CONF-771110--3

The Nitrogen Pressure in A Vacuum System*

D. Edwards, Jr.
Brookhaven National Laboratory
Upton, New York 11973

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Abstract

MASTER

Since the N_2 outgassing rate from walls of a vacuum system is generally recognized to be very small, the presence of a N_2 pressure in the system is a useful indication of the presence of real leaks to the atmosphere. A technique of thermal desorption mass spectrometry is introduced which allows the N_2 pressure in the background gas of a baked system to be separated from a CH_4 background to a minimum level of N_2 detectability of $\sim 1\%$ of the CO background pressure. In typical baked systems (of which an example is given), it is possible to readily determine the sum of all real N_2 leaks to atmosphere at a level of $\sim 10^{-11}$ Torr liter/s. This feature makes the technique ideally suited to large complex systems since the leak rate can be evaluated before detailed leak finding procedures are initiated.

* Work performed under the auspices of the U. S. Energy Research and Development Administration.

I. Introduction

It is generally recognized that the nitrogen pressure in a vacuum system is considerably less than many other gas components provided that there are no real leaks from the atmosphere into the system. Thus by measuring the N_2 pressure (knowing the N_2 pumping speed) the leak rate from the sum of all leaks to atmosphere may be determined before any elaborate leak testing procedures are initiated.

At present the following techniques are available for a N_2 pressure measurement:

- i) the excitation of the background gas and the subsequent observation of the characteristic N_2 light emitted in the de-excitation process.
- ii) a very high resolution mass spectrometer capable of separating $N_2(28)$ from either $CO(28)$ or $C_2H_4(28)$.
- iii) a low resolution mass spectrometer used in connection with a cracking pattern analysis.
- iv) a low resolution mass spectrometer combined with a thermal desorption technique as described below.

II. Description

Consider that the N_2 pressure is to be determined in a vacuum system which has a 10-20 liter/s N_2 pump and a mass spectrometer. The problem of using a low resolution (~ 1.0 AMU) mass spectrometer is clear from Fig. 1. This spectrum is typical of a 10^4 cm^2 system previously baked 24 hours at 200°C with a 20 liter/s conductance connecting the system to an $\sim 10^3$ liter/s cryopump. For the instrument resolution of ~ 1 AMU, N_2^+ of mass 28 is degenerate with CO^+ of mass 28. Using mass 14 (N^+) eliminates the C^+ and O^+ background from

the CO parent but is again degenerate primarily with CH_2^+ (from a CH_4 parent) and less significantly with CO^{++} (doubly ionized CO).

Using a low resolution mass spectrometer the separation of N_2 from CO and CH_4 may be made in the following manner. At $t = 0$ let an auxiliary tungsten filament (not necessarily in line of sight to the mass spectrometer) be flashed clean. The background gas of the system is then allowed to adsorb on the cleaned filament for T seconds. At the end of the adsorption interval the filament is rapidly flashed while mass 14 is monitored. Let the peak current in amperes of the mass 14 desorption transient be denoted by F_{14} in units of "flash amperes" (FA). Since CH_4 does not adsorb on a clean tungsten surface at ~ 300 K there is no contribution to the mass 14 desorption signal from CH_2^+ . Since both N_2 and CO will adsorb on the filament during the adsorption interval there will be a rise in the CO pressure during the desorption and hence a CO^{++} signal will contribute to the mass 14 current. Thus $F(14)$ must be corrected for this contribution and is written:

$$(1) \quad F_c(14) = F(14) - 1.9 \times 10^{-3} F(28, \text{CO})$$

where $F(28, \text{CO})$ is the mass 28 amplitude due to CO and the coefficient (1.9×10^{-3}) is determined from the cracking pattern of CO (see Table I).

The N_2 pressure is then determined by calibrating the device by establishing a known N_2 pressure and measuring $F_c(14)$. For our system and flash procedure the calibration coefficient was found to be ~ 4.6 Torr/FA. Thus the N_2 pressure is found to be:

$$(2) \quad P_{\text{N}_2} = 4.6 F_c(14) \text{ Torr}$$

2.1 An example

The filament which was found convenient to use was one of the tungsten filaments in a standard ionization gauge. The flash consisted of a constant

3 A current supplied for 5 s. The mass spectrometer output during the flash, shown in Fig. 2, was recorded for masses 28 and 14. From these simple measurements the N_2 pressure is easily determined from Eqs. (1) and (2). The result was that in a background pressure of $\sim 1.2 \times 10^{-10}$ Torr (H_2), 1.5×10^{-10} Torr (CO) the N_2 pressure was found to be 1.25×10^{-11} Torr.

2.2 Cracking pattern analysis

As a check of the above result the N_2 pressure was determined by a cracking pattern analysis. The cracking patterns (at $\sim 10^{-9}$ Torr) for various gases were measured with the present mass spectrometer and are given in Table I. The listed gas purities were at least 99.97.

Immediately prior to the N_2 pressure determination discussed in paragraph 2.1, the background gas spectrum was recorded and is shown in Fig. 3. From this data the cracking pattern analysis proceeded in the following manner with $I(M)$ denoting the mass spectrometer current of mass M :

1. $I(13)$ is corrected for C_{13}^+ using $I(12)$. [$\sim 0.01 I(12)$]
2. Using $I(13)$, $I(CH_2^+)$ is found. [$2.1 I(13)$]
3. Using $I(15)$, $I(CH_2^+)$ is found. [$0.179I(15)$] Note that steps 2 and 3 are redundant.
4. From $I(28)$ find $I(CO^{++})$. [$0.0019I(28)$]
5. $I(N^+) = I(14) - I(CH_2^+) - I(CO^{++})$
6. Find $I(N_2^+)$ from $I(N^+)$. [$I(N^+)/0.104$]
7. $P_{N_2} = K I(N_2^+)$

K being determined from a separate calibration.

Using the data from Fig. 2, the result obtained from the cracking pattern analysis is:

$$P_{N_2} = \begin{cases} 1.49 \\ 1.25 \times 10^{-11} \text{ Torr} \end{cases}$$

the two values depending upon whether I(13) or I(15) was used to determine the CH_2^+ background present in I(14). The above result is in reasonable agreement with that made by the desorption technique.

2.3 Dependence on N_2 exposure

The linearity of $F_c(14)$ to the N_2 pressure for a constant adsorption interval is shown in Fig. 4. Seen is that $F(14)$ is proportional to the N_2 pressure, the largest N_2 exposure being 0.07 L. In Fig. 5 the dependence of $F_c(14)$ to the adsorption interval during background gas adsorption is shown. Again no significant deviation of $F_c(14)$ from linearity was observed even for exposure times as large as 10^3 s. The N_2 pressure in the measurements presented in Fig. 5 was 1.6×10^{-12} Torr* corresponding to a N_2 flux into the system of 3×10^{-11} Torr liter/s.† Thus the technique seems capable of measuring the N_2 pressure in a CO background 100 times larger and for a baked system with modest pumping of determining the total leak rate from atmosphere to $\sim 10^{-11}$ Torr liter/s. It should in fact be possible by using a selective getter (i.e., one which pumps CO but not N_2) to reduce the minimum detectable leak rate to a level significantly less than 10^{-11} Torr liter/s.

3. Summary

A desorption technique has been introduced which permits the N_2 pressure in a vacuum system to be measured by an operationally simple procedure with a modest resolution mass spectrometer. This technique may be extended to situations in which a small amount of adsorbable gas is to be measured in the presence of a large nonadsorbable background such as D_2 in He_4 .

* The lower N_2 pressure found in Fig. 5 as opposed to Figs. 2,3 resulted from cleaning the cryopump of condensed N_2 .

† If the influx of N_2 was from the interior surface of the vacuum chamber uniformly outgassing N_2 , an outgassing rate of $\sim (2-3) \times 10^{-15}$ Torr liter/s cm^2 would result. However, it is not possible in the present situation to separate the contribution of real leaks to atmosphere to the measured influx of N_2 .

TABLE I

CH_4	%	
16 (CH_4^+)	100	
15 (CH_3^+)	84	$14/15 = 0.179$
14 (CH_2^+)	15	
13 (CH^+)	7	$14/13 = 2.14$
12 (C^+)	~ 2	
N_2		
28 (N_2^+)	100	
14 (N^+)	10.2	
CO		
28	100	
16 (O^+)	2	
14 (CO^{++})	0.19	
12 (C^+)	6	
A		
40 (A^+)	100	
20 (A^{++})	4-6	

FIGURE CAPTIONS

- Fig. 1 Mass spectra of a 20 L, 10^4 cm^2 system previously baked to $\sim 200^\circ \text{C}$.
- Fig. 2 In the first 3 flashes mass 28 is recorded, whereas in the second 3, mass 14 is recorded. From this measurement the N_2 pressure is determined as described in the text.
- Fig. 3 Mass spectra used in the cracking pattern analysis.
- Fig. 4 $F(14)$ is plotted as a function of N_2 pressure for a constant adsorption interval.
- Fig. 5 $F_c(14)$ for various adsorption intervals.

6-23-1

SYSTEM SPECTRA, $S_{N_2} \approx 20 \text{ l/S}$

N₂ DETECTION

6-51-4

M.S. CURRENT

2

1

0

$$P_{N_2} = 4.59 \left\{ F_{14} - 1.9 \times 10^{-3} F_{28} \right\} \text{ TORR}$$

10⁻⁸ A/IN

10⁻¹¹ A/IN

GAIN

28

14

MASS

F₂₈

F₁₄

$$P_{N_2} = 1.25 \times 10^{-11} \text{ TORR}$$

0

1

2

3

TIME (100S)

CRACKING PATTERN ANALYSIS

ΔF_{14} VS $P(N_2)$

55S EXPOSURE
3A, 5S FLASH

$\Delta F_{14} \times 10^{-10}$ FA

$P(N_2) \times 10^{-9}$ TORR

ΔF_{14}^C VS EXPOSURE TIME

BACKGROUND

 $P(H_2) = 1.0 \times 10^{-10}$ TORR $P(CO) = 1.0 \times 10^{-10}$ TORR $P(N_2) = 1.6 \times 10^{-12}$ TORR $\Delta F_{14}^C \times 10^{-12}$ FA