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"RAMAN SCATTERING OF NEAT AND MIXED CRYSTALS OF IrF_6: THE JAHN-TELLER INTERACTION IN THE GROUND STATE"

by

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Raman scattering data for neat and mixed crystal IrF_6 samples at 77 K are reported. It is found that ν_2 ($\nu(2)$) (e_g) and ν_5 ($\nu(5)$) (t_{2g}) both evidence non-vanishing Jahn-Teller interactions for the Γ_{8g} ($^4A_{2g}$) ((4) $A(2g)$) ground state. The

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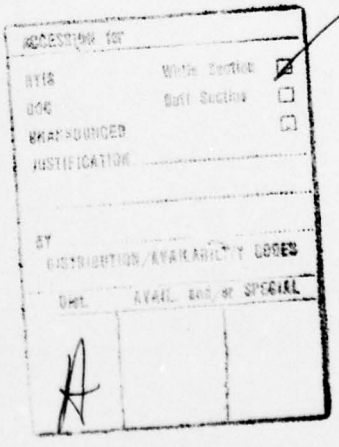
20. (continued...)

linear Jahn-Teller parameters for these modes are $D_{(2)} < 0.001$ and $D_{(5)} = 0.02$. The $\nu_{(5)}$ Jahn-Teller interaction is large enough to quench an expected exciton splitting of ~20 cm^{-1} almost completely. The $\nu_{(2)}$ Jahn-Teller interaction is small enough so that exciton interactions predominate; however, normal $\nu_{(2)}$ $\underline{k}$ = 0 sharp line exciton structure is not observed. Anomolous phonon behavior is observed and discussed with respect to other hexafluoride phonon data. The non-Jahn-Teller active modes $\nu_{(1)}$, $\nu_{(3)}$, $\nu_{(4)}$, $\nu_{(6)}$ are found to behave in a fashion consistent with other members of this series.

approx. 1/20 cm



I. INTRODUCTION

Raman scattering and infrared absorption studies have been the important experimental methods in understanding the Jahn-Teller (JT) effect in the ground states of paramagnetic transition metal hexafluoride systems.[1-6] Raman spectroscopy is, however, the superior experimental technique in this instance as selection rules allow direct observation of the relevant vibrational modes [ν_2 (e_g), ν_5 (t_{2g})]. Experimental studies reported here encompass Raman spectra of the ground state manifold of neat and mixed crystals of IrF_6 at 77 K.

The JT interaction in the ground state of IrF_6 is thought to be small.[1,3,7] Weinstock and Goodman predict, on the basis of an approximate calculation which includes only t_{2g} orbitals, that the JT interaction is two orders of magnitude smaller than that in ReF_6.[3] There exist two experimental observations which support the contention that JT interactions are small in the ground state of IrF_6:

1) Unlike ReF_6[3,6] and OsF_6[3], for which large ground state JT effects have been identified, the [ν_2 (e_g) + ν_3 (t_{1u})] combination band observed in the infrared absorption spectrum of IrF_6 is not broader than that found for non-JT active hexafluorides such as WF_6; and

2) The ν_5 (t_{2g}) frequency of IrF_6 fits into the general MF_6 frequency systematics for non-JT active systems. In JT active hexafluorides (e.g., ReF_6[3] and OsF_6[3]), the ν_5 frequencies obviously do not fit hexafluoride systematics.

These data do not, however, indicate how much smaller the JT interaction actually is. The main purpose of the present paper is to address this question.

Available Raman spectra of IrF_6 vapor[4] are not as informative as they might be since important but weak vibronic features may have been missed due to poor

signal-to-noise ratio. Such was found to be the case in the ReF_6 spectrum.[5,6]

There are three distinct advantages to a low temperature, solid state Raman study over gas phase work. First, larger Raman signals obtain due to the increased molecular density. Second, better spectral resolution is achieved due to the absence of rotational structure and most hot bands. Third, as discussed in reference 6 for neat ReF_6, a low-lying electronic state (ca. 30 cm^{-1} for ReF_6 and ca. 5 cm^{-1} for IrF_6) can have important effects on observed vibrational exciton band structure. In the absence of JT vibronic coupling (e.g., ν_1 (a_{1g}), ν_3 (t_{1u}), ν_4 (t_{1u}), ν_6 (t_{2g})) it is apparent that vibrational and electronic excitons are independent, giving rise to sharp $\underline{k}$ = 0 vibrational exciton structure much like that which is observed for non-paramagnetic (non-JT) hexafluoride neat crystals.[8] If vibronic coupling is important, however (e.g., ν_2 (e_g) and ν_5 (t_{2g})) and the low-lying electronic state is thermally populated, a much more complicated overall spectrum is found. This spectrum consists of band-to-band transitions,[6,8,11,12] hot bands, and interference effects between exciton and JT splittings.[6] Interestingly enough, this additional complexity in the ν_2 and ν_5 bands may serve as a diagnostic for recognizing small or obscured JT effects.

In addition to JT active modes, the present study yields information concerning non-JT active modes (ν_1 (a_{1g}), ν_3 (t_{1u}), ν_4 (t_{1u}), ν_6 (t_{1u})) and phonons of neat IrF_6.

II. EXPERIMENTAL

The samples used in these Raman studies were the following: neat IrF_6, 3% IrF_6/WF_6, and 5% IrF_6/MoF_6. The details of sample preparation and crystal growth are given in reference 9.

The Raman apparatus consisted of an Ar^+ laser (Spectra Physics 170) with special optics to permit use of the 5287 Å line, an f/5.8 0.5 m double monochrometer (McPherson 285) with 1800 gr/mm holographic gratings, micropositioning devices for maximizing the Raman signal and diffraction limited focusing and collection optics. The sample was mounted in a small Pyrex dewar to allow 77 K spectra to be obtained. The detector was a cooled photomultiplier tube (RCA C31034A-02) operated in the photon counting mode. The 5287 Å Ar^+ laser line was used as the exciting source to minimize sample absorption. Typical experimental parameters were the following: 0.5-1 W of 5287 Å laser power, and 0.5-2 cm^{-1} slitwidths. Calibration was achieved by recording Fe-Ne hollow cathode emission lines[10] over the entire spectral region studied. Accuracy of sharp lines is $\pm$ 0.1 cm^{-1}.

III. RESULTS AND DISCUSSION

A summary of the 77 K Raman spectrum of neat IrF_6 is given in Table 1. A summary of the 3% IrF_6/WF_6 and 5% IrF_6/MoF_6 data is given in Table 2. A survey of Raman spectrum of neat IrF_6 is presented in Figure 1; more detailed spectra are given in Figures 2-8.

Before proceeding to the principal theme of the paper, which concerns the JT interaction in the ground state of IrF_6 and the information which the ν_2 (e_g) and ν_5 (t_{2g}) data give in this regard, some other aspects of the spectra will be discussed. A number of these points bear indirectly on the JT discussion.

A. Phonons. The frequency of observed phonons of neat IrF_6 may be found in Table 1 (see Figure 2 also). According to the theory presented in reference 8, these frequencies should be similar to those of WF_6.

Using the notation of reference 8 in which T_i and L_i label the translational and librational (rotational) phonon modes, a comparison shows that L_4 (L_5 and/or T_7), and T_5 are missing from the IrF_6 spectrum and that "extra" peaks are found at 61.0 and 63.9 cm^{-1}. It might be noted that the ReF_6 spectrum is also missing the same phonons, but apparently does not have the additional peaks that IrF_6 has.[8] The "extra" phonons in IrF_6 cannot be explained as being (T_3 and/or L_7) which is the only missing phonon in the WF_6 spectrum. Although the absence of a peak may be rationalized as being caused by variations in scattering intensity, appearance of a peak where none is predicted seems a more serious discrepancy. Thus, it is concluded that the detailed behavior of some of the phonons of IrF_6 does not coincide with the behavior of those of WF_6, UF_6, or MoF_6.

The paramagnetic nature of IrF_6 suggests two possible reasons for this difference: magnetic interaction or a Jahn-Teller effect. Alternatively, the fact that Ir is in a different periodic group than W suggests that differences in phonon behavior might be correlated with differences in IrF_6 and WF_6 electronic distribution and structure. Lower temperature Raman spectra (77 K - 1.6 K) might shed additional light on this subject.

B. ν_1 (a_{1g}), ν_3 (t_{1u}), ν_6 (t_{2u}). Assignments for non-JT active vibrations ν_1, ν_3, and ν_6 may be made by comparison with the gas phase data (Table 3). The totally symmetry vibration ν_1 is observed, as expected, close to the gas phase value.[11]

Assignment of the ν_3 $\vec{k}$ = 0 components at 692.6, 707.3, and 741.3 cm^{-1} is consistent with the gase phase ν_3 value of 719 cm^{-1}, large dipole-dipole type exciton interactions,[11] and the broad (90 cm^{-1}) band observed at $2\nu_3$.

Intensity of the 692.6 and 706.3 cm^{-1} ν_3 components has been increased, apparently by Fermi resonance with ν_1 (see Figure 3); the 741.3 cm^{-1} component is less intense by more than an order of magnitude. Intensity of ν_6 is low; nonetheless it can be observed at 221.1 cm^{-1}, an increase of 15 cm^{-1} over the gas phase value of 206 cm^{-1}. Such an increase in the ν_6 frequency is consistent with other hexafluoride data.[11]

The ν_1, ν_3, and ν_6 data corroborate the discussion in Section I concerning the behavior of non-JT active vibrations and low-lying electronic exciton bands in that sharp $\underline{k}$ = 0 structure is observed and no hot bands are found.

C. ν_2 (e_g). The peaks at 647.7 and 643.0 cm^{-1} in neat IrF_6 may be identified as ν_2 components by a comparison with the gas phase ν_2 frequency of 645 cm^{-1}. A notable characteristic of the neat IrF_6 ν_2 spectrum is its broad, band-like nature (Figure 4). As discussed above, this is indicative of

a JT interaction in the ν_2 coordinate. Observation of two ν_2 components in the neat IrF_6 spectrum may be interpreted in either of two ways: these peaks are maxima in the ν_2 exciton density-of-states function, or they are associated with a JT splitting of ν_2. The data appear to support the former view. Figure 5 shows the $\nu_1 + \nu_2$ peak for neat IrF_6; considerations in reference 12 indicate that the band shape for $\nu_1 + \nu_2$ should approximate the exciton density-of-states function for ν_2. Thus, similarity of the IrF_6 ν_2 and $\nu_1 + \nu_2$ band shapes with those of MoF_6, WF_6 and UF_6 supports the first interpretation. Additionally, the ν_2 spectrum of IrF_6 in a WF_6 mixed crystal (Figure 6), as would be expected if exciton interactions are dominant in the neat crystal, is quite different from that observed in neat IrF_6.

Both neat and mixed crystal spectra of IrF_6 imply that the JT interaction is small for the ν_2 mode. A rough upper limit on the magnitude of the interaction may be set by supposing that the 6 cm^{-1} bandwidth of ν_2 in 3% IrF_6/WF_6 (Table 2) is due to a ~3 cm^{-1} JT splitting. Using Child's[13] perturbational expression, a $D_2 \leq 0.001$ is obtained.

D. $\underline{\nu_4\ (t_{1u}),\ \nu_5\ (t_{2g})}$. The gas phase IrF_6 data (Table 3) suggest that the 264.4 cm^{-1} peak in neat IrF_6 is a ν_5 component and that those at 278.6 and 287.6 cm^{-1} are ν_4 components. The assignment of the major feature at 298.5 cm^{-1} requires more detailed considerations (Figure 7). As in the ν_2 spectrum, the broad bandwidths observed here are indicative of a JT effect.

The proximity of ν_4 and ν_5 in the gas phase (276 and 267 cm^{-1}, respectively) suggests that crystal-induced Fermi resonance between ν_4 and ν_5 might be important. In neat UF_6, for example, ν_4 and ν_5 each appear to be repelled ~15 cm^{-1} by a Fermi resonance interaction.[11] If such were the case in neat IrF_6, then the peak at 298.5 cm^{-1} might be assigned as a Fermi resonance-perturbed ν_4 component. It may be seen, however, that the Fermi resonance

interaction is not as important in the present case because the ν_5 peak at 264.4 cm^{-1} lies quite close to the ν_5 gas phase value (267 cm^{-1}). The peak at 298.5 cm^{-1} may thus tentatively be assigned as a ν_5 component, consistent with its Raman intensity and broad line width. Presumably the difference in behavior of UF_6 and IrF_6 is related to a larger molecular distortion in the UF_6 crystal.

Verification of JT split ν_5 components appearing at 298 and 264 cm^{-1} can be found in mixed crystal data (Table 2 and Figure 8). As has been noted previously for ReF_6 ground state ν_2 and ν_5 modes,[6] the virtual identity of pure and mixed crystal features conclusively points to a JT interaction. Thus the 34 cm^{-1} ν_5 splitting observed in both pure and mixed IrF_6 crystals can be assigned as due to a linear JT interaction.

In linear JT theory, which should be applicable in this case, the 264.4 cm^{-1} feature is designated as ν_5 (J_5 = 3/2) and the 298.5 cm^{-1} peak is ν_5 (J_5 = 1/2). The linear JT parameters are: D_5 = 0.02 and ν_5^0 = 276 cm^{-1}.[13] Note that the ν_5^0 value is similar to the ν_5 (287 cm^{-1}) observed in the Γ_{7g} ($^2T_{2g}$) electronic state of IrF_6 [14] and thereby strengthens the assignment. The gas phase spectra of ν_5 must then contain only the J_5 = 3/2 component. It should be noted that a roughly 10 cm^{-1} quadratic JT shift of ν_5^0 ($Q[a_{1g}] \sim -0.08$) could be assigned for the ground state ν_5 based on a comparison with the Γ_7 ν_5 value.[6,14,15] However, since the shift is small and the comparison to $\nu_5(\Gamma_7)$ is not quantitatively reliable, we hesitate to make a firm assignment of a quadratic JT effect at present.

The prediction of Weinstock and Goodman noted in Section I that the JT effect is vanishingly small in the ground state of IrF_6 seems to be more nearly correct for the ν_2 mode than for the ν_5 mode. A possible reason for the larger JT interaction in the ν_5 mode is configuration interaction of e_g and charge-transfer[16] states with the t_{2g} states; Weinstock and Goodman did not account for these in their calculation.[3]

IV. CONCLUSION

The principal conclusions of these Raman studies of IrF_6 in various solids are that an easily observable Jahn-Teller interaction is present in the ν_5 (t_{2g}) vibrational mode of the Γ_{8g} ($^4A_{2g}$) ground state of IrF_6 (D_5 = 0.02) and that a vanishingly small Jahn-Teller effect is found in the ν_2 (e_g) mode ($D_2 \leq 0.001$).

It is also found that the phonon frequencies in neat IrF_6 are not completely consistent with those of other (non-paramagnetic) hexafluorides.

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Table 1. Summary of neat IrF_6 Raman spectrum at 77 K. The accuracy of the sharp lines is ±0.1 cm^{-1}.

Stokes Shift (cm^{-1})	I[a]	FWHH (cm^{-1})[b]	Assignment	
25.7	-	1	T_1	Phonons[c]
30.0	-	1	L_1, T_2	
34.3	-	1	L_2, T_6	
61.0	-	2		
63.9	-	-		
65.2	-	2	L_3 (?)	
80.5	-	2	L_6, T_4	
221.1	VW	-	ν_6	
264.4	W	10	ν_5 (J_5 = 3/2)	
278.6	W	-	ν_4	
287.6	W	-		
298.5	W	16	ν_5 (J_5 = 1/2)	
643.0	M	3	ν_2	
647.7	M	4		
692.6	W	1		ν_3
701.2	S	≤0.5	ν_1	
707.3	W	1		
741.3	VW	-		
1343.7	W	-	$\nu_1 + \nu_2$	
1348.1	W			
1402.7	W	90	$2\nu_1$	$\nu_1 + \nu_3$, $2\nu_3$
1435.6	W			
1472.4	VW			

(a) Intensity: VW = very weak; W = weak; M = medium; S = strong.
(b) FWHH = full width at half height.
(c) The assignments which are made are in analogy with the assignments for WF_6 in reference 8. T_i are translational phonons and L_i are librational (rotational).

Table 2. Summary of Raman spectra of IrF_6 in various mixed crystals at 77 K.

	Stokes Shift (cm^{-1})	FWHH (cm^{-1})(a)	Assignment
IrF_6/WF_6	263.6	-	$\nu_5(J_5 = 3/2)$
	297.6	-	$\nu_5(J_5 = 1/2)$
	644.3	6	ν_2
	702.0	<1.4	ν_1
IrF_6/MoF_6	263.8	6	$\nu_5(J_5 = 3/2)$
	297.6	4	$\nu_5(J_5 = 1/2)$
	701.8	<1.2	ν_1

(a) FWHH = full width at half height.

Table 3. Gas phase values of the vibrational frequencies of IrF_6[a].

ν_1 (a_{1g})	ν_2 (e_g)	ν_3 (t_{1u})	ν_4 (t_{1u})	ν_5 (t_{2g})	ν_6 (t_{2u})
701.7	645	719	276	267	206

(a) Reference 5.

Figure 1.

Survey Raman spectrum of neat IrF_6 at 77 K. The ordinate is a logarithmic scale. A rough idea of relative intensities can be obtained by noting that the intensity of ν_1 (701.2 cm^{-1}) is ~10^5 cps while the background is ~10^2 cps.

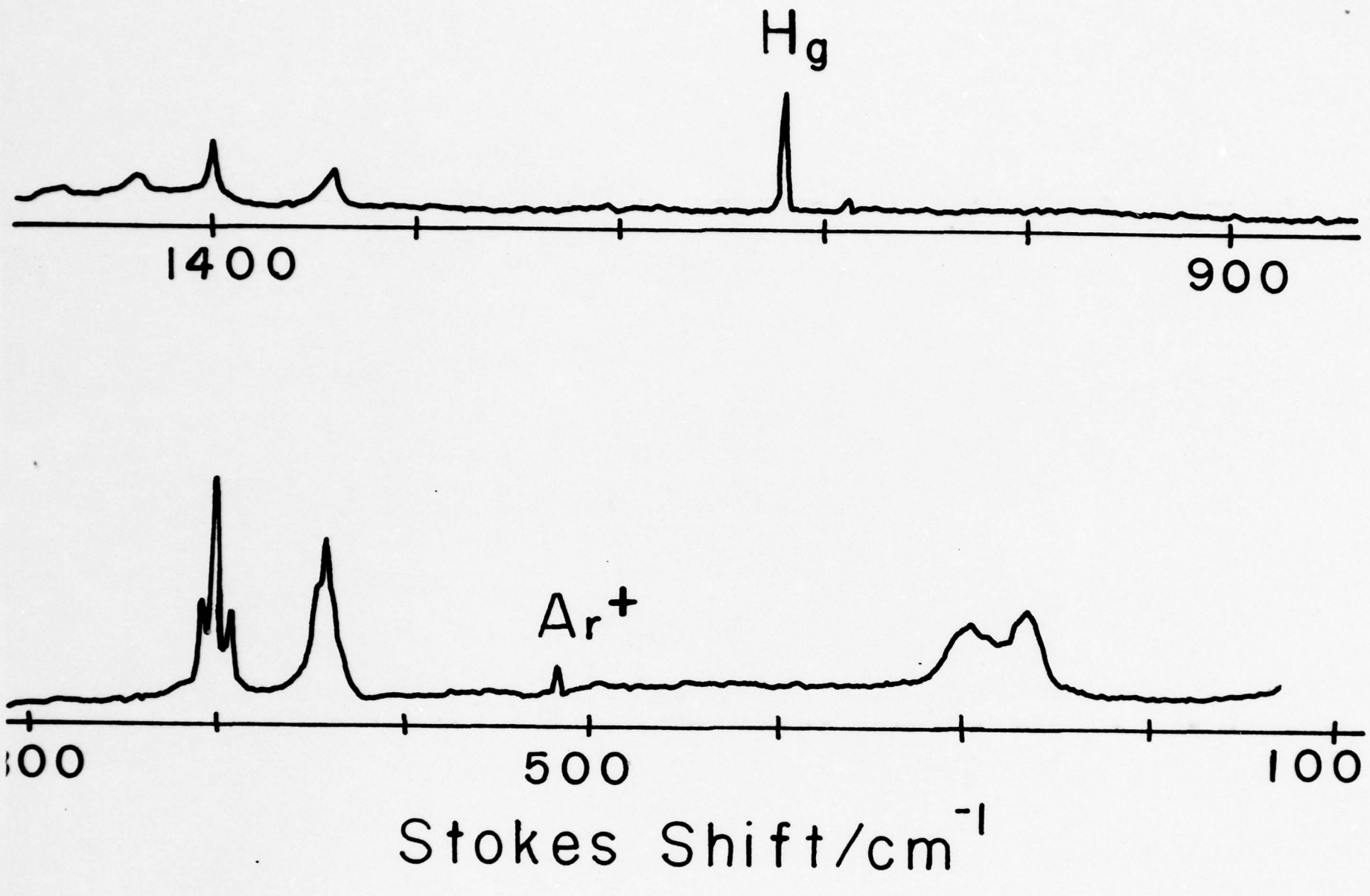

Hg
1400
900
Ar+
500
100
Stokes Shift/cm-1

Figure 2.

Raman spectrum of neat IrF_6 at 77 K in the phonon region.

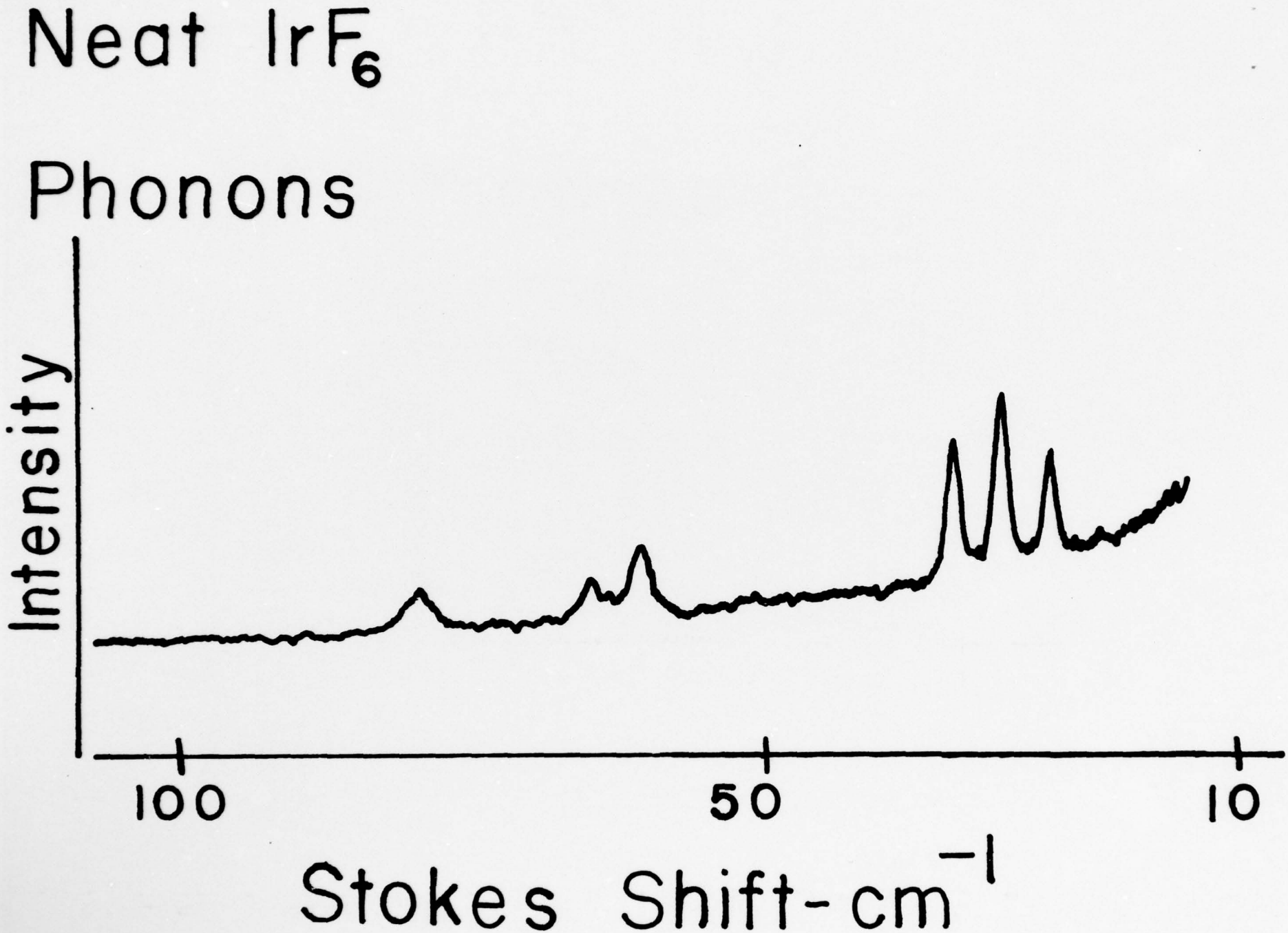

Neat IrF_6
Phonons
Intensity
100
50
10
Stokes Shift - cm^{-1}

Figure 3.

The 77 K Raman spectrum of neat IrF_6 in the region of the totally symmetric vibration, ν_1. Note the sharp and relatively intense ν_3 $\vec{k} = 0$ components. Their intensity has been enhanced by crystal-induced Fermi resonance with ν_1.

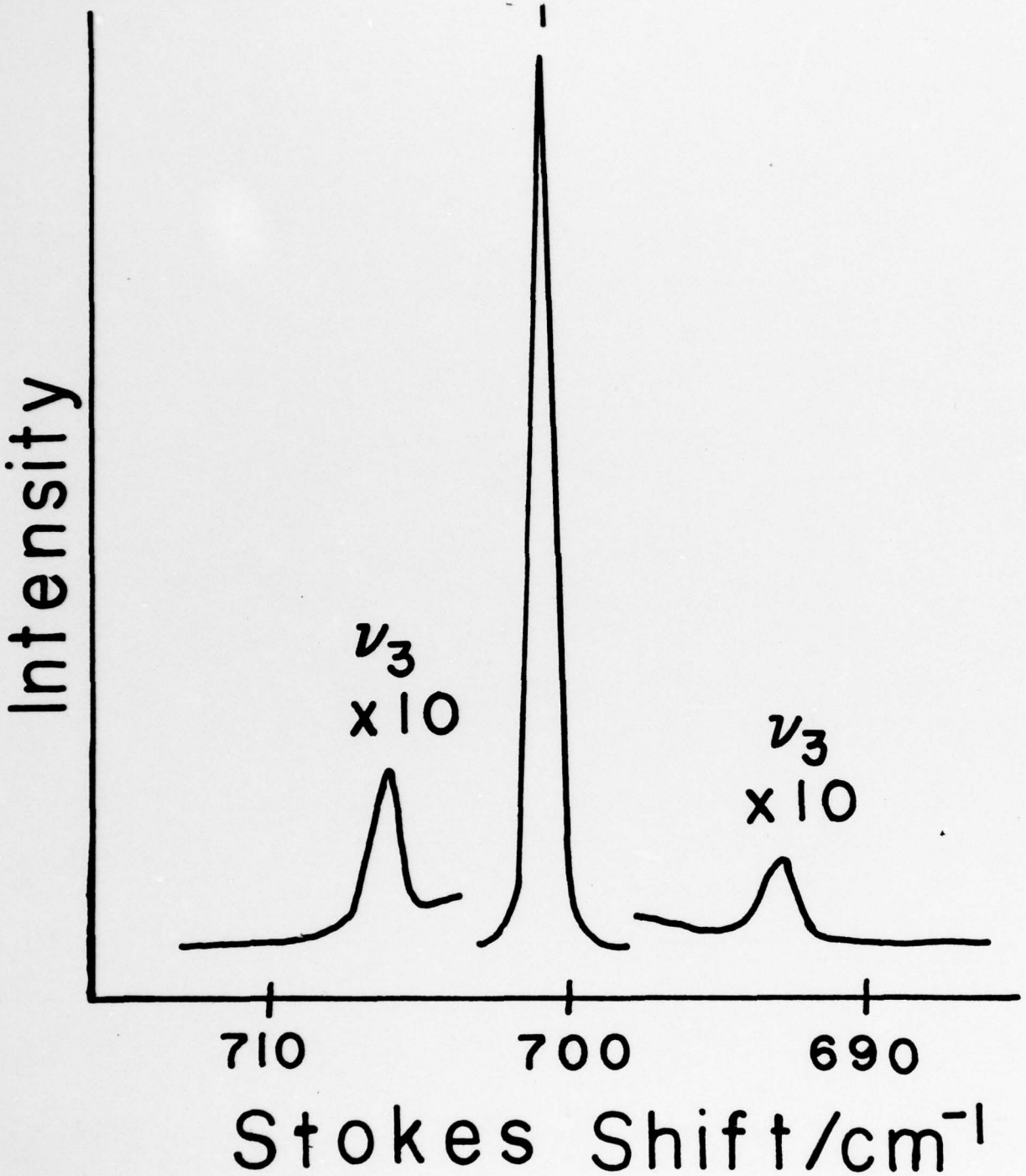

1
Intensity
ν_3
x10
ν_3
x10
710
700
690
Stokes Shift/cm^{-1}

Figure 4.

The Raman spectrum of the ν_2 (e_g) vibration of neat IrF_6 at 77 K.

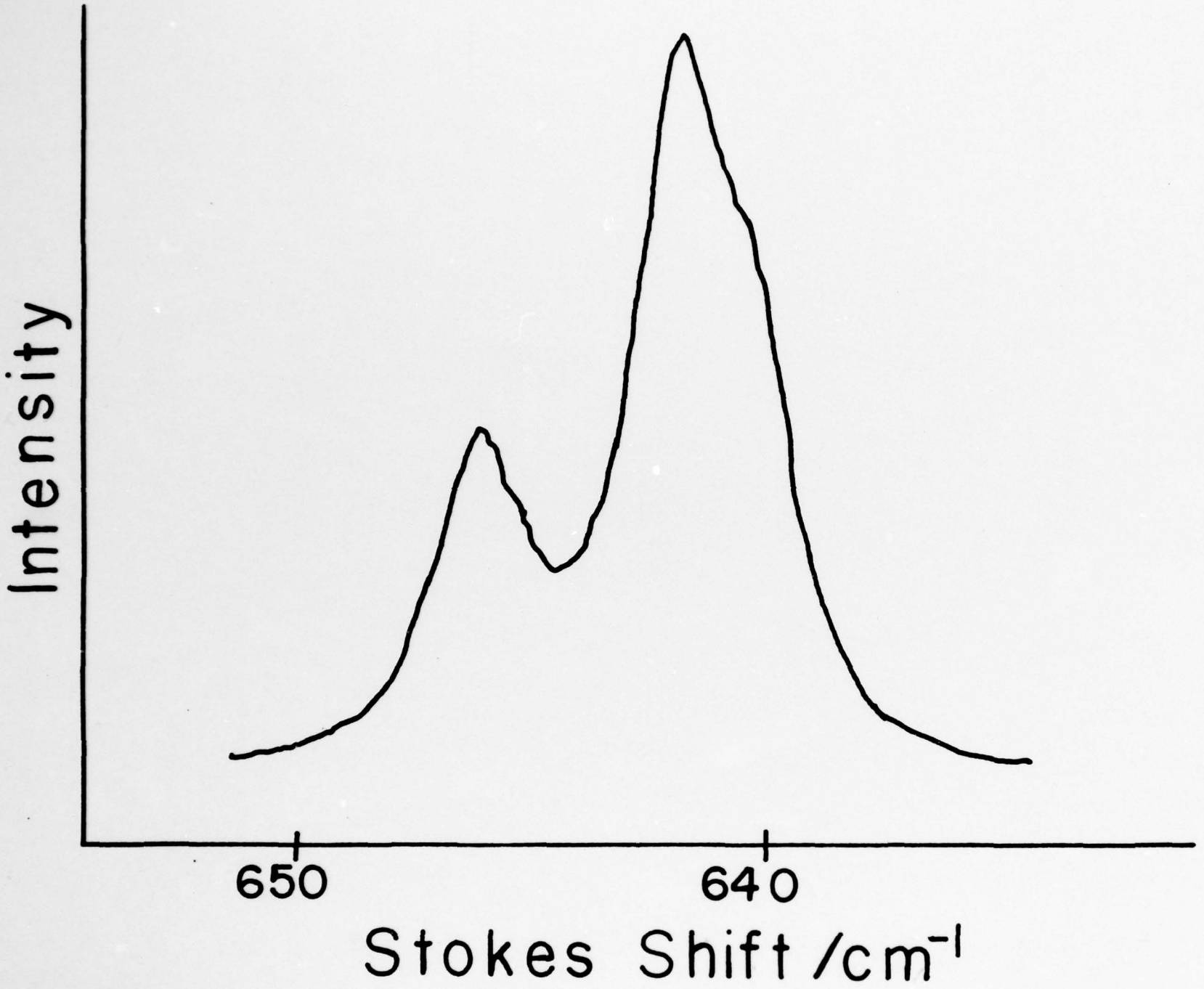

Intensity
650
640
Stokes Shift /cm⁻¹

Figure 5.

The Raman spectrum of $\nu_1 + \nu_2$ in neat IrF_6 at 77 K. Note the resemblance to ν_2 in Figure 5.

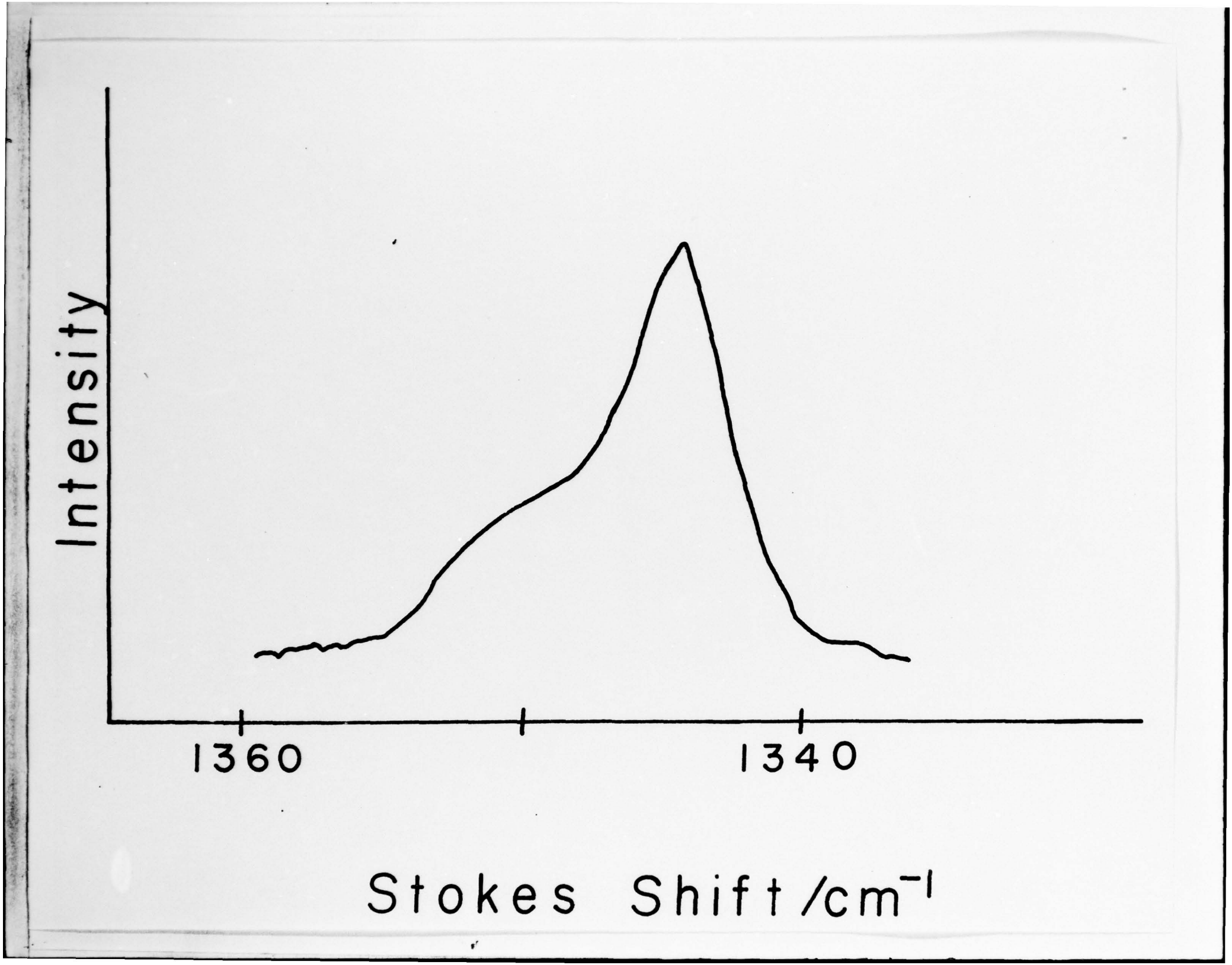

Intensity
1360
1340
Stokes Shift /cm⁻¹

Figure 6.

The Raman spectrum of the ν_2 (e_g) vibration of IrF_6 in a mixed crystal (3% IrF_6/WF_6) at 77 K.

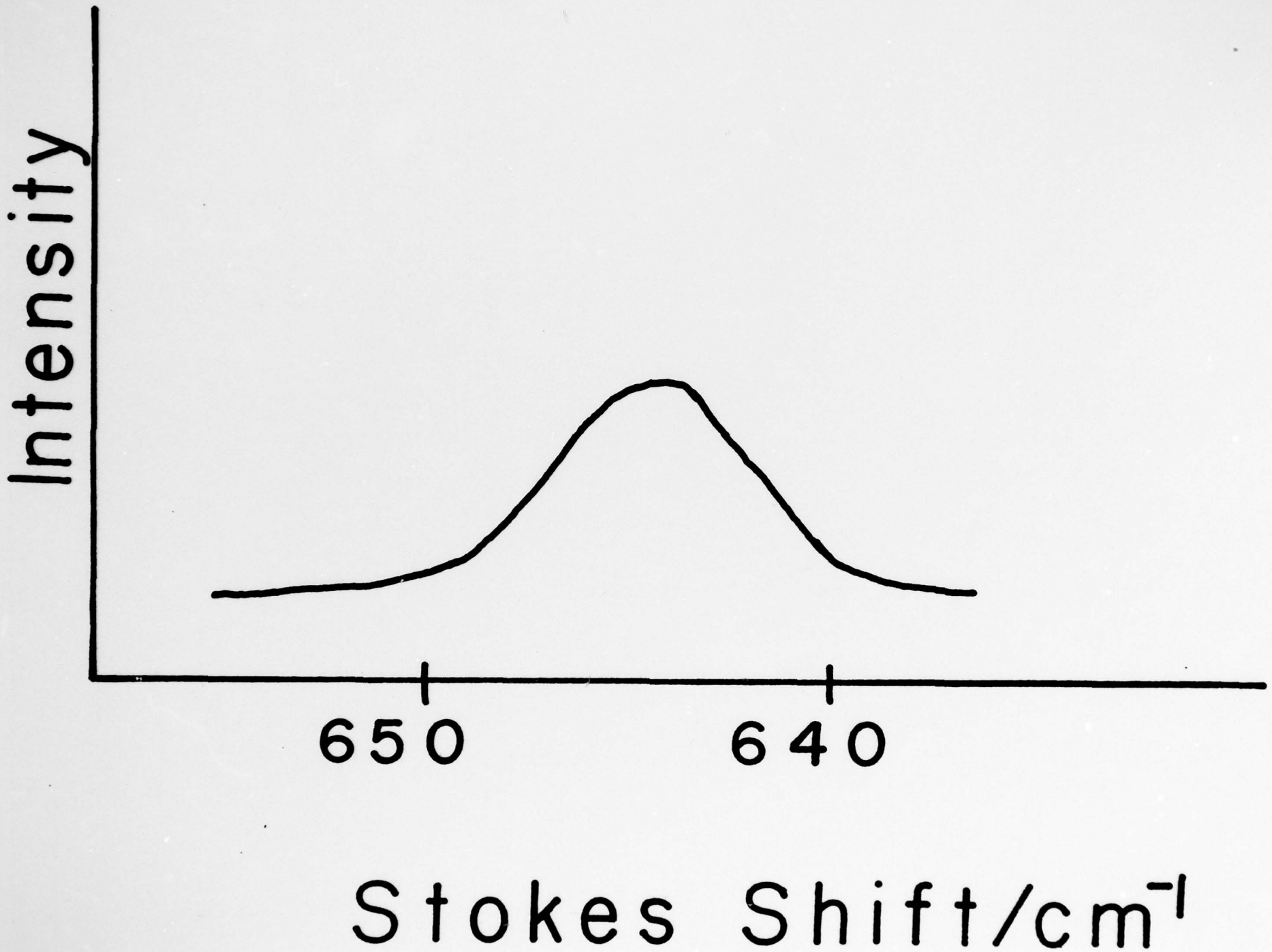

Intensity
650
640
Stokes Shift/cm⁻¹

Figure 7.

The Raman spectrum of the vibrational bending region (ν_4 and ν_5) of neat IrF_6 at 77 K. Note the broad nature of the bands in contrast to those in Figure 3.

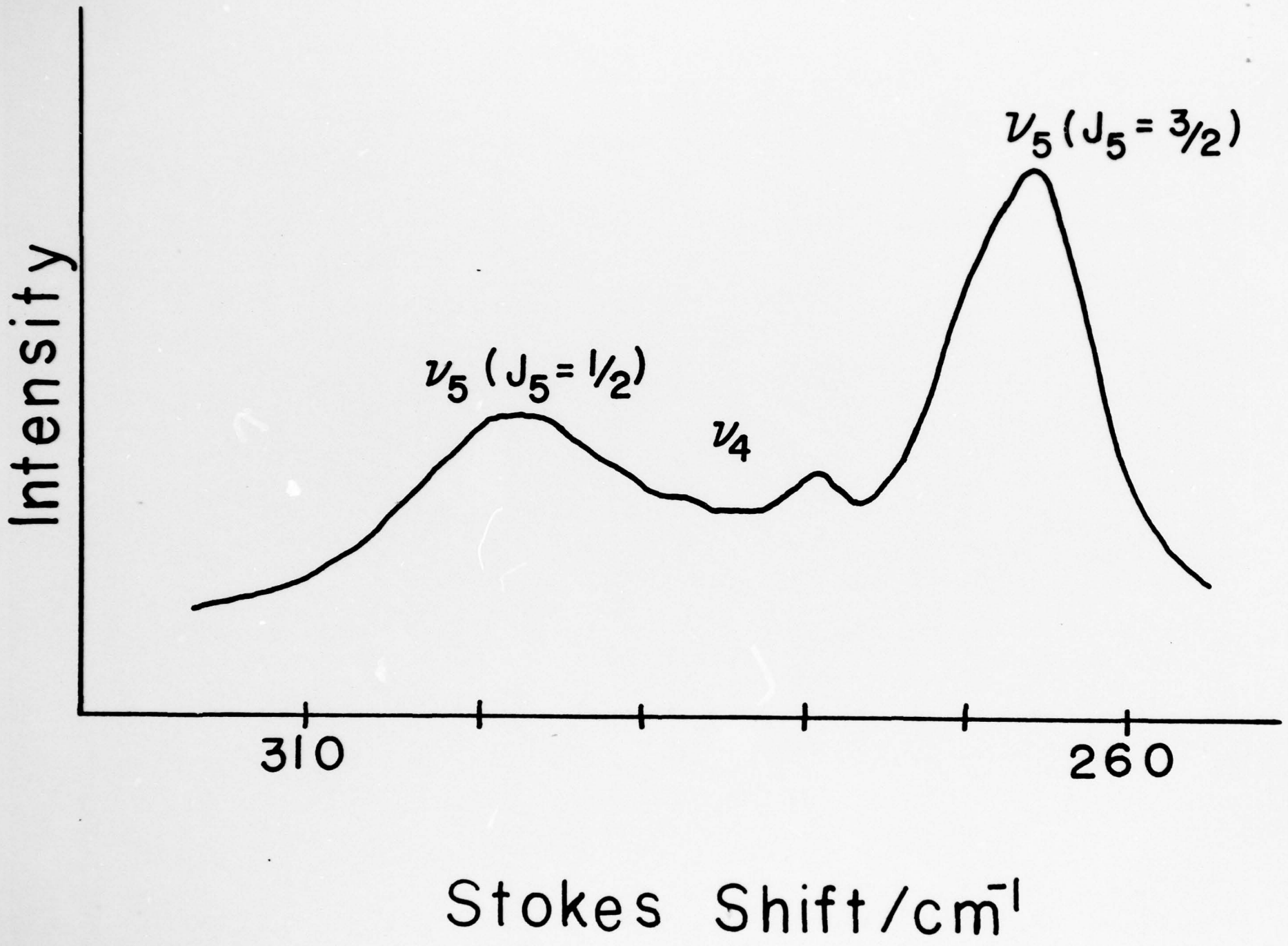
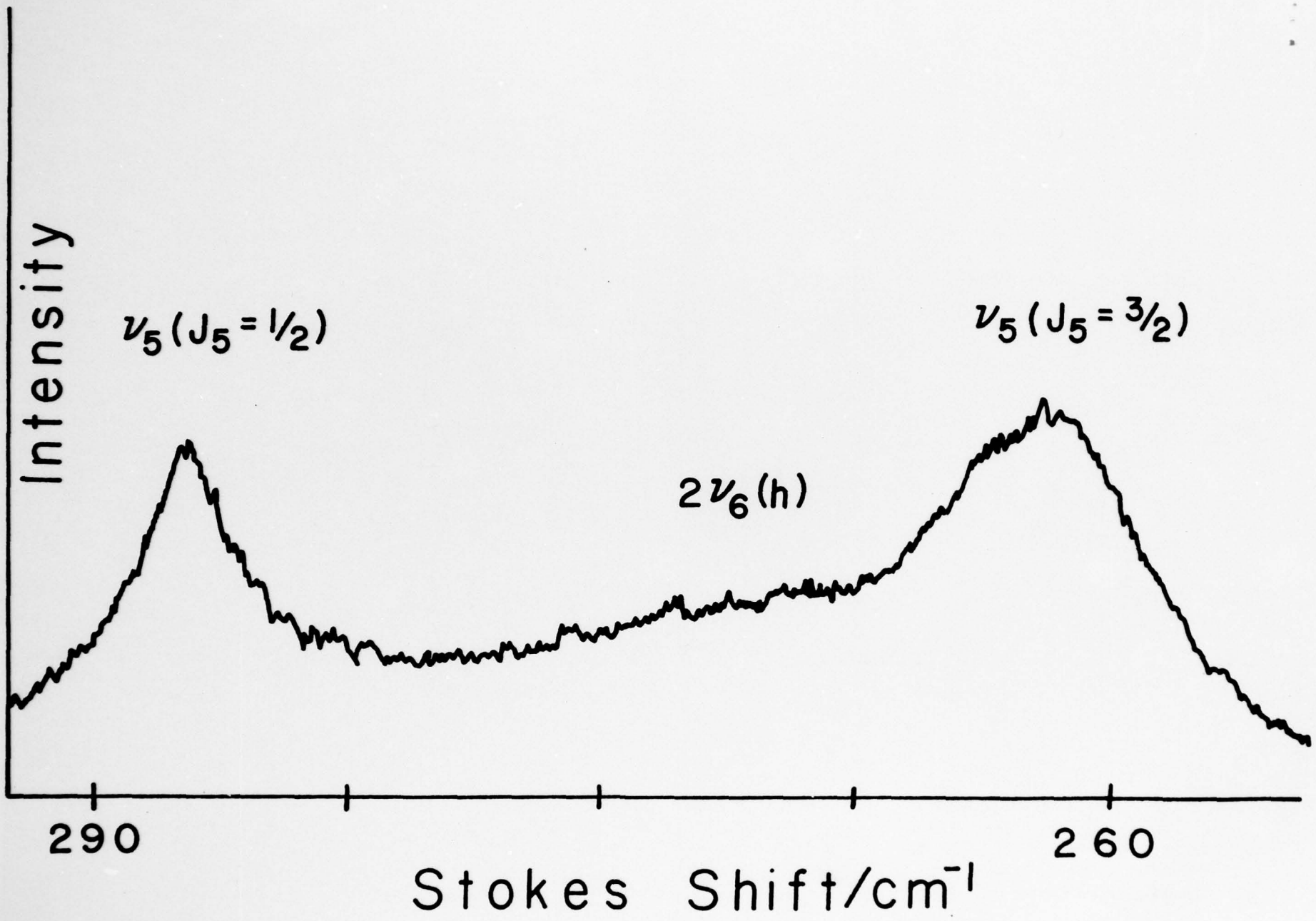

Intensity
ν_5 ($J_5 = 3/2$)
ν_5 ($J_5 = 1/2$)
ν_4
310
260
Stokes Shift/cm^{-1}

Figure 8.

The Raman spectrum of the Jahn-Teller split ν_5 (t_{2g}) vibration of IrF_6 in a mixed crystal (5% IrF_6/MoF_6) at 77 K. $2\nu_6$ (h) is a peak due to the host, MoF_6.


Intensity
$\nu_5(J_5 = 1/2)$
$\nu_5(J_5 = 3/2)$
$2\nu_6(h)$
290
260
Stokes Shift/cm^{-1}

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