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ELECTRON AFFINITY OF UF$_6$

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ABSTRACT

Ionization reactions are observed in crossed molecular beams, usually of thermal energy, of alkalis and MoF_6, WF_6 and UF_6. Previous studies have indicated large electron affinities for these hexafluorides, and this is confirmed here. Ionization at thermal energies proceeds with the alkali dimers, A_2, for all three hexafluorides, but with alkali atoms, A, only for UF_6. Several ionization paths are observed, allowing the deduction of molecular energies. A few experiments are done with eV-range beams. Lower limits for the electron affinities are 4.5, 3.3, 4.9, 4.3 and 1.9 eV for MoF_6, MoF_5, WF_6, UF_6 and UF_5, respectively. Possible mechanisms are discussed.

OBJECTIVE

The objective of our experiments was to resolve a controversy which had been extensively discussed at the Atomic Collisions Conference in Seattle, 1975: i.e., to determine whether a number of transition metal hexafluorides MF_6 had electron affinities sufficiently large to chemi-ionize with alkalis at thermal energies. Of primary interest to ERDA was the question: Does the reaction $Cs + UF_6 \rightarrow Cs^+ + UF_6^-$ proceed without the use of high translational energies? Work reported by R. Compton (ORNL) at the Seattle Conference indicated that a number of hexafluorides would chemi-ionize with Cs, and in several cases with K and Na as well. While Compton did not report UF_6 results at the meeting, we learned privately that he had also observed chemi-ionization for this system. On the other hand, S. Datz (ORNL) and K. Lacmann (Berlin) suggested that these results might be due to surface effects.

We suggested to ERDA that we might resolve this dispute, since much of the relevant apparatus was at hand. The results are given in the subsequent technical discussion. It turns out that for UF_6 the reaction proceeds, while with MoF_6 and WF_6 it does not. Chemi-ionization occurs with all three species when alkali dimers are used. Even with UF_6 the dimer reaction is more efficient than the atom reaction.

INTRODUCTION

Positive-negative ion-pairs which result from collisions of alkali atoms, A, with molecules have been frequently studied.[1] In most cases the reaction endoergicities were overcome by kinetic energies in the electron-volt range. However, alkali ionization with some metal hexafluorides MF_6 is exoergic and proceeds at thermal energies.[2-4] This was previously interpreted[4] as the alkali atom process

$$A + MF_6 \rightarrow A^+ + MF_6^- \quad . \tag{R1}$$

In order for R1 to occur at thermal energy, the electron affinity $EA(MF_6)$ must be larger than the atomic ionization potential IP(A). In this paper we find that in some cases dimeric alkalis are the cause of the observed ion pairs, even when the process with atoms is also exoergic.

Lin, Wharton and Grice[5] originally found chemi-ionization processes in collisions of near-thermal K_2 (≈ 0.5 eV) with several halogen-containing molecules. Three types of alkali-dimer ionization reactions[6] occur with halogen molecules X_2 at thermal kinetic energies:

$$A_2 + X_2 \rightarrow A^+ + AX + X^- \tag{R2}$$

$$\rightarrow A^+X^-A^+ + X^- \tag{R3}$$

$$\rightarrow A^+ + X^-A^+X^- \quad . \tag{R4}$$

Exoergicity for R2 is provided mainly by the high binding energy in AX. Paths R3 and R4 have approximately 1.5-2 eV greater exoergicity than R2.[6] A rough generalization of our observations is that path R2 is more probable when it is energetically possible despite the still higher exoergicity from paths R3 and R4. However, these latter two are found when R2 is energetically prohibited.

In the study reported here, chemi-ionization reactions are measured, at thermal energy, between A or A_2 and MF_6. Because beams of A frequently have some unwanted A_2, we wished to investigate the possibility that some results previously attributed to A might have involved A_2 instead.

Possible paths involving dimers include those that are analogous to R1-R4:

$$A_2 + MF_6 \rightarrow A_2^+ + MF_6^- \qquad (R5)$$

$$\rightarrow A^+ + AF + MF_5^- \qquad (R6)$$

$$\rightarrow A^+F^-A^+ + MF_5^- \qquad (R7a)$$

$$\rightarrow A^+MF_5^-A^+ + F^- \qquad (R7b)$$

$$\rightarrow A^+ + F^-A^+MF_5^- \;, \qquad (R8)$$

as well as

$$A_2 + MF_6 \rightarrow A^+ + A + MF_6^- \;. \qquad (R9)$$

Some of these processes are observed and reported for MoF_6, WF_6 and UF_6 at thermal energy. In addition, a few results are given for A-MF_6 collisions in the electron volt energy regime.

EXPERIMENTAL

The thermal-energy 90° cross beam apparatus has been previously described.[6] Briefly, an alkali beam emerges from a 0.1 mm × 6 mm slit in a two-chamber effusion oven, is collimated, passes through an inhomogeneous electromagnet and an 8.5 Hz chopper before reaching the collision region. The MF_6 beam is formed in a capillary array. Product ions are extracted into a quadrupole mass filter and the analyzed ions are counted.

The magnet deflects A but not A_2. It is used to determine which of these caused the ionization. Usually, the apparatus is aligned to allow an undeflected alkali beam to reach the beam interaction region. In this case, product ions are caused by A and/or A_2. If the magnet is energized, some ion signal may be lost, which indicates that those ions originated from collisions with atoms. This is the "deflect out" mode. Alternatively, some experiments are conducted with the beam aligned so that no undeflected alkali reaches the interaction region and no product ions are formed. However, the magnet can divert atoms into the interaction region and any resulting ion signal is <u>solely</u> attributable to A. This is the "deflect in" mode.

Another test for the origin of the product ions involved variation of the alkali source temperatures. The source pressure P is approximately the alkali vapor pressure[7] corresponding to reservoir temperature T_1, which is usually adjusted so that $P < 10^{-1}$ torr. The final alkali temperature T_2 is separately controlled in an exit chamber. For the endothermic process $A_2 \rightleftarrows 2A$, simple equilibrium considerations indicate that y, the mole fraction of A_2, should increase linearly with P but decrease with T_2. Under our conditions y is calculated[7] to vary in the range 10^{-4} to 5×10^{-3}. Previous studies of the beam velocity distribution from this source were well characterized by T_2. However, impurities may cause P to be less than the vapor pressure, which corresponds to T_1. Accordingly, the calculated y's are upper limits. There is also uncertainty in the thermodynamic data, so that y is known only within a factor of ≈ 5. However, there are dramatic changes in dimer intensity and in y with source temperatures. The effect of these changes qualitatively supports the conclusions about the origin of ions as determined by the magnet.

The efficiency of atom removal by the magnet is typically 80-99%, depending on the beam geometry and source conditions. It becomes poorer with increasing P, probably because of "cloud" formation in front of the oven slit. Because we could not measure y, even with 99% removal of atoms, the calculated value is used subsequently. Typically, a 5% change in product intensity is observable. Because the upper limit for y is 5×10^{-3} and our lower limit for atom deflection is 80%, it may be inferred that failure to see a change in ion signal with the magnet implies that the cross section for ionization with dimers is more than 3 orders of magnitude greater than that with atoms.

The alkali chopper, with 50% open time, is used to test the possibility that MF_6^- signals arise from surface ionization. An MF_6^- signal from a surface should be little affected by the chopper, but the intensity is reduced by 50%, which indicates that two-body collisious account for our results.

The ion collection system is subject to mass discrimination.[6] In the X_2 experiments we partially corrected for this by considering the partitioning of kinetic energy between observed ionic fragments. The complexity of MF_6 makes these corrections less plausible here, and they are not attempted. Accordingly, the present results for intensity ratios of ionic products are more qualitative.

The arrangement for producing atomic beams in the eV region, the method of obtaining relative cross sections as a function of energy and the energy calibration using SO_2 as a reference standard has also been described previously.[8] Only a few eV-range measurements were made because an extensive series of such experiments has been completed at Oak Ridge National Laboratory.[4]

RESULTS

MoF_6

With thermal cesium the product ions were Cs^+, MoF_5^- and MoF_6^-. The measured intensity ratio MoF_6^-/MoF_5^- was ≈ 0.6. This ratio, as well as the total intensity, was unaffected by the magnetic field. Accordingly, the ions were exclusively the result of dimer reactions R6 and R9.

Only positive ion data were obtained with potassium, and without magmetic analysis. An intensity ratio K_2F^+/K^+ of ≈ 0.3 was found. In contrast, no Cs_2F^+ was observed with cesium.

With Cs or K in the 5-20 eV region, the only negative ions observed were F^- and MoF_5^- in comparable amounts. An upper limit to the c.m. threshold for the appearance of MoF_5^- from Cs is 4.66 eV. However, there is evidence for the existence of a lower energy tail, which may considerably decrease the actual threshold value.

WF_6

With thermal cesium the observed ions were Cs^+ and WF_6^-. Although F^- or WF_5^- as small as 5% of WF_6^- should have been seen, none was found. Again, tests with the magnet, using both "deflect in" and "deflect out" modes, show clearly that there were no ions from Cs atoms. Accordingly, we conclude that path R9 accounts for the data. Some K^+ was observed in the reaction with potassium. As with MoF_6, this data was for positive ions only, and had no magnetic analysis.

The dominance of WF_6^- persists in high energy K or Cs reactions. With K between 30 and 100 eV (LAB) we find the ratio WF_6^-/WF_5^- to be about 20. With Cs the c.m. threshold for F^- formation is 5.45 eV. At 15 eV (LAB), with Cs the ratio WF_6^-/F^- is about 30, and with 30 eV (LAB) K this ratio is near 50.

UF_6

Most of our data are for UF_6, because the initial objective of the study was to determine whether ionization occurs with atoms at thermal energy. With cesium, both "deflect in" and "deflect out" magnet tests show an atom reaction which produces Cs^+ and UF_6^- according to R1. Potassium atoms also react, although this was tested only with the "deflect out" mode.

As the cesium source is adjusted to increase y, Cs_2F^+ and UF_5^- also appear, in accord with R7a. The ratios Cs_2F^+/Cs^+ and UF_5^-/UF_6^- are in the range 0-3% depending on source conditions. These ratios are increased as much as a factor of 5 by magnetically removing Cs from the beam, which is consistent with an atomic path. Unfortunately, the higher source pressures which favor dimer formation also serve to decrease the efficiency of the magnet. However, the equilibrium calculation yields $y \approx 0.005$ for the source condition at whith $Cs_2F^+/Cs^+ = 0.03$, suggesting that the dimer ionization cross section R7a is greater than that for atomic process R1.

Similar measurements were done with lithium and sodium. The cations produced were Li^+ and Li_2F^+ with the former and Na^+ and Na_2F^+ with the latter. In both cases, small quantities of UF_6^- and UF_5^- were produced. In contrast to the case with Cs, where only a few percent of Cs_2F^+ and UF_5^- were found, the ratios of Li_2F^+/Li^+ and Na_2F^+/Na^+ are close to unity, and more UF_5^- is produced than UF_6^-. None of these ion signals is significantly affected by the magnet indicating that the ions were produced from dimers. However, only two such experiments were done and were of only fair quality. Accordingly, we place less weight upon the lithium and sodium results.

Another such measurement with potassium exhibited intermediate behaviour. There is clearly a reaction with atoms, but increase of y causes the product ratio K_2F^+/K^+ to increase to as much as 0.46. The calculated y at the maximum product ratio is 5×10^{-3}, and consequently the ionization cross section for R7a is much higher than that for R1.

With an eV-range Cs or K beam we found mainly reaction R1 to yield A^+ and UF_6^-, but also some UF_5^-. This is presumably formed by atom reaction

$$A + UF_6 \rightarrow A^+ + UF_5^- + F \qquad \text{(R10)}$$

since no A_2F^+ is observed. With K, the intensity ratio UF_5^-/UF_6^- rises sharply with beam energy from zero at threshold to ≈ 0.12 at 20 eV, falls to ≈ 0.06 at 50 eV and remains approximately this value to 150 eV. Compton[9] has reported data from 0-12 eV. At 11 eV his ratio is about 2 times greater than ours. Given the possibility of error due to mass discrimination, the agreement is reasonable.

DISCUSSION

Energetics

MoF_6

Since ions are produced from MoF_6 with Cs_2, but not with Cs, path R9 leads to a lower limit for $EA(MoF_6)$ of 4.28 eV, i.e., the atomic ionization potential IP(Cs) plus the dissociation energy of the dimer $D(Cs_2)$. The production of MoF_5^- via R6 implies that $EA(MoF_5) > D(Cs_2) + IP(Cs) + D(MoF_5\text{-}F) - D(CsF) = 3.03 \pm 0.4$ eV. The error limit is mainly attributable to the effect of internal energy in MoF_6.

The reaction with potassium was carried out without magnetic analysis of the reagent molecules. However, the analogy with Cs suggests that this is a K_2 reaction, which implies that $EA(MoF_6) > 4.51$ eV. In contrast to the cesium case, significant quantities of A_2F^+ are found with potassium, presumably from reaction R7a. We have not proved the occurrence of R6 with K_2, and thus cannot obtain a higher limit for $EA(MoF_5)$.

Ionic products were also observed with thermal cesium by Cooper, Compton and Reinhardt (CCR).[4] They interpreted the data as evidence for path

R1 and thus concluded that EA(MoF_6) > IP(Cs) = 3.89 eV. We find no evidence of a cesium reaction via path R1. Our threshold measurement with the eV range Cs beam yields 3.3 eV as a lower limit for EA(MoF_5).

$\underline{WF_6}$

Because, for cesium, WF_6 ionizes only with Cs_2, and only via path R9, we obtain the limit EA(WF_6) > 4.28 eV. With potassium we had no magnetic analysis. Both potassium and cesium ionization reactions were reported by CCR. We fail to find any thermal reactions with Cs atoms and have no information about K atoms. However, it is reasonable to suppose that the potassium ionization, both ours and that of CCR, is also caused by dimers. If so, these data imply that EA(WF_6) > IP(K) + D(K_2) = 4.85 eV. An endoergic reaction threshold with Na was found by CCR and from this EA(WF_6) was determined to be ≈4.5 eV. We consider this to be reasonable agreement.

With the accelerated Cs beam, the appearance potential for F^- implies a dissociation energy D(WF_5-F) of 5.0 eV, in good agreement with Hildenbrand's value[10] of 5.25 eV.

Dispert and Lacmann (DL)[11] have reported results of chemi-ionization of WF_6 with K and Na. For the dissociation energy WF_5-F they obtain 5.11 ± 0.3 eV.

There is a disagreement with the electron affinity reported by DL of 3.7 eV and by us (4.85 eV) or CCR (4.5 eV). The data of DL and CCR are very similar, but DL have assumed a linear c.m. cross section. A glance at their Figure 3 shows that the appearance potential determined in this way is an upper limit and so their electron affinity should be considered a lower limit. Our experience with similar data is that the calculated threshold is very sensitive to the assumed form of the c.m. cross section, and to any "constant" background subtraction.

A previous one-sentence statement[3] from this laboratory reported thermal ionization between cesium and WF_6 and has been cited[4,9,12] as support for R1. However, no analysis for a Cs_2 component in the beam was done at that time and we now presume that those ions were due to Cs_2.

UF_6

The reaction of K_2 with UF_6 on path R9 implies that $EA(UF_6) > 4.85$ eV. However, the difficulty of separating the dimer and monomer contributions (via R9 and R1, respectively) to the K^+ and UF_6^- signals suggests that our upper limit be confined to that of R1, i.e., $EA(UF_6) > 4.34$ eV. The energetics for R7a yield a limit $EA(UF_5) > 1.90$ eV.

Small amounts of UF_6^- and A^+ were also produced with Li_2 and Na_2 (the signals were approximately independent of the magnet). Straightforward application of the energetics would then lead to the conclusion that $EA(UF_6) > 6.42$ eV. However, the small quantity produced suggests that possibly this is the result of internal energy in the UF_6. This point requires further investigation.

Our lower limit $EA > 4.34$ eV is in accord with Compton[9] who finds > 5.1 eV on the basis of ionization with thermal socium beams, and Beauchamp,[13] who estimated 4.9 ± 0.5 eV from an ICR study. Our most reliable values, and those of previous investigators, are contained in Table I.

TABLE I

LIMITS FOR ELECTRON AFFINITIES AND DISSOCIATION ENERGIES (eV) OBTAINED FROM PRESENT EXPERIMENTS AND FROM LITERATURE

(See Text for Details)

	Present Results		Literature Values	
	EA	$D(MF_5{-}F)$	EA	$D(MF_5{-}F)$
MoF_6	$>4.5 \pm 0.4^a$		$>3.9^c$ $(>4.5)^d$	
MoF_5	$>3.0 \pm 0.4^a$ $>3.3 \pm 0.4^b$			
WF_6	$>4.9 \pm 0.4^a$	$<5.0 \pm 0.4^b$	$>4.5^3$ 3.7^g	5.3^f 5.1 ± 0.3^g
UF_6	$>4.3 \pm 0.4^a$		$>5.1^h$ $>4.9^i$	
UF_5	$>1.9 \pm 0.4^a$	—	$>4.0^h$	

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a. Thermal energy alkali beam.

b. eV-range alkali beam.

c. Original value, Ref. 4.

d. Ref. 4, assuming results due to Cs_2.

e. Ref. 4, threshold measurement.

f. Hildenbrand, see Ref. 10.

g. See Ref. 11.

h. See Ref. 9.

i. See Ref. 13.

Mechanism

Annis and Datz (AD)[12] have studied the neutral products from the thermal reaction of K and Cs with UF_6, WF_6, MoF_6, TeF_6 and SeF_6. The cross sections, Q_r, to form AF (and possibly AMF_5) from UF_6 and WF_6 are ≈ 140 Å^2 and are considerably smaller for MoF_6. The angular distributions were consistent with complex formation. They found no ionic products and estimated the cross section for ion formation Q_i to be less than 0.01 Å^2. Our experiment does not yield a value for Q_i and it is conceivable that the AD limit is correct. In any case $Q_r >> Q_i$. With WF_6 and MoF_6 we find no atomic ionization reaction at thermal energy and estimate that the Q_i's are at least an order of magnitude less than that for UF_6.

Both AD and Compton[9] have treated the Cs + UF_6 reaction as a statistical decomposition of an intermediate complex. A very low Q_i is calculated by AD. Compton finds the branching ratio (UF_6^-/CsF) to be low, but very sensitive to EA(UF_6). He argues that it would be higher if the centrifugal barrier for the neutral products had been included in the calculation. If we assume that the ions result from a statistical decomposition, then the very low Q_i with MoF_6 and WF_6 might be attributed to lower electron affinities for these molecules.

The metal hexafluorides have the largest electron affinities yet measured. The harpooning theory of electron transfer involves crossings of neutral and ionic potential energy surfaces. However, when EA(MF_6) > IP(A), the ground-state ionic surface lies entirely below the neutral surface and there are no crossings. Compton[9] suggests that crossings with UF_6 might involve excited UF_6^- states. Such surfaces would consist of varying levels of vibrational excitation or of electronically excited UF_6^-. Boring, Wood and Moskowitz[14] have predicted the existence of bound, electronically-excited states of UF_6^-. Because Cs ionizes with UF_6, but not with MoF_6 or WF_6, and because the vibrational manifolds are likely to be similar for all three species, electronic excitation seems more likely to play a role in the ionization. Because the Q_r for Cs with UF_6 and WF_6 are similar and are considerably larger than that for MoF_6, while we find that only UF_6 ionizes, there may be different initial steps in the ionization reaction than that with neutral products.

Ionic products are obtained from MoF_6 and WF_6 only with Cs_2, but not with Cs. Because the atomic reaction R1 to form $A^+ + MF_6^-$ would be more exoergic than the dimeric reaction R9 with the same products, the mechanism should explain the preference for path R9. An obvious possibility is a surface crossing with an electronically excited state of Cs_2^+. Fig. 1 shows the potential curves taken from Ref. 15. A Franck-Condon transition to the ungerade state has a sufficiently high range of vertical ionization energies to lead to a reasonable value for the crossing radii of neutral and ionic surfaces. This Cs_2^+ state is repulsive, which accounts for production of Cs^+ rather than Cs_2^+. The latter has never been seen in these experiments. Its absence is an argument against a complex intermediate in path R9 as the production of Cs_2^+ would be somewhat more exoergic than that of Cs^+. On the other hand, the very exoergic path R7a to form Cs_2F^+ might emerge from a complex. With MoF_6, for which both MoF_6^- and MoF_5^- are observed, only Cs^+ is produced, again suggesting a direct mechanism.

The general conclusion of our study is that the ionic process R1, although energetically preferred over the dimeric reaction R6, only occurs

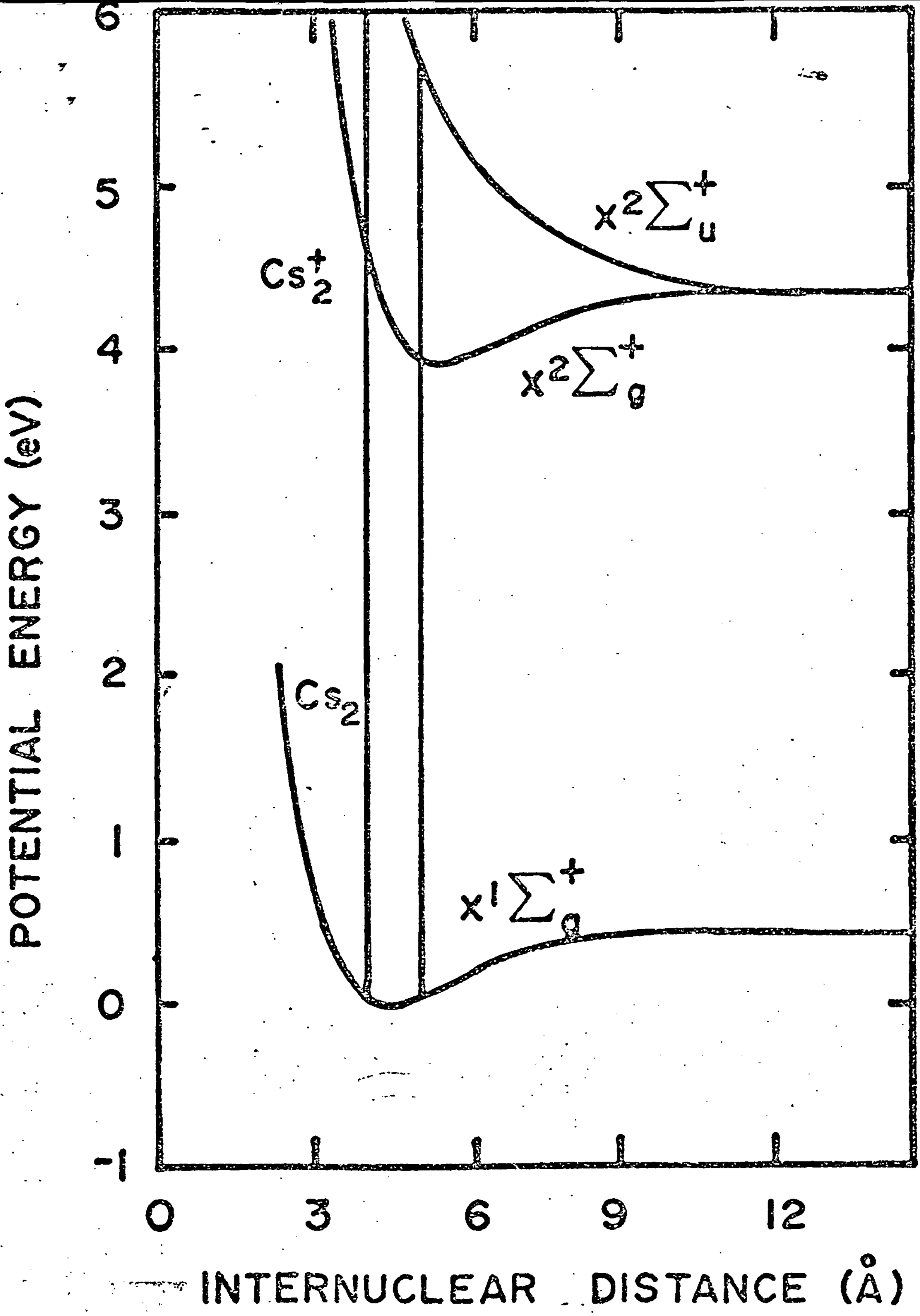

FIGURE 1. Potential energy states for the ground states of Cs_2 and Cs_2^+, taken from data assembled in Ref. 14. The vertical lines indicate the Franck-Condon region. The figure suggests that the ionization cross section might be quite sensitive to internal energy in Cs_2. This point was not investigated here.

in the case of Cs-UF_6 and K-UF_6, possibly through the medium of excited electronic states of UF_6^-. The ions observed in the other cases result from dimeric reactions.

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