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Technical Report 19

A LOW ENERGY ALKALI ATOM BEAM SOURCE

By: Donald C. Lorents Graham Black

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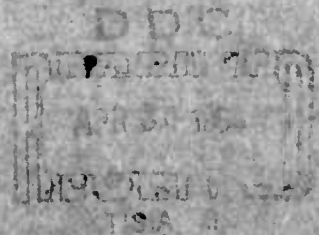
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February 1964

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*By: Donald C. Lorents Graham Black
 Molecular Physics Department*

SRI Project No. 3857

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ABSTRACT*

A method of obtaining beams of alkali atoms in the eV energy range has been developed. The energy range and beam intensities make this a useful tool for studying ionizing reactions of importance in re-entry physics. The source has been used to produce a lithium atom beam but can be used equally well with any other alkali.

Lithium ions emitted thermally from a β -eucryptite source are accelerated and focused into a ribbon beam by an electric field designed to compensate for the space charge field. Beams with current densities near the theoretical space charge limit have been attained at mean energies of 2 eV and above with a total energy spread of ± 0.3 eV. The β -eucryptite source emits impurity ions of Na and K, but at temperatures above 1000°C the impurity ions comprise less than 0.2% of the beam.

The ion beam traverses at right angles a high-density collimated thermal lithium vapor beam issuing from an oven. Neutralization of the ion beam occurs by charge transfer interactions between the ions and vapor atoms and conversion efficiencies of 10-20% are easily attained. The vapor beam is frozen out on the cooled walls of the chamber which surrounds the charge transfer region so that the fast atom beam which emerges is not contaminated by any appreciable thermal flux. With the present geometry neutral beam intensities range from about 1×10^{-10} Amperes (ion equivalent) at 5 eV to about 2×10^{-9} Amperes at 30 eV and then gradually increase to 2×10^{-7} Amperes at 500 eV. The measured ion beam densities and conversion efficiencies demonstrate that neutral beam densities of the order of 10^{-9} Amperes/cm² (ion equivalent) can be obtained at energies below 10 eV with a more ideal geometry.

The total cross section for charge transfer between lithium ions and atoms has been measured for relative energies of 14 eV to 1000 eV. The slow ions from charge transfer interactions were collected in a negatively biased cage

*This report describes work completed as of November 30, 1963 in the Chemical Physics Division of SRI under contract SD-103, ARPA order 281-62.

surrounding the intersection volume of the two beams. The vapor beam density for the absolute cross section measurements was obtained by a titration technique. The energy dependence of the cross section shows the expected behavior for resonant charge transfer.

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I INTRODUCTION

Involvement of military and space efforts in high temperature gas technology has brought to light many problems of a basic nature which must be solved. One of the most pressing of these is that of gaining an understanding of the mechanisms that control ionization in high temperature gases. These mechanisms can best be understood by studying the individual reactions of the gas species. However, the reactions are usually difficult to study directly, and frequently reactions and reaction rates must be inferred from the macroscopic behavior of an ionized gas. New and various techniques to study the reactions directly are needed. An example is the problem of ionization in a shock wave. A large number of experiments in which ionization has been observed by a variety of techniques in shock waves has not clearly elucidated the initiating mechanism.¹⁻⁴ Direct measurements of collisional ionization and collisional excitation cross sections at energies near the threshold of the reaction would clearly aid the interpretation of these shock tube observations.

The subject of this report is the development of a beam source of low energy monoenergetic alkali atoms. Although the intensities at the lowest energies are low, many direct measurements of cross sections needed to understand ionized gases can be made using this beam; collisional ionization and excitation cross sections at energies near threshold are examples. Because of their low ionization potentials the alkalis play a significant role in many ionized gases either as contaminants or as directly ionizable species.

II NEUTRAL ALKALI BEAMS BY CHARGE TRANSFER

A. Introduction

To obtain a well-collimated intense atom beam by charge transfer requires that these same properties be present in the incident ion beam. Intense alkali ion beams are readily available from thermal sources. Surfaces coated with alkali aluminosilicates can be used as ion sources and yield current densities of 1 ma/cm^2 when heated to sufficiently high temperatures.⁵ Since the ions are emitted thermally, relatively low energy ion beams ($\geq 1 \text{ eV}$) having a small energy spread can be obtained.

Converting the ion beam into a neutral beam with high efficiency requires a dense target gas with a high cross section for charge exchange. The highest charge transfer cross sections occur for symmetric charge transfer reactions (that is Li^+/Li , Na^+/Na , etc.). This process has been applied to the conversion of lithium ions to neutrals by directing the ion beam through a dense vapor beam of lithium. The cross section for charge transfer of Li^+ in Li has been measured as a function of energy.

Views of the equipment are shown in Figs. 1 and 2 and a schematic of the apparatus is shown in Fig. 3.

B. Ion Source

Of the alkali ion sources, the one most studied is β -eucryptite,^{6,7} lithium aluminosilicate ($\text{Li}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), which emits lithium ions above 1000°C . The thermal emission properties of this mineral make it an ideal choice for a low energy ion source.

The ion gun consists of an emitter and accelerating electrodes, mounted on a lavite holder designed to provide freedom to adjust the parameters of the source. Its principal features are shown in Fig. 4. The electrode geometry was based on the Pierce principle^{8,9} and was developed in accordance with a design of Von Ardenne.¹⁰

The β -eucryptite was prepared from natural lithium carbonate (Li_2CO_3), alumina (Al_2O_3), and silica (SiO_2) as described by Allison and Kamegai⁶ and by

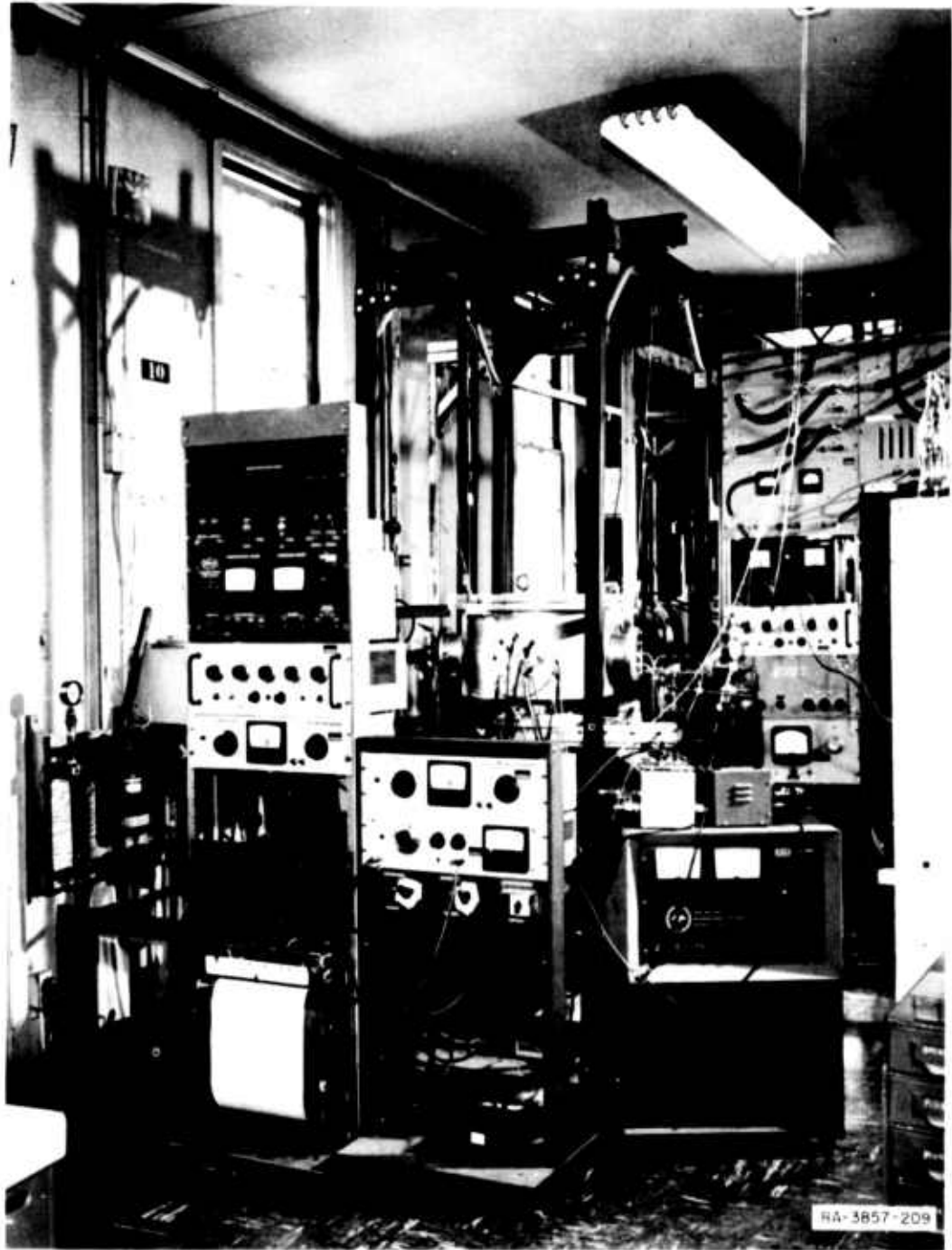


FIG. 1 GENERAL VIEW OF THE APPARATUS

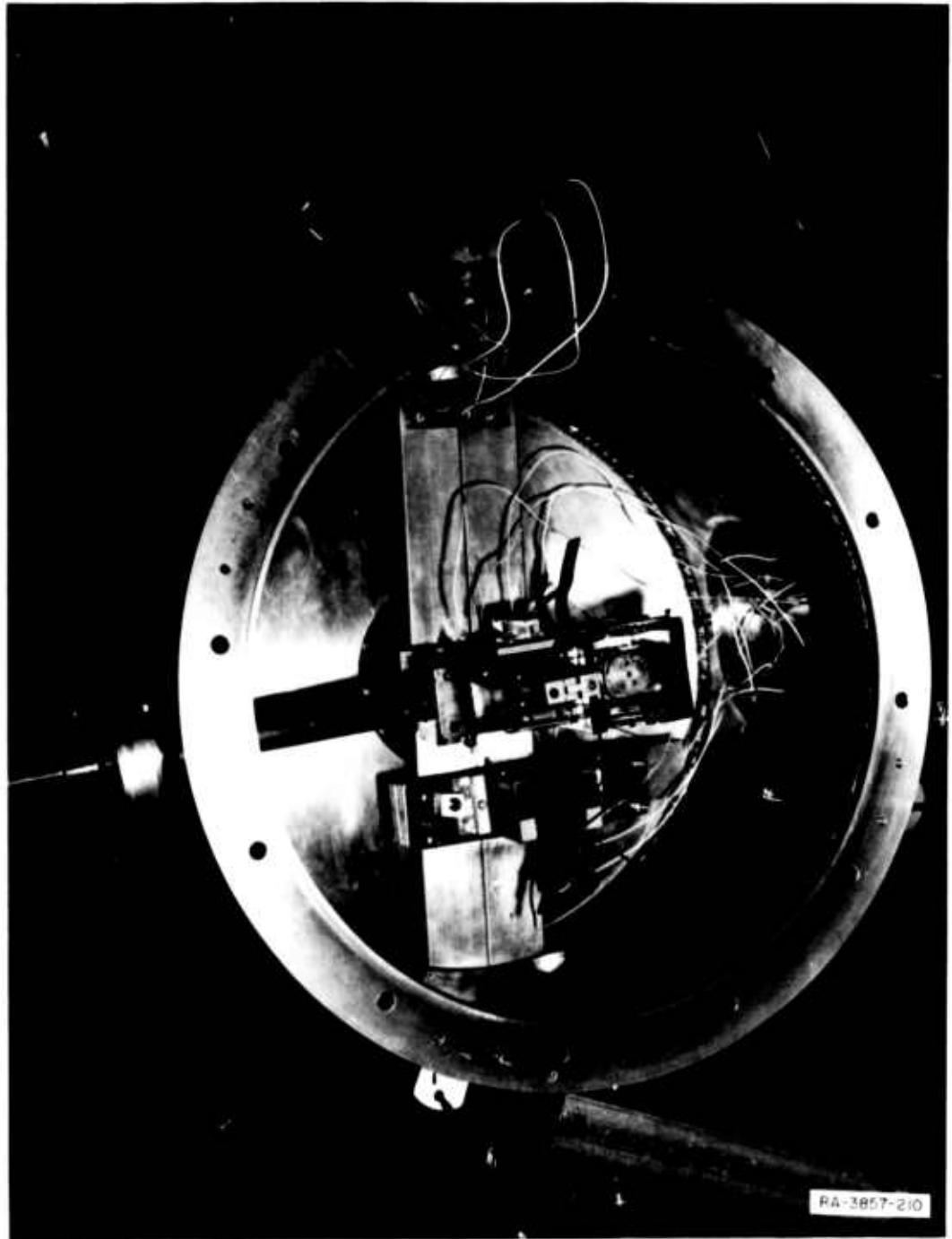


FIG. 2 VIEW OF THE EQUIPMENT INSIDE THE VACUUM CHAMBER

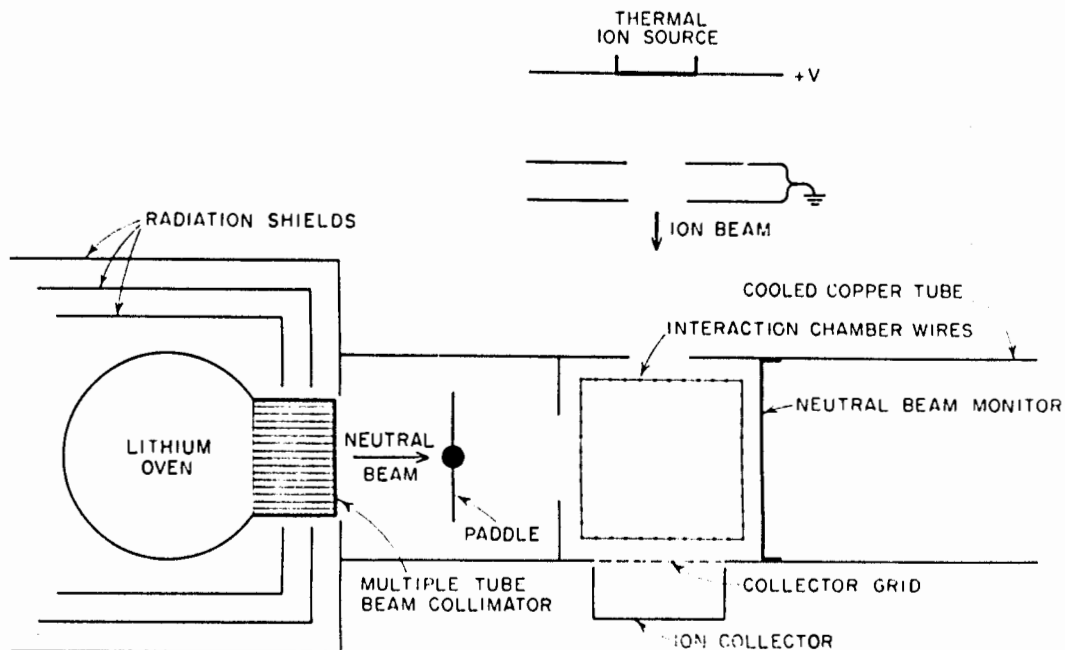


FIG. 3 SCHEMATIC OF THE APPARATUS

Waldron.¹¹ It was applied as a suspension in amyl acetate containing a little "parlodion," to the 1.5 x 1 cm surface of a tantalum boat which had been scoured. A tungsten wire grid spot-welded to the boat assisted adhesion of the coating and served to maintain the β -eucryptite surface at the same potential and temperature as the boat.

The boat was heated by a 0.007-in. tungsten filament wound through alumina tubing and embedded in Alundum cement. Heat losses were minimized by two nickel radiation shields on top of the boat.

After assembling the source, installing in the vacuum chamber and evacuating, the heater power was raised very slowly to allow slow outgassing of the source. A temperature of 600°C at the β -eucryptite surface (determined with an optical pyrometer) was reached with 15 watts supplied to the heater. At

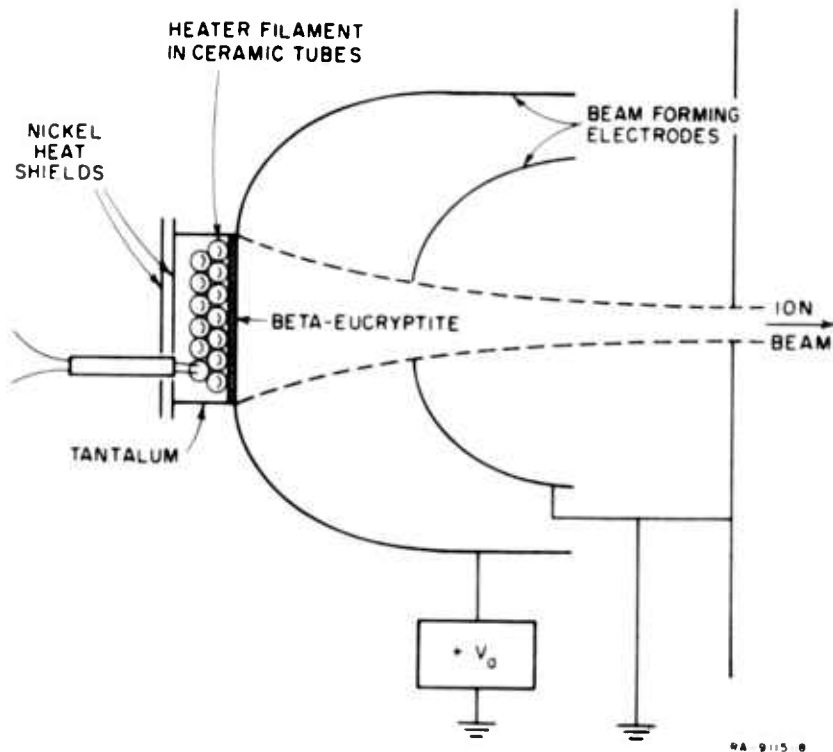


FIG. 4 LITHIUM ION SOURCE

this temperature the first ion emission was observed. The emission continued to increase as the temperature was raised to 1050°C with 75 watts input to the heater. The source was then installed in a 0.1 to 5 keV ion accelerator which has been developed in the Molecular Physics Department of SRI¹² and the ion beam was mass-analyzed at several source temperatures. The data, shown in Fig. 5, indicate that the β -eucryptite emits impurity ions of sodium and potassium which predominate at the lower temperatures, but above 1000°C the impurity ions comprise less than 0.2% of the beam.

At 1000°C , with a heater power of 70 watts, the lifetime of the source was governed by heater failure. This usually occurred after a few tens of hours operation and appeared to result from some reaction of the tungsten with the

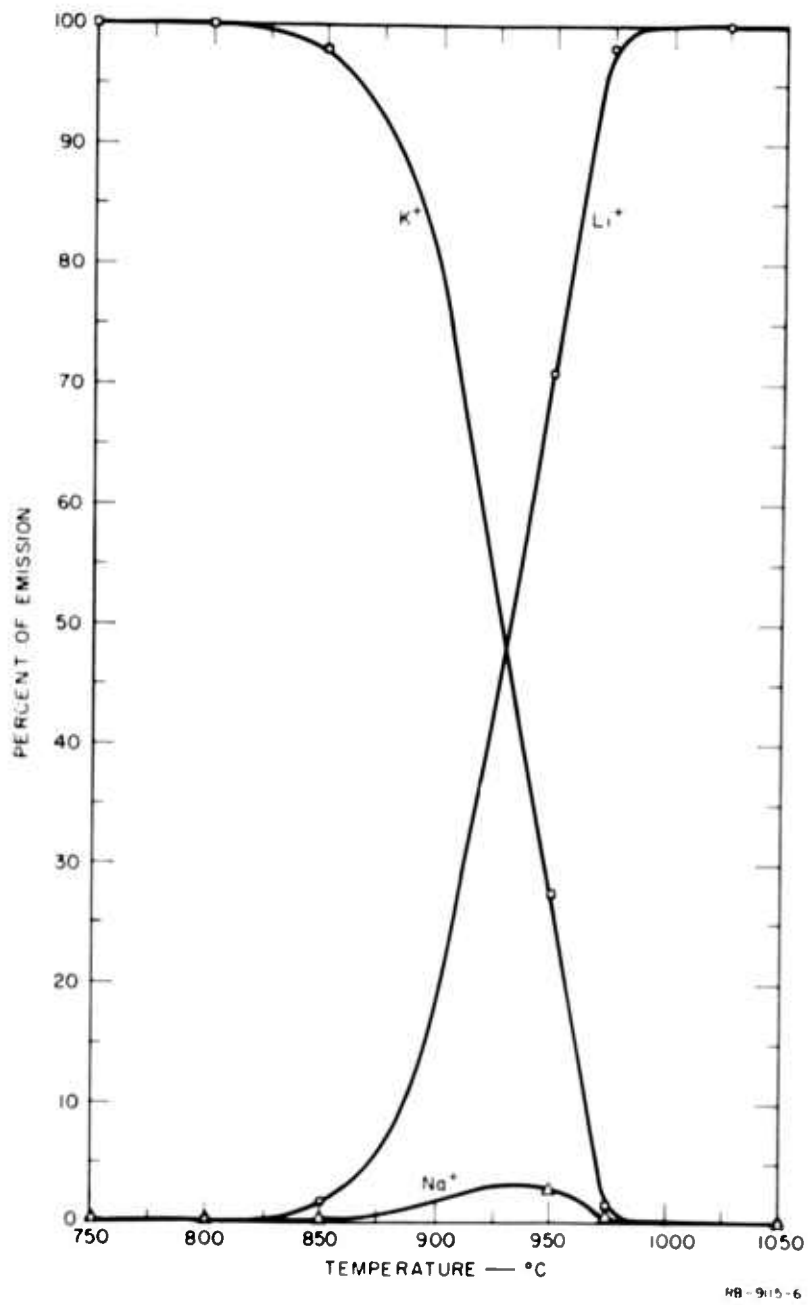


FIG. 5 THE MASS-ANALYZED EMISSION OF THE ION SOURCE FROM 750 TO 1050 °C

Alundum cement. This lifetime was deemed satisfactory for the present experiments and no attempt was made to improve it. An order of magnitude improvement would have to be made in the heater lifetime before lithium exhaustion from the β -eucryptite limited the source lifetime.

For the geometry of our source, substitution of the various parameters in the equations given by Von Ardenne¹⁰ predicted a maximum beam current of lithium ions of

$$I = 1.7 \times 10^{-8} V_a^{3/2}$$

for the 1.5 cm² emitter employed in this experiment. This relationship, the corresponding one for potassium ions, and the experimental values are shown in Fig. 6. The experimental beam currents were early data determined with a source temperature of 825°C and refer to potassium ion emission. Thus the source can produce beams near the theoretical space charge limit at mean energies of 3 eV and above.

C. Lithium Vapor Beam-Oven

The most efficient conversion of the lithium ion beam to a neutral beam requires a target of lithium vapor. A collimated vapor beam was generated in an oven-collimator system operated at about 700°C. The vapor beam was directed across the ion beam and subsequently condensed on a cooled surface.

The soft iron cylindrical oven was capped with a threaded cover using a metal O-ring as a seal. A chromel-alumel thermocouple spot-welded to the cover measured the oven temperature. The heater, wound with 0.015-in. tantalum wire through alumina tubing (to insulate it from the oven), was confined to a side arm, located near the top of the oven, which contained the collimating system. This positioning of the heater ensured that no lithium condensed in the collimator. A triple-walled radiation shield surrounding the oven reduced heat losses so that a power input of 125 watts raised the oven temperature above 700°C.

The collimator presented an area 2 x 1/2 cm for the effusion of lithium and was 1.5 cm long. It was made from alternate layers of plane and crinkled

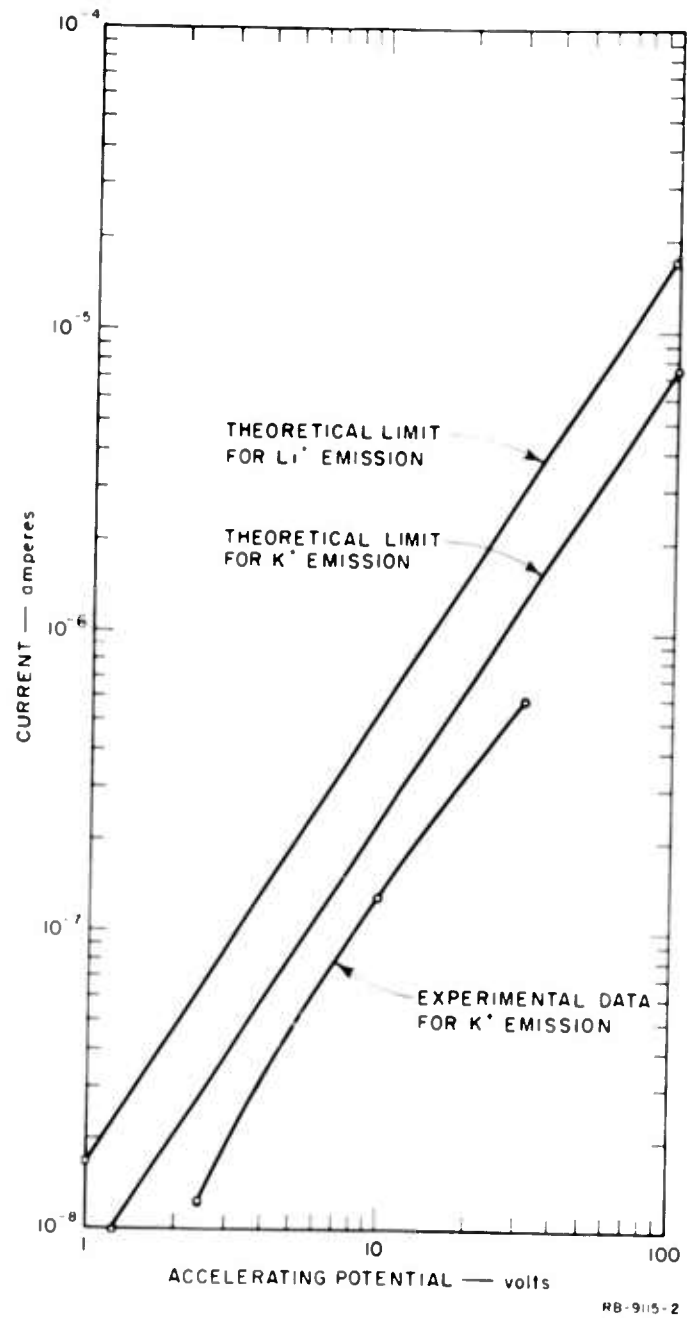


FIG. 6 ION SOURCE EMISSION AS A FUNCTION OF ACCELERATING POTENTIAL

0.0003-in. tantalum foil. The final collimator thus resembled a bundle of small diameter (~0.020-in.) tubes. Such an array should be capable of functioning as an efficient collimator for oven pressures around 0.1 mm Hg.¹³ The vapor beam was further collimated with a 2 x 1/2 cm slit just in front of the interaction region. Between this slit and the oven a paddle, controlled from outside the vacuum, served to interrupt the beam. After passing through the interaction region, the vapor beam was condensed on the copper shell which connected with a 1-in.-diameter copper rod passing outside the vacuum into a dry ice/acetone mixture.

D. Lithium Vapor Beam-Flux Measurements

To measure the vapor beam flux, a surface ionization detector was first built. This operates on the principle that the ratio of ions to atoms leaving a metal surface is governed by¹⁴

$$\frac{n^+}{n^0} = \frac{w^+}{w^0} e^{-(I-\phi)/kT}$$

where w^+/w^0 = ratio of statistical weights of the ion and atom = 1/2 for alkalis, I = ionization potential of the atom, and ϕ = work function of the surface. This ratio can become very large if ϕ is larger than I . For lithium ($I = 5.36$ volts), a tungsten oxide surface ($\phi = 5.9$ volts)¹⁵ is normally used. Such a surface can convert incident lithium atoms into ions with an efficiency of 100%. The ion current thus monitors the neutral beam striking the surface.

The detector consisted of a 0.001-in. tungsten wire mounted concentrically in a 1-in.-diameter cylinder of 0.005-in. tantalum. Holes were cut in the cylinder wall to allow the neutral beam to enter and leave. It was mounted in the copper shell on the downstream side of the interaction chamber in the path of the vapor beam.

Before using the detector, it was necessary to determine the conditions under which a tungsten oxide surface could be produced on the tungsten wire. This was done by investigating the work function of the wire surface under various conditions.

The work function of a surface can be determined from its electron emission using the Richardson-Dushman equation¹⁶

$$j = AT^2 e^{-\phi/kT}$$

where

j = electron emission (amps/sq cm)

and

ϕ = work function of the surface.

Thus a plot of $\ln(j/T^2)$ against $1/T$ has a slope of $(-\phi/k)$.

Such an investigation for the detector wire, increasing the temperature of the wire by increasing the current through it, showed that under a vacuum of 1×10^{-6} mm Hg pressure, the work function of the surface below 1900°C was about 5.9 volts and reverted to that of tungsten ($\phi = 4.7$ volts)¹⁵ above 2000°C . It therefore appeared that no special efforts need be taken to continuously oxygenate the wire under the pressures prevailing during an actual run (with the oven and ion source operating, pressures of 1×10^{-5} mm Hg were normal).

To determine the efficiency of the detector as a function of its temperature, a constant lithium flux of about 10^{13} atoms/sq cm/sec (from the oven at $\sim 400^\circ\text{C}$) was allowed to fall on the wire. The resulting ions were collected on the surrounding cylinder biased at -50 volts and measured as a function of wire temperature.

The response of the detector as a function of its temperature is shown in Fig. 7. This was drawn by interpreting the constant signal obtained in the temperature range 1150°K to 1450°K as 100% conversion of neutrals to ions and normalizing the rest of the data to this. The experimental data of Datz and Taylor¹⁷ for lithium on tungsten are included together with a theoretical curve calculated from the work of Hague and Donaldson¹⁵ on the work functions of tungsten and tungsten oxide surfaces. Deviations from the theoretical curve probably are mainly due to a temperature gradient along the wire.

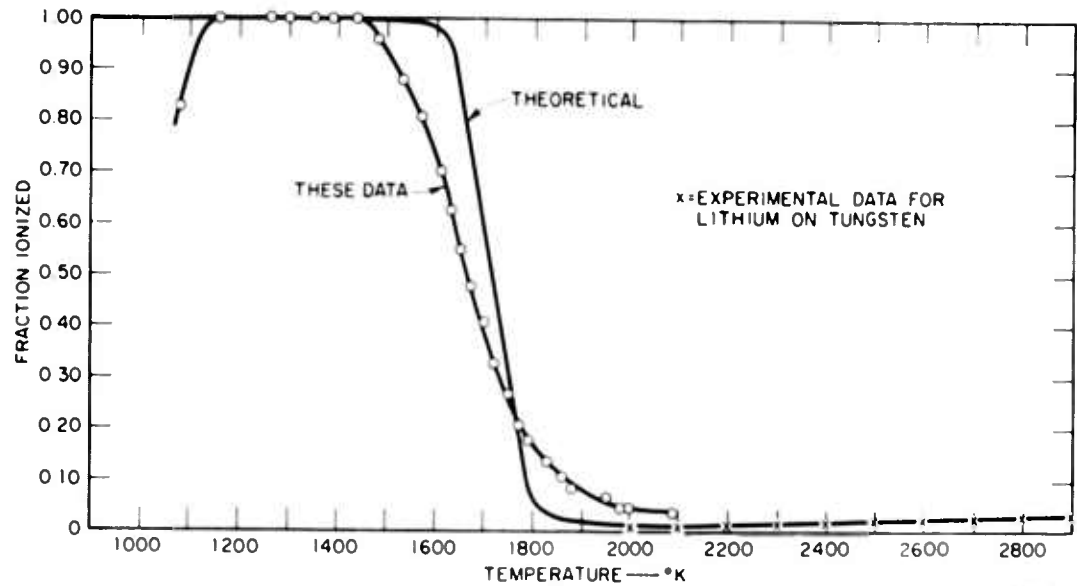


FIG. 7 IONIZATION OF LITHIUM ON TUNGSTEN AND OXYGENATED TUNGSTEN SURFACES

With the detector operating at 100% efficiency at 1300° K, the flux incident on the detector was gradually increased by further raising the oven temperature. At a flux of about 10^{16} atoms/sq cm/sec the response of the detector levelled off and then decreased as the flux was further increased. It appeared therefore that the residence time of the atoms on the surface was sufficiently long to effect some fractional surface coverage under this flux and thus reduce the efficiency of the detector. Under the maximum flux required during a charge transfer experiment ($\sim 10^{18}$ atoms/sq cm/sec) it was therefore impossible to operate the detector at a temperature giving 100% conversion of neutrals to ions. At higher temperatures, the uncertainty in the efficiency of neutral to ion conversion would introduce an unnecessarily large error into the experiment.

An integrating chemical method was therefore developed and used to measure the neutral beam density. All the lithium passing through the interaction chamber was condensed on a neutral beam monitor (Fig. 3) consisting of a copper plate with 1-mm-diameter copper wires spaced 1 mm apart mounted approximately 1/8-inch above its surface. When assembled in the copper tube

the wires were horizontal. Measurement of the amount of Li condensed on each of the wires and on the plate enabled a profile of the neutral beam to be established (Fig. 8) as well as the total flux through the interaction chamber.

After an experiment the neutral beam monitor was removed from the copper shell. The lithium on each wire and on the plate was dissolved in water using separate flasks. This converted the lithium to lithium hydroxide (any surface nitride on the lithium would be similarly converted). Any ammonia (from the nitride) was removed by boiling and the lithium determined as the hydroxide by titrating with standard acid using phenolphthalein as indicator. The accuracy of this method for determining the amount of lithium deposit was $\pm 3\%$.

To relate the total flux at the monitor to the lithium concentration in the interaction region a probe was devised. This consisted of a 2-mm-wide strip of 0.020-in. thick copper (2 mm being the width of the ion beam). With the ion beam collector and its grid removed, the probe was inserted along the center

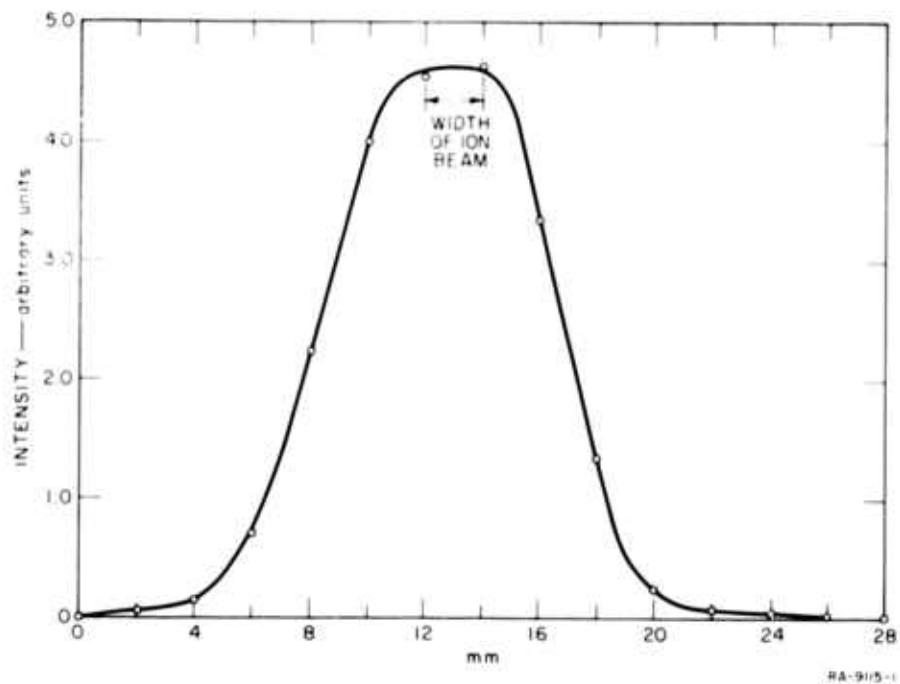


FIG. 8 NEUTRAL BEAM PROFILE

of the interaction region. By measuring the Li flux using the probe and the beam monitor, the monitor was calibrated in terms of the average flux in the interaction region.

Measurements with the probe in position showed that an average thermal lithium flux of 4×10^{17} atoms/sq cm/sec over the 1 1/2-inch width of the interaction chamber was produced at an oven temperature of about 700°C. This would indicate a flux $> 10^{18}$ atoms/sq cm/sec at the center of the chamber.

E. Experimental Apparatus and Procedures

Figure 1 is a view of the electronic and external parts of the vacuum system. Figure 2 is a top view of the vacuum chamber with the top cover removed. The ion source, oven, and the cooled copper box surrounding the interaction region are seen mounted in the chamber. Figure 3 is a schematic layout of the apparatus.

The volume of intersection of the ion and vapor beams is surrounded by a cage consisting of a pair of tantalum plates mounted above and below the beams and connected by fine vertical grid wires. Slow charge transfer ions created in the interaction volume are collected on the cage by biasing it a few volts negative. The grid wires intercept about 2% of the ion beam as it traverses the cage. Since the Li vapor beam monitor was calibrated with the cage in place, interception of Li by the wires was accounted for.

The ion beam was defined by two 1 x 0.2 cm slits mounted 5 cm apart. The second of these slits was mounted on the copper shell surrounding the interaction volume. After traversing the interaction volume the ion beam was collected in a 2 x 1 cm Faraday cup. The size of the cup insured that the ion beam was not attenuated appreciably by scattering. A grid in front of the cup, negatively biased, suppressed any secondary electron emission from the cup.

The oven was charged with 2 to 3 grams of lithium in an argon atmosphere in a dry box. Before removing from this atmosphere, the lithium was covered with benzene to reduce chemical reaction with the atmosphere while assembling the apparatus. The oven was then removed from the dry box and the top secured. It was mounted in the radiation shields and the thermocouple and heater wires connected. Meanwhile, the ion source had been aligned with the two slits in the copper shell prior to attaching the ion collector and its grid.

After checking all the electrical connections, the top plate of the vacuum chamber was placed in position and the apparatus carefully evacuated (to allow slow removal of the benzene). Before any experiments were performed the oven and ion source were outgassed. This was done by raising the oven temperature to about 250° C for twelve hours and the ion source to 1000° C for two hours.

With the oven temperature steady, and hence a steady thermal beam of lithium vapor, the paddle was raised to allow the thermal lithium beam to intersect the steady ion beam in the center of the interaction chamber. The geometry of the intersection is shown in Fig. 8. The chamber was biased negatively to collect the slow ions resulting from charge exchange when the two beams intersected. The amount of bias required was determined by increasing the negative potential on the chamber until the slow ion current became saturated. Raising the paddle gave rise to an increase in the current to the interaction chamber and an attenuation of the collector current. These two signals, found to agree within experimental error, measured the total fast neutral beam. This result was evidence that the ion beam attenuation was due only to charge transfer and did not include a contribution from scattering. Its magnitude was determined as a function of the ion beam energy.

A 3-gram charge of lithium in the oven was sufficient for several hours' experimentation. After an experiment the apparatus was allowed to cool before opening the equipment to air and determining the lithium on the monitor. The exposure time of the monitor was determined by addition of the separate exposure times as they were presented on the recorder. Thus the average flux could be determined.

F. Conversion Efficiencies and Beam Densities

For relative cross section or conversion efficiency measurements, it was sufficient to operate the oven at constant temperature thereby maintaining constant vapor beam flux. The conversion efficiencies were measured as a function of energy and the results of three runs were normalized to obtain one relative curve. Typical ion-to-neutral conversion efficiencies at an oven temperature of about 700° C ranged from 8.5% at 1000 eV to 15% at 120 eV and 25% at 15 eV. The resulting fast neutral beams ranged from about 1×10^{-10} A (ion equivalent) at 5 eV to about 2×10^{-9} A at 30 eV and then gradually increased to 2×10^{-7} A

at 500 eV. The ion beam intensity was severely limited by the collimation required for reliable charge transfer measurements. For the purpose of obtaining maximum beam intensity the collimation can be reduced allowing the ion source to be moved closer to the vapor beam. By this means space charge spreading losses will be reduced and the resulting neutral beam intensities can be increased by about a factor of 10. Quantitative conversion efficiency measurements were not extended below 14 eV because scattering effects became noticeable, but it is clearly possible to obtain useful neutral beams to energies of 1 eV.

III RESONANCE CHARGE TRANSFER CROSS SECTION

No information on the absolute cross section for Li^+/Li charge exchange could be found in the literature; hence, measurement of it was undertaken because little additional effort was required. To determine absolute cross sections, the concentration of Li in the interaction region relative to the total flux must be determined. It was at this stage, therefore, that the probe measurements described in Section IID were performed.

Charge exchange measurements were performed at one energy to determine the absolute cross section with minimum error. The ion source and oven were allowed to stabilize for a time before starting the measurement to insure constant beams for the run. With the incident ion energy fixed at 100 eV and -15 V on the interaction chamber (that is, for 115 V ions passing through the interaction chamber), the charge transfer was monitored for some time (say, 30 minutes) at this one energy. The charge transfer current and the ion beam current were averaged for the run and these numbers, together with the average vapor flux, were used to calculate the cross section. The results of two runs were found to be within $\pm 6\%$ of the mean which is within the estimated error limits.

These measurements established the absolute cross section at 115 eV as $1.43 \pm 0.11 \times 10^{-14} \text{ cm}^2$. The relative data at other energies were then normalized at this energy. The variation of the cross section with energy is shown in Fig. 9.

For a symmetric resonance process of the type



the cross section, Q , can be represented¹⁸ by

$$Q^{1/2} = a - b \log E \quad (E = \text{ion energy}) .$$

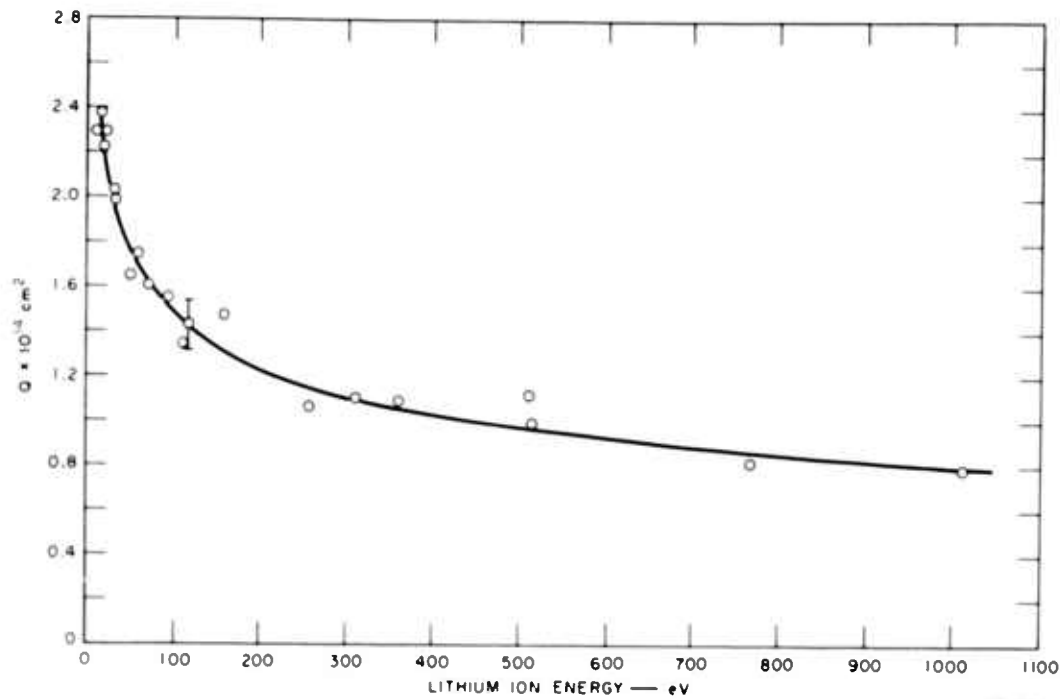


FIG. 9 CROSS SECTIONS FOR CHARGE TRANSFER BETWEEN Li^+ IONS AND ATOMIC Li

Such a relationship was found for the data of Fig. 9, a plot of $Q^{1/2}$ against $\log E$ yielding a straight line (Fig. 10).

Evaluation of the constants a and b gives

$$Q_{(\text{Li}^+/\text{Li})}^{1/2} = (19.3 \pm 0.3) \times 10^{-8} - (3.5 \pm 0.2) \times 10^{-8} \log E$$

where the cross section Q is given in units of square centimeters, and E is expressed in electron volts.

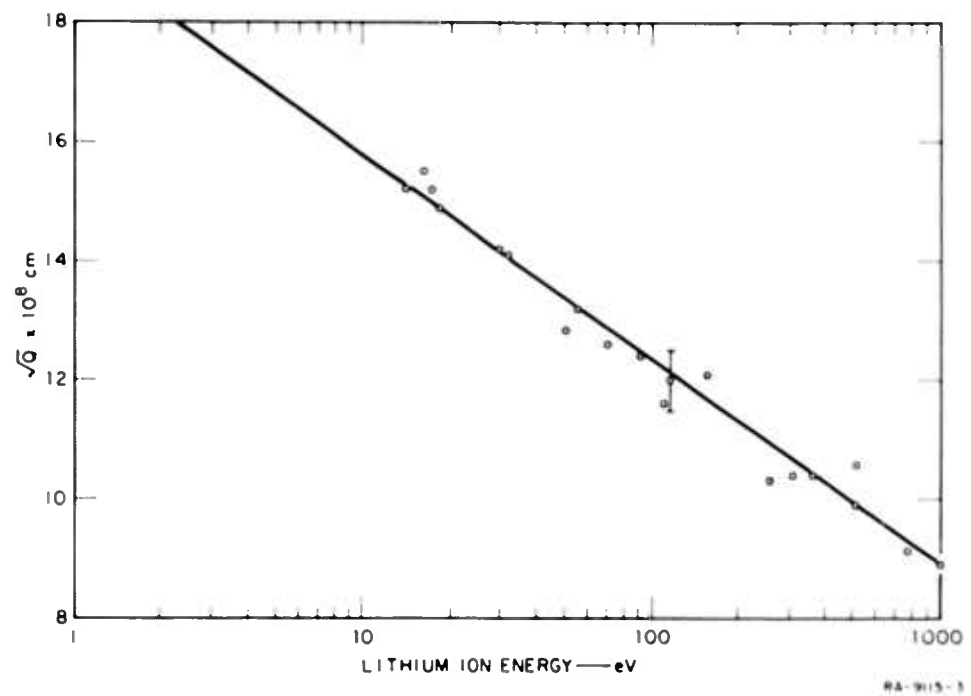


FIG. 10 SYMMETRIC RESONANCE CHARGE TRANSFER IN LITHIUM

IV CONCLUSIONS

These experiments have demonstrated the feasibility of the charge exchange method for producing monoenergetic beams of lithium atoms in the eV energy range. Beams of sufficient intensity to study differential scattering, and inelastic interactions such as ionization and excitation at energies of concern in high temperature gases have been obtained. The technique has been developed for lithium and is applicable to any of the other alkalis.

The absolute cross section for resonant charge transfer in the Li^+/Li system has been obtained as a function of energy from 14-1000 eV.

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