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SULFUR HEXAFLUORIDE AS A FUEL FOR IONIC PROPULSION

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- ABSTRACT -

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This report describes experiments on the generation of an intense beam of SF." negative ions by an electron bombardment method. Some theoretical considerations are given on the basic processes for the generation of negative ions, the characteristics of an electron bombardment ion source and the relevant properties of the Sulfur Hexafluoride molecule. A detailed description of the experimental system is presented and the important features in the design of the ion source are discussed. In particular the feasibility of introducing the gas in the ion source in the form of a well defined stream is demonstrated thus ensuring a high ion conversion efficiency for this ion source.

Time-of-Flight-Mass Spectrometer measurements have indicated that only  $SF_5^-$  and  $SF_6^-$  ions were produced in that source and that the  $SF_5^-$  contribution to the total ion current was negligible. The negative ion current as function of the electron bombarding energy exhibits a complex resonance spectrum. The limitation to the maximum negative ion current produced by this method is set by space charge effects in the bombarding electron beam. It is found that the negative ion current is still increasing as the maximum handling capacity of the gas collimator is reached.

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It is concluded that if space charge limitations can be eliminated a negative ion current adequate for electrical propulsion could be generated with Sulfur Hexafluoride.

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### SULFUR HEXAFLUORIDE AS A FUEL FOR IONIC PROPULSION

#### I INTRODUCTION

This research programme was initiated to study the generation of an intense beam of negative ions using Sulfur Hexafluoride (SF<sub>6</sub>). The ultimate aim of this research is the use of negative ions to neutralize the positive ions at the exhaust of an ionic rocket.

The use of negative ions, instead of electrons, has been suggested as a possible solution to the very important problem of the charge neutralization associated with ionic rockets. The mass of the negative ions being essentially the same as that of the positive ions, leads to mixing of the charges much more quickly than if positive ions and electrons were present.

The state of the art of generating positive ions is at a point where small cesium ion motors are already being developed for testing in outer-space. In the case of negative ions the problem is still at the stage of basic research and, except for particle accelerator applications, little effort\*

\* R. J. Sunderland, J. R. Radbill and R. D. Gilpin, "The Development of a Negative Ion Source", Paper presented at the ARS Electrostatic Propulsion Conference, Monterey, November 1960.

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has been made yet to produce intense beams of negative ions. The development of negative ion sources for particle accelerators has been directed toward the generation of rather light ions such as hydrogen and oxygen. For electrical propulsion, the interest is in the direction of heavier ions and this aspect has led us to the experimental study of the generation of negative ions using Sulfur Hexafluoride of molecular mass 146. The SF, molecule is particularly attractive for ionic rocket application since its mass is comparable with that of the cesium (133) positive ion.

The research programme reported hereafter describes experiments aimed at producing an intense beam of negative ions by an electron bombardment method. The ion source was designed in such a way as to enable examination of the influence of the electron bombardment current, the electron energy and the SFs gas density on the generation of SFs<sup>-</sup> negative ions. It will be shown that, by introducing the gas in the form of a molecular beam, a high percentage ion conversion efficiency can be attained.

The production of  $SF_6$  negative ions as function of electron bombardment energy exhibits a multiple resonance spectrum. The presence of several peaks in the energy spectrum is attributed in part to instrumental effects and in part to multiple collisions of the electrons with the SF<sub>6</sub> molecules across the ion chamber. The negative ion current corresponding to the maximum

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value in this energy spectrum represents a total pegative ion current in the range of  $1 \ \mu$  A at an energy of 1 Kev. It is found that the negative ion current that can be attained by this method is limited by space charge effects in the bombarding low energy electron beam. In the region of the energy spectrum where the SF<sub>6</sub><sup>-</sup> ion peak is a maximum it is observed that the SF<sub>5</sub><sup>-</sup> ion is not contributing significantly to the total negative ion current. The ion intensity appears to increase almost linearly with the SF<sub>6</sub> gas flow rate in the region where the gas collimator reaches its full handling capacity. This indicates that still higher negative ion current could be attained by this method by increasing the gas flow rate in the ion source.

Some theoretical considerations in connection with the basic processes for generation of negative ions, the general characteristics of an electron bombardment ion source and the interesting properties of Sulfur Hexafluoride will be presented in Section II. A description of the construction and operation of the ion source designed for this research programme will be given in Section III followed, in Section IV, by a presentation and discussion of the measurements on negative ions. Section V will be devoted to some concluding remarks in connection with the results obtained and some recommendations for future research in the field of negative ions for electrical propulsion.

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## II THEORETICAL CONSIDERATIONS

## (a) Basic Processes for Negative Ion Formation

There are two basic processes by which a molecule may be converted into a negative ion. A neutral molecule may capture an electron and become a stable negative ion or it may, by collision with another particle, split into a pair of positive and negative ions.

The electron capture process may lead to an unstable negative ion with subsequent splitting into a neutral particle and a stable negative ion. The electron capture process is a resonance phenomenon in the sense that it usually takes place for a finite energy range of the colliding electrons. If the energy of the interacting electrons is not within this energy range, then the probability of electron capture is essentially zero.

All negative ions previously studied\* have shown that the maximum probability for the electron capture process to occur takes place at very low values of electron energies i.e. between 0 and 10 ev. This characteristic is thus very attractive when considering the energy involved in producing negative ions. However, for most molecules or atoms previously

\* F. H. Field and J. L. Franklin, "Electron Impact Phenomena", Academic Press, N.Y. (1957). investigated, it has been found that the probability of electron capture is much smaller than that for the formation of positive ions by electron bombardment. Nevertheless few exceptions, among them sulfur hexafluoride molecules, are characterized by an electron capture cross-section at least as large as that of the positive ions.

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The process of ion pair formation by electron impact is in principle very attractive as a potential source of positive and negative ions for electrical propulsion. However the main disadvantage of this process is that the electron energy required is relatively high i.e. at least 15 ev. This energy corresponds to the dissociation energy of the molecule plus the ionisation potential of the positive fragment minus the electron affinity of the negative fragment: this latter is usually very small. Experiments have also indicated that the cross-section for this process to take place is normally much smaller than that for electron capture.

It is thus believed that at present the most promising process for producing intense beams of negative ions is the electron capture process.

# (b) General Characteristics of a Negative Ion Source

To gain insight into the physical processes within an electron bombardment ion source an attempt has been made to describe theoretically the performance of this ion source. The model studied here although it is not an exact representation of the actual ion source described below should be useful in analysing the characteristics of that source in terms of the various parameters involved. The geometry of the theoretical model is equivalent to that of the experimental ion source but the effects of scattering and density gradient of the particles within the ion source have been neglected.

The cross-section  $Q(\mathbf{E})$  for ionization of a neutral molecule by electron impact is defined through the relation

$$dI_{e} = -N_{g} Q(E) I_{e} dx$$
 (1)

where dI<sub>e</sub> (amp/cm<sup>2</sup>) is the change in electron current density due to electron capture by the molecules as the electrons travel a distance dx through the gas, N<sub>g</sub> is the gas density (no. of molecules/cm<sup>3</sup>) I<sub>e</sub> is the electron current density (amp/cm<sup>2</sup>) and Q(E) (cm<sup>2</sup>) is the electron capture cross-section; it is in general a strong function of electron energy.

Integrating Eqn. (1) leads to

$$I_{e} = I_{eo} e^{-N_{g}Q(B)x}$$
(2)

where  $I_{eo}$  is the electron current density at x = 0.

If each electron lost to the beam is captured by a neutral molecule then the number of negative ions produced per second is given by

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$$\frac{\partial n_{i}}{\partial t} = \dot{n}_{i} = \frac{I_{eo} - I_{e}}{e} = \frac{I_{eo}}{e} \left(1 - e^{-N_{g}Q(\mathbf{E})\mathbf{x}}\right)$$
(3)

where e is the charge of the electron.

Consider an ion source as shown schematically in Fig. 1. If the density of the electron beam incident on  $A_1$  is



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 $I_{eo} = N_e e v_e \qquad (4)$ 

where  $N_e$  is the electronic charge density (no. of electrons/cm<sup>3</sup>) and  $v_e$  is the velocity of the electrons (cm/sec) then the number of electrons entering the volume per second is

$$\frac{\partial n_{\theta}}{\partial t} = \hat{n}_{\theta} = \frac{I_{\theta} A_{1}}{\theta}$$
(5)  
= N\_{\theta} v\_{\theta} A\_{1}

The number of electrons leaving the box per second is, using Eqn. (2)

$$\frac{I_1}{e} = \frac{I_{ec}}{e} e^{-N_g Q l_3}$$
(6)

where N is the gas density (no. of molecules/cm<sup>3</sup>).

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The number of negative ions formed per second which is the same as the number of electrons disappearing in the box is thus given by

$$\dot{n}_{io} = \frac{I_0}{e} - \frac{T_1}{e} = N_e v_e A_1 \left(1 - e^{-NgQl_3}\right)$$
(7)

Under steady-state conditions the rate at which the ions are formed must equal the rate at which they are removed from the source. The process by which the ions are formed is assumed to be by electron attachment. The negative ions may be removed from the source in two ways: diffusion to the walls and extraction into a useful ion beam. Hence by Eqn. (7) the following equation for the steady state operation of the ion source may be written

$$N_{e}v_{e}A_{1}\left(1-e^{-NgQl_{3}}\right)-\dot{n}_{ib}-\dot{n}_{id}=0$$
(8)

where  $\dot{n}_{ib}$  is the rate at which the negative ions are extracted as a useful beam and

 $\dot{n}_{id}$  is the rate at which the ions are lost to the walls by diffusion.

The efficiency of extracting the negative ions  $(\eta_{ex})$  from the source is mainly dependent upon the geometry of the source

and the electrostatic potential of the extracting electrode with respect to the source. It can be defined as

$$\eta_{\text{ex}} = \frac{\dot{n}_{\text{ib}}}{\dot{n}_{\text{ib}} + \dot{n}_{\text{id}}}$$
(9)

Eliminating  $n_{id}$  between Eqns. (8) and (9) leads to

$$\dot{\mathbf{n}}_{ib} = \eta_{ex} N_{e} v_{e} A_{1} \left[ 1 - e^{-NgQl_{3}} \right]$$
(10)

The ion current density  $I_i$  in  $(amp/cm^2)$  is given by

$$I_{i} = \frac{\underline{n}_{ibe}}{A_{3}}$$

where  $A_3$  is a cross-section of the negative ion beam at the exit plane of the ion source (see Fig. 1).

In terms of Eqn. (10) and using Eqn. (4) the ion current density is given by

$$I_{i} = \eta_{ex} \left(\frac{A_{1}}{A_{3}}\right) \left[1 - e^{-NgQl_{3}}\right]$$

or

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$$\frac{I_{i}}{I_{e}} = \eta_{ex} \left( \frac{A_{1}}{A_{3}} \right) \left[ 1 - e^{-N_{g}Q_{1_{3}}} \right]$$
(11)

Eqn. (11) represents the conversion ratio of an electron beam into a negative ion beam. It should be noted that the factors  $\eta_{ex}$  and  $[1 - e^{-NgQl_3}]$  are always smaller than one but the

ratio  $A_1/A_3$  can be greater than one. In the electron bombardment ion source described below this ratio is of the order of two; two electron beams of cross-sectional area equal to that of the ion beam are used in the ion source. However it may be shown that if the electrostatic extraction of the ions is neglected the factor  $\eta_{ex} \frac{A_1}{A_3}$  is always smaller than one. This may be estimated by means of a simple approximation; it is assumed that the diffusion of the negative ions to the walls of the source is isotropic. It is then possible to express the ion extraction efficiency  $\eta_{ex}$  as follows:

$$\eta_{ex} = \frac{\text{Area of the ion exit aperture}}{\text{Total area of the walls of the ion source}}$$
 (12)

$$= \frac{A_3}{2(A_1 + A_2 + A_3)}$$

Inserting Eqn. (12) into Eqn. (11) leads to

$$\frac{I_1}{I_0} = \frac{A_1}{2(A_1 + A_2 + A_3)} \left[ 1 - e^{-N} g^{Q_{1_3}} \right]$$
(13)

and it is evident that this relation is always smaller than one. However it is expected that the electrostatic extraction method will greatly increase this ratio.

The efficiency of conversion of gas molecules into negative ions  $\eta_g$  is defined as the ratio of the number of negative

ions extracted from the source per second to the number of neutral molecules entering the source per second. It may be expressed by

$$\eta_g = \frac{\dot{n}i}{\dot{n}_g}$$
(14)

with  $n = N \nabla A_2$  where  $\nabla_{-}$  is the gas linear

 $\mathbf{v}_{\mathbf{g}}$  is the gas linear flow velocity (cm/sec) and  $A_2$  is the cross-sectional area of the gas jet.

By means of Eqn. (10) an expression for  $\eta_g$  may thus be obtained in terms of the electron current, the gas flow rate, the ion extraction efficiency and the geometry of the source; it is given by

$$\eta_{g} = \eta_{ex} \frac{N_{e} \mathbf{v}_{eA_{1}}}{N_{g} \mathbf{v}_{g} A_{2}} \left[ 1 - e^{-N_{g} Q \mathbf{l}_{3}} \right]$$
(15)

By definition  $\eta_g$  cannot be greater than one but this is not evident in Eqn. (15). Indeed the gas flow  $(N_g v_g A_2)$  is independent of the electron flow  $(N_g v_g A_1)$  and thus the ratio  $\frac{N_e v_e A_1}{N_g v_g A_2}$  may be made greater than one. The value of  $\eta_g$  as expressed in Eqn. (15) may in principle exceed one. This situation results from the fact that in the derivation of Eqn. (15) the gas density N<sub>g</sub> has been assumed constant throughout the ion source. This is, of course, only valid if a small fraction of the gas molecules incident upon the ion source are transformed into ions. A better approximation may be obtained by replacing  $N_g$  appearing in the exponent  $N_gQl_3$  by the average value  $(\overline{N_g})$  of the gas molecules density within the source (after substracting the number of gas molecules transformed into negative ions). The efficiency  $\eta_g$  for the conversion of neutral molecules into negative ions may be increased by recirculating the gas molecules which have not been ionized in their first passage through the ion source. The experimental arrangement used during this research programme has permitted testing of this possibility and it will be shown that by this method the efficiency  $\eta_g$  may attain a value close to unity.

## (c) Properties of Sulfur Hexafluoride

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The properties of sulfur hexafluoride  $(SF_6)$  will now be discussed in relation to the desirable properties of fuels for a high intensity source of negative ions.

Sulfur Hexafluoride is gaseous at normal temperature and pressure but can be frozen at a temperature of  $-50.8^{\circ}$ C. Its vapour pressure is less than  $10^{-9}$  mm of Hg at  $-195^{\circ}$ C (liquid nitrogen temperature). It is essentially chemically inactive and can be handled easily without risk of corrosion. SFe is relatively inerpensive (many times less than cesium) and is widely available.

The molecular weight of the SF<sub>6</sub> molecule is 146. This value falls in the range of molecular masses considered practical

for electrostatic propulsion. The molecular weight of SFe compares favorably with that of cesium (133) which at present is considered the best fuel in positive ion sources for electrostatic propulsion. The possibility of operating an ion rocket with a pair of cesium and SFe ion ejectors is thus quite attractive.

One of the most interesting properties of SF<sub>6</sub> as a source of negative ions is its exceptionally large electron capture cross-section  $(10^{-15} - 10^{-16} \text{ cm}^2)^*$ . It should also be noted that the maximum of the resonance curve for electron capture in SF<sub>6</sub> occurs at very low electron energies (between 0 and 1 eV). This characteristic may be an important feature in regards to the overall power efficiency (kilogram/kilowatt) of an ionic rocket. It should finally be noted that electron impact experiments<sup>\*\*</sup> with SF<sub>6</sub> have indicated the formation of SF<sub>5</sub><sup>-</sup> ions as well as SF<sub>6</sub><sup>-</sup> ions. Although it has been found that the abundance of the SF<sub>5</sub><sup>-</sup> ions does not exceed 20% of that of the SF<sub>6</sub><sup>-</sup> ions, it is expected that these SF<sub>5</sub><sup>-</sup> ions may reduce the effectiveness of the SF<sub>6</sub><sup>-</sup> ion source.

W. M. Hickam and R.E. Fox, J. Chem. Phys. 25, 642 (1956).
 A. J. Absorp and N. B. Hannah, Chem. Phys. 25, 642 (1956).

A. J. Ahearn and N. B. Hannay, J. Chem. Phys. 21, 119 (1953).

#### III. THE EXPERIMENTAL SYSTEM

The basic operation of the ion source investigated in this research programme is illustrated in Fig. 2. The neutral SF, gas is introduced into the source in the form of a molecular beam and is collected at the far end of the source. The electrons emitted from an incandescent tungsten ribbon are launched into the ion chamber in a direction perpendicular to the gas flow. The SF,<sup>-</sup> negative ions formed by electron bombardment are then extracted from the ion source and launched into a mass spectrometer or an ion collector in a direction normal to both gas and electron flows. A small magnetic field is used to collimate the electron beam and to prevent the electrons from being extracted with the negative ions.

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There are three main reasons for introducing the SF. molecules in the form of a collimated beam. Firstly, it allows the pressure in the ion analyzer section to remain low and thus prevents scattering of the negative ion beam by the ambient gas. Secondly it prevents dissociation of the SF. molecules on the hot parts of the ion source and thirdly it permits the recirculation of the SF. molecules not ionized in their first transit through the ion source. This last characteristic is directly related to the possibility of attaining high percentage propellant utilization for ionic

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rocket applications.

A rather complex experimental apparatus had to be constructed in order to realize the concepts involved in the ion source just described and to investigate its performances. For convenience and clarity the description of the experimental system will be presented under three main headings: the gas flow system, the ion source and electron supply system and the negative ion analyzer system.

#### 1. The Gas Flow System

The main components of the gas flow system are presented in the schematic diagram of Fig. 3. They comprise the vacuum system, the gas-handling system, the gas collimator and the gas collector.

(a) The Vacuum System

A 2-inch oil diffusion pump in conjunction with a mechanical pump serves to evacuate the 4-inch wide diameter Pyrex cross-test chamber. A large liquid nitrogen trap is located on the top of the diffusion pump and can be isolated from the test chamber by means of a 2-inch diameter butterfly valve. The differential pumping system for the gas collimator is formed by a two-inch diffusion and liquid nitrogen cold-trap connected to the gas collimator by means of two-inch diameter

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pyrex glass tubing. A 2-inch butterfly valve is also used to isolate the differential pumping system from the gas collimator. A set of ionization and thermocouple gauges serves to monitor the pressure at various locations in the vacuum system. During the tests a pressure of  $5 \times 10^{-7}$  mm of Hg could be maintained in the test chamber when the cold traps were filled with liquid nitrogen.

(b) The Gas Handling System

A schematic diagram of the gas handling system is shown in Fig. 4. It is constructed entirely from glass except for a plastic tube connecting the main gas reservoir to the gas handling system. The SF, gas from a high pressure tank is introduced into the gas handling system through a needle valve and a mercury manometer and bubbler. A small glass reservoir forms part of the system and is also used to purify the sulfur hexafluoride as supplied from the main reservoir. This reservoir may be cooled to liquid nitrogen temperature and non-condensable impurities may be removed by vacuum pumps. The gas is then allowed to flow through a differential pressure flow indicator before reaching a fine control needle valve which is used to adjust the flow of SF, to the gas collimator.





(c) The Gas Collimator System

The function of the gas collimator is to limit the width of the gas stream within the walls of the ion chamber. In the present apparatus the size of the ion chamber and the distance between the collimator and the ion source is such that an appreciable beam divergence may be tolerated. A SF<sub>6</sub> beam intensity higher than that limited by molecular flow conditions is thus permitted; the SF<sub>6</sub> gas flow rate may be increased to a value intermediate between that of molecular and viscous flows.

The efficient operation of the collimator depends on two main factors: its geometry and the efficiency of the differential pumping system.

The size of the slits and the distance between them must be such as to limit the profile of the beam within the walls of the ion chamber. The geometry of the various collimators used in the experiment reported here as well as the various dimensions of interest are shown in Fig. 5. The size of the first aperture is limited by the condition that, at the operating feed line pressure, the mean free path of the SF. molecules must not exceed the diameter of the aperture. For the aperture size (0.5 mm diameter) chosen in the collimators used in the present experiments the upper limit to the gas pressure in the feed line is in the region of 100 microns of Hg.









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Fig. 5. Various designs of gas collimators.

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The respective diameters of the inner and outer tubes of the collimator must be such as to allow good conductance for the gas removal from the collimator region. A high differential pumping speed is also necessary to maintain the pressure in the region between the two slits at a sufficiently low value to prevent excessive beam scattering within the collimator region.

A qualitative evaluation of the collimator efficiency may be carried out by observing the pressure in the test chamber as the SF<sub>6</sub> is allowed to flow through the system. If the collimator allows only a well defined beam of molecules to flow through the test chamber and if the gas collector pumping efficiency is adequate, then no appreciable change of pressure in the test chamber should be observed.

Such a test was performed with each of the three collimator arrangements shown in Fig. 5-a,b and c. With the gas collimator shown in Fig. 5-a the differential pumping system was made up of a cold trap of the type shown in Fig. 6-a. When the SF, gas was allowed to flow at a pressure of 20 microns of Hg in the feed line the pressure in the test chamber increased from  $1 \times 10^{-6}$  mm of Hg to  $2 \times 10^{-5}$  mm of Hg. This appreciable rise of pressure in the test chamber indicated that very little gas collimation was indeed obtained due presumably to insufficient pumping speed in the collimator system. This



collimator was then changed to that shown in Fig. 5-b in which a smaller feed tube and a larger diameter pumping line was used. In this case the "inverted finger" cold trap shown in Fig. 6-c was used to evacuate the collimator. This arrangement has permitted the pressure to be maintained in the test chamber at  $3 \times 10^{-6}$  mm of Hg while feeding the SF, to the collimator at a pressure of 20 microns of Hg. In an attempt to further improve the performance of the collimator the design shown in Fig. 5-c was adopted. This change produced only a slight improvement in the steady-state pressure of the test chamber for the same conditions of SF, flow in the collimator.

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In the course of these experiments it was realised that the presence of even small amounts of non-condensable impurities could cause serious difficulties in the operation of the SF, gas collimator. Indeed, when using a cold trap as a differential pumping system, the nor-condensable molecules in the collimator can only escape this region through the second slit of the collimator. Since this aperture represents a high impedance path for the molecules the pressure in the collimator may increase to such a level as to produce excessive scattering of the SF, beam within the collimator itself. The collimator cold trap was then removed and replaced by an oil-diffusion pump to permit the non-condensable gases as well as the SF, gas to be quickly removed from the collimator region. The gas collimator shown in Fig. 5-d was then constructed and was connected to the two-inch pumping system described earlier. With this arrangement it was found that the pressure in the test chamber could be maintained below  $10^{-6}$  mm of Hg even when the SF<sub>6</sub> pressure in the feed line was as high as 500 microns of Hg.

An attempt was made to examine the profile of the SF. beam by introducing a cold target in front of the gas collimator. By condensing the SF. emitted by the collimator on a surface at liquid nitrogen temperature, as shown in the insert of Fig. 7, it was possible to observe directly the crosssection of the molecular beam after running the gas for a period of approximately twenty minutes. Some pictures of the SF. deposit on the cold target are shown in Fig. 7 for various gas pressures in the feed line. It is seen that, indeed, at pressures below 500 microns of Hg the beam is well defined and corresponds to the size expected from the geometry of the gas collimator. It is also observed that at a pressure of 600 microns of Hg the beam begins to diffuse and is completely deteriorated at a pressure above 800 microns of Hg.

The calibration of the gas flow through the ion source as function of feed line pressure is also shown in Fig. 7. This curve was obtained by running the SF, through the collimator for

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Fig. 7. Calibration of the SFs gas collimator.

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a fixed period of time and by expanding the gas collected on the target in the entire test chamber and measuring the pressure of the gas filling the test chamber. From a knowledge of the volume of the test chamber, as determined by expanding a known amount of gas, it was possible to calculate the number of molecules per second rejected by the gas collimator for various pressures in the gas feed line.

(d) The Gas Collector System

The function of the gas collector system is to recover the SF, molecules which have crossed the ion source without being ionized. In the course of this research programme various types of gas collector systems were tried. Two of these gas collector systems are shown in Fig. 8a and b and were used to carry out volumetric measurements of the gas ejected by the collimator. In the final set-up it was found more convenient and efficient to use a cold (liquid nitrogen) target close to the ion source as shown in Fig. 8-c. With this arrangement it was possible to run the SF, gas in the collimator at pressures in the range of  $500\mu$  of Hg without observing appreciable increases in the pressure  $(5 \times 10^{-7} \text{ mm}$ of Hg) of the main test chamber.







Fig. 8. Designs of the collector systems.

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(c)

### 2. The Ion Source and Electron Supply System

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The operation of the ion source was outlined earlier in connection with the schematic diagram of Fig. 2 illustrating the basic arrangement of the ion source. This arrangement, whereby the three types of particles flow along mutually perpendicular directions, was chosen to allow a close control of the various parameters, such as gas flow, electron flow and ion extraction, affecting the performance of the ion source. In particular since higher gas densities than usually encountered in mass spectrometry studies were to be used, a special attention was given to the geometry of the source in connection with the gas flow system.

(a) Construction of the Ion Source

The various parts comprising the source may be identified in the sketches of Fig. 9-a,b and c.

(i) Electron Beam Source

A tungsten ribbon having a cross-section of 0.002"× 0.020" extending between stainless steel support posts some 0.75" apart from the electron emitting elements. Two filaments in all are located on either side of the tunnel forming the main section of the ion chamber in juxtaposition with the control grid and collimating slits in the walls of the tunnel. Each pair of support posts pass through glass seals which in turn are secured



Fig. 9-a. Layout of ion source and mass spectrometer.

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Fig. 9b. Exploded view of the ion-source assembly.





in filament support blocks. These seals allow for some movement of the support posts, whilst the support blocks themselves are secured to the base plate of the ion source with bolts that pass through slotted holes. In this manner two degrees of freedom of adjustment are possible for accurate alignment of each filament with its corresponding slit. To avoid any increase in noise level due to stray electrons in the vicinity of the source, cylindrical tantalum shields were attached behind the filaments reflecting the electrons in the direction of the slit.

Each control grid was made of a .010" thin tantalum plate of size  $0.375" \times 2.5"$ . A slit of dimensions  $0.093" \times 0.625"$  was cut in the centre and using gold plated (0.001")tungsten wire, a grid was formed by spot welding lengths of wire across the slit. The spacing of the wire mesh fabricated by this technique was about 0.040". The grid was mounted on teflon spacers to provide electrical insulation.

(ii) Ion Chamber

The ion chamber embodies a  $1/32^{"}$  nickel plated brass plate bent to form a rectangular tunnel of depth 0.875" and width 0.75". Slits 0.093"  $\times$  0.625" are symmetrically located in the walls of the tunnel to allow entry of the electron beam.

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Completing the ion chamber is the cover or extracting electrode, insulated from the lower plate, made of the same materials and in addition having a centrally located slit  $0.8" \times 0.625"$  in size to allow for removal of the negative ion beam.

(iii) Ion Focussing Electrodes

The focussing arrangement for the ions is achieved by having two half sections insulated from one another but together forming a single plate similar in size to the extracting electrode.

Brass support rods incorporating 0.25" diameter teflon bushing contain the stack of electrodes. Quartz spacers placed between the electrodes maintain the following distances:-

- (a) Mass spectrometer entrance slit to ion focussing electrodes: 0.250"
- (b) Ion focussing electrodes to extracting electrode:
   0.187"
- (c) Extracting electrode to ion chamber: 0.032"

(b) Electrical Operation of the Ion Source

Since the ion source was used in conjunction with a time-of-flight mass spectrometer, extraction of the negative ions from the ion chamber had to be performed in short bursts. To achieve this type of operation the extracting electrode was normally held slightly negative with respect to the ion chamber to prevent the ions from being drawn into the mass spectrometer. A large positive voltage pulse of short duration was then applied on the extracting electrode to launch the ions in bunches into the mass spectrometer. The kinetic energy of the ions entering the drift tube of the mass spectrometer was then given by the voltage applied between the ion chamber and the drift tube. The implications behind this mode of operation will be discussed more fully in a later section dealing with the Time-of-Flight Mass Spectrometer.

The circuits associated with the electrical operation of the ion source are shown in Fig. 10.

Except for the ion chamber supply, the power requirements for the source come from dry cell batteries and in the case of the heaters, from large capacity storage batteries.

It is convenient to consider the electrons and ion flow separately.

(i) Electron Flow

A two ohm fifty watt potentiometer control is used in both filaments to maintain the currents within desired limits. Heater currents were in the range of 4 amperes and both filaments could be operated separately or together as desired. Heaters are biassed negatively with respect to the ion chamber by a



Fig. 10. Circuit diagram for the Ion Source.

combination of battery and potentiometer (see Fig. 10) to furnish a continuously variable bias. The extent of this bias potential provides a direct measure of the electron bombardment energy of the source.

The electron control grid circuit (E.G.) provides a continuously variable positive voltage with respect to the heater potential and essentially acts as the anode of a diode thus controlling the current drawn from the filament.

(ii) Ion Flow

For most convenient operation the drift tube of the time of flight mass spectrometer is operated at ground potential. This necessitates the ion chamber to be operated at a negative voltage in order to accelerate the negative ions into the drift tube at the required kinetic energy.

Accordingly the ion chamber is connected to a variable 1.5 kilovolt negative supply. In operation a typical value for the ion chamber potential was one thousand volts negative.

The small negative bias required by the extracting electrode consists of a similar battery and potentiometer arrangement referred to previously and is variable from zero to 12 volts negative. The positive pulse required at the extracting electrode to draw out the negative ions is derived from a Hewlett Packard Model 212A pulse generator AC coupled to the main system through an isolating condenser. The purpose of the diode seen in Fig. 10 is to maintain the D.C. potential at the back edge of the pulse.

The focussing electrodes are connected to a 360 volt positive supply. Each electrode potential is variable over the whole range of available voltage and operates independently of the other providing a large range of focussing for the negative beam.

The complete source is located between the poles of a magnet to utilize the focussing effect of the magnetic field in the separation of the negative ions and electrons. The magnetic field in the center of the source is approximately 100 gauss.

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To insure reliable and reproducible measurements, both current and voltages of each circuit were monitored. The monitoring points shown in the sketch (Fig. 10) were all brought out to panel connections arranged below the corresponding potentiometers controlling the current or voltage levels. Closing of a circuit was made by connecting with short leads the current monitoring points of the circuits or alternatively introducing a current meter between those points.

# 3. Time of Flight Mass Spectrometer

The operation of the ion source in conjunction with the Time of Flight Mass Spectrometer has already been explained in section [2b]. Negative ion bunches are launched into the drift tube during the positive pulse portion of the cycle of operation. The time T that the ions spend in the drift tube depends on the length L of the drift tube, the electronic charge to mass ratio  $(\frac{\Phi}{M})$  and the kinetic energy at the entrance to the drift tube. This time T is given by the relation

$$T = \frac{L}{\sqrt{2V(e/M)}}$$
(1)

In this context V is the voltage through which the ions have been accelerated.

Any mixture of ions will contain different charge to mass ratios, so that the ions will separate themselves into bunches of constant  $(\frac{e}{N})$  whilst drifting along the length of the tube.

In accordance with equation (1) the resolving power may be obtained in the usual manner by investigating the change in time  $\Delta T$  for an incremental change in mass.

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$$\mathbf{T} + \Delta \mathbf{T} = \frac{\mathbf{L}}{(2 \bullet \nabla)^{\mathsf{T}\mathbf{T}}} \left(\mathbf{M} + \Delta \mathbf{M}\right)^{\frac{1}{2}}$$
(2)

- 40 -

Dividing equation (2) by (1)

i.e.

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 $\frac{\mathbf{T} + \Delta \mathbf{T}}{\mathbf{T}} = \left(1 + \frac{\Delta \mathbf{M}}{\mathbf{M}}\right)^{\frac{1}{2}}$ 

$$\frac{\Delta \mathbf{T}}{\mathbf{T}} \approx \frac{\Delta \mathbf{M}}{2\mathbf{M}} + \text{ correction term } 0 \left(\frac{\Delta \mathbf{M}}{\mathbf{M}}\right)^2$$

Therefore the resolving power is given by:

$$\frac{M}{\Delta M} \approx \frac{T}{2\Delta T}$$
(3)

In the present application of this instrument, the ion beam is expected to contain both  $SF_5$  and  $SF_6$  as principal constituents so the design of the Time of Flight Mass Spectrometer hinges around the separation of these two molecules.

Insertion of values for  $SF_5$  and  $SF_6$  in (3) yields a value between 7 and 8 for the Resolving Power. It may be noted from equation (3) that resolving power is inversely proportional to  $\Delta T$ , the measure of the interval during which ions of mass M arrive at the collector. Clearly, this time  $\Delta T$  is proportional to the thickness of the ion packets at the collector which in turn is a combination of the thickness of the packets leaving the source together with the broadening of the packets during the drift time. Accordingly, estimating the resolving power of the instrument requires a knowledge of all those effects which lead to spread of ion velocities within their packets. These effects include:

- The spread in the time of flight for ions which start simultaneously from different parts of the ionisation chamber.
- (2) The spread due to the variation in magnitude and direction of the initial ion velocities. This spread will depend on both the thermal distribution of the velocities of the molecules before ionization together with the energy acquired from the field after ionization.
- (3) The spread in starting time of the ions leaving the source.
- (4) The influence of the field produced by space charge effects.
- (5) Conditions of detection of ion bunches in the receiving equipment.

Bearing in mind the detrimental effect these factors will have in the overall resolving power a design value for the resolving power of the instrument was chosen as 20 for atomic masses of 150 A.M.U.

(a) Mechanical Construction

The mechanical construction and pertinent dimensions of the Time of Flight Mass Spectrometer appear in Fig. 9-a.

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The drift tube portion consists of a nickel plated brass tube machined to form a slip joint near the source and having brass flanges silver soldered at both extremities. A third flange located nearer the collector end is used to support the system and form a vacuum seal with the main chamber. By mounting the tube off centre in this flange, rotation of the instrument about this position results in some lateral movement of the source. The vertical and lateral movement permits lining up of the source with the molecular beam during the final assembly.

The collector is a modified multiplier phototube (RCA 6810) having the photo cathode removed and mounted in a brass flange to permit easy separation of the instrument.

(b) Detecting Equipment

A bandwidth greater than 10 megacycles per sec is required to furnish adequate resolution of the mass peaks. Low signal levels at this bandwidth imposes severe demands on the detecting equipment. To overcome some of the effects of stray capacity, a transistorized emitter follower (see Fig. 11) circuit was located immediately on the collector of the







Fig. 12. Block diagram of the negative ion measuring system.

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multiplier tube. The action of this circuit is to drop the impedance of the signal source looking back at the emitter follower by the A.C. current gain of the transistor, whilst maintaining the same signal swing. Output from the emitter follower was via a coaxial cable terminated at the emitter follower end by the characteristic impedance of 50  $\Omega$  in this instance. The signal was fed into the type L plug-in unit of a 545A Tektronix Oscilloscope that includes a built-in delay.

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A block diagram of the complete circuit is shown in Fig. 12. The pulse generator on receiving the trigger pulse applies a positive 100 volt pulse to the ion extractor plate and a synchronizing pulse that starts the run down of time base B of the oscilloscope. At a point determined by the delay dial setting, time base A is triggered by time base B so that by arranging this delay to be equal to the time the ions take to drift down the tube the resulting waveform may be displayed on the oscilloscope with the required time scale. A satisfactory mass resolution (see Section IV, Fig. 14) was obtained when the width of the pulse applied to the extracting electrode was adjusted to  $0.5\mu$  sec.

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A view of the experimental set-up is shown in Fig. 13-a while close-up of the test chamber containing the ion source, the mass spectrometer and the gas collimator may be seen in Fig. 13-b.



Fig. 13(a). A view of the Experimental Set-up.



Fig. 13(b). Close-up of ion source and mass spectrometer assembly.

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#### IV NEGATIVE ION MEASUREMENTS

#### (a) Preliminary Measurements

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Some preliminary tests were first carried out on the ion source without using the mass spectrometer. A Faraday cup was used to collect the ions and no pulse technique was required for these measurements. These tests were carried out to ascertain that the ion source was properly mounted and that negative ion currents could be detected. These measurements were also done to establish the order of magnitude of the negative ion currents to be measured with the mass spectrometer. The total ion current was measured with a Hewlett-Packard Model 475A Micro Volt-Ammeter.

While maintaining the electron emission current constant at a value in the range of a  $100 \mu$  A, the butterfly values isolating the test chamber from the vacuum pumps were closed and the SFs gas was allowed to flow into the vacuum chamber. As the pressure was raised in the test chamber the current measured by the meter was observed to increase. The electron energy was then adjusted to obtain a maximum reading. In this way it was found that an ion current of the order of  $0.25 \mu$  A was obtained for a SFs gas pressure of  $2 \times 10^{-4}$  mm of Hg in the test chamber.

To ascertain that the current measured was not due to electrons extracted from the source, the test chamber was evacuated and the current reading was observed to fall back to zero. The possibility of measuring negative ion current due to other ions than those produced by  $SF_6$ , (i.e., by the gas emanating from the walls of the test chamber) was eliminated in the following way. The butterfly valves were again closed and the pressure in the test chamber was allowed to increase without feeding any SF<sub>6</sub> gas. No deflection of the ion-micro-ammeter was observed during this operation, thus confirming that the current detected originally was due to negative ions produced from SF<sub>6</sub>.

#### (b) Mass Spectrometer Measurements

The time-of-flight mass spectrometer was first tested by allowing the SF<sub>6</sub> gas to fill up the test chamber at a pressure in the range of  $10^{-4}$  mm of Hg. An ion spectrum was observed on the oscilloscope and although in general the SF<sub>6</sub><sup>-</sup> peak was the most important, the SF<sub>5</sub><sup>-</sup> was found to be at least 50% as large as the SF<sub>6</sub><sup>-</sup> peak. At the same time, it was observed that a small F<sup>-</sup> ion started to appear at a pressure above  $2 \times 10^{-4}$  mm of Hg.

When at a later stage the SF. gas was introduced in the ion source in the form of a molecular beam it was found, as seen in Fig. 14, that the SF.<sup>-</sup> peak was considerably smaller than the SF.<sup>-</sup> peak and that no F ions could be observed. From these tests it was concluded that when the SF. gas is introduced without a net directivity, an appreciable fraction of the gas is dissociated or excited on the hot parts of the ion source, and as a result a



Fig. 14. SF ion current as function of Electron Bombardment Energy.

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larger amount of  $SF_5$  and F ions are produced. These tests have thus permitted to establish one definite advantage of introducing the gas in the form of a molecular beam.

## (c) Energy Spectrum of the Negative Ions

Some typical curves of negative ion current as a function of the electron bombardment energy are shown in Fig. 14. The complexity of the spectrum is difficult to explain in view of the fact that other (\*) mass spectrometry studies of SF6 at low pressures have established the existence of a single resonance process for electron capture by SF6 at an electron bombardment energy between 0 and 1 ev. Of course, the conditions in which the measurements reported here were made, are quite different from those in which these electron capture cross-sections were obtained. The electron bombardment current is at least 2 orders of magnitude larger and the gas density in the ion chamber is quite high. It is believed that the multiple resonances in the electron bombardment spectrum are due in part to the electrons travelling across the ion source and producing on the electron collectors secondary electrons which then enter into the ion source. These secondary electrons may have energies in the appropriate resonance range for the electron capture process. Another effect which may explain the occurrence of electron capture resonances at higher electron energy is the fact that due to

\* A. J. Ahearn and N. B. Hannay, J. Chem. Phys. <u>21</u>, 119 (1953) W. M. Hickam and R. E. Fox, J. Chem. Phys. <u>25</u>, 642 (1956)

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the multiple collisions of the primary electrons with the SF<sub>6</sub> molecules their energy is sufficiently reduced to reach the resonance point for electron capture by SF<sub>6</sub> molecules. The lens action of the slits of the electron gun may also change the pattern of the secondary electrons hitting the opposite wall of the ion source in such a manner as to produce this complex energy spectrum.

In Fig. 14 is also presented a curve of the electron bombardment as function of the electron energy. This curve was measured under the same conditions after which the ion curves shown in the same figure were obtained. It is seen that due to the well known space charge effects, the bombardment current at low energy is reduced to a very low value. This effect is believed to set the limit to the maximum negative ion current that can be attained by this method.

In fact this situation may be illustrated by normalizing the experimental curves of negative ion current as a function of electron energy to a constant electron bombardment current. This has been done for the curve corresponding to an SF, gas pressure of  $195\mu$  of Hg in the gas feed line and is shown in Fig. 15. The normalized curve was calculated for an electron bombardment current of  $100 \mu$  A. It is seen that on this basis the lower energy part of the spectrum, in contrast with the measured spectrum, forms the preponderant part of the spectrum. This difference effectively illustrates the negative ion current limitations due to space charge

(ARBITRARY UNITS) FEEDLINE PRESSURE 195 4 Hg ION CURRENT NORMALIZED TO CONSTANT BONBARDMENT | CURRENT MEASURED °õ A ELECTRON ENERGY IN ۰V



effects in the electron beam.

Another remark that must be made in connection with these curves is the fact that the first measured resonance at 3.5 ev corresponds effectively to the primary process for electron capture by SF.. The fact that this maximum occurs at 3.5 ev rather than below 1 ev is due to the fact that the electron energy scale shown in Fig. 14 and 15 was not corrected for contact and space potentials between the various electrodes. It should also be noted that in the normalized curve the resonance occuring in the range of 15 to 21 ev is much less important than those appearing at 3.5 and 8.5 ev.

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## (d) <u>Negative Ion Current as Function of SF. Gas Flow Rate</u>

The family of curves of negative ion current vs electron energy shown in Fig. 14 effectively illustrates the general variation of the negative ion energy spectrum in terms of the gas flow rate in the ion source. By means of the calibration curve of Fig. 7 it is then possible to convert the feed line pressure into absolute gas flow rate (molecules/sec) and thus obtain the variation of ion current with gas flow rate. Such a dependence is shown in Fig. 16 where the ion current corresponding to the highest peak in the spectrum (Fig. 14) is plotted as a function of the SF, gas flow rate. The absolute value of ion current plotted along the ordinate axis corresponds to the ion current received at the first dynode of the electron multiplier and is not accurate since the current gain





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calibration of the electron multiplier tube was done using an electron beam rather than a negative ion beam. It should be noted that due to interception of negative ions at the entrance and exit slit of the mass spectrometer and due to beam divergence along the drift tube a great fraction of the ions emitted by the ion source does not reach the first dynode of the electron multiplier. For this reason the current scale shown in Fig. 16 should in fact be multiplied by a factor which is estimated to lie between 10 and 100. This value is based on the fact that ion currents in the range of one microampere were measured when using a Faraday cup as an ion detector.

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The upper limit of the scale for the SF<sub>6</sub> gas flow rate in Fig. 16 corresponds to the condition where the molecular beam begins to deteriorate. It is observed that at this upper limit, set by the instrument, the negative ion current is still increasing. This result suggests that quite possibly much higher negative ion current could be obtained if a higher capacity gas collimator were used.

#### (e) <u>Ion Conversion Efficiency</u>

In Fig. 16 is also shown a plot of ion conversion efficiency as a function of the gas flow rate. This ion conversion corresponds to the ratio of the number of ions per second collected at the first dynode of the multiplier to the number of SF<sub>6</sub> gas molecules entering the ion source persecond. The values shown in the curve were calculated from the measurement of maximum ion current vs SF<sub>6</sub> gas flow rate. It is seen that the ion conversion efficiency decreases with increasing SP. flow rate up to  $3 \times 10^{414}$  molecules/sec and then levels off for the higher flow rate. The fact that ion interception occurs at the entrance and exit slits of the mass spectrometer contributes to making the values of ion conversion efficiency shown in Fig. 16 lower than they are actually when one considers the true ion source efficiency. This factor, which for the same reasons as given above, may be in the range of 10 to 100, thus bringing the ion conversion efficiency to quite a substantial value.

In fact, the ion conversion efficiency quoted here corresponds to one transit of the gas molecules through the ion source. Since it has been shown in Section III that a good collimation of SF. gas flow was attained and an efficient gas collector could be operated, it is then possible to recirculate the gas not ionized in its first transit through the ion source. In this way it is thus realized that an ion conversion efficiency at least as high as 90% (allowing for negative ions formed in the source but lost to walls of the ion chamber by diffusion) can be attained with the ion source investigated here.

#### V CONCLUSIONS

The important features of this experimental study may be summarised as follows.

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- Using a gas collimator it has been possible to introduce the SFe gas into the ion source in the form of a well-defined molecular stream and thus demonstrate the feasibility of recirculating the SFe gas through the ion source.
- 2. A significant negative ion surrent was obtained by direct bombardment of SF<sub>6</sub> molecules with electrons. The basic process for the generation of negative (SF<sub>6</sub>) ions by this method was the capture of an electron by the SF<sub>6</sub> molecules.
- 3. The negative ion mass spectrum was found to be composed of SF<sub>5</sub><sup>-</sup> and SF<sub>6</sub><sup>-</sup> negative ions. It was observed that when introducing the gas in the form of a stream the SF<sub>5</sub><sup>-</sup> contribution to the total ion current was negligible.
- 4. The generation of SF<sub>6</sub><sup>-</sup> ions as function of the electron bombardment energy exhibits a multiple resonance spectrum. The presence of several peaks in the spectrum is attributed to instrumental effects and to multiple collisions of the electrons with the gas molecules in the ion source.

- 5. Due to space charge effects, it is found that the maximum negative ion current that can be produced by this method is limited by the electron bombardment current that can be injected at low electron energies. The negative ion current produced by this source was in the range of  $1 \ \mu A$ .
- 6. The negative ion current increases almost linearly with the SF<sub>6</sub> gas flow rate in the region where the gas collimator reaches its maximum handling capacity. This indicates that still higher negative ion currents could be generated if a higher capacity gas collimator were used.
- 7. It is estimated that the ion conversion efficiency for one transit of the SF<sub>6</sub> gas through the ion source may be as high as 3%. By including the fact that the SF<sub>6</sub> gas not ionized in its first transit through the ion source can be recirculated, it is then estimated that the overall ion conversion efficiency for this ion source is as high as 90%.

From these results it may be concluded that a high ion conversion efficiency may be attained and still larger ion currents are possible by flowing more SF. gas through the ion source. The main limitation to this method of producing an intense beam of negative ions resides in the space charge effects taking place in the bombarding electron beam. For this reason it is recommended that further investigations on intense negative ion sources using  $SF_6$  should be carried out using techniques for eliminating space charge effects in the low energy electrons (e.g. by using positive ions). It is believed that if the space charge effects can be eliminated, the generation of intense negative ion beams useful for electrical propulsion is feasible.

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