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Vol. I

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FOREWORD

The study of the chemical changes consequent upon the nuclear transformation of an atom that is linked with other atoms in a molecule and surrounded by other similar or dissimilar molecules has intrigued chemists for a number of years. This interest is certainly not static but if anything is increasing. The main theme of this meeting was a discussion of the suggestions and theories that have been advanced to explain the wealth of experimental observations on the behaviour of atoms at energies and in situations not normally accessible in the laboratory. Though the subject has some practical implications in the preparation of radioisotopes, this was not an important consideration at this Symposium.

The first Symposium on hot-atom chemistry organized by the Agency was held in Prague in October 1960. Comparison of the past and the present state of the subject shows that a greater variety and sophistication of techniques are now being applied as the simpler approaches used in the past have been shown to be inadequate. Progress has been made in the understanding of the simpler gas system, but in liquids and solids there is still much to clarify. It is also of interest that for the majority of the work reported in these Proceedings a reactor was the radiation source, and in this field much experimental work still remains to be done.

The Symposium on Chemical Effects Associated with Nuclear Reactions and Radioactive Transformations was held from 7 to 11 December 1964 in Vienna, and was attended by 136 participants from 29 countries and 4 international organizations. It was organized by the International Atomic Energy Agency in co-operation with the Joint Commission on Applied Radioactivity. The publication of these Proceedings makes the content of the papers and discussion available to a wider audience than was possible at the meeting in Vienna.

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IONIZATION EFFECTS AND TRITIUM HOT ATOM STUDIES IN THE GAS PHASE
(Sessions 1 and 2)

MASS SPECTROMETRIC STUDIES OF REACTIONS OF RECOIL IONS WITH MOLECULES*

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Abstract — Résumé — Аннотация — Resumen

MASS SPECTROMETRIC STUDIES OF REACTIONS OF RECOIL IONS WITH MOLECULES. Mechanisms of the reactions of recoil species from nuclear transformation have previously been inferred from the distribution of the stable products. Two alternative experimental approaches, which use mass spectrometric techniques to identify the transient ionic intermediate products of recoil reactions, are described. These experiments provide (1) a direct study of modes of energy loss by high-energy tritium recoils during slowing down to the range of chemical reaction energies and (2) evidence for the mechanism of the chemical reaction with CH_4 of the low-energy $(THe^3)^{\dagger}$ daughter from β^- decay of T_2.

Ionic products from interactions of 0.8 - 3.75 MeV protons (as stand-ins for tritons) with several isolated noble gas atoms and hydrocarbon molecules (methane, acetylene, ethylene, ethane, propane and n-butane) were observed in a portable mass spectrometer that had special constructional features. Species in various charge states were produced from the noble gases. Partial ionization cross-sections determined for the individual charge states varied between 10^{-15} and 10^{-20} cm^2 $molecule^{-1}$. Collision of a proton with a polyatomic hydrocarbon molecule resulted in ionization and extensive fragmentation. Only singly-charged fragments were found. Comparisons were made with the effects provoked by high energy electrons with the same velocity as that of the protons. The results show that energetic tritium recoils from the $He^3(n, p)T^6$ and $Li^6(n, \alpha)T$ nuclear reactions must lose kinetic energy through inelastic ionizing and exciting collisions with the medium, and that the recoils behave much like any ionizing agent. Consequences of these processes in "hot-atom" chemistry are discussed.

ÉTUDE, A L'AIDE D'UN SPECTROMÈTRE DE MASSE, DES RÉACTIONS ENTRE IONS DE RECUL ET MOLÉCULES. Jusqu'à présent, on a déterminé les mécanismes des réactions provoquées par les espèces chimiques de recul provenant de transformations nucléaires, en se fondant sur la distribution des produits stables. L'auteur décrit deux autres méthodes expérimentales qui font appel à un spectromètre de masse pour identifier les produits ionisés intermédiaires des réactions de recul. Ces expériences permettent: 1. d'étudier directement les voies par lesquelles les tritons de recul dotés d'une haute énergie perdent une partie de celle-ci lorsqu'ils sont ralentis au point d'atteindre la gamme des énergies des réactions chimiques; 2. d'obtenir des indications sur le mécanisme de la réaction chimique entre CH^4 et $(T^3He)^+$ de faible énergie, produit de la décroissance β^- de T_2.

Les produits ionisés provenant des interactions entre protons de 0,8 à 3,75 MeV (utilisés à la place de tritons) et plusieurs atomes de gaz rares et molécules d'hydrocarbures isolés (méthane, acétylène, éthylène, éthane, propane et n-butane) ont pu être observés à l'aide d'un spectromètre de masse portatif spécialement conçu à cet effet. Les gaz rares ont donné des espèces présentant différents états de charge. Les sections efficaces partielles d'ionisation, déterminées pour les différents états de charge, varient de 10^{-15} à 10^{-20} cm^2 par molécule. La collision d'un proton avec une molécule d'hydrocarbure polyatomique a provoqué une ionisation et une fragmentation poussée. L'auteur n'a trouvé que des fragments à charge unique. Il a comparé ces effets avec ceux qui sont provoqués par des électrons de haute énergie ayant la même vitesse que les protons. Les résultats montrent que les tritons de recul de haute énergie, qui proviennent des réactions nucléaires $^3He(n, p)T$ et $^6Li(n, \alpha)T$ doivent perdre une partie de leur énergie cinétique du fait de chocs inélastiques ionisants et excitants avec le milieu, et que les atomes de recul ont un comportement très semblable à celui de tous les agents ionisants. L'auteur examine l'intérêt que ce phénomène présente pour la chimie des atomes chauds.

Il a utilisé des méthodes de spectrométrie de masse à «haute pression» pour prouver que l'espèce polymère $C_2H_4T^+$ constitue le principal intermédiaire ionique de longue période qui soit formé lors de la réaction

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* Based on work performed under the auspices of the United States Atomic Energy Commission.

entre le méthane et (T^{3}He)$^{+}$, produit de la décroissance β de T_2. Le précurseur CH_4T^+ de $C_2H_4T^+$ semble avoir une période trop courte pour pouvoir être observé. Les données confirment la validité du mécanisme envisagé par Pratt et Wolfgang pour expliquer l'échange isotopique entre le tritium et le méthane qui est provoqué par la décroissance de T_2.

МАСС-СПЕКТРОМЕТРИЧЕСКИЕ ИССЛЕДОВАНИЯ РЕАКЦИЙ ИОНОВ ОТДАЧИ С МОЛЕКУЛАМИ. Механизмы реакций образцов отдачи в результате ядерных преобразований раньше выводились из распределения устойчивых продуктов. Описываются два других экспериментальных метода, при которых для распознавания переходных ионных промежуточных продуктов реакций отдачи применялись методы масс-спектрометрии. В результате проведения этих экспериментов обеспечивается 1) непосредственное изучение форм потери энергии атомами отдачи трития высокой энергии во время замедления до диапазона энергий химических реакций и 2) подтверждение в отношении механизма химической реакции дочернего продукта низкой энергии (THe^{3})$^{+}$, образовавшегося в результате β-распада T_2, с CH_4.

Ионные продукты, образуемые в результате взаимодействия протонов с энергией 0,8—3,75 Мэв (например временные заменители тритонов) с некоторыми изолированными атомами благородных газов и молекулами углеводорода (метан, ацетилен, этилен, этан, пропан и n-бутан), наблюдались в переносном масс-спектрометре со специальными конструкционными особенностями. Образцы в разном зарядовом состоянии производились из благородных газов. Парциальные поперечные сечения ионизации, определяемые для отдельных зарядовых состояний, менялись в диапазоне между 10^{-15} и 10^{-20} см^{2} молекула$^{-1}$. Столкновение протона с молекулой многоатомного углеводорода приводило к ионизации и значительной фрагментации. Были обнаружены только осколки с единичным зарядом. Проводились сравнения с эффектами, вызываемыми электронами высокой энергии с такой же скоростью, как и протоны. Результаты показывают, что атомы отдачи трития большой энергии, образуемые в результате ядерных реакций He^{3}(n,p) T и Li^{6}(n, α) T, должны терять кинетическую энергию через неупругую ионизацию и возбуждение столкновений со средой и что атомы отдачи ведут себя в значительной степени так же, как ионизирующий агент. Обсуждаются результаты этих процессов в рамках химии "горячих атомов".

Методы масс-спектрометрии "высокого давления" применялись для показа, что полимерные образцы $C_2H_4T^+$ представляют собой основной долгоживущий ионный промежуточный продукт, образуемый при реакции дочернего продукта (THe^{3})$^{+}$, образовавшегося в результате бета-распада T_2 с метаном. Частица CH_4T^+ первичная в отношении C_2H_4T, по-видимому, является слишком короткоживущей, чтобы ее можно было наблюдать. Данные подтверждают предложенный Праттом и Вольфгангом механизм наведенного распада изотопного обмена трития с метаном. (Большая часть данного доклада представляет собой новый материал, часть же—обсуждение недавно опубликованных результатов).

ESTUDIO POR ESPECTROMETRIA DE MASAS DE LAS REACCIONES DE IONES DE RETROCESO CON MOLECULAS. Hasta ahora, el mecanismo de las reacciones de las especies de retroceso provenientes de transformaciones nucleares se ha deducido de la distribución de los productos estables. El autor describe otros dos procedimientos experimentales basados en la espectrometría de masas que sirven para identificar los productos iónicos intermedios de carácter transitorio de las reacciones de retroceso. Estos experimentos permiten: 1) estudiar directamente las modalidades de la pérdida de energía del tritio de retroceso de alta energía en el transcurso de su moderación hasta alcanzar los niveles de energía del orden de las reacciones químicas; 2) obtener pruebas del mecanismo de la reacción química del descendiente (T^{3}He)$^{+}$ de baja energía, proveniente de la desintegración β^{-} del T_2, con el CH_4.

En un espectrómetro de masas portátil de características especiales se has estudiado los productos iónicos provenientes de las interacciones de protones de 0,8 - 3,75 MeV (empleados en lugar de tritones) con varios átomos de gases nobles y moléculas de hidrocarburos aislados (metano, acetileno, etileno, etano, propano y n-butano). Los gases nobles produjeron especies en estados diferentes de carga. Las secciones eficaces parciales de ionización determinadas para cada estado de carga oscilaron entre 10^{-15} y 10^{-20} cm^{2}/molécula. El choque de protones con moléculas poliatómicas de hidrocarburo produjo como resultado ionización y amplia fragmentación. Sólo se observaron fragmentos de una sola carga. Se efectuaron comparaciones con los efectos provocados por electrones de elevada energía animados de la misma velocidad que los protones. Los resultados muestran que el tritio energético de retroceso proveniente de las reacciones nucleares ^{3}He(n, p)T y ^{6}Li(n, α)T debe perder energía cinética por choques inelásticos de ionización y excitación con el medio, y que las especies

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de retroceso deben tener comportamiento muy parecido al de cualquier agente ionizante. Se examinan las repercusiones de estos procesos en la química de los «átomos calientes».

Se han aplicado técnicas de espectrometría de masas a «elevada presión» para demostrar que la especie polimérica $C_2H_4T^+$ constituye el principal ion intermedio de período largo formado en la reacción del descendiente $(T^3He)^+$, proveniente de la desintegración β del T_2, con el CH_4. El ion CH_4T^+, precursor del $C_2H_4T^+$, parece escapar a la observación por tener un período demasiado corto. Los datos confirman el mecanismo propuesto por Pratt y Wolfgang para el intercambio isotópico entre el tritio y el metano inducido por desintegración.

1. INTRODUCTION

Experimental studies of the chemistry of energetic atoms have almost exclusively involved the identification of the stable products formed by reaction with molecules of the radioactive recoils from spontaneous nuclear transformation or nuclear reaction. Mechanisms of these reactions have been inferred from the product distributions as affected by scavengers, molecular structure and state [1]. However, the information required for an adequate understanding of the mechanisms and kinetics of these often unique chemical interactions includes:

(a) The properties (charge, state of excitation, kinetic energy and molecular composition) of the recoil species at the instant of formation in the nuclear process;

(b) The modes of translational-and excitation-energy loss and charge change during the slowing down of the energetic recoils to the range of chemical reaction energies (<~20 eV);

(c) The identity of the transient intermediate species (ionic, free radical or molecular) taking part in the possible consecutive chemical reactions leading to the observed products; and

(d) The ways (e.g. dissociative charge neutralization, charge exchange, ion recombination and collisional de-excitation) by which the molecular species are stabilized as the observed products.

Much is known about the primary physical and chemical characteristics of the nuclidic entities produced by radioactive decay, and some results have been obtained on the initial products of nuclear reactions [2]. Consequently, the next steps in this approach in hot atom chemistry should be the study of the mechanisms of energy loss during the slowing-down phase and the identification of transient intermediates formed by reactions of the hot species with molecules of the medium. For these types of investigations direct observation of the interactions of the recoils with isolated molecules is desirable and often required. This paper reports on a start that has been made on these lines. Two different mass spectrometric techniques have been employed to study the transient ionic intermediates formed by interactions of recoil species with atoms and molecules. One method has given direct evidence for modes of kinetic energy loss by high-energy tritium recoils during part of their slowing down to the range of chemical reaction energies. The other has provided experimental proof for a proposed mechanism of the sequence of chemical reactions beginning with methane and the $(THe^3)^+$ daughter from beta decay of molecular tritium.

Ionizing collisions of 0.8 to 3.75 MeV protons (as stand-ins for tritons) with the isolated noble gas atoms He, Ne, Ar and Kr, and with the hydrocarbon molecules methane, acetylene, ethylene, ethane, propane and n-butane, were studied in a portable mass spectrometer that had special constructional features. The slow positive fragments formed in these collisions were analysed as to chemical nature, charge state and abundance. Partial cross sections for formation of several of the charge states of each of the noble gases were determined and compared with corresponding probabilities obtained by FEDORENKO et al. [3] for impact by 5 to 180 keV protons. The patterns of positively charged fragments resulting from bombardment by 2.25 MeV protons with the hydrocarbons were compared with the spectra observed with 1225 eV electrons. At these energies the two projectiles possess the same velocity. This comparison provided an assessment of the behaviour of recoil tritons as ionizing agents.

The second experimental approach used the techniques of "high pressure" mass spectrometry to establish the existence of the long-lived ($> 10^{-5}$ s) polymeric intermediate involved in the decay-induced isotopic exchange of tritium with methane [4]. The mechanism of tagging organic molecules with tritium by the widely used and simple technique of mixing T_2 and the compound, and allowing the two to stand for a time ("Wilzbach labelling" [5-7]), has been investigated for several simple compounds by GANT and YANG [8]. Methane in particular has been studied by PRATT and WOLFGANG [9]. Kinetic and scavenger procedures have led both groups of workers to conclude that there are two modes of labelling organic molecules with tritium: decay-induced labelling, which is initiated by reaction of the $(THe^3)^+$ daughter ion from radioactive transformation of T_2; and radiation-induced labelling, which is provoked by the excitation and ionization of the target molecule and T_2 by the betas emitted in the nuclear decay and by secondary electrons. The mechanisms proposed for the two paths of isotopic tagging involve the reactions of positively-charged transient species.

Search for the ions suggested as intermediates in the decay-induced mechanism was made by introducing mixtures of T_2 and CH_4 with and without added D_2 into the source chamber of a "high-pressure" mass spectrometer [10] operated at a source pressure sufficiently high ($\sim$0.1 mm) so that consecutive ion-molecule reactions can occur. At this density of gas a primary ion may be expected to undergo several collisions between formation by nuclear decay and departure from the source chamber. As an illustration, in a gas at 0.1 mm an ion traversing a length of 3.2 mm between point of origin and exit slit may collide with three molecules on the average. These experiments, then, serve to test the ionic mechanism proposed by Pratt and Wolfgang for the decay-induced isotopic exchange of tritium with methane. Investigation of the suggested radiation-induced mechanisms of tritiating CH_4 has also been made [4].

2. EXPERIMENTAL

The object of this research necessitated the use of two completely different techniques of mass spectrometry. For the study of the behaviour of high energy recoils, a portable mass spectrometer was constructed on a frame provided with castors and jacks so that it could be readily moved into position and aligned with the beam of high energy protons from the 4.5-MeV Van de Graaff electrostatic generator of this laboratory. For the investigation of the chemical reactions following beta decay of molecular tritium, a mass spectrometer characterized by very large pumps, a vacuum-tight source chamber (except for the necessary tiny apertures) and a scintillation ion detector was designed. The spectrometer could be operated with pressures in the source as high as several tenths of a mm, and very low signals could be detected.

The design of the spectrometer source section used in most of the high-energy projectile experiments is shown in Fig. 1. The energy-analysed and collimated beam entered the source chamber through a knife-edge slit 2.0 mm high and 4.0 mm wide, and passed through the target gas on a path midway between the repeller plate and exit slit before being monitored in a deep air-cooled Faraday cup. It was stopped by a tantalum-coated iron plug that could be manoeuvered by an external magnet so as to permit its observation through an end window. The position and profile of the beam could be judged from the fluorescence emitted when it struck a quartz plug adjacent to the window. A negative voltage on an adjacent electrode containing a slit 2.4 mm high and 3.2 mm wide served to prevent secondary electrons produced by the protons at the edges of the entrance slit from entering the chamber. The Faraday cup was also maintained at a positive potential to suppress secondary electrons.

Slow positive ions formed in the ionizing collisions were repelled from the source chamber through a 6.3×18.8 mm slot covered by a grid of 60% open area. Two more electrodes containing slots of similar dimensions, but covered with grid material with 98% of its area unobstructed, accelerated and focused the ion beam. The ions then passed through the 0.5×15.9-mm object slit of the spectrometer before being analysed by a 90°-sector single directional focusing magnetic field of 9-in radius. They then entered the detector chamber through a 1.0×15.9-mm image slit, and their intensities were measured by a plate surrounded by guard rings and an electrode to suppress secondary electrons. A vibrating-reed electrometer whose background was $\sim 5 \times 10^{-14}$ Å measured the current to the detector. The spectrometer could resolve fragments differing by one mass unit at a mass of 115. The pressure of the target gas in the source was approx. 10^{-4} mm. Generally, less than 10^{-6} mm was observed for the pressures in the other compartments of the spectrometer.

A high-energy electron gun designed by MONAHAN and STANTON [11] was attached to the source chamber for the studies of the fragmentation of polyatomic molecules by bombardment with electrons of the same velocity as that of the protons. The conditions of this part of the experiment (direction and profile of the electron beam, the source pressure and the electric fields for extracting, focusing and accelerating the ions) were held as nearly the same as possible as those used in corresponding measurements with the proton beam. To minimize the deflection of the ionizing beams by the fringe field, the magnet was enclosed by a box of soft iron, and several layers of soft iron and aluminium were placed alternately between the magnet and the source of the spectrometer. A more complete description of the experiment has been published [12].

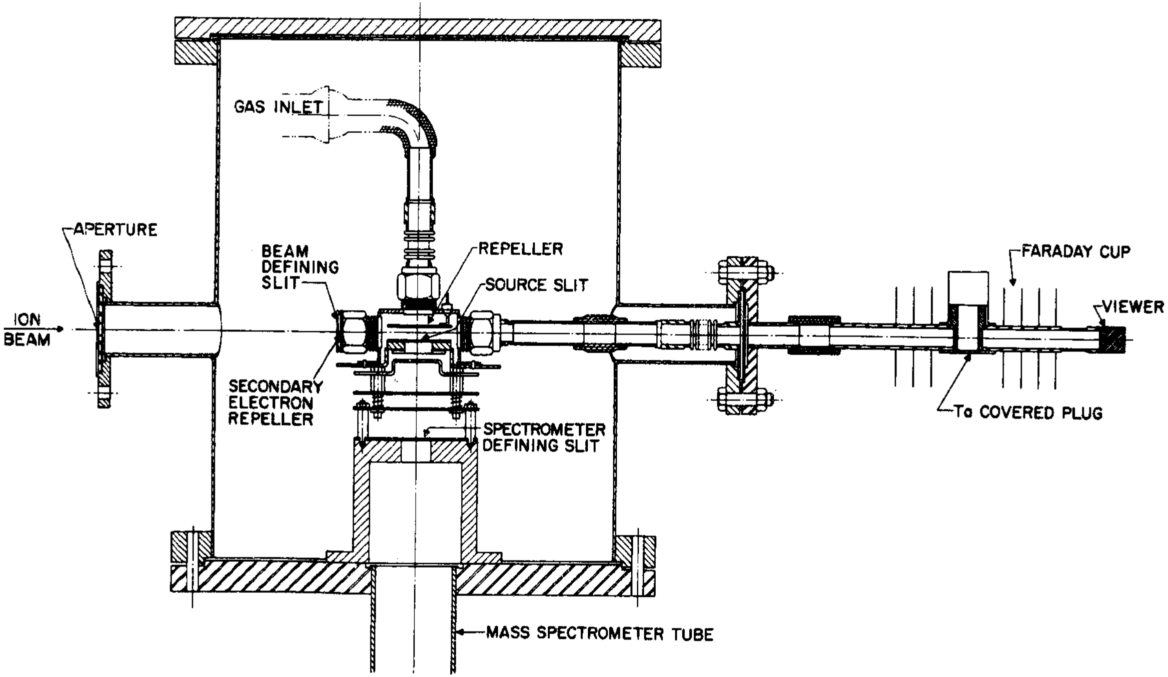

Fig. 1

Source section of portable mass spectrometer for investigating ionizing collisions of high-energy protons with atoms and molecules

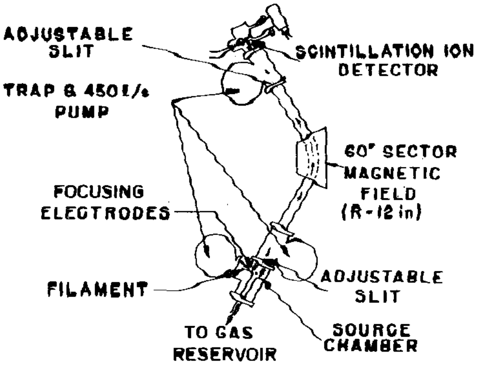

Fig. 2

"High pressure" mass spectrometer

A sketch of the "high-pressure" mass spectrometer appears in Fig. 2. The magnetic deflection is conventional; a 60° sector of 12-in radius of curvature with single directional focusing analysed the positive species. But the spectrometer has several distinguishing features which made the studies described here possible. Three 450-l/s mercury-diffusion pumps, each exhausted through its own fore-pump, were connected to the spectrometer through short tubes of 5-in diam. The great pumping speed effected sharp decreases in pressure between the source chamber and other compartments of the instrument. Thus, when the pressure of a gas mixture in the source was 0.15 mm, that in the chamber surrounding the source was 3×10^{-6} mm, while the pressure in the tube was 5×10^{-7} mm. A hemi-cylindrical repeller electrode established a field of 10 to 16 V/cm to impel positive ions produced in the chamber towards an exit slit 0.025 mm high and 2.0 mm wide. The only other aperture to the mass spectrometer in the otherwise vacuum-tight chamber was a hole, 0.025 mm in diam., which, for testing purposes only, served to introduce an electron beam into the box. The low intensities (5 to 800 counts/min above background were measured) of the ionic species found were determined by pulse counting with a scintillation ion detector [13] extensively modified from the design of DALY [14].

In the experiments designed to search for transient ionic intermediates of the decay-induced exchange of tritium with methane, mixtures of T_2, D_2 and CH_4 of known composition entered the source chamber through large-diam. tubes from an 11.4-l reservoir. Pressures in the source (of about 0.1 mm) were calculated from the pressures in the reservoir and the rate of flow in the inlet system. Ionic species of positive charge were looked for by magnetic scanning. Only those ions resulting initially from the beta decay and from the accompanying internal radiation field in the source were observed. The electron gun was, of course, not used.

TABLE I

FRACTIONAL INTENSITIES OF NOBLE GAS IONS PRODUCED BY IMPACT OF 2.0 MeV PROTONS

Gas	Charge state				
	+1	+2	+3	+4	+5
He	0.997	0.0025			
Ne	0.97	0.033	0.0015		
Ar	0.94	0.047	0.010	0.0021	0.00032
Kr	0.82	0.096	0.069	0.0155	0.0040

3. RESULTS

The slow positive ions formed in collisions of high-energy protons with noble gas atoms reveal themselves in the mass spectrometer in a spectrum of charge states. Table I presents the charge spectra (expressed as fractional intensities) produced by impact of 2.0 MeV protons on He, Ne, Ar and Kr, respectively. The error in the data is estimated to vary between 5 and 15%, increasing with the charge state of the species. The relative abundances of the different states of ionization of each target gas are seen to decrease rapidly with charge state. They also vary with the kinetic energy of the ionizing projectile in the range from 0.8 to 3.75 MeV. Although the fractional intensity of the +1 ion changed only slightly, the yield of a multiple-charged species could decrease progressively as much as a factor of 2.5.

The present data on the magnitude of the charge n of the struck atom and its fractional current $i_n/\Sigma i_n$ were used in combination with the values of HOOPER et al. [15] for the total ionization cross section to compute the partial cross section σ_n for the production of ions of this charge [16]. Typical results for proton energies from 0.8 to 3.75 MeV, as illustrated for the ionic states of neon, appear in Fig. 3. Included in the figure are curves representing the data of FEDERENKO et al. [3] for the range of proton energies extending from 5 to 180 keV. Although a large gap still remains to be explored between the two sets of data, the results are seen to be reasonably concordant. The magnitude of σ_n for singly-charged neon ions decreases from a high of 3×10^{-16} cm^2 molecule^{-1} for proton energies in the tens of kilovolts to an order of magnitude lower when the energy is a few MeV. The probabilities of forming higher states decrease rapidly with charge, the values for Ne^{+3}, for example, being 10^{-19}–10^{-20} cm^2 molecule^{-1} at a proton energy in the MeV region. Note that there are appreciable cross sections for production of positively charged species over an extensive range of proton energies. Similar behaviours of partial cross sections were observed for the other noble gases [16].

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