

K_{α} X-ray Spectra of Sodium Compounds Excited by High Energy, Heavy
Charged Particles

by

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Submitted in Partial Fulfillment of the Requirement of the
University Undergraduate Fellows Program

1982-193

Approved by:



R.L. Watson

April 1983

ABSTRACT

K α X-ray Spectra of Sodium Compounds Excited by High Energy
Heavy Charged Particles. (April 1983)

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The K α x-ray spectra of sodium compounds produced by bombardment with 5.2 MeV He⁺ and 20 MeV C³⁺ were measured using a curved crystal spectrometer. The average probability of electron transfer to the L shell of sodium was found to be 0.20 ± 0.01 for carbon ion excitation and ranged from 0.27 to 0.45 for helium excitation. X-rays emitted by the radiative Auger process were measured and a previously unreported line at 951 eV, corresponding to a KL₁L_{2,3} Auger transition, was seen.

Acknowledgements

I wish to thank Dr. R.L. Watson, my advisor, for his guidance and assistance throughout this project. I also gratefully acknowledge Dr. G. Pedrazzini, Dr. K. Parthasaradhi, R. Maurer, B. Bandong, and T. Ritter for their assistance in the experiments.

TABLE OF CONTENTS

SECTION	Page
I INTRODUCTION	1
II EXPERIMENTAL PROCEDURE	5
III EXPERIMENTAL RESULTS	8
IV DISCUSSION	18
A $K\alpha$ Satellite Intensities	18
B Radiative Auger Emission	26
V CONCLUSION	30
REFERENCES	32
VITA	33

LIST OF TABLES

Page	
7	Table I. Absorption Coefficients for Sodium Compounds (cm^2/g).
7	Table II. Fluorescence Yields for Neon.
8	Table III. $K\alpha$ Satellite Intensities for Sodium Excited by 20 Mev Carbon Ions.
8	Table IV. $K\alpha$ Satellite Intensities for Sodium Excited by 5.2 Mev Helium Ions.
9	Table V. q Values for Sodium Compounds.
18	Table VI. $K\alpha_4/K\alpha_3$ for Sodium Compounds Excited By 5.2 MeV He and 20 MeV C.
24	Table VII. Experimental p'_L and p_f Values for Na Compounds Excited by 5.2 MeV He and 20 MeV C.
30	Table VIII. Theoretical and Experimental Auger Energies.

LIST OF FIGURES

Page	
2	Figure 1. Energy Level Diagram for X-ray Production.
4	Figure 2. Spectra of Neon Excited by Different Projectiles.
6	Figure 3. Diagram of the Spectrometer.
10-13	Figure 4. Spectra of Sodium Compounds Excited by 5.2 MeV Helium Ions.
14-17	Figure 5. Spectra of Sodium Compounds Excited by 20 MeV Carbon Ions.
20	Figure 6. Spectra of Fluorine Compounds Excited by 5.5 MeV Helium Ions.
22	Figure 7. Comparison of Spectra of Neon and Fluorine Excited by Carbon Ions.
25	Figure 8. Comparison of Experimental Intensities with the Binomial Distribution for Sodium Fluoride Excited by 20 MeV Carbon Ions.
27	Figure 9. The Auger and Radiative Auger Processes.
29	Figure 10. Spectrum of the RAE X-rays of Sodium Fluoride Excited by 5.2 MeV Helium Ions.

I. Introduction

X-ray spectroscopy has been recognized for many years as a powerful tool for determining the composition and structure of matter. It has been applied to measurements of the structure of crystals and especially to studies of the electronic structure of atoms and molecules. Since the advent of accelerators capable of producing high energy beams of heavy charged particles, considerable effort has been directed toward the study of ion-atom collisions using x-ray spectroscopy. It was soon found that the chemical and solid-state environment could have large effects on the x-rays produced after inner-shell ionization by heavy ions. It was also found that several other processes compete with x-ray production.

X-rays are produced by the radiative decay of ions having a vacancy in an inner shell. An electron from an outer shell, with high energy, fills the vacancy in a low energy inner shell, and the difference in energy is carried off by the x-ray. Of particular importance to this research is the $K\alpha$ x-ray which is emitted when a 2p electron in the L shell fills a 1s vacancy in the K shell. The energy of a $K\alpha$ x-ray is equal to the difference in energy between the 1s and the 2p subshells of the atom. This energy difference can be changed by creating additional vacancies in the K or L shells. For example, if there is a vacancy in the L shell in addition to the vacancy in the K shell, the difference in energy between the 2p and 1s subshells increases, so the x-ray energy is greater than when the initial L vacancy is absent (see Fig. 1). As a result, a series of $K\alpha$ x-ray lines is possible; each line corresponding to a particular number of initial L vacancies. These lines, called $K\alpha$

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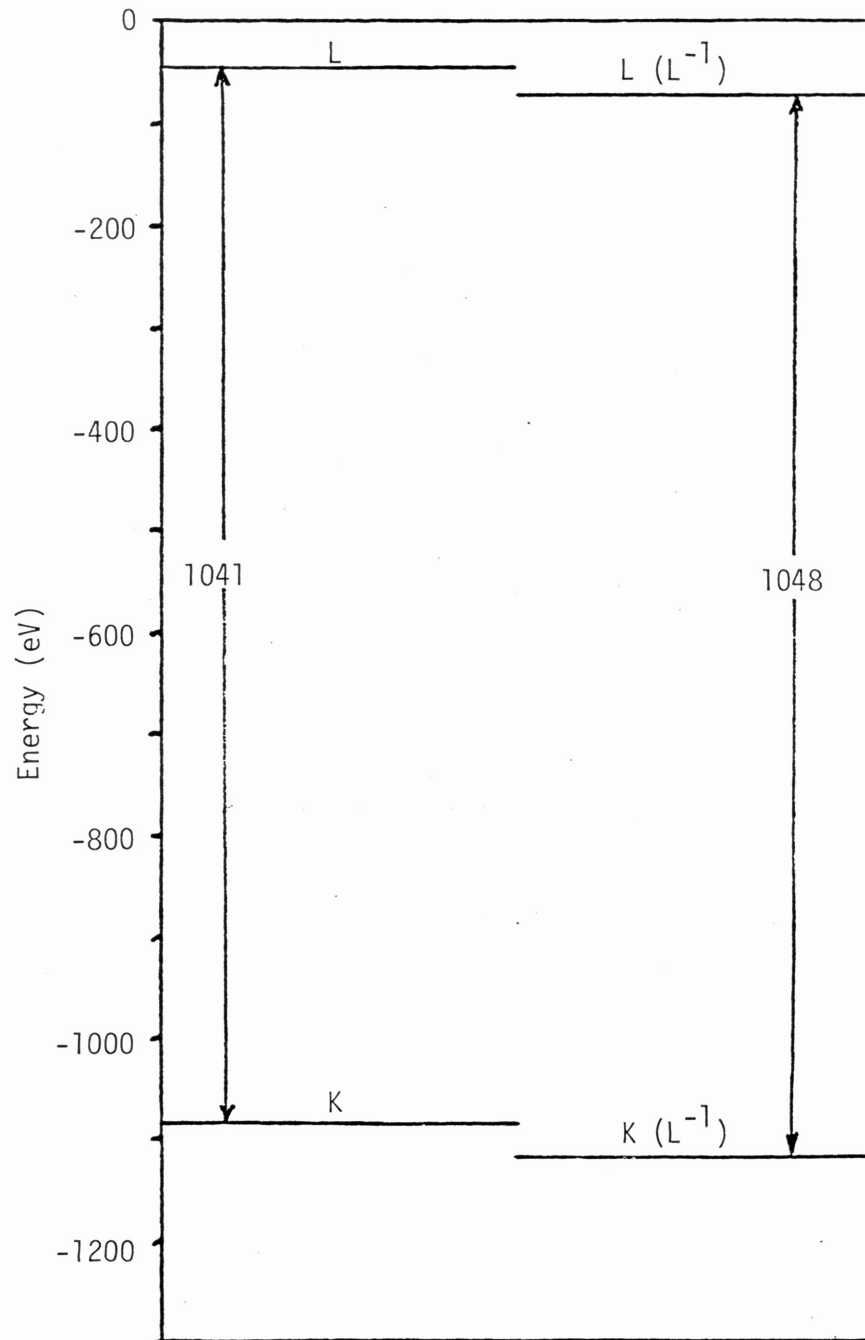


Figure 1. Energy Level Diagram For X-ray Production. The first pair of levels correspond to a KL^0 transition, and the second pair to a KL^1 transition.

satellites, are denoted by their initial state electron configurations, KL^n , where K indicates there is one K vacancy and L^n indicates n L vacancies. In this notation, n can have values of 0 to 7 for the eight possible configurations leading to a 2p to 1s transition.

The intensity of a particular KL^n satellite line is proportional to the number of target atoms having the KL^n initial configuration, and the number of atoms having that configuration is related to the probability of producing n vacancies in the L shell in a collision which also ionizes the K shell. Therefore, if the intensity distribution of the K_α satellites is known, the distribution of L vacancies and the probability of producing an L vacancy can be deduced.

Two factors which affect the probability of producing an L vacancy in an ion-atom collision are the charge and the velocity of the projectile ion.¹ The probability of an L electron being ionized increases as the ion charge increases and as the ion velocity decreases. In Fig. 2, the K_α satellites of neon produced by different projectiles are shown.¹ The intensities of the satellites associated with higher numbers of L vacancies (KL^5 , KL^6 , and KL^7) clearly increase from helium ions with a charge of 2 to argon with an average charge of about 13.

Recent work by Benka et. al.^{2,3} has shown that the chemical environment greatly influences the intensities of fluorine satellites. Deconninck and Van den Broek⁴ observed small variations in some sodium compounds, but only a few compounds were investigated and only one mode of excitation was used. The object of the present work was to examine some of these chemical and solid state effects in more detail, as well as to investigate alternate decay modes, in sodium atoms following inner-

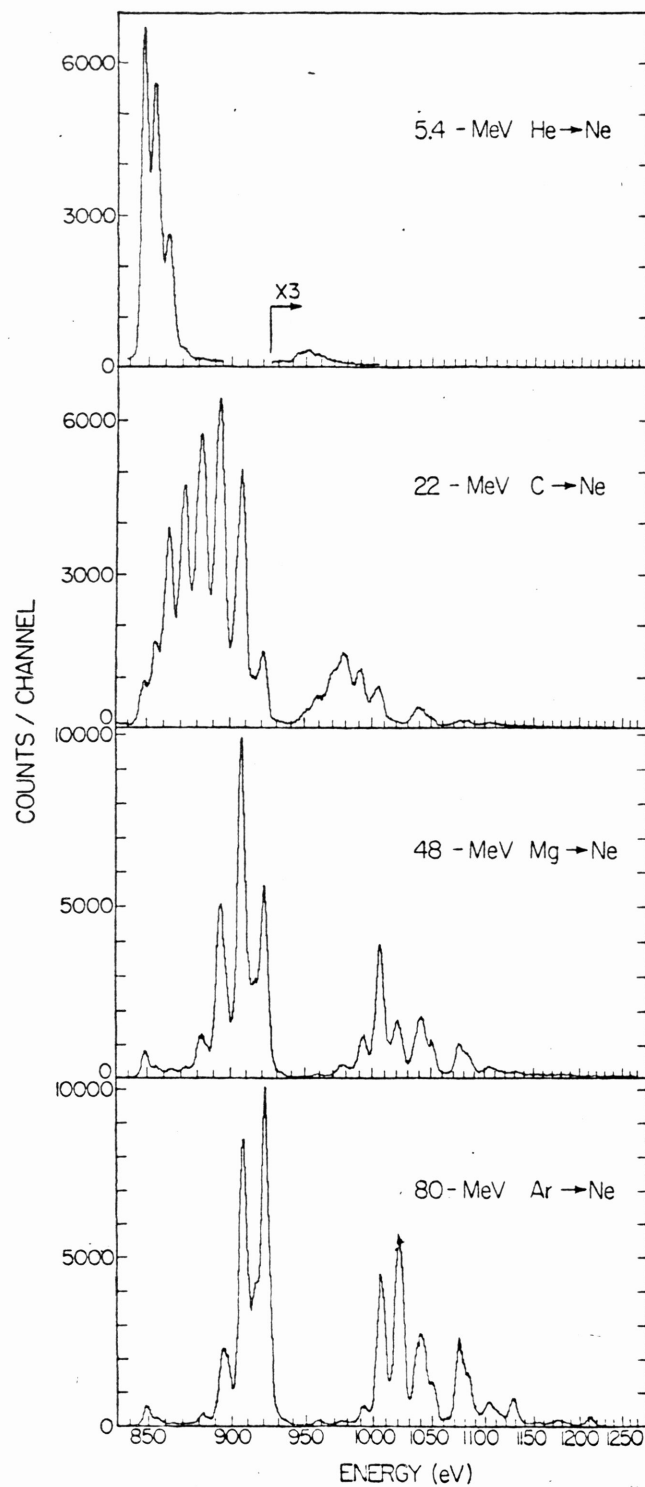


Figure 2. Spectra of Neon Excited by Different Projectiles. The $K\alpha$ satellites are those lines below 950 eV. The lowest energy line is the KL. (from ref. 1)

shell ionization by high energy, heavy ions. In pursuit of this objective, a series of sodium compounds excited by bombardment with two different projectile ions have been studied. Comparisons of the spectra of different sodium compounds yielded information regarding chemical effects, while a comparison of sodium spectra with spectra of neon demonstrated the effect of the solid-state environment.

II. Experimental Procedure.

Beams of 5.4 MeV He^+ and 22 MeV C^{3+} beams were extracted from the Texas A&M Variable Energy Cyclotron. The beams were focused by means of two quadrupole magnets and directed through a 3 mm diameter collimator placed 1 cm in front of the target position. X-rays emitted from the target were analyzed with a 12.7 cm Johansson-type curved crystal spectrometer using a thallium acid phthalate (TAP) crystal (see Fig. 3). The spectrometer was operated to obtain second order diffraction from the crystal, and gave an energy resolution of 1.0 eV at 1040 eV. The diffracted x-rays were detected by a flow proportional counter with a $65\mu\text{g}/\text{cm}^2$ thick polypropylene window and which contained a 90% methane-10% argon gas mixture. The integrated current from a thin ($\sim 10\mu\text{g}/\text{cm}^2$) carbon foil located between the collimator and the target was used to determine the counting time at each Bragg angle. A DEC VAX 11/780 computer with a CAMAC interface was used for data acquisition, storage, and analysis.

The spectra were analyzed by fitting Voigt functions to the peaks using a non-linear least squares fitting program (FACELIFT). The program provided the peak centroids, widths, and areas. The areas were corrected for differences in absorption in the target by multiplying the

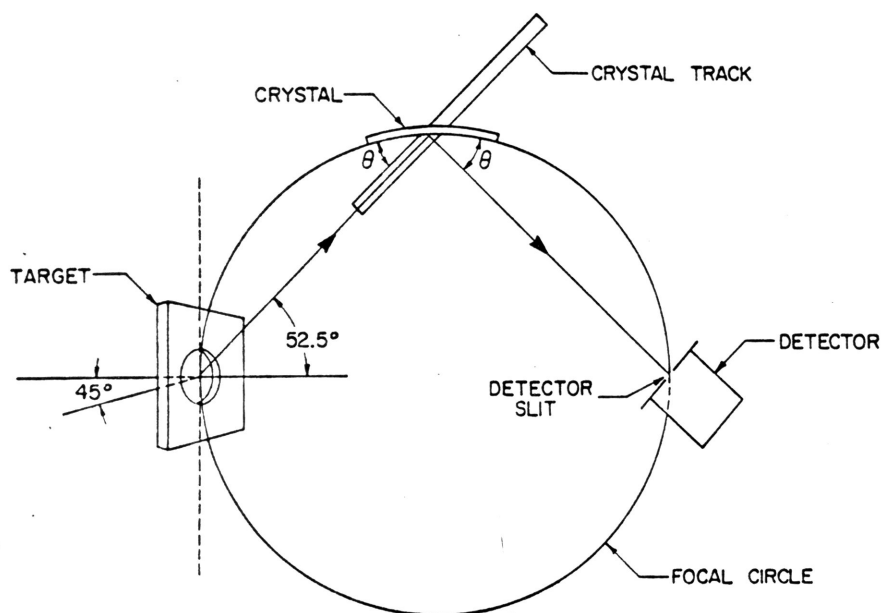


Figure 3. Diagram of the Spectrometer. This view is looking down the beam-line from the cyclotron.

uncorrected peak area by the absorption coefficients presented in Table I. An additional correction was made for differences in

Table I. Absorption Coefficients for Sodium Compounds (cm^2/g)^a.

Compound	KL ⁰	KL ¹	KL ²	KL ³
Na	498	488	478	466
NaF	2507	2454	2404	2344
NaCl	1747	1710	1676	1634
NaBr	2464	2412	2363	2304
NaI	7214	7066	6927	6758
NaOH	1163	1139	1116	1088
Na ₂ S	1706	1670	1636	1595

^aRef. 5.

fluorescence yield by dividing the areas by the fluorescence yields of neon shown in Table II. Since only relative intensities were considered,

Table II. Fluorescence Yields for Neon^a

Peak	KL ⁰	KL ¹	KL ²	KL ³
Fluorescence Yields	0.0159	0.0175	0.0199	0.0248

^aRef. 6.

no correction for dependence on atomic number was applied to the fluorescence yields. Also, no corrections were made for variations in crystal reflectivity or absorption in the proportional counter window, since the net correction over the energy range from KL⁰ to KL³ was only 3% which is negligible compared to the experimental errors.

The energy loss of the projectile beam in the target was calculated

from range tables given by the program STOPRANGE. The beam energies at the average depth of x-ray production were 5.2 MeV for He and 20 MeV for C.

III. Experimental results.

The experimental satellite intensities which for the sodium compounds studied are listed in Tables III and IV. These intensities have

Table III. $K\alpha$ Satellite Intensities for Sodium Excited By 20 MeV Carbon Ions.

Compound	KL ⁰	KL ¹	KL ²	KL ³
Na	0.093	0.209	0.431	0.282
NaF	0.094	0.224	0.393	0.290
NaCl	0.083	0.212	0.413	0.292
NaBr	0.092	0.204	0.411	0.293
NaI	0.074	0.215	0.421	0.289
NaOH	0.079	0.208	0.427	0.286
Na ₂ S	0.073	0.198	0.434	0.294

Table IV. $K\alpha$ Satellite Intensities for Sodium Excited By 5.2 MeV Helium Ions.

Compound	KL ⁰	KL ¹	KL ²	KL ³
Na	0.525	0.382	0.080	0.013
NaF	0.606	0.322	0.064	0.008
NaCl	0.598	0.329	0.063	0.010
NaBr	0.588	0.344	0.059	0.009
NaI	0.592	0.324	0.071	0.007
NaOH	0.555	0.353	0.086	0.006
Na ₂ S	0.495	0.390	0.101	0.015

been corrected for self-absorption and fluorescence yield and are given relative to the sum of the intensities of the first four satellite peaks. The spectra are presented in Figs. 4 and 5. As can be seen in the spectra, a discontinuity occurs around 1075 eV due to the K-absorption edge of sodium. X-rays above 1075 eV are highly absorbed in the target and therefore the peaks above KL^3 are greatly depressed. Due to uncertainties in the exact magnitude and position of this absorption edge, only the intensities of KL^0 through KL^3 were used in the analysis.

The intensity distribution of each spectrum was characterized by a parameter, q , which was obtained from the formula

$$q = \frac{1}{3} \sum_{n=0}^3 n y'(n) \quad (1)$$

where $y'(n)$ is the intensity of the KL^n satellite relative to the sum of the intensities of the first four peaks. Values of q for the sodium compounds excited by He^+ and C^{3+} projectiles are presented in Table V.

Table V. q Values for Sodium Compounds.

Compound	q with 5.2 MeV He	q with 20 MeV C
Na	0.073	0.240
NaF	0.059	0.235
NaCl	0.060	0.239
NaBr	0.061	0.238
NaI	0.061	0.241
NaOH	0.068	0.240
Na ₂ S	0.079	0.244

By virtue of the excellent resolution obtained using the second order diffraction from the TAP crystal, the KL^1 satellite was resolved into three components called $K\alpha'$, $K\alpha_3$, and $K\alpha_4$ in order of increasing

Figure 4. Spectra of Sodium Compounds Excited by 5.2 MeV Helium Ions.
a) Na and NaF.

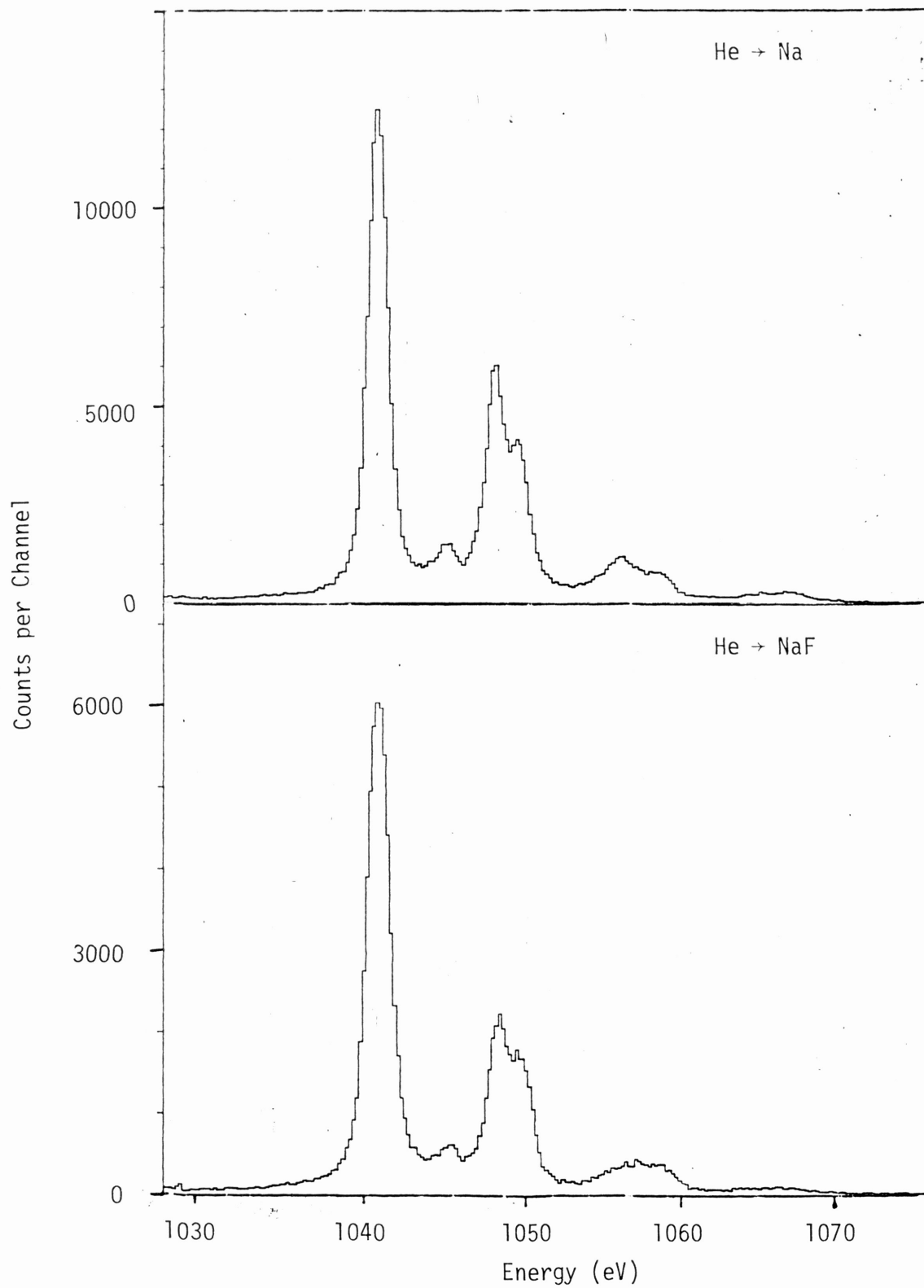


Figure 4. Spectra of Sodium Compounds Excited by 5.2 MeV Helium Ions.
b) NaCl and NaBr

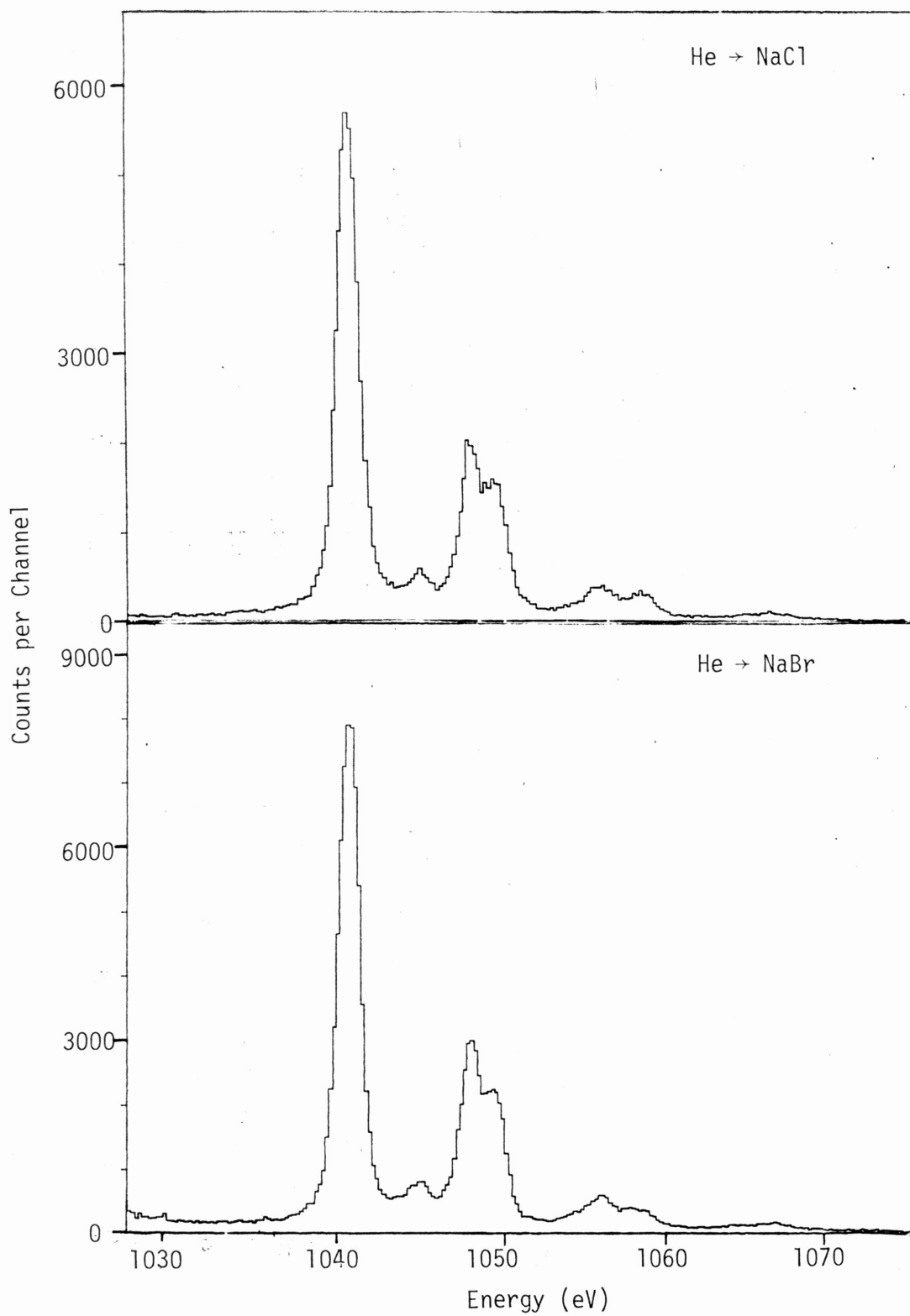


Figure 4. Spectra of Sodium Compounds Excited by 5.2 MeV Helium Ions.
c) NaI and NaOH

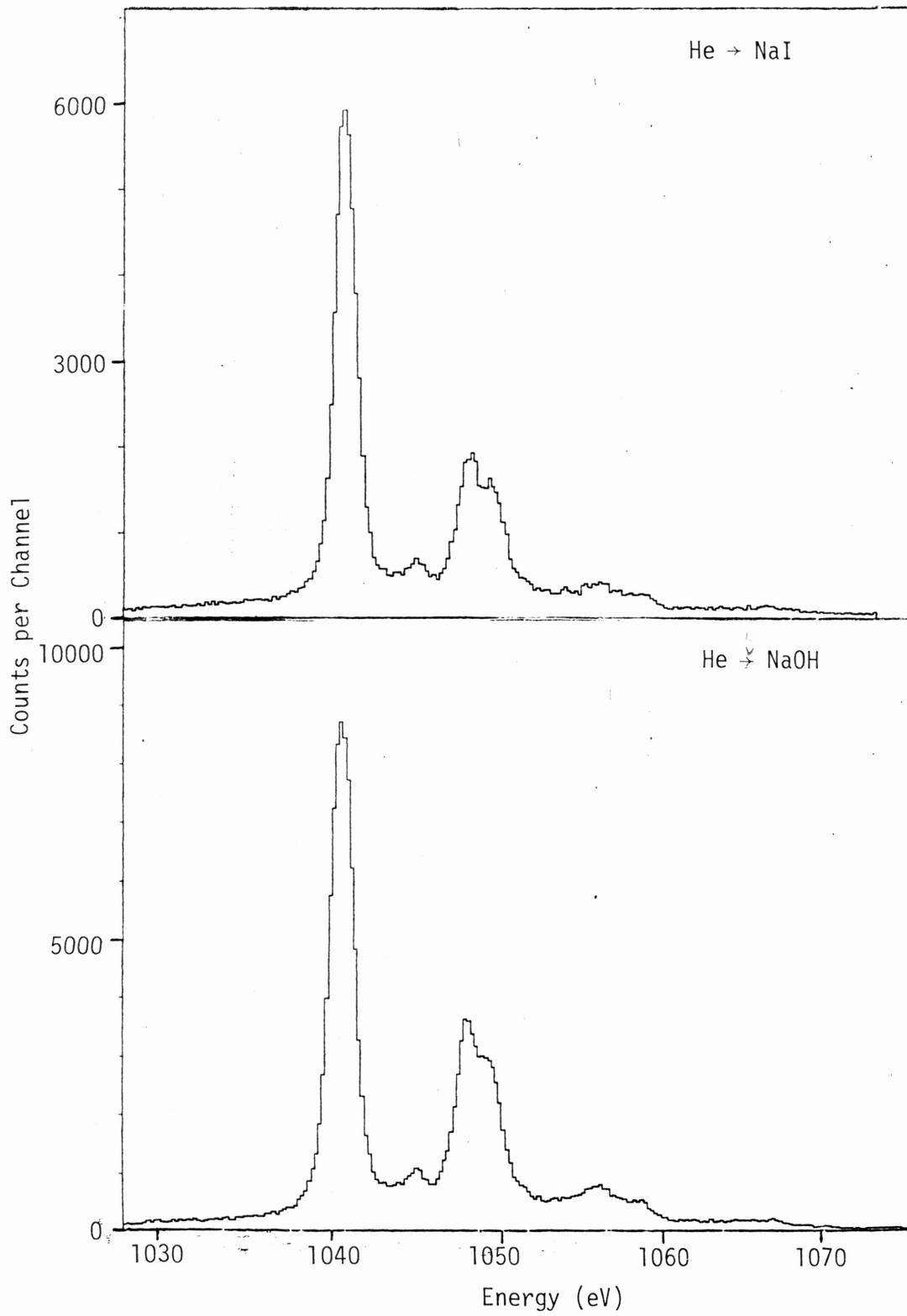


Figure 4. Spectra of Sodium Compounds Excited by 5.2 MeV Helium Ions.

d) Na_2S

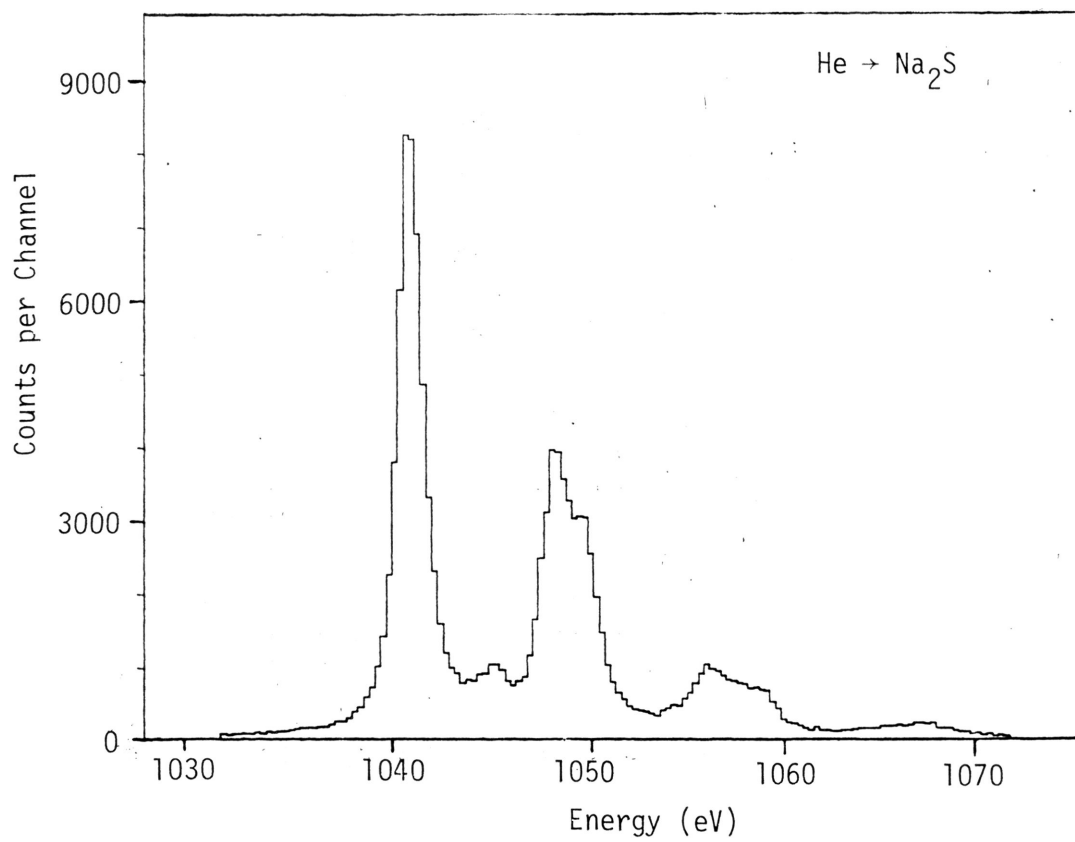


Figure 5. Spectra of Sodium Compounds Excited by 20 Mev Carbon Ions.

a) Na and NaF

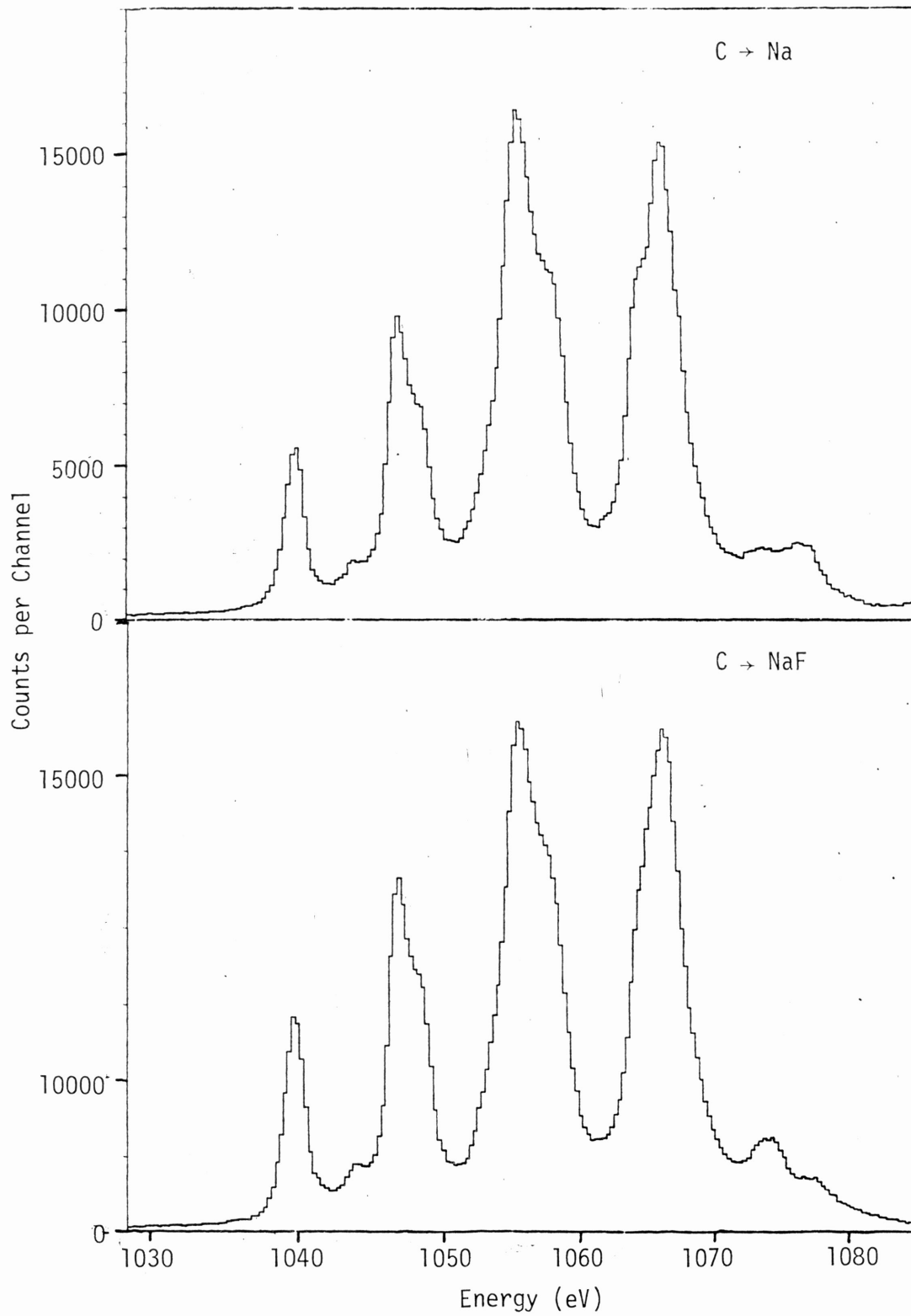


Figure 5. Spectra of Sodium Compounds Excited by 20 MeV Carbon Ions.

b) NaCl and NaBr

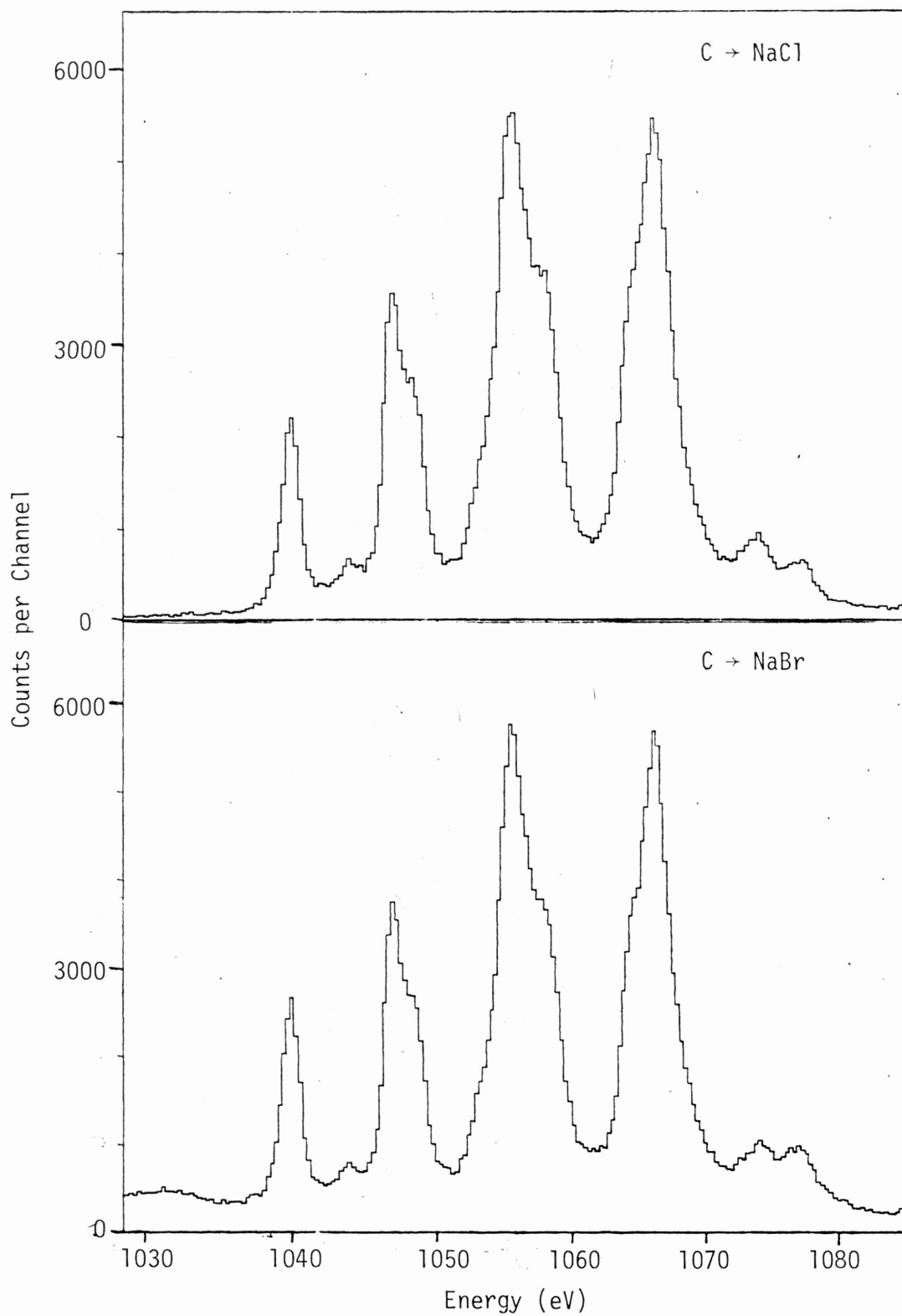


Figure 5. Spectra of Sodium Compounds Excited by 20 MeV Carbon Ions.

c) NaI and NaOH

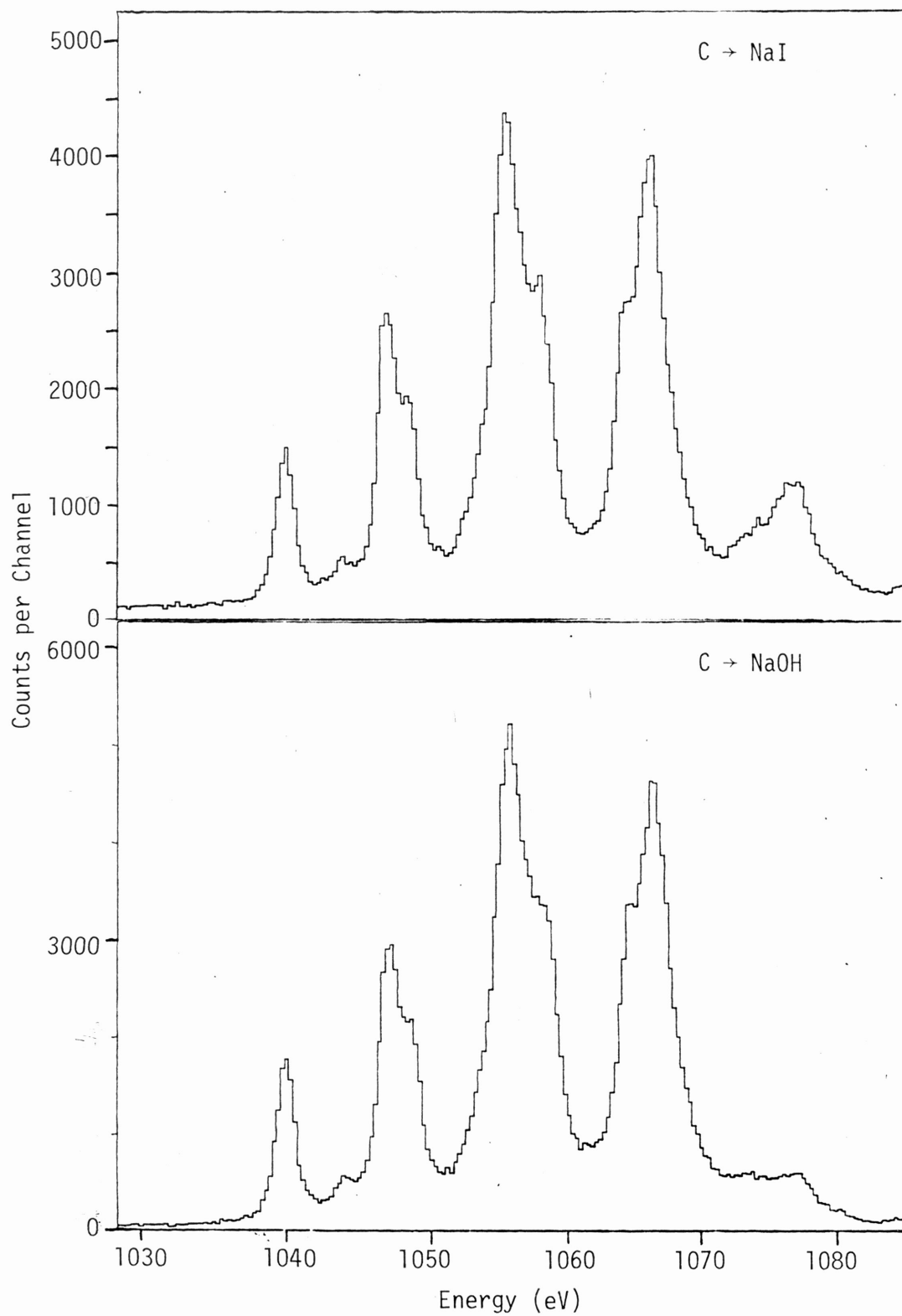
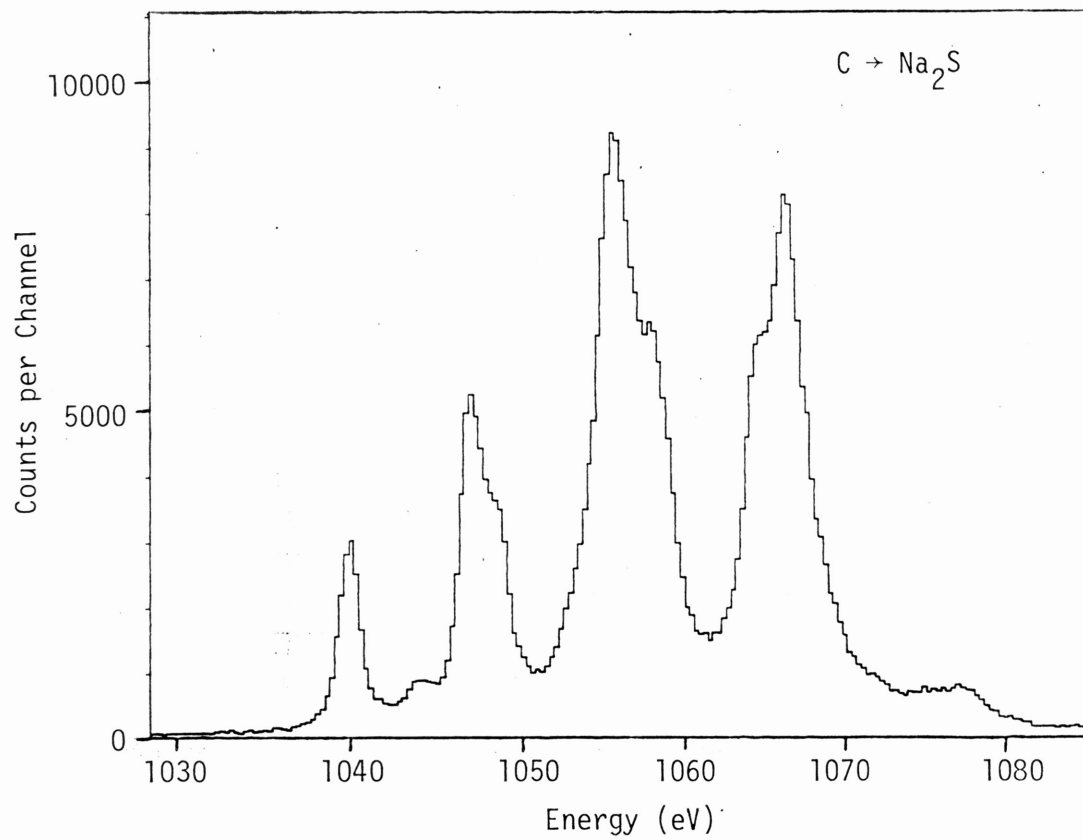


Figure 5. Spectra of Sodium Compounds Excited by 20 MeV Carbon Ions.

d) Na_2S



energy. The ratio of the intensity of $K\alpha_4$ to $K\alpha_3$ was computed for each compound, and the results are tabulated in Table VI. These intensities were corrected for self-absorption but not for fluorescence yield.

Table VI. $K\alpha_4/K\alpha_3$ for Sodium Compounds Exited By
5.2 MeV He and 20 MeV C.

Compound	$K\alpha_4/K\alpha_3$ with He	$K\alpha_4/K\alpha_3$ with C
Na	0.66	0.77
NaF	0.81	0.84
NaCl	0.81	0.86
NaBr	0.71	0.88
NaI	0.75	0.93
NaOH	0.74	0.95
Na ₂ S	0.58	0.94

IV. Discussion.

A. $K\alpha$ Satellite Intensities.

The intensity distribution of the $K\alpha$ satellites is related to the distribution of L vacancies in the atoms of the target. Certain processes, which depend on the chemical or solid-state environment, can fill vacancies in the L shell during the lifetime of the initial state (about 10^{-14} sec.).² If these processes fill L vacancies prior to the emission of an x-ray, the satellite intensity distribution will be skewed toward the lower energy satellites, which are associated with fewer L vacancies.

One process which fills L vacancies, resonant electron transfer (RET), was recently observed in compounds of fluorine.² The KL^1 satellite of KF and SrF_2 was found to be greatly suppressed, and KL^0 was correspondingly enhanced, compared with other fluorine compounds (see

Fig. 6). The explanation of these effects is that, for the KL^1 configuration of KF and SrF_2 , there is an energy of the fluorine 2p levels with the np level of the companion metal ions. This enables an electron from the metal ions to easily fill the single vacancy in the 2p level of the fluorine ion. In the present study, however, no evidence of RET was observed for any of the sodium compounds examined. An examination of the KL^1 intensities in Tables III and IV (p. 8) clearly shows that there are no compounds for which the KL^1 intensity is anomalously low, as it is in the case of fluorine. The reason that RET does not occur for sodium is probably associated with the fact that the binding energy of its 2p electrons is far below the top of the potential energy barrier separating a sodium ion from its neighboring anions. This makes it very improbable that an electron would be able to tunnel from the anion site.

An examination of the $K\alpha_4$ to $K\alpha_3$ ratios in Table VI (p. 18) shows that the ratio for Na metal is consistently lower than those of the compounds. Langenberg and Watson⁷ observed this difference between the metal and its compounds in magnesium, and proposed that it was due to the rearrangement of electrons in the L shell by Coster-Kronig transitions prior to x-ray emission.

A non-resonant filling process was also observed in fluorine when carbon ions were used to produce the initial vacancy distribution.³ The spectra of fluorine in different compounds were found to be strikingly different from that of neon. The fluorine ion is isoelectronic with neon and should, in the absence of filling processes, have approximately the same satellite intensity distribution. Therefore the differences, which

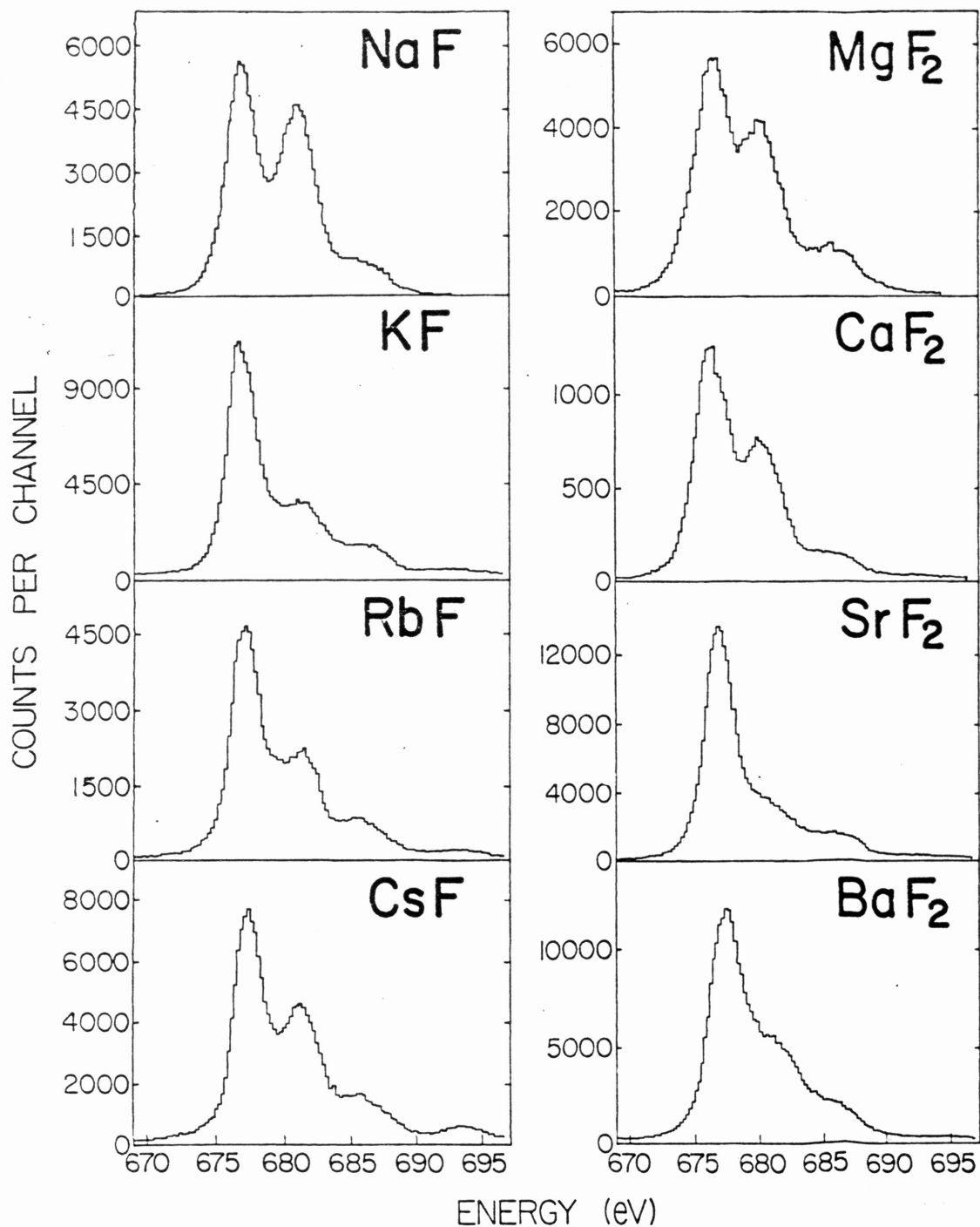


Figure 6. Spectra of Fluorine Compounds Excited by 5.5 MeV Helium Ions. The low intensity of the KL_1 peak in KF and SrF_2 . Is due to resonant electron transfer RET. (from ref. 2)

are clearly visible between the high energy satellites of neon and those of fluorine in Fig. 7, must be due to filling of L vacancies. Furthermore, the suppression of the high energy satellites in fluorine occurs for all compounds and hence must be due to a non-resonant filling process associated with the solid state.

The parameter used to characterize the distribution of L vacancies present at the time of x-ray emission is p'_L . The usual formula used to calculate p'_L is

$$p'_L = \frac{1}{8} \sum_{n=0}^7 n y(n) \quad (2)$$

where $y(n)$ is the relative intensity of KL^n (normalized to the sum of all the satellite intensities) corrected for variations in absorption and fluorescence yield. A comparison of p'_L values with the primary vacancy distribution p_L value provides a measure of the probability of electron transfer to the L shell prior to x-ray emission. Since filling processes have the effect of enhancing the intensity of the low energy satellites, the p'_L of a spectrum will be less than p_L . As stated above, sodium and fluorine ions are isoelectronic with neon, and hence it may be assumed that their initial vacancy distributions would be nearly identical. Furthermore, the neon $K\alpha$ satellite intensities should directly reflect the initial vacancy distribution, since electron transfer cannot occur for this monatomic gas atom. An experimental p'_L may therefore be assumed to represent the p_L of the initial L vacancy distribution. On the other hand, with fluorine and sodium in the solid state, there are many electrons available to fill L vacancies before x-ray emission.

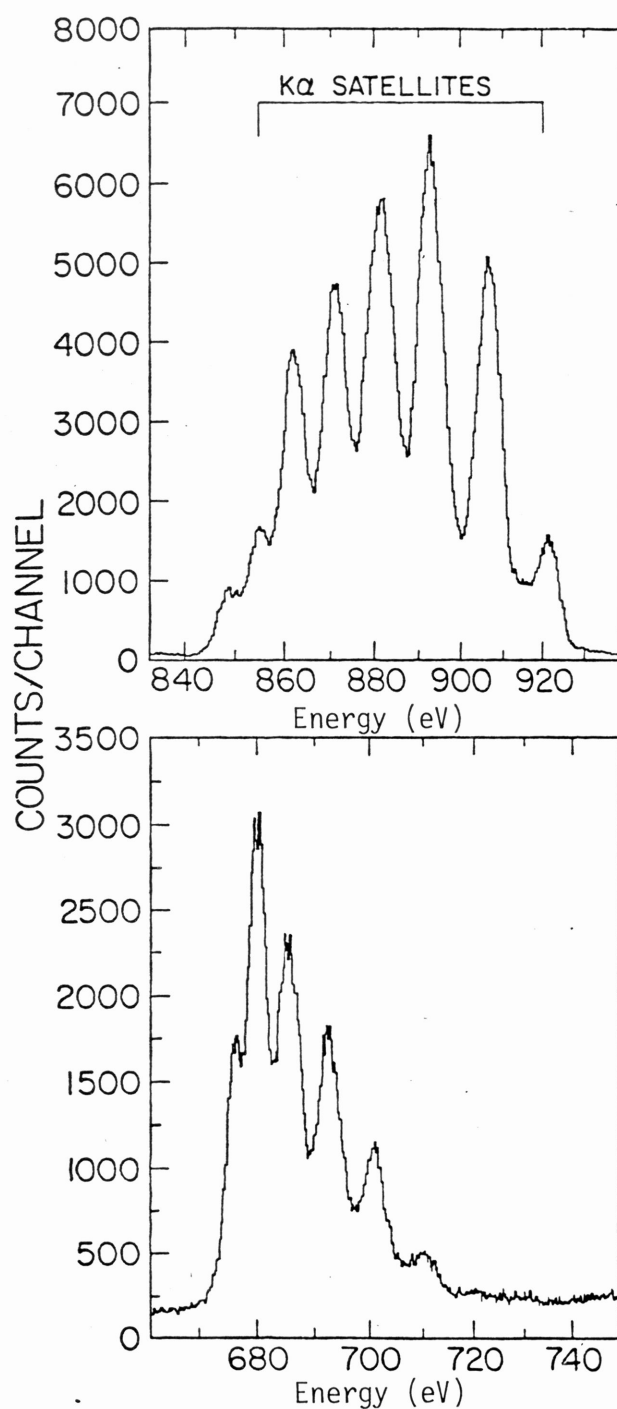


Figure 7. Comparison of Spectra of Neon and Fluorine Excited by Carbon Ions. The suppression of the high energy satellites in fluorine is due to general electron transfer to the L shell. (from ref. 17)

Consequently, the experimental p'_L value calculated from a spectrum of sodium or fluorine will differ from the p_L value obtained from the neon spectrum. These two quantities are related by the formula⁸

$$p'_L = p_L (1 - p_t) \quad (3)$$

where p_t is the average probability that an L vacancy, once created, will be filled before the emission of an x-ray.

For neon, the primary vacancy distribution is approximately given by the the binomial formula⁹

$$B(n, p_L) = \binom{8}{n} p_L^n (1 - p_L)^{8-n} \quad (4)$$

where

$$\binom{8}{n} = \frac{8!}{n! (8 - n)!} , \quad (5)$$

p_L is the average probability for creating a single L vacancy, and $B(n, p_L)$ is the relative yield of KL^n configurations produced in ion-atom collisions. As mentioned above, the intensities of the satellite lines will mirror the L vacancy distribution as given by equation 4 if no additional process creates or fills vacancies before the x-ray is produced. The usual calculation of p'_L by equation 2 requires a knowledge of the relative intensities of all eight peaks. Due to the interference of the sodium K-absorption edge, reliable relative intensities could not be calculated for the satellites above KL^3 , and so p'_L could not be calculated using equation 2. However by assuming that the x-ray intensity distribution is also binomial, p'_L was deduced by finding the binomial distribution which gave the same value of q as the experimental distribution. Such a distribution must satisfy the

requirement

$$\frac{1}{8} \sum_{n=0}^3 n B(n, p'_L) \text{ ----- } = q \quad (6)$$

$$\sum_{n=0}^3 B(n, p'_L)$$

where q is the parameter given by equation 1 and $B(n, p'_L)$ is the binomial yield as given by equation 4. The experimental p'_L values for the sodium compounds, obtained in this way, are presented in Table VII and

Table VII. Experimental p'_L and p_t Values for Na Compounds Excited by 5.2 MeV He and 20 MeV C.

Compound	He proj.		C proj.	
	p'_L	p_t	p'_L	p_t
Na	0.073	0.34	0.30	0.20
NaF	0.060	0.45	0.29	0.19
NaCl	0.061	0.45	0.30	0.20
NaBr	0.062	0.44	0.29	0.19
NaI	0.061	0.45	0.30	0.20
NaOH	0.068	0.38	0.30	0.20
Na ₂ S	0.080	0.27	0.30	0.20

comparisons of the experimental intensity distributions with the binomial distribution are presented in Fig. 8. The calculation of p_t from p'_L requires the p_L of the initial vacancy distribution. Ignoring the Z dependence of p_L (which is expected to vary by only 10% or so over the Z interval 10 to 11), the previous results for neon have been used for this purpose. The neon p_L values of 0.11 for 5.2 MeV He and 0.36 for 20 MeV C were obtained from reference 1. Using the neon p_L values, the sodium p_t

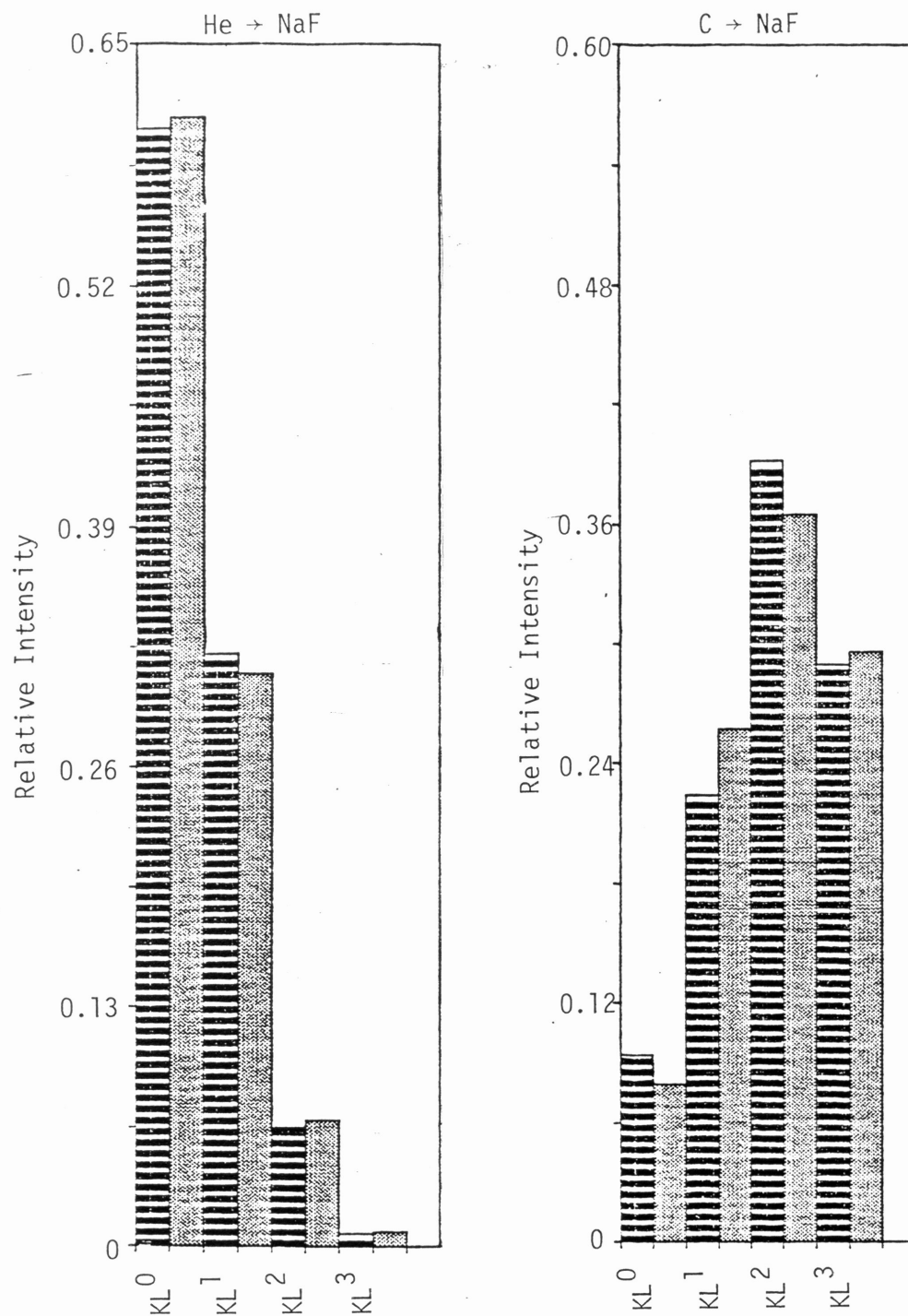


Figure 8. Comparison of Experimental Intensities with the Binomial Distribution for Sodium Fluoride Excited by 20 MeV Carbon Ions and 5.2 MeV Helium Ions. The striped bars are the experimental intensities. The gray bars are the intensities predicted by the binomial distribution.

values listed in Table VII were obtained. It should be noted that the p_t values for fluorine in NaF (from ref. 3) is 0.36 while the corresponding value for sodium is 0.19.

The difference of a factor of two in the p_t values of sodium and fluorine may be due to the difference in the lifetimes of the initial states. A state which has a long lifetime has a larger probability of being filled. The total radiative decay lifetime of a K vacancy in sodium is found to be 1.09×10^{-13} seconds, while the radiative decay lifetime of a fluorine K vacancy is 3.56×10^{-13} seconds.¹⁰ Clearly the longer lifetime of the fluorine initial state correlates well with its higher p_t .

B. Radiative Auger Emission.

In addition to x-ray emission, there are other processes by which an atom excited by a projectile ion can de-excite. Two of these processes are diagrammed in Fig. 9. In an Auger transition, an electron from the L shell fills the initial K vacancy, just as in x-ray emission, but instead of emitting an x-ray, a second L electron is ejected from the atom. These ejected electrons, called Auger electrons, have a kinetic energy equal to the difference in the energies of the L and K shells minus the energy required to remove the electron from the atom. Another decay process (which occurs only rarely) is radiative Auger emission (RAE) in which both an x-ray and an Auger electron are emitted.¹¹ The sum of the x-ray and the Auger electron kinetic energies is equal to the energy that an ordinary Auger electron would have. The maximum energy the x-ray may have in an RAE transition is therefore equal to the energy of the Auger electron in ordinary Auger decay. This corresponds to the

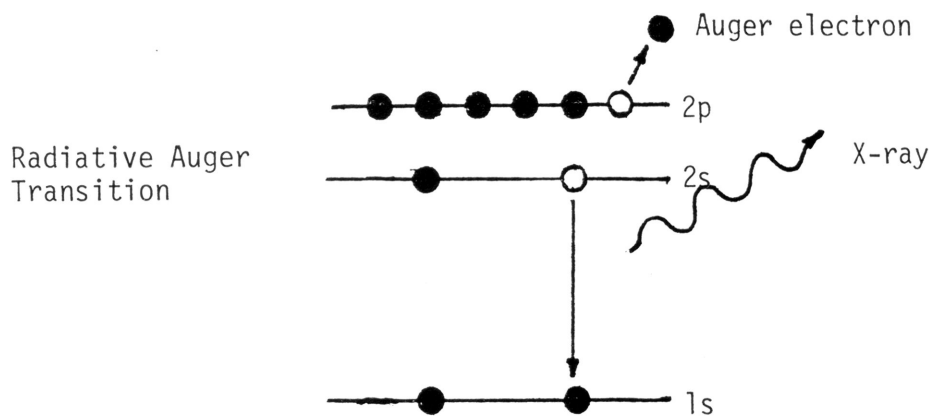
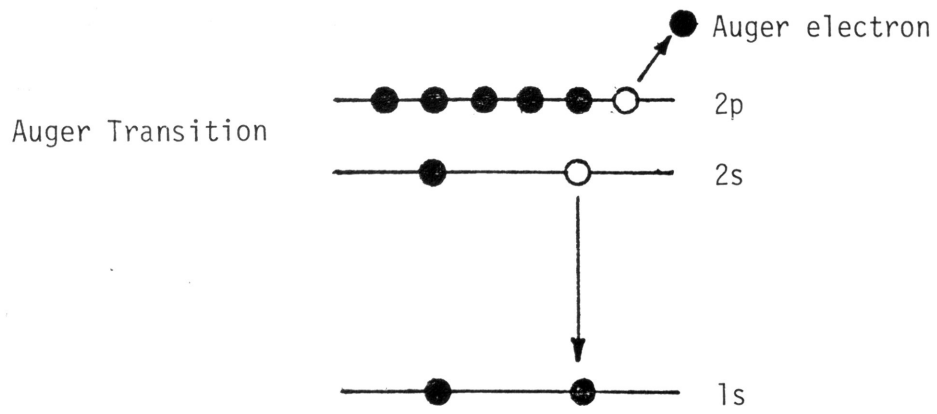


Figure 9. The Auger and Radiative Auger Processes. The closed dots represent electrons, and the open dots are vacancies.

RAE electron being ejected from the atom with zero kinetic energy. From another point of view, an RAE x-ray will have a maximum energy equal to the energy of the KL^0 transition minus the energy required to remove an additional L electron from the atom. Therefore, one should look for RAE x-rays below the KL^0 peak.

A spectrum of NaF excited by 5.2 MeV He^+ projectiles is shown in Fig. 10. The region scanned is just below the KL^0 peak and the spectrum clearly shows two broad peaks; one at 951 eV and another at 984 eV. These two peaks agree in energy with the kinetic energies of the $KL_1L_{2,3}$ and $KL_{2,3}L_{2,3}$ Auger transitions respectively.¹² The first transition, $KL_1L_{2,3}$, occurs when a 2s electron fills the K vacancy while a 2p electron and an x-ray are emitted. In the $KL_{2,3}L_{2,3}$ transition, a 2p electron fills the K vacancy and a second 2p electron and an x-ray are emitted. Although the $KL_{2,3}L_{2,3}$ RAE x-ray has been observed before¹³, the $KL_1L_{2,3}$ has not been previously reported. Moore et. al. used a potassium acid phthalate (KAP) crystal which has lower reflectivity than our TAP crystal. This probably accounts for their inability to see the low intensity $KL_1L_{2,3}$ x-ray.

The identification of these RAE x-rays with their respective Auger transitions was done by comparing the measured x-ray energy with experimental Na Auger energies. Attempts to calculate these energies theoretically using the Hartree-Fock method¹⁴ did not yield good agreement with sodium. Similar calculations were made for other elements as well and are tabulated in Table VIII.

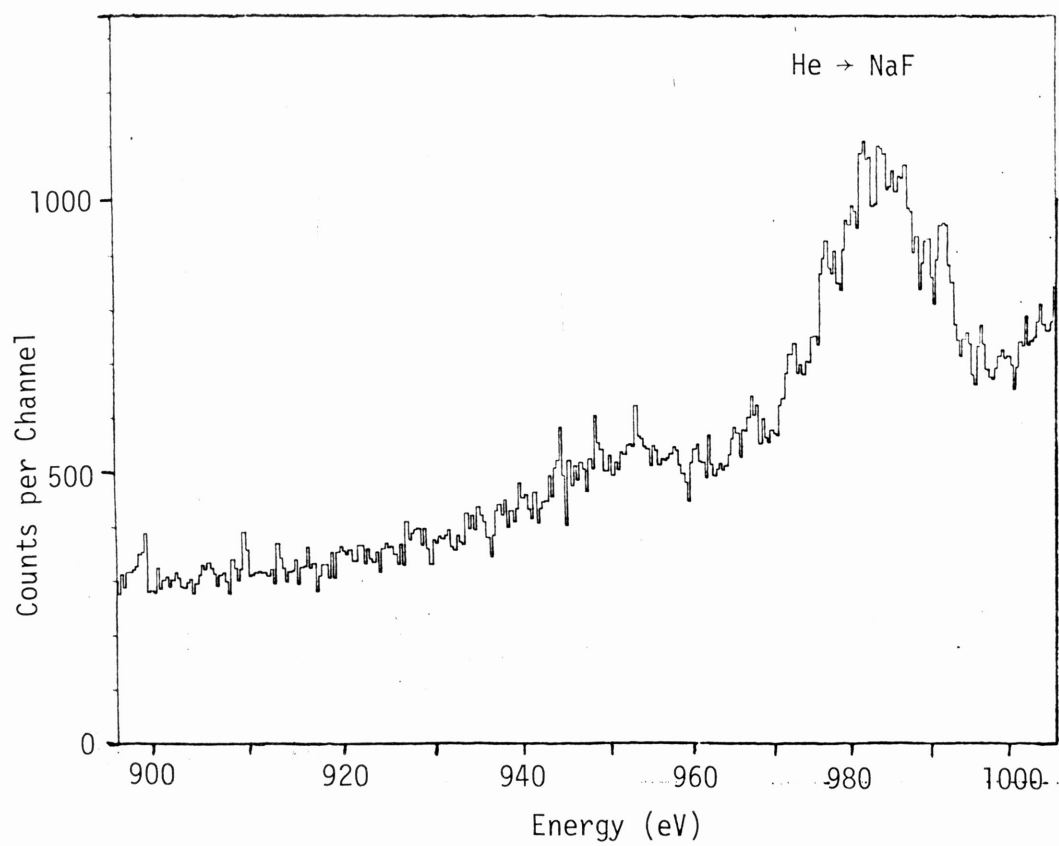


Figure 10. Spectrum of the RAE X-rays of Sodium Fluoride Excited by 5.2 MeV Helium Ions.

Table VIII. Theoretical and Experimental Auger Energies.

Element	Transition	Theory	Experiment	
F (NaF)	KL1L2,3	(1P)	629.23 ^d	623.7 ± 0.4 ^a
		(3P)	639.64	632.0 ± 0.4
	KL2,3L2,3	(1S)	654.35	646.7 ± 0.4
		(1D)	658.56	649.6 ± 0.4
Ne (gas)	KL1L2,3	(1P)	770.67	771.1 ± 0.1 ^b
		(3P)	782.89	781.9 ± 0.1
	KL2,3L2,3	(1S)	800.72	800.5 ± 0.1
		(1D)	805.83	804.15
Na (Na ₂ O)	KL1L2,3	(1P)	925.77	945.8 ± 0.4 ^c
		(3P)	939.73	958.4 ± 0.4
	KL2,3L2,3	(1S)	960.83	980.6 ± 0.4
		(1D)	966.85	994.8 ± 0.4
Mg (MgO)	KL1L2,3	(1P)	1094.41	1129.7 ± 0.4 ^c
		(3P)	1110.08	1144.6 ± 0.4
	KL2,3L2,3	(1S)	1134.47	1169.8 ± 0.4
		(1D)	1141.37	1179.7 ± 0.4

^aRef. 15.^c Ref. 12.^bRef. 16.^d Ref. 14

A correction of 5 eV for the work function was subtracted from all experimental values except for neon, which, being a gas, needs no such correction. Although the agreement between theory and experiment is good for neon and fluorine, large deviations between theory and experiment occur for sodium and magnesium. The average difference between theory and experiment is 19 eV for sodium and 35 eV for magnesium.

V. Conclusion.

The intensities of the K_α satellites of sodium and some of its compounds have been studied in ion-collisions with beams of 5.2 MeV He

and 20 MeV C. An analysis of the spectra indicated that no resonant electron transfer to the L shell of sodium occurs, but that non-resonant transfer takes place. An average filling probability was calculated for each compound and was found to be 0.20 ± 0.01 for 20 MeV C excitation. The filling probability for 5.2 MeV He excitation ranged from 0.27 in Na₂S to 0.45 in NaCl. The filling probability of fluorine in NaF was calculated to be about twice that of sodium in NaF, and a correlation was observed between filling probability and initial state lifetime.

Spectra of RAE x-rays of NaF excited by 5.2 MeV He were taken, and two peaks were observed. One peak, corresponding to the Auger transition $KL_1L_{2,3}$, has not been reported previously. A comparison of theoretical and experimental Auger energies was made and large discrepancies of the order of 20 to 40 eV were found for sodium and magnesium.

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