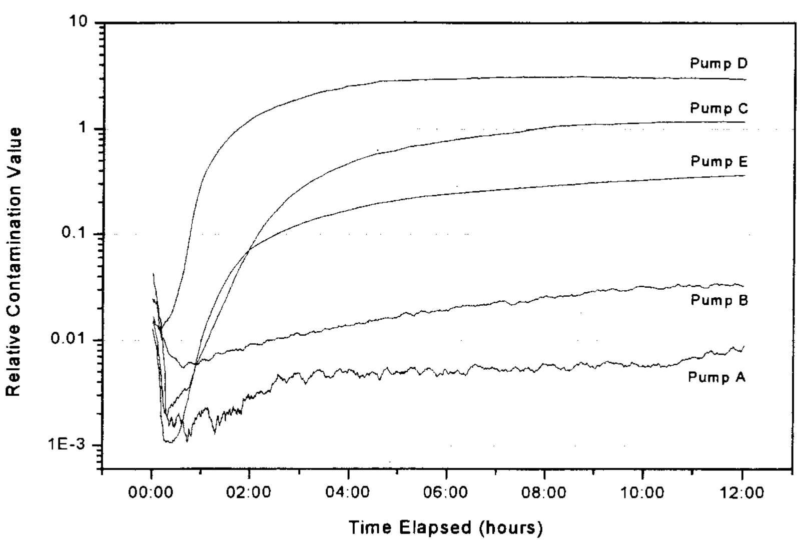

FIG. 4. Calculated relative contamination value in the residual atmosphere at the inlet of scroll pumps over a 12-h period.

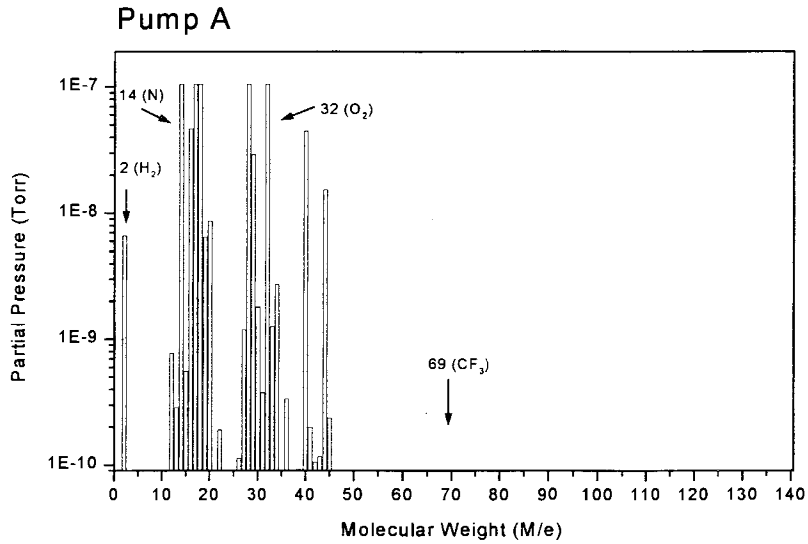

FIG. 5. Mass spectrograph of the residual atmosphere of pump A after 24 h continuous operation at ultimate.

consistent. It is at least an order of magnitude greater for *pump* E, see Fig. 9. The partial pressures measured at M/e 69 and M/e 2 after 12 and 24 h continuous pumping at ultimate are used to calculate relative contamination values for each pump, see Table II. After 24 h the relative contamination value for *pumps* A and B are 0.01 (i.e., background) and 0.02, respectively. The value for *pump* C has increased slightly from 1.12 after 12 h to 1.61 after 24 h. Similarly *pump* E shows an increase in the relative contamination value from 0.36 after 12 h to 0.68 after 24 h. The value calculated for *pump* D after 12 h is 3.04 and decreases to 2.66 after 24 h.

IV. DISCUSSION

From the plots in Fig. 3 it is noted that the contamination measured in the residual atmosphere of *pumps* C, D, and E rises from an initial background level, of the order of 10^{-11} Torr, to equilibrium over a period of approximately 6–12 h. A complex process exists whereby oil vapor backmigrates through the pump to appear as a contaminant in the residual atmosphere. The degree of contamination present at equilibrium is thought to be a function of two competing pump parameters. These are the ability to pump the fluorocarbon vapor against the rate of vapor backmigration from the pump. The quantity of contamination in the residual atmosphere is considered to be a measure of these competing processes and hence gives an indication of the pump cleanliness characteristic.

Table II contains the partial pressures measured at M/e 2 and M/e 69 and includes the calculated relative contamination value (i.e., ratio M/e) 69/2 after 12 and 24 h pumping at ultimate. *Pump* A shows the lowest level of contamination in the residual atmosphere. The absolute partial pressure measured at M/e 69 is 8.8×10^{-11} Torr. The relative contamination value below 0.01 that is calculated for *pump* A is also the lowest value of all the pumps under test. It is to be noted that these numbers for *pump* A are equal to the background noise. This is consistent with the design of this pump

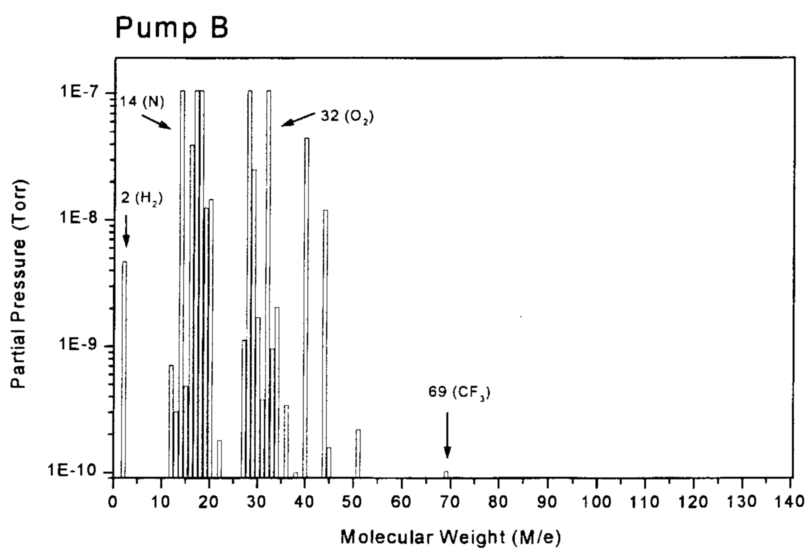

FIG. 6. Mass spectrograph of the residual atmosphere of pump B after 24 h continuous operation at ultimate.

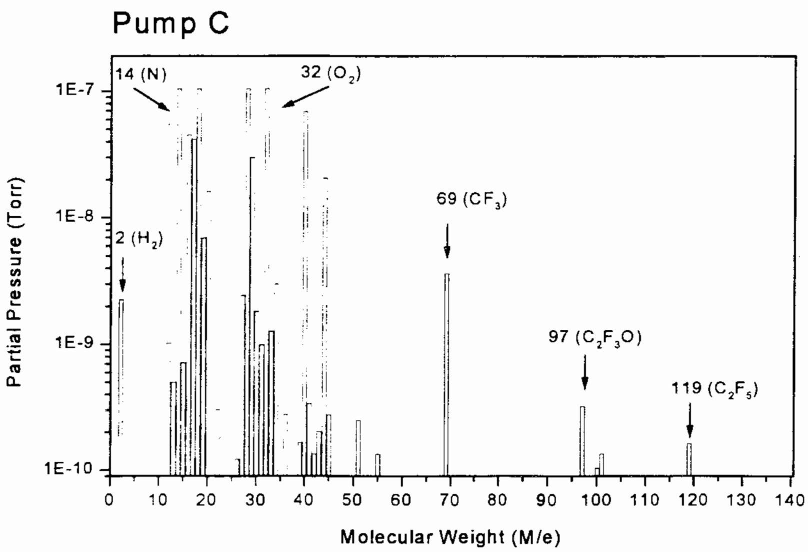

FIG. 7. Mass spectrograph of the residual atmosphere of pump C after 24 h continuous operation at ultimate.

whereby the lubricated bearings are (uniquely) held outside the vacuum space. The level of contamination at the inlet to *pump* A is expected to be zero above background which was indeed the case measured. The quantity of contaminating species in the residual atmosphere of *pump* B is comparable to that of *pump* A. However, the bearings on *pump* B were found to have run dry and a higher level of contamination would be expected from properly greased bearings. The absolute partial pressure measured at mass 69 is 1.0×10^{-10} Torr and a relative contamination value of 0.02 is calculated.

In contrast, the partial pressure measured at M/e 69 in the residual atmosphere of *pump* C is an order of magnitude greater than that of *pumps* A and B. The relative contamination value calculated for *pump* C with intrinsic ballast is 1.61 and *pump* D (i.e., *pump* C without interstage ballast but with bearing purge) is 2.66. The presence of the interstage ballast serves to reduce the level of contamination appearing in the residual atmosphere at the inlet to this pump. It is believed that the additional flow of gas serves to restrict the backmigration of the fluorocarbon vapor and may enhance the ability to pump the fluorocarbon vapor.

Pump E achieved the best ultimate of all the pumps under test, i.e., 4.1×10^{-3} Torr. In order to maintain a consistent sampling rate of 7.0×10^{-5} Torr l s^{-1} and an analysis chamber pressure of 1.0×10^{-6} Torr it was necessary to further open the fine leak valve connecting the analysis chamber to the foreline. The partial pressure measured at the main contamination peak at M/e 69 is 6.5×10^{-8} Torr and the partial pressure at mass 2 is 9.6×10^{-8} Torr, see Fig. 9. The relative contamination value is calculated to be 0.68.

The partial pressure at M/e 69 measured at the inlet to *pump* E of 6.5×10^{-8} Torr is significantly larger in magnitude than the partial pressures measured for *pump* C, 3.7 $\times 10^{-9}$ Torr, and *pump* D, 8.5×10^{-9} Torr. This value in isolation from a reference point indicates that the cleanliness characteristic of *pump* E is inferior to that of *pumps* C and D. However, relating the measurement to the partial pressure

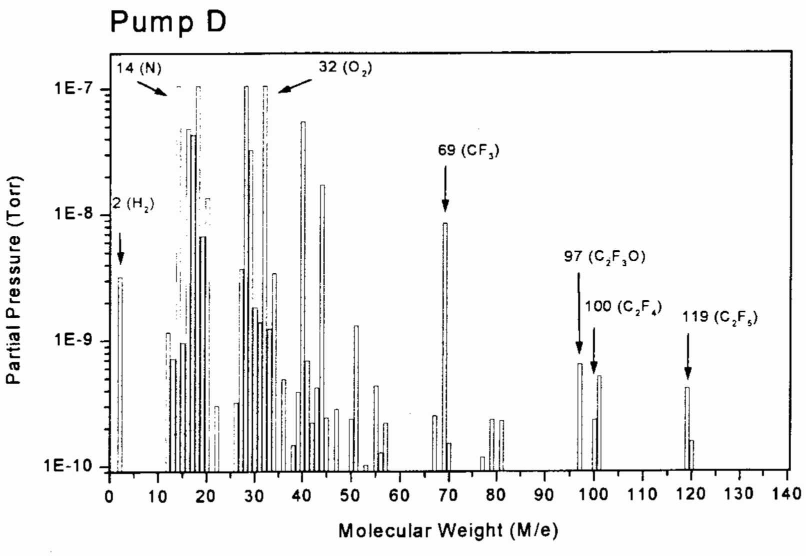

FIG. 8. Mass spectrograph of the residual atmosphere of pump D after 24 h continuous operation at ultimate.

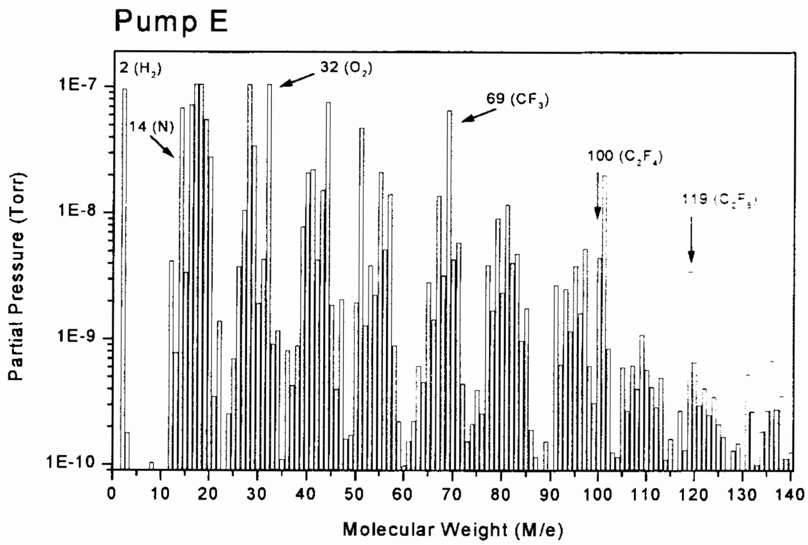

FIG. 9. Mass spectrograph of the residual atmosphere of pump E after 24 h continuous operation at ultimate.

measured at the reference peak of M/e 2 gives a calculated contamination value for *pump* E of 0.68. This represents a significant improvement over values of 1.61 for *pump* C and 2.66 for *pump* D. The improved relative contamination value can be attributed to the partial pressure of 9.6×10^{-8} Torr measured at M/e 2 in the residual atmosphere of *pump* E compared to 2.3×10^{-9} and 3.2×10^{-9} Torr for *pumps* C and D, respectively. The increased hydrogen partial pressure at the inlet of *pump* E may be due in part to the improved ultimate vacuum attained coupled with the requirement to maintain a consistent pressure in the analysis chamber. Adjustments to the fine leak valve are required to maintain a consistent pressure of 1.0×10^{-6} Torr in the analysis chamber. These adjustments will differentially improve the conductance of H_2 through the valve that will lead to an increase in the magnitude of the M/e 2 signal. As discussed earlier, *pump* E attains the best ultimate with a coincident relative reduction in atmospheric O_2/N_2. In contrast, the partial pressures of many other residual gases increase with respect to the overall residual atmosphere, see Fig. 9. Hence, the increase in the partial pressure measured at M/e 2 in the residual atmosphere of *pump* E is indicative of the low base pressure attainable by the test pump and improved H_2 conductance through the fine leak valve while maintaining consistent conditions within the analysis chamber (i.e., opening the fine-leak valve). Subsequent chemical analysis of the lubricating oil from *pump* E has shown that it contains a small quantity of hydrocarbon components. The spectrum of Fig. 9 clearly shows the presence of these hydrocarbon components manifested by the presence of the characteristic CH_2 peak groupings (i.e., at intervals of M/e 14). The presence of hydrocarbon components can affect the M/e 2 measurement and lead to spurious calculations for the relative contamination value. The improved relative contamination level of *pump* E over *pumps* C and D as shown in Fig. 4 when compared with the M/e 69 measurements plotted in Fig. 3 may be due in part to the presence of the hydrocarbon components.

Consequently, measuring the contamination peak at M/e 69 in isolation from a suitable reference peak would be subject to influences that could affect the proper evaluation of the pump cleanliness characteristic. The method employed here to calculate a relative contamination value from the ratio of the contamination peak at M/e 69 to the reference hydrogen peak at M/e 2 compensates for instrumentation variations such as drift and for the differing ultimate pressure attainable by each of the pumps. However, the presence of hydrocarbon components that contribute to the M/e 2 signal will affect the calculation of the relative contamination value and possibly generate a misleading result.

TABLE II. Partial pressures for the 69(CF_3) and 2(H_2) measured in the residual atmosphere at each pump inlet after 12 hours and 24 hours, and the calculated relative contamination values.

	After 12 h at ultimate			After 24 h at ultimate		
Pump	$M/e=69$ (Torr)	$M/e=2$ (Torr)	M/e Ratio 69/2	$M/e=69$ (Torr)	$M/e=2$ (Torr)	M/e Ratio 69/2
A	8.1×10^{-11} (background)	9.4×10^{-9}	<0.01	8.8×10^{-11} (background)	6.7×10^{-9}	<0.01
B	1.2×10^{-10}	4.0×10^{-9}	0.03	1.0×10^{-10}	4.7×10^{-9}	0.02
C	6.3×10^{-9}	5.4×10^{-9}	1.12	3.7×10^{-9}	2.3×10^{-9}	1.61
D	8.1×10^{-9}	2.7×10^{-9}	3.04	8.5×10^{-9}	3.2×10^{-9}	2.66
E	2.6×10^{-8}	7.1×10^{-8}	0.36	6.5×10^{-8}	9.6×10^{-8}	0.68

V. CONCLUSIONS

In this work our previous technique has been further developed to calculate a relative indicator to the level of contamination in the residual atmosphere at the inlet of dry pumps and applied to small commercial scroll pumps. The results show that this technique can be applied successfully to quantify pump performance in terms of the contamination content of the residual atmosphere at the pump inlet. The results presented in this article show that significant differences can exist in the cleanliness performance of scroll pumps from different manufactures.

Pump A showed contamination levels equal to the background. The absolute partial pressure of the background at M/e 69 in the residual atmosphere was 8.8×10^{-11} Torr and a background relative contamination value of <0.01 was calculated. This is to be expected since the internal construction and design of this pump is such that bearing lubricants are entirely isolated from the vacuum space making it impossible for contaminating species to find their way through the pump and into the residual atmosphere. The bearings of all the other pumps (i.e., pumps B, C, D, and E) are exposed to the vacuum space. The measured partial pressure at M/e 69 for the residual atmosphere at the inlet to pump B was 1.0×10^{-10} Torr after 24 h. The relative contamination value was 0.02—noticeably above background. The bearings in pump B were found to have run dry, as discussed earlier, and hence the contamination level could be reasonably expected to be significantly higher with the bearings correctly greased.

Pumps C, D, and E exhibit significantly larger partial pressure contamination signals at M/e 69 thereby indicating that the pumps are a contributing source of contamination to the residual atmosphere. The partial pressure measured at M/e 69 for pumps C, D, and E were 3.7×10^{-9}, 8.53×10^{-9} Torr, and 6.53×10^{-8} Torr, respectively after 24 h pumping at ultimate. The calculated relative contamination values are; pump C (1.61), pump D (2.66), and pump E(0.68); continuous gas ballast accounts for the improved cleanliness of pump C, over pump D though it is still relatively high. These results show the relative cleanliness of several scroll pumps under the same conditions and illustrate that this technique can be a useful tool in quantifying the degree of dry in the residual atmosphere at the inlet to commercial dry pumps.

ACKNOWLEDGMENTS

The authors gratefully acknowledge technical assistance provided by J. Clarke and J. Norman, and also to N. Condon who took part of the data set.

[1]W. Wong, L. Laurenson, R. G. Livesey, and A. P. Troup, J. Vac. Sci. Technol. A **6**, 1183 (1988).
[2]L. Laurenson and D. Turrel, Vacuum **38**, 665 (1988).
[3]N. T. M. Dennis, L. J. Budgen, and L. Laurenson, J. Vac. Sci. Technol. **18**, 1030 (1981).
[4]R. P. Davis, Vacuum **53**, 239 (1999).
[5]D. Sianesi, V. Zamboni, R. Fontanelli, and M. Binaghi, Wear **18**, 85 (1971).
[6]V. L. Woodfin Jr., Anal. Chem. **50**, 1228 (1978).
[7]L. Holland, L. Laurenson, P. N. Baker, and H. J. Davis, Nature (London) **238**, 36 (1972).