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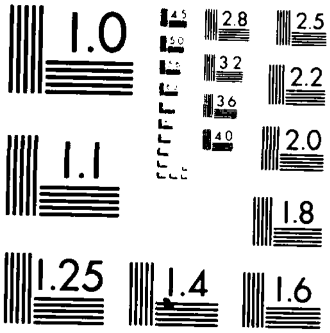
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THE ASSEMBLY AND DEMONSTRATION OF THE FUNCTIONALITY OF A FIELD IONIZATION MASS SPECTROMETER/MASS SPECTROMETER (FIMS/MS) SYSTEM

by S.E. Buttrill, Jr.
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May 1986

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The purpose of this project was to assemble and demonstrate the functioning of a mass spectrometer/mass spectrometer (MS/MS) system that could be used eventually to study collisions between electrons and positive ions. In this phase of the program, fragmentation of ions between the first and second mass spectrometers was accomplished by collisions with a neutral gas. SRI International used existing mass spectrometer equipment to construct the new MS/MS system. This report describes the design and construction of the MS/MS instrument and gives the results from the early instrument performance tests.

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Gas collision
Collision chamber
Collision gas jet

PREFACE

The work described in this report was authorized under Contract No. DAAD05-81-C-0181. This work was started in September 1981 and completed in November 1982.

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THE ASSEMBLY AND DEMONSTRATION OF THE FUNCTIONALITY OF A FIELD IONIZATION MASS SPECTROMETER/MASS SPECTROMETER (FIMS/MS) SYSTEM

1. INTRODUCTION

The purpose of this project was to assemble and demonstrate the functioning of a mass spectrometer/mass spectrometer (MS/MS) system that could be used eventually to study collisions between electrons and positive ions. In this phase of the program, fragmentation of ions between the first and second mass spectrometers was accomplished by collisions with a neutral gas.

SRI International (SRI) used existing mass spectrometer equipment to construct the new (MS/MS) system. This report describes the design and construction of the MS/MS instrument and gives the results from early, instrument performance tests.

2. BACKGROUND AND OBJECTIVES

2.1 Background.

Previous work at SRI showed that field ionization mass spectrometry (FIMS) can be used to detect and monitor low concentrations of chemicals in the air. Laboratory prototype instruments have demonstrated a sensitivity < 0.01 mg/m^3 for nerve agent simulants. Others* have shown that two mass spectrometers can be used in tandem to provide increased specificity for the detection and identification of specific compounds present in complex mixtures. This section describes the basic elements of a mass spectrometric detector for chemicals in the air and compares the single and tandem mass spectrometer concepts.

Using mass spectrometry to analyze compounds in the air can be completed in four steps.

a. Sample Inlet. The chemical molecules present as a dilute vapor in the air must be introduced into the mass spectrometer ion source or vacuum system at a pressure of 10^{-6}-10^{-4} torr. This is accomplished by allowing the chemical vapors to diffuse through a heated, dimethyl silicone membrane, which passes chemical molecules of interest more rapidly (three orders of magnitude) than the major constitutents of air. Therefore, the concentration of the chemicals of interest is increased relative to the

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*Kondrat, R. W. and Cooks, R. G. Direct Analysis of Mixtures by Mass Spectrometry. Analytical Chemistry, 50, 81A-92A (1978).

concentration of air, and the total pressure is reduced to a level which is compatible with the operation of the mass spectrometer vacuum system.

b. Sample Ionization. Although there are many different ways to ionize molecules, field ionization (FI) is unique in its ability to produce unfragmented ions from most chemical compounds. The FI sources developed at SRI also require very little power, are very sensitive, and were used exclusively for the work conducted for this project.

c. Mass Separation. At SRI, quadrupole mass filters and magnetic sector mass spectrometers have been used with the air sampling system and FI sources. Of the commercially available mass spectrometers, the quadrupoles are the most compact and easiest to use for a programmable monitor. However, for a mass spectrometer system with the ultimate in low weight and power consumption, a miniature, double-focusing magnetic sector would be preferred.

d. Data Recording. For a chemical agent monitor or detector, the only ions that need to be monitored are those corresponding to the molecular weight of agent materials. These signals can be displayed as a concentration of agent or simulant.

The overall concept of a FIMS chemical monitor is shown in Figure 1 which shows the various steps involved in going from chemical molecules in the air to a final record of the chemical composition. The inlet system itself is not specifically shown. In Figure 1, it is assumed that the entire mass range is scanned by the mass spectrometer to display the relative amounts of different chemicals present in the air. However, interest is limited to those masses in the spectrum that correspond to known chemical agents.

The tip of the dimethyl silicone membrane interface used for sampling chemicals from the air is shown in detal in Figure 2. The complete interface is approximately 12 inches long and is designed to fit through the 1/2-inch-diameter air lock of the FIMS. The membrane interface can be added to the mass spectrometer at any time without breaking the vacuum or performing any extensive instrument modifications. Air to be sampled is pulled through the interface by a small, mechanical vacuum pump, and flows through a glass-lined stainless steel tube to the dimethyl silicone membrane. The whole tube is heated to reduce holdup and interface response time for polar materials such as chemical agent simulants. Because the tip of the interface is inside the mass spectrometer vacuum envelope, the membrane's temperature is essentially the same as the temperature read by the thermocouple at the end of the air inlet tube. The membrane's active area is approximately 46 mm^2.

For the past 6 years, SRI has been working with and improving an FI source originally developed by Mr. Capp Spindt. This FI source is called the volcano-style source because of the volcano-like appearance of the active element in the early models. In more recent versions, the ionizing

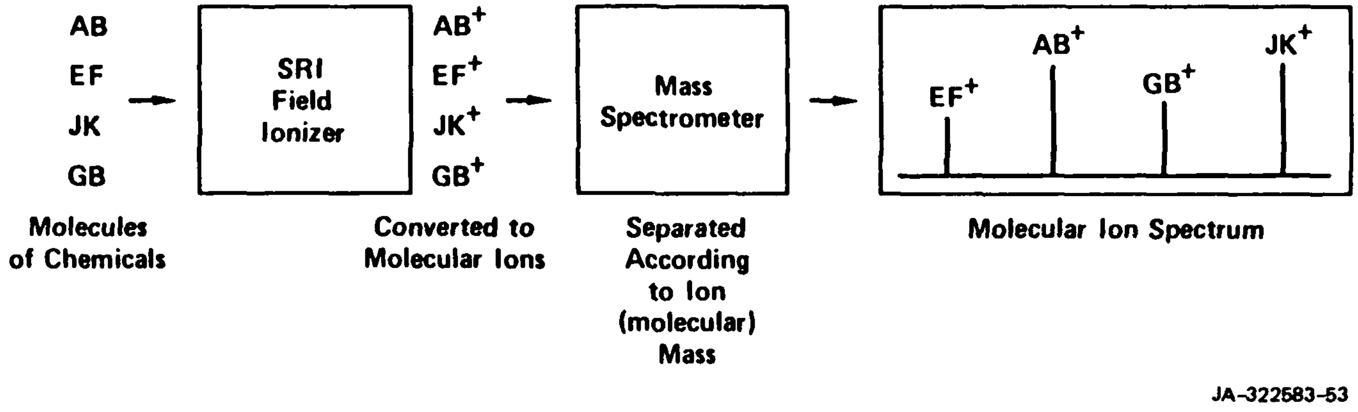

FIGURE 1. FIELD IONIZATION MASS SPECTROMETRY (FIMS) ALARM CONCEPT

The mass spectrometer can be rapidly set to monitor the chemical(s) of interest.

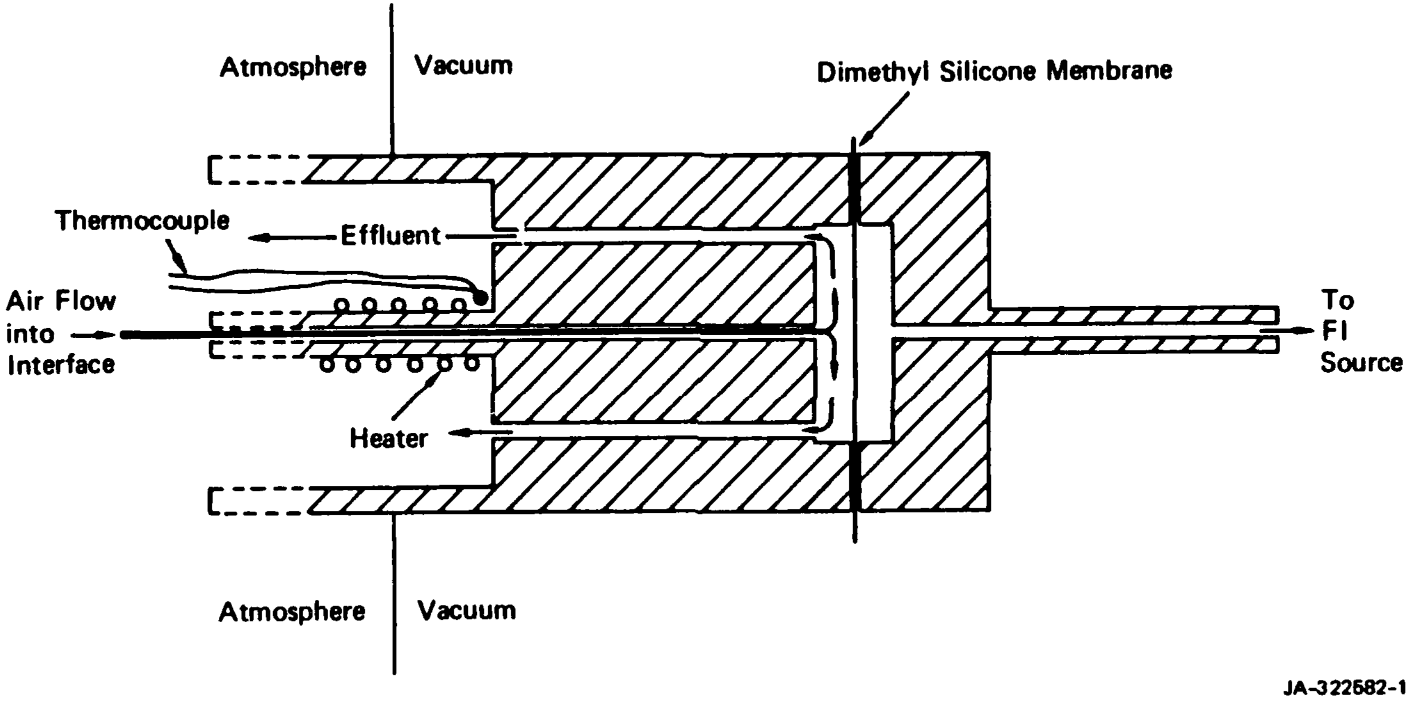

FIGURE 2. MEMBRANE INTERFACE FOR SAMPLING CHEMICALS FROM AIR INTO A MASS SPECTROMETER

element closely resembles an inverted funnel with a throat diameter of 15 microns. Figure 3 shows an electron micrograph of a volcano-style, field ionizer located beneath a circular opening in its associated counter electrode.

The FI mechanism may be viewed as an electron tunneling from the sample molecule into the field ionizer, in this case, the inverted funnel structure or volcano. Normally, this requires electric field strengths of 10^7 to 10^8 v/cm. In the volcano-style, field ionization source, this very high field strength is achieved using applied potentials of only 1-2 kv. The smallness of the volcano structure and its closeness to its counter electrode, together with submicroscopic structures on the rim of the opening, combine to produce the high, field strength needed for FI, even with these modest potentials.

Figure 4 shows a scale drawing of the entire volcano FI source. The source assembly provides mechanical supports for both the volcano and its counter electrode and also has provisions for separately heating both of these components. To support the necessary potential difference required for FI, the components are electrically insulated from each other. This particular design of FI source is highly sensitive because all sample molecules entering the source are forced to leave through the volcano itself. Thus, all of the sample molecules are forced through the high field region where FI occurs.

Because FI produces only molecular ions from most compounds, the FI mass spectrum of a mixture is normally quite simple. Each component in the mixture contributes to one peak in the FI spectrum. The amount of a single component can be determined from the size of the single peak which corresponds to that component.

Because the volcano-style FI source produces ions over such a small area, the resulting ion beam is very bright. The beam's brightness allows almost all of the ions to be focused into the mass spectrometer. As a result, the overall instrument sensitivity is higher. At SRI, volcano-style FI sources operating on quadrupole and magnetic sector mass spectrometers have demonstrated overall instrument sensitivities of 2×10^{-6} ions/molecules. On occasion, even higher sensitivities have been obtained. This sensitivity means that only 10^{-14} gram of sample introduced into the ion source will yield a mass analyzed signal of 100 ions at the detector. With this high sensitivity, it is obvious that only very small amounts of sample need to enter the mass spectrometer ion source to produce detectable signals.

Although the existing FIMS monitor provides a highly selective method of detecting chemicals in the air, situations occur in which a greater specificity is desirable. Knowing the molecular weight of a compound is not enough information to identify it. Background concentrations of naturally occurring and man-made substances range from 0.5 ppb to approximately 5 ppb at most masses. Since the detection of specific chemical agents below these levels requires more specificity than just knowing the

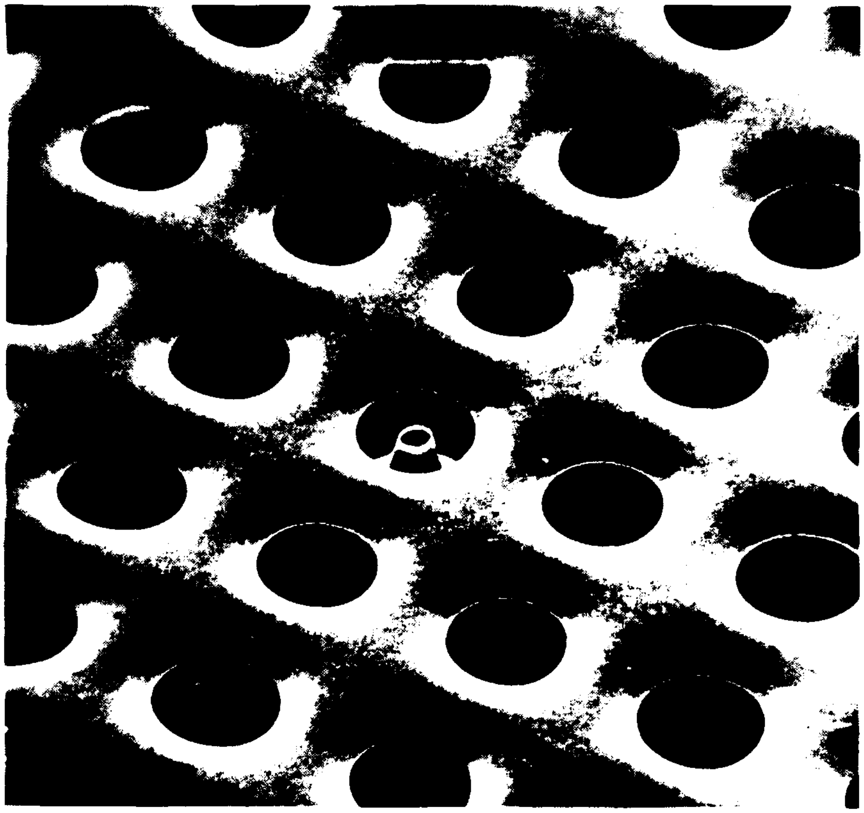

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FIGURE 3. VOLCANO FIELD IONIZATION SOURCE

Volcano throat 15 μm

Counterelectrode hole spacing 100 μm center to center.

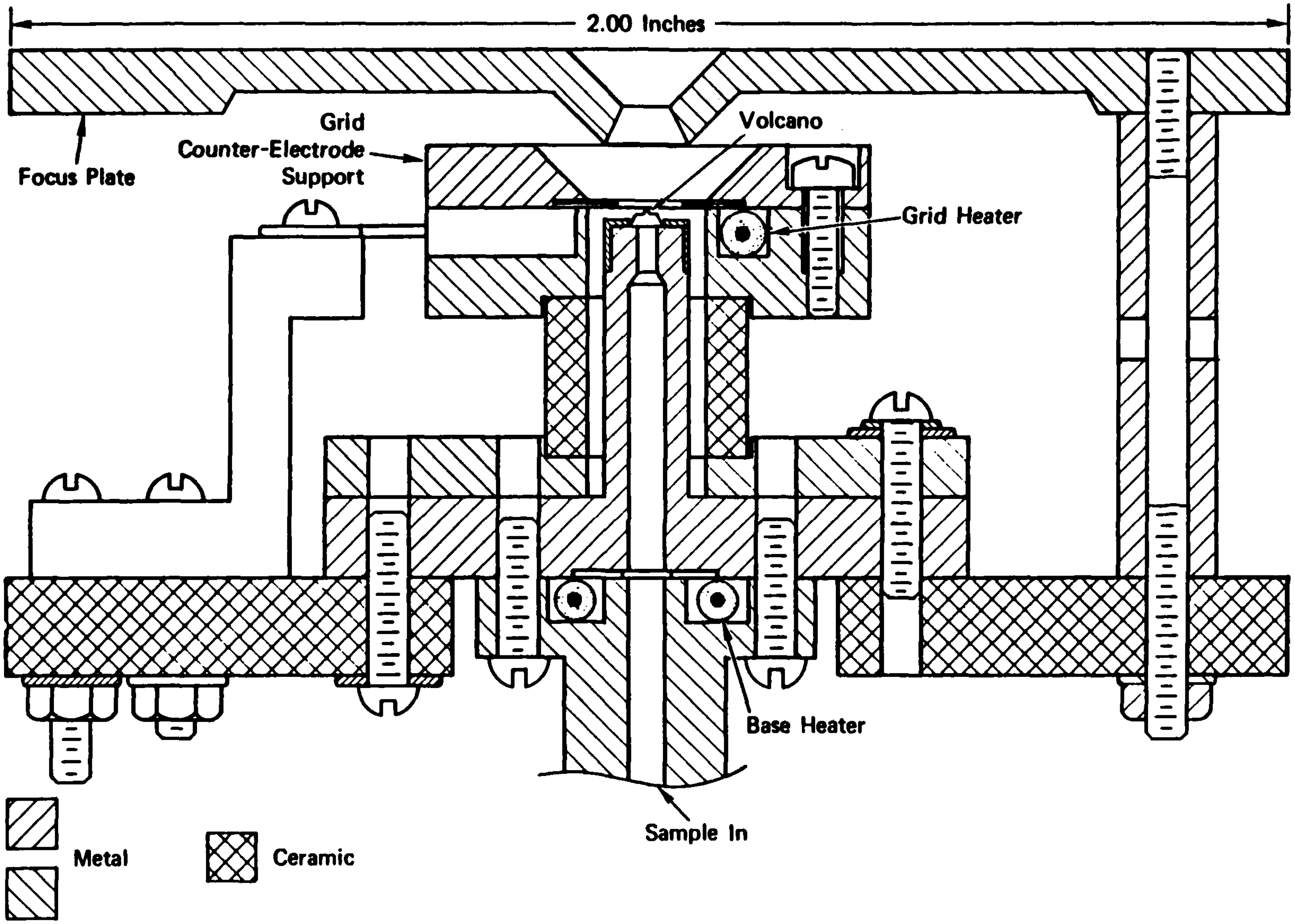

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FIGURE 4. IMPROVED VOLCANO-TYPE FIELD IONIZATION SOURCE

molecular weight of the compound, more information than that will be required to detect an unknown substance in the air.

Using two mass analyzers in tandem provides a means of attaining the additional chemical information needed for the two above applications. Such tandem mass spectrometry is termed MS/MS. The concept of a FIMS/MS detector for chemical agents is shown in Figure 5. Molecules from the air enter the instrument by a membrane inlet. The molecular ions produced by the FI source are separated according to mass by the first mass spectrometer, MS(1). A molecular ion of selected mass is then fragmented to produce a pattern of fragment ions. The spectrum of fragment ions is recorded by the second mass spectrometer, MS(2). The fragment ion spectrum produced from a molecular ion of the compound is very closely related to and often identical with the conventional electron impact mass spectrum of the same compound. Therefore, the fragment ion spectrum will be a great aid in identifying unknown substances. An alternative application would use the FIMS/MS device as a highly specific monitor for a particular compound by tuning MS(1) to pass all ions with a mass corresponding to the molecular weight of the compound of interest. These molecular ions would then be fragmented, and MS(2) would pass a fragment ion mass that was highly characteristic of the compound. Thus, the only signals detected would come from compounds that had the correct molecular weight and the ability to produce the fragment ion at the characteristic mass.

2.2 Objectives.

The main objective of the overall program, of which this project is the first phase, is to field a transportable MS/MS system which will detect and identify chemical agents. Reducing the size, weight, and power consumption of mass spectrometer systems is primarily a matter of reducing the size, weight, and power consumption of their associated vacuum systems. The usual method for fragmenting ions in an MS/MS device is to collide the ions at high energy with a beam of neutral gas molecules. The neutral gas in the collision region places a heavy load on the vacuum system and requires large vacuum pumps that use substantial amounts of electrical power.

The specific objective of this project was to assemble and demonstrate a FIMS/MS instrument that could be used to compare the sensitivity and efficiency of MS/MS instruments using the gas collision method and new, ion fragmentation methods (e.g., fragmentation of ions by electron impact). Future projects will develop and test these novel methods of ion fragmentation.

3. MS/MS DESIGN

Two mass spectrometer systems were available at SRI for use in this project. The first was a quadrupole mass filter manufactured by Extranuclear Laboratories of Pittsburgh, Pennsylvania. The quadrupole mass spectrometer was already equipped with an SRI, volcano-style FI source. This instrument had been developed earlier to detect explosive vapors in air and had received extensive service as the mass spectrometer component of a GC/FIMS system. The second mass spectrometer available was a 90°, 10-in.-radius,

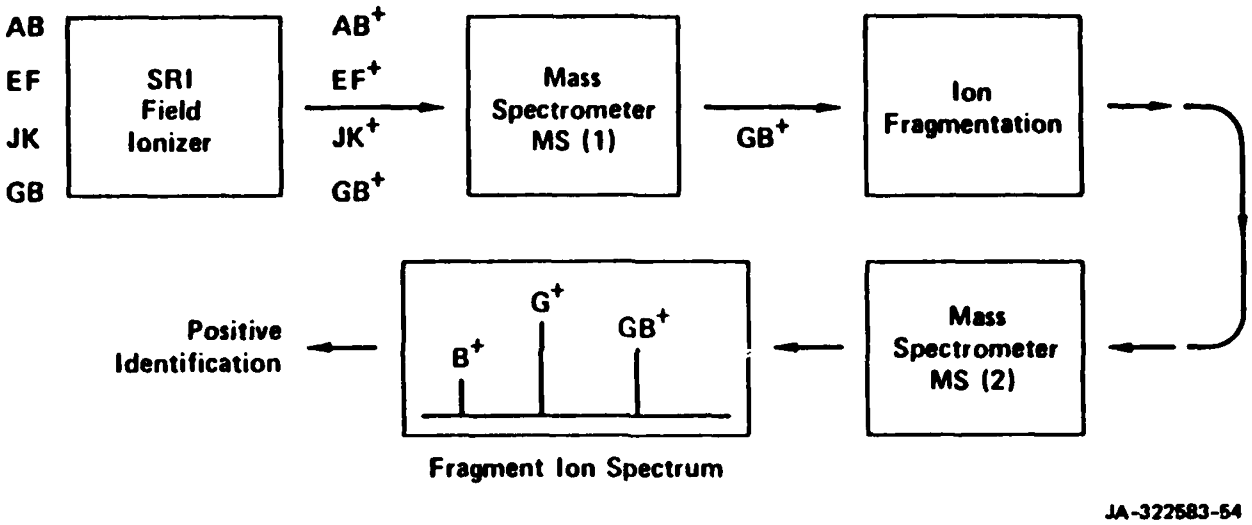

FIGURE 5. FIMS/MS CONCEPT

Pattern of ion fragments from mass selected molecular ion allows positive identification of chemicals, even in complex mixtures.

single-focusing magnetic sector that had also been built several years earlier at SRI and was also equipped with a volcano-style FI.

In designing the MS/MS system, it was necessary to decide which of the two mass spectrometers would serve as the first stage of the tandem instrument. The design with the quadrupole first and the magnetic sector second is called the QB geometry, whereas the reverse design is called the BQ geometry.

The advantages of the QB geometry are as follows:

a. The sample introduction system and ion source are at a low ground potential, and introducing samples through a membrane air sampling inlet, a direct vapor inlet, or a solids probe is performed easily.

b. Because the ions are accelerated to higher and higher energies as they progress through the instrument, the ion lens design is less critical and high, overall ion transmission can be achieved.

c. The production of a well-focused, low-kinetic-energy primary ion beam of a geometry suitable for electron impact fragmentation is achieved readily.

d. The magnetic sector can have very high transmission for fragment ions if z-direction focusing is provided.

e. Greatly accelerating the fragment ions immediately after they are formed means that acceptable resolution can be obtained from the magnetic sector and the peak width provides information on the amount of energy released in the fragmentation process.

The BQ geometry has the following advantages:

a. The mass range and mass resolution of the magnetic sector is higher at high mass, and therefore is better suited for the first stage of the tandem instrument.

b. The quadrupole mass filter is almost unaffected by several electron volts in the ion energy, making it well suited for separating the fragment ions that have an energy distribution typically broadened by the dissociation process.

c. The collisions between the mass-selected primary ions and a neutral gas jet can occur at higher relative kinetic energies, thereby increasing the probability of fragmenting the most stable of the molecular ions.

Primarily, because of its suitability for testing the electron impact fragmentation of ions, the QB geometry was chosen. Figure 6 shows a schematic drawing of the FIMS/MS instrument assembled under this project. At the left of the figure, the first item is the air lock for sample introduction. It provides three different means for introducing samples into the ion source of the mass spectrometer. Chemical vapors may be introduced through a leak valve connected to a piece of tubing designed to fit through

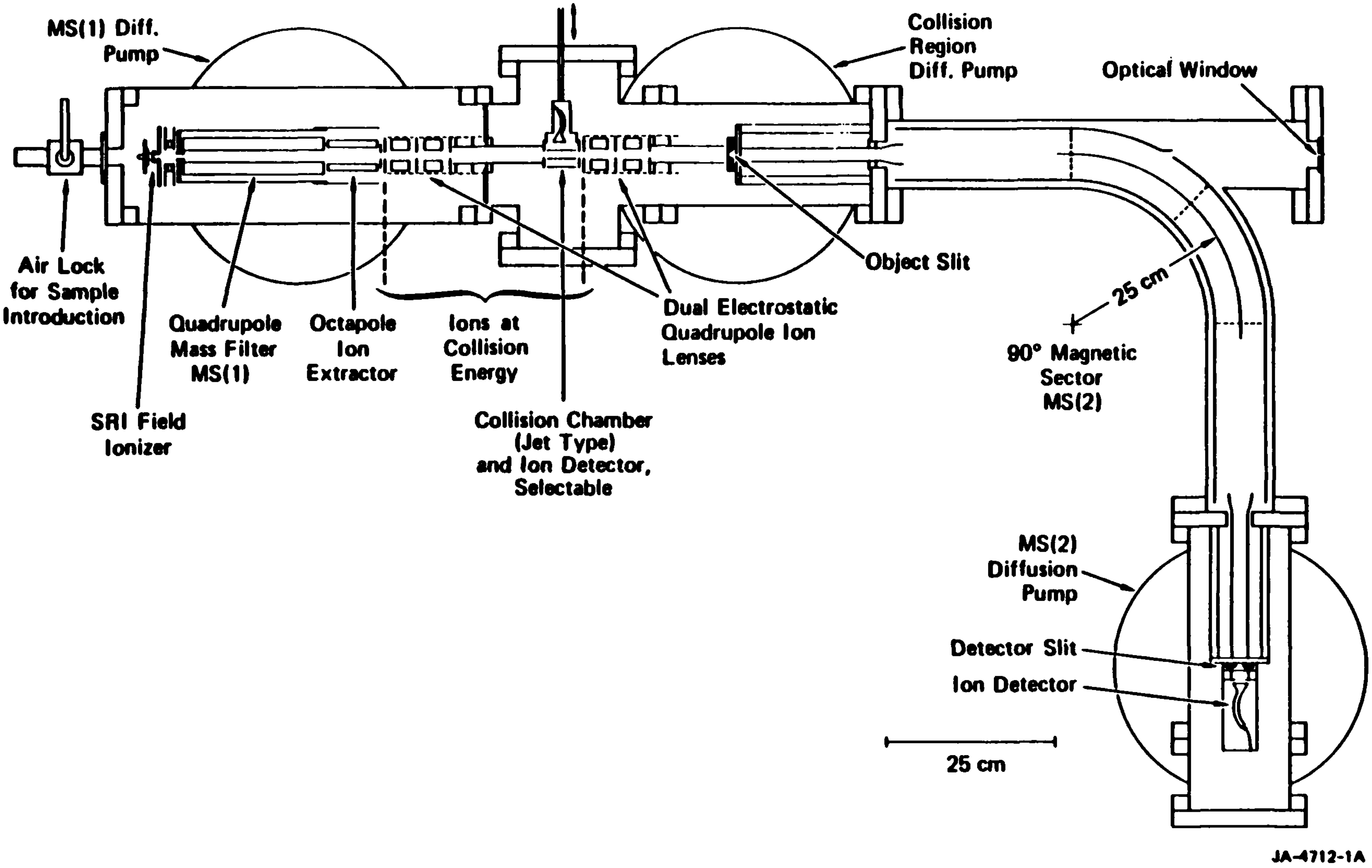

FIGURE 6. SRI FIMS/MS (QB Type)

the air lock and mate directly with the FI source. Samples in solid form may be introduced on the tip of a heatable probe which also mates with the back of the FI source. Finally, a silicone-membrane organic vapor separator can sample chemical vapors directly from the air in the ion source without introducing large quantities of air.

Ions are produced by the SRI FI source developed by Mr. Capp Spindt. These ions are focused with a reentrant focus electrode, and the resulting beam is directed by a single, electrostatic, quadrupole steering lens into the Extranuclear, quadrupole mass filter. The rods in this mass filter are 3/4 inch in diameter. Ions are removed from the quadrupole with an octopole lens ion extractor. To rapidly accelerate the ions exiting the quadrupole mass filter through the rf fringing fields, the octopole is normally operated at a mean potential approximately 100 volts negative. The octopole also reshapes the ion beam from the rough cross shape produced by the quadrupole into an approximately circular, cross section beam. The ions are then accelerated to the selected collision energy before being focused and steered by a set of dual, electrostatic, quadrupole ion lenses.

The quadrupole ion lenses have two separate functions. They convert the nearly round ion beam into a ribbon-shaped beam which is better suited for interaction with the neutral collision gas jet (the future electron beam for ion fragmentation) and the object slit of the magnetic sector. The second function of the quadrupole lenses is to correct small mechanical misalignments in the long ion beam path. The beam can be steered in both the y and z axes (the direction of ion travel is in the x direction).

The ion beam then enters the collision chamber which is shown in detail in Figure 7. The collision chamber contains three separate sections: (1) an ion multiplier section in which the primary ion beam is intercepted and deflected into a conventional ion multiplier for detecting and recording at this point, (2) a gas-jet collision region in the middle in which the focused ion beam collides with a jet of neutral gas, and (3) a third region in which the electron-impact, fragmentation experiment will be mounted. Figure 8 shows a schematic diagram with further details of the geometry of these three regions. The collision chamber is mechanically movable in the y-direction; thus, any one of the three regions may be moved into the path of the ion beam without opening the mass spectrometer vacuum system. The central region containing the neutral gas jet was the only one used in the experiments for this project. The gas jet is fabricated from a short length of 0.007-inch-ID stainless steel tubing. Large slots are cut in the top and bottom to allow excess collision gas to escape and be pumped away. When this region is aligned with the ion beam, the estimated conductance between the gas jet and the large, collision-region diffusion pump is about 30 liters/sec. By moving the gas jet across the ion beam while monitoring the signal from a fragment ion, it is possible to demonstrate that about one half of the ion fragments are formed as a result of collisions with gas in the jet and one half are caused by collisions with gas molecules that are inside the collision cell but not part of the jet itself.

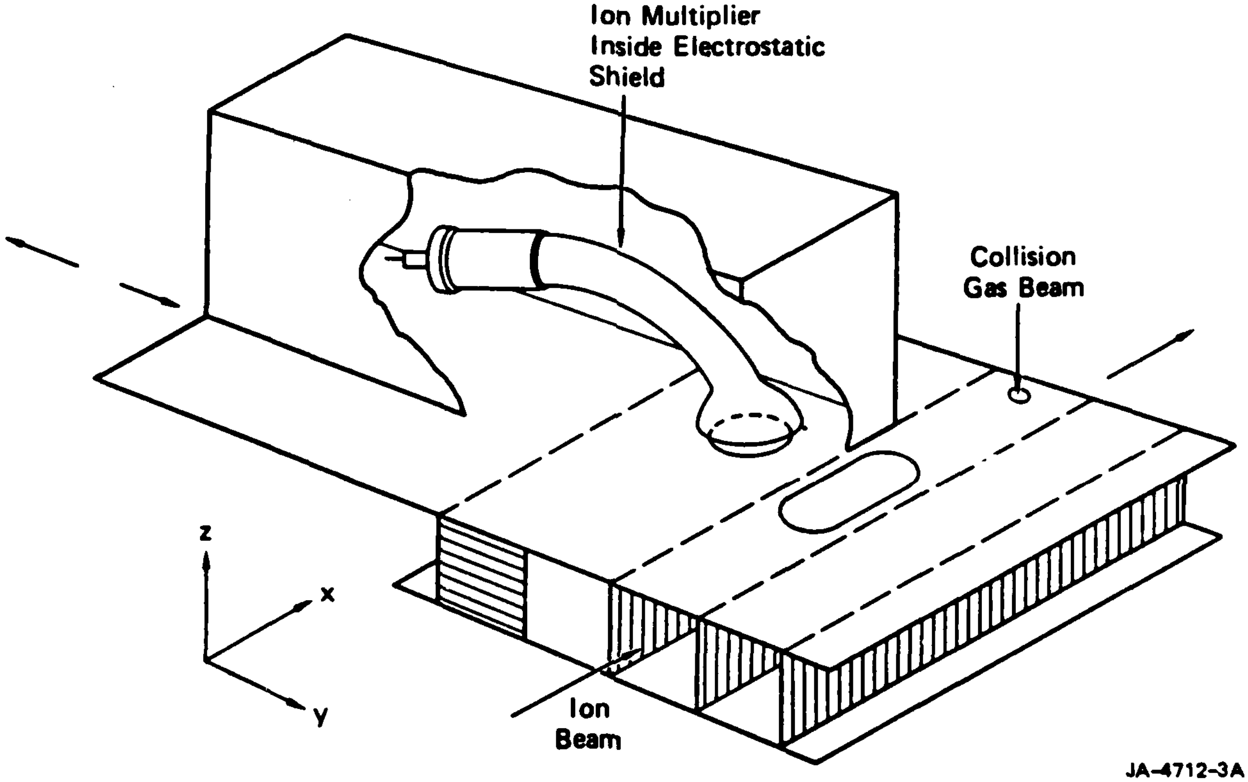

FIGURE 7. SCALE DRAWING OF THREE-SECTION COLLISION CHAMBER OF THE MS/MS SYSTEM

The ion beam is shown passing through the central gas collision section. Moving the chamber to the right brings the ion multiplier/electrometer detector into line with the ion beam. Moving the chamber to the left sends the ions into the electron collision region.

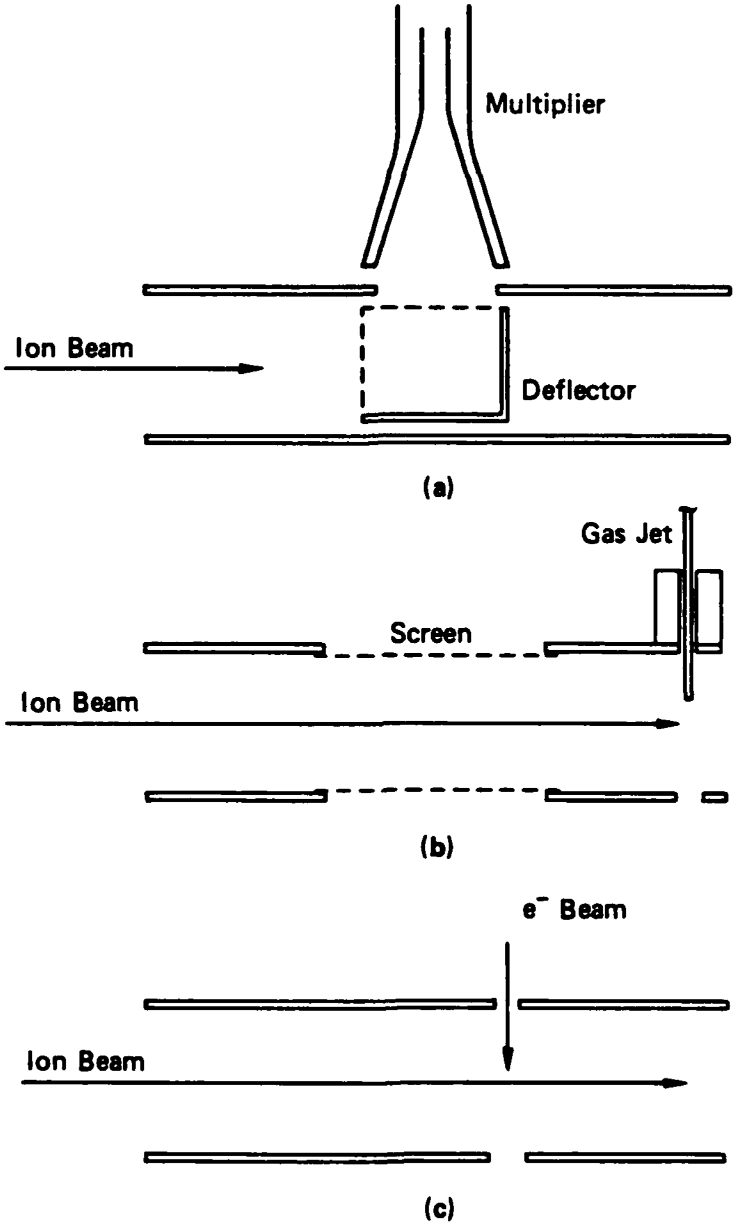

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FIGURE 8. SCALE DRAWINGS OF CROSS SECTIONAL VIEWS OF THE THREE PARTS OF THE MS/MS ION COLLISION CHAMBER

After passing through the collision chamber, the ion beam is accelerated to the final analysis energy. A second set of dual, electrostatic quadrupole lenses then refocuses the fragment ions and provides additional steering to center the ribbon-shaped ion beam on the object slit of the magnetic sector. Because the entire vacuum system is at ground potential, it was necessary to completely enclose the ion flight path with an electrically isolated shield biased at the appropriate potential. The magnetic-sector flight tube contains a liner constructed from 0.019-inch-thick stainless steel in four separate sections that can be inserted into the flight tube from one end. A side arm on the magnetic-sector flight tube is fitted with an optical quality window which makes it possible to use a small He-Ne laser to facilitate the mechanical alignment of the entire ion optical train. The magnetic-sector liner, which is operated most often at -3 kv, can operate at potential variables to -5 kv.

Ions leaving the magnetic sector pass through the variable width detector slit and directly onto the continuous dynode (Channeltron) multiplier. The multiplier is normally operated in the pulse counting mode with the output signal at ground potential.

The vacuum housing of the quadrupole mass filter is pumped by a Varian VHS-6 diffusion pump with a rated pumping speed of 1600 liters/sec. The other two diffusion pumps shown in Figure 1 are rated at 1200 liters/sec. All three pumps are equipped with water-cooled baffles and pneumatically operated gate values which make it possible to vent the vacuum system quickly to air for any necessary modifications during the development of the electron-impact, fragmentation method.

Figure 9 shows a block diagram of the ion detection electronics, the mass spectrometer control units, and the data recording equipment used to test the FIMS/MS. Samples of chemical vapor were introduced into the FI source by a direct vapor inlet. The pressure of the sample in the ion source was controlled by a variable leak valve. MS(1) was operated manually and set to pass the molecular ion of the compound under study. Argon was used as the collision gas in all cases. The amount of gas going to the collision jet was adjusted to produce a reading of 3×10^{-5} torr on the ion gauge located directly above the diffusion pump for the collision chamber. In practice, this meant that the primary ion beam was attentuated by 15-40 percent. MS(2) was scanned over a range that included the molecular ion and all of the fragments.

After mass analysis by MS(2), fragment ions enter the ion detector and produce negative going signal pulses. These are amplified and detected in the usual way using commercially available, pulse-counting electronics (Ortec Model 9301 preamplifier, Model 9302 amplifier/discriminator). The scanning of MS(2) is controlled by the x-axis output of the multichannel analyzer. As MS(2) is scanned, the ions detected are counted for a constant time into each of the 4,096 channels.

After several scans have been accumulated, the resulting spectrum can be plotted on an x-y recorder using the standard readout capabilities of the multichannel analyzer. The x-axis drive of the multichannel analyzer

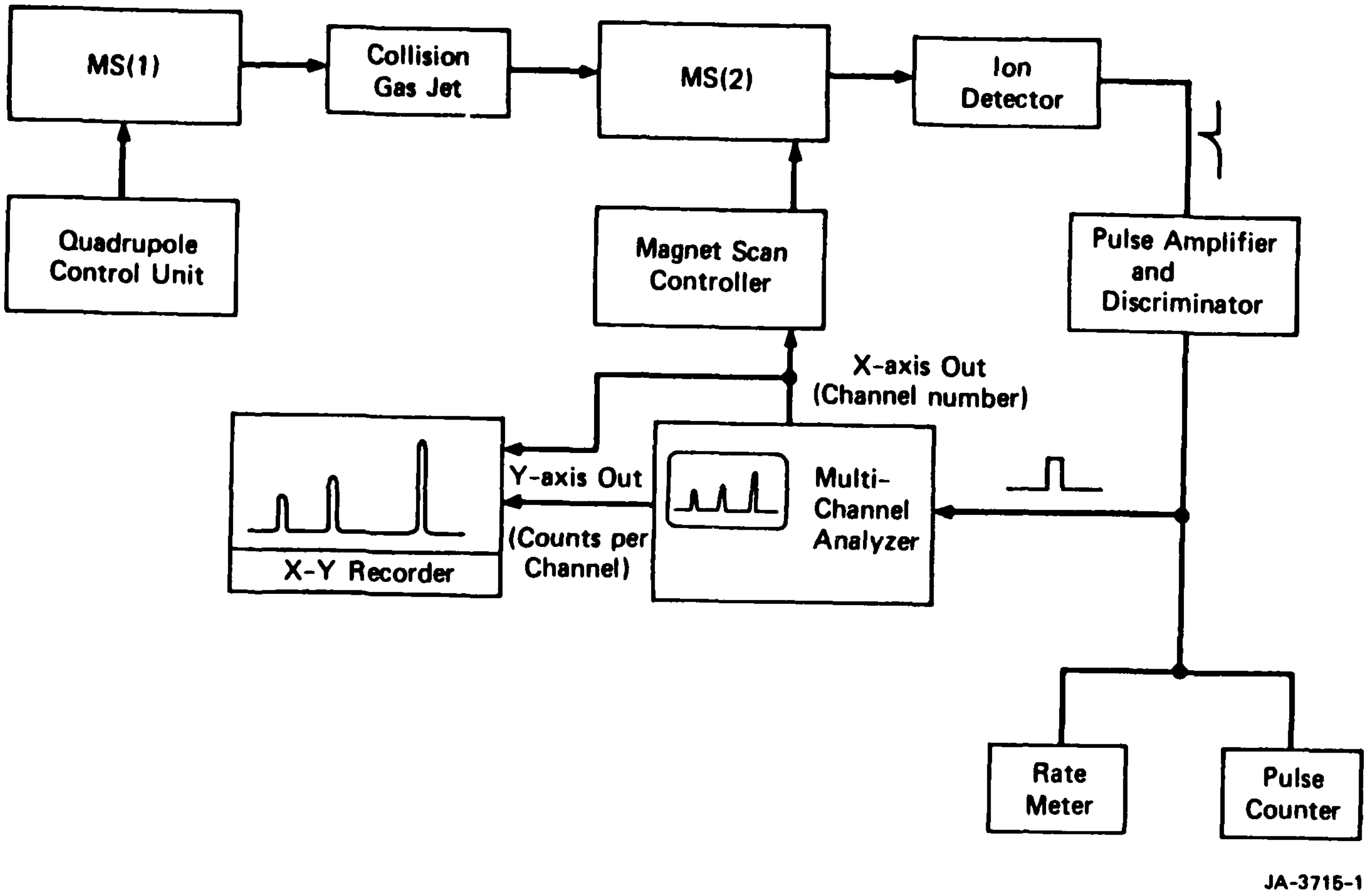

FIGURE 9. BLOCK DIAGRAM OF THE ION DETECTION, MASS SPECTROMETER CONTROL, AND DATA RECORDING ELECTRONICS FOR DEMONSTRATING THE MS/MS SYSTEM

is conditioned by the magnet scan control to produce a magnet scan that is approximately linear in mass for a normal, single-focusing mass spectrometer. However, because MS(2) is analyzing fragment ions that retain only part of the original energy of the molecular ion, the translational energy of a fragment ion as it passes through MS(2) depends upon the mass of the fragment ion and the mass of the molecular ion from which it was formed. Therefore, the mass scale for MS(2) is not linear. It changes when the mass of the molecular ion beam examined changes or when the collision energy changes. Although the situation is not satisfactory for routine operation, the system shown in Figure 9 demonstrated the performance of the FIMS/MS system.

4. RESULTS

Initially, we attempted to demonstrate the functioning of the FIMS/MS system using diisopropylmethyl phosphonate. Although we were able to obtain routinely FI spectra of this compound 2 years earlier, we found that the current versions of the volcano-style FI source lost all sensitivity when exposed to either diisopropylmethyl phosphonate or dimethylmethyl phosphonate. Therefore, other chemical compounds were used to demonstrate the operation of the FIMS/MS system.

Figure 10 shows the FIMS/MS spectrum of the molecular ion of hexylsulfide. In acquiring this spectrum, there was no mechanism for fragmenting the molecular ion because we turned the collision gas jet off. As a result, the spectrum shows only the molecular ion of hexylsulfide at m/z 202. The fragment ion spectrum is completely absent in the absence of collision gas. In Figure 10, this mass region is shown with 100 times greater sensitivity than that used to plot the hexylsulfide, molecular ion peak. The few small spikes on the baseline are the result of random background ion counts.

When the collision gas jet is turned on, the expected fragment ion spectrum appears, as shown in Figure 11. The fragment ion masses are understood readily in terms of the single bond cleavages indicated in the figure. This fragment ion spectrum is characteristic of hexylsulfide and bears a strong resemblance to the electron impact spectrum of this compound.

5. CONCLUSIONS

The FIMS/MS instrument was assembled and tested. Although the performance of the overall system was adequate, difficulties were encountered in ionizing the alkylphosphonates. To determine the source of this difficulty and to find ways to correct it, further investigative work is required.

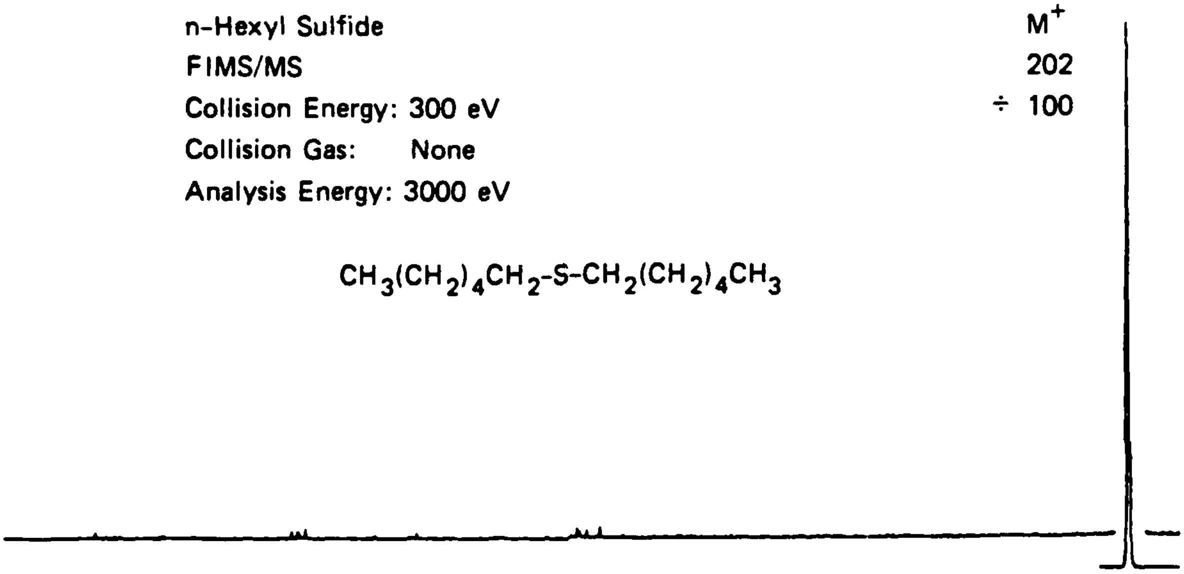

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FIGURE 10. FIMS/MS SPECTRUM OF THE MOLECULAR ION OF HEXYL SULFIDE WITHOUT ANY COLLISION GAS

There is no fragmentation. The small spikes in the spectrum are a few background ion counts.

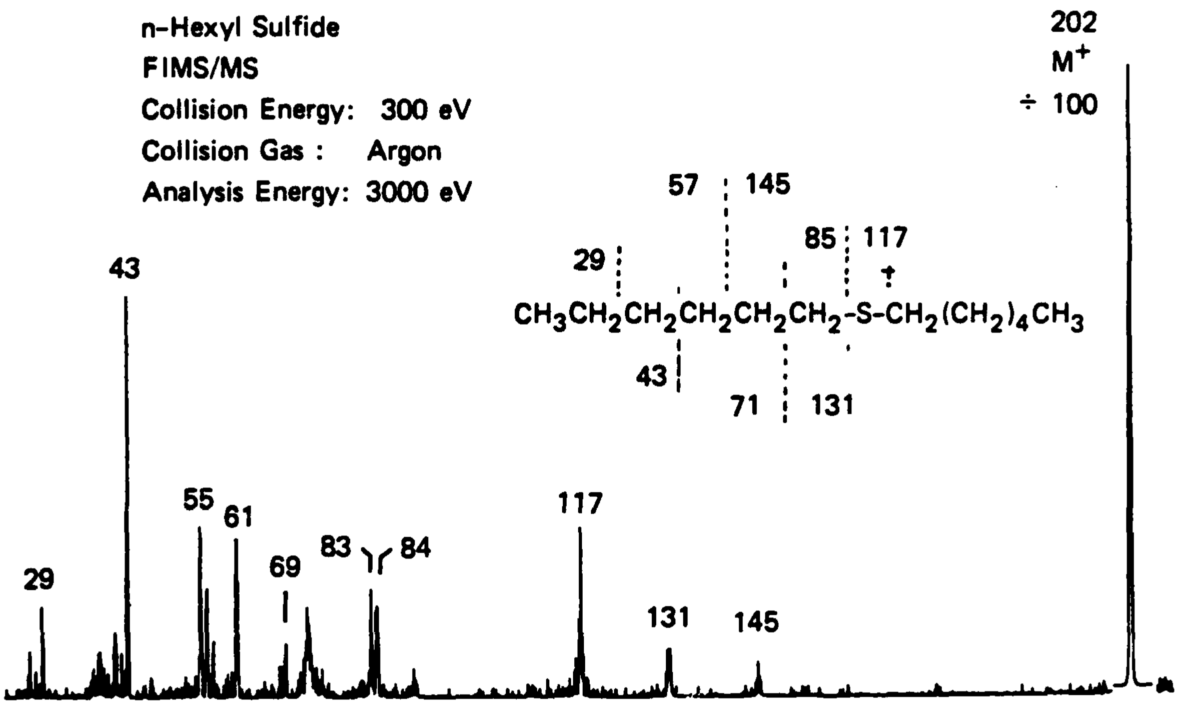

FIGURE 11. FIMS/MS SPECTRUM OF HEXYL SULFIDE WITH ARGON COLLISION GAS

The fragment peaks are the result of the indicated bond cleavages.

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