

# Application Note #7

## Vacuum Diagnosis With a Residual Gas Analyzer

### Introduction

Residual Gas Analyzer (RGA) is the term for a class of quadrupole mass spectrometers that typically cover mass ranges from 1 to 100 or 200 amu (atomic mass units) and are intended to be used for the analysis of the gasses present in high and ultra high vacuum. The RGA's resolution is sufficient to clearly distinguish peaks that are 1 amu apart. These specifications are a perfect match for the requirements of vacuum diagnosis. Not many materials with a mass greater than 200 amu will be volatile and the high resolution of research grade mass spectrometers is not necessary for the analysis of low molecular weight species. Overall, RGA's are affordable instruments that can be permanently attached to a vacuum system.

The purpose of vacuum is to remove molecules that would interfere with a process or experiment. Although the reduction of total pressure is always a concern in vacuum systems, near operating pressure the real concern becomes the presence of certain species, e.g. oxygen, water, and hydrocarbons. When operating a vacuum system with only a total pressure vacuum gauge, one must rely on the assumption that the total pressure is direct indicator of the partial pressure of these crucial impurities. This assumption presents two problems. First, a total pressure measurement cannot tell the user whether the vacuum system is filled with water, nitrogen, carbon dioxide or hydrogen. Second, total pressure measurement is not very precise—measurements better than 10% are difficult and expensive. If a system is operating at a standard pressure of  $1.0 \times 10^{-7}$  and the pressure rises to  $1.1 \times 10^{-7}$  is there a cause for concern? The additional 10% could be harmlessly inert or it could be oil vapor. The RGA is designed to address exactly this question. It immediately tells the user **what** is in their vacuum system.

The primary application of the RGA is to analyze the composition of a vacuum system. The composition can be used to detect impurities, monitor gas fills, or analyze chemistry that is occurring. The second application of the RGA is as an intrinsic leak detector. It serves this purpose very well and is in many ways superior to a portable helium leak tester. In the remainder of this application note we will illustrate the usefulness of an RGA. The data shown are real results from experiments designed to illustrate certain points.

### Composition Analysis

Stanford Research Systems' RGA software allows the composition of a vacuum system to be analyzed by two

methods. The most common is to measure the mass spectrum of the vacuum. This provides a "fingerprint" of the vacuum composition. A second method is to track specific species or peaks of the mass spectrum. The first method, analog scan mode, is most useful when the user does not know what is present in the chamber. Once the identities of the species have been determined, individual peaks can be tracked using either pressure vs. time, table or annunciator mode.

### The Mass Spectrum

The fundamental operation of the RGA is as a mass spectrometer. Figure 1 shows a graph of partial pressure versus mass, which was measured with an RGA with an electron multiplier detector. The scan was taken of a vacuum system near its ultimate vacuum. The pressure axis is plotted on a logarithmic scale so that a

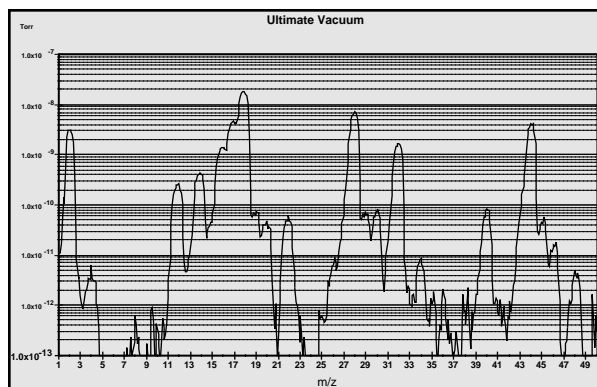


Fig.1: Partial Pressure vs. Mass

large range can be seen. The log scale makes the peaks appear wider than when plotted on a linear scale. This scan from 1 to 50 amu shows some gasses commonly present in vacuum chambers. There are many peaks, but they are caused mainly by 7 species. Hydrogen is at 2 and helium at 4. Water gives primary peaks at 16, 17, and 18 due to the species  $O^+$ ,  $HO^+$ , and  $H_2O^+$ . The smaller peaks at 19 and 20 are due to  $^{18}O$  which is naturally present at 0.2%. Nitrogen is at 28 and also causes the peaks at 14 by atomic  $N^+$  and the doubly ionized  $N_2^+$ . Molecular oxygen shows a peak at 32 and an isotope peak at 34. Argon shows a peak at 40. Carbon dioxide shows a peak at 44 and a peaks for  $CO_2^{++}$  and  $C^+$  at 22 and 12. The other peaks are caused by fragments of these species and contaminants.

The presence of air components in the spectra might lead us to believe that the system is leaking, but this is untrue. The hybrid turbomolecular pump has simply

reached its compression limit. The foreline of the pump was operating at a total pressure of 0.5 Torr; therefore the compression ratio is about 108 (as the pump specifications indicate). Nitrogen, oxygen and argon are all present in the same ratios as standard atmosphere. The presence of helium is interesting, because it is present in the atmosphere at about 7 ppm. Its peak might be expected 6 decades smaller than the nitrogen peak. The low compression ratio of the turbo pump for helium ( $10^5$ ) explains why the peak is only three decades smaller.

The ability to detect these common species and many others is the essence of the RGA. The fragmentation of molecules in the ionizer of the RGA gives each molecule a distinct fingerprint. The fragmentation patterns for many molecules is available from the library of the SRS RGA software. Keeping a historical record of the typical spectrum of a vacuum system allows the appearance of new peaks to be instantly detected. For instance, the peak at 48 in Figure 1 is SO from  $\text{SO}_2$ . On occasion this peak is seen in our chambers. In addition to simple gasses such as  $\text{SO}_2$ , we are also interested in molecules with higher weights

### Oil Contamination

Figure 2 contains a mass spectrum of a common contaminant of vacuum systems: oil. Figure 2a shows measured data while figure 2b shows a spectrum of pump oil from the library. The presence of mechanical pump oil is immediately obvious. The peaks at masses 39, 41, 43, 55, and 57 are caused by mechanical pump oil backstreaming into the vacuum chamber during a load lock sequence. The total pressure in the chamber was dominated by water and was less than  $2 \times 10^{-8}$ . In this case, the total pressure might satisfy operating conditions but the spectra reveals that the system is heavily contaminated with oil. This could have been caused by improper valve sequencing or a saturated oil trap. Without an RGA, only operating procedures for valves and a maintenance schedule for traps can ensure that the cleanliness of a vacuum system is maintained. With the RGA, cleanliness can be proven **before** a process or experiment begins.

### Solvent Contamination

Oil contamination is common in vacuum systems. Cleaning parts with solvents is a common approach to removing this contamination. We have observed that organic solvents such as acetone and TCE are more tenacious contaminants than the oil they are designed to remove. Figure 3 shows a spectrum indicating conta-

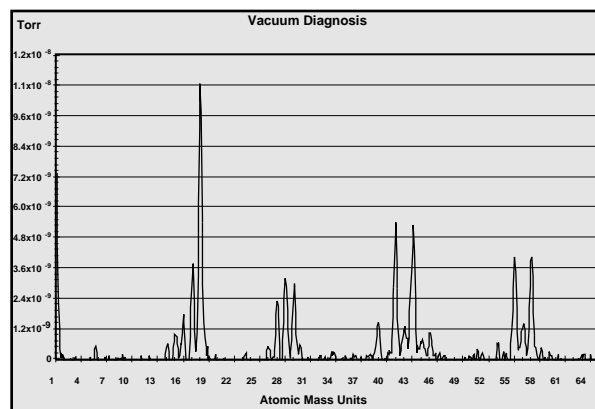


Figure 2a: Pump Oil Contamination

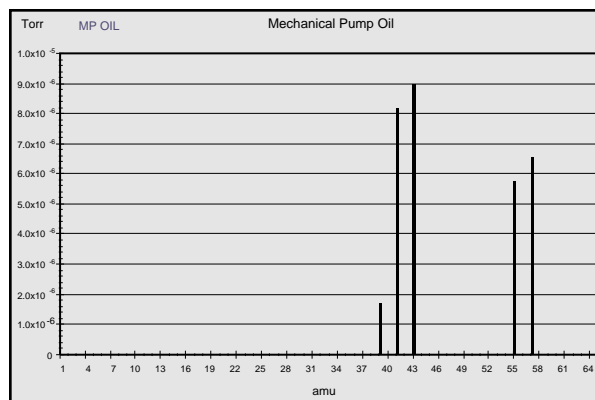


Figure 2b: Library Pump Oil Data

mination with 1,1,1-trichloroethane as shown by the major peaks at 97 and 99, and the minor peaks at 61, 63, 117 and 119. The paired peaks are caused by the natural isotopic occurrence of  $^{35}\text{Cl}$  and  $^{37}\text{Cl}$  (75% and 25%). This spectrum was measured one week after the initial contact with the solvent. The TCE permeated into the o-rings in the system during a cleaning step. The TCE continued to outgas from the O-rings for two weeks and showed no signs of stopping. At that time they were removed and baked in an oven, which eventually removed the TCE.

Data like this is invaluable to the development of cleaning procedures. While the TCE successfully removed the oil, the vacuum chamber was left more contaminated than it would have been without the cleaning. The mass spectrum provides a more accurate evaluation of cleaning procedures than pump down time and base pressure. Just because a system pumps down quickly does not guarantee that an undesirable contaminant is not present. The large dynamic range also allows eval-

uations to be made more quickly. The user does not have to wait several hours for the water to pump away to determine if a system is contaminated. The RGA can make measurements in the presence of a high water partial pressure.

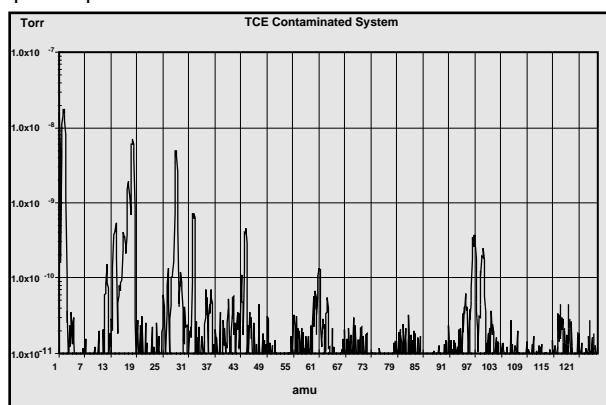


Figure 3: TCE Contaminated System

### Peak Measurement

For vacuum systems that only need to be clean, the mass spectrum is the most useful measurement. During experiments and processes the partial pressure of certain species is of more interest. The RGA software provides three modes that are used to measure selected peaks. The selection of which mass is associated with which species is usually straightforward, i.e. the mass of the molecule is chosen. When two species have overlapping peaks patterns, the user chooses the strongest peak that does not interfere. For example  $N_2$  and  $CO$  both have a mass of 28. In a system with a large  $CO$  interference,  $N_2$  could be measured at mass 14. Because the peak at 14 is smaller than the major peak, a scaling factor is required. The set of peaks of interest are entered into the RGA software by the user. During measurements, the RGA measures only each peak. Because the whole spectrum is not recorded, data is acquired much faster. The two examples that follow show an interesting example of pressure vs. time measurements and a method of increasing the dynamic range of measurements.

### Pressure vs. Time

Figure 4 shows the use of the P vs. T Mode to monitor an airlock sequence. The process opens an air lock, places the sample in it, and moves the sample into the main vacuum chamber. The air lock is pumped from atmospheric to rough vacuum using the same mechanical pump as the main vacuum chamber. This requires

isolating the foreline of the turbo pump during the time the sample is in the airlock. To keep oxygen out of the main vacuum chamber, the airlock is flushed with dry nitrogen then pumped to rough vacuum. The flush is repeated two additional times before the sample is finally transferred. If this procedure is successful, the main vacuum chamber should not be disturbed. The text which follows gives a detail of the events.

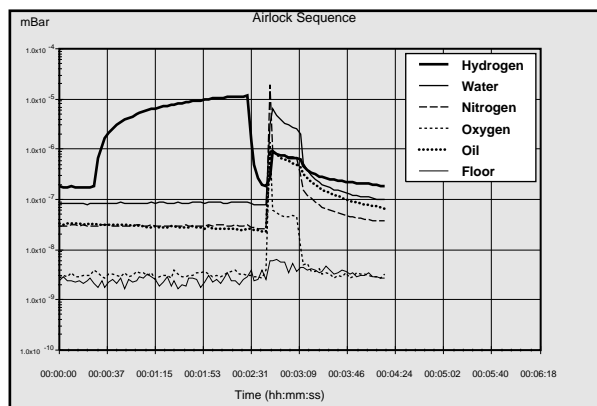


Figure 4: Airlock Sequence

To make these measurements, the electron multiplier detector has been used with a gain of 100, which allows all six channels to be recorded every three seconds. The standard Faraday cup detector is able to detect these partial pressures, but not at this rate. The "floor" channel is set to mass 21. There is rarely anything present at this mass, which allows it to be used as an indicator of the noise floor.

The sequence starts with the main vacuum chamber at its base pressure of  $2 \times 10^{-8}$ . The chamber is pumped by a turbomolecular pump that is backed by a rotary vane pump. A small load lock is attached to the chamber, which can be rough pumped by the same mechanical pump and purged with nitrogen.

At 0:30, the isolation valve between the turbo pump exit and mechanical pump is shut so that the mechanical pump can be used to rough pump the load lock. During this time, the load lock is repeatedly filled with nitrogen and pumped down. Of interest in the data, is the rise in the hydrogen partial pressure during this step of the sequence. The partial pressure of hydrogen increases by a factor of 100 while the partial pressure of the other gasses barely increases. This difference is caused by the low compression ratio that turbo pumps have for light gasses. The heavier gasses are being compressed into the dead volume between the turbo pump exit and isolation valve. But, the turbo pump has insuffi-

cient compression ratio to store hydrogen in this manner, causing the partial pressure of hydrogen to rise.

At 2:30, the load lock has been roughed and the turbo pump foreline isolation valve is opened. The pressure of H<sub>2</sub> immediately drops back to the base pressure value. At 2:40, the load lock is opened to the main chamber causing a jump in pressure. The rise in oxygen and oil pressure indicates that the procedure is operating poorly. Even though the load lock was purged three times with 99.999% nitrogen, oxygen was still introduced into the chamber. This was either caused by a small air leak into the load lock, or permeation of oxygen out of the elastomer seals on the load lock. The rise in oil partial pressure indicates that the trap on the mechanical pump is exhausted and allowed oil to backstream into the load lock.

At 3:09, the valve between the load lock and main chamber is closed, and the pressures begin to return towards their base values. Oxygen is pumped out of the chamber. Hydrogen, water, and nitrogen recover their original values, but slowly. The oil is alarming because it persists at a higher concentration. If this sequence occurred several more time, the oil would continue to step up. The RGA allows it to be detected before reaching undesirable values.

**Table Mode**

The noise floor of the Faraday cup detector is about 10<sup>-10</sup> mbar. Since the maximum operating pressure is 10<sup>-4</sup> mbar, the dynamic range of the RGA is 6 decades or 1 ppm. The noise floor of the channel electron multiplier (CEM) is lower, but its maximum operating pressure also decreases with the noise floor. The two pressure limits change such that the dynamic range of the CEM is still 6 decades. By switching between the two detectors, measurements covering more than 6 decades can be made. The table mode of the SRS RGA software allows such a measurement to be made. The CEM status can be set independently for each mass being monitored. Figure 5 shows a configuration where the prevalent gasses are detected with the FC and the low pressure gasses are detected with the CEM. A comparison of the value for nitrogen and “floor” show that the apparent dynamic range is 8 decades or 10 ppb. The program will automatically sort the channels so that all the measurements requiring the CEM are made as a group, which minimizes the switching on and off of the detector. Without the ability to choose between FC and CEM detector for each channel, the CEM would have to be used for all channels so that the low pressure gasses could be detected. Operation like

Ch#	Name	Mass	Value	Alarm	Speed	Cal	CEM
1	Hydrogen	2	3.8E-07	NORMAL	1	1.00	OFF
2	Water	18	7.1E-08	HIGH	1	1.00	OFF
3	Nitrogen	28	1.4E-05	HIGH	1	1.00	OFF
4	Oxygen	32	4.6E-10	NORMAL	3	1.00	ON
5	CO2	44	3.4E-11	NORMAL	3	1.00	ON
6	Oil	55	1.6E-12	NORMAL	3	1.00	ON
10	Floor	21	1.5E-13	NORMAL	1	1.00	ON

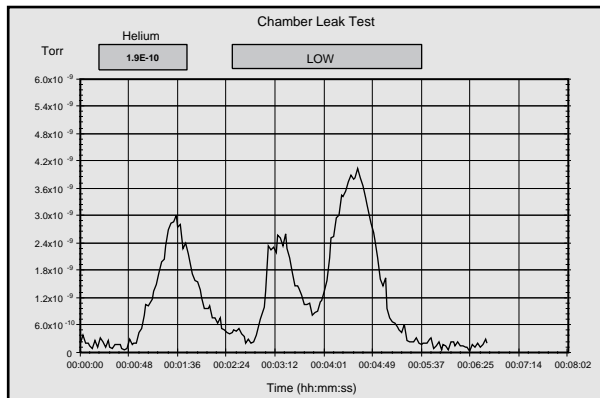
**Figure 5: SRS RGA Table Display**

this would cause the CEM detector to saturate at the high pressure peaks. Saturation of the detector makes the value useless, and also increases the physical wear rate of the CEM.

**Leak Testing**

In addition to the diagnosis of vacuum systems, the RGA is invaluable as an intrinsic leak detector. It is always available and does not require perturbing the system. The user does not have to roll up a large leak detector and attach it to the vacuum system. The system does not have to be brought up to atmospheric pressure. The RGA can operate in leak detection mode using any gas, so it does not require helium. For moderate leaks, argon or tetrafluoroethane (a typical gas in cans of aerosol dust remover) can be used. Only for the smallest leaks is helium necessary. Having a built-in leak detector makes working with vacuum systems much easier and faster, and the SRS RGA is far less expensive than a leak detector.

The process of leak detection with an RGA is the same as with a traditional helium leak detector. Place the software in leak detection mode, indicate the mass of the test gas and watch the partial pressure as various joints in the vacuum system are sprayed with the test gas. When the leak is sprayed with the test gas, the partial pressure will rise. The response is immediate if the leak is a direct path from the outside to the inside of the system. Figure 6 contains the result of a leak test with helium on a vacuum chamber. The tester moves the helium probe towards and then past the leak, causing the first peak. Once the location of the leak is bounded, the tester goes back to exactly locate the leak. For most situations leak testing is straightforward and no different than traditional methods. In the following sections, we discuss a few situations where traditional methods fail and how the RGA provides a better method.



**Figure 6: Helium Leak Test Data**

The partial pressure of the test gas is directly related to the leak rate into the chamber. Assuming that the vacuum pump is not operating near its compression limit, the throughput of the test gas is equal to the product of the partial pressure and the effective speed of the pump at the RGA ionizer ( $Q = S \cdot P$ ). In Figure 5, the partial pressure was measured in Torr. The effective speed of the turbo pump for helium was approximately 50 liter  $s^{-1}$  and the largest peak of  $4 \times 10^{-9}$  represents a leak rate of  $1.5 \times 10^{-7}$  scc/sec. From this figure we can estimate a minimum detectable leak of  $1 \times 10^{-8}$  scc/sec which is measured with the Faraday Cup detector. To measure smaller leaks, the electron multiplier detector can be used, or the turbo pump can be throttled to decrease the pumping speed.

### Supply Gas Valve Seats

Leaks across valves that supply gasses to a vacuum system cannot be detected with conventional helium leak testers (unless the valve supplies helium). To test a suspect valve would require removing it and attaching it to the leak tester. Because the RGA can monitor any gas, this is unnecessary; the valves on gas supply lines can be tested in situ. The procedure is simple: monitor the composition of the vacuum system with a high pressure and low pressure behind the valve seat in question. If the partial pressure of the gas in question changes, the valve seat is leaking.

### Supply Gas Manifolds

Leak testing supply gas lines can be a very trying experience. The difficulty is largely because compression type fittings do not have a leak test port. Another difficulty is that supply manifolds commonly have a large number of connections in close proximity. Because the leak in a compression fitting is inside the fitting, transporting the test gas to the leak requires a large flowrate

and waiting for an extended time for the gas to diffuse into the fitting. Because of the flowrate and time, it is possible that the test gas can travel to adjacent tube fittings and cause a misleading indication of a leak. On many occasions we have observed "fugitive" leaks that appear and disappear at a specific fitting. What happens is that the test gas inadvertently flows to another fitting of the manifold. Confining the test gas to the fitting under question can help, but the RGA provides an easier solution: use a gas other than helium. Helium will spread in air quickly and diffuse into many fittings. A heavy gas like argon or tetrafluoroethane is far easier to confine to a specific fitting. Once the moderate leaks have been located and eliminated, a follow up with helium to check for tiny leaks is warranted.

### Bellow Valves

Bellow valves can be difficult to leak test due to the large volume of gas contained between the bellow and the valve body. To perform a quick leak test it is required to change the composition of the gas in this trapped volume quickly. Unfortunately, for some valves, this volume of gas is not highly accessible. This greatly reduces the response time of a leak test. For a leak causing a base pressure of  $10^{-7}$  mbar in a vacuum system with a 70 l/s pump, the volumetric flowrate of gas entering from the atmospheric side of the leak is 7 nl/s. For a bellow with a trapped volume of 1 ml, the response time constant would be over 40 hours. This emphasizes the importance of leak test ports on vacuum hardware.

In such situations it is common to place a bag over the body of the valve and fill the bag with helium. The RGA allows us to consider using gasses other than helium. The permeability of helium through elastomeric seals can give a false leak reading. And as in manifolds, unless the helium can be strictly confined to the valve body, it may spread to adjacent connections. Given the amount of work and lost time required to remove and repair large valves, false leak readings are expensive. A second test with another gas, such as argon, can confirm that a suspect valve is leaking before starting out on the repair.

### Conclusion

An RGA is a real eye opener for users of vacuum systems. With an RGA the process of working with vacuum systems is elevated from an empirical trial and error approach to a systematic approach. It is hard to imagine anybody who has worked on a vacuum system with an RGA ever wanting to "go back" to a system without one.