An ion source for molecular effusion studies

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AN ION SOURCE FOR MOLECULAR EFFUSION STUDIES

By
Don Merrill Jackson
D. E. Hudson

May 1959

Ames Laboratory
Ames, Iowa
F. H. Spedding, Director, Ames Laboratory.

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An Ion Source for Molecular Effusion Studies*

Don Merrill Jackson and D. E. Hudson

Abstract

An ion source which utilizes a beam of monoenergetic electrons as the ionizing agent was designed and built for use in a 60° sector general utility mass spectrometer. The source was made to be used without a magnetic field to collimate the electron beam. The source is interchangeable with a surface ionization source that was already utilized in the mass spectrometer. Various operating characteristics of the source were obtained, and the source was tested for linearity. If the source were perfectly "linear", the recorded ion current would be directly proportional to the atom current effusing into the ionizing region of the source. The source was estimated to be linear to within one percent.

Latent heats of sublimation of silver and thulium were obtained with the new source in the mass spectrometer. A value of 70.2 ± 0.7 kcal/M was found for the latent heat of sublimation of silver at 298°K. A value of 57.7 ± 0.8 kcal/M was found for the latent heat of sublimation of thulium at 298°K.

*This report is based on an M. S. thesis by Don Merrill Jackson submitted May, 1959, to Iowa State College. This report was done under contract with the Atomic Energy Commission.
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I. INTRODUCTION

A. Mass Spectrometry

A mass spectrometer is an instrument which separates a beam of ions according to mass. The mass spectrometer in its various forms (1) has a wide range of application. It is used in isotope identification or gas analysis work, in the determination of relative isotopic abundance, and in packing fraction and ionization potential experiments (1, 2, 3, 4, 5, 6, 7). The mass spectrometer is also used in the investigation of composition and thermodynamic properties of solids and liquids by observing the ion beam which represents the vapor in equilibrium with these solids (8, 9, 10). The work described in this report deals with one phase of mass spectrometer research—the design, construction and testing of an electron bombardment ion source to be used in the investigation of thermodynamic properties of various metals and metallic vapors.

A mass spectrometer is composed of four basic components: a sample system which introduces molecules from the material to be investigated into the mass spectrometer; an ion source which ionizes a certain fraction of these molecules thereby enabling them to be deflected in electric and magnetic fields and to be detected by electrical methods; an analyzer to separate the ions according to mass; and a collector-recorder.
system which measures and records the magnitude of the ion currents. A schematic drawing of the mass spectrometer used in this problem is shown in Figure 1. This figure shows the ion source region (a), the mass tube through which the ions are drawn into the analyzer (b), the analyzer magnet (c), and the collector region (d). The following sections describe briefly the individual components of the mass spectrometer.

1. The sample system

In order to investigate the vapor in equilibrium with a solid sample, the sample system shown in Figure 2 was utilized. The sample is contained in the small, closed tantalum crucible (c). The metallic vapor effuses from the small orifice (o) in the top of the cell. Under these conditions, the rate of effusion of molecules from the orifice is proportional to the equilibrium vapor pressure inside the cell which in turn increases with the cell temperature. The temperature of the Knudsen cell is controlled by varying the amount of electrical power dissipated in the coil windings (b). Lava insulators are indicated at a, d, s, and p in the figure, and molybdenum heat shields at e. The temperature of the Knudsen cell is measured by means of a Pt - 13 per cent Pt Rh thermocouple (t). Figure 3 shows how the cell and heater unit are mounted in the source chamber at k.
Figure 1. A block-schematic diagram of the mass spectrometer
Figure 2. The Knudsen effusion cell and heater unit

SCALE 4" = 1"

Figure 2. The Knudsen effusion cell and heater unit
Figure 3. A schematic view of the surface ionization source
2. The ion source

The only type of ion source used in this mass spectrometer had been a surface ionization source. This source utilizes a very hot tungsten ribbon as the ionizing agent. Figure 3 is a schematic view of the surface ionization source. The neutral atoms effuse from the Knudsen cell (k), pass through the hole in the slit plate (s), and strike the filament (f). A fraction of the neutral atoms are ionized at the surface of the filament and are drawn through the 0.006-inch wide upper slit (c), into the mass tube of the mass spectrometer. The ion beam passes through a 1/8-inch wide lower slit (d) before entering the mass tube. Theory concerning surface ionization can be found in the literature (1, 2, 11).

3. The mass analyzer

The mass analyzer (c in Figure 2) used in this spectrometer is a 60° sector, variable field electromagnet. The magnetic field may be varied from 2000 to 8000 gauss. In actual practice, the range of observed mass has been from mass 16 to mass 208. This range is mainly limited by the range of the magnet power supply.

4. Ion detection

Two methods of ion detection have been used in this mass
spectrometer. The first technique is the straight collection of ions in a simple Faraday collector. This collector is a cup-shaped electrode with a secondary suppressor grid. The second method of ion detection utilizes an electron multiplier ion detector which was developed especially for use in this mass spectrometer (12). Both ion detectors have output currents which are amplified by a vibrating reed electrometer. The ion current is recorded continuously on a Brown, strip-chart recorder (see Figure 1). When the Faraday collector is used, ion currents as low as $10^{-14}$ ampere have been recorded; whereas with the multiplier, it is possible to record ion currents as low as $10^{-18}$ ampere. In actual practice, the lower limit of ion current has been $10^{-16}$ ampere.

5. Vacuum system

During the operation of the mass spectrometer, three mercury diffusion pumps maintain a pressure of from $3 \times 10^{-6}$ to $2 \times 10^{-5}$ mm. of mercury in the source chamber and a pressure of $5 \times 10^{-7}$ mm. of mercury in the collector chamber. The pressure in the source chamber when the system is at room temperature is lower than $1 \times 10^{-6}$ mm. of mercury.

B. Objectives of this Research

The first object of this experiment was to design and
build a working electron bombardment ion source to be used in place of the surface ionization source mentioned in the preceding section. It should be stressed that it was not the object of this experiment to study ionization by electron bombardment, or the operation of electron bombardment ion sources in general.

The second object of this experiment was to test the completed ion source and to obtain as many of the operating characteristics of the source as possible. In connection with the testing of the source, attempts were to be made to obtain reliable latent heat of sublimation data. This experimental data when compared with that from other experiments would give an idea of the reliability of the source (see Sec. II).
II. DESIGN

A. A Brief Theory of Electron Bombardment Ion Sources

The operation of the mass spectrometer depends upon a source of ions all of which have the same electrical energy $qV$, where $q = ne = \text{net charge of the ion}$ ($e$ is the electronic charge and $n$ is an integer signifying the degree of ionization), and $V$ is the voltage through which the ions are accelerated into the analyzer field. These ions, which represent the various gas molecules present in the ionizing region, may be produced by several methods (1). Each method of ionization is capable of supplying sufficient energy to the molecule to cause it to lose one or more of its electrons. It is obvious that most of the molecules must be ionized to the same degree in order to maintain the quantity $qV$ constant. This report describes a type of ion source which employs a beam of monoenergetic electrons as the ionizing agent.

1. Ionization by electron collision (1, 13, 14, 15, 16, 17)

When a fast electron moves past an atom or molecule, the force between it and the electrons of the atom may result in the transfer of energy to the electrons, thus causing the excitation or ionization of the atom. The resulting energy loss slows down the fast particle. Eventually, after enough of such energy transfers, the fast particle will have too
little energy to excite or ionize an atom. In an ionization chamber containing a given density of neutral molecules, the rate of occurrence of this process is proportional to the number of colliding electrons, i.e. the rate of ionization is proportional to the electron current. In case a molecule is excited and not ionized, the excitation energy is lost through radiation, or the molecule can be ionized by a lesser transfer of energy in a second collision.

2. **Differential ionization**

Differential ionization is defined as the number of ions produced per centimeter of path by an electron as it passes through a gas at a given density. Differential ionization, when plotted as a function of electron energy, climbs sharply from zero at an energy corresponding to the ionization potential of the atom or molecule to a rather broad maximum ranging from 70 eV to 200 eV for elements from helium to mercury (2). In this report, the term "ionization efficiency" will be used synonymously with "differential ionization".

The term "ionization region" is used to denote a small volume within the ionization chamber of an electron bombardment ion source. This volume is the central portion of the electron beam directly above the ion beam defining slit (c in Figure 3). It has a length and width equal to the length and width of the slit. Its height corresponds to that of the
electron beam. It will be assumed that the measured ion beam originates in this region.

For a constant electron flux and gas density within the ionization region, the rate of ion production in this region, and therefore the ion current, would indicate the relative ionization efficiency of the source.

3. Extraction of ions from the source

Once an ion is formed, it must be drawn from the ionization chamber and through the mass analyzer. Only then can it contribute to the measured ion current. One technique used to extract the ions is to use a positively charged "pusher" electrode above the ionization region. Another method, and the one used with the source described in this report, makes use of the fraction of the ion accelerating field that leaks into the ionization chamber (15). The electron beam is deflected by these fields, thus it is necessary to carefully regulate the field in order to minimize this beam deflection and still draw the ions from the chamber.

The extraction of the ions is affected by space-charge effects within the chamber. Space-charge effects are present due to both the electrons in the electron beam and the positively charged ions.
4. **Linearity of the source**

It is assumed that in the mass spectrometer, the ion current reaching the collector after passing through the mass analyzer and slit systems is directly proportional to the ion current leaving the ionization region. The proportionality factor in this case is just a function of the geometry of the system.

Two types of linearity of an ion source must be defined. The first type of linearity gives an indication of space-charge effects within the electron beam. If the electron beam were well defined and the neutral atom density remained constant, an increase in electron beam current should bring about a proportional increase in ion current leaving the ionization region (assuming a constant ratio between ions formed and ions that leave this ionization region). When space-charge effects begin to occur, an increase in electron beam current would cause the beam to spread and thus the electron density in the ionization region would remain more or less constant. This space-charge effect would show up as a leveling off of an ion current-electron current plot.

The second and most important linearity of ion sources is the direct proportionality between the atom current entering the ionization region and the ion current leaving this region. An ion source without this linearity would be of limited worth in mass spectrometry. This second use of
the term "linearity" will be employed in this report. A way to check this type of linearity would be to control and measure the ingoing atom current directly and measure the resultant ion current. However, such a test is not entirely feasible because an independent measure of the neutral atom beam is difficult to obtain.

The resultant slope of a plot of ln (ion current) vs reciprocal Knudsen cell temperature yields (after thermodynamic treatment) the latent heat of sublimation of the sample inside the Knudsen cell (8, 9, 10, 18). If such latent heat data were obtained using a source that was not linear, the results would clearly be in error. Hence, agreement with more accurate experiments would be a fair indication that a source was at least linear to within experimental error of the instrument in which it was utilized.

5. Source limitations on resolution

In any electron bombardment ion source, it is imperative that the electron beam be confined to small enough a volume inside the ionization chamber so that the ions are formed essentially at an equipotential of the electric field used to withdraw the ions. Too large a distribution in ion energies due to the ions being formed at different equipotentials results in a loss of mass resolution at the ion collector. Another reason for keeping the electron beam
confined to as small a volume as possible is the fact that the electrons in collision with the walls of the ionization chamber give rise to secondary electrons and soft X-rays. These secondary ionizing agents would in turn give rise to a non-linearity in ion current since the experiment would essentially be carried on at a varying electron current. Secondary electrons released by collision of the electrons with the collector electrode would also contribute to the above effect.

In order to confine the electron beam to a small volume and to avoid space-charge effects, the electron beam is first collimated by a slit or hole system and then in addition to this, a magnetic field can be directed axially along the electron beam path. Most electron bombardment ion sources employ such a magnetic field (2, 3). The chief difficulty in using such a magnetic field lies in the fact that due to the crossed electric and magnetic fields, there is a mass discrimination within the source. This discrimination would perhaps affect the linearity of the source in that the number of a particular kind of ions leaving the ionization chamber would not be directly proportional to the number of neutral molecules entering the chamber from the sample.

6. **Background**

One of the main difficulties in using an electron bom-
bardment ion source is the fact that all varieties of molecules within the ionization region have a certain fraction of their number ionized. This means that a molecule of complex structure and the same mass as a sample molecule can contribute to the total ion current measured as representative of the sample. High resolution mass spectrometers (resolution being defined as $\frac{A m}{m}$) can distinguish between molecules of the same N+Z but different N to Z ratios. In other instances, it is advantageous to have a method for determining the background ion current which is superimposed upon the ion current representing molecules under examination.

Another contribution to the total background comes from multiply ionized heavier molecules or atoms. A doubly ionized atom would contribute to the ion current representing a singly ionized atom with half the mass.

B. Design Considerations

In order to build a working electron bombardment ion source, it was first necessary to examine critically the basic properties that such a source must have in order to be of practical use.

First, the source had to be designed to be used with the sample and slit systems already in use (see Figure 3). This would mean that it had to be more or less interchangeable with the surface ionizing source. Making the source
interchangeable with another type of source also would give indication of the reliability of the data obtained with the new one.

The new source had to be made simple and rigid. The various source components should be made in units, thereby allowing removal of one of the components without changing the rest of the source. Thus, it would be possible to change the electron filament or check the collector for beam alignment without removing the entire source. Simplicity and rigidness of construction would allow for easy replacement of parts. Also, the source could be cleaned and re-assembled without loss of geometrical configuration. This would be important in work which relies upon calibration of the source with a standard that is dependent upon geometrical factors.

It was decided that, although some researchers seem to feel that magnets are indispensable in an electron bombardment ion source (3), the source would be made so that a magnetic aligning field could be used only if necessary.

In addition to these major considerations, a technique for removing the ions from the ionization chamber and an efficient means of collimating the electron beam had to be developed. The parallel plate geometry of the entire source was also to be preserved.
III. CONSTRUCTION

A. Introduction

Schematic views of the electron bombardment ion source are seen in Figures 4 and 5. In general, the source consists of five basic parts: a source of electrons in the form of a directly heated filament (a); an electron beam collimating unit (b); an ionization chamber (c) in which the electrons collide with a beam of neutral atoms; an electron beam collector unit (d); and some means of extracting the ions from the ionization chamber which in this instrument is a grid plate (e) directly beneath the ionization chamber. Sources vary according to physical dimensions, techniques for extracting the ions, and methods used in the collimation of the electron beam.

The source works as follows. The electrons are accelerated from the hot filament through the collimating system, which defines the beam, and into the ionization chamber. A beam of neutral atoms (j) from the Knudsen cell (Figure 5, K) is directed into the ionization chamber. The electron beam and the neutral atom beam collide in the central region of the chamber. A fraction of the atoms which suffer collisions with electrons are ionized. A portion of the electric field between the ionization chamber and the grid plate (e) leaks into the ionization chamber and extracts a fraction of
Figure 4. A diagram of the major components of the electron bombardment ion source
Figure 5. A schematic view of the electron bombardment ion source
these ions. The ion beam (i) is defined by an upper slit (t) and lower slit (Figure 5, l) before it goes into the mass tube. After traversing the ionization chamber, the electrons pass through a grid across the end of the chamber and are then accelerated to the collector electrode. The neutral atom beam can be kept from reaching the ionization chamber if the shutter (Figure 5, g) is moved across the hole in the slit plate through which the beam must pass on the way to the chamber. A more complete description of this shutter follows in another section. The ion beam is centered on the lower slit when the electric field between the deflecting plates (Figure 5, n) is properly adjusted.

B. Description of Components

Detailed drawings of all source parts are included in Appendix B. Except for the lava insulators, all parts are made from non-magnetic stainless steel.

Figure 6 includes schematic drawings of the electron filament and collector units and the ionization chamber. Similar drawings of the collimating unit and the grid plate are found in Appendix B.

1. The electron filament unit

The source of electrons is a heated, 0.050" wide, 0.0005" thick, tungsten ribbon. The filament (Figure 6, a)
Figure 6. Diagrams of the various source components.
is mounted between two stainless steel pressure jaws (q). These jaws are mounted on a specially designed lava insulator that is fastened in position onto the upper mounting plate (Figure 5, m). The filament holder was designed so that it could be removed as a unit and the filament changed without having to remove the entire source from its vacuum chamber. The photographs in Figures 7 and 8 show the filament holder (a) in place on the upper mounting plate.

2. The electron beam collimating unit

The first attempts at electron beam collimation were to make use of a system of small holes in shaped electrodes as electro-lenses to focus the beam. This technique was not satisfactory (see Section IV-B). Further investigation showed the most efficient means of collimation to be a system of three, co-axial, one-mm. diameter holes with all three hole plates connected electrically to the ionization chamber. The electrons would thus acquire all of their energy between the filament and the first hole plate. The photograph in Figure 8 shows the collimating unit (b) in place on the ionization chamber (c). A detailed drawing of the collimating unit is found in Appendix B. The unit consists of three hole plates separated by 1/8-inch long stainless steel spacers. The holes were knife-edged in order to lessen beam dispersion due to collisions of the electrons with the hole
Figure 7. The electron bombardment ion source.
Figure 8. The major components of the ion source as seen from beneath the ionization chamber.
walls. The holes were aligned so that the electron beam was in the lower half of the ionization chamber in order to partially compensate for the pushing effect of the ion accelerating field, which leaked into the chamber.

3. The ionization chamber

Upon the suggestion of Dr. H. J. Svec of this Laboratory, the ionization chamber was made from a solid block of stainless steel. A two-dimensional, large scale model was made of the cross-section of the proposed ionization chamber. A rubber membrane was stretched over this cross-section and a gravitational potential was developed in the membrane. This potential was to represent the electrical potential between the ionization chamber and the grid plate. A small steel ball representing an ion in the chamber was placed on the membrane. The ball was found to roll, from rest, across the line on the membrane which represented the slot in the bottom of the chamber. Thus, by analogy, it was assumed that sufficient electrical field would leak into the chamber to extract the ions.

Figure 6 includes a schematic drawing of the ionization chamber (c). The 1/4 inch long notches (y) at either side of the slot in the bottom of the chamber allow the neutral atom beam to enter the chamber. The hole (s) in the chamber block is the hole through which the Knudsen cell is observed.
This hole was also used to observe the action of the shutter (Figure 5, g). The photograph in Figure 8 shows the ionization chamber (c) and the sighting hole (s).

4. **The collector unit**

Figure 6 includes a schematic drawing of the electron beam collector (d). The collector is held in position by means of two rods (u) secured in a small stainless steel bar (v). The bar is fastened to an L-shaped bracket (w). The bracket is then fastened to a lava insulator which is mounted onto the top mounting plate. The photographs in Figures 7 and 8 show the collector unit (d) in position as part of the source.

It was necessary to suppress the secondary electrons ejected from the surface of the collector. A small grid across the end of the ionization chamber serves to keep these secondaries from entering the chamber. The photograph in Figure 8 shows this grid (r) in place in front of the collector. A detailed drawing of this grid is included in Appendix B.

5. **Grid plate**

In the first tests of the ion source, the source was mounted directly over the upper slit plate (Figure 4, f). An electric field of approximately 3000 volts per cm. between
the slit plate and the chamber caused the electron beam to be deflected so badly that the collector current dropped to zero. When a grid (Figure 4, e) was placed between the slit plate and the chamber, it was possible to control the electric field effect on the electron beam. Thus, a field of approximately 200 volts per cm. between the chamber and the grid was found to have little effect on the beam, and the ions were still drawn from the chamber. The photographs in Figures 7 and 8 show the grid plate (e) in position. A detailed drawing of the grid plate is included in Appendix B.

C. Assembly Details

The assembled source is seen in the photograph in Figure 7. The main mounting apparatus consists of two stainless steel plates (Figure 5, m). The mounting plates are joined by four stainless steel spacers (Figure 7, z). The ionization chamber is fastened to the top mounting plate by means of two L-shaped brackets (Figure 6, x). The main function of the bottom mounting plate is to preserve parallel plate geometry in the source.

All electrical insulators and spacers were made from grade M lava. "Lava" is the trade name for hydrous aluminum silicate. Unfired lava can be machined like wood or brass. Firing converts lava into a very hard material with good electrical insulating properties. Detailed drawings of the
lava insulators are included in Appendix B.

D. The Shutter System

The basic design of the shutter system used in the source for the determination of the magnitude of the background ion current was developed by O. C. Trulson of this Laboratory. The shutter system is shown in place on the upper slit (Figure 5, f) plate in the photograph in Figure 9. The shutter (B) is actuated by the solenoids (A). The solenoid winding forms (F) are pyrex tubing, and the core (E) is a steel rod one millimeter in diameter. The shutter is fastened to the steel rod. By passing a surge of current of approximately six amperes through the solenoids, the shutter can be moved across the holes (C) in the slit plate through which the atom beam (Figure 4, j) from the Knudsen cell must pass to reach the ionization chamber. An arrangement of push-button switches and a filament transformer are used to activate the shutter.
Figure 9. The shutter assembly mounted on the upper slit plate.
IV. SOURCE OPERATING CHARACTERISTICS

A. Introduction

In the operation of the electron bombardment ion source described in this experiment, there are three variable parameters. The filament temperature, electron accelerating bias, and the collector bias can be varied independently. The electron beam current is closely related to both the filament temperature and the electron energy and will be considered to be an important secondary variable of the source.

The primary operating characteristics of the source were observed by noting the effect of the variation of these parameters on the ion beam and on the electron beam. These observations, or preliminary tests, were necessary for several reasons. The dependence of the electron beam current upon filament temperature would indicate optimum filament temperature for the operation of the source. A plot of ion current as a function of electron beam current would give an indication of one type of source linearity and also would show any space-charge effects within the ionization region. A plot of ion current as a function of electron energy should indicate whether or not the ionization efficiency of the electrons is nearly maximum in the region of 100 volts electron energy (2).

Perhaps the most important test of the source was its
actual production of an observable ion beam within the mass spectrometer. This phase of the tests came after most of the operating characteristics were obtained. Only the ion current vs. electron beam current plot was obtained with the source operating in the mass spectrometer. The other tests were made with the source placed in an auxiliary vacuum system. In these instances, the entire ion current drawn from the ionization chamber was collected at the upper slit plate itself (Figure 4, f). This ion current was large enough to be measured with an RCA ultrasensitive D.C. microammeter.

Figure 10 is a schematic drawing which shows the electrical circuitry necessary for the operation of the source. (The schematic represents the final design of the source and any variations in this design that were necessary in the different tests will be described in the following sections.) The current used in the heating of the filament (a) is furnished through a filament transformer (P) and averages approximately five amperes. The electrons are accelerated from the surface of the filament to the first hole (l) plate by means of the accelerating bias (bias 1) between the filament and the ionization chamber (c). The electrons then "coast" into the ionization chamber and pass through the suppressor grid (r) across the end of the chamber. They are then accelerated to the collector electrode (d). The col-
Figure 10. A schematic-wiring diagram of the ion source
lector bias (bias 2) serves to suppress secondary electrons that are ejected from the collector surface. This bias also serves to draw positive ions that are formed between the suppressor grid and the collector into the ionization chamber, so it must be carefully chosen. During the tests to obtain most of the operating characteristics of the source the collector bias was set at 16 volts. The power supply designed for the source (see Appendix A) sets the collector bias at 50 volts. These biases appear to be sufficient upon referring to charts of the ratio of secondary to primary current as a function of primary energy for various electrode materials (19). The lower bias was chosen in the preliminary tests since the primary electron energy would be varied from zero.

The total electron emission current from the filament is indicated by meter 2 (Figure 10). The electron beam current reaching the collector is indicated by meter 3. The accuracy of meters 1 and 2 is not important in the operation of the source. It is not essential to accurately know the magnitude of either the filament-heating current or the total electron emission current. The five per cent accuracy of meter 3 (see Appendix A) is not entirely adequate because the relative magnitude of the ion current would be controllable to within only five per cent, and this accuracy in ion beam control would not be adequate in certain experiments (such as direct vapor measurements which would depend on a cali-
A regulated power supply designed by Dr. H. J. Svec (20) of this Laboratory was built for use with the ion source. The power supply is set to furnish the electrons with sufficient energy (bias 1) for optimum ionization efficiency of the heavier elements - 85 volts (1, 2). A complete description of this power supply is included in Appendix A.

The linearity of the source (see Section II) was best determined by observing the magnitude of the ion current as a function of the controlled current of neutral atoms which entered the ionization chamber from the Knudsen cell. This test made up a major part of this experiment and will be included in the next section in which the latent heats of sublimation of different metals are discussed. Also, the use of magnets within the source will be discussed in the next section since these magnets have a marked effect on latent heat data.

B. Preliminary Tests

The first tests that were made on the electron bombardment ion source were to determine whether or not it would be possible to focus the electron beam in much the same way as the electron beam is focussed in a cathode ray oscilloscope. A series of three cylindrically shaped electrodes (Figure 10, holes 1, 2, 3) which had diameters of 0.25 inch, and
hole diameters of one mm., were connected so that any combination of potentials totaling 100 volts could be placed between the different electrodes and the electron filament (Figure 10, a). The focusing, and thus dispersion, of the electron beam was determined by placing a probe inside the ionization chamber. This copper wire probe was made in the shape of a ring. The inside diameter of the probe was 0.25 inch, and it was adjusted so it would not touch the ionization chamber. The electron beam would pass axially through the center of the probe, which was positioned in the ionization chamber approximately three-fourths of the distance from the focusing unit, to the suppressor grid. It was placed at the same electrical potential as the ionization chamber so that the electrons would not "see" it. A low resistance ammeter (RCA, Model WV-84A, ultra-sensitive, D.C. microammeter) was used to measure the current to the probe. Using dry cells and high resistance potentiometers (5 meg.-1 W.), different biases were placed on the focusing electrodes (Figure 10, holes 1, 2, 3) and the probe and collector currents measured and compared. The minimum probe-to-collector current ratio was obtained when all of the focusing electrodes were connected (as shown schematically in Figure 10) and the electrons received all of their energy between the filament and the first electrode (Figure 10, hole 1). Under these conditions, when the filament was observed to have a temperature of 2030°C.
and the electrons had an electrical energy of 75 volts, the probe to collector current ratio was found to be $3.6 \times 10^{-5}$. The ratio of the cross section area of the probe to the open area enclosed by the probe is approximately 0.345. It is evident that most of the current was therefore going through the center of the probe and thus the amount of current striking the walls (i.e., the dispersion of the beam) was very small compared to the current reaching the collector electrode. Actual observation of the collector electrode showed that the impinging electron beam left a spot on the electrode surface that was approximately 3/16 inch in diameter. After these results were obtained, the final design of the electron beam collimating unit was worked out. The collimating unit was described in Section II.

C. Operating Characteristics

1. Electron beam current vs. filament temperature

For this test, the complete source (Figure 5) was set up in an auxiliary vacuum system. The circuit of Figure 10 was utilized. The accelerating bias (bias 1) was maintained by a combination of dry cell batteries—depending on the required voltage. The collector bias (bias 2) was set at 16 volts through the use of a dry cell battery. The electron beam current was measured on an RCA, Model WV-84A, ultra-sensitive D.C. microammeter (Figure 10, meter 3).
filament temperature was measured with an L & N number 8622-C optical pyrometer.

The actual data in these runs were taken by setting the filament temperature, varying the electron voltage and observing the resulting electron beam current. The data were plotted as electron current vs. filament temperature with the electron energy held constant. Figure 11 shows a family of these curves. The electron energy for each curve is indicated.

The most useful result of this test is the fact that if the filament is run at a temperature higher than 2000°C, there is no gain in electron beam current.

2. Ion current vs. electron energy

For this test, the source was placed in an auxiliary vacuum system. The source was wired as shown schematically in Figures 10 and 12. The accelerating bias (Figure 10, bias 1) was maintained by a regulated, metered, 0 - 300 vdc, power supply (Kepco, Model 815 B). The collector bias (Figure 10, bias 2) was set at 16 volts through the use of a dry cell battery. A 90-volt grid bias (Figure 12, bias 3) was placed between the ionization chamber (Figure 12, c) and the grid plate (Figure 12, e). This bias furnished the electric field which caused the ions to be drawn from within the ionization chamber. The total voltage through which the ions
Figure 11. The electron collector current as a function of filament temperature
Figure 12. A schematic-wiring diagram showing the biases used to accelerate the ions to the upper slit plate.
were accelerated (Figure 12, bias 4) was set at 1890 volts through the use of a regulated, 0 - 5000 vdc, power supply (Instrument Development Laboratories, Model 1090). The ion current was collected at the slit plate (Figure 12, f) and measured with an ultra-sensitive dc microammeter (RCA, Model WV - 84A).

Only one curve of ion current vs. electron energy was obtained. The electron beam current was affected by changes in electron energy; therefore, it was necessary to have sharp control over the filament temperature in order to maintain a constant beam current. This plot of ion current vs. electron energy is shown in Figure 13.

It is evident from the curve that the ionization efficiency of the electrons begins to reach a maximum at energies near 100 volts. This efficiency is representative of the entire background gas within the ionization chamber.

3. Ion Current vs. Electron Current

Since the electron energy (Figure 10, bias 1) is fixed in the power supply used with the source, the only control over the magnitude of the ion current in the mass spectrometer is through the electron beam current. Therefore, it is necessary to know the manner in which the ion current is affected by changes in the electron current. In particular, if space-charge effects were occurring within the ionization
Figure 13. Ion current collected at the upper slit plate as a function of electron energy.
chamber, an increase in electron current would not cause a corresponding change in ion current. A plot of ion current vs. electron current would give an indication of any space-charge effects since under these effects such a curve would show a maximum.

For these tests, the source was placed on the mass spectrometer (so naturally the source was found to work previous to these tests). The power supply was used during these tests and the wiring circuit of the source was exactly as shown in Figure 10. A bias of 85 volts (Figure 12, bias 3) was placed between the ionization chamber and the grid plate. The ion accelerating bias (Figure 12, bias 4) was maintained at 1890 volts by a regulated dc power supply (this power supply is part of the electronics system of the mass spectrometer). The ion current was recorded on a Brown strip-chart recorder (see Figure 1). The electron current was varied in steps from zero to 65 μa (which represents the maximum regulated electron current obtainable with the power supply). The electron current was measured on a 0-100 μa dc microammeter (Simpson, model SK-525-9).

A plot of ion current as a function of electron current is shown in Figure 14. This curve represents several such curves which were obtained. The ion current shown in Figure 14 represents a background gas that was assumed to be at a constant density within the ionization chamber. It is evident
Figure 14. Recorded ion current as a function of electron collector current.
that no space-charge effects set in within the range of operation of the source.

D. Operation of the Source in the Mass Spectrometer

When the source is in the mass spectrometer, it operates at a potential of 1890 volts positive with respect to the upper slit plate which is grounded (see Figure 12). The high voltage lead to the source is fastened directly to the ionization chamber, so that the biases described in Figure 10 are relative to the chamber and not to ground.

One of the objectives of this experiment was to build an ion source that is capable of furnishing a recordable beam of ions in the mass spectrometer. The source was found to work, and this section will contain a discussion of its operation.

With the source in operation, the mass range was scanned. This was done through the continuous variation of the current through the coils of the analyzer magnet. The records of the various ion peaks were made on the Brown strip-chart recorder. A tracing of the recorder chart of a typical scanning run is shown in Figure 15.

1. The mass spectrometer constant

In order to be able to identify the various mass peaks, it was first necessary to determine the mass spectrometer constant \( k \) given in the equation
Figure 15. Recorded ion current as a function of analyzer magnet field
\[ M = k \frac{H^2}{V} \]  

where \( M \) is the mass of the collected isotope in atomic mass units, \( H \) is the analyzer magnet flux density in gauss, and \( V \) is the voltage through which the ions were accelerated. The constant was found through the identification of the most abundant isotope \((208)\) of mercury. The value was found to be:

\[ k = (1.03 \pm 0.04) \times 10^{-2} \]

Once the constant had been determined, it was possible to identify the various mass peaks according to the mass, in atomic mass units, of the molecules they represented. In Figure 15 the larger mass peaks have been identified, and in some cases an attempt was made to name the most probable element or compound represented. It must be remembered that various combinations of elements can give rise to a given mass peak. These complex molecules are the main cause of background in the source.

2. **Resolution**

Two different elements were used in the calibration of the mass spectrometer—silver and thulium. Figure 16 shows tracings of recorder chart records of these two elements. The two isotopes of silver and the one isotope of thulium are evident. Since the isotopes of silver (which differ by two
Figure 16. Tracings of the Brown recorder chart records for thulium and silver
a.m.u.) are distinctly separated, it would seem that it would be possible to distinguish between two isotopes that differ by only one a.m.u. at this point in the mass range. Thus, an optimistic value of the resolution ($\Delta m/m$) would be 0.01, while a value of 0.02 would be an upper limit.
V. THE DETERMINATION OF SOURCE LINEARITY -
LATENT HEATS OF SUBLIMATION

A. Introduction

The obtaining of reliable latent heat of sublimation data would serve two purposes. First, these data would give an indication of the linearity of the source; and second, if the data were found to be accurate, the actual latent heat values would be useful in themselves.

In connection with this phase of the testing of the source, the latent heats of sublimation of silver and thulium were obtained. These elements were investigated because the vapor pressures of both elements had been measured in other experiments (21, 22), and the latent heat of sublimation of thulium had been determined by mass spectrometric methods which utilized a surface ionization source (9).

A complete theory underlying the technique used to find latent heats of sublimation by mass spectrometric methods is found in the literature (8, 9).

B. Theory

The fundamental equation of this method for obtaining latent heats is:

$$\Delta H_T = -R \frac{d \ln I}{d(1/T)} + RT_m,$$

(2)

where $T_m$ is the mid-range temperature of the run, $\Delta H_T$ is the
latent heat of sublimation corresponding to that temperature, $I_0$ is the recorded ion current, and $R$ is the universal gas constant. Thus, latent heats of sublimation are obtained from plots of $\ln I_0$ vs. $(1/T)$ curves.

The above relation relies on the fact that the ratio of collected ion current ($I_0$) to the atom current ($I_a$) effusing into the ionization region is constant, or, $I_0/I_a = \alpha$.

Therefore,

$$\frac{d \ln I_a}{d (1/T)} = \frac{d \ln I_0}{d (1/T)} - \frac{d \ln \alpha}{d (1/T)} .$$

(3)

If $\alpha$ were not constant but a function of the Knudsen cell temperature, Equation 2 would become:

$$- R \frac{d \ln I_0}{d (1/T)} + RT_m = (\Delta H_T - \beta) = \Delta H_T(\text{Obs}) ,$$

(4)

where $\beta = R \frac{d \ln \alpha}{d (1/T)}$ is a non-linearity term. Hence, the difference between the true and observed values of the latent heat would give an indication of the linearity of the source, or:

$$\Delta H_T(\text{True}) - \Delta H_T(\text{Obs.}) = \beta(T) .$$

(5)

Clearly, if $\alpha$ is constant, $\beta$ is zero and $\Delta H_T(\text{Obs.}) = \Delta H_T(\text{True})$.

C. Procedure

Ion peaks were identified as representative of the Knud-
sen cell sample in the following way: The shutter (Figure 5, g) was adjusted so that the neutral atom beam from the Knudsen cell was cut off before it reached the ionization chamber. If, when this was done, the magnitude of the recorded ion peak dropped off sharply, the peak was assumed to represent the cell sample. Figure 17 shows what happened when the shutter was used to interrupt a beam of neutral silver atoms which were effusing from the cell.

The magnitude of the background under the silver mass peak is indicated in Figure 17. The absolute magnitude of the silver ion beam was obtained by subtracting the background from the current that was observed when the shutter was open.

The procedure for the obtaining of latent heat data was as follows: The mass peak that represented the cell atoms was found, and the cell temperature was raised until a maximum value was reached in either cell temperature or ion current. The temperature was then decreased in small steps, and, when equilibrium was reached within the cell at each temperature, the temperature and ion current were recorded on the Brown strip chart recorder. Equilibrium conditions were observed when the recorder trace of the ion current remained steady. Several runs were made with each cell sample. The temperature was measured with a Pt-Pt 13 per cent Rh thermocouple (Figure 2, t).

Instead of one's subtracting it from the ion current, the
Figure 17. A tracing of the Brown recorder chart record showing the effect of the interruption of the neutral silver atom beam effusing from the Knudsen cell.
background could be accounted for in the following way: in this case, the shutter was closed, and the recorder itself was zeroed. This zeroing-out of the background made it possible for a wider range of ion current to be covered. During these experimental runs, the ion current ranged from $1 \times 10^{-16}$ to $1 \times 10^{-13}$ ampere.

D. Evaluation of Data

The general method for the treatment of the data and a discussion of errors are found in reference (9). In addition to the error analysis included therein, there was an error ascribed to the latent heat values due to the reduction of these values to the reference temperature ($298^\circ$K). The data were reduced according to information given in tables compiled by Stull and Sinke (22). O. C. Trulson (10) has suggested that values for errors in data reduction be assumed according to the reliability of the Stull and Sinke data. This error was assumed to be negligible for silver because the thermodynamic data tabulated for silver were considered to be accurate to less than 1 per cent. Since the thermodynamic data were largely estimated in the case of thulium, the error due to reduction was assumed in the manner used by Trulson (10) in similar cases. Thus, the error in data reduction for thulium was estimated to be 0.02 kcal/M.

The orifice (Figure 2, o) in the Knudsen cell expands
slightly in the process of heating the cell during an experimental run. A small correction must be made in the latent heat to account for this expansion. The corrections used were obtained from information compiled by Savage (9).

E. Results

1. The latent heat of sublimation of silver

Figure 18 is a plot of the data for one of the experimental runs on silver. Table 1 contains the experimental data, list of errors and final value of the latent heat of sublimation of silver. The weighted mean value of the latent heat of silver was found to be $70.2 \text{ kcal/M at } 298^\circ\text{K}$. The error in $\Delta H_T$ arising from temperature measurement was 0.29 kcal/M. The standard deviation of the mean was found to be 0.64 kcal/M. The error in data reduction for silver was assumed to be negligible. The final value for the latent heat of sublimation of silver is

$$\Delta H_{298} = 70.2 \pm 0.7 \text{ kcal/M}. \quad (6)$$

This result can be compared with latent heat values obtained from at least one other source: from Stull and Sinke (22) data, a value of

$$\Delta H_{298} = 66.6 \pm 1.0 \text{ kcal/M}$$

was calculated for the latent heat of sublimation of silver.
Figure 18. A typical plot of silver sublimation data

Silver Run 4

\[ \text{SILVER RUN 4} \]

\[ R_x b = R_x \text{ SLOPE} = -63.19 \text{ K CAL/M} \]

\[ T_m = 1307^\circ \text{K} \]

\[ \Delta H = 64.48 \text{ K CAL/M} \]

\[ R_x S_b = 0.58 \text{ K CAL/M} \]
Table 1. The experimental slopes, corrections, and errors used to determine the latent heat of sublimation of silver metal

<table>
<thead>
<tr>
<th>Run</th>
<th>Midrange temperature $T_m$ (°K)</th>
<th>Experimental slope $-bR$ (kcal/M)</th>
<th>Temperature term $R T_m$ (kcal/M)</th>
<th>Latent heat $\Delta H_T$ (kcal/M)</th>
<th>Weight $w_i$</th>
<th>$1/S_b^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1306</td>
<td>62.787</td>
<td>2.592</td>
<td>65.379</td>
<td>21.505</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1293</td>
<td>64.674</td>
<td>2.570</td>
<td>67.244</td>
<td>1.999</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>1272</td>
<td>66.084</td>
<td>2.528</td>
<td>68.612</td>
<td>6.609</td>
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<tr>
<td>4</td>
<td>1307</td>
<td>63.186</td>
<td>2.596</td>
<td>65.782</td>
<td>11.775</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1345</td>
<td>60.966</td>
<td>2.672</td>
<td>63.638</td>
<td>15.926</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1330</td>
<td>65.316</td>
<td>2.642</td>
<td>67.958</td>
<td>13.443</td>
<td></td>
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<tr>
<td>7</td>
<td>1346</td>
<td>62.167</td>
<td>2.674</td>
<td>64.841</td>
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<tr>
<td>Mean</td>
<td>1314</td>
<td></td>
<td></td>
<td>65.656</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Standard deviation of mean, $S_m^b$ 0.644 kcal/M
Instrumental error, $e_m$ 0.294 kcal/M
Net experimental error, $E_m$ 0.708 kcal/M
Orifice correction -0.050 kcal/M
Error in data reduction, $e_r$ negligible
Latent heat of sublimation at 1314°K 65.7 ± 0.7 kcal/M
Latent heat reduced to 298°K 70.2 ± 0.7 kcal/M

\[ a\Delta H = \frac{\sum w_i \Delta H_i}{\sum w_i} \]

\[ b_{S_m} = \left[ \frac{\sum w_i d_1^2}{(n-1)\sum w_i} \right]^{1/2} \]

where $d_1 = (\Delta H_i - \Delta H)$. 

2. The latent heat of sublimation of thulium

Figure 19 is a plot of the data for one of the experimental runs using thulium. Table 2 contains the experimental data, list of errors and final value of the latent heat of sublimation of thulium. The weighted mean value of the latent heat of thulium was found to be 57.7 kcal/M at 298°C. The error in $\Delta H_T$ arising from temperature measurement was 0.31 kcal/M. The standard deviation of the mean was found to be 0.69 kcal/M. The error in data reduction for thulium was assumed to be 0.02 kcal/M. The final value for the latent heat of sublimation of thulium is

$$\Delta H_{298} = 57.7 \pm 0.8 \text{ kcal/M}.$$  \hspace{1cm} (7)

This result can be compared with information obtained from at least two other sources:

Savage (9) quotes a value of

$$\Delta H_{298} = 57.6 \pm 0.4 \text{ kcal/M}$$

for the latent heat of sublimation of thulium. This result was obtained through an experiment which utilized the mass spectrometric technique but which employed the surface ionization source.

Spedding, et al. (21) quote a value of

$$\Delta H = 57.4 \pm 0.20 \text{ kcal/M}$$
THULIUM RUN 2
RxB = Rx SLOPE = -56.83 K CAL/M
Tm = 1053 °K
ΔH_T = 57.88 K CAL/M
RxSb = 0.677 K CAL/M

Figure 19. A typical plot of thulium sublimation data
Table 2. The experimental slopes, corrections, and errors used to determine the latent heat of sublimation of thulium metal

<table>
<thead>
<tr>
<th>Run</th>
<th>Midrange temperature $T_m$ (°K)</th>
<th>Experimental slope $-bR$ (kcal/M)</th>
<th>Temperature term $RT_m$ (kcal/M)</th>
<th>Latent heat $\Delta H_T$ (kcal/M)</th>
<th>Weight $1/S_b^2$</th>
<th>Instrumental error, $e_m$ (kcal/M)</th>
<th>Net experimental error, $E_m$ (kcal/M)</th>
<th>Orifice correction (kcal/M)</th>
<th>Error in data reduction, $e_r$ (kcal/M)</th>
<th>Latent heat of sublimation at 1073°K (kcal/M)</th>
<th>Latent heat reduced to 298°K (kcal/M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1076</td>
<td>56.737</td>
<td>2.138</td>
<td>58.875</td>
<td>3.983</td>
<td>0.308 kcal/M</td>
<td>0.756 kcal/M</td>
<td>-0.030 kcal/M</td>
<td>0.020 kcal/M</td>
<td>56.1 ± 0.8 kcal/M</td>
<td>57.7 ± 0.8 kcal/M</td>
</tr>
<tr>
<td>2</td>
<td>1053</td>
<td>56.836</td>
<td>2.092</td>
<td>58.928</td>
<td>2.933</td>
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<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>3</td>
<td>1082</td>
<td>53.19</td>
<td>2.150</td>
<td>55.609</td>
<td>0.834</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>1082</td>
<td>54.04</td>
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<td>56.190</td>
<td>25.292</td>
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<td></td>
</tr>
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<td>5</td>
<td>1074</td>
<td>52.365</td>
<td>2.134</td>
<td>54.499</td>
<td>12.849</td>
<td>0.308 kcal/M</td>
<td>0.756 kcal/M</td>
<td>-0.030 kcal/M</td>
<td>0.020 kcal/M</td>
<td>56.1 ± 0.8 kcal/M</td>
<td>57.7 ± 0.8 kcal/M</td>
</tr>
<tr>
<td>Mean</td>
<td>1073</td>
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<td></td>
<td>56.114</td>
<td></td>
<td>0.690 kcal/M</td>
<td>0.308 kcal/M</td>
<td>0.756 kcal/M</td>
<td>0.020 kcal/M</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Standard deviation of mean, $S_m$ = 0.690 kcal/M

Instrumental error, $e_m$ = 0.308 kcal/M

Net experimental error, $E_m$ = 0.756 kcal/M

Orifice correction = -0.030 kcal/M

Error in data reduction, $e_r$ = 0.020 kcal/M

Latent heat of sublimation at 1073°K = 56.1 ± 0.8 kcal/M

Latent heat reduced to 298°K = 57.7 ± 0.8 kcal/M

*aRefer to Table 1.

for the latent heat of sublimation of thulium at 973°K. This result was obtained through direct vapor pressure measurements. When this value of the latent heat was reduced to 298°K (using Stull and Sinke data), a value of

$$\Delta H_{298} = 58.8 ± 0.20 \text{ kcal/M}$$

was obtained.
F. The Effect of Source Magnets

Experimental data were also obtained from the source when small permanent magnets were used in an attempt to further collimate the electron beam. Although no yoke was used, these magnets caused the size of the spot on the collector to be reduced: i.e., the electron beam was collimated better when the magnets were used. A magnetic field effect on the latent heat data of silver was observed. The experimental runs made with the magnets in place yielded slopes that were consistently 5 to 10 kcal/M less than slopes obtained when the magnets were not in place. In order to show the contrast between the two sets of data, Table 3 which contains data obtained with the magnets follows:

Since the data in Table 3 were compiled just to give an idea of the non-linearity, the individual experimental runs were not analyzed according to the standard least-squares method. The data for each run were plotted on semi-log graph paper, and the slopes were determined from the best curves drawn through the data points (see Figures 18 and 19). The resulting value for the latent heat is 7.5 kcal/M less than the value obtained for the same element when no magnets were used in the source. The second value is also far from agreement with the Stull and Sinke result for silver.

The values of $\Delta H_T(Obs.)$ obtained when there were no magnets in the source were assumed to be closer to $\Delta H_T(True)$. 
Table 3. The experimental slopes, corrections, and errors used to determine the latent heat of sublimation of silver metal. (These data were obtained with the source magnets in place)

<table>
<thead>
<tr>
<th>Run</th>
<th>Midrange temperature °K</th>
<th>Experimental slope kcal/M</th>
<th>Temperature term kcal/M</th>
<th>Latent heat kcal/M</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 B</td>
<td>1404</td>
<td>60.7</td>
<td>1.4</td>
<td>62.1</td>
</tr>
<tr>
<td>2 B</td>
<td>1450</td>
<td>52.3</td>
<td>1.4</td>
<td>53.7</td>
</tr>
<tr>
<td>3 B</td>
<td>1414</td>
<td>48.5</td>
<td>1.4</td>
<td>49.9</td>
</tr>
<tr>
<td>4 B</td>
<td>1418</td>
<td>53.3</td>
<td>1.4</td>
<td>54.7</td>
</tr>
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<td>5 B</td>
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<td>55.6</td>
<td>1.5</td>
<td>57.1</td>
</tr>
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<td>6 B</td>
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<td>1.5</td>
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<tr>
<td>7 B</td>
<td>1442</td>
<td>60.5</td>
<td>1.4</td>
<td>61.9</td>
</tr>
<tr>
<td>Mean</td>
<td>1440</td>
<td></td>
<td></td>
<td>56.6</td>
</tr>
</tbody>
</table>

Standard deviation of mean, $S_n$ 1.66 kcal/M
Orifice correction -0.05 kcal/M
Latent heat reduced to 298°K -61.4 ± 1.7 kcal/M

than were the ones obtained when the magnets were in place.

A brief, qualitative idea of the cause of the temperature dependent non-linearity may be gained by considering two details. Figure 20 is a large-scale schematic drawing of the ionization chamber showing the electron beam, the neutral atom path, and a possible ion path. The path shown for the ion is not meant to be exact, but it is just to show that the ion is deflected away from the center of the chamber opening. The first assumption to be made is that the electron density is not homogeneous across the beam. The density is assumed to decrease from a maximum at the center of the electron beam...
Figure 20. A schematic drawing of the ionization chamber showing a hypothetical ion path when a magnetic field is present in the chamber.
to a minimum at the outer edges. The second assumption has
to do with the initial thermal velocities of the ions (Figure
20, $U_0$). The force ($F_0$) due to the presence of the magnetic
field ($B$) is a function of this velocity. $F_0$ is considered
to be an impulsive force for simplicity. In Figure 20, the
ion is shown to be formed at the center of the electron beam.
In order for an ion with $U_0$ to be drawn out of the chamber
across the center mark (and hence to pass through the mass
analyzer), it must be formed at a point farther to the right
of the center of the electron beam. This would mean that the
recorded ion beam would originate in a region of the electron
beam that is less dense than the center region. Ions with
lower initial thermal velocities would be formed in a more
dense electron region than that where ions of higher initial
thermal energy originate. Hence, the ion current representing
ions of higher thermal energies would be effectively "too
low". This effect would in turn cause the slope of the $\ln I_0$
vs. $(1/T)$ curve to be too low also.

No attempt was made to calculate possible deflections
because this incomplete analysis is meant only to call at­
tention to the fact that it could be possible for a non­
linearity to develop in a source due to the presence of a
magnetic field. Evidently, something of this nature actually
occurs in this source when a rather crude magnet set-up is used.
G. Conclusions

It would be difficult to assign an accurate value for the source non-linearity ($\mathcal{S}$ in Equation 5) for two reasons. First, the accuracy of the latent heat values obtained outside of this laboratory has not been definitely established. Therefore, one can only say that the latent heat results correspond closely to values obtained in other experiments. In the case of thulium, the agreement is very close. For silver, there is a 1.9 kcal/M gap between the ranges of error. Second, while the resulting value obtained for silver was higher than the reference value, the thulium results were lower than one reference value. If a systematic error (or non-linearity) were affecting the data, the results would be either consistently high or low, but not likely both high and low. This argument assumes that the reference values are accurate.

We can conclude that the source is nearly linear in the range of ion currents covered in this experiment and for the particular elements involved. Numerically, it is estimated that source non-linearity probably accounts for an error somewhat less than 1 per cent of the observed latent heat value. Furthermore, it is entirely possible that the source is really much better than that figure, and in fact, we suspect that this is the true situation. However, a positive guarantee of a non-linearity effect smaller than 1 per cent cannot be
given.

It must be observed that source linearity is a very critical property in the measurement of cohesive energies by the present method. If accuracies of a few tenths of a percent are to be guaranteed, a more extensive test program than the one reported here must be carried out.
VI. DISCUSSION

A. Evaluation of Design

The electron bombardment ion source is evidently one which works without the aid of a source magnetic field. In fact, a crude magnetic field seems to introduce a marked non-linearity. However, it may be possible to improve the efficiency of the source if a well-aligned magnetic field were used.

The entire source construction allows for ease in the dismantling and cleaning of the different parts. The source was thoroughly cleaned at least twice during the experiment and there appeared to be no changes in operating characteristics. The filament aged considerably and had to be changed several times, but this was easily done.

The source is completely and easily interchangeable with the surface ionization source. Figure 21 shows the two sources side-by-side. It is also evident that the electron beam is approximately at the same point as the ionizing filament (Figure 21, F). This point is observed also by comparing Figures 3 and 5.

B. Component Evaluation

1. Ionization chamber

The solid block ionization chamber seems to be a very
Figure 21. The electron bombardment and surface ionization ion sources.
satisfactory construction technique. This feature also allows for a wide range of different sample systems to be used, while the same general ionization chamber and electron beam are retained. This versatility would allow the exact placement of the Knudsen cell with respect to the electron beam, thereby facilitating a careful calibration of the source. Then it would be possible to measure the vapor pressure of a Knudsen cell sample as closely as a known sample can be used to calibrate.

2. Filament unit

The electron filament unit has also worked without difficulty. It would be useful if a jig were devised to use in the actual positioning of the filament in the jaws. During the testing of the source it was necessary to remove the entire source in order to assure proper filament alignment, whereas if a jig were used, it would be necessary to remove only the filament unit itself. The pressure jaw technique for holding the filament is satisfactory for filament material 0.001 inch thick or more.

A maximum ratio of electron beam current to total emission current was obtained when the jaws were placed very close together, and, therefore, the surface of the filament parallel to the collimating plates was large enough just to cover the collimating hole. This ratio is approximately $10^{-2}$; or one-
hundredth of the total current emitted from the filament goes to make up the ionizing beam. It would make the source more efficient if this ratio could be increased.

3. Collector unit

The collector unit appears to be satisfactory, but there is some question as to whether or not it is necessary to have quite so complicated a mechanism for simple beam collection. Actually, the collector unit is rather easily made and is very rigid in construction. It does allow a certain freedom in positioning the collector, although no tests were made to actually determine the effect of changing the position of the collector.

4. The grid plate

The biased grid plate used to draw the ion beam from the ionization chamber was satisfactory. The 0.003-inch grid wires themselves were not too difficult to spot-weld in place if a very small spot-welder was used. A grid plate with a wider slot was tried, but in this case when the high voltage was turned on, the electron beam was excessively distorted.

C. Evaluation of Source Operation

Although the source in general is operational, some problems arose during the testing which will eventually have to
be solved. One of the major problems was that not all cell samples tested yielded an observable ion beam. Dysprosium, holmium and copper were tried without success. Dysprosium and holmium simply did not show up on the recorder chart, but this may have been because the Knudsen cell temperatures were too low. At the point where copper was expected, there were many very large peaks, none of which appeared to be coming from the Knudsen cell. The shutter was moved across the exit hole while the recorder was held on each peak, but there was no noticeable drop in recorded ion current for any peak. Thus, in order to be able to observe molecules of low ionization efficiency, the large background must first be eliminated.

D. Latent Heat Measurements

From the results obtained in Section V it will be assumed that the latent heat measurements made with the electron bombardment ion source are reasonably accurate. At least the errors in the results overlap or almost overlap the errors in results obtained in other experiments. It would be wise to make a greater number of experimental runs in order to obtain latent heat values that are to be published. It is felt that systematic errors arising from the source when no magnetic fields are present amount to less than 1 per cent of the cohesive energy.

In conclusion, one can assume that the electron bombard-
ment ion source described in this report is a working ion source for molecular effusion studies.
VII. LITERATURE CITED


VIII. APPENDIX A: A REGULATED POWER SUPPLY

The necessary biases and filament current for the electron bombardment ion source are supplied by the power supply shown in Figure 22. This power supply is total emission current regulated. That is, any small fluctuations in total emission current are amplified and the signal used to vary the filament temperature in such a way as to cancel these fluctuations. Figure 23 is a circuit diagram of the power supply. The circuit is divided essentially into two sections, the first being a regulated 300-volt power supply which provides the proper biases to the different parts of the ion source. These biases are tapped off the load at the right of the lower section, as is seen in Figure 23. The second section controls the total electron current emitted from the filament. The means of regulation is essentially as follows: The emission current passes through the grid resistor of the 5693 pentode. Any fluctuations in this grid bias are amplified, and the amplified signal is impressed on the grid bias of the 6336 triode. A drop in emission current causes the bias on the pentode control grid to become less positive with respect to the cathode, thereby causing a decrease in pentode plate current. A drop in pentode plate current causes the control grid of the 6336 triode amplifier to become less negative with respect to the cathode, and thus more plate current flows through this triode. This increase in plate current causes
Figure 22. The front panel of the regulated power supply
Figure 23. Wiring diagram of the regulated power supply
more current to flow through the primary circuit of the transformers, and thus the current flowing through the filament is increased, and the drop in emission current is canceled. The degree of regulation is controlled by setting the reference bias on the control grid of the 5693 amplifier. This is done by varying the 100K rheostat labeled "Adjust" on the front panel of the power supply. The "Shield" bias in Figure 23 is the bias to the ionization chamber. The collector bias is so labeled in Figure 23.

When the power supply was regulating properly, variations in the 150Ω rheostat in the primary circuit of the filament transformer caused no change in the emission current. This rheostat is designated by "Filament Adjust" in Figure 22. When the power supply was regulating, a variation from 85 to 135 volts in line voltage produced no noticeable change in the emission current.

Since the entire ion source must operate at a potential of +1900 volts, it was necessary to mount the components of the power supply onto an insulating board. A screen was placed over the components to lessen the danger of electrical shock. The meter movements are at high voltage, and the rheostat controls are connected through insulated couplings.

The power supply operates satisfactorily for most purposes. At higher electron currents, there is a tendency for the emission current to undergo short jumps. Also, there
seems to be quite a bit of noise which originated in the power supply and showed up in the ion current records. Both of these effects were possibly due to the contamination of the electron filaments which caused its thermionic work function to continually change.

The meters in this power supply are assumed to have 5 per cent accuracy. The meter used to measure the collector current was calibrated in the following way: The current through the meter was varied and fed through a 1 per cent precision resistor. The resulting voltages across the resistor were measured with an L & N type K potentiometer. Thus, the electron beam current values quoted in this report were assumed to be accurate to the precision to which the meter could be read, which was 2 per cent.
IX. APPENDIX B: SOURCE PARTS

The following figures are dimensioned, scale drawings of each part in the electron bombardment ion source.

All screws used in the source are number 1-72 stainless steel screws.
Figure 24. Source parts
Figure 25. Source parts
COLLECTOR UNIT

Figure 26: Source parts