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ADVANCED MASS SPECTROMETERS FOR HYDROGEN ISOTOPE ANALYSES

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ABSTRACT

Two advanced prototype mass spectrometers for the accurate analysis of mixtures of the hydrogen isotopes were evaluated by Du Pont personnel at the Savannah River Laboratory. One, a VG-Isotopes GAZAB, was developed for the Mass Spectrometer Technical Group, a group representing ten DOE contractors. The other, a MAT 250 HDT, was developed independently by Varian MAT (now Finnigan MAT) after extensive discussions with Du Pont personnel.

The GAZAB is a large double-focusing instrument with a resolution of 2,000 at mass 4 and an abundance sensitivity of $>100,000$ for the HT-D₂ doublet. The MAT 250 HDT is a smaller, simpler, stigmatic focusing instrument with exceptionally high ion intensities ($>1 \times 10^{-9}$ A at 600 and about 1×10^{-10} A at 1,300 resolution) for high signal-to-noise ratios.

Both instruments are computer controlled. Once a scan is started, peak switching, scanning, mass discrimination control, data collection and data reduction are done without operator intervention. Utility routines control hysteresis effects and instrument calibration.

A containment facility, with dual inlet systems and a standard distribution system, permits testing with tritium mixtures. Helium flow standards and tritium activity meters provide independent verification of the mass spectrometer calibrations. A recovery system prevents the release of tritium to the environment.

The performance of the mass spectrometers was essentially equal under simulated process control conditions. Precision and accuracy for the D/T ratio was $<0.5\%$ (rel 2σ limits). Performance factors were: sample equilibration <300 ppm; linearity within $\pm 0.3\%$; and gas interference $<0.1\%$. Mass discrimination was controlled reliably by the computers. Therefore, single focusing instruments are suitable for routine control laboratory operations. Double-focusing instruments are only needed for special applications.

SRL/SRP personnel worked closely with both manufacturers in this program. Both have incorporated resulting improvements in their commercial instruments. Both now market competitive single-focusing mass spectrometers.

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ADVANCED MASS SPECTROMETERS FOR HYDROGEN ISOTOPE ANALYSIS

INTRODUCTION

This report is a summary of the results of a joint Savannah River Laboratory (SRL) - Savannah River Plant (SRP) "Hydrogen Isotope Mass Spectrometer Evaluation Program." The program was undertaken to:

- Evaluate two prototype hydrogen isotope mass spectrometers
- Obtain sufficient data to permit SRP personnel to specify the mass spectrometers to be purchased under Schedule 44 Budget Project 81-SR-010 to replace obsolete instruments in Buildings 232-H and 234-H.

ANALYTICAL PROBLEM

Accurate mass spectrometric analyses of mixtures of the three hydrogen isotopes (D, H, and T) are difficult to make because the mixtures contain six molecular species and ^3He (from the decay of T). The heaviest species has three times the mass of the lightest, and mass discrimination is particularly severe.

In a mixture that contains two species with the same mass number, small mass differences and large concentration differences complicate the analysis. The mass difference between HD and ^3He is one part in 520, while that between HT and D_2 at mass 4 is only one part in 980. The concentration ratio of the two species is often more than 1000:1. The scatter of the more-abundant species interferes with the measurement of the less-abundant species. To separate the D_2 -HT doublet reliably, the resolution of the mass spectrometer should be at least one part in 1300, and the abundance sensitivity should be at least 10,000.

Monatomic and polyatomic ions formed in the ion source (Table 1) further complicate the mass spectral analyses. The T^+ and ^3He differ by only one part in 155,000 and probably cannot be separated with any practical analytical mass spectrometers. Fortunately, T^+ ion production is a small constant fraction of the T_2 , DT, and HT concentrations, and the error in correcting the ^3He mass peak is trivial. Trimer formation is a function of the pressure in the ion source and is usually very small.

TABLE 1

Important Ions in the Mass 2 to 6 Range*

<u>Species</u>	<u>M/e</u>	<u>Specimen Analysis (Atom %)</u>	<u>Resolution Required</u>
D ⁺	2.0140	-	1,220
H ₂ ⁺	2.01565	0.004	2.5
³ He ⁺	3.01603	1.118	155,000
T ⁺	3.01605	-	520
HD ⁺	3.021825	1.036	1,830
H ₃ ⁺	3.023475	-	3.5
⁴ He ⁺	4.00260	-	160
HT ⁺	4.023875	-	980
D ₂ ⁺	4.028	86.039	4.5
DT ⁺	5.03005	11.259	5.5
T ₂ ⁺	6.032	0.382	610
D ₃ ⁺	6.0420		

* Isotope masses from **Handbook of Chemistry and Physics** (56th ed.). Robert C. West, ed. CRC Press, Cleveland, OH p. B-253 (1975).

<u>Isotope</u>	<u>Mass, amu</u>	<u>Isotope</u>	<u>Mass, amu</u>
H	1.007825	³ He	3.01603
D	2.0140	⁴ He	4.00260
T	3.01605		

Hydrogen isotopic mixtures can be analyzed with low (<200), medium (~600), or high (>1300) resolution mass spectrometers. When either low- or medium-resolution mass spectrometers are used, the sample must be at equilibrium. The relative amounts of D₂ and HT at mass 4 are calculated using the equilibrium constants published by Jones.¹ When high-resolution instruments are used, all of the species of interest are measured directly.

Helium-3 content is measured directly with medium- and high-resolution instruments but must be measured by some other method, such as absorption of the hydrogen fraction of the sample on uranium or titanium, with low-resolution instruments. This technique is relatively imprecise as shown in Figure 1. The error in the ³He determination causes a corresponding error in the HD value. The HD error is propagated directly to the HT value via the equilibrium calculation.

The specimen analysis in Table 1 is an example of a particularly difficult type of mixture to analyze accurately. About 0.5% of the total T is present as HT. With low resolution methods, gross errors can occur in the ³He determination. These errors are propagated to the HD measurements; then to the HT value via the equilibrium calculation. The resulting errors in the HT value make it impossible to determine either total T or the D/T ratio accurately.

MASS SPECTROMETER DEVELOPMENT PROGRAM

In the past, hydrogen isotopic analyses usually were made with commercially available low- or medium-resolution mass spectrometers. Equilibrium among the isotopic species was assumed for mixtures containing tritium, but was not usually verified. ³He was either estimated from the mass spectrum or determined separately by an auxiliary technique. The limit of accuracy was about ±2 to ±4% and was adequate for many programs.

The available high-resolution mass spectrometers were expensive and produced low ion intensity; they were difficult and time consuming to operate and, judging by the work of Schott and Beau,² were no more accurate than the less expensive, low-resolution instruments.

By the 1970's, all of the hydrogen isotope mass spectrometers in the DOE complex had become obsolete. Modern replacements were sought. There were none that could be used for accurate analyses of mixtures of the hydrogen isotopes. The best of the available commercial instruments were designed for organic analyses and could not be used for quantitative hydrogen isotopic analyses without extensive modifications. It became evident that new instruments would have to be developed.

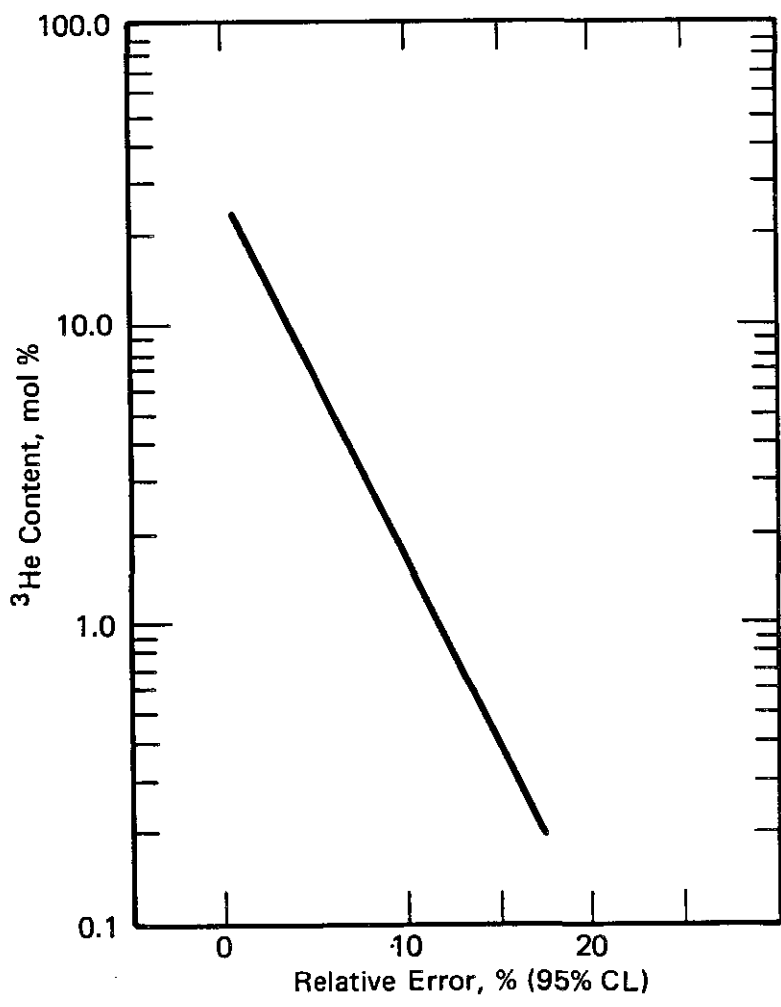


FIGURE 1. Determination of ^3He by Titanium-Sublimation Pumping

At SRL, Chastagner,³ determined that construction of a practical high-resolution (>1300) was within the state-of-the-art of mass spectrometry. Ferguson and Chastagner⁴ demonstrated the concepts that had been developed. A group led by Chastagner drew up tentative specifications for new hydrogen isotope mass spectrometers and contacted mass spectrometer manufacturers. None were willing to develop an instrument for such a limited market.

MASS SPECTROMETER TECHNICAL GROUP

The Mass Spectrometer Technical Group (MSTG) was formed to develop a hydrogen isotope mass spectrometer for the DOE complex. The group represented, and was funded by ten DOE contractors (Appendix I). Organization of the group followed program proposals to ERDA/ALO in 1975 by Alec Willis, SNLL, Clarence Sandy, SNLA, and approval by Vincent Vespe, Director, Weapons Development Division, ERDA/ALO. Sandia National Laboratory, Albuquerque, NM, was named as the coordinating agency, and James Guthrie, SNLA was selected to coordinate the group.

MSTG DEVELOPMENT PROGRAM

The MSTG drew up final specifications (Appendix II) and contracted with VG-Isotopes, the only responsive bidder, for the development and construction of a prototype hydrogen isotope mass spectrometer. The goal of the program was to develop a reliable, affordable mass spectrometer capable of accurately analyzing: mixtures of the hydrogen and helium isotopes regardless of the equilibrium state of the hydrogen isotopes; and mixtures of air components and various fixed gases. The instrument was to be; easy to operate, electrically and mechanically modular for easy maintenance, and adaptable to computer control and data handling.

The MSTG program consisted of four phases:

- paper design
- component and systems testing on a leased prototype instrument called the GAZAB
- refurbishing and purchase of the prototype instrument
- testing the prototype with tritium mixtures at a DOE laboratory.

The MSTG selected SRL/SRP to receive and test the prototype.

INDEPENDENT HYDROGEN ISOTOPE MASS SPECTROMETER DEVELOPMENT

In the late 1970's, Varian MAT (now Finnigan MAT) developed a new mass analyzer design for geological and thermal ionization mass spectrometers. The analyzer appeared to be suitable for a hydrogen isotope mass spectrometer. The Varian MAT technical staff in Bremen was thought to have the technology required to design and build a satisfactory ion source. Extensive discussions between SRL/SRP personnel and Varian MAT personnel in the United States and Germany convinced MAT management that there was a market for hydrogen isotope mass spectrometers and that MAT probably could build a competitive instrument. MAT personnel then designed and built a prototype called the MAT 250 HDT. SRP purchased the prototype and added it to the hydrogen isotope mass spectrometer test program.

The MSTG GAZAB MASS SPECTROMETER

Summary

The MSTG prototype, GAZAB, is a large reversed-geometry double-focusing mass spectrometer. It features an ion source that was modified especially for hydrogen isotope analyses, high resolution and abundance sensitivity, and high transmission. The instrument is controlled by an HP desktop computer throughout the analytical cycle. Data is collected and reduced to final form by the computer.

The GAZAB is very versatile and can be used for a variety of analytical problems. It can be operated at a resolution of 1300 for routine analyses of mixtures of the hydrogen and helium isotopes, regardless of their state of equilibrium. It can also be operated at a resolution of 2000 for sophisticated analyses where all of the molecular species must be separated.

Instrument Description

The hydrogen isotope mass spectrometer prototype (GAZAB) that was developed for the MSTG is shown in Figure 2. It is a gas version of the VS-Isotopes ZERO ALPHA AND BETA (ZAB) mass spectrometer. The ZAB was designed primarily for organic applications that require both high sensitivity and high resolution. It is a reversed geometry double-focusing mass spectrometer with a 30 cm radius magnetic sector followed by a 38 cm radius electric sector (Figure 3). The analyzer is designed to make all the first and second order aberration coefficients equal to zero. Thus the instrument is capable of retaining high resolution and sensitivity, with a gas source of good linearity and high repeller voltage, which would inevitably produce a strong energy aberration in a

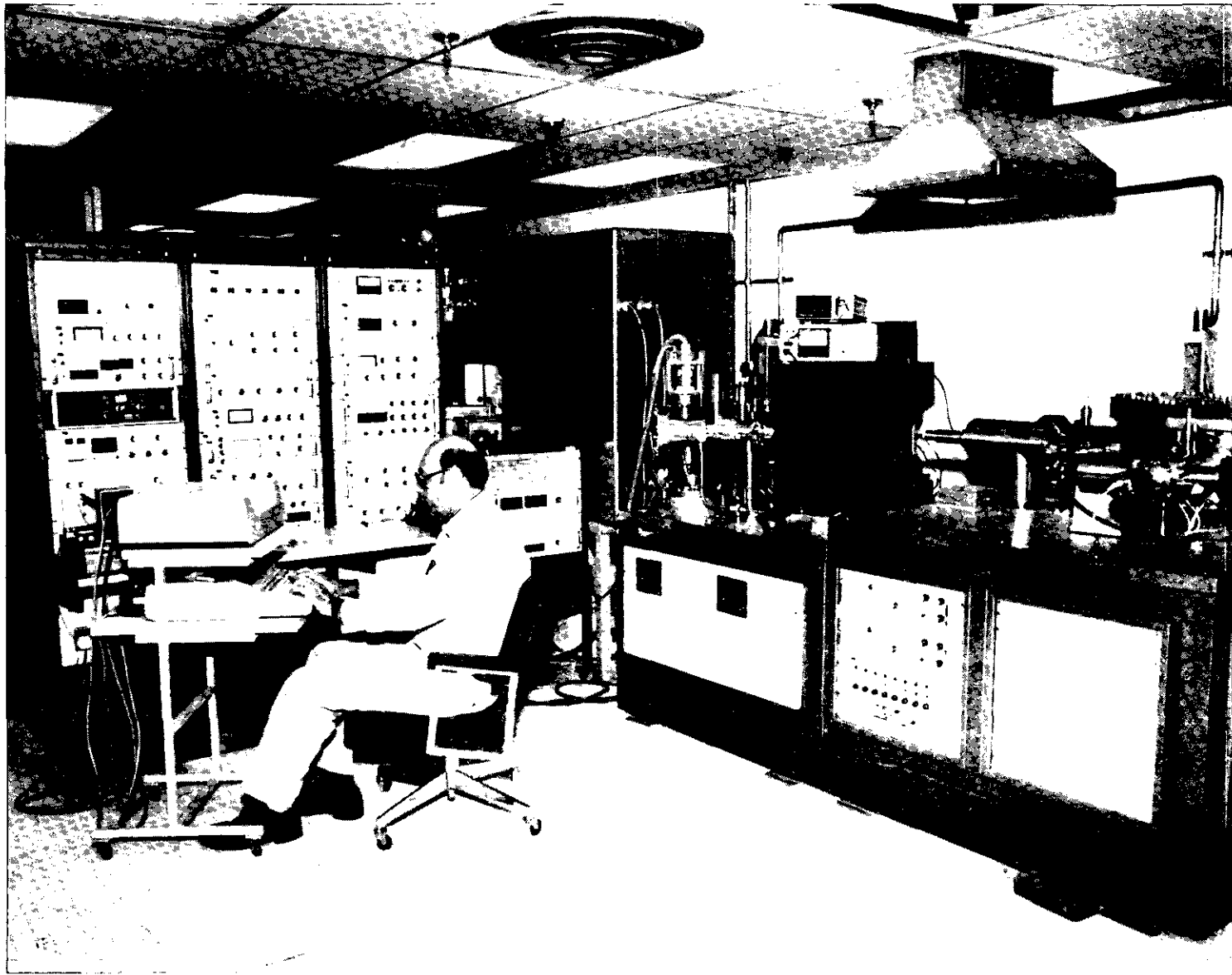


FIGURE 2. GAZAB Mass Spectrometer

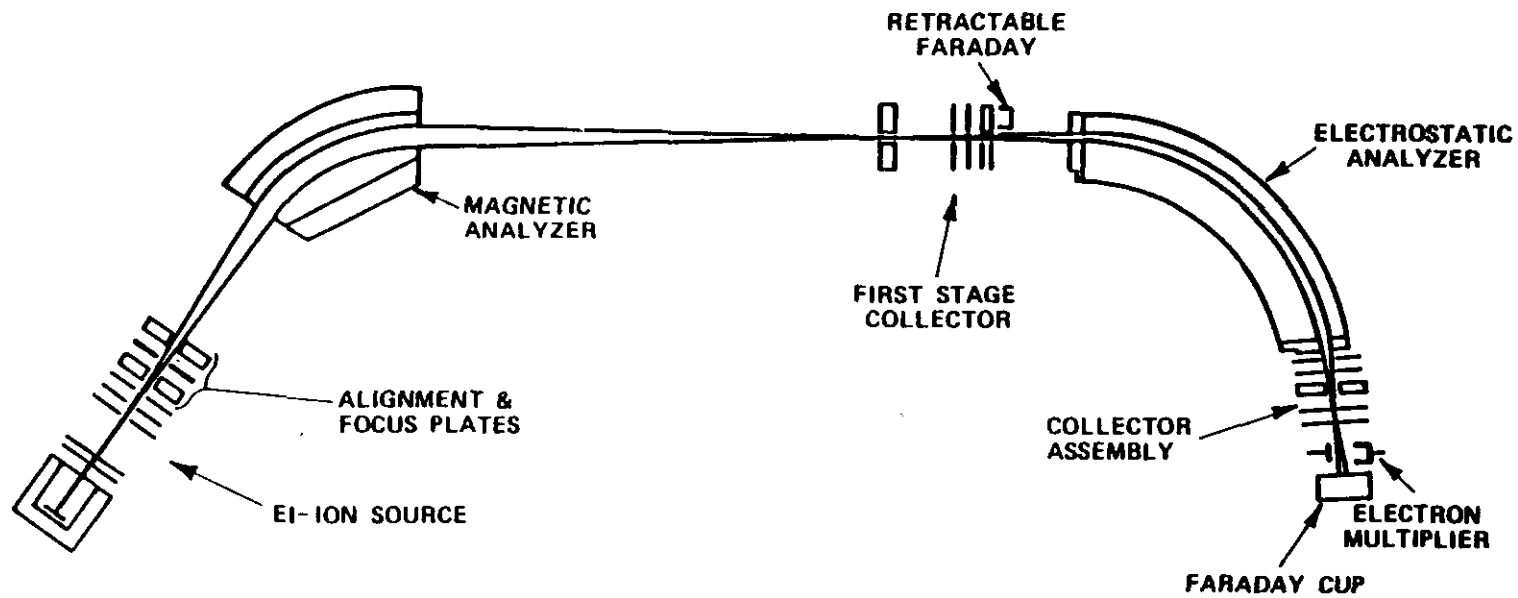


FIGURE 3. GAZAB Ion Optical System

single focusing mass spectrometer. In addition, image curvature correction is incorporated which allows higher sensitivity to be achieved at a given resolution than would otherwise be the case. Also, by fitting slits which are variable in both the dispersion and magnetic field directions, optimum sensitivity can be established at a given resolution.

Deflector plates are incorporated after the alpha slit and before the intermediate slit so that the ion beam may be steered along a shorter or longer path through the magnet as desired. These steering voltages are fully programmable and are used to preserve resolution and peak shape as the mass numbers change. This feature is especially important in a hydrogen isotope mass spectrometer. Mass discrimination caused by magnetic fields in the ion source causes ions of different masses to diverge as they exit the source, and only one mass is in focus at a time.

The ion source is a modified Nier-type. An electron beam passes through a box-shaped ion chamber close to a narrow exit slit. Behind the electron beam is an ion repeller which creates a field of few electron volts per millimeter between the ionizing region and the ion exit slit. The ions also encounter an extraction field penetrating into the ionization chamber from the next electrode which is a pair of half plates with mean potential several hundreds of volts closer in potential to earth, and are steered by the differential voltage on these half plates through an adjustable source slit at earth potential. Further steering then occurs in both the y and z directions before the beam emerges from its alpha slit, which in the ZAB is contained in a separate "stack" of electrodes but is still in principle, a part of the ion source.

All the voltages applied to the source can be fully programmed. This feature gives the source great flexibility when run in its automatic mode. If necessary, each mass can have its own set of source parameters for best sensitivity and resolution, and lowest mass discrimination. However, the source is usually run with the ion repeller and focus voltages fixed during the peak jumping sequence. Only the voltages which deflect the beam in the y-direction are varied with mass.

The GAZAB is equipped with both Faraday cage and Daly scintillation-type ion collectors. Only the Faraday cage collector is used for accurate hydrogen isotopic analyses. The Daly collector is used for measuring trace level impurities during survey analyses.

Modern solid-state technology is used in all of the electronic units. All of the source, magnet, beam control, and electric sector power supplies are designed for computer control. Manual control is also provided.

The analyzer system is evacuated by four polyphenyl ether-charged diffusion pumps. The pumps are equipped with integral water-cooled baffles. A liquid nitrogen trap is provided in the source pumping line. Isolation valves permit the source to be vented without venting either the analyzer or the pumping system. Dual stage mechanical pumps are provided for rough pumping and to back the diffusion pumps. Ion and Pirani vacuum gages monitor pressures and shut down the vacuum system and electronic supplies when excessive pressure is detected.

The entire mass spectrometer operates under control of a Hewlett-Packard HP-9831 with software provided by VG-Isotopes. This software was limited by the capacity of the HP-9831 computer to the hydrogen isotopes and two impurities. A complete version that runs on an HP-9845 computer is available.

Because the GAZAB was an experimental instrument, many of the components were prototypes and were the source of some operating problems. Initially, VG-Isotopes personnel replaced or repaired these units as problems developed. In June 1982, they overhauled all of the electronic units and brought the GAZAB up to current production standards at VG-Isotopes expense. From then until the end of the program, the GAZAB operated flawlessly.

INSTALLATION AND ACCEPTANCE

The GAZAB mass spectrometer was shipped by air from England to New York and by van from there to SRL. It arrived in good condition on February 2, 1981. Graham Wells and Barry Wright, VG-Isotopes Engineers, arrived from England at the same time. They immediately started to assemble the instrument, and had an ion beam through it by February 9. Only minor difficulties were encountered: source arcing; a source cable breakdown; an ESA supply problem; and failure of the vibrating reed electrometer (VRE). All problems except that with the VRE were fixed. The VRE could not be repaired on plant and was sent offsite for repair and recalibration. The VG-Isotopes solid-state amplifier in the GAZAB was used instead of the VRE in all the subsequent tests.

Acceptance testing began February 19 and was concluded successfully February 28. As shown in the Appendices, all requirements of Specification SLA-XMS-8-79 as amended in December 1980 were met without exception. Martin Elliott, the VG-Isotopes director in charge of the GAZAB development work, arrived February 23. He assisted Wright in the final phases of acceptance testing and remained another week to teach SRP personnel how to operate the instrument.

CHARACTERIZATION TESTS

Cold testing to characterize the GAZAB was started immediately after its acceptance. The modified ion source performed reliably and data were within specification limits. The sensitivity at a resolution of 2,000 was about 110 mV/ μ for hydrogen (with a 1×10^{11} phm resistor). When the resolution was set to 1,300 and the instrument was set for maximum transmission at each mass, the sensitivity was doubled (Appendix II). The mass discrimination under those conditions was about 10%, the resolution was adequate to separate the HT-D₂ doublet at mass 4, and the abundance sensitivity was about 27,000. The sensitivity was very stable with time (Table 2). The sensitivity measurements were made nine days apart.

TABLE 2

Variation of GAZAB Sensitivity with Time

Molecular Species	Sensitivity, mV/ μ		Diff., %
	3/24/81	4/2/81	
H ₂	199.57	199.17	-0.20
³ He	56.17	56.02	-0.27
HD	209.44	208.62	-0.39
D ₂	216.28	215.74	-0.25

Several changes in operating conditions improved the performance of the GAZAB mass spectrometer. The changes included revised ion source parameters and operation with liquid nitrogen in the ion source trap. The precision was excellent and was reproducible (Table 3). In these tests, Mound Laboratory mixture No. 1, and an SRP mixture with the same ³He content ($4 \pm 0.25\%$ ³He, the balance being D₂) were analyzed. Analyses of Mound Laboratory mixture No. 1 yielded ³He values slightly below the theoretical value and H₂ values slightly above the theoretical value. These results were consistent with previous data for Mound mixture No. 1. The ³He value for the SRP mixture agreed with the theoretical value within the error of the latter. This test confirmed the accuracy of the GAZAB for ³He at the 4M % level. These data, together with the results of previous tests with Mound mixture No. 1 suggest that the theoretical values for that mixture are not correct.

The precision of calibrating the GAZAB mass spectrometer was evaluated for H₂, ³He, HD, and D₂. Calibration measurements were repeated at a rate of about two per minute for ten minutes. The ³He calibration precision (Table 4) is typical of these tests.

TABLE 3

Tests of the Precision and Accuracy of the GAZAB Mass Spectrometer

SRP Mixture			Mound Lab Mixture No. 1			Mound Lab Mixture No. 1		
Avg. ^3He , mole %*	Avg. ^3He - Theor. ^3He † Precision, %**	Bias, Δ %	Avg. ^3He , mole %*	Avg. ^3He - Theor. ^3He †† Precision, %**	Bias, Δ %	Avg. H_2 , mole %*	Avg. H_2 - Theor. H_2 ††† Precision, %**	Bias, Δ %
4.030	±0.38	0	3.985	±0.35	-0.40	6.052	±0.23	+ 0.73
4.042	±0.41	+ 0.30	3.986	±0.33	-0.37	6.045	±0.24	+ 0.62
4.039	±0.30	+ 0.22	3.982	±0.39	-0.47	6.044	±0.20	+ 0.60
4.038	±0.34	+ 0.20	3.980	±0.38	-0.52	6.050	±0.17	+ 0.70
			4.006	±0.38	-0.12	6.049	±0.20	+ 0.68
Average		+ 0.18			-0.33			+ 0.67

* Average of 10 runs.

** $4.2s$, where s = estimator of relative standard deviation (%) for a small population; $4.2s$ corresponds to >99.7% confidence limits.

† Theoretical ^3He content = 4.03 mole %.

†† Theoretical ^3He content = 4.001 mole %.

††† Theoretical H_2 content = 6.008 mole %.

TABLE 4

Precision of GAZAB Calibration with ^3He

<u>Inlet Pressure, microns</u>	<u>Sensitivity, mV/micron</u>
93	64.00
92	64.04
92	64.08
91	64.10
90	64.10
90	64.06
89	64.07
88	64.11
88	64.07
87	64.05
87	64.02
86	64.08
86	64.14
84	64.18
84	64.19
83	64.12
82	64.12
82	64.19
Average	64.096 \pm0.054
Relative Standard Deviation	\pm0.08%

In the course of this work, Hess found that the linearity of the GAZAB mass spectrometer for hydrogen isotopes varied with the energy of the ionizing electrons. At 68 V energy, the D₂ response was linear within ±10%. As the energy was either increased or decreased, the response became non-linear. The amount of the non-linearity depended on the difference between the electron energy and 68 V. D₂ sensitivity was directly proportional to pressure at electron energies below 68 V and inversely proportional to pressure at electron energies above 68 V (Figure 4). H₂ sensitivity underwent a similar transition at about 72 V, but was linear within ±0.1% at electron energies below 70 V. ³He linearity was not affected by electron energy.

THE FINNIGAN (FORMERLY VARIAN) MAT 250 HDT MASS SPECTROMETER

Summary

The Finnigan MAT 250 HDT is a modern single-focusing mass spectrometer. It features a high brightness ion source and an off-axis extended geometry magnetic analyzer with very high transmission. The ion source is linear over ten times the pressure range and emission current that we consider conventional. The instrument is controlled with an HP desktop computer.

The MAT 250 HDT is well adapted to routine analytical work. At a resolution of 600 it is used for the analysis of equilibrium mixtures of the hydrogen isotopes and helium. It is also used routinely at a resolution of 1300 to analyze mixtures in which the hydrogen isotopes may not be in equilibrium.

Instrument Description

The Finnigan MAT 250 HDT prototype is a refined single-focusing mass spectrometer (Figure 5) that was developed from the recently-designed MAT 250 and 260 mass spectrometers. The analyzer (Figure 6) is a stigmatic-focusing 23 cm radius magnetic sector. The stigmatic design doubles the dispersion to that of a 46 cm radius normal entry analyzer. The design also provides "Z" focusing which increases the transmission.

The ion source is similar to that of the standard MAT 250 source except that it has been shortened to reduce mass discrimination and to improve transmission. An electron beam passes through a box-shaped ion chamber close to an ion exit slit. Ions are drawn out of the chamber by an electric field created by a "drawout" plate located close to the ion exit slit. An ion lens system collimates the ions into a beam. The ion beam is focused in the "Y" and "Z" directions for maximum transmission through the source

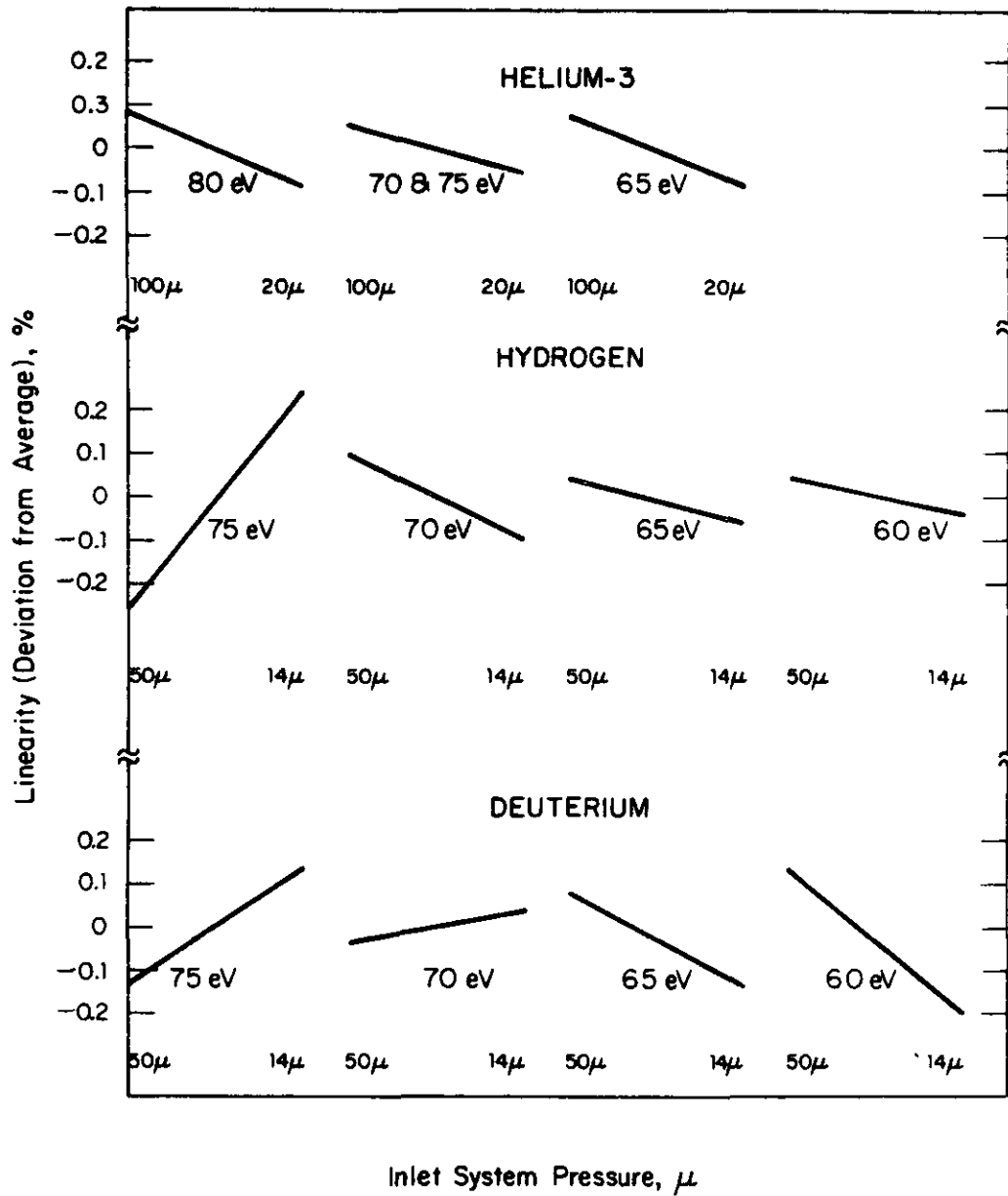


FIGURE 4. GAZAB Linearity as a Function of Ionizing Electron Energy

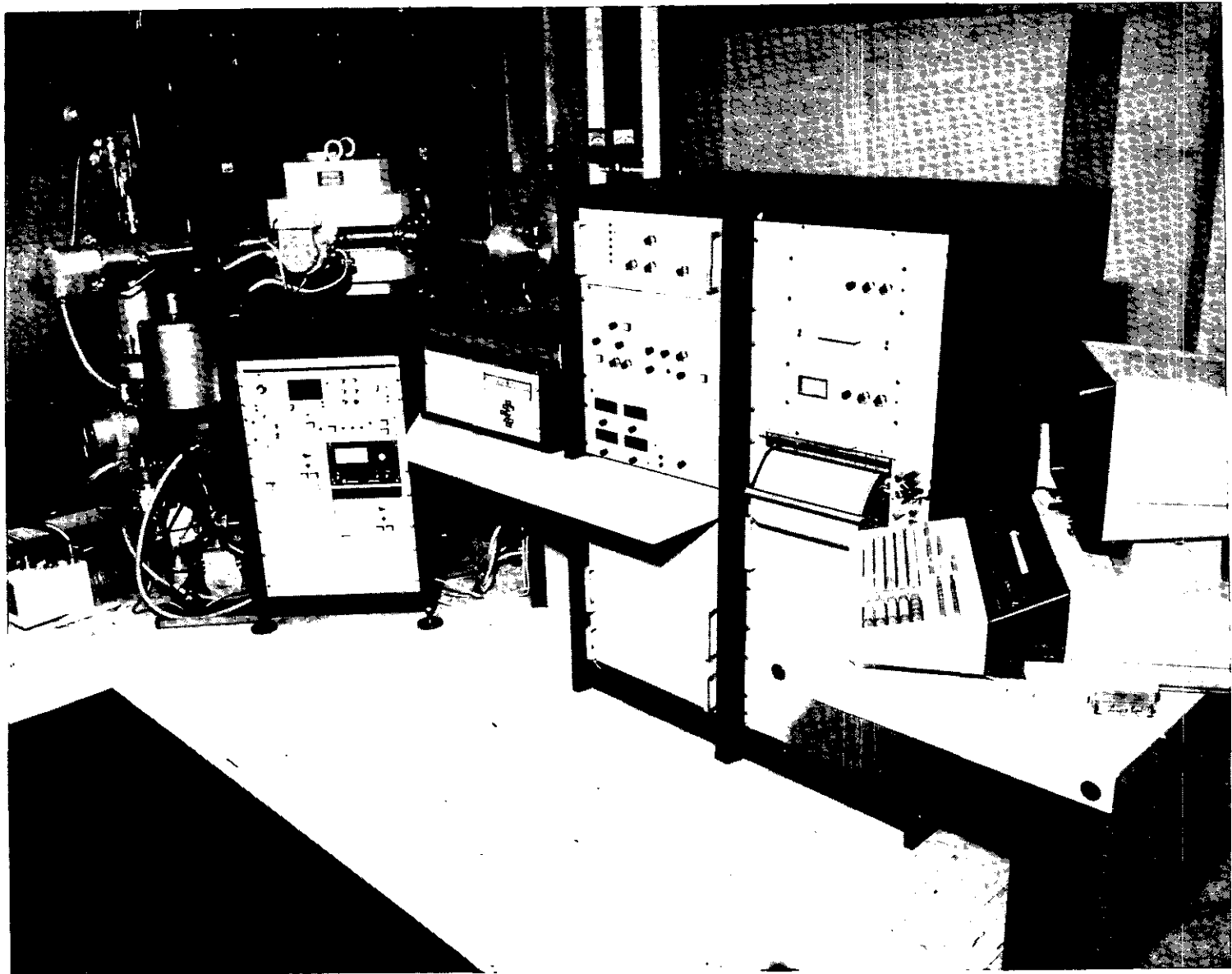


FIGURE 5. MAT 250 HDT Mass Spectrometer

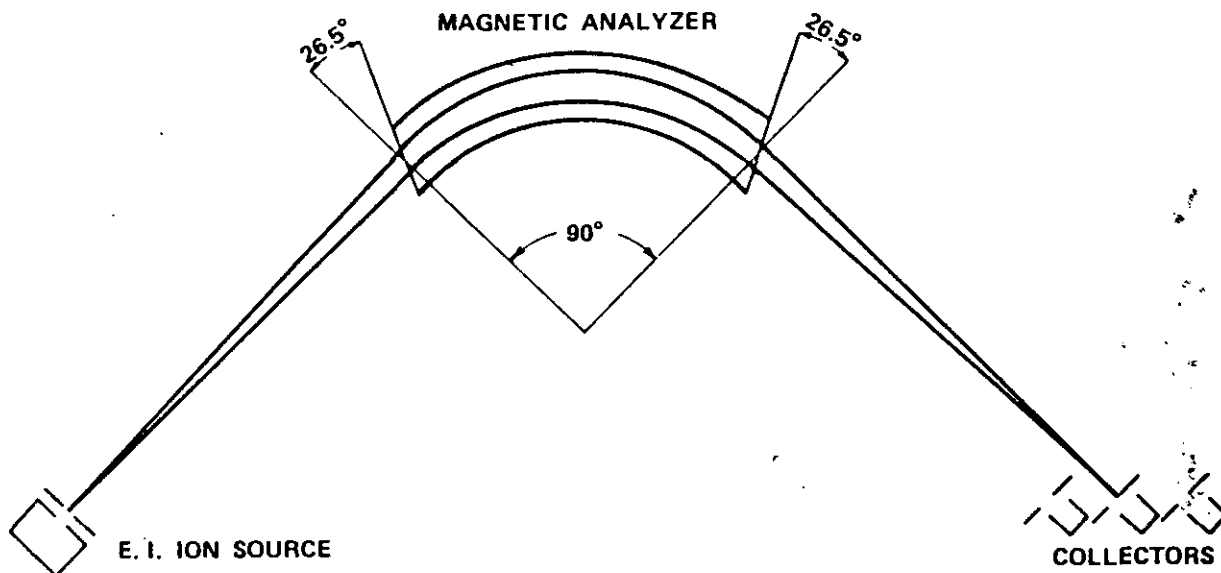


FIGURE 6. Ion Optical System Finnigan MAT 250 HDT

exit slit and is directed into the analyzer. An einzel lens between the source and the analyzer further improves the "Z" focusing.

The instrument is equipped with the triple Faraday cage ion collector system of the standard MAT 250. Only one collector is used at a time when analyzing H, D, and T mixtures. Therefore, the collectors have been equipped with different sized slits. This allows the operator to run the instrument at different resolutions without having to vent the analyzer to change slits.

The voltages in the ion source are fixed, except for the single change needed to compensate for mass discrimination. A series of voltage taps, adjusted for masses 2, 3, 4, 5, 6, and a mass >12, are provided for one focusing half plate. The taps are switched automatically when the mass spectrum is scanned.

Modern solid-state technology is used in all the electronic units. The high voltage and magnet power supplies are designed for both manual and computer control.

The analyzer system is evacuated by two turbomolecular pumps backed by a small diffusion pump and a dual-stage mechanical pump. The diffusion pump increases the hydrogen compression ratio of the pumping system. A liquid nitrogen trap is provided in the source pumping line. Ion and thermocouple gages monitor the pressure. Automatic controls shut down and vent the instrument safely if a pressure rise or a power failure occurs.

The entire mass spectrometer operates under control of a Hewlett-Packard HP-9845 computer with software provided by Finnigan MAT. The prototype software was satisfactory for H-D ^3He mixture tests at a resolution of 600. It was not satisfactory for D-T mixture tests. Final software for operation at a resolution of 600 was being written by the Finnigan MAT staff in Bremen, but it was not completed before the end of the SRL/SRP mass spectrometer evaluation program.

The MAT prototype software was modified for operation at a resolution of 1300 by T. Mehrhoff, General Electric Co., St. Petersburg, FL (GENE). The modified software proved satisfactory for the analysis of D-T mixtures with a MAT 251 HDT, the production version of the MAT 250 HDT mass spectrometer.

FACTORY ACCEPTANCE TESTS

The Finnigan MAT 250 HDT mass spectrometer successfully passed all the specified acceptance tests shown in Appendix VI. P. Chastagner and H. L. Daves witnessed the tests in Bremen, Germany, on September 8-10, 1980. Additional tests in other modes

of operation were conducted September 11 and 12. The instrument was shipped to SRL and arrived October 3.

Acceptance test conditions for the MAT 250 HDT were unusually severe in that magnetic collimation of the electron beam in the ion source was prohibited. Under these conditions, there is no mass discrimination, but ion intensity is usually diminished and other characteristics are degraded. The result is a "worst case" test which established the minimum capabilities of the instrument. As shown in Table 5, the sensitivity was almost four times the specified value and the instrument equalled or exceeded all other specifications.

TABLE 5

Performance Test Results for Varian MAT 250 HDT Mass Spectrometer

<u>Operating Characteristic</u>	<u>Specification*</u>	<u>Result**</u>
H ₂ sensitivity	>10 ⁻⁹ A/torr	3.8 x 10 ⁻⁹ A/torr
H ₂ linearity	<0.01	0.006
Ar linearity	>0.01	0.004
H ₂ -D ₂ mass discrimination	None detected	None detected
HD- ³ He abundance sensitivity	>20,000	40,000
D ₃ production	<120 ppm at 10 ⁻⁹ A	58 ppm
Peak flatness	<0.001 over ±5% width	0.00062

* Appendix VII

** Measured at Bremen, Germany on September 8-12, 1980

During the course of the testing, Chastagner and Daves concluded that, despite the good results, routine operation of the instrument without source magnets would not be practical. Without the magnets, only about 10% of the electrons emitted from the filament reach the trap. The other 90% impact the walls of the ionization chamber. Insulating deposits build up on the chamber walls during normal operation. These deposits become charged and deflect the electron beam; the source becomes unstable and frequent cleaning is required. However, when collimating magnets are in place, 80 to 90% of the electrons reach the trap, and few electrons impact the walls of the ionization chamber. Consequently, the effect of charging the deposits is minimized, stability is maintained, and normal source life is attained.

With collimating magnets in place in the ion source, sensitivity is improved about 25%, the ion peak shape is markedly better (Figure 7), and the resolution is substantially improved. When the collector slit was reduced from 0.6 mm to 0.34 mm, the resolution was increased to about 850 (Figure 8) without loss of sensitivity. The sensitivity for H_2 and D_2 , corrected to equivalent SRP and MSTG terms ($0.2 \text{ cm}^3 \text{ N}_2/\text{sec}$ leak and 1×10^{11} ohm input resistor) was about $920 \text{ mV}/\mu$ with a total emission of 1 mA. This is equivalent to about $90 \text{ mV}/\mu$ with conventional electron beam intensities ($100 \mu\text{A}$ total emission). The high sensitivity and high ion output of the instrument are obtained with high ionizing currents and high sample pressures. The Varian source is linear at ionizing currents and sample pressures that are each ten times higher than those we consider conventional.

The collimating magnets introduced 15 to 20% mass discrimination between H_2 and D_2 . Tests showed that a small voltage adjustment to one ion source focus lens would restore optimum transmission and eliminate the mass discrimination.

Equilibration, the formation of HD or HT in the ion source by reaction with the H_2O background, produced about 0.2% HD when several hundred microns of D_2 gas was introduced into the mass spectrometer. Although this was significantly less than observed with Varian's prototype instrument, this much equilibration is still undesirable. Further reduction of equilibration was made a major goal during the evaluation program.

Extrapolation of then current performance data suggested the possibility of increasing the resolution of 1500 to 1800. The resolutions obtained in the tests at Bremen were about 90% of the theoretical values, which indicated very good focusing of the ion source and the stigmatic-focusing analyzer system. If this 90% figure is maintained with smaller slits, the resolution would be about 1600 with a 0.05-mm source exit slit and a 0.20-cm collector slit. Ion intensity, which is in theory proportional to the area of the ion-source exit slit, could be about 1×10^{-9} A. Even allowing for unspecified experimental losses, the intensity should be at least 3×10^{-10} to 5×10^{-10} A. Under such conditions, abundance sensitivity at the mass 4 HT- D_2 doublet should be 5,000 to 7,000. Varian furnished a variety of slits with the instrument so that higher-resolution performance could be tested at SRL.

INSTALLATION AND ACCEPTANCE AT SRL

Installation of the MAT 250 HDT was completed in January 1981 after a long series of delays. The primary cause of the installation problems appeared to be that the Varian engineer had little or no experience with the instrument. Problems included: shipping

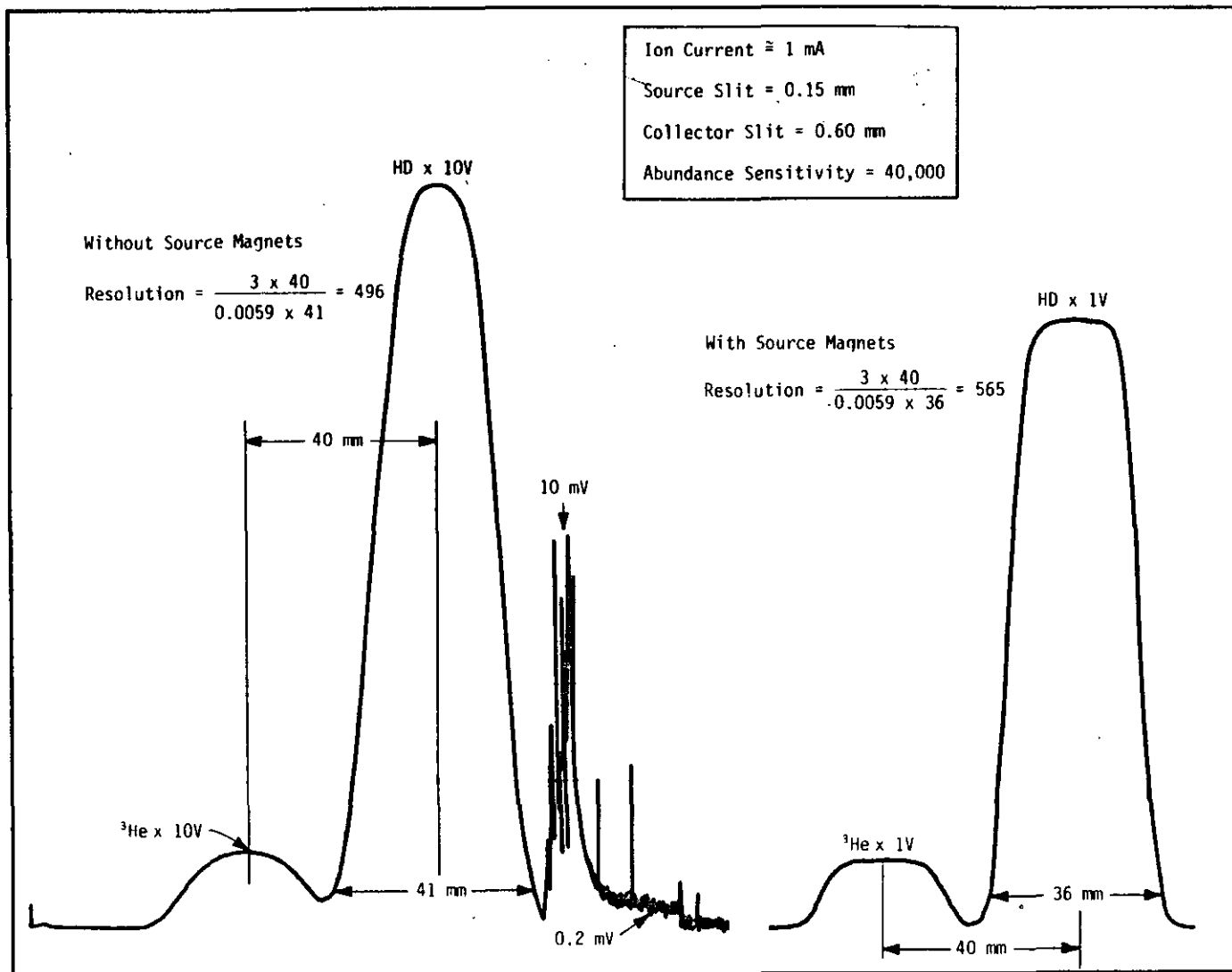


FIGURE 7. Use of Collimating Source Magnets Improves Ion Peak Shape

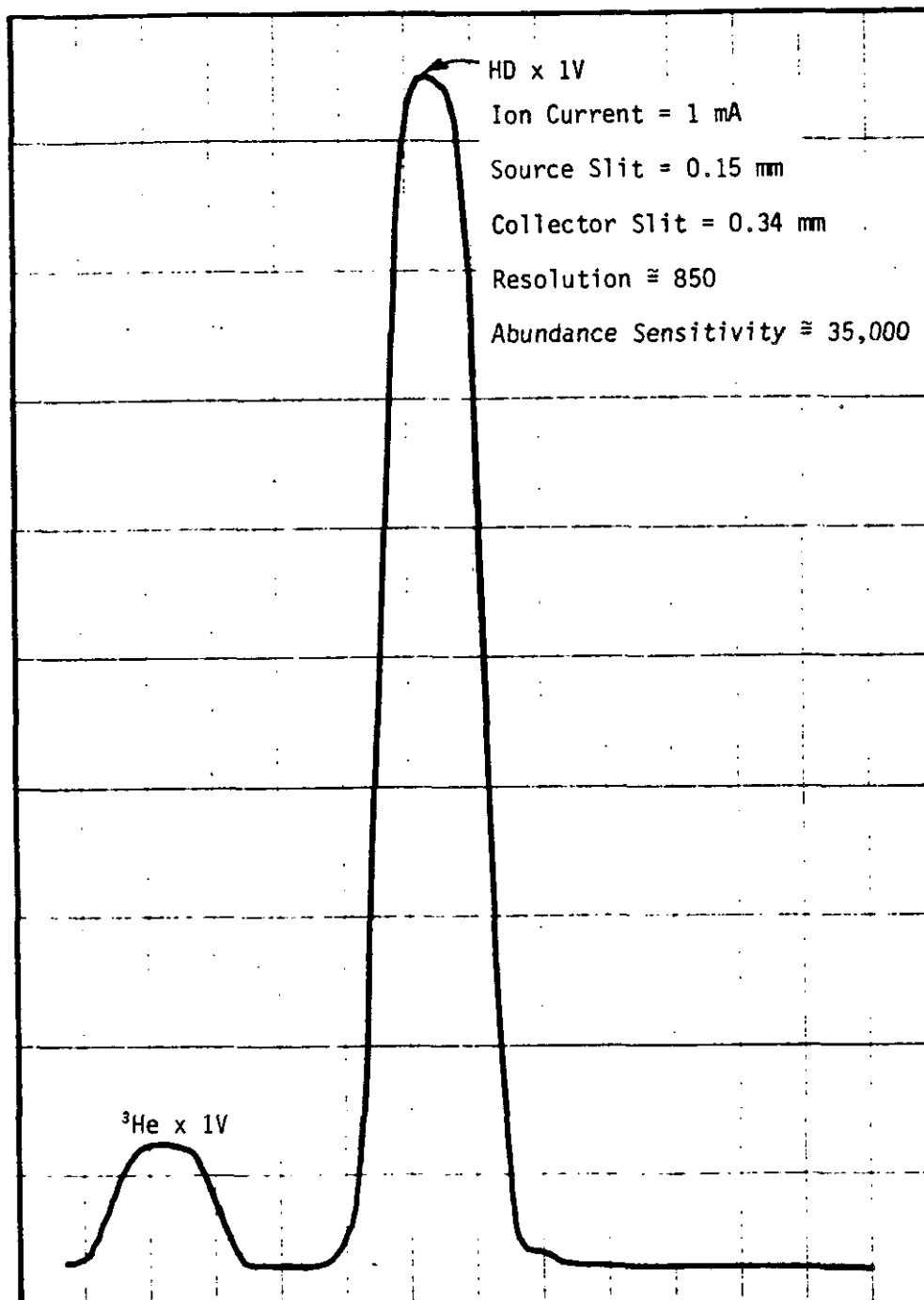


FIGURE 8. Improved Resolution Achieved by Use of Collimating Source Magnets

damage; delays in obtaining an inlet system; vacuum system accidents caused by improper operation; failure to order replacements for damaged parts; and inability to align the analyzer.

The MAT 250 HDT was accepted from Varian February 19, 1981 after a series of specification tests in which barely acceptable data were obtained (Appendix VII). An earlier set of test data was rejected because the instrument was not operated under a single set of conditions. The results of these tests were markedly poorer than those obtained in the Bremen tests. The sensitivity was less than half that obtained in Bremen and all other factors were worse. In post acceptance tests with source magnets in place, the sensitivity was still low, the peak shape was poor, and the linearity varied from about 0.4% to more than 1% in consecutive tests under constant conditions. Subsequently, Du Pont personnel found that the analyzer was out of alignment mechanically, and that the einzel lens system was "floating", i.e., was not connected to the electrical control system. When the analyzer was realigned and the einzel lens system was connected, the peak shape and the linearity were improved. But the sensitivity was still low and the resolution was poor.

TRANSITION FROM VARIAN TO FINNIGAN MANAGEMENT

In February 1981, Finnigan and Varian announced that the MAT operation would be sold to Finnigan. The transition was completed in May and Finnigan management immediately moved to rectify the problems with the MAT 250 HDT. In June, a factory engineer restored the instrument to the performance level obtained in the Bremen tests. In September, factory engineers upgraded, at Finnigan's expense, the instrument for full digital control. Essentially all of the original analog control circuitry was replaced. The instrument was then functionally equivalent to the MAT 251 HDT. The prototype software for operation at a resolution of 600 was written at that time and full computer control was implemented.

EQUILIBRATION

Equilibration of H and D in the ion source was reduced to 300 ppm from the 2700 ppm level when the original ribbon filament was replaced with a wire filament. Equilibration with the wire filament in place was essentially the same as that of the GAZAB ion source.

CHARACTERIZATION TESTS

An SRP blended mixture, 4.03M % ^3He , balance D_2 , was analyzed at five different times to evaluate the precision and accuracy of the MAT 250 HDT. The ^3He data from all of the runs are summarized in Table 6. A typical set of runs is given in Table 7. The precision obtained for the ^3He determination is a factor of two better than that obtained with the GAZAB mass spectrometer.

One of the Mound Laboratory mixtures, mixture No. 1, that was used for acceptance testing of the GAZAB, was analyzed repetitively over a four-day period on the MAT 250 HDT. The results are in Tables 8-11. Each day the mass spectrometer was calibrated in triplicate before the analyses were made. The results are consistent with those made with other mass spectrometers. All instruments have shown that the ^3He concentration is lower than the theoretical value and that the total H concentration is higher. The systematic changes in closure, becoming more negative in each successive analysis, as if the sensitivities were changing, were caused by the source contamination described above.

TABLE 6

Replicate Analyses of SRP Mix by Finnigan MAT 250 HDT
Mass Spectrometer

Run #	Precision-Accuracy SRP Mix (4.03% ^3He - Balance D_2)				
	Time 1	Time 2	Time 3	Time 4	Time 5
1	4.028	4.033	4.022	4.007	4.016
2	4.028	4.033	4.026	4.012	4.018
3	4.027	4.034	4.027	4.012	4.015
4	4.026	4.033	4.025	4.012	4.014
5	4.029	4.040	4.023	4.011	4.016
6	4.027	4.031	4.023	4.013	4.016
7	4.026		4.024	4.014	4.016
8	4.026		4.025	4.013	4.014
9	4.026		4.024	4.011	4.014
10	<u>4.023</u>		<u>4.026</u>	<u>4.012</u>	<u>4.016</u>
Average	4.026	4.032	4.024	4.012	4.016
RSD %	0.04%	0.04%	0.04%	0.05%	0.03%
Accuracy	-0.08%	+0.05%	-0.14%	-0.45%	-0.36%

TABLE 7

Typical Set of Runs with SRP Mix by Finnigan MAT 250 HDT
Mass Spectrometer

Precision Runs SRP Mix (4.03% ^3He — Balance D_2)

Run No.	He	HD	D	N	Closure Baratron - Σ_{pp}^* Percent
1	4.022	0.778	95.166	0.030	-0.22
2	4.026	0.731	96.211	0.031	-0.37
3	4.027	0.731	95.212	0.031	-0.52
4	4.025	0.730	95.214	0.030	-0.63
5	4.023	0.730	95.217	0.029	-0.52
6	4.023	0.729	95.217	0.030	-0.40
7	4.024	0.730	95.216	0.030	-0.25
8	4.025	0.729	95.215	0.030	-0.36
9	4.024	0.729	95.216	0.030	-0.39
10	4.026	0.730	95.213	0.030	-0.42

* Σ_{pp} = sum of partial pressures

TABLE 8

Precision — Accuracy Finnigan MAT 250 — Mound Mixture No. 1,
4/19/82

Run No.	H ₂	³ He	HD	D ₂	Total H	Closure,* $\left(\frac{\text{Baratron}-\Sigma_{pp}}{\text{Baratron, \%}}\right)$
1	0.461	3.996	11.253	84.277	6.088	+0.36
2	0.452	3.995	11.249	84.288	6.077	+0.12
3	0.454	4.000	11.251	84.285	6.079	-0.01
4	0.460	3.995	11.250	84.275	6.085	-0.05
5	0.461	3.997	11.247	84.247	6.084	-0.29
6	0.460	3.996	11.250	84.278	6.085	-0.39
7	0.461	3.998	11.248	84.280	6.085	-0.48
8	0.463	<u>3.994</u>	11.250	84.274	<u>6.084</u>	-0.49
Average		3.996			6.084	
Std. Dev.		±0.0019			±0.0039	
Rel. Std. Dev.		±0.05			±0.06	
ΔTheor.		-0.12			+1.26	

* Σ_{pp} = sum of partial pressures

TABLE 9

Precision — Accuracy Finnigan MAT 250 — Mound Mixture No. 1,
4/20/82

Run No.	H ₂	³ He	HD	D ₂	Total H	Closure,* $\left(\frac{\text{Baratron} - \Sigma_{pp}}{\text{Baratron, \%}} \right)$
1	0.457	3.983	11.231	84.317	6.072	+0.19
2	0.463	3.981	11.212	84.332	6.069	+0.12
3	0.462	4.981	11.211	84.335	6.067	-0.32
4	0.462	3.981	11.210	84.336	6.067	-0.47
5	0.462	3.982	11.209	84.335	6.066	-0.57
6	0.463	3.982	11.209	84.333	6.068	-0.68
7	0.458	3.982	11.207	84.341	6.062	-0.75
8	0.462	3.983	11.207	84.338	6.066	-0.83
9	0.450	<u>3.983</u>	11.207	84.350	<u>6.054</u>	-0.90
Average		3.982			6.066	
Std. Dev.		±0.0009			±0.0051	
Rel. Std. Dev.		±0.02			±0.08	
$\Delta_{\text{Theor.}}$		-0.47			+0.96	

* Σ_{pp} = Sum of partial pressures

TABLE 10

Precision — Accuracy Finnigan MAT 250 — Mound Mixture No. 1,
4/21/82

Run No.	H ₂	³ He	HD	D ₂	Total H	Closure,* ($\frac{\text{Baratron}-\Sigma_{pp}}{\text{Baratron, \%}}$)
1	0.492	4.006	11.409	84.067	6.196	+0.75
2	0.474	3.994	11.260	84.246	6.104	+0.13
3	0.473	3.993	11.234	84.276	6.090	-0.09
4	0.471	3.993	11.228	84.286	6.085	-0.21
5	0.472	3.993	11.225	84.288	6.084	-0.34
6	0.472	3.993	11.231	84.279	6.088	-0.40
7	0.471	3.994	11.230	84.282	6.087	-0.48
8	0.471	3.994	11.227	84.286	6.084	-0.53
9	0.470	3.995	11.228	84.286	6.084	-0.58
10	0.467	3.993	11.226	84.292	6.080	-0.65
11	0.469	3.996	11.226	84.287	6.082	-0.69
12	0.471	<u>3.995</u>	11.226	84.287	<u>6.083</u>	-0.68
Average		3.994			6.086	
Std. Dev.		±0.0010			±0.0064	
Rel. Std. Dev.		±0.03			±0.11	
$\Delta_{\text{Theor.}}$		-0.17			+1.29	

* Σ_{pp} = Sum of partial pressures

TABLE 11

Precision — Accuracy Finnigan MAT 250 — Mound Mixture No. 1,
4/22/82

Run No.	H ₂	³ He	HD	D ₂	Total H	Closure,* $\left(\frac{\text{Baratron}-\Sigma_{pp}}{\text{Baratron, \%}}\right)$
1	0.471	3.985	11.231	84.279	6.086	+0.35
2	0.471	3.983	11.215	84.298	6.078	+0.02
3	0.472	3.982	11.203	84.309	6.074	-0.17
4	0.472	3.982	11.199	84.313	6.072	-0.33
5	0.472	3.981	11.195	84.317	6.070	-0.38
6	0.477	3.992	11.209	84.286	6.082	-0.07
7	0.472	3.982	11.196	84.315	6.070	-0.35
8	0.471	3.982	11.194	84.318	6.068	-0.44
9	0.472	3.982	11.194	84.317	6.069	-0.46
10	0.471	<u>3.982</u>	11.194	84.319	<u>6.068</u>	-0.55
Average		3.983			6.074	
Std. Dev.		±0.0032			±0.0063	
Rel. Std. Dev.		±0.08			±0.10	
$\Delta_{\text{Theor.}}$		-0.45			+1.10	

* Σ_{pp} = Sum of partial pressures

EMISSION REGULATION MODE

The mode of regulating the emission of ionizing electrons from the filament in the ion source was changed from the "total emission" mode originally used to the "trap current" mode in mid-July 1982. In the "total emission" mode, the total number of electrons that leave the filament is controlled. It is assumed that a constant percentage traverse the ionizing region. In the "trap current" mode, the number of electrons that traverse the ionizing region and arrive at the "trap" (ionizing electron collector) is controlled. The change improved the day-to-day stability of the ion source. Day-to-day changes in sensitivity for the July 15 to August 24 period are shown graphically in Figure 9. No values were plotted for the August 12-16 period because daily power outages prevented the source from stabilizing.

The source temperature regulator was also turned off in mid-July. The source cooled and stabilized at a temperature below the 150 to 250°C range of the source monitor. The temperature was not measured. At the lower source temperature, the sensitivities were about 20% greater than they were when the source was stabilized at 150°C.

GEND TESTS AT 1300 RESOLUTION

Norman Parsons, GEND, informed us that their MAT 251 HDT mass spectrometer passed its acceptance tests in Bremen. After the low resolution tests (resolution >500), Finnigan MAT personnel installed small slits and demonstrated a resolution of 1300 with good peak shape. Parsons furnished Figure 10, which shows the $H_2^+-D^+$ doublet. Sensitivity for H_2 was 7.2×10^{-11} A/torr at high resolution inlet. 1.26×10^{-9} A/torr at a low resolution with about 0.1 cc/SPC (ford N_2) molecular leak in the inlet system. Ion output at high resolution was linear within 0.7% (extreme spread) over the inlet system pressure range of 0.12 to 1.02 torr.

After installation at GEND the MAT 251 HDT was put into tritium service. Norman Parsons reported that it was being operated routinely at a resolution of 1300. Parsons sent the set of initial results shown in Table 12. These runs were made with a partially plugged molecular leak in the sample inlet system which is the reason for the relatively low sensitivity. Results obtained with the GEND Du Pont 21-104 mass spectrometer are given in Table 13 for comparison. The sample, SRP mix #3-MR was made up to be 50% D - 50% T in 1976. The values obtained with the MAT 251 HDT agree very well with the theoretically decayed values when corrected for protium ingrowth. Tom Mehrhoff, GEND, modified the low resolution Finnigan MAT software extensively. The modified software operates the MAT 251 HDT reliably at 1300 resolution.

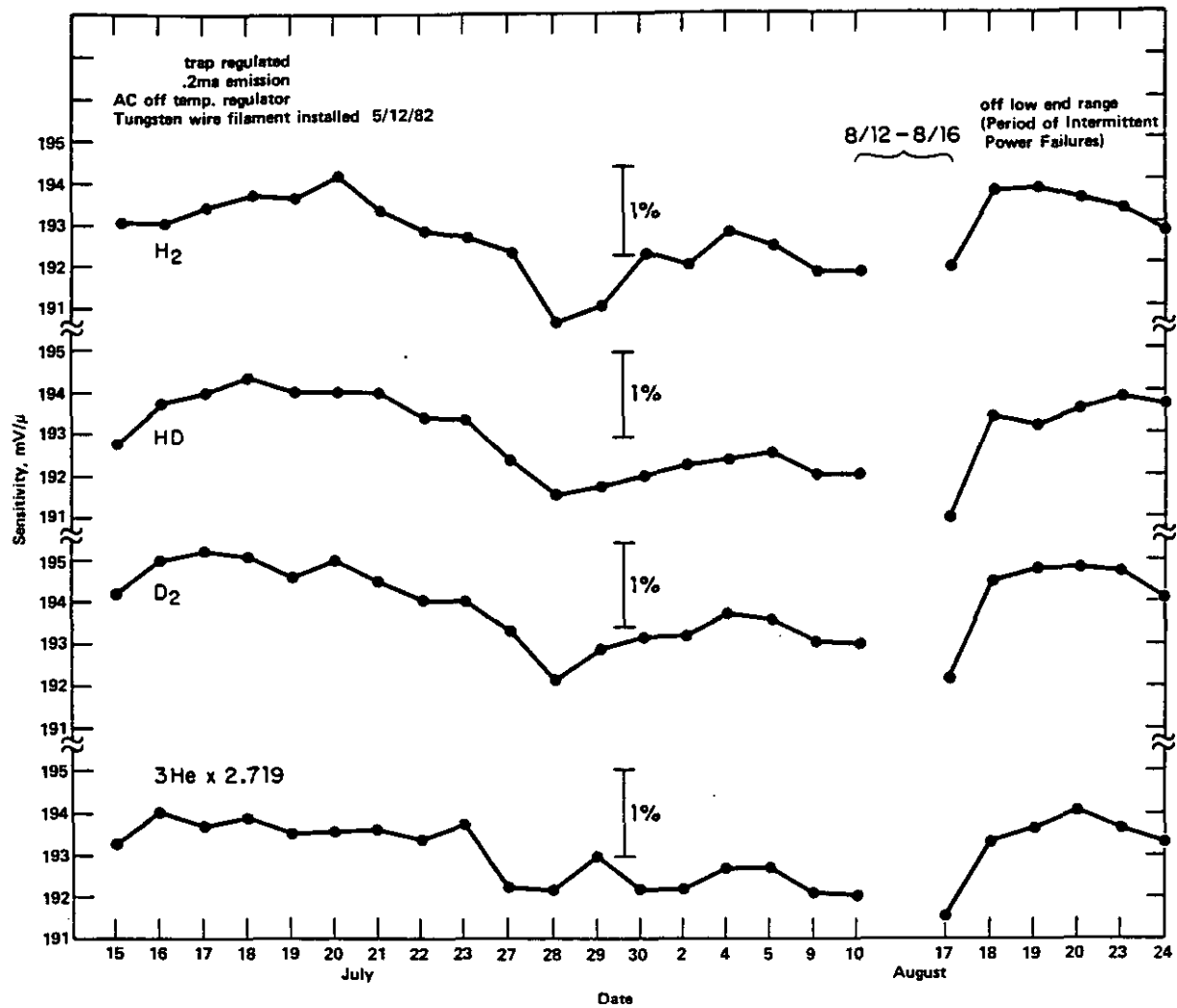


FIGURE 9. Sensitivity Change vs Time SRP MAT 250 HDT

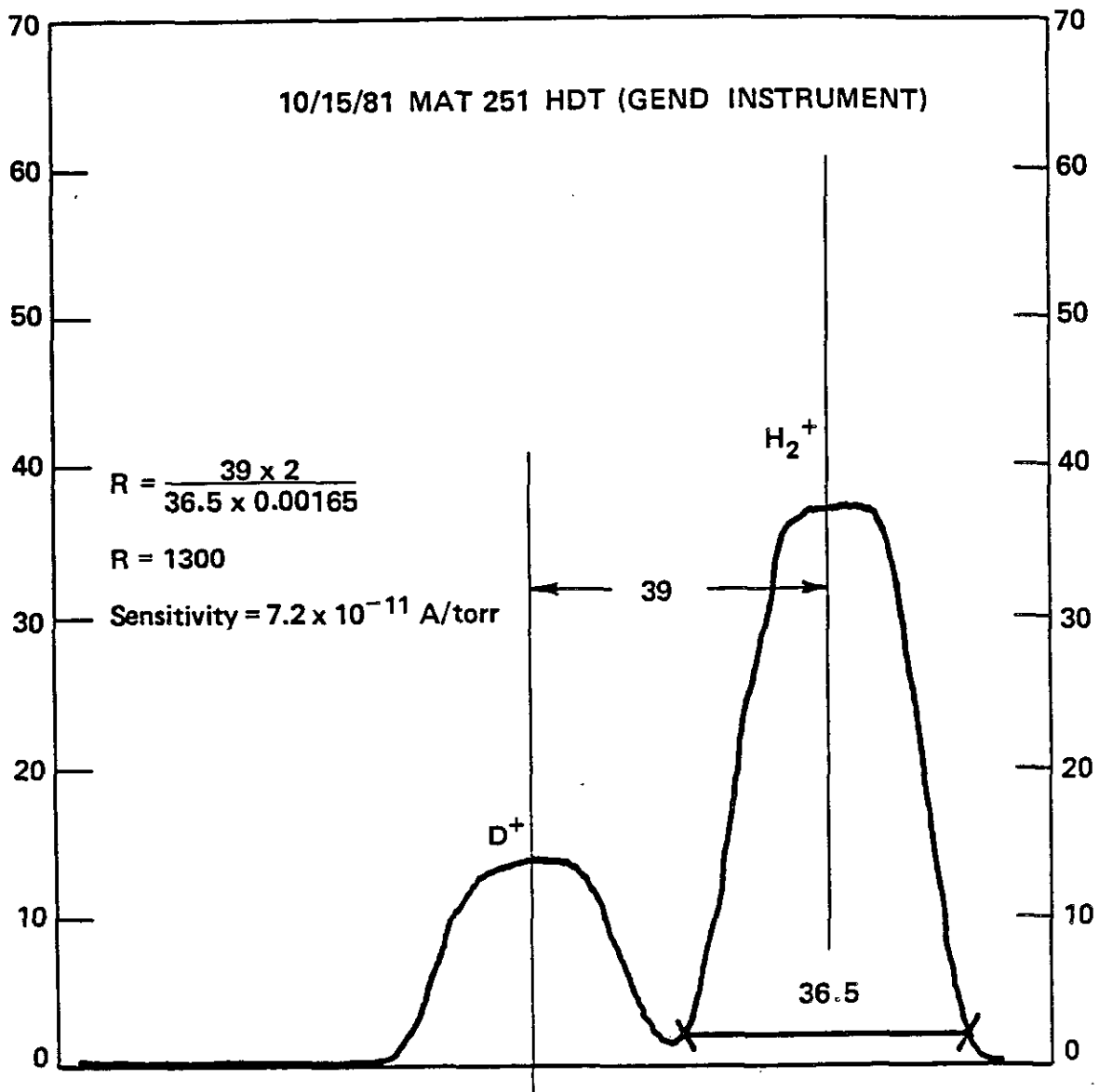


FIGURE 10. High-Resolution Test (in Bremen)

TABLE 12

Operation of Finnigan MAT 251 Mass Spectrometer at GEND*

Operating Parameters

Resolution - ~1300
 Ribbon Filament; Emission - 0.400 MA
 Ion Source Temperature - 149.6°C
 Trap - 149.6 volts
 Electron Energy - 80.1 volts
 Electron Plate - 96.9 volts
 Amplifier IV - 1×10^{11} ohms
 Sensitivity - $D_2 = 3.65 \text{ mV}/\mu$

Calibrated 3/29/82 using SRP Mix #5 T330

Analysis of SRP Mix #3-MR (50-50) Vessel #1274-07, 3/30/82

	<u>Total H₂, mol %</u>	<u>Total D₂, mol %</u>	<u>Total T₂, mol %</u>	<u>³He, mol %</u>	<u>Inlet Press., mT</u>
	0.383	44.306	29.341	25.971	275.9
	0.386	44.229	29.313	26.072	238.0
	0.377	44.289	29.281	26.053	166.0
	0.369	44.258	29.263	26.111	164.1
	0.375	44.228	29.320	26.078	161.8
	0.391	44.308	29.333	25.968	251.2
	0.394	44.236	29.317	26.054	211.8
	0.390	44.235	29.281	26.096	178.5
	0.398	44.273	29.279	26.050	150.5
	0.391	44.215	29.318	26.077	151.8
Mean	0.385	44.258	29.305	26.053	
S.D.	0.009	0.034	0.026	0.048	
S.D. %	2.34	0.18	0.08	0.09	
Std. Value	0.105**	44.312	29.541	26.038	
Difference %		-0.12	-0.80	+0.06	

* GEND data courtesy of N. H. Parsons.

** This mix has been in use since October 1975. Pressure is 52 torr. Hydrogen ingrowth has been confirmed to be higher than theoretical. Theoretical value appears to be close to value obtained in the analyses.

TABLE 13

Operation of Du Pont 21-104 Mass Spectrometer at GEND*

Operating Parameters

Resolution - ~1300

Analysis of SRP Mix #3-MR (50-50) Vessel #1274-07

	<u>Total H₂, mol %</u>	<u>3He, mol %</u>	<u>Total D₂, mol %</u>	<u>Total T₂, mol %</u>
	0.420	25.575	44.335	29.670
	0.387	25.328	44.331	29.954
	0.324	25.255	44.443	29.977
	0.386	25.293	44.470	29.850
	0.402	25.449	44.368	29.781
Mean	0.384	25.380	44.389	29.847
S.D.	0.029	0.135	0.059	0.123
S.D. %	7.55	0.53	0.13	0.41
Std. Value	0.105	26.038	44.312	29.541
Difference %		-2.53	+0.17	+1.04

* GEND data courtesy of N. H. Parsons.

THE SAVANNAH RIVER LABORATORY TEST FACILITY

Summary

The test facility was a large air conditioned laboratory that was equipped with duplicate inlet systems for the mass spectrometers and containment facilities for tritium. A series of tests established that the inlet systems were identical and that meaningful comparisons could be made between the two mass spectrometers. T/D/³He and D/H/³He standards were provided to allow assessment of both the precision and the absolute accuracy of the mass spectrometers.

Facility Description

The Savannah River Laboratory Test Facility (Figure 11) was designed so the GAZAB and MAT 250 HDT mass spectrometers could be tested simultaneously with the same standard materials. The facility is air conditioned and has:

- Duplicate mass spectrometer inlet systems supplied by common standard manifolds
- A tritium recovery system
- Hood exhausts for the ion sources
- A tritium monitoring system
- Full containment for all tritium handling operations.

The dual inlet systems and standard distribution systems were designed and built by H. L. Daves and W. B. Hess. The systems are located in a 8-foot long glove box (Figure 12) that is located between the mass spectrometers.

As shown in Figure 13, each mass spectrometer has its own dedicated inlet system that includes a 3 liter volume, Baratron pressure sensors, a tritium activity meter for measuring total tritium, a molecular leak and a ^4He flow standard. Manifolds for nonradioactive gases and for tritium mixtures are shared by both mass spectrometers. The manifold for nonradioactive gases is isolated from the rest of the system by valve, V2, and is located outside the glove box. All valves except the dispensing valves on the tritium containers are pneumatic and can be operated from either mass spectrometer console.

The various sampling and storage vessels used on the system are diagrammed in Figure 14. The "cold gas bottles" are used for all nonradioactive pure gases except HD. They are equipped with internal capillary leaks to limit the gas flow and with pneumatic valves for operator convenience. D-T mixtures and HD are stored in "passivated stainless steel standard bottles". A T_2 storage vessel equipped with a "palladium diffuser" is used to provide pure T_2 to the mass spectrometers. The total tritium inventory, $\sim 7,000 \text{ Ci}$, is stored in these vessels at subatmospheric pressures.

A tritium recovery system, designed by P. Chastagner, was installed in a 4-foot-long glove box (Figure 15). As shown in Figure 16, effluents from the mass spectrometer and inlet system pumps are piped into the recovery system. Traps are provided in the collection system to remove entrained oil and a ballast volume reduces pressure surges during pumpdown from atmospheric pressure.

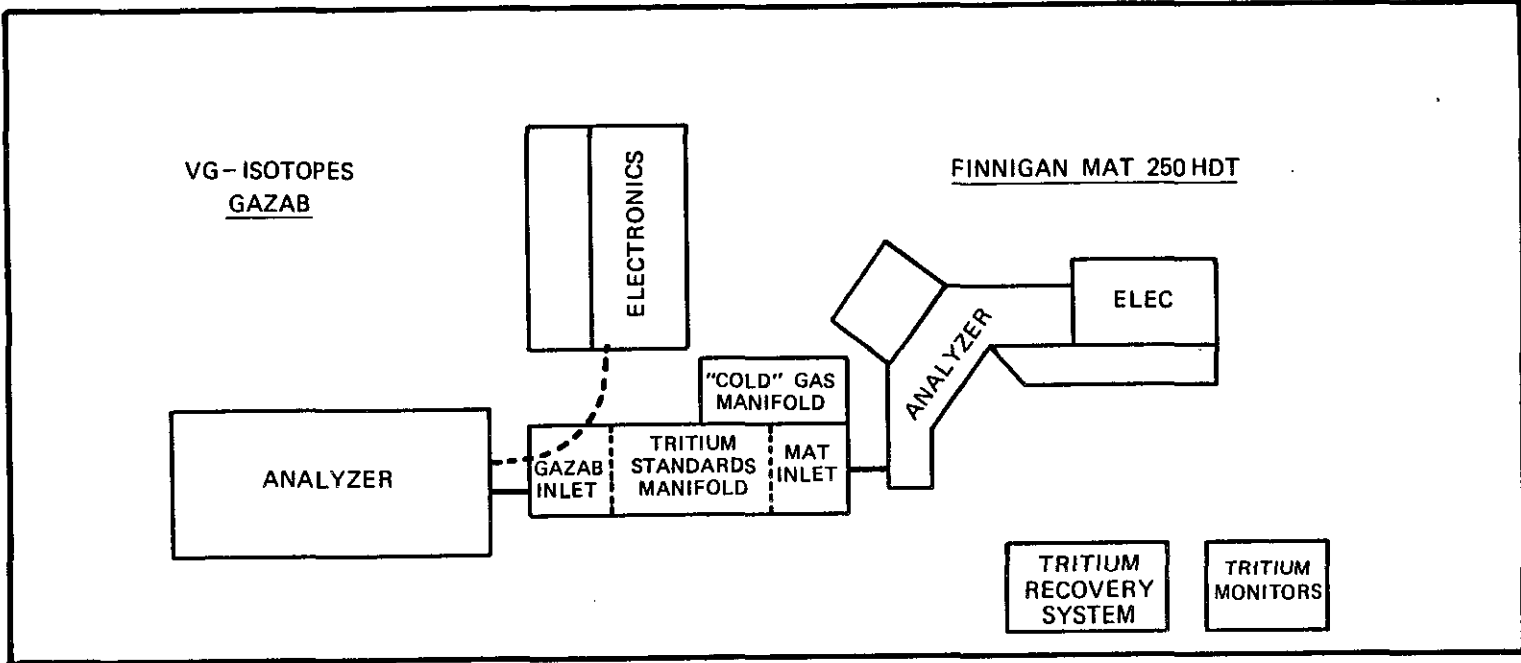


FIGURE 11. Savannah River Laboratory Test Facility

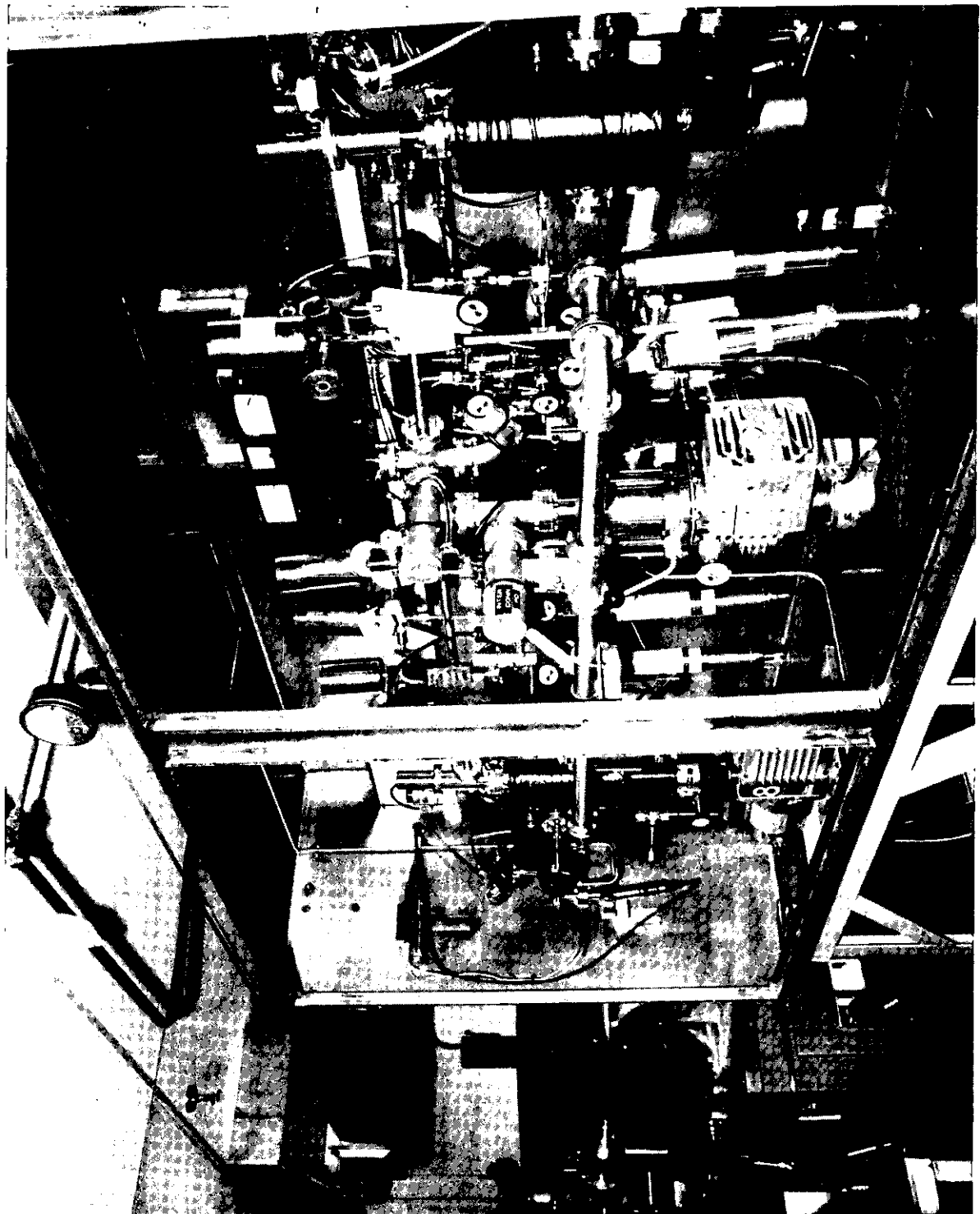


FIGURE 12. Sample Inlet System

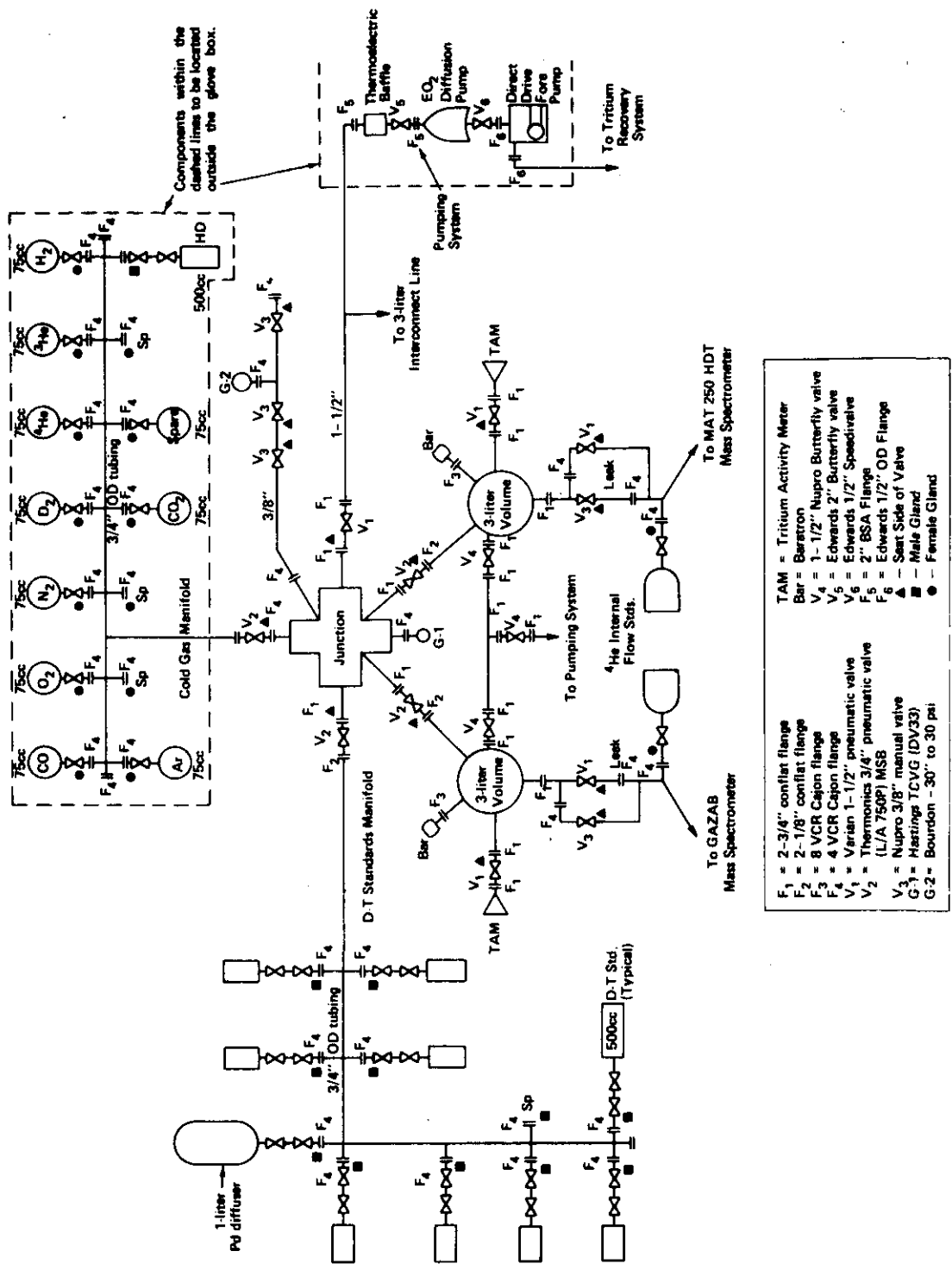
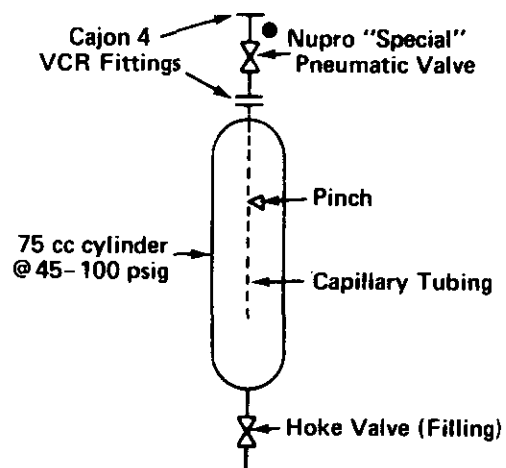
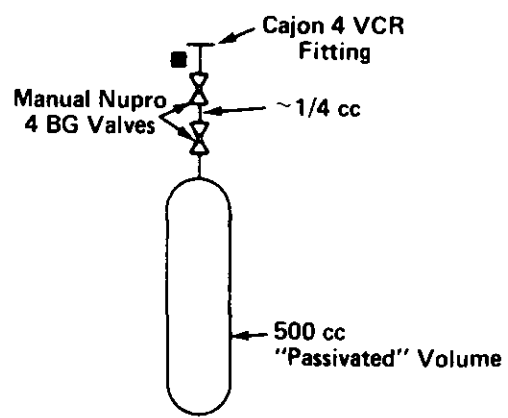


FIGURE 13. Sample Inlet System for GAZAB and MAT 250 HDT Hydrogen Isotope Mass Spectrometers

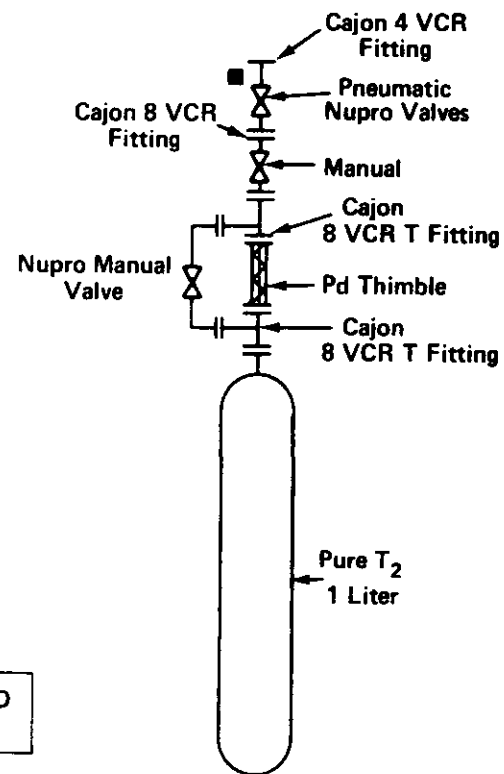
COLD GAS BOTTLE



D-T AND HD STANDARDS BOTTLE



PALLADIUM DIFFUSER



● FEMALE 4 VCR GLAND
 ■ MALE 4 VCR GLAND

FIGURE 14. Details of Sampling Vessels in Mass Spectrometer Inlet System

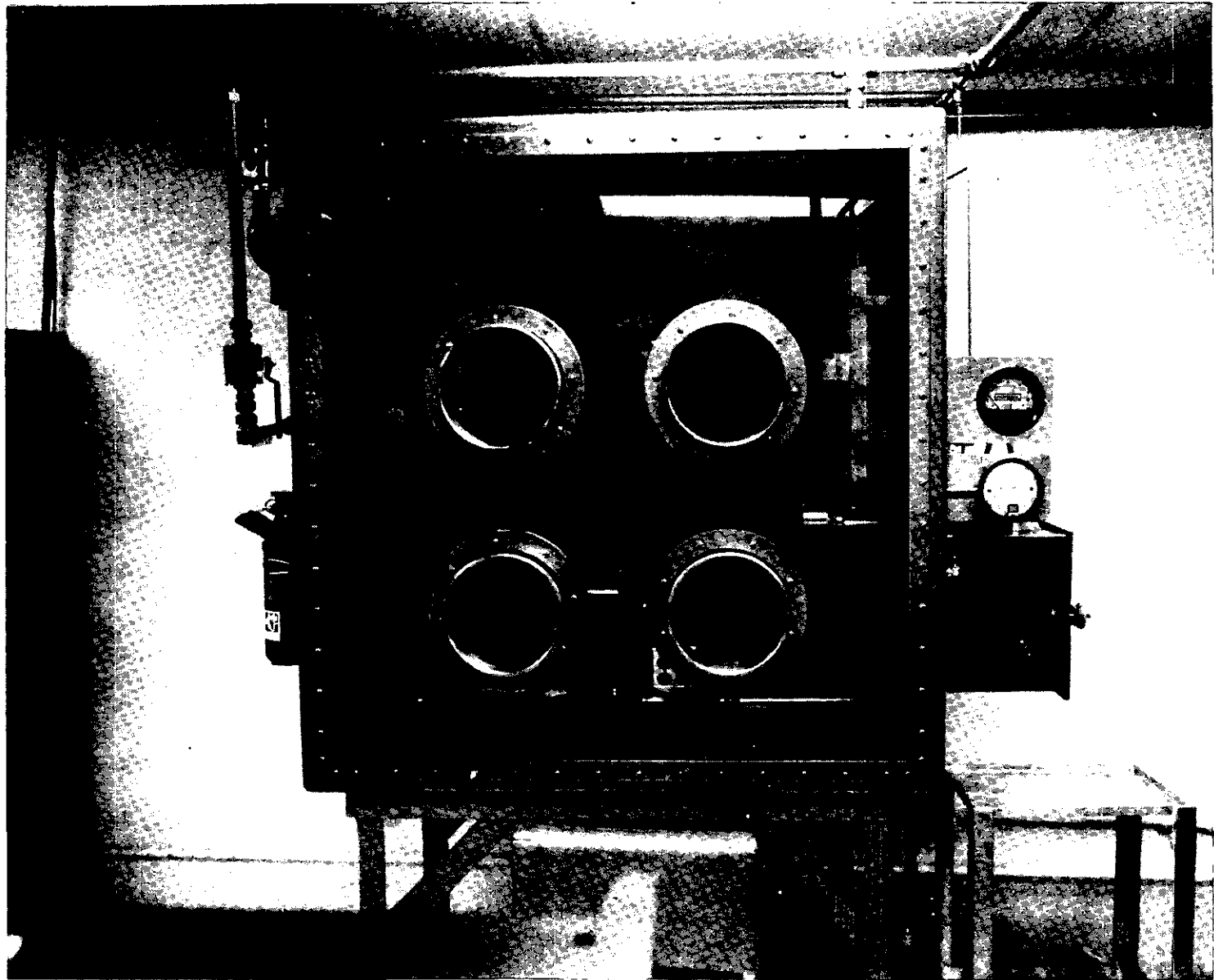


FIGURE 15. Tritium Recovery System

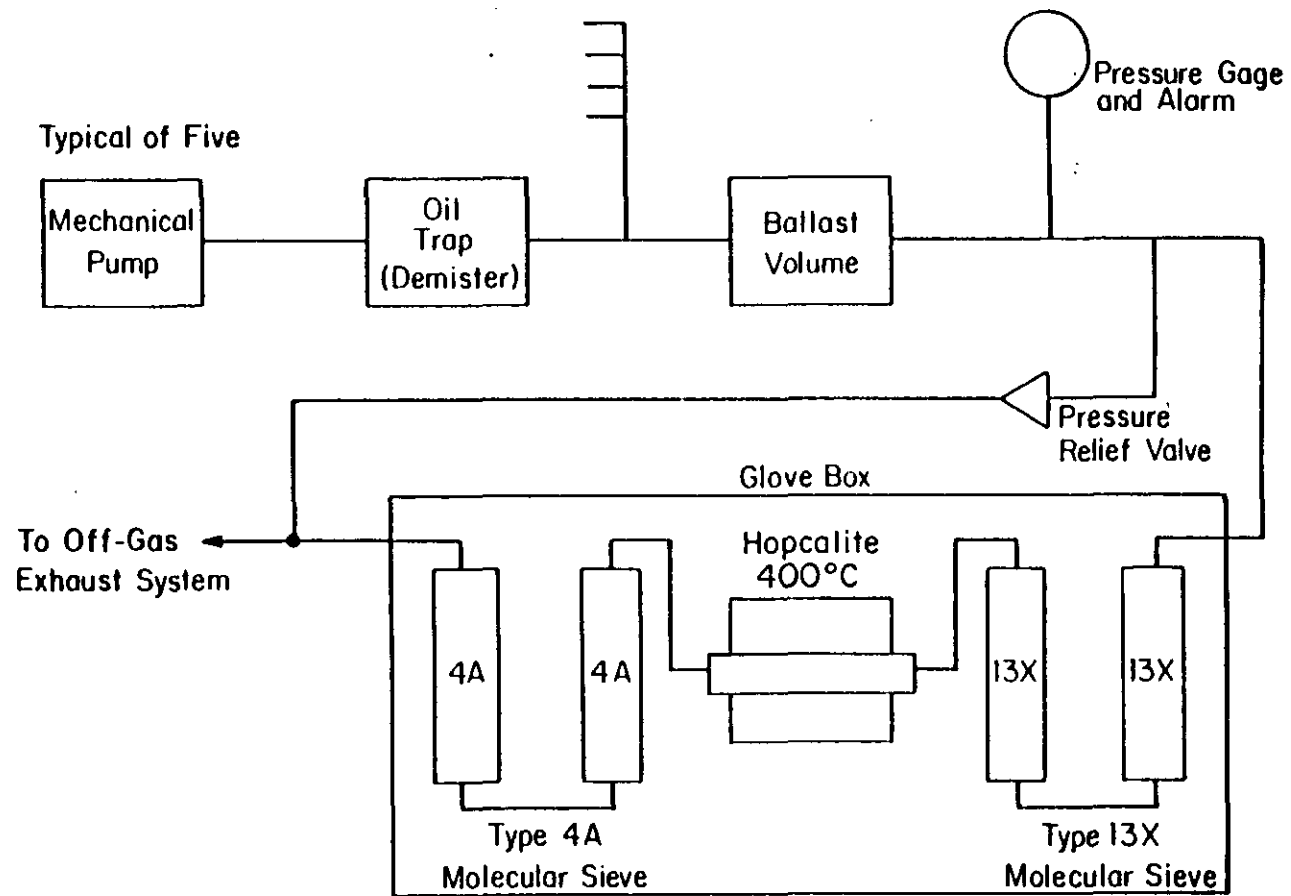


FIGURE 16. Diagram of the Tritium Recovery System

A bypass of the off-gas exhaust system opens automatically if a major air leak causes the system to become pressurized. In the recovery unit, organics are removed by beds of Type 13X molecular sieve and the hydrogen passes through heated Hopcalite where it is converted to water. The water is trapped on tandem Type 4A molecular sieve beds and gases such as N₂, Ne, and Ar pass through into the off-gas exhaust system.

The effluents from the tritium recovery system and the two glove boxes are monitored continuously by a pair of Kanne ionization chambers. A manifold system permits cross-monitoring of the inlet and recovery systems. Local hoses are provided for monitoring individual pieces of equipment anywhere in the laboratory module. The outputs of the two monitors are recorded on strip chart recorders and alarms sound if tritium is detected in the exhaust system.

BARATRON COMPARISON

After the new inlet systems were connected to the GAZAB and MAT 250 HDT mass spectrometers, Hess compared the Baratron to each other. As shown in Table 14, he found that they were linear within 0.1% in the <150 micron range. They were less linear at higher pressures. The nonlinearity was about 0.5% in the 250 to 350 micron range.

TABLE 14

Baratron Comparison (pressure in microns)

<u>VG Baratron</u>	<u>Finnigan MAT Baratron</u>	<u>Delta VG Baratron, %</u>
24.39	24.09	-1.2
46.21	45.67	-1.2
68.25	67.44	-1.2
100.13	98.92	-1.2
155.36	153.6	-1.1
176.32	174.3	-1.2
197.16	194.9	-1.2
208.3*	204.7	-1.7
304.3	299.5	-1.6
350.3	345.0	-1.5
498.7	491.6	-1.4
715.5	705.9	-1.3
970.9	958.7	-1.3

* VG Baratron has a range change at 200 microns.

The inlet system was checked for fractionation effects with a ⁴He-Ar mixture. The mixture was analyzed after different expansions between the GAZAB inlet system three liter volume and other parts of the inlet system. The data are given in Table 15. The data show that fractionation effects are <0.2% for the ⁴He-Ar mixture. Therefore, there should be no fractionation problem with D/T mixtures.

TABLE 15

Inlet System Fractionation Tests with Mixture of 30% ⁴He and 70% Ar

Day	No. Runs	Measured ⁴ He, %		Δ (Rel), 1 Expansion, %
		1 Expansion	3-4 Expansions	
1	9	29.972	29.909	-0.21
2 (a.m.)	4	29.969	29.985	+0.05
2 (p.m.)	3	29.903	29.815	-0.29
3	3	29.961	29.957	-0.01
4	4	29.960	29.923	-0.12
Theoretical		30.01		

DEUTERIUM -- TRITIUM STANDARDS

The four D-T standards used in the evaluation program were made in the SRP gas blending system. Their compositions are listed in Table 16. Standard #4 is a 50% D-50% T mixture intended for calibrating the mass spectrometers accurately for DT. Standards #1 and #3 are typical of D-T mixtures with low and high concentrations of T. Standard #2 contains 0.44% (relative) more T than Standard #1 and is designed to test the ability of the mass spectrometers to detect and measure small differences between samples. The standards were loaded into double-valved passivated 500 cm³ containers. The vessels were made by Quantum Mechanics (QM) and by Western Air Products (WAP) as shown in the table.

TRITIUM ACTIVITY METER

The tritium activity meter (TAM) located on the three-liter volume in the MAT 250 HDT inlet system was tested with a 7% T standard. The standard-sample-standard sequence of analyses was followed in this test. SRP Standard T 500 (~50% total T) was used

as a calibration standard. The results are given in Table 17. The 0.14% relative standard deviation and +0.19% relative bias are approximately the same as the precision and bias values obtained previously with a TAM in the SRP tritium facilities.

TABLE 16

D-T Standards for Evaluation of the GAZAB - MAT 250

	<u>Standard #1</u> SRP TO700	<u>Standard #2</u> SRP TO702	<u>Standard #3</u> SRP T930	<u>Standard #4</u> SRP T500
D ₂	93.000	92.969	7.000	50
T ₂	7.000	7.031	93.000	50
Vessels	WAP #1	WAP #3 WAP #4	QM #3 QM #4	WAP #2 QM #2

Double-valved 500 cc passivated vessels

Aliquot volume .25 - .3 cc

TABLE 17

Tritium Analysis by Tritium Activity Meter TAM Located on MAT 250 HDT Inlet System

<u>Run</u> <u>No.</u>	<u>Mole %</u> <u>Total T</u>
1	6.913
2	6.919
3	6.901
4	6.902
5	6.912
6	6.917
7	6.931
8	6.921
9	6.915
10	6.914
11	6.913
12	<u>6.935</u>
Average	6.916
S	±0.0099
RSD	±0.14
Rel. Bias	±0.19

New Container for D-T Standards

A newly designed passivated shipping container was evaluated for protium ingrowth, CT_4 formation and other potential problems. The new container is a double-valved 500 cm³ stainless steel vessel. The volume between the valves is ~0.25 cm³. This feature allows the user to meter the gas out in small aliquots with essentially no waste.

The new container was designed to replace the one-liter, single-valved passivated stainless steel containers currently used for D-T standards. The single-valved design is inherently wasteful. The volume between the valve and the user's sample system is always much larger than the volume of gas needed for an analysis. Most of the gas released from the container is not used and must be discarded.

Two of the newly designed containers were loaded with 99.8% pure T₂ at 275 torr. The contents were analyzed 53 days and 174 days after loading. The data shown in Table 18 show that there are neither protium ingrowth nor CT_4 formation problems. The valves also functioned properly and there were no leaks or other mechanical problems. This design will now be evaluated for smaller vessels, some as small as 10 cm³.

TABLE 18

Evaluation of "Passivated" 500 cm³ Containers for Protium and CT_4 Ingrowth

<u>Day No.</u>	<u>Total H</u>	<u>CT_4</u>
0	0.068%	0.014%
53	0.078	0.021
174	0.074	0.019

COMPARISON OF THE GAZAB AND MAT 250 HDT MASS SPECTROMETERS

Results and Conclusions

This hydrogen isotope mass spectrometer evaluation program established that both the VG-Isotopes GAZAB and the Finnigan MAT 250 HDT prototype mass spectrometers are superior to older instruments used for hydrogen isotope analyses. The results also showed that single-focusing instruments are suitable for routine for routine control laboratory operations. As shown in Table 19, factors that were serious problems in older mass spectrometers have

been addressed successfully. Linearity is very good with both mass spectrometers when they are properly adjusted. Mass discrimination is minimal for the mass optimized. Automatic, computer-controlled refocusing is required and provided with both instruments. With automatic control, linearity and mass discrimination errors are <0.5% with either instrument. Gas interference and trimer formation are not problems, and equilibration, i.e., the production of excess HD in a mixture of H₂ + D₂ is <300 ppm.

TABLE 19

Potential Problems

<u>Problem</u>	<u>GAZAB</u>	<u>MAT 250 HDT</u>
Linearity*	<0.5%	<0.5%
Mass discrimination*	<0.5%	<0.5%
Gas interference	<0.1%	N.D.
Sample equilibration	270 ppm	<300 ppm

* Optimum conditions for each mass

The two prototype mass spectrometers proved to be very competitive as shown in Table 20. The higher resolution and abundance sensitivity of the GAZAB facilitates the analysis of mixtures that are not at equilibrium. At a resolution of 600, the less expensive MAT 250 HDT has an exceptionally high signal-to-noise ratio. It is capable of excellent precision and accuracy for mixtures that are at equilibrium. At a resolution of 1300, it is competitive with the GAZAB for many applications. Both instruments are fully automated and are well adapted to routine analytical work.

In the course of this program, SRL/SRP personnel worked with both manufacturers to resolve problems that developed. Both manufacturers have incorporated the resulting improvements in their commercial instruments, and both now market competitive single-focusing mass spectrometers.

As a direct result of this program, single-focusing mass spectrometers were specified for hydrogen isotopic analyses at SRP and several other DOE sites. To date, a total of 17 single-focusing and one double-focusing hydrogen isotope mass spectrometers have been ordered in the United States, and additional instruments have been sold in Europe.

TABLE 20

Comparative Factors

Factor	GAZAB		MAT 250 HDT	
Resolution	2,000	1300	1300	600*
Abundance sensitivity				
³ He - HD	>100,000	90,000	>40,000	-0.000
HT - D ₂	>100,000	50,000	10,000	--
Low intensity	5 x 10 ⁻¹¹ A	1 x 10 ⁻¹⁰ A	1 x 10 ⁻¹⁰ A	5 x 10 ⁻⁴ A
Signal-to noise ratio	1 x 10 ⁵	2 x 10 ⁵	2 x 10 ⁵	>1 x 10 ⁶
Precision for D/T (2σ)	0.5%	0.5%	0.5%	0.41
Approximate cost	\$600,000		\$195,000	

* Minimum SRP requirements

Test Data Summaries

Tests to compare the performance of the GAZAB and MAT 250 HDT mass spectrometers were made with HD-³He mixtures and with pure HD. The GAZAB was also tested with DT standards. Comparative tests with the MAT 250 GDT were not possible because of the software problem noted in Section 3. Informal tests were made to ensure that the precision with tritium mixtures was the same as that with HD mixtures. Tests with SRP-blended HDT standards were made at GEND with the MAT 251 HDT mass spectrometer operating at a resolution of 1300. The data are included here by courtesy of Norman Parsons, GENA.

Mound Mixtures

All four cylinders of each of the two HD-³He mixtures Mound made for the acceptance testing of the GAZAB mass spectrometer were analyzed with the GAZAB and MAT 250 HDT mass spectrometers in June (Tables 21-24). The four cylinders of Mixture No. 2 were reanalyzed in July (Tables 25 and 26). The between-day relative standard deviations (RSD) for total H in Mixture No. 1 were <0.07% with both instruments. But the between-day RSDs for total D in the June data for Mixture No. 2 were about 0.3%. The July data for Mixture No. 2 show between-day RSD values <0.05%. The improvements in the July data are due to extensive upgrading of the GAZAB in June, and the conversion of the MAT 250 HDT ion source to trap regulation and improved operating techniques.

TABLE 21

Mound Mixture No. 1 Analyzed - June 1982*

Theoretical values
$^3\text{He} = 4.001 \text{ mol } \%$
Total H = 6.008 mol %

Component	Cylinder 1				Cylinder 2			
	GAZAB		MAT 250 HDT		GAZAB		MAT 250 HDT	
	6/4	6/8	6/4	6/8	6/4	6/8	6/4	6/8
Total mol %								
Average of 6								
^3H , mol %	3.970	3.977	3.994	3.993	3.947	3.953	3.964	3.964
SD ₆	±0.0101	±0.0043	±0.0028	±0.0019	±0.0044	±0.0073	±0.0046	±0.0038
RSD ₆ , %	±0.35	±0.11	±0.07	±0.05	±0.11	±0.18	±0.12	±0.10
% Δ Theo	-0.77	-0.59	-0.18	-0.20	-1.34	-1.20	-0.92	-0.92
Total H, mol %	6.053	6.070	6.051	6.034	6.056	6.072	6.054	6.038
SD ₆	±0.0122	±0.0079	±0.0031	±0.0040	±0.0107	±0.0089	±0.041	±0.0020
RSD ₆ , %	±0.20	±0.13	±0.05	±0.07	±0.18	±0.15	±0.07	±0.03
% Δ Theo	+0.75	+0.03	+0.71	+0.43	+0.79	+1.06	+0.76	+0.50
Component	Cylinder 3				Cylinder 4			
Total mol %	GAZAB		MAT 250 HDT		GAZAB		MAT 250 HDT	
Average of 6	6/4	6/8	6/4	6/8	6/4	6/8	6/4	6/8
^3He , mol %	3.946	3.954	3.974	3.971	3.954	3.965	3.986	3.984
SD ₆	±0.0075	±0.0049	±0.0012	±0.0026	±0.0082	±0.0060	±0.0023	±0.0033
RSD ₆ , %	±0.19	±0.12	±0.029	±0.07	±0.21	±0.15	±0.06	±0.08
% Δ Theo	-1.39	-1.17	-0.69	-0.74	-1.17	-0.90	-0.37	-0.41
Total H, mol %	6.052	6.076	6.058	6.044	6.058	6.070	6.060	6.041
SD ₆	±0.0073	±0.0061	±0.0027	±0.0078	±0.0130	±0.0099	±0.0042	±0.0043
RSD ₆ , %	±0.12	±0.10	±0.04	±0.13	±0.21	±0.16	±0.07	±0.07
% Δ Theo	+0.73	+1.14	+0.83	+0.60	+0.83	+1.04	+0.87	+0.55

* Note: No LN₂ was used in traps 6/4 on either instrument. LN₂ was used in both instruments 6/8.

TABLE 22

Mound Mixture No. 1 -- Average of June Analyses

	Cylinder 1		Cylinder 2		Cylinder 3		Cylinder 4	
	GAZAB	MAT	GAZAB	MAT	GAZAB	MAT	GAZAB	MAT
^3He , avg., mol %	3.974	3.994	3.950	3.964	3.950	3.973	3.960	3.985
% Δ Theo	-0.69	-0.18	-1.27	-0.92	-1.27	-0.71	-1.04	-0.40
Total H, avg., mol %	6.062	6.043	6.064	6.064	6.064	6.051	6.064	6.051
% Δ Theo	+0.89	+0.57	+0.93	+0.63	+0.93	+0.72	+0.93	+0.72

TABLE 23

Mound Mixture No. 2 Analyzed -- June 1982*

Theoretical values
$^3\text{He} = 3.924 \text{ mol } \%$
Total D = 6.026 mol %

Component	Cylinder 5						Cylinder 6					
	GAZAB			MAT 250 HDT			GAZAB			MAT 250 HDT		
	6/9	6/10	6/11	6/9	6/10	6/11	6/9	6/10	6/11	6/9	6/10	6/11
<u>^3He</u>												
\bar{x}_o , mol %	3.898	3.888	3.890	3.945	3.958	3.947	3.890	3.887	3.893	3.932	3.937	3.931
σ	± 0.0076	± 0.0082	± 0.0079	± 0.0058	± 0.0123	± 0.0056	± 0.0047	± 0.0055	± 0.0050	± 0.0041	± 0.0042	± 0.0128
RSD, %	± 0.20	± 0.21	± 0.20	± 0.15	± 0.31	± 0.14	± 0.12	± 0.14	± 0.13	± 0.10	± 0.11	± 0.33
% Δ Theo	-0.66	-0.92	0.87	+0.54	+0.87	+0.59	-0.87	-0.94	-0.79	+0.20	+0.33	+0.18
<u>Total D</u>												
\bar{x}_o , mol %	6.028	6.022	6.026	6.050	6.063	6.048	6.024	6.027	6.030	6.044	6.047	6.041
σ	± 0.0074	± 0.0069	± 0.0076	± 0.0026	± 0.0133	± 0.0129	± 0.0049	± 0.0069	± 0.0037	± 0.0037	± 0.0061	± 0.0150
RSD, %	± 0.12	± 0.11	± 0.13	± 0.40	± 0.61	± 0.37	-0.03	+0.02	+0.07	+0.30	+0.35	+0.25
Component	Cylinder 7						Cylinder 8					
	GAZAB			MAT 250 HDT			GAZAB			MAT 250 HDT		
	6/9	6/10	6/11	6/9	6/10	6/11	6/9	6/10	6/11	6/9	6/10	6/11
<u>^3He</u>												
\bar{x}_o , mol %	3.922	3.905	3.913	3.927	3.935	3.926	3.930	3.920	3.925	3.917	3.935	3.923
σ	± 0.0089	± 0.0039	± 0.0043	± 0.0071	± 0.0038	± 0.0039	± 0.0099	± 0.0039	± 0.0032	± 0.0069	± 0.0051	± 0.0084
RSD, %	± 0.23	± 0.08	± 0.11	± 0.18	± 0.10	± 0.10	± 0.25	± 0.10	± 0.08	± 0.18	± 0.13	± 0.21
% Δ Theo	-0.05	-0.48	+0.28	+0.01	+0.28	+0.05	+0.15	-0.10	+0.03	-0.18	+0.28	-0.03
<u>Total D</u>												
\bar{x}_o , mol %	6.067	6.059	6.065	6.034	6.042	6.031	6.054	6.049	6.053	6.007	6.027	6.016
σ	± 0.0063	± 0.0050	± 0.0033	± 0.0116	± 0.0062	± 0.0084	± 0.0101	± 0.0050	± 0.0066	± 0.0075	± 0.0071	± 0.0131
RSD, %	± 0.10	± 0.08	± 0.05	± 0.19	± 0.10	± 0.14	± 0.17	± 0.08	± 0.11	± 0.12	± 0.12	± 0.22
% Δ Theo	+0.68	+0.55	+0.65	+0.13	+0.27	+0.08	± 0.46	± 0.38	± 0.45	-0.32	+0.02	-0.17

* All analyses made with liquid N_2 in traps.

TABLE 24

Mound Mixture No. 2 — Average of June Analyses

	Cylinder 5		Cylinder 6		Cylinder 7		Cylinder 8	
	GAZAB	MAT	GAZAB	MAT	GAZAB	MAT	GAZAB	MAT
^3He , avg., mol %	3.892	3.950	3.890	3.933	3.913	3.929	3.925	3.925
% Δ Theo	-0.82	+0.66	-0.87	+0.24	-0.27	+0.14	+0.03	+0.03
Total D, avg., mol %	6.025	6.054	6.027	6.044	6.064	6.036	6.052	6.017
% Δ Theo	-0.01	+0.46	+0.02	+0.30	+0.63	+0.16	+0.43	+0.15

TABLE 25

Mound Mixture No. 2 — Analyzed July 1982*

Component Total mol % Avg of 6	Cylinder 5						Cylinder 6					
	GAZAB				MAT 250		GAZAB				MAT 250	
	7/13	7/14	7/15	7/16	7/22	7/23	7/13	7/14	7/15	7/16	7/22	7/23
<u>^3He</u>												
\bar{x}_0 , mol %	3.904	3.904	3.904	3.905	3.923	3.925	3.900	3.895	3.890	3.901	3.915	3.917
RSD, %	.07	.08	.08	.09	.12	.08	.17	.07	.08	.05	.13	.11
% Δ Theo	-.51	-.51	-.51	-.48	-.02	+.03	-.61	-.74	-.87	-.59	-.23	+.18
<u>Total D**</u>												
\bar{x}_0 , mol %	6.031	6.037	6.043	6.043	6.031	6.049	6.036	6.037	6.042	6.046	6.035	6.050
RSD, %	.09	.04	.03	.06	.04	.05	.12	.12	.09	.07	.02	.05
% Δ Theo	+.08	+.18	+.28	+.28	+.08	+.38	+.16	+.18	+.26	+.33	+.14	+.40
<u>$^3\text{He}^{**}$</u>												
\bar{x}_0 , mol %	3.908	3.904	3.902	3.909	3.928	3.930	3.916	3.907	3.899	3.909	3.955	3.926
RSD, %	.11	.13	.04	.09	.14	.09	.06	.14	.16	.09	.10	.07
% Δ Theo	-.42	-.51	-.56	-.38	+.10	+.14	-.19	-.43	-.64	-.38	+.02	+.05
<u>Total D**</u>												
\bar{x}_0 , mol %	6.055	6.046	6.058	6.055	6.052	6.067	6.042	6.037	6.035	6.037	6.032	6.041
RSD, %	.04	.11	.08	.07	.08	.04	.04	.07	.03	.06	.05	.11
% Δ Theo	+.48	+.33	-.53	+.48	+.42	+.68	+.26	+.18	+.15	+.18	+.10	+.26

* Each value (7) is the average of 6 analyses in mol %.

** Liquid N_2 was used in both instruments for all analyses.

TABLE 26

Mound Mixture No. 2 — Average of July Analyses

Component Total mol %	Cylinder 5		Cylinder 6		Cylinder 7		Cylinder 8	
	GAZAB	MAT 250	GAZAB	MAT 250	GAZAB	MAT 250	GAZAB	MAT 250
Avg. ³ He	3.904	3.924	3.896	3.916	3.906	3.929	3.908	3.926
% Δ Theo	-.51	0	-.71	-.20	-.46	+.13	-.41	+.05
Total avg. D	6.038	6.040	6.040	6.042	6.054	6.060	6.038	6.036
% Δ Theo	+.20	+.23	+.23	+.26	+.46	+.56	+.20	+.17

The tests with the Mound mixtures confirm that despite identical pure gas calibration techniques, there is a consistent bias in ³He determinations between the GAZAB and the MAT 250 HDT. At the 4 mol % level, ³He analyses with the GAZAB are about 0.5% (relative) below analyses made with the MAT 250 HDT. The reason for the bias is not known at this time.

The June data (Table 22) show that the gas in all four cylinders of Mixture No. 1 is identical. The data for Mixture No. 2 (Table 24) are inconclusive. However, the July data (Table 26) show that the total D content in cylinder 7 is about 0.2% (relative) greater than that in the other three cylinders.

Storage of Pure HD

On August 18, 1978, the SRP Production Department loaded ~250 liters of pure HD (>99.95% HD) into various vessels. This was done to:

- aid in the determination of the $\frac{D^+}{HD}$ cracking pattern
- determine the long-term stability of HD in various types of vessels
- meet future requests for high purity HD and HD mixtures from other DOE sites
- supply HD for the testing of the GAZAB mass spectrometer.

Nineteen months later (March 1980) the HD was analyzed on MS-15, a cycloidal focusing mass spectrometer in Building 232-H. Four years after loading, it was analyzed with the GAZAB and MAT 250 HDT mass spectrometers. The results of these analyses are summarized in Table 27. They show that in passivated vessels, there is essentially no portium ingrowth and the re-equilibration rate ($HD \neq H_2 + D_2$) is very low. The data also indicate that the re-equilibration rate in untreated vessels is some function of the volume-to-surface ratio; the re-equilibration rate in the 50-liter vessels being substantially lower than that in the 500 cm³ vessels.

TABLE 27

HD Analysis (Loaded 8/18/78 - >99.95% HD)

Analysis March 1980 Mol % (MS No. 15-232H)							
	5-Liter Passivated Vessel-Ion Devices		50-Liter PC No. 1C		50-Liter PC No. 1B		Whitey Cylinder 500 cc Mound Lab-Filled Vessel
							Mound - 1/23/78
	SRP						
H ₂	0.17	0.63	1.21	9.70	0.93		
HD	99.77	98.87	97.72	81.46	98.64		
D ₂	0.05	0.49	1.06	8.84	0.41		

Analysis July 1982							
	5-Liter Passivated Vessel-Ion Devices		50-Liter PC No. 1		50-Liter PC No. 1B		Whitey Cylinder 500 cc Mound Lab-Filled Vessel
	GAZAB	MAT 250*	GAZAB	MAT 250*	GAZAB	MAT 250*	GAZAB
H ₂	0.084	0.096	1.19	1.20	2.67	2.69	26.2
HD	99.83	99.81	97.63	97.60	94.66	94.63	47.6
D ₂	0.077	0.096	1.18	1.20	2.67	2.69	26.2

* Calculations of H₂ based on GAZAB analyses which indicate H₂ = D₂. GAZAB resolves D⁺ and H₂. MAT 250 cannot resolve D⁺ and H₂.

HD Cracking Pattern

As shown in Table 28, tests with high purity HD on the MAT 250 HDT and GAZAB mass spectrometers showed that the cracking fraction

$$\frac{D^+}{HD^+}$$

is ~75% of the cracking fraction

$$\frac{D^+}{D_2^+}$$

routinely measured in pure D_2 gas calibrations.

Thus, the historical assumption of equimolar contributions

$$\frac{D^+}{HD^+} = 50\% \text{ of } \frac{D^+}{D_2^+}$$

was shown to be incorrect. This assumption was necessary because neither ultra high purity HD nor high resolution mass spectrometers were available before now. The importance of high resolution is indicated by the change in the D^+/HD^+ value measured with the MAT 250 HDT (resolution ~600) as a function of HD concentration. With the higher resolution of the GAZAB (~1200), the D^+/HD^+ value remains constant.

The GAZAB mass spectrometer was used for a variety of tests with DT standards. Multiple calibrations were made with pure D_2 , pure T_2 , and T-500 (50 mol % D-50 mol % T) standard to obtain comparative sensitivities. The data in Table 29 show sensitivity biases between pure D_2 and D_2 in the mixture, and between pure T_2 and T_2 in the mixture. The fact that the biases have opposite signs suggests a slight error in the theoretical values for the T-500 standard. It is not known why the T_2 bias is double the size of the D_2 bias.

In each of the D_2 and T_2 pure gas analyses listed in Table 29, the $\frac{D^+}{D_2^+}$ and $\frac{T^+}{T_2^+}$ ratios, respectively were measured. The average values obtained were:

$$\frac{D^+}{D_2^+} = 0.00384$$

$$\frac{T^+}{T_2^+} = 0.00289$$

TABLE 28

Summary of HD Cracking Pattern Data (7/22/82)

Sample*	MAT 250 HDT			GAZAB			Vessel
	$\frac{H^+}{HD^+}$	$\frac{D^+}{D^+}$	$\frac{D^+/HD^+}{D^+/D^+} \times 100$	$\frac{D^+}{HD^+}$	$\frac{D^+}{D^+}$	$\frac{D^+/HD^+}{D^+/D^+} \times 100$	
99.8% HD	0.00493	0.00654	75.4%	0.00302	0.00386	78.2%	5-liter SS passivated by ion devices
97.6% HD	0.00450	0.00659	68.3%	0.00294	0.00384	76.6%	50-liter PC No. 1C
94.6% HD	0.0040	0.00643	62.2%	0.00296	0.00382	77.5%	50-liter PC No. 1B

Conditions

Ion source temperature <150°C
 Resolution 600
 Electron energy 85 eV
 Anode to ion source V + 42V
 10 kV 3.6 A cathode
 0.2 mA emission (trap current)

Conditions

Ion source temperature 180°C
 Resolution 1200
 Electron energy 77 eV
 Anode to ion source V ~59V
 Trap current 50 μA
 Total emission 0.62 mA
 Block current 0.38 mA
 repeller + 3.2 V

* Isotopic analyzes of these samples are given in Table 10.

TABLE 29

GAZAB Sensitivities, mV/ μ

Vessel	Pure	Standard T-500 (50% D - 50% T)						Pure gas
		QM #2	WAP #2	QM #2	WAP #2	QM #2	WAP #2	
Species	D ₂	D ₂	D ₂	DT	DT	T ₂	T ₂	T ₂
	158.64	158.43	158.35	157.33	157.36	155.34	155.30	154.22
	158.87	158.81	158.54	157.41	157.34	155.43	155.27	154.43
	159.01	158.44	158.42	157.34	157.00	155.22	155.21	154.53
	158.74	158.51	158.42	157.34	157.22	155.04	155.19	154.56
	158.81	158.46	158.52	157.34	157.32	155.17	155.22	154.47
	158.88	158.46	158.62	157.23	157.35	155.22	155.14	154.35
	159.09	158.63	158.53	157.41	157.30	155.19	155.05	154.42
	158.79	158.54	158.50	157.30	157.26	155.05	154.98	154.16
Avg. sens.	158.85	158.50	158.52	157.34	157.27	155.21	155.17	154.39
RSD	±0.09%	±0.04%	±0.09%	±0.04%	±0.08%	±0.08%	±0.07%	±0.09%
Δ sensitivity (between vessel) (relative)		Δ QM #2 - WAP #2 0.01%		Δ	QM #2 - WAP #2 0.04%		Δ QM #2 - WAP #2 0.02%	
Δ sensitivity* (mix - pure)		-0.22%	-0.21%			+0.53%	+0.50%	

$$* \frac{(\text{Sens. Mix} - \text{Sens. Pure})}{\text{Sens. Pure}} \times 100$$

Multiple runs were made on each vessel containing DT standards T-0702 and T-930 to determine if there were any differences between vessels. The GAZAB was calibrated with standard T-500 for all of these runs. The results in Table 30 indicate that the difference in the total T (T-0702) and total D (T-930) are less than .05% relative. This data and the comparison data in Table 29 for the vessels containing DT standard T-500 show that both the Quantum Mechanics vessels and the Western Air Products vessels are well passivated. Neither type affects the composition of the stored standards.

Multiple analyses of standards T-0700, T-0702, and T-930 were made over a six week period. The results (Tables 31, 32, and 33) show that analyses with the GAZAB are generally within 0.25% (relative) of the theoretical values.

The ability of the GAZAB to distinguish between two samples of nearly the same composition was tested by comparing the data for standards T-0702 and T-0700. As shown in Table 34, the GAZAB reliably detected and measured the ~0.4% relative difference between the two standards.

Norman Parson, GEND, reported that their MAT 251 HDT continues to operate very well. The instrument is set up to operate routinely at a resolution of 1300. T. Mehrhoff, GEND, modified the MAT low resolution (~600) software to operate the instrument at high resolution. As shown in Table 35, the instrument is precise within 0.3% (relative 95% confidence limits) and accurate within 0.5% (relative) for the major components. Reproducible nonlinear effects in the DT measurements were observed in some of the data. Gross differences in sample inlet pressures between individual analyses of standards 12-T-200 and 2-L caused variations in the measured DT values. The differences caused the 0.36% and 0.63% precisions shown for the total D/total T ratio measurements for these standards. When the DT values were corrected for the pressure effect, the precision values for the total D/total T ratio determination fell to 0.28% and 0.25%, respectively.

TABLE 30

GAZAB Tests to Determine DT Differences Between Vessels*

T-930 QM #3 Total D%	10/18/82 QM #4 Total D%	T-930 QM #3 Total D%	10/21/82 QM #4 Total D%	T-0702 WAP #3 Total T%	10/19/82 WAP #4 Total T%
7.050	7.050	7.032	7.040	6.874	6.873
7.046	7.052	7.034	7.040	6.874	6.874
7.052	7.054	7.036	7.037	6.873	6.870
7.048	7.050	7.036	7.043	6.865	6.866
7.048	7.049	7.040	7.036	6.872	6.872
<u>7.046</u>		<u>7.036</u>	<u>7.036</u>	6.870	6.872
7.048 ±0.002	7.051 ±0.002	7.036 ±0.003	7.039 ±0.003	6.868	6.866
RSD ±0.03%	±0.03%	RSD ±0.04%	±0.04%	6.870	6.862
				6.872	6.871
				<u>6.875</u>	
Δ QM #3-QM #4 = 0.04% rel		Δ QM #3-QM #4 = 0.04%		6.871 ±0.003	6.870 ±0.00
				RSD ±0.04%	±0.06%
				ΔWAP #3 - WAP #4 = 0.01%	

* QM - vessels fabricated by Quantum Mechanics.
WAP - vessels fabricated by Western Air Products.

TABLE 31

Analysis of Standard Mixture SRP T-0700 by GAZAB

Date	Total Mol %		H ₂	D	T	Theoretical Δ*
	³ He	Theoretical ³ He				
9-3-82	0.122	0.131	0.082	92.881	6.906	+0.27%
9-3-82	0.122	0.132	0.082	92.886	6.894	+0.10%
					6.900	
9-9-82	0.134	0.144	0.083	92.874	6.894	+0.19%
9-9-82	0.134	0.143	0.082	92.891	6.890	+0.14%
9-9-82	0.134	0.144	0.086	92.868	6.889	+0.12%
					6.891	
9-14-82	0.145	0.155	0.084	92.877	6.885	+0.14%
9-14-82	0.145	0.156	0.084	92.875	6.883	+0.12%
9-14-82	0.145	0.156	0.082	92.874	6.887	+0.17%
9-14-82	0.145	0.155	0.084	92.876	6.876	+0.01%
					6.883	
9-15-82	0.147	0.157	0.080	92.878	6.884	+0.17%
9-15-82	0.147	0.154	0.082	92.884	6.882	+0.13%
9-15-82	0.147	0.157	0.081	92.880	6.882	+0.13%
9-15-82	0.147	0.157	0.081	92.880	6.882	+0.13%
					6.854	+0.15%

* Difference from theoretical T value.

TABLE 32

Analysis of Standard Mixture SRP T-0702 by GAZAB

Date	Total Mol %		H ₂	D	T	Δ T-0700**	Theoretical Δ*
	³ He	Theoretical ³ He					
9-3-82	0.150	0.139	0.090	92.830	6.930	+0.45%	+0.23%
9-3-82	0.150	0.139	0.090	92.825	6.932		+0.26%
					6.931		
9-9-82	0.162	0.152	0.091	92.874	6.894		+0.19%
9-9-82	0.157	0.152	0.090	92.891	6.890		+0.14%
9-9-82	0.157	0.152	0.090	92.868	6.924		+0.12%
9-9-82	0.161	0.152	0.091	92.826	6.917		+0.15%
					6.918	+0.39%	
9-14-82	0.174	0.162	0.091	92.822	6.909		+0.11%
9-14-82	0.172	0.162	0.091	92.828	6.910		+0.13
9-14-82	0.172	0.162	0.090	92.823	6.910		+0.13%
9-14-82	0.172	0.162	0.090	92.826	6.909		+0.11%
					6.910	+0.40%	
9-15-82	0.175	0.165	0.086	92.827	6.907		+0.10%
9-15-82	0.176	0.165	0.087	92.827	6.907		+0.10%
9-15-82	0.174	0.165	0.086	92.826	6.910		+0.15%
9-15-82	0.176	0.165	0.087	92.825	6.910		+0.15%
					6.908	+0.37%	
10-13-82	0.236	0.224	0.090	92.792	6.884		+0.23%
10-13-82	0.234	0.224	0.090	92.798	6.882		+0.20%
10-13-82	0.232	0.224	0.091	92.805	6.878		+0.14%
10-13-82	0.234	0.224	0.089	92.799	6.882		+0.20%
					6.882	+0.41%	
10-19-82	10 runs		Each vessel		6.871		+0.14%
							+0.16%

* Average vessel WAP #3 - WAP #4.

** Theoretical Δ 0.44% relative between T-0700 and T-0702.

† Δ = difference from theoretical T value.

TABLE 33

Analysis of Standard Mixture SRP T-930 by GAZAB

Date*	Total Mol %		H ₂	D	T	Δ	Theoretical
	³ He	Theoretical ³ He					
9-3-82	1.490	1.498	0.55	7.084	91.378	D ₂	+0.12%
9-3-82	1.484	1.498		7.082			+0.09%
9-9-82	1.674	1.665		7.094			+0.35%
9-9-82	1.679	1.665		7.095			+0.36%
9-9-82	1.688	1.665		7.109			+0.26%
9-9-82	1.684	1.665		7.096			+0.37%
9-14-82	1.817	1.804		7.080			+0.22%
9-14-82	1.815	1.804		7.078			+0.19
9-14-82	1.819	1.804		7.083			+0.26%
9-14-82	1.820	1.804		7.079			+0.20%
9-15-82	1.850	1.832		7.080			+0.23%
9-15-82	1.851	1.832		7.084			+0.28%
9-15-82	1.849	1.832		7.082			+0.25%
9-15-82	1.852	1.832		7.080			+0.23%
10-13-82	2.626	2.604		7.053			+0.24%
10-13-82	2.622	2.604		7.054			+0.26%
10-13-82	2.631	2.604		7.060			+0.34%
10-13-82	2.638	2.604		7.058			+0.31%
10-18-82	2.764	2.742		7.050	avg. 12 runs		+0.27%
10-21-82	2.847	2.824		7.038	avg. 12 runs		+0.14%
					Average		+0.26%

* Average vessel QM #3 - QM #4.

TABLE 34

Detection of Small Differences

<u>Date</u>	<u>Total T, Mol %</u>		<u>ΔT, Mol %</u>	<u>ΔT, Rel %</u>
	<u>T-0702</u>	<u>T-0700</u>	<u>(T-0702)-(T-0700)</u>	
9/3/82	6.931	6.900	0.031	0.45%
9/9/82	6.918	6.891	0.027	0.39%
9/14/82	6.910	6.883	0.027	0.39%
9/15/82	6.908	6.883	0.025	0.36%
10/13/82	6.882	6.854	<u>0.028</u>	<u>0.41%</u>
		$\bar{\Delta}$	0.028	0.40% ± 0.04 (RSD)
	Theoretical	Δ	0.031	0.44%

TABLE 35

Summary of MAT 251 HDT (GEND) Data — Resolution 1300*

Standard	Total H	Total D	Total T	D/T Precision**
ST 330	35.49 mol %	35.44 mol %	29.07 mol %	0.27%
Precision**	±0.22%	±0.12%	±0.24%	
Rel bias †	±0.17%	0.00%	-0.07%	
3-MR=1274-02	0.28	60.11	39.61	0.17%
Precision	±5%	±0.09%	±0.14%	
Rel bias	+100%	+0.12%	-0.50%	
12-T-900	0.07	11.16	88.77	0.35%
	±37%	±0.36%	±0.05%	
		(±0.28%)††		
	+133%	+2.2%	-0.30%	
2-L	0.32	92.77	6.91	0.63
Precision	±0.9%	±0.04%	±0.63%	
			(±0.35%)††	0.25%††
Rel bias	+220%	-0.26%	+0.29%	
10-11-T-300	0.05	72.00	29.75	0.21%
Precision	±15%	±0.08%	±0.19%	
Rel bias	-150%	+0.17%	-0.30%	

* GEND data courtesy of N. H. Parsons

** Precision = relative 95% confidence limits for a single determination

†

$$\text{Relative bias} = \frac{(\text{observed mol \%} - \text{theoretical mol \%})}{\text{theoretical mol \%}} \times 100$$

†† Corrected for nonlinearity

REFERENCES

1. W. M. Jones. "Thermodynamic Functions for Tritium Deuteride. The Dissociation of Tritium Particles. Equilibrium Among the Isotopic Hydrogen Molecules." *J. Chem. Phys.* 17, 1062 (1949).
2. R. Schott and G. Beau. "Quantitative Analysis of Hydrogen Isotopes by Mass Spectrometry." *Method. Phys. Anal.* 7, 165 (1971).
3. P. Chastagner. Mass Spectrometer for Accurate Hydrogen Isotopic Analyses. USDOE Report DP-1492, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, SC (1978).
4. R. B. Ferguson and P. Chastagner. "High Resolution Mass Spectrometer for Accurate Analysis of Mixtures of Hydrogen and Helium Isotopes." *Int. J. Mass Spectrom. Ion Phys.* 24, 403 (1977).

APPENDIX I

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APPENDIX II

MSTG SPECIFICATIONS FOR GAZAB MASS SPECTROMETER

A. GENERAL DESCRIPTION

1. The instrument should have an overall mass range of at least m/e 1-600. The resolution should be 2000 at mass 4 and the abundance sensitivity for the $H_2^+ - D^+$ doublet should be at least 40,000. It should be easily adjustable to a stable resolution of at least 3000 at mass 28.
2. After a pure gas standardization, the instrument should have an overall capability of determining the total amount of an H-isotope present at the 6% level in a 2 std cc sample of a mixture (regardless of isotopic species) and a 15:1 ratio of two major components (e.g., total D \approx 90% and a total H \approx 5%) according to the tests described in the Performance Tests (C.I.k.). The normal sample analysis rate should not be less than six samples per hour.
3. The mass spectrometer should be constructed entirely of metal with metal-gasketed joints (except as specified) and be bakeable to at least 250°C. Standard UHV joints compatible with Varian and Ultek joints are performed.
4. The high voltage distribution system will be designed to operate continuously at altitudes of up to 7600 feet above sea level.

B. DETAILED SPECIFICATIONS

1. Sample Inlet System. The main purpose of this inlet system is for demonstrating the performance specifications. The inlet system will be all stainless steel and will be bakeable to at least 150°C. In normal operation the inlet system will be maintained at a temperature of about 40°C and will be held constant within $\pm 0.1^\circ C$. It will consist of a sample inlet volume equipped with a 0-200 torr bourdon tube gage, (or equivalent), an expansion volume (reservoir) a molecular leak connected to the ionization chamber of the source, a bypass valve around the molecular leak, an acceptable capacitance type manometer such as the MKS 145 AH-1 (whose chamber is included in the temperature controlled system), a bypass valve around the capacitance manometer, an ionization pump to maintain the reference side of the capacitance manometer at a pressure

$<1 \times 10^{-7}$ torr, a polyphenyl ether diffusion pump with water, air, freon, or thermoelectrically cooled baffles, molecular seive, optically dense foreline trap, or an adequate forepump and an isolation valve between the forepump and diffusion pump. The inlet system will essentially be as shown in the attached sketch.

The conductance of the gold foil molecular leak (easily interchangeable) will be less than 0.2 cc/sec for nitrogen gas.

All valves will be constructed to provide minimum surface area in contact with the sample and for ease of maintenance. When possible, the valves will be mounted with their seats facing the active volume as shown in the sketch.

No device that allows mercury to contact the sample gas may be used.

2. Ion Source. The ion source is the key element in the mass spectrometric analysis of mixtures of the H-isotopes.
 - a. The ion source will be a heated, electron bombardment, short focal length, high-brightness type.
 - b. The accelerating voltage will be at least 8,000 V.
 - c. The source materials must be chosen to minimize memory and to facilitate cleaning and maintenance.

Nichrome V is the preferred material for the metal parts and aluminum oxide is preferred for the insulators.

- d. The filaments will be self-aligning.

The source will function correctly with either tungsten or rhenium filament material. (See Item 6.a.).

- e. The ion source region will be differentially pumped with respect to the analyzer.
 - f. An adequate isolation valve is required between the ion source housing and the analyzer to permit the source to be vented for filament changing and repair without venting the analyzer.
 - g. Z axis focusing plates will be provided after the ion source exit slit plate to optimize transmission from the ion source through the analyzer.

- h. The ion source temperature will be variable from ambient temperature (with the filament on) to at least 250°C and will be regulated within $\pm 0.2^\circ\text{C}$.
- i. The ionizing voltage will be precisely adjustable over a range of at least 10 to 150 V.
- j. The ionizing current will be trap regulated and will be adjustable over a range of at least 10 to 300 μA .
- k. The ion source controls will be simple and the settings must be precisely reproducible.
- l. The ion source exit slit(s), if any, will be externally adjustable (precisely) without breaking the vacuum.

3. Analyzer. The analyzer must have high transmission and sharp focus for good resolution and abundance sensitivity. The magnet should be a low-impedance high-current electromagnet.

The analyzer will be all metal and will be bakeable to at least 150°C.

Any realignment of the analyzer magnet (if moved during the bakeout procedure) must be simple and precise.

4. Ion Collector(s)

- a. The principal ion collector will be a Faraday cage type with a reliable, effective secondary electron suppression system.
- b. The collector slit will be adjustable (precisely) from outside the vacuum system over a range of about 500 μm to less than 2 μm .
- c. The collector slit or the ion beam will be rotatable (precisely) from outside the vacuum system.
- d. An electron multiplier ion detector will be provided.
- e. A total ion monitor collector will be provided.

5. Vacuum System

a. Pressure Measurement

Ionization gages will be located in the high vacuum pumping lines to monitor the pressure in all parts of the system that are equipped with diffusion pumps and they must not

contribute to background spectra in the m/e 2 to 44 range. Pirani gages will be provided in all fore pressure and roughing lines.

Isolation valves will be used between diffusion pumps and forepumps.

b. Source Housing

The ion source housing will be maintained at a pressure less than 1×10^{-7} torr at all times during operation. It will be pumped by a diffusion pump with polyphenyl ether fluid and will have a liquid nitrogen trap with a minimum holding time of 12 hours. A viton-sealed and gasketed isolation valve will be provided between the LN₂ trap and the source housing. An isolation valve will be used between the diffusion pump and the forepump. Differential pumping is required between the source housing and the analyzer to prevent degradation of the pressure in the analyzer.

c. Analyzer

The pressure in the analyzer will be maintained less than 2×10^{-8} torr at all times. The analyzer will be pumped between the source housing and the magnetic analyzer and between the magnetic analyzer and the intermediate slit and between the intermediate slit and the electrostatic analyzer. Polyphenyl ether filled diffusion pumps with adequate trapping and isolation valves between diffusion pumps and forepumps will be used.

d. Mechanical Pumps

The mechanical forepumps will be quiet running, have readily available parts, and require minimum maintenance. The forepumps will be isolated by adequate foreline traps to prevent the forepump oil from backstreaming into the system. These pumps will also be equipped with automatic safety valves to isolate them during power failures or whenever isolation is desired.

e. The sample inlet system will have a separate pumping system similar to the source housing pumping system.

f. Protection System

An adequate protection system will be provided to protect all components in the event of failure of power, water (or other collants), or vacuum.

6. Electronics

All electronic supplies and controls will be of all-solid state design.

a. Emission Regulator

The emission regulator will regulate the trap current of the electron bombardment ion source within $\pm 0.05\%$. The trap current range will be at least 10 to 300 μA with precise settings and readout. The ionizing potential will be precisely adjustable over the range of at least 10 to 150 V and will be stable within ± 0.1 V. The regulator will operate satisfactorily with all normally used filament materials including, but not limited to, tungsten and rhenium.

b. High Voltage Supply

The high voltage supply to the ESA supply will be precisely adjustable over a minimum range of 500 to 10,000 V and will be stable within $\pm 0.002\%$ (20 min.) at 8 kV accelerating voltage. Manual and repetitive sweep modes at several different sweep speeds will be provided. The unit will be capable of being controlled by a computer (analog signals 10 V maximum). The supply and all associated high voltage wiring and control circuitry will be suitable for continuous operation at altitudes 7,600 feet above sea level.

c. Magnet Power Supply and Control

The magnet power supply and control unit will be stable within $\pm 0.0005\%$ (2 min. at m/e 2 and above). The unit will be precisely field controlled with a Hall probe. It will be resettable within ± 20 m gauss. Manual and bidirectional sweep modes at several different sweep speeds will be capable of being controlled by a computer (analog signals 10 V maximum).

d. Amplifier

The amplifier will have a dynamic range of at least $5 \times 10^5:1$, a response time of 0.3 seconds or less, a noise level less than 2×10^{-15} A, and be linear within 0.1% over the entire range (with a 1×10 -inch ohm resistor). The unit will have two outputs; one for a recorder, and the other for a DVM or a computer.

C. PERFORMANCE TESTS

The instrument will be tested with hydrogen and helium and mixtures of their isotopes to demonstrate satisfactory low mass performance. Testing will be limited to the nonradioactive isotopes. The Faraday cage collector will be used for all of the testing except where the electron multiplier is specified.

The instrument will be tested for satisfactory high mass performance with nitrogen and other inorganic gases either singly or in mixtures.

1. Performance Tests with Hydrogen and Helium Isotopes

- a. The source will be adjusted with pure D_2 and pure H_2 for a resolution of at least 2000 (10% valley definition) with flat-topped peaks and this setting will be used for all of the performance testing (hydrogen and helium). No manual readjustment will be necessary over the $m/e = 2-44$ mass range (see N_2 sensitivity test below).

When the instrument is calibrated with pure D_2 gas, the H_2 and HD sensitivities* should agree with the D_2 sensitivity within 0.5% without manual readjustment to the ion source. Sensitivities will not require redetermination more than once every eight hours.

When the ion source is adjusted for optimum performance with the H isotopes, the sensitivity for N_2 will not be more than 20% less during automatic scans than when the ion source is adjusted for optimum performance with N_2 gas.

- b. Absolute sensitivity of the instrument will be tested with pure D_2 gas flowing into the ion source. The ion intensity measured at the Faraday cage ion collector for D_2^+ will be not less than 3×10^{-11} A when the D_2 gas flow into the ion source is 2.5×10^{-5} atm-cc/sec.
- c. The abundance sensitivity will be at least 40,000 for the $H_2^+-D^+$ doublet at mass 2 and for the $HT^+-D_2^+$ doublet at mass 4.

The abundance sensitivity at mass 2 will be demonstrated with appropriate D_2-H_2 mixtures at a pressure that produces a 1×10^{-10} A H_2^+ ion beam at the ion collector. The height of the tail of the H_2^+ ion peak under the D^+ ion peak will not be more than one part in 40,000 of the height of the H_2^+ ion peak. With the same sample, at the same time, the height of the tail of the D_2^+ ion peak at the

* Sensitivity here (and elsewhere when not qualified) means output current per \therefore pressure in the inlet system.

calculated peak HT^+ mass position will not be more than one part in 40,000 of the height of the D_2^+ ion peak.

- d. The absence of memory will be demonstrated by introducing a sample of pure D_2 into the system at a pressure that yields a mass 4 ion beam of 1×10^{-10} A. The sample will then be pumped out for ten minutes. At the end of the ten minute period, an equal pressure of pure H_2 will be put into the system. The peak at mass 4 will not be larger than 3×10^{-15} A (30 ppm of the previous D_2^+ ion peak) and the HD^+ ion peak at mass 3 will be no larger than that attributable to the pure H_2 gas.
- e. The background of the instrument will be determined by pumping out the inlet system and the ion source overnight so the pressure in the ion source housing is 2×10^{-8} torr or less, and measuring the residual ion peaks in the mass 2-44 range with the inlet system valved off. A series of 10 analyses, such as those required for the precision and accuracy test, will be made and the ion source and sample system will be pumped out for a period of ten minutes, and the background will again be measured. No background ion peak will show an increase under these conditions of more than 100 ppm of the major peak when a sample from the series of 10 is flowing into the ion source.
- f. The absence of gas interference will be demonstrated by flowing pure D_2 gas into the ion source at a rate of approximately 1×10^{-5} atm-cc/sec. along with an Ar leak of similar proportions. The gas flows will be obtained from Sandia standard leaks. The D_2^+ ion current will be monitored for another ten minute period. The D_2^+ ion outputs for the two periods will agree within 0.1%.
- g. Trimer formation will be measured by observing the D_3^+ mass position during the abundance sensitivity test. The trimer formation at mass 6 should be less than 0.005% of the D_2^+ ion peak.
- h. Linearity will be measured by determining the sensitivity for pure D_2 (also for N_2 , O_2 , and Xe) over a pressure range of 15 to 100 μ in the inlet system. All of the sensitivities will agree within $\pm 0.15\%$.
- i. Satisfactory sensitivity for trace impurities will be demonstrated by measuring the ^4He content of air in a sample of not more than 100 μ behind the standard leak using the electron multiplier ion detector.

j. The absence of gas equilibration will be demonstrated by monitoring the mass 3 HD⁺ peak while a freshly-mixed 50% H₂ - 50% D₂ sample is flowing into the ion source. There should not be any observable increase in the HD⁺ peak. The height of the HD⁺ peak should not increase more than 100 ppm of the total signal.

k. Precision and Accuracy

The precision and accuracy will be demonstrated by analyzing two mixtures supplied by the contractor, consisting of a nominal 90.0 mol % D, 6.0 mol % H, and 4.0 mol % ³He and 6.0 mol % D, 90.0 mol % H and 4.0 mol % ³He, respectively. The H and D will be at or near equilibrium. The instrument will be calibrated with pure gases, and ten consecutive analyses of each standard mixture will be made at a rate of at least six analyses per hour (six separate introductions of sample gas into the inlet system). The major component's ion beam intensity (from the mixtures and calibration gases) will be made at a rate of at least six analyses per hour (six separate introductions of sample gas into the inlet system). The major component's ion beam intensity (from the mixtures and calibration gases) will be at least 5×10^{-11} A at the collector. Sixteen hours or more later, the instrument will be recalibrated with the pure gases and another set of ten analyses of each standard mixture will be made. The total H, D, and ³He contents will be calculated for each analysis. The following steps will be done:

1. For each set of ten analyses the sample mean and standard deviation will be calculated for each component. The mean for each set is given by:

$$\bar{X}_j = \frac{\sum_{i=1}^n X_{ji}}{n} \quad j = 1, 2$$

where \bar{X}_j is the sample mean of the jth set. X_{ji} are the individual measures of the component and n is the number of observations (10). The standard deviation is given by:

$$s_j = \sqrt{\frac{\sum_{i=1}^n X_{ji} - \bar{X}_j^2}{n-1}}$$

where s_j is the sample standard deviation of the jth set.

- Each of the sets of 10 analyses will be tested for outlying observations.

$$\frac{|X_{\text{ex}j} - \bar{X}_j|}{s_j} > 2.29$$

Where $X_{\text{ex}j}$ is the extreme observation of the j th set (high or low), the observation will be discarded.

In discarding an observation, the entire analysis will be discarded. Not more than one "outlier" will be allowed in each set of 10 analyses.

If an observation is discarded in a set, a new sample mean and standard deviation will be calculated based upon the remaining nine observations.

- The remaining data will be pooled to give a mean and standard deviation for the test. The pooled mean is:

$$\bar{X}_p = \frac{n_1 \bar{X}_1 + n_2 \bar{X}_2}{n_1 + n_2}$$

where \bar{X}_p is the pooled mean, n_1 and n_2 are the number of observations remaining (9 or 10) after possible outlier discarding and \bar{X}_1 and \bar{X}_2 are the sample means again after possible discarding.

The pooled standard deviation is:

$$s_p = \sqrt{\frac{(n_1 - 1) s_1^2 + (n_2 - 1) s_2^2}{n_1 + n_2 - 2}}$$

where s_p is the pooled standard deviation and s_1 and s_2 are the sample standard deviations after possible discarding of outliers.

- An upper confidence for the population standard deviation will be calculated by:

$$U = K s_p$$

where U is the upper bound for the population standard deviation and K is a value from the following table:

$n_1 + n_2$	K
20	4.153
19	4.200
18	4.253

The ratio U/\bar{X}_p must be less than or equal to the relative precision limits given in Table II-1. Thus, for example, in mixture No. 1, $U/\bar{X}_p < .002$ or 0.20% for total H.

TABLE II-1

	<u>Component</u>	<u>Nominal Concentration</u>	<u>Maximum Precision Limit (Relative)</u>
Mixture No. 1	Total H	6.0 mol %	±0.20% (±0.012 mol %)
	Total D	90.0 mol %	±0.04% (±0.036 mol %)
	³ He	4.0 mol %	±0.40% (±0.016 mol %)
Mixture No. 2	Total H	90.0 mol %	±0.04% (±0.036 mol %)
	Total D	6.0 mol %	±0.20% (±0.012 mol %)
	³ He	4.0 mol %	±0.40% (±0.016 mol %)

5. Using the values, as supplied by the contractor for the mixtures, the sample means for each component must satisfy the following:

$$\frac{|\bar{X}_j - KC|}{KC} < .005 \quad j = 1, 2$$

where KC is the specified concentrations for the components.

In addition, for each component the following must also be satisfied:

$$\frac{|\bar{X}_1 - \bar{X}_2|}{\bar{X}_p} < .005$$

Approximate equilibrium distribution of test mixture
No. 1

Total D	90.0 mol %
Total H	6.0 mol %
³ He	4.0 mol %
D ₂	84.4 mol %
HD	11.13 mol %
H ₂	0.45 mol %

Approximate equilibrium distribution of test mixture
No. 2

Total D	6.0 mol %
Total H	90.0 mol %
³ He	4.0 mol %
D ₂	0.45 mol %
HD	11.13 mol %
H ₂	84.4 mol %

2. Higher Mass Performance Tests

- a. The source will be adjusted with a nominal 50% N₂, 50% CO gas mixture for a resolution of at least 3000 (10% valley definition) at mass 28 with an N₂⁺ ion beam intensity of at least 1×10^{-10} A at the collector. This setting will be used for all of the higher mass performance testing.
- b. The abundance sensitivity will be demonstrated with normal Xe gas at a pressure in the inlet system that produces a 3×10^{-11} A or larger ¹³⁴Xe ion intensity at the collector. The combined contribution of ¹³²Xe and ¹³⁴Xe at the mass 133 position will not be greater than one part in 10⁶ of the height of the ¹³⁴Xe ion peak and the combined contribution of ¹³⁴Xe and ¹³⁶Xe at the mass 135 position will not be greater than one part in 10⁶ of the height of the ¹³⁴Xe ion peak.
- c. The precision and accuracy will be demonstrated by analyzing 10 successive samples of pure natural Xe gas at a rate of at least four analyses per hour (four separate introductions of Xe gas into the inlet system). The data will be treated as in Section C.1.k (above) and the values obtained will be equal to or less than the values in Table II-2.

TABLE II-2

<u>Isotope</u>	<u>Natural Abundance, mol, %</u>	<u>Maximum Precision Limits, Relative, %</u>	<u>Maximum Deviation of Average from Known Abundance, Relative, %</u>
^{124}Xe	0.096	± 1.0	± 1
^{126}Xe	0.090	± 1.0	± 1
^{128}Xe	1.919	± 0.25	± 0.5
^{129}Xe	26.44	± 0.12	± 0.3
^{130}Xe	4.08	± 0.20	± 0.4
^{131}Xe	21.18	± 0.13	± 0.3
^{132}Xe	26.89	± 0.12	± 0.3
^{134}Xe	10.44	± 0.17	± 0.3
^{136}Xe	8.87	± 0.18	± 0.4

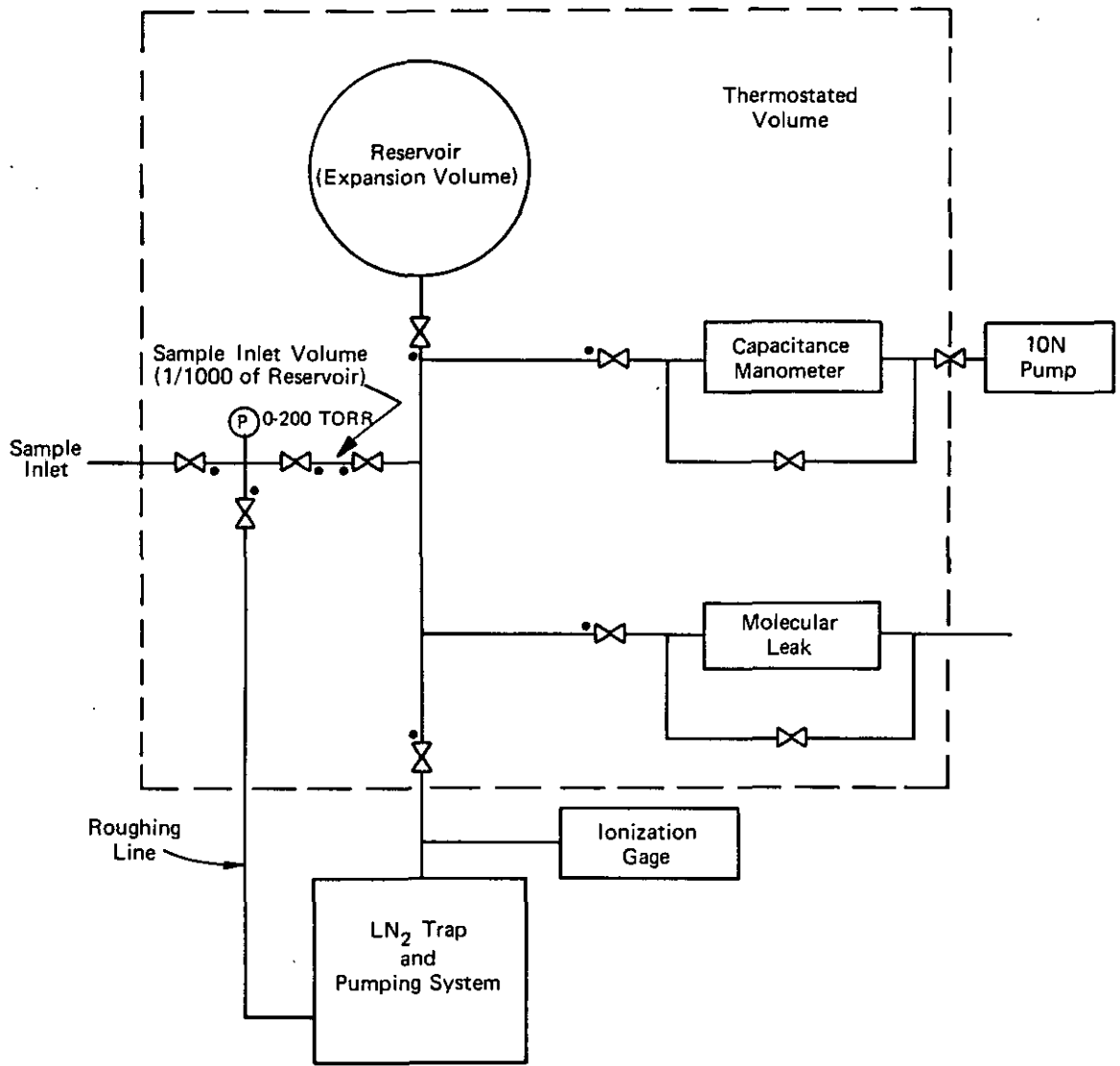


FIGURE II-1. Sketch of Sample Inlet System

APPENDIX III

MEMORANDUM (JANUARY 6, 1981) FROM J. W. GUTHRIE (SNLA) TO
G. L. THOMPSON (SNLA PURCHASING)

Sandia Laboratories

Albuquerque, New Mexico
Livermore, California

date: January 6, 1981

to: G. L. Thompson, 3713

J. W. Guthrie
from: J. W. Guthrie, 1551

Coordinator, DOE Mass Spectrometer Development Program

subject: Contract 13-9152 with VG Isotopes Ltd., England via
The Kearns Group, Gaithersburg, Maryland

Evaluation by DOE's Savannah River Facility of the latest (December 1980) test information from VG Isotopes, Ltd. has resulted in the following recommendations concerning contract 13-9152.

1. Waive the right to again witness specification tests in England (witnessed by W. B. Hess, SRP in April and October 1980).
2. Authorize shipment of the mass spectrometer system to the Savannah River Facility as per contract 13-9152. (Reference TWX dated 1-6-81 to G. L. Thompson and J. W. Guthrie, SNLA, from J. Beatty and W. B. Hess, SRP).
3. Specify March 1, 1981 as the date for completion of installation and acceptance testing at the Savannah River Facility.
4. Proceed at the Savannah River Facility with final acceptance tests using specification SLA-XMS-8-79-SR dated November 1, 1979 (as per contract 13-9152) with the following specification changes as authorized by the Savannah River Facility. (Reference, TWX dated 12-18-80 to G. L. Thompson and J. W. Guthrie, SNLA, from J. Beatty and W. B. Hess, SRP).
 - a. Item C.1.d (page 9)

The absence of memory will be demonstrated by introducing a sample of pure D_2 (from the inlet system) into the MS at a pressure that yields a mass 4 ion beam of 1×10^{-10} A. The D_2 flow will be stopped (close inlet valve) and replaced by an equal flow of pure H_2 (from the flow standard) for 10 minutes. After the ten minute period the peak at mass 4 will not be larger than 3×10^{-15} A (30 ppm of the previous D_2^+ ion peak) and the HD^+ ion peak at mass 3 will be no larger than that attributable to the pure H_2 gas.

b. Item C.1.e (page 10)

The background of the instrument will be determined by pumping out the inlet system and the ion source overnight so the pressure in the ion source housing is 2×10^{-8} torr or less and measuring the residual ion peaks in the mass 2-44 range with the inlet system valved off. A series of 10 analyses, such as those required for the precision and accuracy test, will be made and the ion source and sample system will be pumped out for a period of ten minutes, and the background will again be measured. No background ion peak (excluding waters) will show an increase under these conditions of more than 100 ppm of the major peak when a sample from the series of 10 is flowing into the ion source.

c. Item C.1.j (page 11)

The absence of gas equilibration will be demonstrated by monitoring the mass 3 HD⁺ peak from D₂ (inlet system) and H₂ (flow standard) flowing into the ion source. The HD⁺ peak will not exceed 600 ppm more than that attributable from the H₂ and D₂ gases.

d. Item C.2.a (page 17)

The tests in C.2.b and C.2.c will be run at the same resolution as the hydrogen tests in C.1.

e. Item C.2.b (page 17)

The abundance sensitivity will be demonstrated with normal Xe gas at a pressure in the inlet system that produces a 3×10^{-11} A or larger ¹³⁴Xe ion intensity at the collector. The combined contribution of ¹³²Xe and ¹³⁴Xe at the mass 133 position will not be greater than 1 part in 10^4 of the height of the ¹³⁴Xe ion peak and the combined contribution of ¹³⁴Xe and ¹³⁶Xe at the mass 135 position will not be greater than 1 part in 10^4 of the height of the ¹³⁴Xe ion peak.

f. Item C.2.c (page 17)

The precision and accuracy will be demonstrated by analyzing 10 successive samples of pure natural Xe gas at a rate of at least 4 analyses per hour. For the set of 10 analyses the sample mean and standard deviation will be calculated for each Xe isotope. The data will be treated as in Section C, pages 13 and 14 (Items 1 and 2). The f sigma (Table II for f values) precision and accuracy (mean for the set) obtained will be equal to or less than the values in Table II.

TABLE II

<u>Isotope</u>	<u>Natural Abundance (mol %)</u>	<u>Maximum Precision Limits (Relative)</u>	<u>Maximum Deviation of Average from Known Abundance (Relative)</u>
^{124}Xe	0.096	$\pm 1.0\%$	$\pm 1\%$
^{126}Xe	0.090	$\pm 1.0\%$	$\pm 1\%$
^{128}Xe	1.919	$\pm 0.25\%$	$\pm 0.5\%$
^{129}Xe	26.44	$\pm 0.12\%$	$\pm 0.3\%$
^{130}Xe	4.08	$\pm 0.20\%$	$\pm 0.4\%$
^{131}Xe	21.18	$\pm 0.13\%$	$\pm 0.3\%$
^{132}Xe	26.89	$\pm 0.12\%$	$\pm 0.3\%$
^{134}Xe	10.44	$\pm 0.17\%$	$\pm 0.3\%$
^{136}Xe	8.87	$\pm 0.18\%$	$\pm 0.4\%$

Values of f for P = 0.05

<u>Degrees of Freedom</u>	<u>f Values</u>
7	2.365
8	2.306
9	2.262
10	2.228
11	2.201
12	2.179

APPENDIX IV

ACCEPTANCE TESTS OF THE MSTG GAZAB, FEBRUARY 1981



E. I. DU PONT DE NEMOURS & COMPANY

ATOMIC ENERGY DIVISION

SAVANNAH RIVER PLANT
AIKEN, SOUTH CAROLINA 29801

(TEL: 803-731-6570, TEL: 803-732-6311, WET: 803-676, 662)

March 4, 1981

J. W. Guthrie, Coordinator
DOE Mass Spectrometer Development Group
Sandia National Laboratories, Albuquerque
Albuquerque, NM

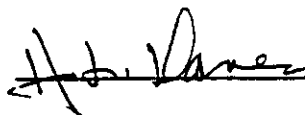
ACCEPTANCE OF THE GAZAB MASS SPECTROMETER

The VG Instruments, Inc. GAZAB Mass Spectrometer was delivered to Savannah River Laboratory February 2, 1981. Installation of the GAZAB has now been completed, having met, without exception, all requirements of the specification SLA-XMS-8-79-SR as amended December, 1980 and as required by the Sandia Contract 13-9152.

All contract materials have been delivered with the following exceptions:

- 1) Remaining flow and mixture standards (presently located at Kennedy Airport, New York)
- 2) Remaining portions of the 8 sets of operating and service manuals and appropriate drawings.

Attached is a copy of the VG Instruments Customer Acceptance Paper.

 3/4/81

 3/4/81

SRP Representatives
E. I. du Pont

VG Isotopes Limited, Ion Path, Road Three, Winsford, Cheshire CW7 3BX. Telephone Winsford (06065) 51121 Telex 669429.

CUSTOMER ACCEPTANCE

Invoice to: VG Instruments Incorporated, 300 Broad Street, Stamford, Ct. 06902, U.S.A.			Ship to: A.T. Labiento, J.F.K. for onward shipment to:- Savannah River Laboratories, Aiken, South Carolina, U.S.A.	
VG Order No. S2538			Customer Order No. 13-9152	
System Type MMZAB			System Serial No. -	
Item	Quantity		Description	Serial No.
	Ordered	Shipped		
1	1	1	MMZAB Mass Spectrometer	
<p>Except as otherwise noted the systems listed above, in accordance with the order numbers, have been installed and satisfactorily completed and acceptance tests, as noted by notation on the order, have been passed.</p> <p>VG Reference No. _____ Date of Acceptance <u>Feb 23, 1971</u> _____ Customer's Representative</p>				

BEST AVAILABLE COPY



MICROMASS
THE MASS SPECTROMETRY COMPANY
VG-Isotopes Limited



GAZAB PERFORMANCE DATA

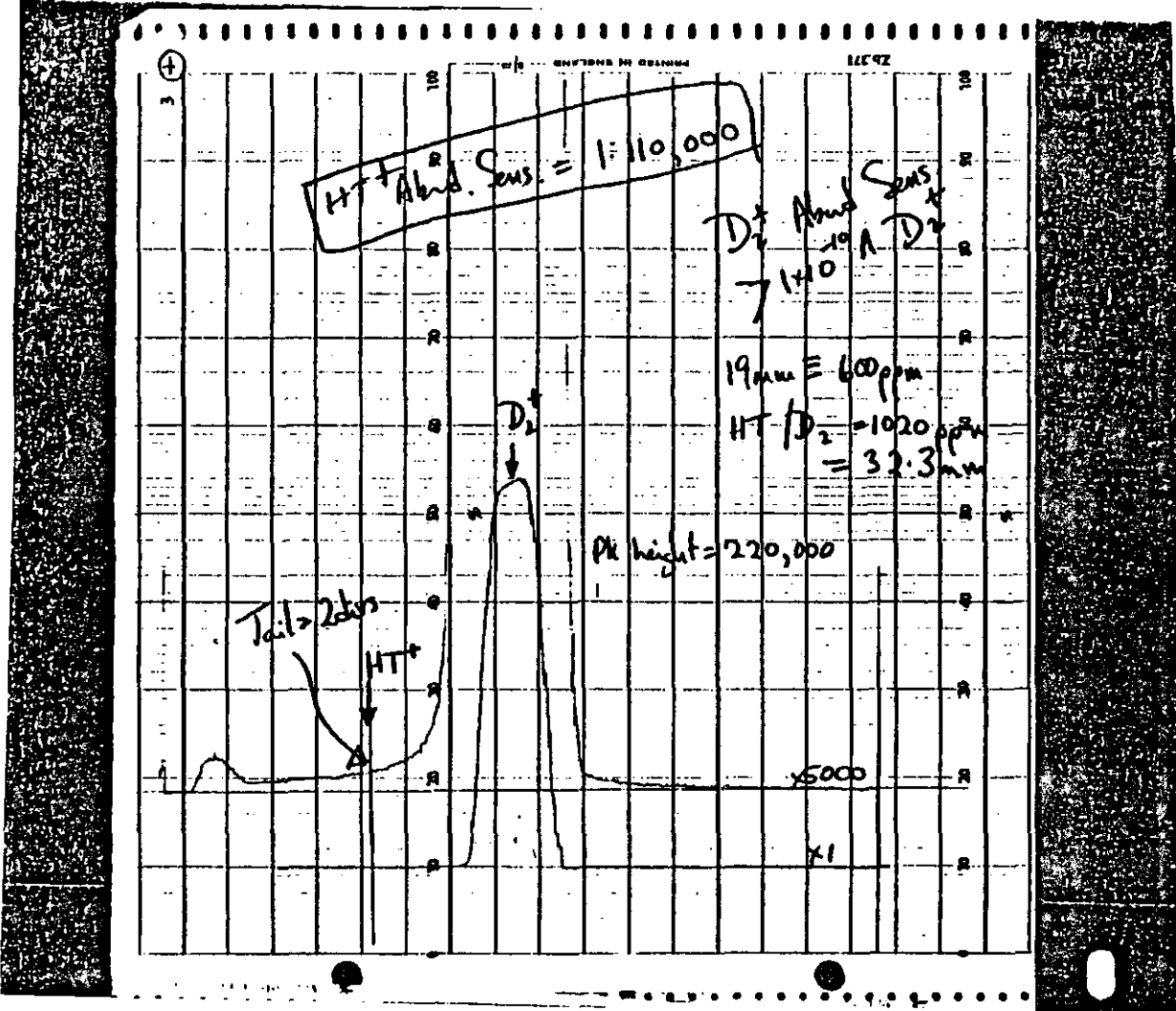
February 1981

**E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, S. C. 29801**

GAZAB CHECKOUT

MASS DISCRIMINATION - ABSOLUTE SENSITIVITY

		<u>Sensitivity $10^{11}\Omega$</u>
H ₂	Purity 99.97%	111.02 mv/ μ
HD	Purity 99.7%	111.06 mv/ μ
D ₂	Purity 99.3%	110.92 mv/ μ



2/20/81

Memory.

D₂ from st. leak.

- 1) D₂ bled in from st. leak 6.5×10^{-8} A. for 4 mins.
- 2) Stopped flow for 10 mins. & pumped source to 7×10^{-9} Torr.
- 3) Placed 65 um H₂ in reservoir and allowed to flow into source : Analysis below :
 HD at natural level
 No observable D₂ memory using Faraday detector.

Peak no. 1

Peak no. 1 ZERO
pk# = 1908.25 Deviation = 41.1235622

Peak no. 2 H-2
peak at 281830 hw = 50 profile(.01%) 11 5 0 0 2 0 1 3 6 5 5
y,z1 = 814687.0072 1908.25

Peak no. 3 HE-3
peak zero or lost
y,z1 = 1908.25 1908.25

Peak no. 4 HD
peak at 332320 hw = 61 profile(.01%) 19 31 48 51 36 75 33 0 71 46 77
y,z1 = 2202.342045 1908.25

Peak no. 5 D-2
peak zero or lost
y,z1 = 1908.25 1908.25

Peak	mf	height	time	Persec	comp
H-2	281830	812558	63.7	78.130	99.963
HE-3	332038	0	0.0	0.000	0.000
HD	332320	294	114.7	0.029	0.037
D-2	374929	0	0.0	0.000	0.000

Memory

- 1) D_2 leak with multiplier $1.92 \times 10^{-7} A$.
For ~ 2 mins.
- 2) Closed CRT 7 Valve and pumped source
for 10 mins.
- 3) After 10 min D_2 ion current on multiplier was

After 4 mins.	$0.6 \times 10^{-10} A$.
6 mins.	$0.2 \times 10^{-10} A$.
8 mins.	$0.06 \times 10^{-10} A$.
10 mins	$0.025 \times 10^{-10} A$.

H_2 put \implies $0.026 \times 10^{-10} A$
in at $55 \mu m$.

Memory \Rightarrow mass. 4 current = $2.6 \times 10^{-12} Amp$.

D_2^+ beam = 1.92×10^{-7} .

$$\text{Memory in ppm} = \frac{2.6 \times 10^{-12}}{1.92 \times 10^{-7}} \times 10^6 = \frac{2.6 \times 10^{-6}}{1.92 \times 10^{-7}}$$

$$= 13.5 \text{ ppm of } D_2 \text{ signal}$$

spec. is 30 ppm of D_2 signal.

2/24/81

Background

Inlet and source housing pumped overnight
 Inlet pressure 3.5×10^{-3} mbar. Source housing

D₂ ion beam with 91 μm in 30 liter
 = 5×10^7 Amp in FA3.
 $100 \mu\text{m} = 5 \times 10^7$ Amp.
 7×10^9 Torr.

Background peaks

Ion current FA3

Ion currents after 10 align
 criterion = $\pm 5 \times 10^{-10}$ A.

2	1.03×10^{-10} A	
3	3×10^{-13} A	
4	not detectable n.d.	
12	3.5×10^{-12} A	1.6×10^{-12} A ✓
13	1.6×10^{-12} A	n.d.
14	1.42×10^{-10} A	1.15×10^{-10} A ✓
15	2.61×10^{-10} A	1.7×10^{-10} A ✓
16	2.56×10^{-10} A	5.2×10^{-10} A ✓
17	2.73×10^{-10} A	2.73×10^{-10} A ✓
18	1.903×10^{-10} A	1.39×10^{-10} A ✓
19	1.74×10^{-10} A	1.74×10^{-10} A ✓
20	5.0×10^{-12} A	4.5×10^{-12} A ✓
21	n.d.	n.d.
22	1.3×10^{-12} A	1.27×10^{-12} A ✓
23	n.d.	n.d.
24	3×10^{-13} A	n.d.
25	25×10^{-12} A	3.6×10^{-12} A ✓
26	1.6×10^{-10} A	1.23×10^{-10} A ✓
27	3.6×10^{-10} A	2.5×10^{-10} A ✓
28	3.39×10^{-10} A	3.26×10^{-10} A ✓
29	2.84×10^{-10} A	2.40×10^{-10} A ✓
30	1.34×10^{-10} A	1.37×10^{-10} A ✓
31	1.2×10^{-10} A	1.3×10^{-10} A ✓
32	2.37×10^{-10} A	2.62×10^{-10} A ✓
33	4.4×10^{-12} A	6.16×10^{-12} A ✓
34	1.6×10^{-12} A	3.4×10^{-12} A ✓
35	2.9×10^{-12} A	4.1×10^{-12} A ✓
36	2.6×10^{-12} A	2.5×10^{-12} A ✓
37	4.0×10^{-12} A	1.0×10^{-12} A ✓
38	1.6×10^{-10} A	1.7×10^{-10} A ✓

2 ppm
 26 ppm
 23 ppm
 24 ppm

10 ⊕

2/24/31

Mass No.

FAS Ion Current

After 10 elyasts

39
40
41
42
43
44

1.00×10^{10} A
 2.24×10^{10} A
 3.43×10^{10} A
 5.37×10^{10} A
 1.69×10^{10} A
 1.44×10^{10} A

9.4×10^{11} A
 2.5×10^{11} A
 2.1×10^{10} A ✓
 5.07×10^{11} A ✓
 1.56×10^{10} A ✓
 1.35×10^{10} A ✓

Deviation range from -0.084324660 to 0.055692615 is = 0.140017275

Peak at 331695 hw= 61 profile(.01%)### 96 7 0 3 5 16 28 19 28 58
 R= 1 bar= 46.31 peak= 148447.618

Peak at 331698 hw= 64 profile(.01%)### 95 73 31 1 0 6 8 41 54###
 R= 1 bar= 46.06 peak= 147496.9581

Peak at 331695 hw= 58 profile(.01%) 21 11 12 10 2 0 0 4 9 24 38
 R= 1 bar= 45.82 peak= 146769.4059

Peak at 331701 hw= 60 profile(.01%) 4 0 0 0 25 48 50#####
 R= 1 bar= 45.58 peak= 145995.3374

3/1/82
 Interference of F. Du
 on D₂

set no. 1	IR= 1		mV/micron	deviation (percent)
paratron (corr)	integrator (corr)			
46.310	148448		32.055	0.063
46.060	147497		32.023	-0.038
45.820	146769		32.032	-0.010
45.580	145995		32.031	-0.014

mean= 32.0350721

Deviation range from -0.038348128 to 0.062821966 is = 0.101170094

Peak at 331699 hw= 60 profile(.01%) 37 16 15 9 0 1 9 41 70 41 91
 R= 1 bar= 44.63 peak= 142814.7298

Peak at 331696 hw= 61 profile(.01%) 53 30 9 2 0 0 26 54 65 87###
 R= 1 bar= 44.4 peak= 142065.2747

Peak at 331687 hw= 61 profile(.01%)### 60 26 5 4 0 4 15 26 47 41
 R= 1 bar= 44.16 peak= 141304.2575

Peak at 331694 hw= 66 profile(.01%)##### 92 29 12 0 9 9 0 26
 R= 1 bar= 43.93 peak= 140652.1141

D₂ = 0.7 x 10⁻¹¹ amp

set no. 1	IR= 1		mV/micron	deviation (percent)
paratron (corr)	integrator (corr)			
46.310	148448		32.055	0.113
46.060	147497		32.023	0.012
45.820	146769		32.032	0.040
45.580	145995		32.031	0.036
44.630	142815		32.000	-0.060
44.400	142065		31.997	-0.070
44.160	141304		31.996	-0.065
43.930	140652		32.017	-0.065

with D₂ flow stop
 +0.050
 -0.050
 0.1%
 w/o D₂

mean= 32.01903253

Deviation range from -0.069735256 to 0.1129

2/5/81

Interference Argon on D₂.

Repeller optimum, D Focus optimum

Repeller + 0.5 volts (indicated in notes), D Focus 10/900

Argon ion current 1.16×10^{-10} Amp.
D₂ ion current $3-4 \times 10^{-10}$ Amp.

D Focus	10/900	(1.30 pm)	Interference +0.5% (+ve with D ₂ only)
D Focus	10/1000	"	-0.8% (-ve with D ₂ only)
D Focus	10/950	"	-0.3% (-ve : ")
D Focus	10/900	(Repeat of 1.30 pm) at 3.11 pm	+0.5% (+ve with D ₂ only)
D Focus	10/925	3.2 pm	+0.2% (+ve with D ₂ only)
D Focus	10/925	Repeat 3.36	-0.1% (-ve - -)
D Focus	10/925	3.56	-0.1% (-ve - -)

GAS INTERFERENCE



peak
IR= 1 bar= 104.11 peak= 358869.9186

set no. 1 baratron (corr)	IR= 1 integrator (corr)	mV/micron	deviation (percent)
35.760	375749	105.355	0.000
35.620	375398	105.390	0.114
35.490	373653	105.284	0.013
35.350	372250	105.304	0.032
35.210	370691	105.290	0.009
34.640	364478	105.219	-0.049
34.500	363001	105.218	-0.050
34.370	361659	105.225	-0.043
34.240	360267	105.218	-0.049
34.110	358870	105.210	-0.058

2/17/81
D₂ Interference
opt. repeller
+ Absc D Flow 10/925
105.32 ~ 0.1%
D₂ only
105.22

mean= 105.270264

Deviation range from -0.057579779 to 0.113580217 is= 0.171159996

peak at 375395 hw= 70 profile(.01%) 7 2 0 2 3 17 42 69 89#####
mean= 105.1768755

Deviation range from -0.158923449 to 0.113580217

peak at 375382 hw= 72 profile(.01%) 6 0 1 0 0 3 3 13 16 17 44
IR= 1 bar= 100.1 peak= 1054537.002

peak at 375382 hw= 68 profile(.01%) 4 2 0 1 4 8 19 31 63 81###
IR= 1 bar= 99.79 peak= 1052029.693

8kV, 50.0H trap

D₂ Linearity
Optimum repeller
D Flow 10/925

set no. 1 baratron (corr)	IR= 1 integrator (corr)	mV/micron	deviation (percent)
19.300	202905	105.132	-0.195
19.240	202284	105.137	-0.1
19.280	513723	105.463	0.119
19.120	510050	105.466	0.122
#####	1E+06	105.375	0.008
99.720	1E+06	105.451	0.108

+ .11 - .19
15/2/81

mean= 105.3374859

Deviation range from -0.112576655 to 0.092335113 is= 0.204911768

peak at 331698 hw= 55 profile(.01%)\$\$\$\$ 28 7 0 9 26 48 42 49 55
 IR= 1 bar= 38.47 peak= 123357.9139

peak at 331697 hw= 65 profile(.01%) 40 19 15 0 4 5 15 31 62\$\$\$\$
 IR= 1 bar= 38.3 peak= 122809.94

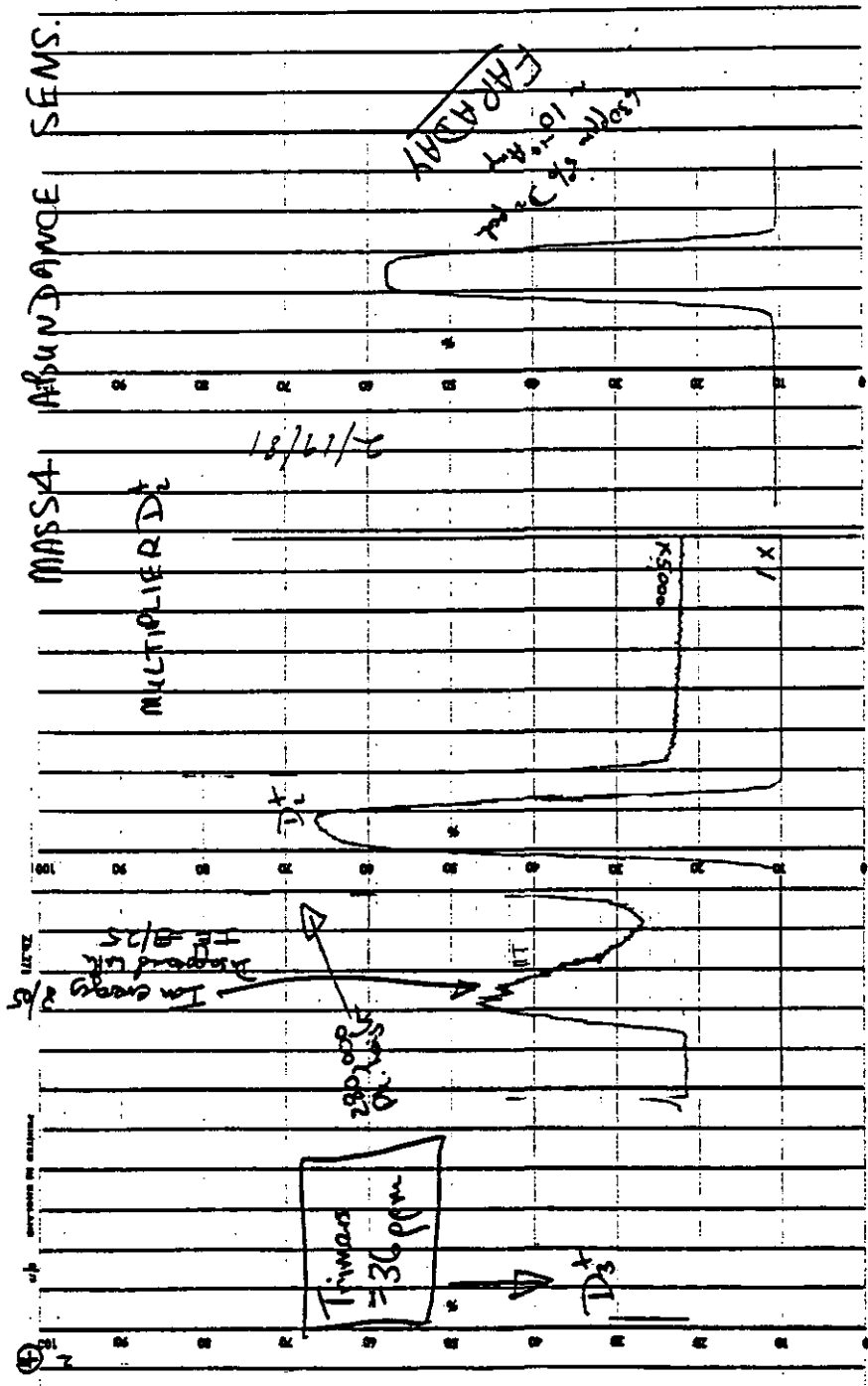
*3/1/81 Interference of
 D₂ on ³He.
 Repeat.*

set no. 1	IR= 1			
baratron	integrator	mV/micron	deviation	(percent)
(corr)	(corr)			
40.980	131280	32.035	0.049	
40.760	130584	32.037	0.056	
40.550	129798	32.009	-0.031	
40.330	129204	32.037	0.054	
39.820	127311	31.972	-0.143	
39.610	126725	31.993	-0.082	
39.410	126003	31.972	-0.146	
39.190	125431	32.006	-0.042	
38.470	123358	32.066	0.146	
38.300	122810	32.065	0.144	

*With D₂ flow std.
 +0.03
 ±0.02
 0.05%
 w/o D₂ flow std.*

mean= 32.01924857

Deviation range from -0.148750219 to 0.146020045 is= 0.294770294



Equilibration

2/20/81

60

11.30 a.m.

H₂ sens. 105.88 mV/μm

HD level

H₂ pk shifted down in mass by ~100 digits
HD moved down by 80 dig

0.045%
0.039%

D₂ sens. 105.19 mV/μm previous day 1/1/81 0.72%
2/1/81 0.732%

Analysis 49.919% H₂

0.411 HD

After lunch 0.428

49.670% D₂

Expected HD level is 0.384%

$$\text{excess} = 0.411 - 0.384 = 0.027\%$$

$$\approx \underline{270 \text{ ppm XS}}$$

$$\text{SPEC} = 600 \text{ PPM}$$

Peak no. 3 HE-3
 peak at 331986 hu= 12 profile(.01%) 20 44 69 0 55 95 99 35 70 63 33
 y,zl= 1922.875873 1890.35

Peak no. 4 HD
 peak at 332261 hu= 59 profile(.01%) 59 20 75 41 30 0 31 25 66 31 11
 y,zl= 2106.600408 1890.25

Peak no. 5 D-2
 peak at 374916 hu= 46 profile(.01%) 2 22 0 67 65 38 52 46 7 22 31
 y,zl= 1903.500638 1890.25

Peak	af	height	time	Ppress	comp%
H-2	281772	559440	72.9	53.884	99.938
HE-3	331986	33	181.8	0.011	0.021
HD	332261	216	139.3	0.021	0.039
D-2	374916	13	160.2	0.001	0.002

Total= 53.918 at time zero

CLOSURE= 1.064028260
 Baratron= 57.37 at time zero (zero corrected)
 Measured= 53.92 corresponding to time zero
 Baratron zero= 0.09
 Pressure= 59.86

Peak no. 1 ZERO
 Sk= 1807.75 Deviation= 11.59446329

Peak no. 2 H-2
 peak at 281722 hu= 48 profile(.01%) 36 0 16 12 0 1 7
 y,zl= 4278.977599 1887.75

Peak no. 3 HE-3
 peak at 332007 hu= 72 profile(.01%) 0 30 39 48 67 39 27 15 40 17 5
 y,zl= 1899 1887.75

Peak no. 4 HD
 peak at 332298 hu= 64 profile(.01%) 77 59 43 19 4 13 26 4 10 0
 y,zl= 6392.709536 1887.75

Peak no. 5 D-2
 peak at 374932 hu= 70 profile(.01%) 13 3 0 0 1 3 4 8 13 21 31
 y,zl= 603666.7698 1887.75

Peak	af	height	time	Ppress	comp%
H-2	281722	2170	63.8	0.209	0.352
HE-3	332007	11	92.1	0.004	0.006
HD	332298	4505	120.4	0.437	0.738
D-2	374932	601779	148.7	58.629	98.904

Total= 59.279 at time zero

CLOSURE= 0.991421564
 Baratron= 59.77 at time zero (zero corrected)
 Measured= 59.28 corresponding to time zero
 factor= 1.00744 se= 10519.50520

*0.38% HD.
 ✓ 0.42*

Peak no. 1 ZERO
 Sk= 1809.75 Deviation= 21.63111794

Peak no. 2 H-2
 peak at 281800 hu= 46 profile(.01%) 12 9 4 1 0 5 3 3 7 22 38
 y,zl= 618302.8462 1889.75

Peak no. 3 HE-3
 peak zero or lost
 y,zl= 1889.75 1889.75

Peak no. 4 HD
 peak at 332300 hu= 62 profile(.01%) 29 24 28 0 7 11 5 5 29 55
 y,zl= 6927.150064 1889.75

Peak no. 5 D-2
 peak at 374920 hu= 70 profile(.01%) 15 0 4 0 2 0 1 0 3 12 0
 y,zl= 607806.7723 1889.75

Peak	af	height	time	Ppress	comp%
H-2	281800	616192	57.5	59.180	49.919
HE-3	332020	0	0.0	0.000	0.000
HD	332300	5037	181.0	0.480	0.411
D-2	374920	605917	129.7	58.884	49.670

Total= 118.552 at time zero

CLOSURE= 0.495730966
 Baratron= 58.77 at time zero (zero corrected)
 Measured= 55.55 corresponding to time zero

CLOSURE=
Baratron zero= 0.09
Pressure= 57.4

Peak no. 1 ZERO
Bk= 1887.5 Deviation= 16.34496542

Peak no. 2 H-2
peak at 281833 hu= 51 profile(.01%) 18 49 42 9 2 1 2 0 2 + 6
yzl= 601435.5594 1887.5

Peak no. 3 HE-3
peak zero or lost
zl= 1887.5 1887.5

Peak no. 4 HD
peak at 332324 hu= 60 profile(.01%) 72 15 13 40 38 23 21 6 0 26 24
yzl= 2157.622074 1887.5

Peak no. 5 D-2
peak at 374953 hu= 81 profile(.01%) 2 21 0 8 28 83 76#####
yzl= 1947.512213 1887.5

Equil. 2/20/81

1/2 H₂ + 1/2 D₂

HD = 0.411

Peak	nf	height	time	Press	comp%
H-2	281833	599327	63.8	57.628	99.944
HE-3	332828	0	0.0	0.000	0.000
HD	332324	270	115.0	0.026	0.045
D-2	374953	60	151.8	0.006	0.010

Total= 57.660 at time zero

CLOSURE= 0.993925419
Baratron= 57.31 at time zero (zero corrected)
Measured= 57.66 corresponding to time zero

factor= 1.00055 se= 10580.25384

Baratron zero= 0.1
Pressure= 57.47

Peak no. 1 ZERO
Bk= 1892.5 Deviation= 21.28008706

Peak no. 2 H-2
peak zero or lost
yzl= 1892.5 1892.5

Peak no. 3 HE-3
peak zero or lost
yzl= 1892.5 1892.5

Peak no. 4 HD
peak no. 2 HD

Peak no. 1 ZERO
Bk= 1890.25 Deviation= 11.26298736

Peak no. 2 H-2
peak zero or lost
yzl= 1890.25 1890.25

Peak no. 3 HE-3
nf=1, hu=2, ir=3, yd=4, hp=5, bc=6, zd=7, fo=8, se=9, bs=11, 10+1200
2 2 150 11

Peak no. 1 HE-3
peak zero or lost
yzl= 1890.25 1890.25

Peak no. 2 H-2
peak at 281784 hu= 53 profile(.01%)##### 0 0 71#####
yzl= 578564.315 1890.25

Peak no. 3 HE-3
peak zero or lost
yzl= 1890.25 1890.25

Peak no. 4 HD
nf=1, hu=2, ir=3, yd=4, hp=5, bc=6, zd=7, fo=8, se=9, bs=11, 10+1200
2 3 50 11
2 1 281800 11

Peak no. 1 HD
peak at 270000 hu= 0 profile(.01%) 5 79 21 36 0 0 0 36 26 21 21
yzl= 1899 1890.25

Peak no. 2 H-2
peak at 281772 hu= 50 profile(.01%) 20 0 1 0 5 10 21 36 54 32###
yzl= 561551.2582 1890.25

2/27/81 15 ⊕

Mixt. #3 Analysis. Calibration 4.30 p.m.
Original Inlet technique

i.e. Single expansion of 3cc vol. into 3 litre vol. $\sim 30\mu\text{m gas}$

	H ₂	³ He	HD	D ₂	Total H	Total D	closure
#1	0.449	3.990	11.159	84.452	6.0285	90.022	-0.18%
#2	0.448	3.908	11.161	84.443	6.0285	90.024	-0.20%
#3	0.449	3.947	11.161	84.443	6.0295	90.024	-0.26%
#4	0.448	3.949	11.166	84.437	6.031	90.020	-0.36%
#5	0.448	3.942	11.165	84.445	6.0305	90.028	-0.29%
#6	0.449	3.942	11.162	84.447	6.030	90.028	-0.18%
#7	0.449	3.942	11.166	84.443	6.032	90.026	-0.26%
#8	0.447	3.948	11.171	84.434	6.0325	90.020	-0.29%
#9	0.449	3.950	11.160	84.441	6.029	90.021	-0.27%
#10	0.448	3.947	11.159	84.447	6.0275	90.021	-0.33%

Mean
4.28%
Spec.

3.446
0.003597
0.38
0.40

6.030
0.001619
0.110%
0.20

Peak data on file No. 11

No.	name	mf	hw	lc	vd	hc	sc	sd	fo	area	det. rate	os
1	ZERO	270000	0	1	150	210	0	150	1	0.0	0.000000	0
2	H-2	281439	53	1	150	210	0	150	1	1102.0	0.000126	285
3	HE-3	331597	55	1	100	210	150	150	1	8136.0	0.000152	0
4	HD	331866	62	1	290	210	150	150	1	1106.0	0.000152	0
5	D-2	374453	70	1	295	210	450	150	1	11042.0	0.000132	0

LOW MASS I.E. IS 700
HIGH MASS I.E. IS 700

Trap SOLA, 8KV
FILAMENT 2.7

10:56 AM

2/28/81

H₂, He₃, HD, D₂ wire RM
For Sens. Check

Peak data on file No. 11

No.	name	mf	hw	ir	vd	hp	bc	zd	fo	sens	dep. rate	of
1	ZERO	270000	0	1	100	210	0	100	1	0.0	0.000000	0
2	H-2	281460	52	1	100	210	0	100	1	11077.0	0.000187	235
3	HE-3	331597	55	1	100	210	150	100	1	3117.0	0.000152	0
4	HD	331866	62	1	290	210	150	100	1	11077.0	0.000152	0
5	D-2	374453	70	1	295	210	400	100	1	11100.0	0.000132	0

LOW MASS I.E. IS 700
HIGH MASS I.E. IS 700

860

Mass Discrim. O.K.

Mix # 1 Cyl # 3

Started 11:00 AM

	H ₂	He ₃	HD	D ₂	TOTAL H ₂	TOTAL D ₂	closure %
#1	.453	3.966	11.186	84.395	6.046	89.988	-.27
2	.453	3.961	11.188	84.397	6.047	89.991	-.31
3	.452	3.966	11.194	84.388	6.049	89.985	-.34
4	.454	3.961	11.188	84.397	6.048	89.991	-.32
5	.451	3.964	11.193	84.391	6.048	89.988	-.46
6	.453	3.963	11.186	84.399	6.046	89.992	-.45
7	.452	3.962	11.187	84.397	6.046	89.992	-.38
8	.453	3.962	11.190	84.394	6.048	89.989	-.51
9	.452	3.963	11.190	84.395	6.047	89.990	-.54
10	.452	3.961	11.197	84.390	6.050	89.988	-.58

* NOTE - Accuracy - A NOMINAL 4 MOL% MIXTURE WAS blended at SRP and verified that Mix # 1 ³He and total H values are in error.

Mean
4.28
Spec.
10

3.9629
.30%
.40
.048

6.0475
.099%
.20
.022

	³ He	TOT H
STD Val	4.001	6.008
OBS	3.954	6.039
%DIF	-1.2%	+0.5%

Mix #1 Pooled Data:
Pooled ± .30
Spec. ± .40

Pooled ± 0.10
Spec. ± 0.20

BEST AVAILABLE COPY

Baratron zero= 0.08
Pressure= 53.01

MIX 1 Cyl #3
(TYPICAL SCANS)

2/27/80

Peak no. 1 ZERO
E₁= 93.5 Deviation= 20.0515136

6.05

Peak no. 2 H-2
peak at 281454 hw= 47 profile(.01%) 51 22 0 25 54 31 9
y₂₁= 2944.283988 93.5

Peak no. 3 HE-3
peak at 331619 hw= 64 profile(.01%) 11 0 7 9 1 33 36
y₃₁= 6560.996332 93.5

Peak no. 4 HD
peak at 331889 hw= 55 profile(.01%) 35 30 0 0 2 17 91
y₄₁= 64673.6578 93.5

Peak no. 5 D-2
peak at 374459 hw= 66 profile(.01%) 0 0 3 6 11 16 29 48
y₅₁= 487552.1195 93.5

Peak	mf	height	time	press	corr%
H-2	281454	2616	57.2	0.238	0.449
HE-3	331619	6467	85.8	2.089	3.948
HD	331889	64580	114.4	5.317	11.159
D-2	374459	487459	143.0	14.781	34.452

Total= 53.025 at time zero

CLOSURE= 0.998200737

Baratron= 52.93 at time zero (zero corrected)
Measured= 53.03 correct using time zero

Baratron zero= 0.09
Pressure= 51.04

Peak no. 1 ZERO
E₁= 89 Deviation= 19.81387074

Peak no. 2 H-2
peak at 281411 hw= 55 profile(.01%) 79 55 36 84 44 0
y₂₁= 2838.274283 89

Peak no. 3 HE-3
peak at 331536 hw= 55 profile(.01%) 1 0 4 31 7
y₃₁= 6327.960944 89

Peak no. 4 HD
peak at 331866 hw= 65 profile(.01%) 40 18 10 11 2 0
y₄₁= 62283.59915 89

Peak no. 5 D-2
peak at 374459 hw= 68 profile(.01%) 41 13 11 5 1 3 0 1 5 5 17
y₅₁= 469390.2643 89

Peak	mf	height	time	press	corr%
H-2	281441	3514	57.0	0.239	0.448
HE-3	331598	6239	85.6	2.015	3.948
HD	331866	62195	114.1	5.699	11.161
D-2	374459	469381	143.7	48.111	24.443

Total= 51.054 at time zero

CLOSURE= 0.997953096

Baratron= 50.95 at time zero (zero corrected)
Measured= 51.05 correct using time zero

PRECISION + ACCURACY DATA

16 ⊕

2/27/81 contd. 2/27/81 7.30 pm - 8.30 pm.

Mix #2 Cyl #7. Started ~ 7.15 p.m. using 4.30 pm calibration
Original Inlet Technique

i.e. single expansion of 3cc vol. into 3 litre res.

	H ₂	³ He	HD	D ₂	Total H	Total D	Closure
#1	84.483	3.908	11.153	0.456	90.060	6.0325	-0.08%
#2	84.482	3.910	11.143	0.456	90.063	6.0275	-0.08%
#3	84.482	3.914	11.148	0.457	90.056	6.0310	-0.10%
#4	84.479	3.915	11.149	0.456	90.053	6.0305	-0.13%
#5	84.482	3.908	11.154	0.455	90.059	6.0320	+0.03%
#6	84.487	3.910	11.148	0.455	90.061	6.0290	-0.12%
#7	84.482	3.909	11.152	0.457	90.058	6.0330	-0.12%
#8	84.480	3.908	11.155	0.456	90.058	6.0335	-0.23%
#9	84.498	3.910	11.136	0.456	90.066	* 6.0240 *	-0.23%
#10	84.470	3.913	11.158	0.458	90.049	* 6.0370 *	-0.19%
Mean		3.911				6.031	
4σ		0.28				0.25	
Spec		0.40				0.20	
SD		0.002593				0.003576	

PRECISION + ACCURACY DATA

2/28/81

Mix # 2 Cyl # 7

	H ₂	³ He	HD	D ₂	Total H	Total D	Change
#1	84.474	3.919	11.154	0.453		6.030	-0.38%
#2	84.474	3.921	11.153	0.453		6.0295	-0.56%
3	84.468	3.923	11.156	0.454		6.032	-0.67
4	84.465	3.929	11.153	0.453		6.0295	-0.54
5	84.475	3.915	11.157	0.453		6.0315	-0.49
6	84.477	3.917	11.153	0.453		6.0295	-0.37
7	84.479	3.924	11.143	0.454		6.0255	-0.32
8	84.479	3.921	11.147	0.453		6.0265	-0.38
9	84.474	3.920	11.153	0.453		6.0295	-0.39
10	84.475	3.920	11.152	0.453		6.0290	-0.32
Mean		3.921				6.029	
4-σ		0.41				0.137	
Spec.		0.40				0.30	
SD		0.003872				0.001986	

Mix # 2 - POOLED DATA

Pooled	± 0.35%	± 0.20
Spec	± 0.40%	± 0.20

	ACCURACY ³ He	TOTAL D
STD VAL	3.924	6.026
OBS	3.916	6.030
% DIFF	-0.2%	+0.07
Spec	± 0.5%	± 0.5%

Baratron zero= 0.08
Pressure= 55.62

Peak no. 1 ZERO
Bkg= 93.25 Deviation= 16.54944744

Peak no. 2 H-2
peak at 281452 hw= 47 profile(.01%) 0 0 2 6 3 4 3 4 3 2 13
v.zl= 515191.0026 93.25

Peak no. 3 HE-3
peak at 331616 hw= 50 profile(.01%) 47 33 0 16 10 13 4 20 36 55.11
v.zl= 6818.070584 93.25

Peak no. 4 HD
peak at 331888 hw= 59 profile(.01%) 11 24 20 13 1 0 19 28
v.zl= 67760.34650 93.25

Peak no. 5 D-2
peak at 374478 hw= 68 profile(.01%) 95 7 17 11 10
v.zl= 2852.374348 93.25

MIX #2 Cy/#7 2/27/81
(TYPICAL SCANS) 7.1 Sp. Run on MIX 2

Peak	wt	height	time	Pressure	comp%	#1
H-2	281452	515063	57.7	46.961	84.453	
HE-3	331616	6725	85.6	2.172	3.908	
HD	331888	67667	114.3	5.200	11.153	
D-2	374478	2759	142.8	0.253	0.456	

Total= 55.587 at time zero

CLOSURE= 0.999156436
Baratron= 55.54 at time zero (zero corrected)
Measured= 55.59 corresponding to time zero

Baratron zero= 0.06
Pressure= 53.8

Peak no. 1 ZERO
Bkg= 88 Deviation= 21.71199038

Peak no. 2 H-2
peak at 281403 hw= 52 profile(.01%) 30 30 20 21 4 1 0 2 7 11 14
v.zl= 498730.3725 88

Peak no. 3 HE-3
peak at 331617 hw= 56 profile(.01%) 7 0 0 0 19 41 50
v.zl= 6589.001299 88

Peak no. 4 HD
peak at 331889 hw= 58 profile(.01%) 16 11 8 13 0 15 37 58
v.zl= 65417.7619 88

Peak no. 5 D-2
peak at 374465 hw= 70 profile(.01%) 34 55 50 20 40 9 41 0 9 31 17
v.zl= 3753.533882 88

Peak	wt	height	time	Pressure	comp%	#2
H-2	281403	498407	55.5	45.444	84.452	
HE-3	331617	6501	84.1	2.100	3.910	
HD	331889	65330	122.7	5.993	11.143	
D-2	374465	2666	151.2	0.245	0.456	

Total= 53.795 at time zero

CLOSURE= 0.999156436
Baratron= 53.74 at time zero (zero corrected)
Measured= 53.79 corresponding to time zero

① 2/24/81. "⊕"

Peak data on file No. 13

No.	name	mf	hw	ir	vd	hp	bc	zd	fo	sens	dec.rate	bs
1	ZERO	838800	8	1	100	210	999	160	1	0.0	0.000000	0
2	XE124	929284	76	1	100	210	999	160	1	100.0	0.000024	0
3	XE126	932153	77	1	100	210	999	160	1	100.0	0.000023	0
4	XE128	935003	78	1	100	210	999	160	1	100.0	0.000023	0
5	XE129	936422	79	1	100	210	999	160	1	100.0	0.000023	0
6	XE130	937833	78	1	100	210	999	160	1	100.0	0.000023	0
7	XE131	939242	79	1	100	210	999	160	1	100.0	0.000023	0
8	XE132	940644	80	1	100	210	999	160	1	100.0	0.000023	0
9	XE134	943435	81	1	100	210	999	160	1	100.0	0.000023	0
10	XE136	946200	81	1	100	210	999	160	1	100.0	0.000023	0

LOW MASS I.E. IS 700
HIGH MASS I.E. IS 700

Xe Analysis
1st. Align + Menu.

Baratron zero= 0.12
Baratron zero= 0.15
Pressure=

Pressure=

Pressure= 42.44

Peak no. 1 ZERO
Bks= 109.25 Deviation= 21.87500376

Peak no. 2 XE124
peak at 929282 hw= 76 profile(.01%) 44 47 23 33 0 40 63 89 96#####
y,zl= 2981.281692 109.25

Peak no. 3 XE126
peak at 932151 hw= 76 profile(.01%) 54 47 22 39 14 0 53 35 49 63###
y,zl= 2787.326569 109.25

Peak no. 4 XE128
peak at 935001 hw= 79 profile(.01%) 76 40 9 0 0 3 19 37 63#####
y,zl= 57335.35053 109.25

Peak no. 5 XE129
peak at 936421 hw= 79 profile(.01%) 75 38 14 7 1 0 3 18 42 75
y,zl= 790506.8397 109.25

Peak no. 6 XE130
peak at 937833 hw= 78 profile(.01%) 82 42 16 1 0 0 7 22 45 75###
y,zl= 122102.2673 109.25

Peak no. 7 XE131
peak at 939241 hw= 79 profile(.01%) 50 26 16 3 0 4 9 30 62 98
y,zl= 634516.9088 109.25

Peak no. 8 XE132
peak at 940644 hw= 79 profile(.01%) 57 30 9 0 0 5 22 39 70 98
y,zl= 802922.5292 109.25

Peak no. 9 XE134
peak at 943435 hw= 80 profile(.01%) 54 27 20 6 0 2 9 20 44 78
y,zl= 311597.4009 109.25

Peak no. 10 XE136
peak at 946210 hw= 81 profile(.01%) 94 57 32 16 3 0 5 12 31 54
y,zl= 264479.7909 109.25

Peak	mf	height	time	Poress	Comp
XE124	929282	2872	175.5	28.840	0.0958
XE126	932151	2678	203.8	26.909	0.0894
XE128	935001	57426	232.2	577.369	1.9179
XE129	936421	790398	260.5	7951.806	26.4139
XE130	937833	121993	298.8	1228.087	4.0794
XE131	939241	634408	317.2	6390.491	21.2276
XE132	940644	802813	345.5	8091.895	26.3792
XE134	943435	311488	373.9	3141.457	10.4351
XE136	946210	264371	402.2	2667.799	8.8617

Total= 30104.651 at time zero

CLOSURE= 1.40477E-03
Baratron= 42.29 at time zero (zero corrected)
Measured##### corresponding to time zero

2/24/81

Xe analysis peak width 600ppm $Xe^{134} = 3 \times 10^{-4} A$, $t_{ap} 50 \mu A$, bandpass 4 μm
 Changed line 500 in Mass 3 (Prog. 9) to give longer wait for gas to
 travel from inlet to source.
 Now reads 500 WAIT T9 = 15000 (i.e. - 150 ~~secs.~~)

	Xe^{124}	Xe^{126}	Xe^{128}	Xe^{129}	Xe^{130}	Xe^{131}	Xe^{132}	Xe^{134}	Xe^{136}	
#1	0.0958	0.0894	1.9179	26.444	4.079	21.228	26.879	10.435	8.862	Fresh gas
#2	0.0956	0.0897	1.9182	26.448	4.078	21.225	26.883	10.431	8.862	
#3	0.0959	0.0898	1.9188	26.420	4.076	21.223	26.893	10.437	8.867	← Not, more like Xe - fresh gas
#4	0.0958	0.0897	1.9187	26.402	4.077	21.215	26.899	10.437	8.868	
#5	0.0957	0.0898	1.9188	26.442	4.077	21.224	26.871	10.440	8.872	Fresh Gas
#6	0.0962	0.0897	1.9187	26.412	4.077	21.223	26.885	10.433	8.865	
#7	0.0959	0.0891	1.9169	26.393	4.077	21.221	26.902	10.438	8.866	Fresh Gas
#8	0.0959	0.0898	1.9177	26.401	4.076	21.214	26.891	10.441	8.873	
#9	0.0959	0.0893	1.9166	26.401	4.079	21.231	26.878	10.440	8.868	Fresh Gas
#10	0.0959	0.0896	1.9167	26.399	4.079	21.221	26.894	10.439	8.867	
Mean	0.09588	0.08969	1.9176	26.4072	4.0775	21.2225	26.886	10.4371	8.867	
σ	0.000132	0.000223	0.00085	0.0091	0.00118	0.00521	0.0107	0.0033	0.00362	
2 σ	$\pm 0.431\%$	$\pm 0.52\%$	$\pm 0.10\%$	$\pm 0.08\%$	$\pm 0.06\%$	$\pm 0.06\%$	$\pm 0.09\%$	$\pm 0.07\%$	$\pm 0.09\%$	
Spec	1.0	1.0	0.25	0.12	0.20	0.13	0.12	0.17	0.18	
Error	-0.12	-0.33	-0.07	-0.12	-0.06	+0.20	\bar{x} 0.01	-0.03	-0.03	
Spec	1.0	1.0	0.5	0.3	0.4	0.3	0.3	0.3	0.4	

13 ⊕

Xe Abundance Sensitivity

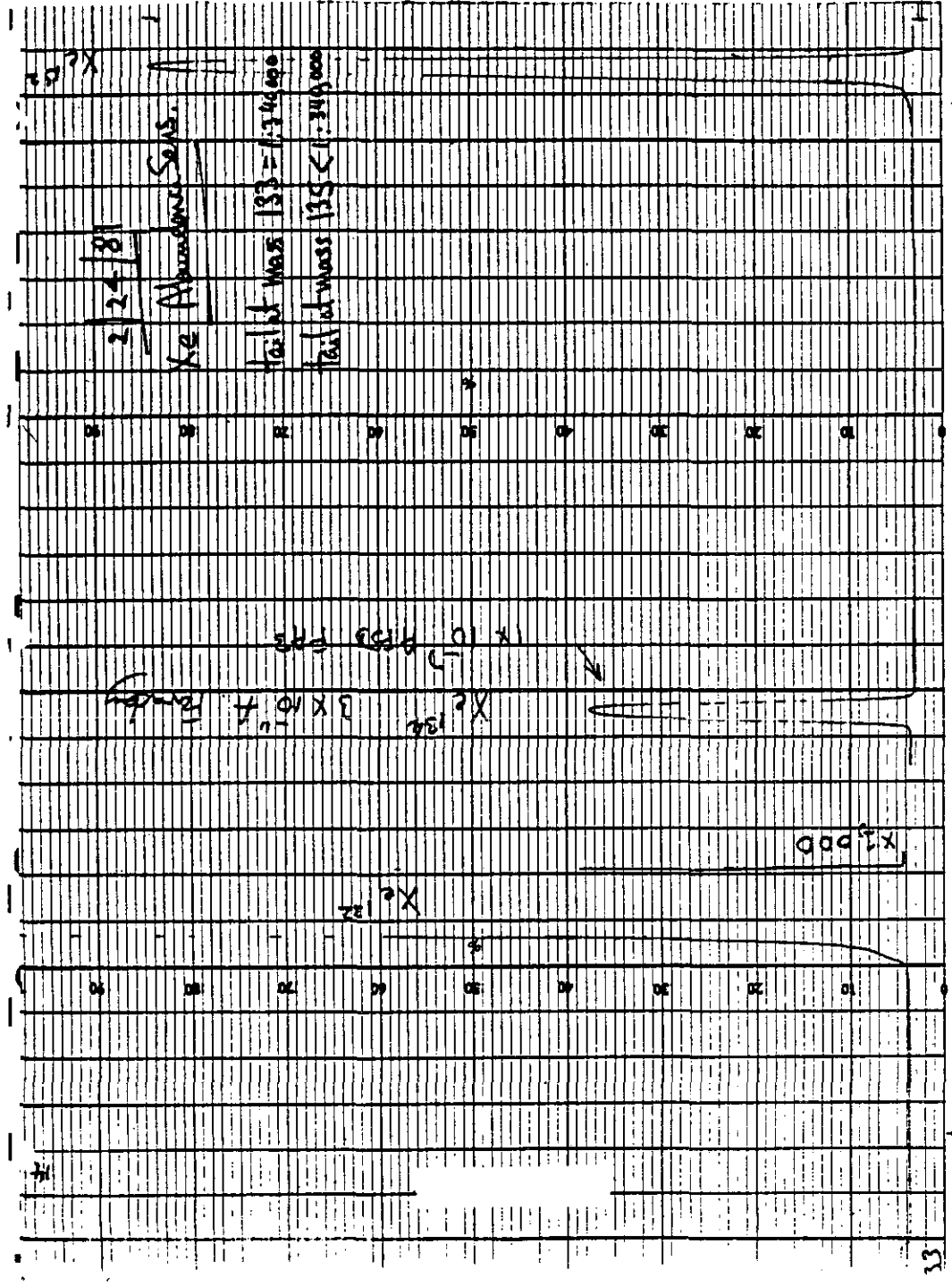
2/24/81

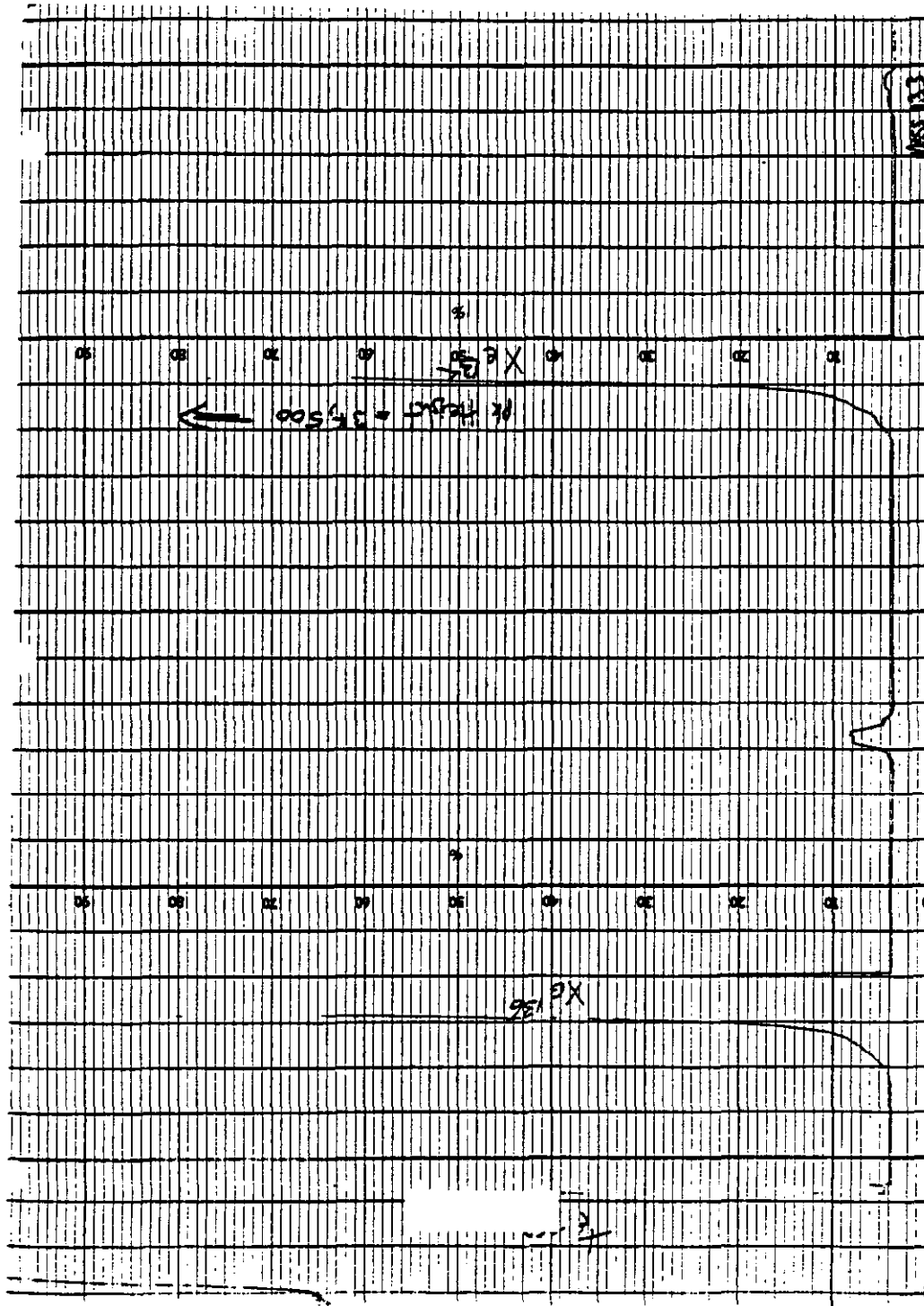
6.55 p.m.

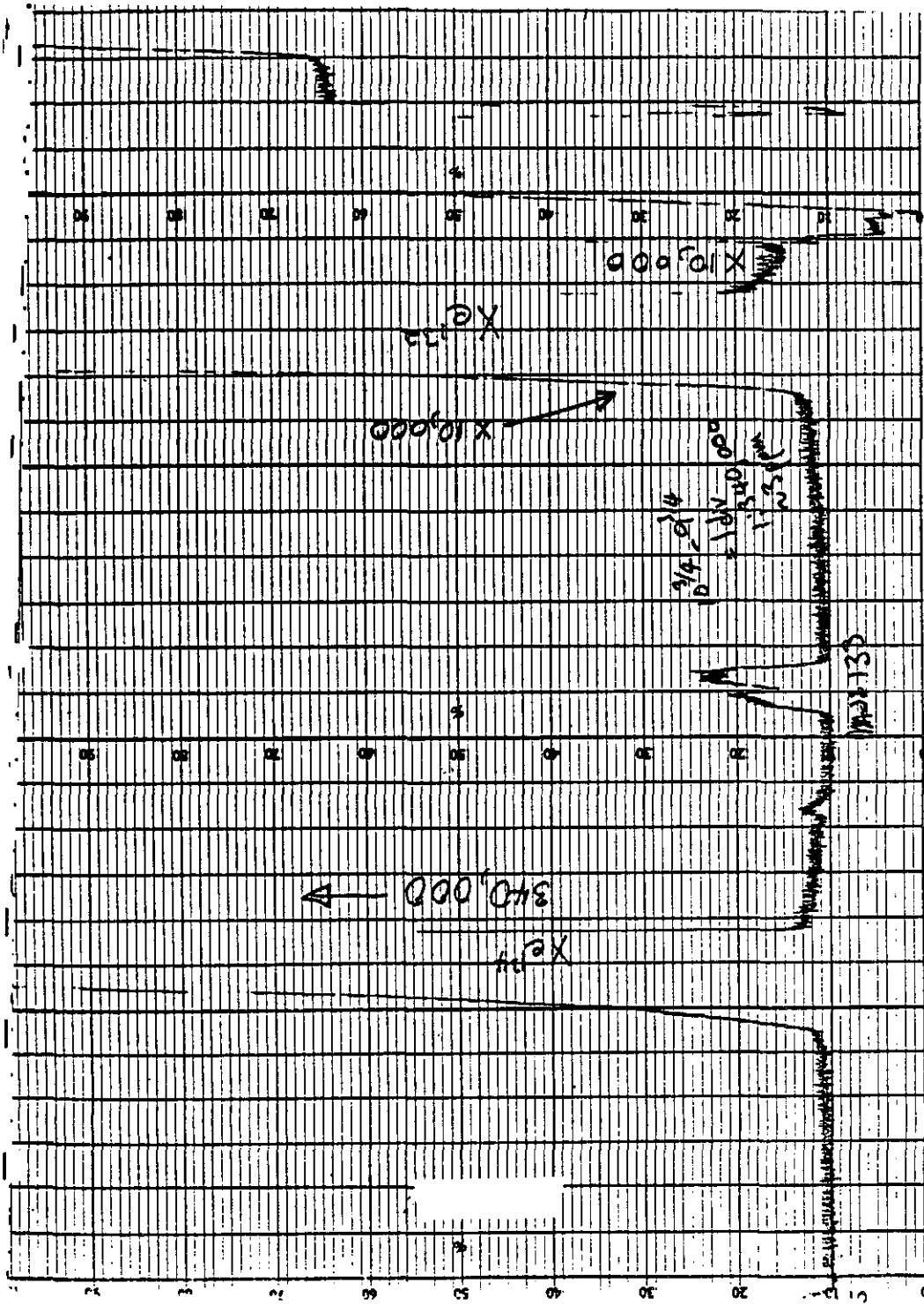
See attached Peak plots from mass 132 thru mass 138
Under maximum gain of $\times 10,000$ relative to $x1$ plot (Xe^{134}
pk. height of 34.5 divs.) , background at \sim Mass 133 is ~ 1 div.
related to 345,000 divs. for Xe^{134} ($3 \times 10^{-8} A$, $40 \mu m$ inlet)

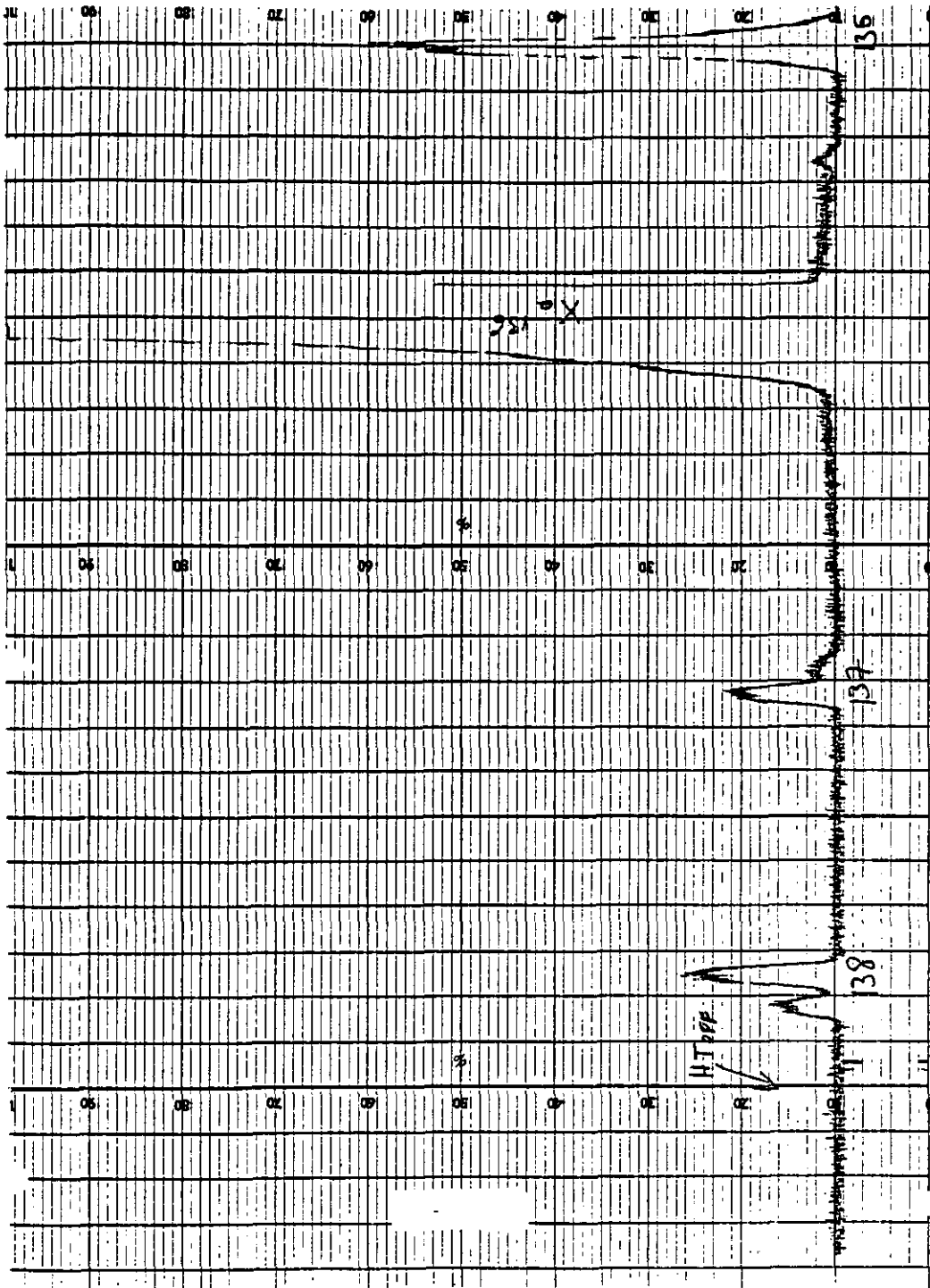
$$\therefore \text{Abund. Sens. (Mass 133)} = \frac{1:345,000}{\sim 3 \text{ ppm}}$$

$$\text{" " (Mass 135)} = \frac{< 1:345,000}{< 3 \text{ ppm}}$$









APPENDIX V

GAZAB PERFORMANCE AT A RESOLUTION OF 1300, MARCH AND APRIL 1981

No Lig N₂ intraps.

MIX #1
Cyl #3
3/24/81
B.T.A

Run	H-2	He-3	HD	D-2
1	0.464	3.984	11.164	84.389
2	0.462	3.989	11.179	84.370
3	0.463	3.988	11.179	84.370
4	0.463	3.986	11.173	84.378
5	0.462	3.989	11.176	84.373
6	0.464	3.997	11.186	84.353
7	0.463	3.992	11.180	84.365
8	0.462	3.992	11.178	84.368
9	0.461	3.986	11.169	84.384
10	0.461	3.988	11.176	84.375

Run	Total H	Total D	He-3
1	6.046	89.971	3.984
2	6.052	89.959	3.989
3	6.052	89.959	3.988
4	6.049	89.964	3.986
5	6.050	89.961	3.989
6	6.058	89.946	3.997
7	6.053	89.955	3.992
8	6.051	89.957	3.992
9	6.046	89.969	3.986
10	6.049	89.963	3.988

Total H :
 AVERAGE= 6.051
 STD DEV= 0.00356 (= 0.059%)
 (4.2SD= 0.247%)

Total D :
 AVERAGE= 89.960
 STD DEV= 0.00709 (= 0.008%)
 (4.2SD= 0.033%)

He-3 :
 AVERAGE= 3.989
 STD DEV= 0.00371 (= 0.093%)
 (4.2SD= 0.391%)

RESOLUTION = 1300

SOURCE FOCUSED FOR MAXIMUM ION BEAM

Sensitivity After mix #1 + mix 2 runs.
 3/24/81 BTB

H ₂	D ₂	HD	He ₃
199.41	216.09	209.43	56.24

Sensitivity Before Runs	H ₂	D ₂	HD	He ₃
	199.57	216.28	209.44	56.18

Run	H-2	He-3	HD	D-2
1	84.492	3.937	11.117	0.454
2	84.503	3.935	11.108	0.453
3	84.513	3.933	11.101	0.453
4	84.493	3.938	11.115	0.454
5	84.499	3.938	11.111	0.453
6	84.517	3.938	11.100	0.452
7	84.505	3.934	11.108	0.453
8	84.506	3.939	11.103	0.453
9	84.511	3.935	11.101	0.452
10	84.516	3.931	11.101	0.453

No $Ly. N_2$

MA # 2

Cy # 7

3/24/81 BTB

Run	Total H	Total D	He-3
1	90.050	6.012	3.937
2	90.057	6.008	3.935
3	90.063	6.004	3.933
4	90.051	6.011	3.938
5	90.054	6.008	3.938
6	90.067	6.002	3.938
7	90.059	6.007	3.934
8	90.057	6.004	3.939
9	90.062	6.003	3.935
10	90.066	6.003	3.931

Total H :
 AVERAGE = 90.059
 STD DEV = 0.00536 (= 0.007%)
 (4.2SD = 0.027%)

Total D :
 AVERAGE = 6.006
 STD DEV = 0.00353 (= 0.059%)
 (4.2SD = 0.247%)

He-3 :
 AVERAGE = 3.935
 STD DEV = 0.00292 (= 0.074%)
 (4.2SD = 0.311%)

RESOLUTION = 1300

SOURCE FOCUSED FOR MAXIMUM ION BEAM

APPENDIX VI

SPECIFIED ACCEPTANCE TESTS FOR THE FINNIGAN MAT 250 HDT MASS SPECTROMETER

The following tests shall be made at the factory in Bremen using Varian's inlet system, and also at SRP using either Varian's or SRP's inlet system. All tests shall be made under the following conditions:

Source slit width	0.15 mm
Collector slit width	0.60 mm
Resolution	500 measured at mass 3 between ^3He and HD
Electron current*	~1 mA
Electron energy	~80 eV
Accelerating voltage	10 kV
Inlet system molecular leak**	3 holes, each 0.02 mm in diameter
No permanent magnets for the electron beam	
Plateau peaks	

The following requirements must be met:

- The sensitivity for hydrogen shall be at least 10^{-9} A/torr. Sensitivity is defined as hydrogen ion current versus pressure inside the inlet system behind the molecular leak.
- Linearity of hydrogen sensitivity shall be less than 0.01 over a pressure range of 0.070 torr to 0.9 torr in the inlet system.† Linearity is defined as the maximum sensitivity deviation divided by the mean sensitivity.
- Linearity of argon sensitivity shall be less than 0.01 over a pressure range of 0.030 torr to 0.6 torr in the inlet system.†

* The electron current is the total emission from the filament. The Germans call this the "ionizing current", thus the use of "IC" in the figures.

** The calculated conductance of the molecular leak is $0.11 \text{ cm}^3/\text{sec.}$, which is 55% of the conductance of the MSTG reference leak ($0.2 \text{ cm}^3/\text{sec.}$).

† A 1000-megohm resistor was used in all tests.

- Mass discrimination between hydrogen and deuterium shall not be detectable.
- The abundance sensitivity shall exceed 20,000, measured at the high-mass side of the HD peak at a distance which is equal to the distance between the ^3He peak and the HD peak. Abundance sensitivity is defined as the ratio of the HD peak height divided by the (imaginary) ^3He peak height.
- D_3 production from D_2 shall be less than 1200 ppm at a D_2 ion current of 10^{-9} A.
- Flatness of the peak plateau shall be better than 0.001 over a width $\pm 5\%$ from the center of the peak. The peak width shall be determined by measuring the peak width at 5% of peak height.

AX 510658N

PURCHASE ORDER ADDENDUM

Copy Frank's specs 2/19/81

<u>ITEM NO.</u>	<u>QUANTITY</u>	<u>DESCRIPTION</u>
1	1	<p>Mass Spectrometer system, Varian MAT Model 250 HDT, specially modified for hydrogen-helium isotopic analyses. The instrument will be fitted with the following:</p> <ul style="list-style-type: none">(a) Triple Faraday collector system with amplifiers. ✓ Collector spacing will be specified later.(b) 270 l/sec Turbomolecular pump on the source. ✓(c) A liquid nitrogen cooled trap between the source ✓ and the Turbomolecular pump. This trap shall have a capacity of \approx 20 hours. ¹² This is 12-15 hr. Frank says is Bremen time ✓(d) A 24 channel peak selector, programmable over ✓ the entire mass range.(e) Temperature stabilized ion source controls with ✓ \pm 0.1 C regulation.(f) Differential pumping for source and analyzer ✓ sections.(g) A closed circuit cooling water system for ✓ all equipment requiring cooling water.(h) An oil booster pump between the Mechanical and ✓ Turbomolecular pump.(i) An accelerating voltage supply capable of 10kv. ✓

The instrument will operate on 60Hz, 480 volt (phase to phase), 3-phase Delta (3 wire) electrical power at a current of not more than 30 amperes/phase.

The instrument will meet the attached performance specifications and will be tested at the vendor's factory in accordance with those specifications before it is shipped. The tests may be witnessed by a representative of the purchaser. Written approval of the tests by the purchaser will be required before the instrument is shipped.

The final acceptance tests will be a repeat of those in the attached performance specification.

<u>ITEM NO.</u>	<u>QUANTITY</u>	<u>DESCRIPTION</u>
2	8	Sets of operating and maintenance manuals, parts lists, drawing and schematic diagrams.
3	1	Set of all alignment jigs, special tools or other equipment not readily available on the open market and needed to service and maintain this instrument.

4 - SHIPPING ADDRESS

*E. I. du Pont de Nemours & Co.
300/700 Receiving Dept.
Dunbarton, SC

*Deliver directly to P. Chastagner, Bldg. 773-A.
DO NOT unload at 300/700 Receiving Dept.

NOTE:

The mass spectrometer system should not include the programmable calculator unless the calculator is of the type that the program is hardwired in the system.

Provide a 1" diameter port in the source housing directly above the filament area of the ion source. This port should be sealed with a Varian standard 2-3/4" OD high vacuum flange.

PRODUCT ACCEPTANCE REPORT NO. _____

varian MAT
mass spectrometry



INSTRUMENT	<u>MAT 250 HDT</u>	SERVICE NO.	<u>8249</u>
INSTITUTION	<u>DuPont / Savannah River</u>	VARIAN S.O. NO.	_____
STREET	<u>Savannah River Plant 773A</u>	TELEPHONE	<u>404 725 6211</u>
CITY/STATE/ZIP	<u>Aiken SC 29801</u>		
CUSTOMER CONTACTS			
NAME/TITLE/EXT.	<u>H.L. Daves / Area Supervisor / 6715</u>		
NAME/TITLE/EXT.	<u>W.B. Hess / Process Staff physicist / 6712</u>		

EXCEPT AS NOTED BELOW, THE DELIVERED ITEMS APPEARING ON THE ABOVE REFERENCED VARIAN SALES ORDER ACKNOWLEDGMENT(S) HAVE BEEN INSTALLED. THE SPECIFIED TESTS FOR THE DELIVERED ITEMS HAVE BEEN COMPLETED SATISFACTORILY AND OPERATION AND MAINTENANCE TRAINING FOR THE DELIVERED ITEMS HAVE BEEN GIVEN TO CUSTOMER PERSONNEL.

DELIVERED ITEMS REQUIRING FURTHER ACTION

ITEM	ACTION REQUIRED
<u>1 Leybold rotary pump (leaking)</u>	<u>replace from F.P. stock</u>
<u>1 computer keyboard documentation</u>	<u>send from F.P.</u>

ORDERED PARTS OR ACCESSORIES NOT RECEIVED

ITEM	ACTION REQUIRED
<u>2 slits (.22, .60 mm)</u>	<u>H. Rache will ship from BLE</u>
<u>1 cathode 46522</u>	<u>ship</u>
<u>1 transistor 29156</u>	<u>ship</u>

CUSTOMER HAS BEEN GIVEN A COPY OF "WARRANTY ON VARIAN/MAT EQUIPMENT SOLD TO UNITED STATES CUSTOMERS" CUSTOMER UNDERSTANDS THAT THE WARRANTY PERIODS ON THE ACCEPTABLE DELIVERED EQUIPMENT BEGIN ON THE SIGNATURE DATES SHOWN BELOW.

FOR CUSTOMER

SIGNED _____
DATE Feb 20 1981

FOR VARIAN

SIGNED SERVICE ENGINEER _____
DATE 20 Feb 81
SIGNED SERVICE MANAGER _____
DATE _____

6480-42-04 1/75 PRINTED IN U.S.A.

- 57 -

DISTRIBUTION: WHITE - SERVICE MANAGER

PINK - SERVICE ENGINEER GOLD - _____

CANARY - BREMEN SERVICE MANAGER

Siv.

S/P

Argon Linearity

300we

Linearity check

Date: 190281

Run: 4

$\bar{x} = 1.2\%$

P(torr) 0.5813

Sig(V) 1.2233

S/P 2.1044

P(torr) 0.4129

Sig(V) 0.8649

S/P 2.0946

P(torr) 0.2454

Sig(V) 0.5111

S/P 2.0826

~~P(torr) 0.1118~~

~~Sig(V) 0.2375~~

~~S/P 2.1242~~

P(torr) 0.1113

Sig(V) 0.2335

S/P 2.0884

P(torr) 0.0549

Sig(V) 0.1150

S/P 2.0944

P(torr) 0.0315

Sig(V) 0.0655

S/P 2.0787

$\bar{x} = 2.0905$

linearity $\pm .60\%$

specification $\pm .5\%$

Hydrogen Linearity

Linearity check

Date: 190281

Run: 2

$\bar{x} = 1.1\%$

P(torr) 0.8872

Sig(V) 1.5309

S/P 1.7255

P(torr) 0.7272

Sig(V) 1.2530

S/P 1.7230

P(torr) 0.5773

Sig(V) 0.9941

S/P 1.7202

P(torr) 0.3547

Sig(V) 0.6086

S/P 1.7156

P(torr) 0.2037

Sig(V) 0.3491

S/P 1.7138

P(torr) 0.1295

Sig(V) 0.2216

S/P 1.7112

P(torr) 0.0728

Sig(V) 0.1242

S/P 1.7060

$\bar{x} = 1.7165$

$\pm .55\%$

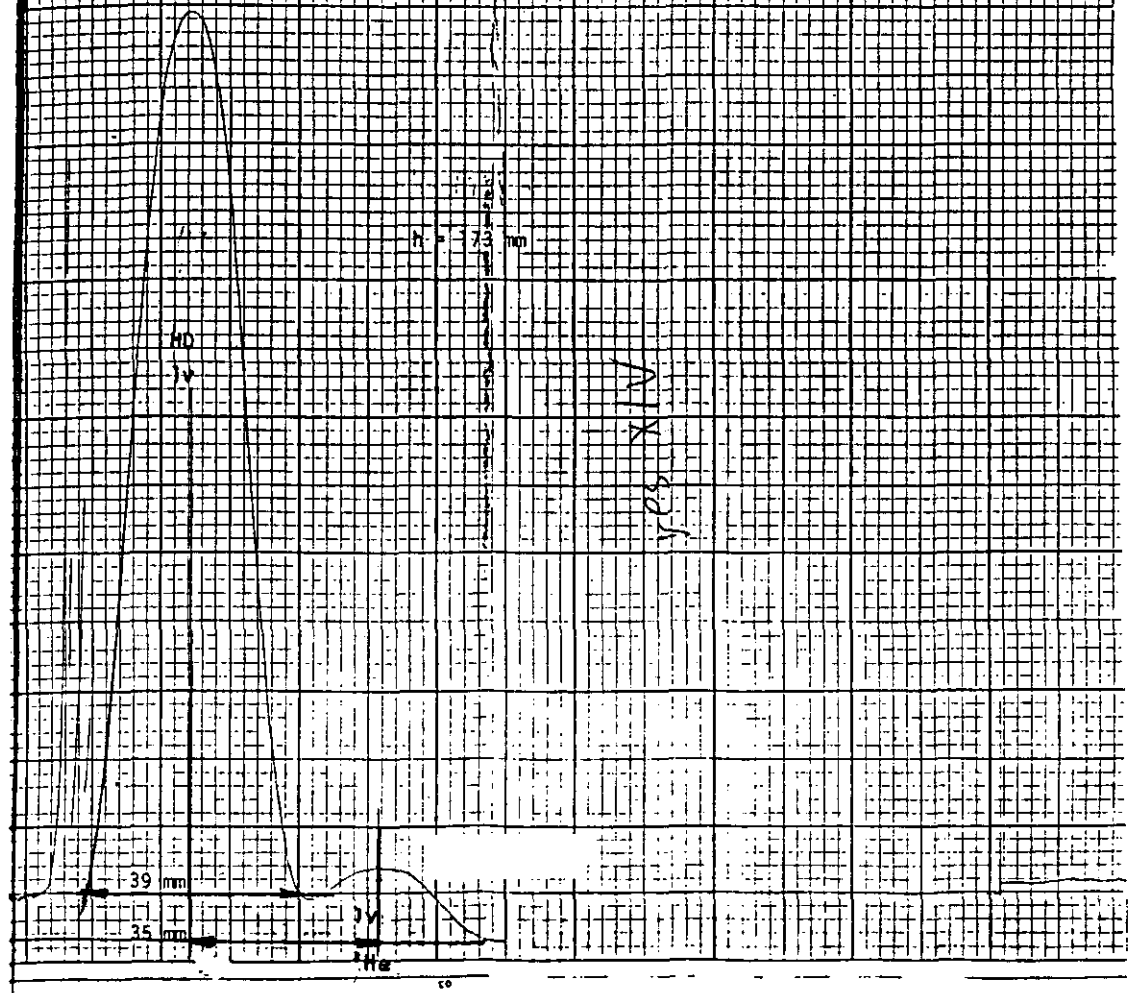
$\pm .5\%$

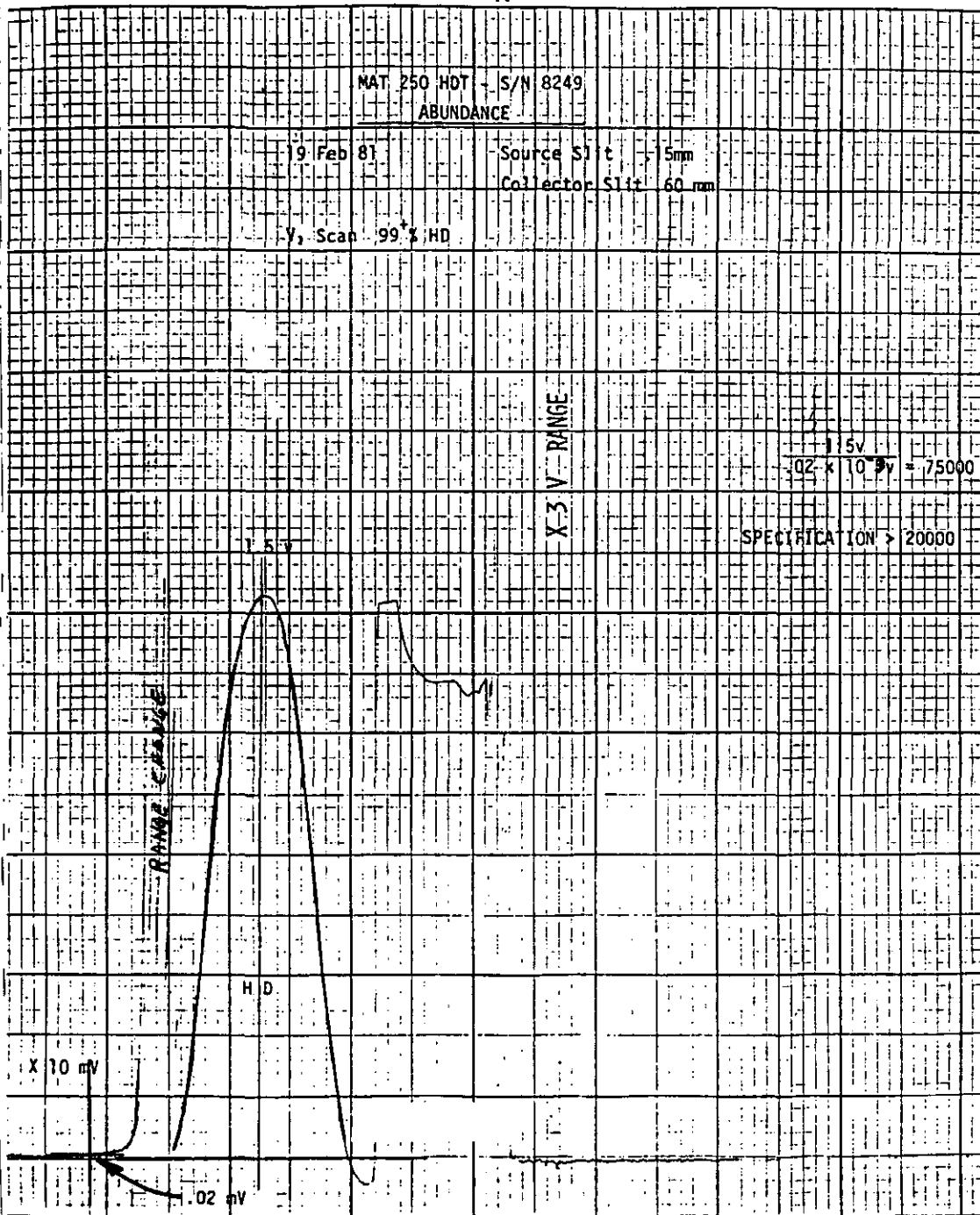
MAT 250 HDI S/N 8249
RESOLUTION & ABUNDANCE

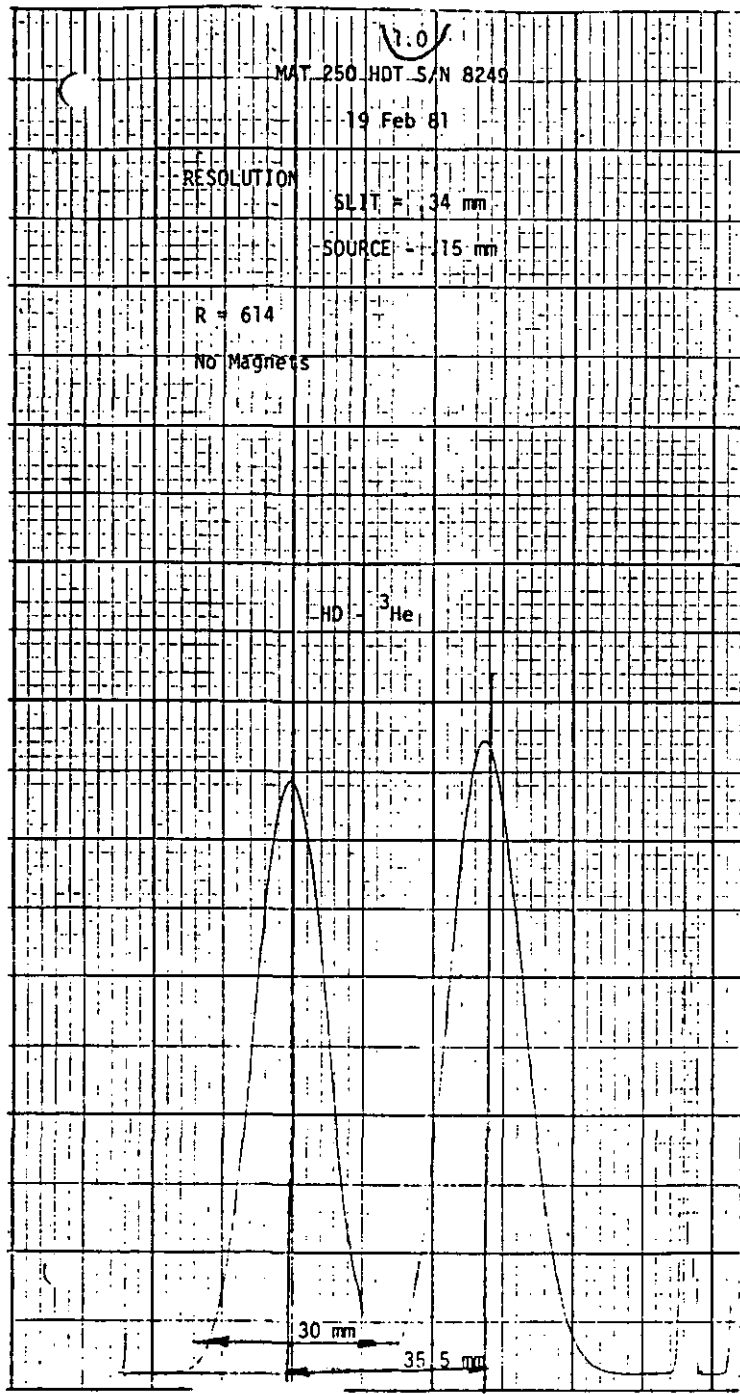
19 FEB 81
Source slit 5 mm
Collector slit 50 mm
10/1 HD/He
1084 gauss
V scan

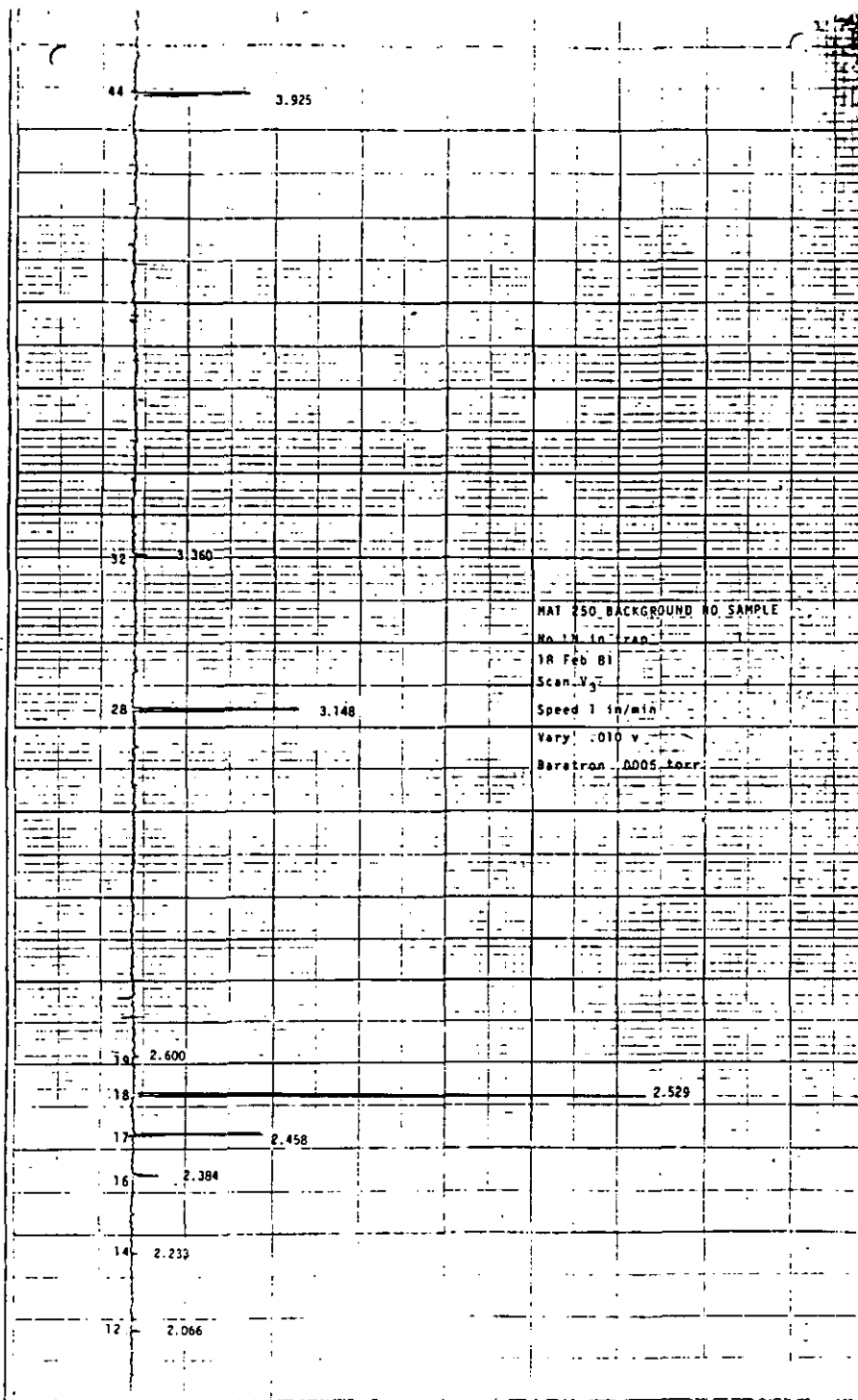
$$R = \frac{3 \times 35}{5.8 \times 10^{-4} \times 39}$$

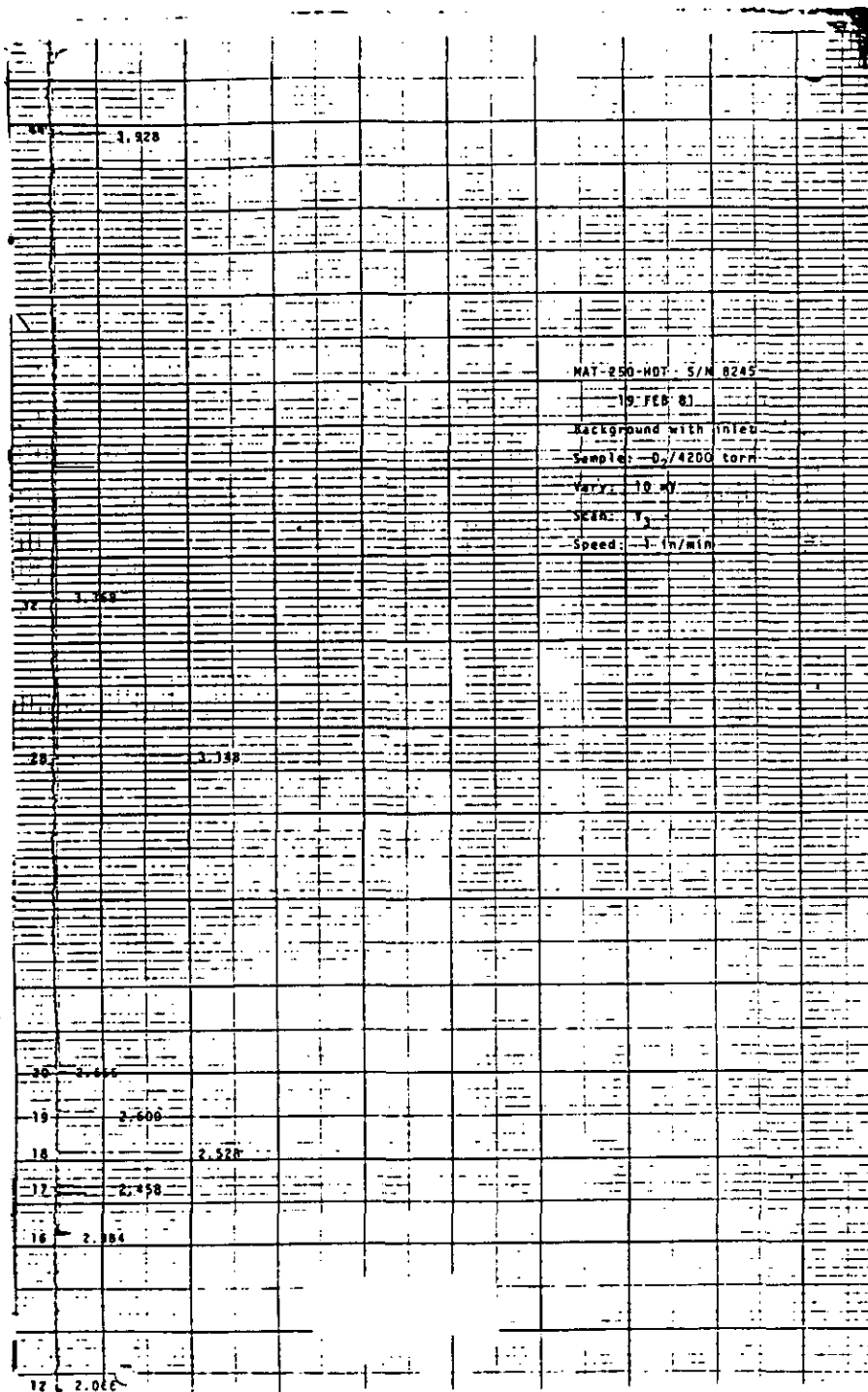
= 465

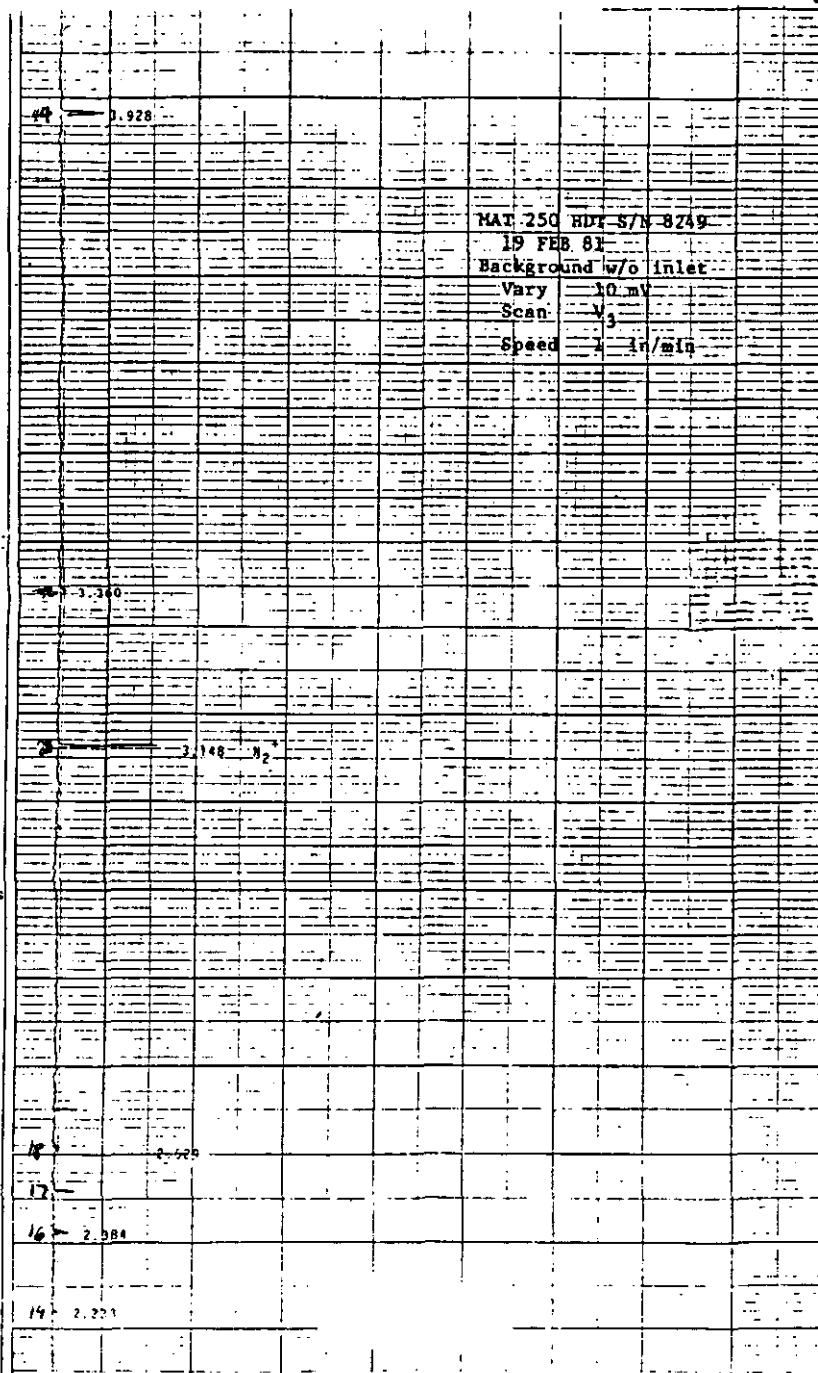












APPENDIX VII

ACCEPTANCE TESTS OF THE FINNIGAN (FORMERLY VARIAN) MAT 250 HDT,
FEBRUARY 1981

PERFORMANCE TESTS AT SRL - VARIAN MAT 250 HDT

February 19, 1981

TEST CONDITIONS

Source slit width	0.15 mm - 0.0059"
Input Resistor	$10^9 \Omega$
II Collector slit width	0.60 mm - 0.0236"
Resolution	465 measured at mass 3 between ^3He and HD
Cathode	6.1 amps
Total Filament Emission	1 ma (300 μa for Argon linearity)
Electron Energy	80 ev.
Trap Voltage	135 volts
Source Temperature	160°C
Accelerating Voltage	10 kV
Molecular Leak (Varian)	3 holes, (.00079") 0.02 mm diameter each. Same molecular leak used in Bremen September, 1980
Base Pressure w/o Sample	Source 2×10^{-8} torr - Analyzer 2×10^{-7} torr
Liquid Nitrogen Trap Filled	
No permanent magnets for the electron beam	
Plateau peaks	

SENSITIVITY FOR HYDROGEN

P inlet system 0.425 torr

H₂ peak height 0.727 v

$$\text{SENS. } \frac{0.727 \text{ v}}{10^9 \Omega \times .425 \text{ torr}} = 1.7 \times 10^{-9} \text{ amps/torr}$$

Specification $\bar{>}$ 10^{-9} amps/torr

MASS DISCRIMINATION

Sensitivity of H_2 at $\sim 420\mu$ = 1.708×10^{-9} amps/torr (inlet pressure)

Sensitivity of D_2 at $\sim 420\mu$ = 1.697×10^{-9} amps/torr (inlet pressure)

$\Delta.6\%$

Specification: Mass discrimination shall not be detectable.

D_3^+ Production from D_2

564 μ D_2 950 mv D_2 $10^9 \Omega$ = $.95 \times 10^{-9}$ amps

D_3 32 mv, $3 \times 10^{11} \Omega$ = 1×10^{-13} amps

D_3 = 105 ppm of D_2^+ peak

Specification: D_3 from D_2 shall be < 120 ppm at 10^{-9} amps D_2 ion current