UNIVERSITY OF WATERLOO

Physics 360/371 – Experiment 13

Mass Spectrometer Gas Analyser

A commercial residual quadrupole gas analyser is used to accomplish the mass spectrometric analysis of several gases.

The Vacuum System: A conventional vacuum system is used to provide the necessary vacuum to operate the mass spectrometer. See page 2 for schematic. The instrument must not be operated at pressures higher than 10^{-3} Torr. The pump down and shut down procedures for the vacuum system are most important. See pages 4 and 5. All operations must be done in the order given.

Caution - to prevent unnecessary contamination of the mass spec head: (a) the cold trap should be N_2 filled whenever H.V. valve is open. (b) close H.V. valve for periods over 1/2 hour so that the instrument will not be in use. Turn quadrupole spectrometer to standby.

The Mass Spectrometer: The Quadrupole technical information manual provides a description of the mass spectrometer and its ion source. This Quadrupole Spectrometer is capable of a scanning from 0 to 64 atomic mass units. See page 7 for outline of scan parameters.

The Experiment:

(a) Follow outline on pages 4 and 5 (steps 1 and 2) to put the vacuum system in the operating condition.

(b) Setting up the mass spectrometer scan. Use the OMG064 Mass Spectrometer Outline (page 6).

Note that default values are present on turning the spectrometer on. Set up a suitable scan (first mass, width, range and speed) by pressing FUNCT until the desired function is indicated and set the value with the numeric or +/- keys. Press MODE to get back to the measurement mode. Pushing the scan button starts or stops scan. Be sure to manually lift the recorder pen before scan ends. Recorder ranges should not be adjusted!

(c) When plotting spectra — do a background run on the residual gas in the system before introducing the sample gas.

The background should be plotted at the bottom of the page; the main scan is then plotted with a vertical offset of about 1/2 inch. There is no need to otherwise adjust the recorder.

(d) Plot Spectra for the following gases. Air, nitrogen, argon, neon, CO_2, and the unknown gases provided. See page 6 for gas handling instructions.

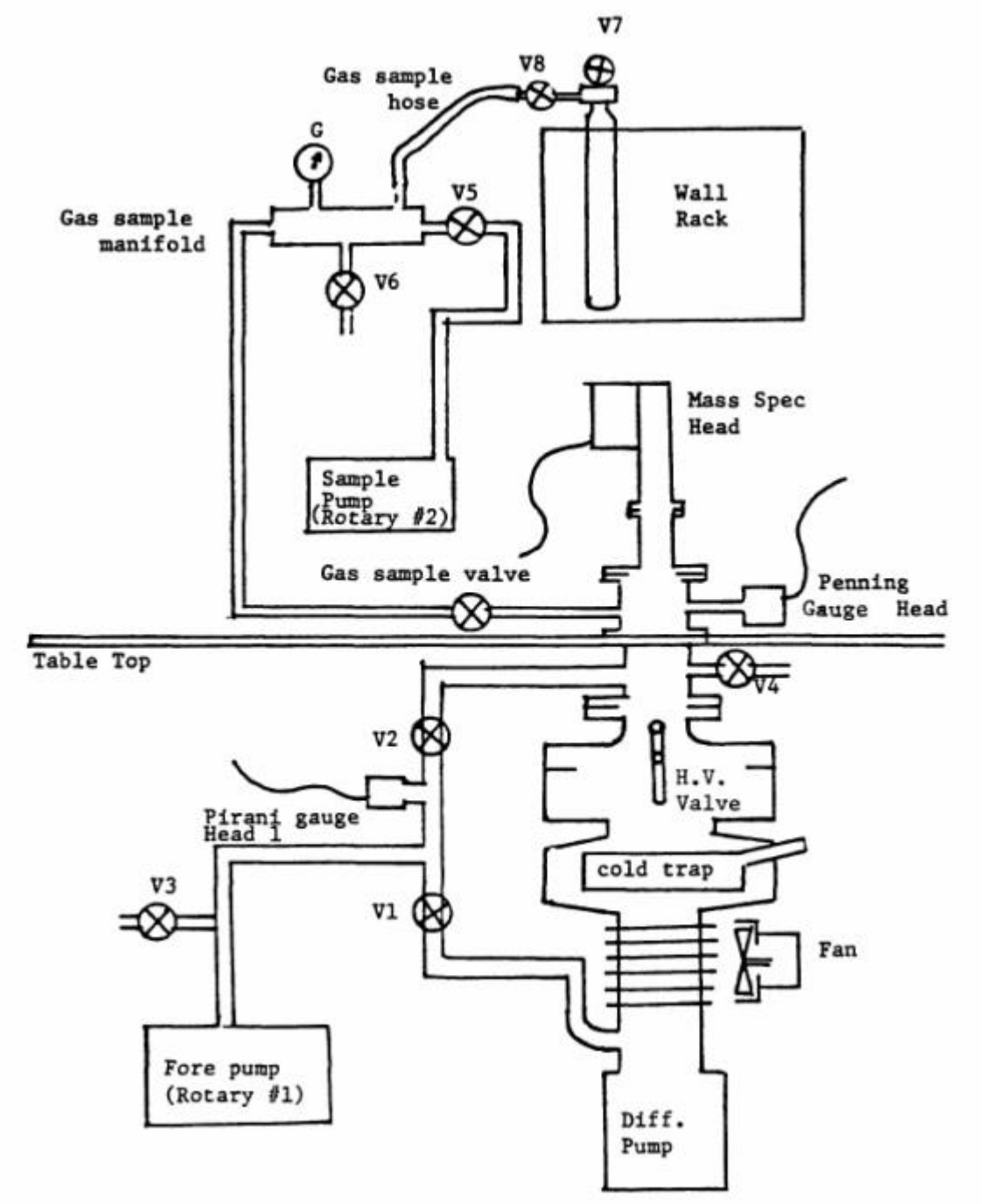
(e) You should compare your mass spectra of known gases with those given in Compilation of Mass Spectral Data, 2nd edition by Cornu and Massot (Heyden & Son, 1975). In this reference, spectral peak heights for a standard ion source are given. You may also identify your unknowns this way. You should check the Neon isotopic ration by comparing your result with that of the nuclear chart on the wall in the lab entrance.

(f) Follow outline on page *5* for shutting down the vacuum system.

Codes

V1 – foreline valve
V2 – roughing valve
V3 – foreline relief valve
V5 – sample pump valve
V6 – manifold relief valve
V7 – main cylinder valve
V8 – control valve
G – sample pressure gauge

Schematic: The Vacuum System



Operating the Vacuum System

1. **Starting up from shut down condition**

(a) The state of system should be set as follows:

- roughing valve - closed
- H.V. relief valve - closed
- foreline relief valve - closed
- H.V. valve (Throttle valve) - closed
- foreline valve - closed
- sample gas valve - closed (do not over tighten this valve: it is a precision needle/shut off combination valve)
- pumps and gauges - off

(b) Turn on Pirani gauge.

(c) Start fore pump. (Rotary #1)

(d) When Pirani gauge reading drops to 100 millitorr, open foreline valve V1.

(e) When the Pirani pressure drops to 100 millitorr or lower turn on the diffusion pump (switch up to heater/fan position).

(f) Wait 20 minutes – fill cold trap with liquid nitrogen.

Observe periodically to ensure liquid nitrogen is maintained in trap. Vacuum system is now in standby condition.

2. **To Change from standby to operating condition**

(a) The initial state of valves etc., is as follows:

- roughing valve - closed
- H.V. relief valve - closed
- foreline relief valve - closed
- H.V. valve - closed
- foreline valve - closed
- gas sample valve - closed
- fore pump (rotary #1) - closed
- diffusion pump - closed
- Pirani gauge - closed
- Cold Trap - N_2 filled

(b) Close foreline valve V1. (see note I, page 5)

(c) Open roughing valve V2, observe Pirani gauge – It may or may not rise quickly to near atmospheric pressure depending upon initial chamber conditions but should drop to < 100 millitorr.

(d) Close roughing valve V2.

(e) Open foreline valve Vi quickly and then open H.V. valve slowly — observing Pirani gauge. Do not allow pressure to rise above 100 millitorr (see note LI). Turn on Penning gauge.

(f) When the Penning gauge comes down to ~ 10^{-5} Torr, you may use the mass spectrometer.

Note I - Foreline valve should not be closed for more than several minutes.

Note II - Pressure should return to original reading very quickly, if it does not, re-close H.V. and get assistance.

Vacuum System is Now in Operating Condition.

3. **Shutting system down to standby condition.**

(a) Turn off Penning gauge and mass spectrometer.

(b) Ensure gas sample valve is closed. Open manifold relief valve V6.

(c) Open the sample pump several seconds to purge the manifold of sample gas. Close manifold relief valve V6. Close sample pump valve. Turn off sample pump. (Rotary #2)

(d) Close H.V. valve. Note that foreline valve is still open and roughing valve is closed.

(e) Any N_2 remaining in the cold trap will gradually evaporate.

The system is now back in the standby condition.

Gas Handling Instructions

1. **Introduction of gas samples for spectra analysis.**

   (a) Connect rubber tube from sample cylinder to gas sample manifold.

   (b) Start sample pump (rotary #2) and open V5.

   (c) Observe vacuum gauge (on manifold) to ensure vacuum is attained. = 28" Hg

   (d) Shut sample pump valve (V5), leaving rotary pump #2 on.

Note: You are now ready to introduce sample gas to system.

2. **Proceed as follows — carefully —**

   (a) Open the main valve on cylinder V7. Under no circumstances is the secondary pressure delivery valve to be adjusted.

   (b) Open control valve V8 (black plastic lever type valve) attached to rubber tube to allow gas into manifold.

   (c) Gauge on manifold should read just above zero, (i.e. just above one atmosphere).

   (d) Put Penning gauge to range 2 if possible.

   (e) Assuming the Penning gauge is on range 2 scale, carefully open gas sample valve to raise the pressure to approximately 2×10^{-5} torr. You are now ready to run a spectrum as described in "Operating Instructions."

   (f) Maintain gas pressure as set in (e) by further opening gas sample valve <u>if required</u>.

   (g) On completion, gently close gas sample valve.

3. **Changing of Gas Samples**

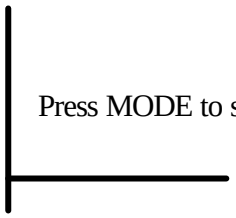
   (a) Ensuring 2(g) above is done — remove rubber tube from gas sample cylinder (this will allow the gas to leave the sample tube).

   (b) Open sample pump valve V5 and run pump for several seconds, this will purge the manifold with air, close sample pump valve V5.

   (c) Connect to the next sample bottle in holder, and connect the hose. Open sample pump valve V5.

   (d) Proceed as above starting at 1(c).

0MG064 Mass Spectrometer Outline



	MODE	STBY	TOTAL	H_2	He	H_2O	N_2+CO	O_2	Ar	CO_2	USER
FUNCT		0	1	2	3	4	5	6	7	8	9

From STBY to TOTAL, gas key, or SCAN turns emission on

press FUNCT to switch from:

WIDTH - Scan width 00.00 to 63.31 (for cycle mode +/- used to enable/disable each gas)
MASS - first mass in 1/32 mass units (i.e. 18.14 = 18 + 14/32 mass units)
CALIB - ionization efficiencies for pressure readings (not used)
RANGE - selected from scan mode (0 to 7 corresponding to 10^{-1} to 10^{-8} mbar full scale)
SPEED - scan speed in scan mode/filter time constant in gas mode (0.5, 1, 2, 5, 10, 20 or 50 sec/amu)
CODE - code number - code parameter