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भारत सरकार
GOVERNMENT OF INDIA
परमाणु ऊर्जा आयोग
ATOMIC ENERGY COMMISSION

A HIGH PRECISION MASS SPECTROMETER FOR
HYDROGEN ISOTOPIC ANALYSIS OF WATER SAMPLES

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भाभा परमाणु अनुसंधान केंद्र
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A B S T R A C T

A high precision mass spectrometer with two ion collector assemblies and direct on line reduction facility (with uranium at 700°C) for water samples for hydrogen isotopic analysis has been designed and developed. The ion source particularly gives high sensitivity and at the same time limits the H_3^+ ions to a minimum. A digital ratiometer with a H_3^+ compensator has also been developed. The overall precision obtained on the spectrometer is 0.07 % ($2\sigma_{10}$ value). Typical results on the performance of the spectrometer, which is working since a year and a half are given. Possible methods of extending the ranges of concentration the spectrometer can handle, both on lower and higher sides are discussed. Problems of memory between samples are briefly listed. A multiple inlet system to overcome these problems is suggested. This will also enable faster analysis when samples of highly varying concentrations are to be analyzed. A few probable areas in which the spectrometer will be shortly put to use are given.

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I. INTRODUCTION

Water sample analysis for its hydrogen isotopic ratio is routinely required in plants producing heavy water, in heavy water moderated reactors and in ground water survey. The analysis is useful in controlling the enrichment factor and the quality of D_2O produced, to locate the possible leaky points on pipes carrying D_2O and for inventory of D_2O leakages and tracing the ground water sources.

Several methods⁽¹⁾ such as infrared spectrometry, measurement of density, refractive index or thermal conductivity are used for the isotopic analysis of water samples. In all these, some physical quantity which is a function of the deuterium concentration in water is measured. But each of these methods has its limitations, like large sample requirement, long time for analysis, stringent maintenance of constancy of temperature of the sample, limited concentration range etc. However, the generally accepted and most widely used method for this work in the concentration range 100 to 10,000 ppm is the use of specially designed double collector mass spectrometer. The ability of such spectrometers in handling a large number of samples/day makes them highly suitable in heavy water plants⁽²⁾. The high precision these mass spectrometers offer at natural concentration ranges entitles them their unique position in the R and D laboratories all over the

world. In the concentration range above 95 % D_2O , infrared spectrophotometers are most convenient and are therefore invariably used.

In hydrology, deuterium concentration studies in water samples help monitoring the movements of ground water (3). Quite often such data will be useful as complementary information in environmental and chemical analysis (4). Studies on the biological effects of deuterium concentrations have been covered by Lacroix (5). Mass Spectrometers because of their high precision are the exclusive machines used in all these studies.

Since the natural concentration of D/H in water is very low (150 ppm) it is necessary to simultaneously collect the HD^+ and H_2^+ beams for getting high precision.

In this report design, development and performance details of the spectrometer are given. Initially a prototype was developed and later a batch of 3 mass spectrometers was produced. The first of the production model is working round the clock since 18 months. In all about 2500 samples in varying concentration have been analyzed.

II. MASS SPECTROMETER

The spectrometer has been designed keeping a number of applications in view. Thus the machine can normally handle samples in the concentration range from 25 ppm

to 1,000 ppm. However, this range can be extended quite easily upto 1 % . It has been designed for routine analysis and is run round-the-clock and has protection against vacuum, power and water failures.

(a) Analyzer : This is made of 304 stainless steel and uses a 6 cm radius 60° angle of deflection symmetric magnetic field and normal incident ion beam geometry (fig.1). Thus the H_2^+ ion beam describes 6 cm radius and the mass 3 beam describes 7.35 cm radius. The ion acceleration voltage is about 1860 and the magnetic field strength (obtained by permanent magnet supplied by Eclipse Major 862) is 1600 Gauss in a gap of 12 mm over an area of 40 cm^2 . The field is found to be quite uniform and the fringing field is assumed to be uniformly spreading over a length equal to 1 pole gap width for calculating the refocusing positions of the ion beams.

The ion source (fig.2) is of the electron bombardment type and is essentially on the lines of Nier's design. However, it is adapted such that the H_3^+ formation, which is due to the ion molecule reaction, $H_2^+ + H_2 \rightarrow H_3^+ + H$ is minimum. This is made possible by using high source magnetic field and better ion focusing with electrostatic fields in the y and z directions. An important parameter in the reduction of H_3^+ ion formation is to keep the H_2^+ ion residence time in the ionizing region to a minimum.

This has been achieved by using high repeller fields and keeping the electron beam only one mm away from the first exit slit of the ionization chamber. The operating electron current is also kept specially high (3mA) to reduce the partial pressure requirement of H₂ to give the same signal. The gas tightness of the ionization chamber and the gas leading tube inside the source ensure high sensitivity of the source and low sample requirement. It was found that a source sensitivity 3×10^{-2} A/torr at 3 mA emission current is easily and routinely obtained. H₃⁺ formation is as low as 2×10^{-14} A when the H₂⁺ ion current is 10^{-9} A, which compares favourably with results of Roth⁽⁶⁾ and Bridger⁽⁷⁾. The final source exit slit size is 1mm x 5mm. The source magnet has field strength of 250 gauss over an area 6 sq.cm.

A collector slit plate (slit size 5mm x 7mm), a secondary electron suppressor (SES) slit plate (7mm x 9mm) and a deep (15mm) trapezoidal shaped copper cup form a collector assembly. Two such assemblies (fig.3) one each for mass 2 and mass 3 ion beam, are mounted such that the ion beams are perpendicular to their slit plates. They are well shielded from each other (both electrostatically and magnetically) for stable and linear operation of the mass 3 amplifier. A voltage of -135 is given to SES plates. The input resistors (10^9 ohms for mass 2 and 5×10^{11} ohms for mass 3) with their respective

condensers 1000 pf and 2pf and the electrometer tubes are housed in two independent chambers and are constantly pumped by a rotary pump.

(b) Vacuum system: The vacuum system (fig.4) consists of an IBFTORR 120 oil (polyphenyl ether) diffusion pump, (120 l/sec) chilled water cooled chevron baffle, liquid nitrogen trap and a high conductance bellows sealed valve with a bypass facility. The diffusion pump is backed by a two stage 50 l/min rotary pump. A molecular sieve trap is used above the rotary pump to minimize the oil contamination in the analyzer. The pressure obtained in the analyser is 3×10^{-8} torr. Gaskets used for the joints are made of PTFE. Liquid nitrogen in the trap lasts for 12 to 16 hours. Liquid nitrogen filling is done automatically using a thermistor sensor and relay circuit.

A high H_2^+ background is normally encountered in all vacuum systems in general due to poor compression ratio (ratio of pressure on the fore vacuum side to pressure on the high vacuum side) of the diffusion pump for H_2 . This can however be improved by increasing the power input to the heater of the diffusion pump. An increase of about 20 % from a normal 325 W was found necessary.

Fig. 5 shows the improvement in the performance. It is clear that the H_2^+ background peak height (at a pressure $\sim 5 \times 10^{-7}$ torr) is reduced from 200 mV to 40 mV

indicating a drastic reduction in H_2 back diffusion. What is even more striking is the fact that the H_2^+ background stability has improved from a noise of 20 mV peak to peak to 0.5 mV peak to peak. This improvement is phenomenal.

III. ON LINE INLET SYSTEM:

This comprises of two symmetrical inlet systems one of them being for the standard and the other for the sample. The system (see fig. 6) has been designed to take liquid samples directly and is especially useful for δ determinations. It is made of glass and is kept at $150^\circ C$ by external heater. An attachment for analyzing gas samples has also been provided. The necessary vacuum is obtained by a separate (50 l/min) rotary pump. V_1 and V_2 are stop cocks and V_3 is a stainless steel diaphragm type valve using PTFE gaskets. The volume of the reservoir is designed such that about 0.5 μl of sample develops a pressure of about 1 torr. The capillary bore size is chosen such that its conductance is about 10^{-5} litres/sec (sample flow is 5×10^{14} molecules per sec.). This ensures that the peak height does not fall by more than 3 % in about 10 min. time.

About 0.5 μl of the sample is injected into the system, through a silicone rubber septum (as shown in figure 6). It instantly vaporizes and reaches the Uranium reduction furnace, where it is reduced to H_2 and HD and

is then let into the ionizing region.

The design of the reduction furnace, (which is shown in Fig.7) is determined by several important factors. Among the several methods that are in use for reduction, the Uranium reduction method is chosen because of its high efficiency (small quantity of uranium is required), quick reduction of sample, low vapour pressure and low cross contamination of samples.

The furnace is 10 cm long with a heating zone 8 cm long. About one gm. clean, dry, grease-free uranium in the form of thin strips, or foil is packed in the centre of quartz tube (bore 5 mm) but not extending beyond a length of 6 cm. The quartz tube diameter is kept at a minimum to reduce the memory effects. The uranium packing should not extend to the ends of the furnace to avoid formation of uranium hydride which is known to contribute much to memory by way of isotopic exchange. The furnace is kept at about 700°C. The flow conditions are so adjusted that the reduction of water is complete in a single passage.

The quantity of uranium is sufficient to last for about 4 months with continuous operation of the machine using the furnace for 8 hours/day.

IV: ELECTRONICS

This consists of the following units

(i) Ion accelerating voltage: A 2.5 KV supply (model HV 216, ECIL make) with a max. load of 1mA and a stability of better than 0.01 %.

(ii) Emission current regulator: This supplies current (max-10A) to ion source filament and regulates the emission current with a stability of better than 0.01 %. The max. emission current that can be regulated is 10 mA. The circuit is a modified version of the one given by Ron Chapman⁽⁸⁾.

(iii) D.C. Amplifiers: The spectrometer has two independent d.c. amplifiers for mass 3 and mass 2 ion current amplification. Low grid leakage Raytheon CK 5889 electrometer pentodes are used in the preamplifier stages (fig.8). The time constants of the two amplifiers are made identical to 1 sec. The maximum output voltage obtainable on these amplifiers is 10V. Long term stabilities of mass 3 and mass 2 outputs are ± 0.5 mV and ± 0.1 mV respectively. Details of these circuits are given in a report by N.V. Rao and C.K. Nazare⁽⁹⁾.

(iv) High voltage scanning supply: This is required only to check the peak shapes and their overlapping and is not used during the analysis. It generates a standard saw-tooth which is superposed on the high voltage. The maximum span of the scan is 250 V with three scan times of 1 min. 5 min. and 10 min.

(v) H_3^+ compensator: The mass 3 output is due to HD^+ and H_3^+ ion currents. While the former is of interest for the

analysis of the sample, the latter, formed because of the ion molecule reaction mentioned before is an unwanted signal. The H_3^+ signal needs therefore to be subtracted from the total M_3 output, before the true HD/ H_2 ratio is computed. Due to its formation in a secondary process the

$$\begin{array}{l} H_3^+ \text{ output} \quad \propto P_2^2 \\ \quad \quad \quad \propto (i_2^+)^2 = K(i_2)^2 \end{array} \quad \left. \vphantom{\begin{array}{l} H_3^+ \text{ output} \\ \propto (i_2^+)^2 = K(i_2)^2 \end{array}} \right\} \begin{array}{l} P_2 \text{ being the partial} \\ \text{pressure of Hydrogen} \end{array}$$

However, normally when analysis is confined to narrow concentration range, a signal $\propto i_2^2$ is subtracted from the mass 3 signal before a ratio of HD/ H_2^+ is determined (fig.9). While the i_2^+ signal is displayed on a 4 digit panel meter, the compensated signal for computing the ratio is fed into a ratiometer unit. The details of the H_3^+ compensator and the ratiometer are given by Handu⁽¹⁰⁾.

(vi) Digital ratiometer: This makes use of BCD type A/D converters and computes and displays the compensated Mass 3/ Mass 2 ratio in 5 digits. The ratio computation is done after the voltage to frequency conversion using highly stable low off-set v/f converters. The ratio calculation time is variable. The linearity of the system is excellent with a stability of 1 in 10^5 i.e. a variation of ± 1 in the last of 5 digits.

Facility to digitally integrate the signals also exists. The system can thus work in Ratio-Integrate and Ratio mode. The integration time is about 10 seconds.

(vii) Other electronics : Power supplies for source electrode potentials and secondary electron suppressors are well stabilized (better than 0.01 %). Other electronic units include those for measurement of fore and high vacuum and protection circuits.

V. PERFORMANCE :

Peak shape and overlap : High precision in ratio measurement demands flat top peak shapes for the mass 2 (M2) and mass 3 (M3) peaks and a collector alignment to give considerable overlap in the region of their flatness. These ensure better precision due to partial cancellation of noise due to fluctuations in the H.T., in electron current or the background. The ion beam width at the source is 1 mm and that at the collector including all aberrations should not be more than 2.5 mm. The collector slit width is 5 mm. Fig. 10 shows M2 and M3 peaks. From the M2 peak shape it is clear that the beam width at the collector is 2.4 mm. The peaks are flat and the overlap of flatness is about 30 V.

Source sensitivity : The sensitivity for hydrogen is 3×10^{-2} A/torr when referred to the analyzer pressure. With a 500 μ A trap current this gives an absolute source sensitivity for hydrogen equal to 60/torr referred to the analyzer pressure. Pressure in the source is estimated to be about 20 times higher. Therefore the source sensitivity is 3/torr when referred to the pressure in the source. The constancy

of source sensitivity can be evaluated from table 1 which shows the figure over a 3 months period. Std. deviation is 10 %. which is well within the accuracy of gauge used for pressure measurement.

Overall efficiency: The overall efficiency of the analyzer is found to be 10^{-4} , that is, 1 ion is collected for every 10^4 gas molecules of the sample. Sample requirement is about 0.5 μ l liquid or 0.3 Std. cc. of gas.

Background stability : The H_2^+ background instability is less than 0.01 % of the peak height. The overall peak instability is also found to be less than 0.01 % of the peak height. This could be achieved by stable (back diffusion free) pumping of the sample with the increased heater power to the diffusion pump as was mentioned earlier.

Detection sensitivity: H_2^+ background output is about 25mV. It was found that a flow of hydrogen at the rate of 10^{14} molecules/sec will give a signal of 1V. Hence the detection sensitivity (assuming 5 mV above the background) $\approx 5 \times 10^{11}$ molecules/sec. Thus, assuming that the signal should be maintained for 10 sec., the quantity of sample required is 5×10^{12} molecules, which is 5×10^{-7} std.cc. The amount of sample requirement is rather high for detection in the context of mass spectrometry. This is due to the large background of hydrogen being desorbed from the internal surfaces.

It is possible to improve this figure by a factor of 100 by using uhv techniques alone and by a further factor of 100 to 10,000 if an electron multiplier is used.

Precision of measurement: For this purpose only one inlet system was chosen and precision of measurement determined independently for three concentration ranges (nominal concentrations are 150, 200 and 300 ppm). After initial stabilization of the whole machine including the inlet system, each sample is injected and a set of 3 observations for the ratio is taken and their average determined. In a similar way 10 average ratios are determined with 10 injections of the same sample. The whole process takes about an hour. The standard deviation σ of these 10 measurements and also $2\sigma_{10}$ are calculated in the standard manner.

$$2 \sigma_{10} = \sigma \times \frac{2.26}{\sqrt{10}}$$

2.26 on the right hand side arises due to the student's distribution assumed and indicates a 95% confidence level.

The same experiment is repeated about 20 times and the average $2\sigma_{10}$ (precision) was found out. Tables 2 to 4 give these figures for the 3 nominal concentration ranges mentioned and it will be seen that the overall precision is found to be about 0.06% .

Accuracy of measurement: This very much depends on the standards used for calibrating the spectrometer and their closeness in concentration to the sample whose concentration

measurement has to be made. In addition it depends much on the operator's skill. In order to determine the accuracy of measurement, four standard water samples with 156.43, 201.9, 240 and 296 ppm HD/H₂ (designated A,B,C and D respectively) were chosen.

All the four standards were analyzed in sequence, For the purpose of accuracy, standards A and C were taken as calibration references and the remaining two standards treated as samples for analysis. The results obtained are shown in columns 2 and 4 of table 5. Standards B and D are taken as reference for calibrating the spectrometer and the remaining two are treated as samples for analysis. The results calculated are entered in columns 1 and 3. The same method is repeated for 16 sets and their averages calculated. It is seen from the table that the deviation of the average value from the true value (accuracy) is found to be about 0.1 %.

VI. SAMPLE ANALYSIS: For very accurate analysis of samples, it is necessary to calibrate the spectrometer with standard samples at least once every day. The calibration is imperative because of some minor drifts in the source operating voltages and inlet surface conditions. However the frequency of calibration can be reduced if the accuracy requirement is not better than $\pm 0.5\%$.

To calibrate the machine, two standards one in the natural and the other around 250 ppm D/H range are taken. The HD/H₂ ratios obtained on the machine for these two standards plotted against their true values which thus serves as calibrating curve. Samples are then analysed and their true values are read from the calibration curve. In an actual analysis about 0.5 µl of sample is taken in a clean prerinsed syringe and injected into the inlet system which is kept at constant temperature and continuously pumped. Just before introduction of the sample pumping is stopped and the injected sample is let into the spectrometer through the reduction furnace. The whole process takes about 10 to 15 seconds and it takes another 1½ to 2 minutes for getting stable peaks. H₃⁺ compensator is adjusted such that with the normal flow of sample, the ratio of mass 3/mass 2 remains more or less constant to better than 1 in 10⁴ as read by the ratiometer. The system is flushed 2 or 3 times with the sample to give stabilized ratio values. After this the actual ratio readings are taken thrice and their mean found. The same procedure of stabilizing the system with 2 or 3 flushings with every sample is followed. Analysis for one injection with 2 flushings and a set of three readings takes about 5 minutes.

VII. DISCUSSION:

The spectrometer is designed to handle samples in

the concentration range between 25 to 1000 ppm (with a precision of better than 0.1 %)) as it is this range which is mostly required for nuclear and hydrological applications.

For some special applications it may be required to increase the range of the spectrometer to about 5 %. This is possible if the operating range of the d.c. amplifier is increased by an order of magnitude. But then in the high output voltage range the circuit has to be modified to maintain the measurement accuracy. The problem can also be solved by switching different (lower value) resistors to the input circuit of the M_3 amplifier. High input impedance, ($> 10^{15}$ ohms) very low leakage thermal time delay switches of the type Victoreen V X 10 can be used. However, some time should be allowed for the amplifier to stabilize after switching.

To improve the range of concentration the spectrometer can handle on the lower side without further losing any precision, the ideal method will be to use an electron multiplier for the Mass 3 ion beam with a nominal stable gain of 100 and a suitable combination of input resistors. The use of multiplier incidentally reduces the requirement of sample by at least an order of magnitude. This would mean that the partial pressure of hydrogen in the source will be $\frac{1}{10}$ th that when multiplier is not used. This helps in reducing the H_3^+ formation to such a low factor as to become

negligible. The use of multiplier also reduces the memory between the samples as the sample quantity injected is itself very small.

If samples, whose D/H concentrations are widely ranging are fed successively to the spectrometer for analysis, memory or cross contamination will be observed. This is due to isotopic exchange between the sample being analyzed and that desorbed from the surfaces of the inlet system. The cross contamination can be reduced by (i) injecting small sample (ion source sensitivity should be high), (ii) reducing the surface area with which the sample comes into contact, (iii) increasing the temperature of the same, (iv) reducing the analysis time and (v) using thinner uranium strips which reduce the thermal diffusion time. Memory between the samples is eliminated by flushing the inlet system a few times with the sample being analyzed. This method has two disadvantages viz - increase in analysis time and reducing the life of the uranium strips because of the idle flushings of the furnace.

Both these problems can be solved by using a multiple inlet system (say about 5 inlets). In such a system 5 identical and independent inlet systems are connected in parallel to the mass spectrometer ion source. The first system, into which all samples are let in, is used only to know the range of concentration of the sample. Among the other systems each one is earmarked for handling a specific concentration range of samples. In this way

memory effect will be reduced and stabilization will be faster. The system thus helps in increasing the number of samples (widely varying in concentration) the machine can handle. Such a system will be especially useful when the ranges of concentrations of samples are totally unknown. eg. in detecting leakages in D_2O systems.

VIII. NEW APPLICATIONS

Although originally intended for routine hydrogen isotopic analysis, new applications were also kept in view while designing the mass spectrometer. By suitable minor modification of the collector geometry, mass 3 and mass 4 ion beams can be collected simultaneously for $^3He/^4He$ measurements, for which a number of applications exist⁽¹¹⁾ Extremely high sensitivity requirement for this application is already met in the source design and with further use of electron multiplier, detection limit can go down to 10^{-12} std. cc. The double collector design gives high precision of measurement even for weak $^3He^+$ signals.

Mass spectrometric analysis (with single collector system) of formaldehyde samples by laser excitation and dissociation for deuterium analysis was reported by Jack Marling⁽¹²⁾. But the accuracy was limited to 1%. However a double collector spectrometer identical to the one discussed, is proposed to be used for similar work in our laboratory. This will give improved accuracy and enable improving the parameters for deuterium enrichment.

In biochemical work, where deuterium labelling is used, this mass spectrometer because of its high accuracy in the natural concentration range, will be helpful to detect small changes in concentrations if the sample can be converted to its hydrogen isotopes.

The spectrometer being highly sensitive, can be useful to detect and estimate the total hydrogen evolved in thermal desorption studies. In this application it acts as a hydrogen monitor.

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TABLE : 1

MASS SPECTROMETER MODEL 660.21 SR# NO. 1

Source sensitivity during 14.9.78 and 29.12.78

S.No.	Date	Sensitivity in $10^{-2}A/torr$
1.	14.9.78	2.6
2.	15.9.78	3
3.	16.9.78	3
4.	18.9.78	2.7
5.	19.9.78	2.5
6.	20.9.78	3
7.	13.10.78	3
8.	16.10.78	3.2
9.	16.11.78	2.5
10.	17.11.78	3
11.	27.11.78	3
12.	28.11.78	2.6
13.	29.11.78	2.7
14.	1.12.78	3
15.	5.12.78	3.4
16.	6.12.78	3.4
17.	12.12.78	3
18.	13.12.78	2.9
19.	14.12.78	2.8
20.	18.12.78	3.2
21.	20.12.78	3.4
22.	26.12.78	3.2
23.	29.12.78	3.5

Average source sensitivity = $2.98 \times 10^{-2}A/torr.$

Std. deviation = 0.29×10^{-2}

Std. deviation = 10.%.

NOTE: Filament assembly and uranium strips were not changed during this period.

TABLE 2

PRECISION OF MEASUREMENTSAMPLE : NATURAL WATER

For each set, 10 measurements of the ratio are taken and their standard deviation calculated. The time taken for these 10 measurements is about 1 hour.

S.No.	date	Std.Deviation ($2\sigma_{10/150}$) %
		σ_{10}
1.	8.5.78	0.12
2.	9.5.78	0.057
3.	10.5.78	0.15
4.	11.5.78	0.15
5.	11.5.78	0.088
6.	12.5.78	0.18
7.	12.5.78	0.057
8.	15.5.78	0.066
9.	16.5.78	0.1
10.	16.5.78	0.09
11.	10.7.78	0.4
12.	12.7.78	0.16
13.	14.7.78	0.19
14.	14.7.78	0.09
15.	15.7.78	0.1
16.	15.7.78	0.06
17.	17.7.78	0.06
18.	24.7.78	0.37
19.	24.7.78	0.25
20.	25.7.78	0.094
21.	25.7.78	0.18
22.	25.7.78	0.057
23.	26.7.78	0.08
24.	26.7.78	0.12
25.	27.7.78	0.06
26.	27.7.78	0.12
27.	27.7.78	0.03
28.	28.7.78	0.05
		0.01
		0.02
		0.06
		0.07
		0.07
		0.037
		0.030
		0.026
		0.031
		0.043
		0.037
		0.17
		0.06
		0.08
		0.04
		0.04
		0.02
		0.02
		0.16
		0.11
		0.04
		0.08
		0.025
		0.04
		0.05
		0.03
		0.05
		0.01
		0.02
		0.048
		Average
		0.0548

TABLE 3
PRECISION OF MEASUREMENT
SAMPLE S 200/8/14-19

Nominal concentration: 200 ppm.

S.No.	Date	σ_{10} Std. Deviation	$(2\sigma_{10}/200) \cdot /.$
1.	14.8.78	0.05	0.02
2.	14.8.78	0.20	0.08
3.	14.8.78	0.04	0.02
4.	16.8.78	0.08	0.03
5.	16.8.78	0.05	0.02
6.	16.8.78	0.1	0.04
7.	16.8.78	0.1	0.04
8.	16.8.78	0.06	0.03
9.	17.8.78	0.04	0.015
10.	17.8.78	0.15	0.056
11.	17.8.78	0.10	0.04
12.	17.8.78	0.05	0.02
13.	19.8.78	0.15	0.05
14.	19.8.78	0.05	0.02
Average			0.034

TABLE 4

PRECISION OF MEASUREMENT

SAMPLE S 300/8/1/9

Nominal concentration = 300 ppm.

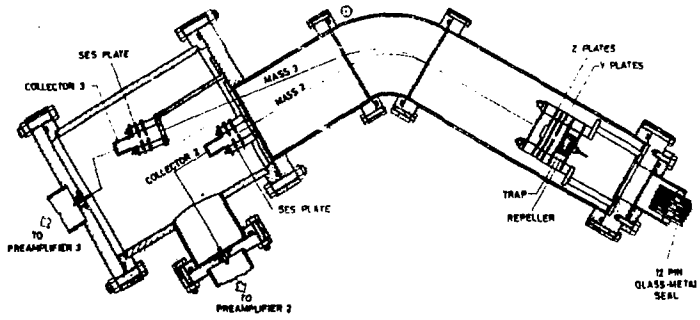
S.No.	Date	Std. Deviation σ_{10}	$(2\sigma_{10}/300)$ %
1.	1.8.78	0.29	0.07
2.	1.8.78	0.21	0.05
3.	1.8.78	0.06	0.01
4.	2.8.78	0.36	0.08
5.	2.8.78	0.23	0.05
6.	2.8.78	0.18	0.04
7.	2.8.78	0.1	0.03
8.	3.8.78	0.54	0.12
9.	3.8.78	0.10	0.03
10.	3.8.78	0.21	0.05
11.	3.8.78	0.21	0.05
12.	3.8.78	0.1	0.03
13.	4.8.78	0.18	0.04
14.	4.8.78	0.25	0.06
15.	4.8.78	0.21	0.05
16.	7.8.78	0.27	0.06
17.	7.8.78	0.27	0.06
18.	7.8.78	0.14	0.03
19.	7.8.78	0.10	0.02
20.	7.8.78	0.14	0.03
21.	8.8.78	0.22	0.05
22.	8.8.78	0.15	0.03
23.	9.8.78	0.23	0.05
24.	9.8.78	0.21	0.05

Average = 0.046

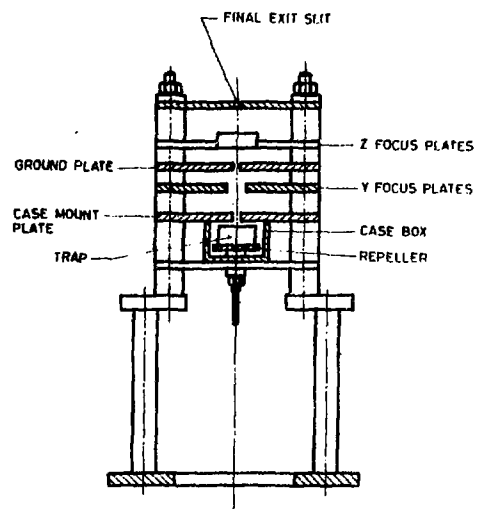
TABLE 5

ACCURACY OF ANALYSIS

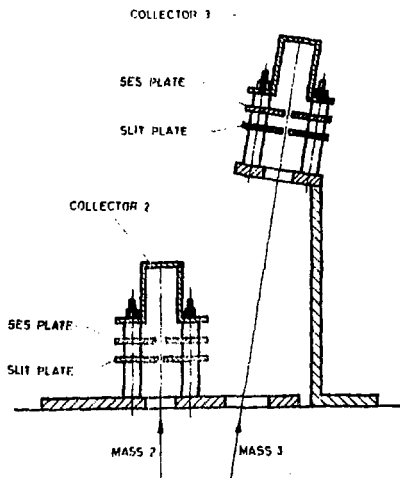
Conc. of Std. used in ppm.	A	B	C	D
	156.43	201.9	240.0	296.0
Experimentally determined values of the concentration of above stds. during 26.9.78 to 7.10.78				
	A	B	C	D
	157.55	201.49	240.24	296.33
	156.46	201.81	240.14	295.78
	156.43	201.77	240.23	295.6
	156.3	202.07	239.9	296.07
	156.96	202.07	239.89	295.99
	156.85	201.53	240.31	295.76
	156.34	201.71	240.42	295.23
	156.89	201.40	240.51	295.45
	156.07	202.03	240.03	295.07
	156.61	201.74	240.14	295.18
	156.58	201.82	240.01	296.07
	156.44	201.76	240.24	295.6
	156.44	201.83	240.02	295.12
	156.09	202.09	239.93	295.89
	156.24	201.87	240.02	295.52
	156.47	201.63	240.45	295.27
Average	156.54	201.79	240.16	295.66
Deviation from Std. value	0.08 %	0.06 %	0.06 %	0.11 %
Std. Deviation σ	0.37	0.21	0.2	0.37



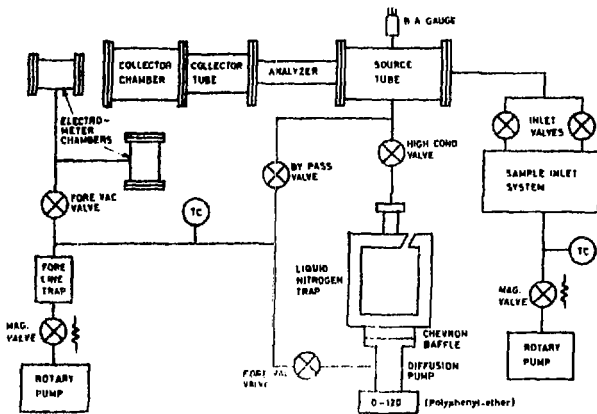
ANALYZER
 FIG. 1



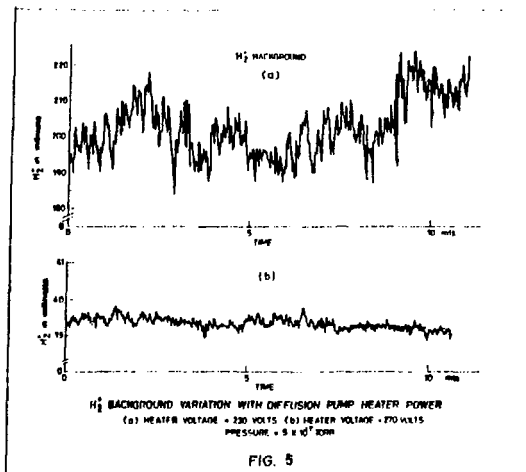
ION SOURCE
 FIG. 2



COLLECTOR ASSEMBLY
FIG. 3



VACUUM SYSTEM
FIG. 4



SAMPLE INLET SYSTEM

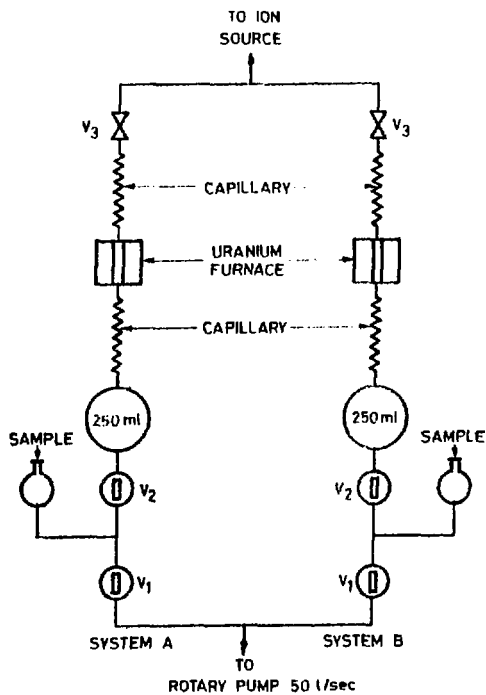
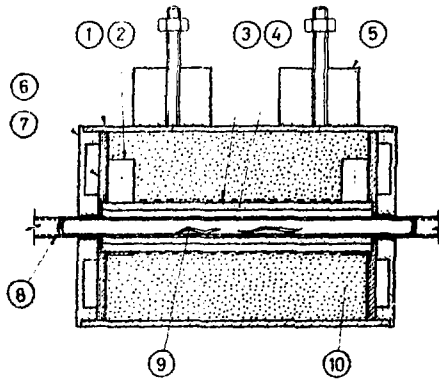


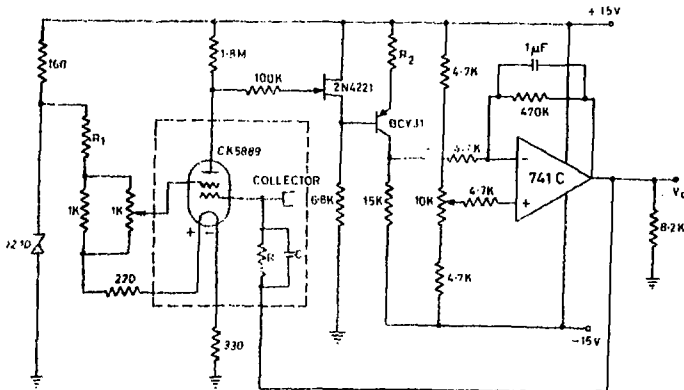
FIG. 6



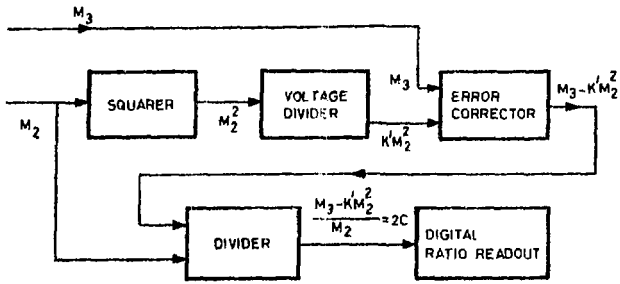
- | | |
|---------------------|--------------------|
| 1 S S CHAMBER | 6 S S COVER |
| 2 S S CLAMP | 7 SYNDANYO PLATE |
| 3 KANFHOR HEATER | 8 QUARTZ TUBE |
| 4 CERAMIC TUBE | 9 URANIUM STRIPS |
| 5 SYNDANYO SUPPORTS | 10 MAGNESIA POWDER |

URANIUM FURNACE

FIG. 7



D C AMPLIFIER
FIG. 8



H_3^+ COMPENSATOR
FIG. 9

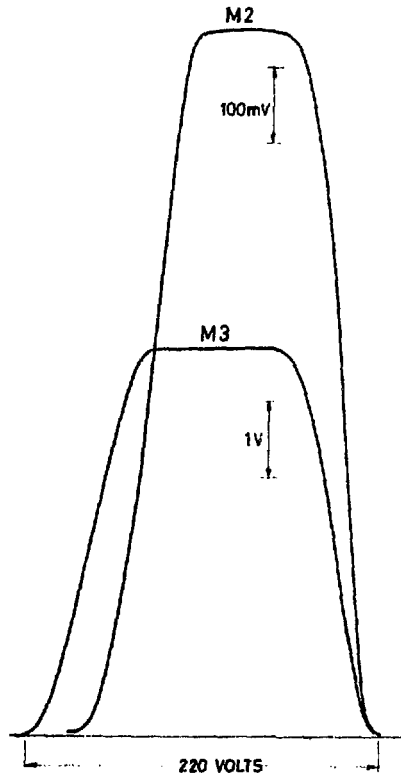


FIG. 10 M2 & M3 PEAKS

