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FABRICATION AND OPERATIONAL DETAILS OF AN ULTRA HIGH VACUUM (UHV) SYSTEM FOR THE SIMULTANEOUS ANALYSIS OF NITROGEN AND NOBLE GASES

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9. a) PURPOSE : To document the procedures to make a UHV extraction system.

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12. ABSTRACT : The fabrication of an UHV extraction system is described in detail. The speciality of this system is its ability to handle nitrogen and all the noble gases simultaneously. This system has been attached to VG 1200 'Noble gas Mass Spectrometer'. The working procedures and the results obtained, are described in detail, using 'Air Standard'.

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Introduction

Static mass spectrometry of noble gases was first developed by Prof. J. H. Reynolds (1956) at University of California, Berkely. This development has enabled, the analysis of noble gases in amounts of the order of 10^{-12} cc STP or less, leading to a number of new discoveries in Earth and Planetary sciences (Reynolds 1960, See Anders 1981, 1987, 1988 for details). In 1981, Frick and Pepin (1981) have extended the static mode of analysis to nitrogen also, making it possible for the first time, isotopic analysis of nitrogen with nanogram amounts. Later, Wright et al. (1983) and Carr et al (1986) [see also Pillinger (1984)] have developed the static analysis mode for carbon dioxide as well. One of the most important advancements due to these sensitive techniques is the ability to analyse the tiny amounts of exotic material (HF-HCl resistant acid residues from primitive meteorites) for C, N, H and noble gas isotopes in order to trace their nucleosynthetic history.

At present, there are only three laboratories in the world, that have the facility for static mass spectrometry of nitrogen. One of them is at Open University, Cambridge and can only analyse nitrogen. The other two are in U.S.A. (Prof. K. Marti's Laboratory, in La Jolla, California and Prof. R. O.

Pepin's laboratory in Minnesota). Both these mass spectrometers can simultaneously analyse nitrogen and noble gases. The author has worked in Prof. Marti's laboratory and is involved in developing the nitrogen analysis system.

A simultaneous study of nitrogen and noble gases helps in better understanding the nitrogen isotopic systamatics and also in deleneating the physico-chemical processes in both terrestrial and solar system objects (Frick and Pepin 1981, Lewis et al 1983, Javoy et al. 1984, 1986). Due to the importance of nitrogen studies, we fabricated a system for our programme in PRL, which is capable of measuring nitrogen and noble gases simultaneously.

The system essentially consists of a mass spectrometer and a gas extraction/purification system, both of which are described below.

2. The Mass Spectrometer (MS):

It is commercially available mass spectrometer VG 1200, made by Vacuum Generators Ltd. U.K. It is equipped with Nier type ion source, with a source magnet to increase ionization efficiency. For ion detection, both a Faraday Cup and an electron multiplier (for small ion currents) are available. The Faraday cup can be retracted from the ion path by an external bellows mounted drive, so that the ion beam hits the multiplier dynode. An oil diffusion pump (for bake out

purpose and also to handle large gas loads) and an ion pump (to keep the MS in UHV all the time) are directly attached to the Mass Spectrometer, through all metal UHV valves, which can be baked upto 400°C. A SAES (SAES is a trade name, which is an abbrevation for Societa Apparecchi Elettrici e Scientifici) getter is also included in the MS volume, through a valve, to keep the hydrogen and hydrocarbon background to a minimum. A bake out oven that goes upto 300°C makes it possible to bake the M.S. to get the vacuum of $<10^{-9}$ torr. An ion gauge, which is also a part of the mass spectrometer, helps in pressure measurement.

3. The Extraction System:

The extraction system has been fabricated in PRL, using commercially available ultra high vauum components. The various components used are as follows:

1. UHV valves of the diaphram type, bakeable to 300°C.

2. A UHV pippette valve bakable to 300°C.

3. An oil diffusion pump and rotary pump for handling large gas loads, during bake-out and degassing.

4. A noble gas ion pump to maintain hydrocarbon free UHV of the order of $<10^{-9}$ torr in isolated system.

5. Ti-Zr and SAES getters to clean the noble gas fractions from traces of hydrocarbons and hydrogen.

6. A Cu/CuO getter to clean N_2 fraction from hydrocarbons and hydrogen.

7. Activated charcoal to adsorb noble gases.

8. Stainless steel mesh (SSM) made from micron sized stainless steel powder, to adsorb nitrogen as well as noble gases.

Even though nitrogen, in its usual form (as N_2 molecule) is inert and behaves like a noble gas under normal conditions of temparature and pressure, in high vacuum, the clean surfaces of certain metals are active to getter nitrogen (Fast 1965). This gettering property is enhanced under conditions of low and high temperatures. Stainless steel, Aluminium, copper-oxide, Mo, W, Ni at room temperature, are inert to N_2, while materials like Ti, Ti-Zr alloy Ti-Pd alloy, SAES getter, some forms of charcoal, hot Ni are some of the materials which scavenge N_2 very fast (Goward 1965). Care has to be taken in selecting the materials to be used in fabricating a system for N_2 processing, with the above problems in mind. When materials that can scavenge N_2 are to be used in the system, a proper isolating mechanism is to be provided, so that when N_2 is being processed, the volumes containing active materials with respect to nitrogen can be valved off. All these requirements are kept in mind, in the

lay-out of the extraction system.

A schematic diagram of the extraction system is shown in Fig. 1. It essentially consists of three parts (excluding pumps and pressure measuring devices).

a) Extraction bottle to heat and extract gases from rock samples.

b) A standard gas mixture (atmospheric air in this case) of known composition, attached to the extraction line through a pipette valve.

c) The main extraction line, wherein gases are purified, separated and transferred to the mass spectrometer for measurement.

The three sub systems are described in some detail below.

The Extraction Bottle

A schematic diagram of the extraction bottle is shown in fig. 2. It is a double vacuum, watercooled bottle. In the inner most vacuum, a Mo crucible is hanged through a seperable quartz tube. The Mo crucible is heated by induction, by sliding a copper spiral (that is connected to a RF generator) over the outer water jacket of the bottle. Only the inner most wall of the bottle is made of quartz and the

rest is made of pyrex glass(This is to minimize the use of expensive quartz tubing) The water flowing in the outer jacket will provide cooling, for the pyrex jacket,from radiative heating from the crucible.The intermediate vacuum layer between the water jacket and the Quartz wall,is intended to serve two purposes.

a) Once the Quartz wall is degassed, it remains clean as it is in vacuum on either side, and there is no possibility of constant loading of gases into Quartz.

b) The diffusion of He through Quartz is minimized as there is no He available to diffuse in, due to Vacuum.

Both these factors lead to lowering of the blank levels to a great extent and enable analysis of small samples.The optically flat view glass, at the top of the bottle will enable viewing the crucible bottom for temperature calibration using optical pyrometer,and also to follow the sample melting by directly viewing.A magnetic ball and the seating are intended to obstruct the path to view glass whenever necessary, to prevent vapor deposits on the view glass. The samples are loaded into the sample tree,and they can be manovoured with small nickel pieces (loaded alongwith samples) by an external magnet.

The Standard Air Reservoir

A precisely known amount of air(few ccSTP) is expanded into a known volume of a reservoir,that is attached to a pipette valve (Fig. 3a). A precisely known amount of an aliquot can be pipetted out from this reservoir to calibrate the sensitivities and mass discrimination of the mass spectrometer,periodically.

I am describing the process of standard preparation in some detail,for the benefit of future users.

a) A Break Seal Ampule (BSA) is made in such a way that the process of sealing it finally does not result in appreciabl volume change. This is achieved by making the sealing end, with a thick capillary pyrex tube with a conical ending (Fig. 3b) By simply putting a softended, bead like pyrex glass mass at the conical end, sealing can be achieved. The volume of the BSA is precisely measured by evacuating and filling the BSA with Hg and weighing the volume of Hg that fills the Ampule. The BSA is then cleaned and evacuated, and through a stop cock, kept under vacuum. It is then taken to a clean and open space.The temperature, relative humidity and atmospheric pressure at that place are recorded and the BSA is filled with air by slowly opening the stop cock, and allowing at least 5 minutes to achieve equilibrium, before closing the stop-cock. Just before sealing only the BSA is detached from the stop cock.

b) the volume of the reservoir bulb is determined, by filling it with distilled water and weighing the water that fills the bulb.

c) The reservoir is attached to the pipette valve. The BSA is attached to the reservoir. The volume changes that resu due to glass blowing, are kept to a minimum.

d) The reservoir and the pipette valve are thoroughly baked before beaking the BSAand expanding the air into reservoir. This reservoir serves as a standard fo Ne, Ar, Kr Xe and N_2 only. For He, due to the diffusion of atmospheric He through pyrex and also due to the very low ^3He, ^4He ratio of 1.4×10^{-6} for air, it is necessary to make a separate reservoir in a metal container and with artifically prepared ^3He, ^4He mixture of around $^3He/^4He = 10^{-4}$. This will be taken up in course of time. The details of the "Air Standard" are as follows:

Volume of BSA : 7.9109 cc

Conditions during Air filling:

Temparature : 35°C

Atmospheric Pressure : 760 torr

Relative Humidity : 20.0%

Vapour pressure of water at 35°C : 42.14 torr

Dry air pressure : 760 -(.2 x 42.14) = 751.57 torr.

7.9109 cc air at 35°C and 751.57 torr $= \frac{751.57 \times 7.9109 \times 273}{760 \times 308}$

$= 6.9342$ cc STP air

Total volume of the reservoir after breaking the ampule $= V_1 + V_2 + V_3$ [see Fig. 3a]

$= 777.57$ cc

Volume of the reservoir after pinching off the BSA $= 762.34$ cc

Amount of air trapped in the reservoir $= \frac{762.34 \times 6.9342}{777.57}$

$= 6.7983$ cc STP air

Volume of Pipette $= 0.1041$ cc

1st pipetted vol. of air $= \frac{0.1041 \times 6.7983}{0.1041 + 762.34}$

$= .9282 \times 10^{-3}$ cc STP air

Depletion factor $= \frac{6.7983 - 0.9282 \times 10^{-3}}{6.7983}$

$= 0.99986$

nth pipetted vol. of air $= (.99986)^{n} \times 0.9282 \times 10^{-3}$ cc STP

Amounts of various gases in the 1st pipetted air are as follo
[the relative volume fractions are taken from Verniani (1966)]
(in units of cc STP).

$N_2 = 7.247 \times 10^{-4}$ $\quad$ $^{28}N_2 = 7.195 \times 10^{-4}$

$Ar = 8.666 \times 10^{-6}$ $\quad$ $^{36}Ar = 2.915 \times 10^{-9}$

$Kr = 1.058 \times 10^{-9}$ $\quad$ $^{84}Kr = 6.030 \times 10^{-10}$

$Xe = 8.074 \times 10^{-11}$ $^{132}Xe = 2.171 \times 10^{-11}$

$Ne = 1.687 \times 10^{-8}$ $^{22}Ne = 1.569 \times 10^{-9}$

The Main Extraction Line

In this part of the extraction line, cleaning and separation of various gases is carried out, before letting the sample into mass spectrometer. The main line consists of Cu/CuO clean up system and a cold finger. Three separable volumes consisting, a) a Ti-Zr getter and charcoal finger b) a SAES getter and charcoal finger c) a stainless stell finger containing SSM are attached to the main line through valves. Additionally, a volume in which a SSM is attached, is provided between the extraction line and the mass spectrometer. The purpose of this volume is to first transfer the gas that is to be let into mass spectrometer, by adsorbing on SSM, and isolating the extraction line, from this volume, before opening the let-in-valve to mass spectrometer.

There are two protocols that are followed: One for handling N_2 sample and the other for noble gases. But before describing these protocols in details, the properties of the SSM need to be discussed.

Stainless Steel Mesh:

The stainless steel mesh (SSM) is a cylinder made from S. S. powder of micron-size. They are originally made by the manufacturers (Nupro Company, Ohio, U.S.A.), intended to be

used as filters. Ithas been found that these meshes have all the adsorption/desorption properties, similar to activated charcoal/zeolite, and in addition, they have an added advantage over either charcoal and zeolite. That is, the bakability of SSM upto >800°C and thereby achieving thorough degassing and obtaining ultra clean conditions is possible, while in the case of charcoal and zeolite, heating beyond 300 to 400°C will result in structural damage which effects the adsorption/desorption properties. Additionally in the case of charcoal, a possibility of CO contamination to the N_2 gas rules out the use of charcoal in the N_2 processing operation.

Fig. 4 provides a cross sectional view of the S.S. finger enclosing SSM. The S.S. finger is made in such a way that the SSM snugly fits into it, so as to provide good thermal contact between the mesh and the S.S. finger, for efficient heating (while degassing and desorption) and cooling (for adsorption). It is necessary to use S.S. only for the outer jacket, to be able to heat to higher temperatures (upto 800°C) and also to avoid differential expansion. The S.S. finger is welded to the covar-end of the glass to metal seal, to fecilitate joining to the glass system. In Table 1, the adsorption/desorption times, for various gases at liquid nitrogen temparature and room temperature respectively are given. These times are determined by directly monitering the signal while the process of adsorption/desorption is going on.

4. Gas Handling Procedure

The gas sample can be either from the standard reservoir, or from the extraction bottle (during the sample analysis). All the valves except V_2 are closed. The gas from either the standard reservoir or the extraction bottle is let into the system by opening V_7 or V_E. By keeping SSM at liquid nitrogen temperature, Ar, Kr, Xe and N_2 are adsorbed into volume [V_2] (The volume behind valve V_2 is defined as [V_2]) and this volume is isolated. Now only the He and Ne will be in the gas phase.

Clean-up of He-Ne fraction

The He-Ne fraction is analysed first. This fraction is exposed to Ti-Zr getter and SAES getter for short times, to minimise processing time in an effort to reduce He diffusion into the system through pyrex glass. Before letting this fraction into mass spectrometer, the Ar and CO_2 (if they are present) are adsorbed onto SSM-4, by keeping it in liquid nitrogen, and left at this temperature throughout the measurement period, so that interference to ^{20}Ne and ^{22}Ne from $^{40}Ar^{2+}$ and CO_2^{2+} are minimized.

After the analysis of He-Ne fraction the line is pumped off. Valves $V_1, V_3, V_4, V_5, V_6, V_7, V_E$ are closed. The gas (Ar, Kr, Xe,

N_2) from $[V_2]$ is expanded into main volume by opening V_2. After allowing 5 minutes for equilibration, V_2 is closed and fraction trapped in volume $[V_2]$ is used for N_2 analysis, while the rest of the gas is used for noble gas analysis. The procedures for processing the N_2 fraction and the Ar, Kr, Xe fractionare as follows:

Clean-up of N_2 fraction

1. Close valves $V_1, V_3, V_4, V_5, V_6, V_7, V_E$. Open V_2 and let the N_2 fraction come in contact with Cu/CuO. Raise the temperature of CuO to 700°C. At the end of 20 minutes, the CuO temperature is reduced to 400°C. In the heating stage, the CuO oxidises any organic contaminants and CO that are present in the sample into CO_2 and H_2O. In the cooling stage, the free oxygen that remains in gas phase is taken back by the Cu/CuO, and also any oxide of N_2 that is formed in the heating stage is reduced back to N_2.

2. After allowing 10 minutes for complete removal of O_2, the condensible products CO_2 and H_2O are trapped on the cold finger at Liquid N_2 temperature, and the non-condensible N_2 and noble gases are adsorbed on SSM-2. Valve V_2 is closed and the condensibles are pumped off by opening V_5. The pressure of the condensibles is noted, from the ion-current of the pump.

If is is felt that the amount of condensibles is large, the Cu/CuO clean up as described above is repeated. In most of the cases, one cycle of Cu/CuO clean up is suffice.

3. N_2 fraction, alongwith the accompanying Ar,Kr,Xe is then transferred to volume [V_4]. There is no need to separate N_2 from Ar, Kr, Xe, as this amount is only a small fraction of the total gas amount. But if it becomes necessary, it is possible to separate N_2 from Ar,Kr,Xe.

4. Before letting N_2 into mass spectrometer the SAES getter in the mass spectrometer volume has to be closed and the ion gauge has to be switched off at least one hour before letting in N_2. Both these measures are very important, to prevent scavening of N_2 gas.

Clean up of Ar,Kr,Xe fraction

1. The noble gas fraction is first cleaned on Ti-Zr getter, by raising its temperature to 800°C and progessively cooling it to 300°C in about 30 minutes time. At temperatures above 700°C, Ti continuously sorbs N_2, O_2, CO_2, CO and decomposes hydrocarbons by taking up

C and leaving H_2 gas. From 400°C and below the H_2 is sorbed also. Zr decomposes H_2O vapours around 300°C and sorbes H_2 and O_2 (Roth 1982).

2. After clean up on Ti-Zr, the gas is transferred to Volume [V_3] and further cleaned on SAES getter to remove remaining traces of contaminants. The particular getter used in our system is AP-10GJ. It contains a non evoparable getter material (ST 101, Zirconinm Aluminium alloy). The gettering action is similar to that of Ti-Zr. The SAES getter is taken upto 750°C and allowed to cool down to 300°C in slow steps over a time span of 20 minutes.

At the end of these two clean up operations, the noble gas fraction is clean enough and is ready for separation into Ar, Kr and Xe fractions. The separation is achieved by differential adsorption on charcoal at different temperatuires, wherein only one gas species from a mixture of gases can be selectively adsorbed. To achieve this, one needs a mechanism by which charcoal can be held at a constant temperature in the range of 25° to -196°C. We have developed a temperature probe in our laboratory for this purpose as described below.

5. Variable temperature Probe:

A miniature variable temperature probe is designed in the laboratory. It is shown in Fig.5.On a copper cylinder a heating wire is wound for electrically heating the copper cylinder. Inside the copper cylinder where the constant temperature is maintained, a liquid that does not solidify at the desirable temperature is placed to act as a medium for sensing the temperature of the volume and to achieve uniformity of temperature. A Fe-constantan thermocouple dipping in the liquid acts as a temperature sensor.The copper cylinder is inserted into a pyrex jacket with a flat bottom, so that there is good contact between the bottom surfaces of the Cu and Pyrex for a good conduction.The wall of the pyrex jacket is insulated from direct contact with Cu, by keeping an asbestos paper. The purpose of this arrangement is to make sure of two aspects,namely when the whole assembly is dipped in liquid nitrogen,the copper cylinder gets cooled only through the bottom face,and when the heater wire heats the copper cyliner,the heat is not transmitted to the glass walls. Required constant temperature is achieved by a balance between heat supplied by electrical heating and the cooling through the bottom surface of copper cylinder.

The control unit is purchased from Powertech Instruments, Bombay.The particular unit that we are using is INDOTHERM-400 temperature controller, which is ideal for applications where precision temperature control is required. Its operation is based on the null balance system.

The thermocouple signal and a precision reference source are compared in a high performance chopper amplifier which produces an output proportional to the deviation from the set point. This output drives a thyristor power control package to maintain precise temperature. The proportional band is made adjustable, so as to permit the matching of heat input to the dynamic characteristics of individual furnace. This prevents overshoot and hunting at set point. A manual reset control eliminates offsets caused by process load variations, without resetting the set point. A centre zero meter is provided to indicate the temperature deviation from the set point. The temperature sensing element is thermocouple and can be selected so as to cover desired range and applications.

6. Ar-Kr-Xe Separation Procedure

Idealy two control units and two probes are needed to complete the process faster. But even with a single unit, the separation quality is not effected, except that it takes little longer time. Also, a liquid which does not solidify down to $-130^{\circ}C$ is necessary to reach $-120^{\circ}C$ (the minimum temperature used in the separation process) for a good separation. Isopentane with melting point of $-159.9^{\circ}C$ (boiling point $27.8^{\circ}C$) will be an ideal liquid for this purpose, but not easily available. Among the liquids easily available and non-toxic, ethyl alcohol with melting point of $-117.3^{\circ}C$, is the best choice we have. But one can only reach

down to $-110^{\circ}C$ or so, in order that the liquid medium does not become highly viscous and give non-uniform temperature. This limits the separation of Kr and Ar. But at the present time we have to be content with the quality of separation that result from using ethyl alcohol as the contact liquid.

The separation procedure, as to the temperature to be used to adsorb a given gasseous species, and the length of time needed to achieve good separation yield are worked out through a trial and error method, in Prof. K. Marti's laboratory at UCSD, La Jolla. I followed the same procedure as described below, and satisfactory separation yields are achieved, keeping in view of the problem with the contact liquid.

The dial setting that correspond to the temperatures of interest are found out a priori, by measuring the temperature with an alcohol thermometer for varying dial settings. The separation operation is carried out in the following sequence.

1. The Ar, Kr, Xe mixture is collected into volume [V_6] and isolated. The probe is filled with ethylalcohol and immersed in a liquid nitrogen dewar. After allowing the alcohol to cool for some time, the dial reading on the control unit is set to 3.5 (corresponding to $-85^{\circ}C$) and the power is turned "on". The probe is then slided

over the charcoal finger in [V_6] and allowed to come into regulation. That probe is regulating is indicated by the temperature deviation meter reading zero, which means that the probe temperature and the set temperature are equal. At this temperature the charcoal adsorbs only Xe, leaving Kr and Ar in the gas phase.

2. After allowing twenty minutes for Xe adsorption, the Kr and Ar in gas phase are adsorbed onto charcoal fingure in [V_3], by keeping Ch-3 in liq. N_2 and opening V_3 and V_6. Ten minutes is enough to adsorb Kr + Ar on Ch-3, at the end of which V_6 and V_3 are closed. Now the probe is removed from Ch-6. Both Ch-3 and Ch-6 are warmed. Separated Xe is now in volume [V_6] and Kr + Ar is in [V_3].

3. The probe is now slid over Ch-3 and the dial setting is changed to 2. 0 (corresponding to $-110^{\circ}C$). After regulation is achieved, fifteen minutes are allowed for Kr to be adsorbed onto Ch-3. Alongwith Kr part of Ar will also get adsorbed on Ch-3, at this temperature.

4. SSM-2 is cooled to Liq. N_2 temperature and V_2 and V_3 are open to adsorb the Ar in the gas phase on to SSM-2. Here the amount of Ar that is adsorbed onto SSM-2 strictly depends on length of time. As liquid N_2

temperature is more favourable for Ar adosorption, as time passes, the Ar partial pressure in gas phase decreases, and to maintain the equilibrium with respect to charcoal at $-110^{\circ}C$, the weakly adsorbed Ar from charcoal desorbs into gas phase and gets transferred to SSM-2. But since $-110^{\circ}C$ is not low enough for Kr to remain as adsorbed phase on charcoal ($-120^{\circ}C$ is required for maintaining Kr preferentially on charcoal), some Kr, invariably comes onto SSM-2. The only way to counter this problem is to strictly follow a fixed length of time for Ar adsorption onto SSM-2, so that reasonably good amount of Ar gets separated from Kr + Ar mixture and then accept the yield of Kr that remains in Kr fraction on Ch-3. A time period of 6 minutes is found to be reasonable. At the end of 6 minutes V_2, V_3 are closed. The probe is now removed from Ch-3, and Ch-3 and SSM-2 are warmed. Now Ar is separated onto SSM-2 and Kr is separated onto Ch-3. The yields are given in table-2.

If $-120^{\circ}C$ regulation had been possible the yields would be as shown in Table - 2. We will procure Isopentane in future, so that $-120^{\circ}C$ regulation becomes possible and better separation can be achieved.

7.Calibration of mass spectrometer

An air standard is processed as per the procedures outlined(Section-4). The gases obtained from the air standard are used to find out the sensitivity and the mass descrimination of the mass spectrometer.These two values are not constant. They keep varying, depending upon the condition of the spectrometer at a given time,and the setting used. These values are periodically determined and the values of the air standard that is last analysed,are used in assessing corrections to the samnple measurements. In Table - 3 are listed the sensitivities and mass descrimination values for the settings mentioned,to give a feeling. In general these values do not change by more than 20% under same operating conditions. The sensitivities mentioned in table - 3 are only for normal operating conditions. An order of magnitude higher sensitivities are easily achievable by using higher emission current for the filament and by using slightly higher voltage for the multiplier dynodes.

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Table - 1: Adsorption/desorption characteristics of Stainless Steel Mesh.

Species	Time (Minutes)	
	For complete adsorption at liq. N_2 Temp.	For complete desorption atroom Temp.
N_2	7.5	.4
Ar	10	1
Kr	11	1.5
Xe	18	4

Table 2: Separation yields of Ar, Kr, Xe in different fractions

Species	Yield (%)		
	In Ar-Fraction	In Kr-Fraction	In Xe-Fraction
a) Using Ethanol as Contact Liquid			
Ar	85	15	< 1
Kr	48	50	2
Xe	< 1	5	95
b) Using Isopentane as Contact Liquid			
Ar	98	2	< 1
Kr	5	95	< 1
Xe	< 1	5	95

Table 3: Sensitivities and Mass discrimination for normal operating conditions*

Species	Detector & Resistor used	Sensitivity (cc STP/Volt)	Mass Descriminatio (%/amu)
^{4}He	M, 10^{8} Ω	-	-
^{22}Ne	M, 10^{8} Ω	7.0×10^{-9}	$1.63 \pm .24$
$^{28}N_2$	F, 10^{10} Ω	9.6×10^{-6}	$2.32 \pm .03$
^{36}Ar	F, 10^{10} Ω	4.5×10^{-6}	$0.90 \pm .05$
^{84}Kr	M, 10^{9} Ω	5.2×10^{-11}	$0.88 \pm .15$
^{130}Xe	M, 10^{9} Ω	6.4×10^{-11}	$0.13 \pm .06$

M = Multiplier, F = Faraday cup.
*Emission current = 100 μA, Electron Energy = 60 eV (for He, Ne) and 80 eV (for the rest).

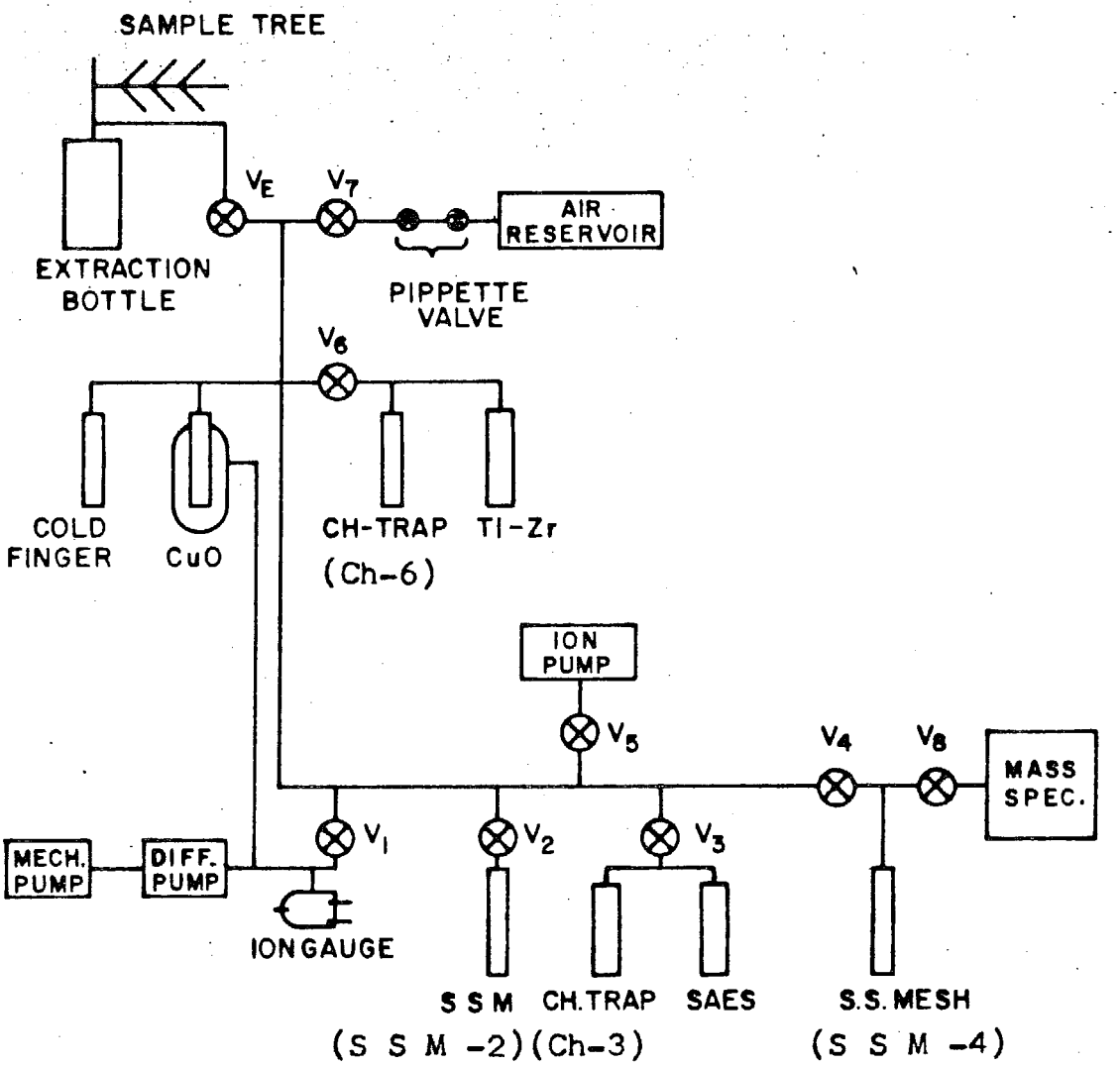

FIG. 1: SCHEMATIC DIAGRAM OF THE EXTRACTION SYSTEM

V_1 - - V_7, V_E ARE U.H.V. VALVES

Ch-n:Charcoal trap-n, SSM-n:S.S.Mesh-n, n=1,2

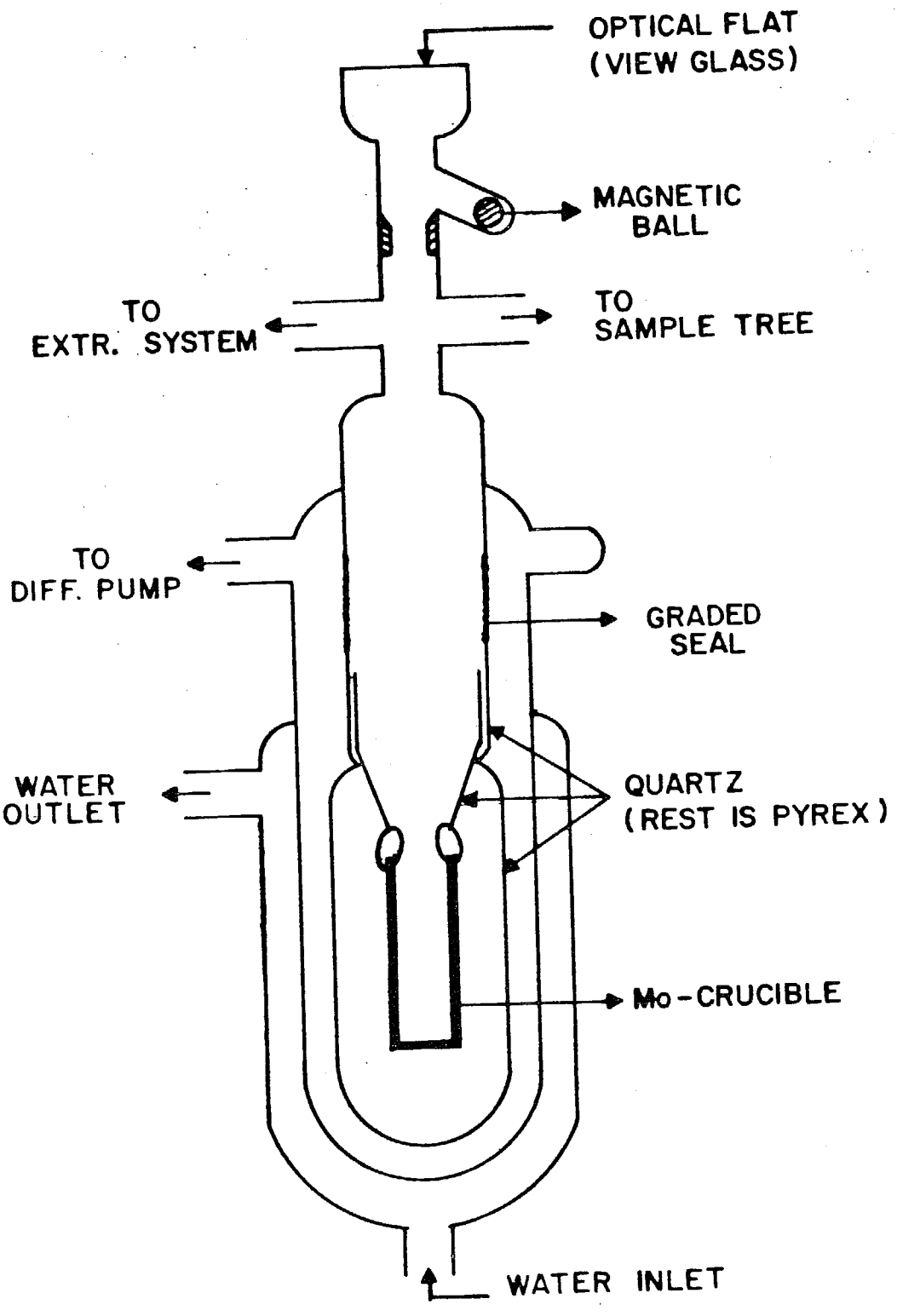

FIG. 2: DOUBLE VACUUM EXTRACTION BOTTLE

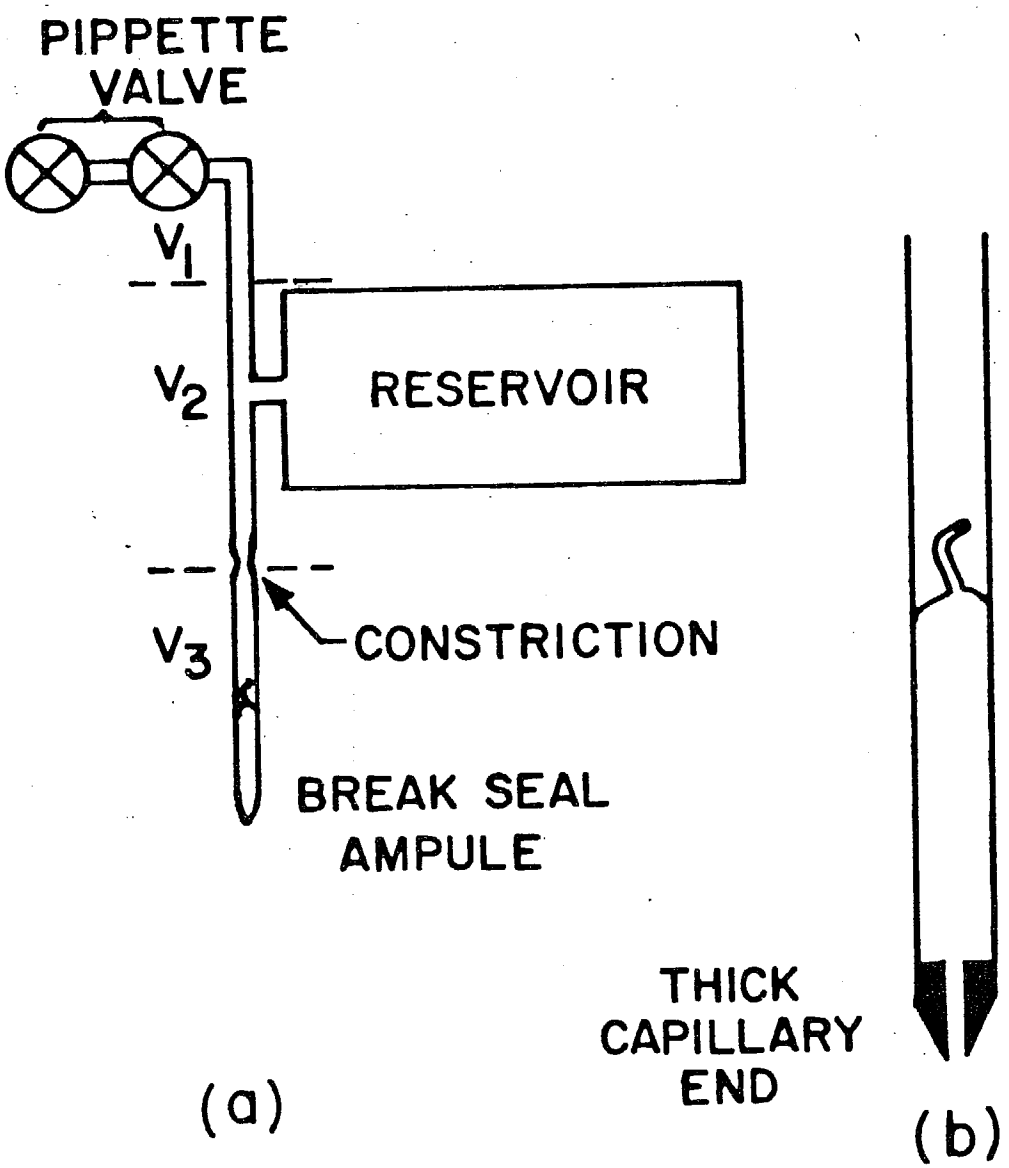

FIG. 3a: THE ARRANGEMENT USED IN PREPARING AIR STANDARD RESERVIOR

3b: THE BREAK SEAL AMPULE CONSTRUCTION IS SHOWN IN DETAIL

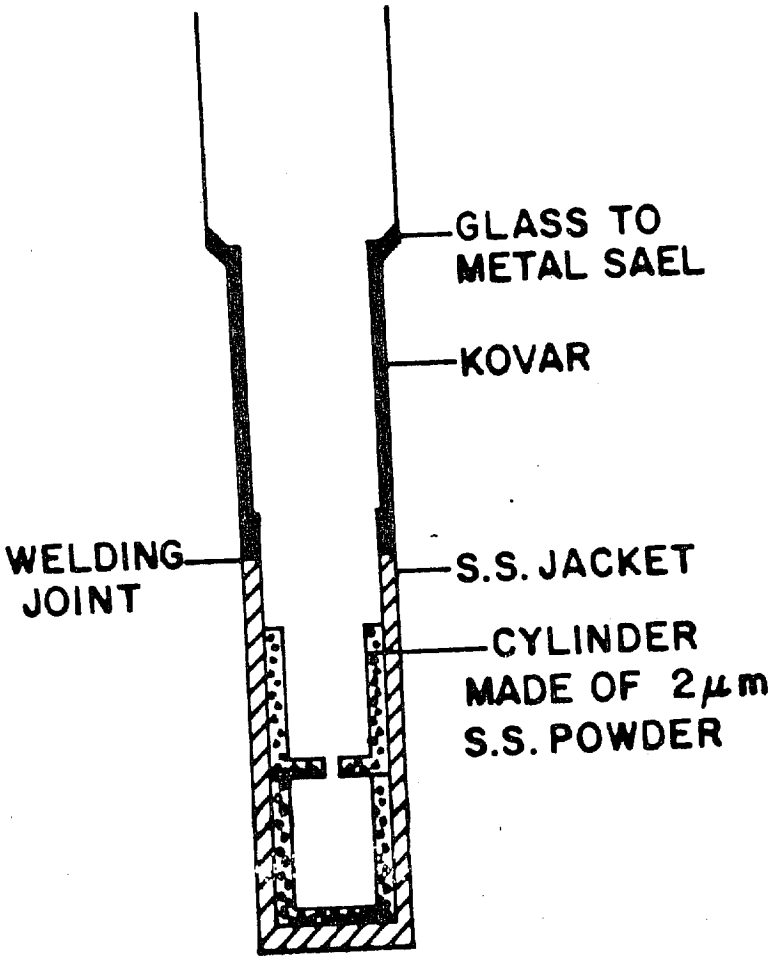

FIG. 4: CROSS - SECTIONAL VIEW OF THE STAINLESS STEEL FINGER HOLDING THE S.S.MESH

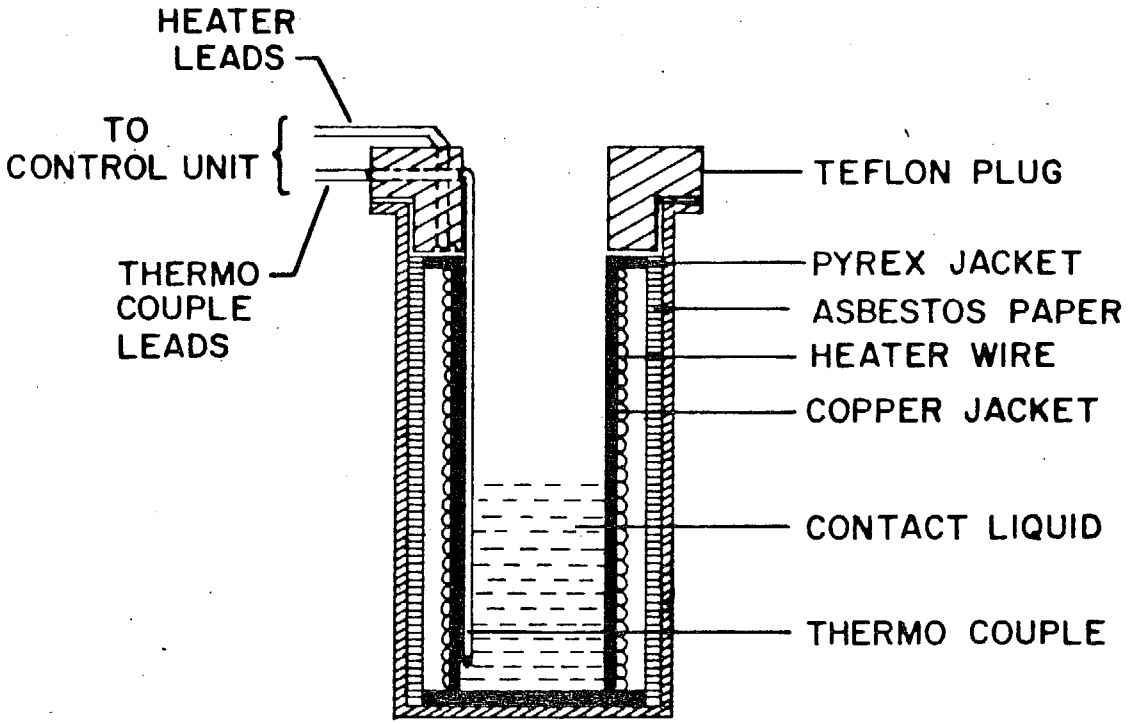

FIG. 5: CONSTANT TEMPARATURE PROBE