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Studies on the Failure of the First Born Approximation in Electron Diffraction

III. Tellurium Hexafluoride

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Tellurium hexafluoride has been studied by electron diffraction. It is demonstrated that a fairly satisfactory agreement between experimental and theoretical intensities is obtained for either of the two sets of complex scattering amplitudes ($f = |f|\ e^{i\eta}$) available when this study was initiated. The experimental data gave $(\eta_{Te} - \eta_F) = \pi/2$ at $s = 16.2$ Å^{-1}, while the corresponding theoretical values are 17.65 Å^{-1} and 18.5 Å^{-1}. There is no evidence for deviation from O_h symmetry. The bond length is found to be 1.824 Å with a standard deviation of 0.004 Å. The corresponding root-mean-square amplitude of vibration (u) is 0.038_5 Å (with a standard deviation of 0.002 Å) which is in agreement with the value obtained from spectroscopic data.

The investigation of gaseous tellurium hexafluoride by electron diffraction constitutes a part of a study of scattering by molecules where the first Born approximation fails. Results for UF_6[1] and OsO_4[2] have already been published.

The theoretical expression for the modified molecular intensity is [1]

$$I(s) = \text{const}\ \{6g_{TeF/FF}(s)\ \exp\ (-\tfrac{1}{2}\ u_1^2 s^2)\ (\sin\ r_1 s)/r_1 + 12\ \exp\ (-\tfrac{1}{2}\ u_2^2 s^2)\ (\sin\ r_2 s)/r_2 + 3\ \exp\ (-\tfrac{1}{2}\ u_3^2 s^2)\ \sin\ r_3 s)/r_3\} \quad (1)$$

assuming Gaussian distance distributions and a regular octahedral molecule. Here

$$g_{TeF/FF}(s) = \frac{|f_{Te}(s)||f_F(s)|}{|f_F(s)|^2} \cos\ (\eta_{Te}(s) - \eta_F(s)). \quad (2)$$

The same sets of scattering amplitudes have been used as in the previous investigations.[1,2] The values later referred to as the first set of scattering amplitudes, have been calculated at Indiana University Research Computing Center as described by Karle and Bonham.[3] The values referred to as the second

set, have been calculated by Ibers and Hoerni.[4] The latter were adjusted to the applied potential according to the formulas proposed in Ref. 4.

Tellurium hexafluoride has already been studied twice by electron diffraction. In both cases an octahedral structure was reported in spite of the use of the first Born approximation. The bond lengths obtained were 1.82 ± 0.04 Å [5] and 1.84 ± 0.03 Å.[6] Spectroscopic investigations strongly indicate an octahedral structure.[7]

EXPERIMENTAL

The sample of tellurium hexafluoride was obtained from Baker and Adamson, New York. The compound was purified by distillation immediately before the data were recorded. Diffraction photographs were taken at a nozzle temperature of about 25°C, applying an accelerating potential of approximately 35 kV. Three sets of plates were used, taken with the nozzle to plate distances 48.02 cm, 19.32 cm, and 13.17 cm. The corresponding s ranges are approximately 1.5—20.0 Å^{-1}, 5.0—42.0 Å^{-1}, and 15.0—60.0 Å^{-1}. Each set consists of six plates of which four were used. The plates were photometered and corrected in the usual way.[8] An experimental background was subtracted from the intensity obtained from each individual plate. Thus three sets of intensity curves, covering different s ranges, were obtained. The curves were scaled and the mean values used in the overlap regions, giving four intensity curves ranging from s = 1.5 Å^{-1} to s = 60 Å^{-1}.

STRUCTURE ANALYSIS

Fig. 1 (curve A) shows the experimental intensity curve. The corresponding radial distribution (RD) curve — obtained by a Fourier transformation of the experimental intensity — is given in Fig. 2 (curve A). An artificial damping constant k = 0.0009 Å^2, was applied. Obviously the two outer peaks correspond to the two different F···F distances, while the Te—F contribution is a double peak. Approximate values for all the interatomic distances and for

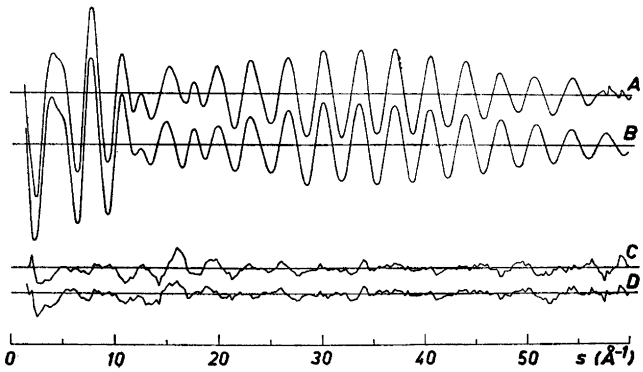

Fig. 1. Experimental (A) and theoretical (B) intensity functions. The theoretical curve was obtained by using eqn. 1 and the first set of scattering amplitudes (see p. 1535). Curve C shows the difference between the curves A and B, while D shows the difference between curve A and an intensity function calculated according to eqn. 1, but with the experimental g function in Fig. 4a.

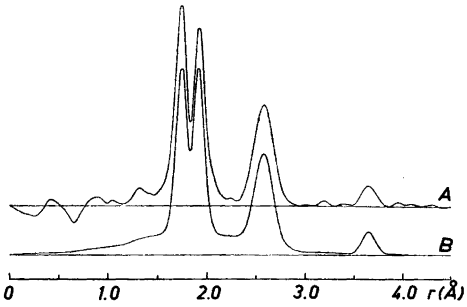

Fig. 2. Experimental (A) and theoretical (B) RD curves calculated with an artificial damping constant $k = 0.0009$ Å^2.

Table 1. Various results for the parameters in TeF_6. Standard deviations are given in parentheses. All values in Å.

	a	b	c	d
r_1	1.826_5	1.826_5 (0.0006)	1.826_7 (0.0006)	1.826_2 (0.0007)
r_2	2.573	2.574 (0.0036)	2.574 (0.0039)	2.575 (0.0043)
r_3	3.643	3.649 (0.0136)	3.647 (0.0144)	3.647 (0.0158)
u_1		0.038_5 (0.0005)	0.039_1 (0.0005)	0.035_5 (0.0007)
u_2	0.091	0.086 (0.0031)	0.086 (0.0033)	0.080 (0.0037)
u_3	0.063	0.065 (0.0111)	0.065 (0.0118)	0.060 (0.0130)

	e	f	g	h
r_1	1.823_6 (0.0014)	1.826_5 (0.0006)	1.826_6 (0.0006)	
r_2	2.574 (0.0036)	2.574 (0.0037)	2.570 (0.0037)	
r_3	3.649 (0.0135)	3.649 (0.0136)	3.650 (0.0134)	
u_1	0.038_5 (0.0005)	0.038_6 (0.0005)	0.038_6 (0.0006)	0.0393
u_2	0.086 (0.0031)	0.083 (0.0039)	0.085 (0.0039)	0.0846
u_3	0.065 (0.0111)	0.063 (0.0113)	0.063 (0.0112)	0.0529

a Results obtained from RD curves.

b-g Results obtained by least-squares refinements:

b Intensity data in the s range 1.5—60.0 Å^{-1}, and the first set of scattering amplitudes (Karle and Bonham) were used. The theoretical intensity was calculated according to eqn. 1. The constants in the weight function [1] were $s_1 = 4.0$ Å^{-1}, $s_2 = 30.0$ Å^{-1}, $W_1 = 0.1$, $W_2 = 0.002$.

c The weight function was changed by using $s_2 = 50$ Å^{-1}. All other conditions as in b.

d The second set of scattering amplitudes was applied (Ibers and Hoerni). All other conditions as in b.

e The theoretical intensity was calculated including $\varkappa_{TeF}$ (*cf.* eqn. (3)) as an additional parameter. All other conditions as in b. Result: $\varkappa_{TeF} = -2.2 \times 10^{-6}$ (0.92×10^{-6}) Å^3.

f α_{TeF} (*cf.* eqn. (3)) was refined as an additional parameter. All other conditions as in b. Result: $\alpha_{TeF} = 1.053$ (0.047).

g The experimental g function (Fig. 4a) was applied and scaled by refining α_{TeF}. The intensity data in the s range 1.5—50.0 Å^{-1} were used. Result: $\alpha_{TeF} = 0.909$ (0.040).

h Spectroscopic u values from Cyvin *et al.*[9]

the F···F amplitudes of vibration are easily obtained from this curve and are shown in Table 1a. The values given for the F···F distances correspond to the position of the maxima of the two outer peaks, and the Te—F distance is taken as the mean of the positions of the two maxima. (This value is used rather than the position of the minimum, since the latter value seems to vary more with the artificial damping constant.) It is seen from Table 1a that the geometrical restrictions for an octahedral arrangement ($r_2 = \sqrt{2}\, r_1$, $r_3 = 2r_1$) are nearly satisfied.

Curve B in Fig. 1 shows the modified theoretical intensity calculated according to eqn. 1 with the first set of scattering amplitudes and the distance- and u values given in Table 1b. Curve C shows the difference between the curves A and B. We notice that the agreement between experimental and theoretical intensities is not quite satisfactory in the region $s = 10.0-20.0$ Å^{-1} (see p. 1542).

Fig. 2 (curve B) shows the theoretical RD curve calculated by a Fourier transformation of curve B in Fig. 1. Some values for the difference between the positions of the two maxima in the double peak ($\Delta\ r$) are given below.

	$k = 0.0$ Å^2	$k = 0.0009$ Å^2
Experimental	0.160 Å	0.177 Å
Theoretical (with first set of f values)	0.153 Å	0.167 Å
» » second » » »	0.148 Å	0.159 Å

The best theoretical values are thus obtained by using the first set of scattering amplitudes.

The distances and u values were refined by least-squares refinement on the intensity data. The program is the same as used previously [1,2] and permits a calculation of the theoretical intensity according to

$$I(s) = \text{const} \sum_{i \neq j} \sum \alpha_{ij} g_{ij|kl}(s) \exp\left(-\tfrac{1}{2} u_{ij}^2 s^2\right) \frac{\sin[(r_{ij} - \varkappa_{ij} s^2)s]}{r_{ij}} \qquad (3)$$

The meaning of the parameters and the applied weight function are discussed in Ref. 1. Table 2 a-d shows the results for the four observed intensity curves with intensity data in the range 1.50—60.0 Å^{-1}. The theoretical intensity was calculated according to eqn. 1. The first set of scattering amplitudes was used, and all the distances were treated as independent parameters. The standard deviations obtained by the least-squares procedure are also given. The mean for each parameter is given in Table 2e. The standard deviations in this column were calculated according to [1]

$$\sigma = \left[\sum_{i=1}^{n} (p_i - \bar{p})^2/(n-1)\right]^{\frac{1}{2}} \qquad (4)$$

The standard deviations obtained in this manner are of course very uncertain since n is only four.

It is worth noting that the standard deviations for r_1 and u_1 obtained by the least-squares refinements (Table 2 a-d) are smaller than the corresponding values in Table 2e. A similar result was found for UF_6.[1]

Table 2. Least-squares results from four observed intensity curves covering the s range 1.5–60.0 Å^{-1}

	a	b	c
r_1	1.829_6 (0.0008)	1.825_8 (0.0007)	1.826_3 (0.0007)
r_2	2.578 (0.0048)	2.577 (0.0044)	2.570 (0.0046)
r_3	3.657 (0.0151)	3.638 (0.0147)	3.656 (0.0192)
u_1	0.037_3 (0.0007)	0.038_6 (0.0006)	0.038_3 (0.0006)
u_2	0.081 (0.0042)	0.086 (0.0037)	0.089 (0.0039)
u_3	0.054 (0.0126)	0.061 (0.0121)	0.073 (0.0157)

	d	e
r_1	1.823_9 (0.0008)	1.826_4 (0.0024)
r_2	2.569 (0.0044)	2.574 (0.0047)
r_3	3.645 (0.0177)	3.649 (0.0091)
u_1	0.039_3 (0.0007)	0.038_4 (0.0008)
u_2	0.086 (0.0038)	0.086 (0.0033)
u_3	0.068 (0.0145)	0.064 (0.0083)

a-d Results obtained from the individual intensity curves using the first set of scattering amplitudes and $s_1 = 4.0$ Å^{-1}, $s_2 = 30.0$ Å^{-1}, $W_1 = 0.100$, $W_2 = 0.002$.
e Mean values and standard deviations (eqn. 4) calculated from the results in the columns a-d.

The four intensity curves were averaged, and a least-squares refinement was carried out using the same conditions as before. The parameters obtained (see Table 1b) are of course practically the same as given in Table 2e, and they are also very close to the results obtained from the RD curve (Table 1a). As observed in the investigation of UF_6[1] the standard deviations are not reduced by a factor of two by averaging four intensity curves.

If we want to compare the standard deviations in Table 1b with the corresponding quantities obtained in the usual way from the four individual intensity curves, we must use $1/\sqrt{4}$ times the values given in parentheses in Table 2e. (These curves are then assumed to be independent.) Keeping this factor of ½ in mind we find that the standard deviations obtained by least-squares refinement on the averaged intensity curve are greater than the corresponding values found from four observations, except for r_1, where the value in Table 1b is too small.

The weight function was considerably changed by adjusting s_2.[1] Table 1c shows the resulting parameters which are practically the same as given in Table 1b, except for u_1, where the shift is greater than the corresponding standard deviation. The same parameter proved to be most sensitive to changes in the weight function in the investigations of UF_6[1] and OsO_4.[2]

The results in the next column (d) were obtained by using the second set of scattering amplitudes (Ibers and Hoerni). The estimate of u_1 is considerably lower than in column b. The higher standard deviations in (d) may indicate

that the first set of scattering amplitudes fits the experimental data somewhat better than the second set. Another indication in the same direction is obtained by comparing the estimates of u_1 in the columns b and d to the corresponding u value obtained by Cyvin *et al.*[9] (column h) from spectroscopic data. (u values for TeF_6 have also been calculated by M. Kimura and K. Kimura.[10] The values reported by these authors are practically the same as those found by Cyvin *et al.*) The agreement is clearly better when scattering amplitudes according to Karle and Bonham are used. Since the potentials used in the calculation of the first set of scattering amplitudes probably are the most accurate ones, this result was perhaps to be expected. The scattering amplitudes according to Karle and Bonham also gave the best agreement between observed and calculated intensities in the investigations of UF_6.[1] However, for OsO_4 the values calculated by Ibers and Hoerni proved to give the best fit.

As in the two previous investigations we find that the inner maximum of the double peak in the RD curve is higher than the outer one. It was shown in the investigation of UF_6[1] that the double peak should be symmetric only if the vibration is harmonic, *i.e.* if there is no distortions of the wave length of the intensity contribution. Least-squares refinements including $\varkappa$ for the bond distance as an additional parameter (eqn. (3)), gave positive $\varkappa$ values for UF_6 and OsO_4. Fourier transformations of the theoretical intensities calculated with $\varkappa$ as a parameter, gave a better agreement between experimental and theoretical RD curves. However, in this case a negative value was obtained by a similar refinement. ($\varkappa_{TeF} = -2.2 \times 10^{-6}$ Å^3 with a standard deviation of 0.92×10^{-6} Å^3). The results are shown in Table 1e. $\varkappa$ is of course particularly sensitive to relative errors in the s scales calculated for plate sets covering different s ranges. Therefore a similar refinement was carried out using the intensity obtained from the shortest camera distance only. Then $\varkappa = 2.2 \times 10^{-6}$ (0.55×10^{-6}) Å^3 was found, which is a reasonable value.

The results in Table 1f were obtained by refining α_{TeF} as an additional parameter (*cf.* eqn. 3). The resulting α value is slightly larger than unity. A similar result was obtained for α_{UF}, while α_{OsO} was found to be less than unity.

As in the previous investigations we have determined experimental values for the function given in eqn. 2 (See the next section.) A least-squares refinement applying this experimental g function gave the results shown in Table 1g. The function was scaled by refining α_{TeF}.

SCATTERING AMPLITUDES

The experimental function $g_{TeF/FF}^{exp}(s)$ was obtained in the same way as described for OsO_4.[2] The double peak in the RD curve, calculated with zero damping, was Fourier transformed giving the Te—F contribution to the experimental intensity. The absolute values of this Te—F intensity were plotted below $s \approx 16$ Å^{-1}, and for larger s all function values were made negative and plotted (Fig. 3). By multiplying the envelope of this curve by $\exp(\frac{1}{2}u_1^2s^2)$ we obtain an experimental function proportional to $g(s)$.

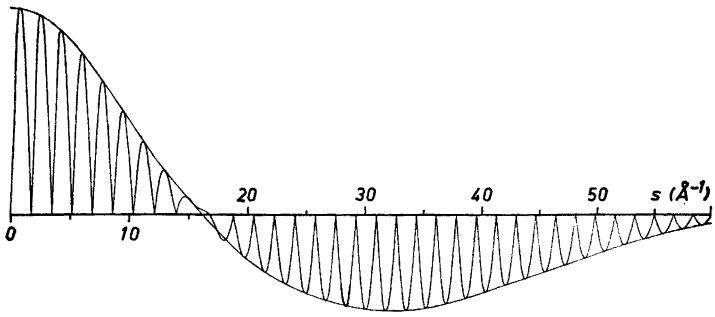

Fig. 3. The Te—F contribution to the experimental intensity plotted after making all values positive below $s \approx 16$ Å^{-1} and all values negative for higher s values.

This experimental function depends on the value chosen for u_1. Fig. 4a (A) shows the result when u_1 is taken from Table 1b. The theoretical g function according to Karle and Bonham is shown for comparison (B). The experimental function in Fig. 4a was scaled by requiring that the minimum value should be equal to the theoretical minimum, though a somewhat smaller scale was found by least-squares refinement (*cf.* Table 1g). Similarly Fig. 4b (A) shows the function obtained by using u_1 from Table 1d, compared to the theoretical function according to Ibers and Hoerni (B). (Since the observed intensity has been multiplied by $s/|f_F|^2$ one has a slight difference in the intensity curves for the two sets of scattering amplitudes. It was therefore necessary to carry out the Fourier transformations separately in the two cases.) The experimental values are very uncertain for high s values and the curves have therefore been plotted to $s = 55$ Å^{-1} only. The point where the intensity vanishes (the beat-out) is at $s = 16.2$ Å^{-1} on the experimental curves compared to 17.65 Å^{-1} (Fig. 4a) and 18.5 Å^{-1} (Fig. 4 b) on the theoretical ones. It is possible that the neglect of multiple scattering causes the difference between the experimental and theoretical beat-out values. A rough calculation based on the formulas given by Bonham [11],* suggests that a correction for the multiple

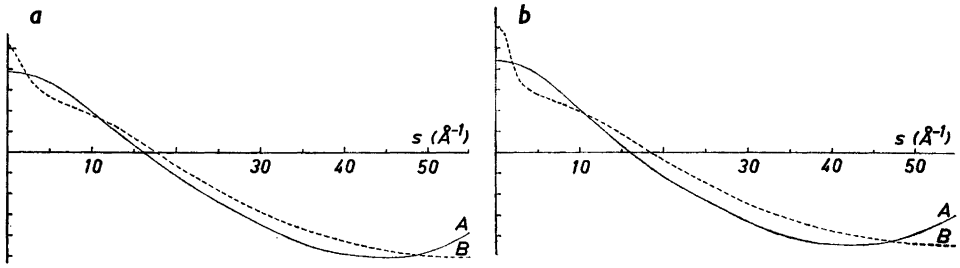

Fig. 4a, b. Comparison of experimental (A) and theoretical (B) curves for $g(s)$. The first set of scattering amplitudes was used in a, the second set was used in b.

* The functions denoted by $\mathbf{h}_n(z)$ in Ref. 11 are not the spherical Struve functions as stated, but some related functions.

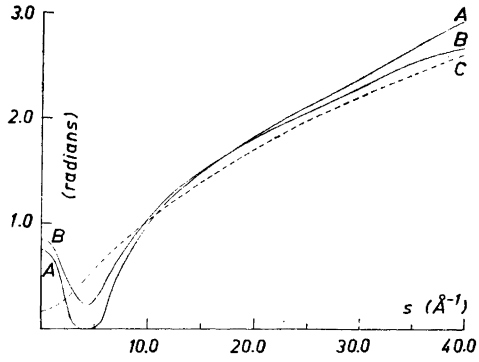

Fig. 5. Experimental (A, B) and theoretical (C) $\Delta\eta_{TeF}(s)$ functions. The curves A and B were obtained using the experimental $g(s)$ function shown in Fig. 4a. The difference between A and B originates from a different scaling of $g_{TeF}(s)$. (See text). Curve C shows $\Delta\eta_{TeF}(s)$ according to the first set of scattering amplitudes.

scattering should shift the theoretical beat-out towards lower s values. For UF_6[1] the experimental beat-out was found at a slightly smaller s value than obtained by theoretical η values while the opposite result was obtained for OsO_4.[2] We feel that more experimental data are needed before this point is discussed more closely.

As in the previous investigations [1,2] a "semi-experimental" $\Delta\eta$ function was obtained. The experimental g function was divided by the theoretical values for $|f_{Te}|/|f_F|$, and the resulting function scaled by requiring exactly (-1) as the minimum value. However, this procedure is more uncertain in this case than in the prevoius investigations, since $\Delta\eta_{TeF}(s)$ reaches the value π at a higher s value than $\Delta\eta_{UF}(s)$ or $\Delta\eta_{OsO}(s)$, and the uncertainty in the experimental g function increases with s. The result using the first set of scattering amplitude is shown in Fig. 5. The dashed curve (C) shows the theoretical $\Delta\eta$ function. Curve A represents the values obtained as described above. Curve B was obtained by normalizing the experimental g function according to the result obtained by least-squares refinement (Table 1f). This procedure may give a better estimate for $\Delta\eta(s)$ for most s values, though it does not work for high s values.

DISCUSSION AND CONCLUSIONS

It was shown in the previous sections that a fairly satisfactory agreement between experimental and theoretical intensity values was obtained by using complex scattering amplitudes. However, the difference curve C in Fig. 1 shows some maxima and minima that require a further discussion. The first minimum ($s < 5$ Å^{-1}) could probably be removed by changing the background, while the maxima and the minima between $s = 10.0$ Å^{-1} and $s = 20.0$ Å^{-1} did not disappear for any reasonable background. Curve D in Fig. 1 shows the difference between the observed intensity (Fig. 1, curve A) and an intensity curve calculated according to eqn. 1, but with the experimental g function (see Fig. 4a). This difference curve is clearly better than curve C in the critical region ($s = 10.0$ Å^{-1}—20.0 Å^{-1}), though the maxima and minima have not quite disappeared.

Table 3. Results obtained by least-squares refinements on intensity data obtained separately from each camera distance.

	a	b	c	d
Nozzle to plate distance (cm)	48.02	48.02	19.32	13.17
Scattering amplitudes	Theoretical (Set I)	Experimental (Fig. 4a)	Theoretical (Set I)	Theoretical (Set I)
s_{min} (Å^{-1})	1.5	1.5	6.0	16.0
s_{max} (Å^{-1})	19.0	19.0	42.0	60.0
s_1 (Å^{-1})	6.0	6.0	8.5	18.5
s_2 (Å^{-1})	12.0	12.0	25.0	30.0
W_1	0.100	0.100	0.100	0.100
W_2	0.020	0.020	0.003	0.002
r_1 (Å)	1.816_4 (0.0024)	1.817_4 (0.0021)	1.823_1 (0.0008)	1.828_4 (0.0003)
r_2 (Å)	2.574 (0.0031)	2.572 (0.0027)	2.570 (0.0047)	2.603 (0.0064)
r_3 (Å)	3.641 (0.0150)	3.639 (0.0132)	3.637 (0.0181)	3.649 (0.0092)
u_1 (Å)	0.075_2 (0.0055)	0.041_2 (0.0093)	0.036_2 (0.0010)	0.041_6 (0.0006)
u_2 (Å)	0.087 (0.0048)	0.082 (0.0045)	0.084 (0.0041)	0.098 (0.0034)
u_3 (Å)	0.073 (0.0171)	0.069 (0.0157)	0.065 (0.0148)	0.061 (0.0062)

As already mentioned, the difference in the heights of the maxima in the double peak in Fig. 2 (curve A) cannot be ascribed to anharmonicity. To investigate this further least-squares refinements were carried out on the intensities obtained from each set of plates. The results are given in Table 3. Two refinements were performed using the long distance plates only. The results in Table 3a were obtained with theoretical values for the function $g_{TeF/FF}(s)$, while the results in column b were found by using the experimental g function (Fig. 4a). The large difference in the estimate of u_1 should be noticed. However, the shift in r_1 is small. The columns c and d show the results obtained using the plates taken with the intermediate and short camera distances. The r values in the last column are systematically higher than the corresponding values in the columns b and c. The reason may be a small error in one or more of the calculated s scales, caused by an error in the wave length or the nozzle to plate distance. The difference between the estimates of r_1 given in the columns b and c is perhaps not real. However, a refinement using the inner part of the intensity data obtained from the plates taken with the intermediate camera distance (s range 6.0 $Å^{-1}$—21.0 $Å^{-1}$), gave also a short r_1 value. Furthermore, preliminary results for MoF_6 and WF_6 indicate a similar effect. It is possible that a distortion of the wave length of the Te—F contribution is caused by multiple scattering or other effects neglected in the theory. Our present calculation of the multiple scattering is not sufficiently accurate to give the effect on the wave length. By a Fourier transformation of an intensity function — calculated according to eqn. 1, but with r_1 as a function of s corresponding to the results in Table 3 — the agreement between theoretical and experimental RD curves was improved.

If the distance values in Table 1b are corrected according to [12] $r_g = r_g(1) + u^2/r$, the values in Table 4a are obtained. Table 4b shows the F···F distances calculated from the bond length assuming a rigid, octahedral molecule, and column c gives the shrinkage calculated by Cyvin *et al.*[9] By subtracting the shrinkage from the values in Table 4b the values in column d are obtained. The agreement between the values in column a and d is seen to be quite good, though r_2 is somewhat higher in column d. This is in

Table 4. Comparison of F···F distances obtained by least-squares refinement (as independent parameters), and the values calculated from the bond length.

	a	b	c	d
Te—F	1.8273			
F···F (1)	2.577	2.584	0.001	2.583
F···F (2)	3.650	3.655	0.005	3.650

a r_g distances calculated from the parameters in Table 1b.
b F···F distances calculated from the bond length assuming a rigid octahedral molecule.
c Shrinkage calculated by Cyvin *et al.*
d F···F distances calculated from the bond length and corrected for shrinkage.

accordance with the s scale difference already mentioned. Because of the possibility of an s scale error a weighted average of the values given in Table 3b, c, and d may give a better estimate of r_1 than the value given in Table 1b. However, it is very difficult to estimate the weights. The standard deviation of r_1 given in Table 3b is much larger than the corresponding values in the columns c and d. The influence on the determination of r_1 of errors in the applied scattering amplitudes and of effects neglected in the theory, is also greatest when the long distance plates are used. The results in column d are on the other hand most sensitive to errors in the nozzle to plate distance.

The best value for the bond length seems to be 1.824 Å (r_g). The standard deviation given in Table 1b for this distance is obviously too low. If the variation shown in Table 3 is taken into account 0.004 Å seems to be a more reasonable value. The standard deviation for u_1 must also be considerably higher than given in Table 1b since this parameter is particularly influenced by errors in the scattering amplitudes and in the applied blackness correction.[2] A reasonable estimate for u_1 seems to be 0.038_5 (0.002) Å.

In agreement with spectroscopic investigations [7] we may conclude that there is no evidence for deviation from O_h symmetry. The agreement between the bond lengths given in Table 4a and d, and the excellent agreement between the u values obtained from spectroscopic data and our results (see Table 1), are strong indications for the octahedral model.

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REFERENCES

1. Seip, H. M. *Acta Chem. Scand.* **19** (1965) 1955.
2. Seip, H. M. and Stølevik, R. *Acta Chem. Scand.* **20** (1966) 385.
3. Karle, J. and Bonham, R. A. *J. Chem. Phys.* **40** (1964) 1396.
4. Ibers, J. A. and Hoerni, J. A. *Acta Cryst.* **7** (1954) 405.
5. Braune, H. and Knoke, S. *Z. physik. Chem. Abt. B.* **21** (1933) 297.
6. Brockway, L. O. and Pauling, L. *Proc. Natl. Acad. Sci. U. S.* **19** (1933) 68.
7. Gaunt, J. *Trans. Faraday Soc.* **49** (1953) 1122.
8. Bastiansen, O. and Skancke, P. N. *Advan. Chem. Phys.* **3** (1960) 323.
9. Meisingseth, E., Brunvoll, J. and Cyvin, S. J. *Kgl. Norske Videnskab. Selskabs, Skrifter* **1964** No. 7.
10. Kimura, M. and Kimura, K. *J. Mol. Spectry.* **11** (1963) 368.
11. Bonham, R. A. *J. Chem. Phys.* **43** (1965) 1103.
12. Bartell, L. S. *J. Chem. Phys.* **23** (1955) 1219.

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