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Crystal Structure of $KTeO(OH)_5 \cdot H_2O$[1]

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Received August 19, 1963

The structure of $KTeO(OH)_5 \cdot H_2O$ crystallizing in the monoclinic space group $P2_1/c$ with 4 molecules in a unit cell with parameters $a = 8.26$, $b = 6.20$, $c = 12.83$ Å., and $\beta = 108°$ has been solved by Patterson and Fourier methods and refined by least-squares technique. The interesting results of this study, which is probably the first detailed study of a tellurate, are: (1) the octahedral oxygen environment of the Te atom, (2) the geometry of this environment, and (3) the 12-fold oxygen coordination of the K atom. The correct chemical composition, namely, $KTeO(OH)_5 \cdot H_2O$, not known in the beginning, was arrived at through the X-ray analysis and is consistent with the subsequent chemical analyses.

Preliminary Studies

The study of potassium tellurate was undertaken to ascertain the shape and size of the tellurate ion. It is surprising that not much information is available in the literature about the crystallography of tellurates. The earliest study was the incomplete work of Pauling[2] on telluric acid. Patry[3] concluded from powder photographs taken of the anhydrous sulfate, selenate, and tellurate of potassium that the three compounds are isomorphous and are orthorhombic. Crystals belonging only to the monoclinic system could be obtained by us whether the crystals were grown from aqueous solutions of either K_2TeO_4 or $K_2TeO_4 \cdot 2H_2O$. We concluded that our substance was different from either the anhydrous tellurate (Patry[3]) or the pentahydrate reported by Gröth.[4] (See Table I for our goniometric data.) X-Ray reflections of type (hkl), $k = 0$ to 4, and $(0kl)$ were photographed at room temperature with Weissenberg and precession cameras employing Mo $K\alpha$ radiation and a needle-shaped crystal of size less than $0.2 \times 0.2 \times 1.0$ mm. The unit cell dimensions are: $a = 8.26 \pm 0.04$, $b = 6.20 \pm 0.03$, $c = 12.83 \pm 0.05$ Å., and $\beta = 108° \pm 20'$. The systematic absences are $(0k0)$, $k = 2n + 1$, and $(h0l)$, $l = 2n + 1$, where n is an integer; the space group is probably C^5_{2h}, *i.e.*, $P2_1/c$. The density is high and could not be measured accurately but is in the range 3.0 to 3.50. The values calculated on the assumptions that there are four molecules of $K_2TeO_4 \cdot 2H_2O$ or $KTeO(OH)_5 \cdot H_2O$ in the unit cell are 3.40 and 3.04. The two-dimensional $(h0l)$ and $(0kl)$ Fourier syntheses, calculated on the basis of the signs from the Te atom located from the two-dimensional Patterson functions, indicated that the substance under study was a monopotassium tellurate and not the dipotassium salt.

Three-Dimensional X-Ray Analysis

In order to unambiguously determine the chemical composition and ascertain the geometry of the tellurate ion, a complete three-dimensional analysis was undertaken. The intensities of 1700 (hkl) reflections were estimated visually, corrected for Lorentz and polarization factors, and reduced to values of $|F_o|^2$ on a common scale through the use of the $(0kl)$ data. No corrections were made for absorption effects, the linear absorption coefficient μ for Mo $K\alpha$ radiation being 18.3 cm.$^{-1}$.

TABLE I

GONIOMETRIC DATA OF $KTeO(OH)_5 \cdot H_2O$

(100) ∧ (001)	71° 50′ ± 10′
(100) ∧ (110)	57° 35′ ± 10′
(100) ∧ (10$\bar{2}$)	64° ± 30′

The three positional parameters of the Te atom were derived from a Patterson synthesis and were refined in two cycles of least-squares calculation. The conventional crystallographic R factor was 0.28. Next a difference Fourier synthesis with coefficients $S(\text{Te})(|F_o| - |F_{Te}|)$ was calculated where $|F_o|$, S(Te), and $|F_{Te}|$ are the observed structure amplitude, the calculated sign, and the numerical magnitude of the amplitude of scattering from the Te atom. In this difference Fourier were one peak with maximum electron density 38 e/Å.^{3} and seven peaks with maximum electron density in the range 10 to 16 e/Å.^{3}. There were, of course, some weaker peaks with electron density less than 5 e/Å.^{3}. The strongest peak was taken to be that of the potassium atom. Six out of seven remaining peaks were taken to be those of the tellurate oxygen atoms because the centers of these six peaks were at the vertices of an octahedron and were at distances of about 1.9 Å. from the Te atom. The seventh peak was identified to be due to a water of hydration after careful consideration of thermal parameters and peak heights in subsequent cycles of least-squares refinements and Fourier syntheses. The three final cycles of refinements were carried out on the assumption that the asymmetric unit contains one Te, one K, and seven O. These refinements incorporated the effects of anomalous dispersion from the Te and K atoms. For Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å.), $\Delta f''$, the imaginary component of the scattering factor, has the value 2.2 for the Te and about 0.3 for the K atom. The effect of $\Delta f''$ in a case where a center of symmetry is present is, as pointed out by Templeton,[5] to make the

(1) Research performed under the auspices of the U. S. Atomic Energy Commission.
(2) L. Pauling, *Z. Krist.*, **63**, 502 (1926).
(3) M. Patry, *Compt. rend.*, **202**, 1516 (1936).
(4) P. Gröth, *Chemische Krist.*, **2**, 292 (1908).
(5) D. H. Templeton, *Acta Cryst.*, **8**, 842 (1955).

TABLE II

PARAMETERS AND ERRORS IN $KTeO(OH)_5 \cdot H_2O$

No.	Atom	X	Y	Z	B, Å.²	ΔX	ΔY	ΔZ	ΔB, Å.²
1	Te	0.2396	0.2093	0.3442	1.07	0.0001	0.0003	0.0001	0.02
2	K	0.4855	0.1740	0.1489	1.96	0.0005	0.0011	0.0003	0.06
3	O_1	0.2219	0.3912	0.2206	1.51	0.0013	0.0032	0.0009	0.16
4	O_2	0.4770	0.2232	0.3626	1.55	0.0014	0.0032	0.0009	0.16
5	O_3	0.0122	0.1985	0.3302	1.82	0.0015	0.0034	0.0010	0.18
6	O_4	0.2827	0.4590	0.4418	2.09	0.0016	0.0035	0.0011	0.21
7	O_5	0.3015	0.0312	0.4750	1.84	0.0015	0.0033	0.0010	0.19
8	O_6	0.2420	−0.0366	0.2536	2.46	0.0018	0.0039	0.0012	0.24
9	O_7	0.9520	0.2316	0.0298	2.14	0.0017	0.0034	0.0011	0.20

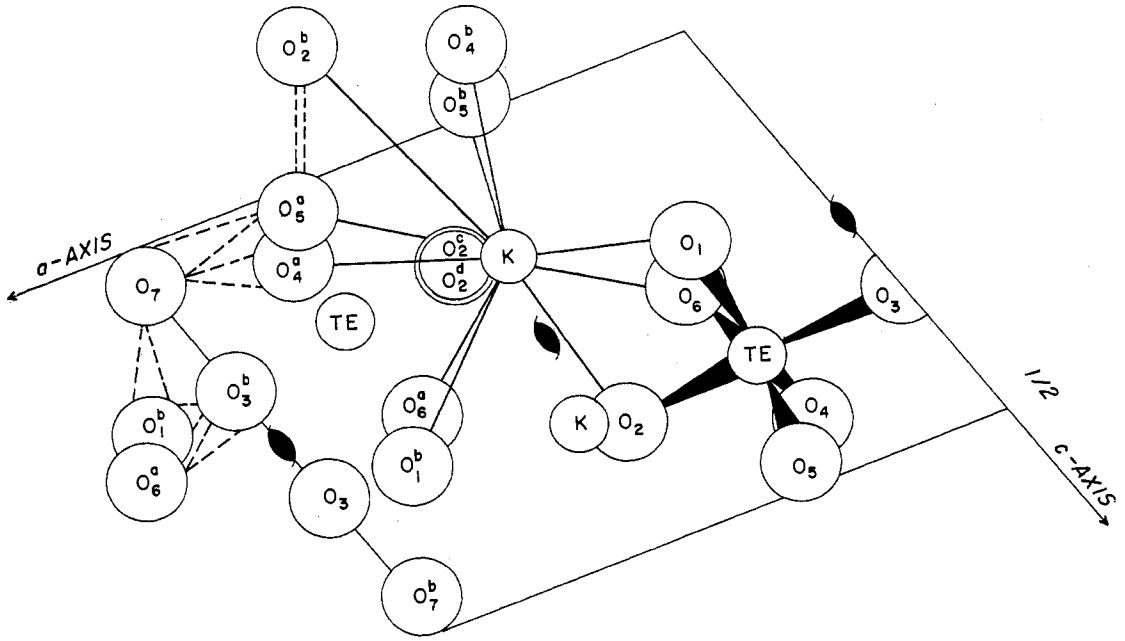

Fig. 1.—Projection of structure of $KTeO(OH)_5H_2O$ down b-axis.

structure factors[6] complex. The resultant correction terms to the derivatives are taken care of by Ibers' modified version of the Busing–Levy least-squares program for the IBM 7090 computer. The atomic scattering factors used were those from the International Tables.[6] An observed amplitude of value less than 18.0 was given a constant weight while an observed amplitude of value $|F_o|$ greater than 18.0 was given a weight inversely proportional to $|F_o|$. The atoms were assumed to have individual isotropic temperature factors. The final parameters, with errors, are given in Table II, and the list of structure factors on an absolute scale is given in Table III. The final value of the R factor is 11%.

Discussion of the Structure

The chief structural feature is the octahedral oxygen environment of the Te atom (Fig. 1). There are six Te–O distances, five of which are distributed about an average of 1.926 Å. with a maximum deviation of 0.026 Å., while the sixth one, namely Te–O_3, is 1.83 Å. The estimated standard deviations are between 0.01 and 0.02 Å. so that Δ, the difference of 0.1 Å. between the distance 1.83 Å. and others, is probably significant. This, together with the distribution of O–O distances, is probably indicative of the chemical formula $K[TeO(OH)_5]H_2O$.[7] The oxide oxygen atom is then plausibly taken to be O_3 so that the Te–O distance is around 1.83 Å. and the Te–OH distance is around 1.93 Å. These

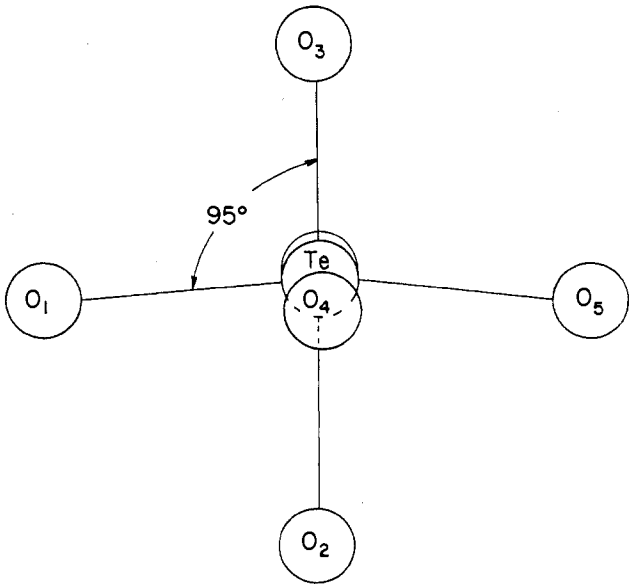

Fig. 2.—The $TeO(OH)_5$ group.

(6) International Tables for X-Ray Crystallography, Vol. 3, 1962, p. 215.

(7) The author is grateful to the referees and the Assistant Editor, Dr. Robert C. Taylor, for suggesting this.

Table III
Observed and Calculated Amplitudes

H	L	FØB	FCAL
*****	K=0	****	
-0	4	55	61
-0	6	72	88
-0	8	12	12
-0	10	75	72
-0	12	32	29
-0	14	22	20
-0	16	38	34
-0	18	7	5
-0	20	16	14
-0	22	12	13
-0	26	9	8
-1	4	55	65
-1	6	16	13
-1	8	80	91
-1	10	35	30
-1	12	33	32
-1	18	26	25
-1	20	16	16
-1	22	10	9
-1	24	16	14
-2	4	10	13
-2	6	9	12
-2	8	10	9
-2	10	42	41
-2	12	27	23
-2	14	14	15
-2	16	24	24
-2	18	8	8
-2	20	10	13
-2	22	11	13
-3	0	30	26
-3	4	3	2
-3	6	45	48
-3	8	51	49
-3	10	2	3
-3	12	39	40
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-3	22	10	11
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-4	4	57	80
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-5	8	56	64
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-5	12	34	37
-5	14	37	31
-5	16	5	7
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-5	20	12	11
-5	22	10	10
-5	24	14	14
-6	0	41	40
-6	2	2	2
-6	4	40	38
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-6	8	29	28
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3	20	9	15
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5	8	39	37
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5	20	10	8
5	24	6	6
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1	6	36	34
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1	11	6	9
1	12	21	22
1	13	12	12
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1	18	16	15
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1	20	4	7
2	2	41	39
2	3	4	2
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2	5	22	17
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2	7	14	13
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2	23	6	4
2	24	10	2
3	1	9	7
3	2	44	47
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3	4	57	65
3	5	32	29
3	6	14	13
3	7	22	18
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3	9	27	23
3	10	30	28
3	12	21	20
3	13	21	20
3	14	28	28
3	15	11	12
3	17	3	6

distances are to be compared with the I–O distance of 1.93 Å. in the IO_6^{-5} ion in $(NH_4)_2H_3IO_6$[8] and a mean Xe–O distance of 1.875 Å. in the XeO_6^{-4} ion.[8] It is to be noted that two types of I–O distances exist in α-HIO_3, namely two I–O bonds of length 1.80 Å. and one I–O (probably I–OH) bond of length 1.89 Å., with a difference $\Delta = 0.09$ Å.[8]

The angles in the octahedron average to 90° with a maximum deviation of 7°, whereas the estimated standard deviations are only 1 to 2°. The octahedron is thus somewhat distorted, and the ligands have an approximate C_{4v} symmetry (Fig. 2), with the 4-fold axis passing through the presumed oxide atom O_3, the Te atom, and the hydroxyl O_2H, the angle O_2TeO_3 deviating from 180° by only 1°. The angles between the oxygen atoms in the basal plane and the central Te

(8) W. C. Hamilton, J. A. Ibers, and D. R. Mackenzie, *Science*, **141**, 532 (1963).

TABLE III (*Continued*)

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4	5	47	43
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4	7	15	16
4	8	3	5
4	10	30	31
4	11	23	21
4	12	24	23
4	14	8	8
4	15	10	11
4	16	20	20
4	17	3	7
4	18	4	6
4	19	4	2
4	20	8	7
5	1	18	18
5	2	43	48
5	3	28	25
5	4	35	29
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5	6	3	3
5	7	8	9
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5	12	8	9
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11	4	20	19
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11	9	10	10
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0	13	17	19
0	14	18	20

H	L	FØB	FCAL
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-9	6	17	18
-9	7	24	23
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-9	9	10	11

H	L	FØB	FCAL
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-9	12	18	19
-9	13	19	19
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-9	17	14	14
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-12	5	14	16
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1	12	9	11
1	13	16	18
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1	15	5	6
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2	3	8	6
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2	7	21	19
2	9	7	6
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6	2	11	12
6	3	15	12
6	5	10	11
6	6	11	14
6	7	23	24
6	10	10	12

H	L	FØB	FCAL
6	11	15	16
6	12	9	10
6	13	10	11
6	17	11	12
7	2	8	10
7	3	28	31
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10	2	8	10
10	3	11	13
10	5	4	8
10	6	6	8
10	7	15	17
10	11	9	9
10	13	11	9

atom average to 89.5°, deviating from the ideal value of 90° by 0.5°, whereas the four angles between the oxide oxygen O_3, the central atom Te, and the four oxygens in the basal plane average to 95° with a maximum deviation of 2°; thus the oxygen atoms in the basal plane buckle toward the presumed hydroxyl O_2H.

There are twelve K–O contacts (Table V) distributed about the average of 3.1 Å. with a maximum deviation of 0.6 Å. The shortest K–O contact is 2.8 Å. The geometry of the K–O coordination (Fig. 1) is best described as follows: eight oxygen atoms approximately occupy the corners of a cube centered on the K atom, with one face parallel to the (100) plane and the other face parallel to (010). The remaining four occupy the corners of a square roughly bisecting the cube and parallel to the (100) plane, with the diagonal of the square passing through the K atom and parallel to the *b*-axis.

TABLE IV

BOND LENGTHS AND ANGLES AROUND THE Te ATOM[a,b]

Atom 1	Atom 2		Distance, Å.
Te	O_1		1.92
Te	O_2		1.90
Te	O_3		1.83
Te	O_4		1.95
Te	O_5		1.94
Te	O_6		1.92
Atom 1	Atom 2	Atom 3	Angle, deg.
O_1	Te	O_2	83
O_1	Te	O_3	97
O_1	Te	O_4	91
O_1	Te	O_5	89
O_2	Te	O_4	85
O_2	Te	O_5	86
O_2	Te	O_6	85
O_3	Te	O_4	94
O_3	Te	O_5	93
O_3	Te	O_6	97
O_4	Te	O_5	87
O_5	Te	O_6	91
O_1	Te	O_5	170
O_2	Te	O_3	179
O_4	Te	O_6	169

[a] The standard deviations in the bond lengths are between 0.01 and 0.02 Å., while those in the bond angles are between 1 and 2°. [b] The numbering of the atoms is the same as in Fig. 1.

TABLE V

POTASSIUM–OXYGEN DISTANCES IN $KTeO(OH)_5 \cdot H_2O$[a,b]

Atom 1	Atom 2	Distance, Å.
K	O_1	2.94
K	O_1^b	3.03
K	O_2	2.78
K	O_2^b	3.71
K	O_2^c	3.43
K	O_2^d	2.82
K	O_4^b	2.79
K	O_4^a	2.86
K	O_5^b	2.92
K	O_5^a	3.50
K	O_6^a	2.85
K	O_6	3.04

[a] The standard deviations are between 0.01 and 0.03 Å. [b] The numbering of the atoms is the same as in Fig. 1.

The three shortest K–Te distances are 3.70, 3.87, and 3.70 Å., with an average of 3.76 Å., the fourth distance being greater than 4 Å.

The intramolecular O–O contacts are of two types. The shorter ones, 12 in number, define the edges of the octahedron. They are distributed between 2.54 and 2.81 Å. with an average of 2.70 Å. The three longer ones define the diagonals of the octahedron and are 3.84, 3.73, and 3.90 Å. with an average of 3.82 Å. There are seven intermolecular O–O contacts between 2.62 and 2.92 Å. and 21 between 3.16 and 4 Å. The shorter seven O–O contacts are probably the OH–O bonds, as there are seven hydrogen atoms available for hydrogen bonding, if the molecule is $KTeO(OH)_5 \cdot H_2O$, as suggested by the Te–O distances. The H-bonding scheme proposed assumes that there are four hydrogen bonds between the tellurate oxygen atoms and water of hydration and three between the tellurate oxygen atoms. O_7 is involved in four bonds, O_3 in three, O_1 and O_5 in two, and O_2 and O_4 and O_6 in one hydrogen bond. The hydrogen-bond angles are nearly tetrahedral (Table VI). The H-bonding scheme presumes the assignment, $O_1H \cdots O_3$; $O_6H \cdots O_3$; $O_7H \cdots O_3$; $O_4H \cdots O_7$; $O_5H \cdots O_7$; $O_2H \cdots O_5$, and $O_7H \cdots O_1$. The above assignment seems to be consistent with the chemical formula $KTeO(OH)_5 \cdot H_2O$.

TABLE VI

HYDROGEN BOND LENGTHS AND ANGLES[a,b]

Atom 1	Atom 2		Distance, Å.
O_1^b	O_3^b		2.65
O_1^b	O_7		2.92
O_2^b	O_5^a		2.79
O_3^b	O_6^a		2.62
O_3^b	O_7		2.79
O_4^a	O_7		2.68
O_5^a	O_7		2.79
Atom 1	Atom 2	Atom 3	Angle, deg.
Te	O_1^b	O_3^b	119
Te	O_1^b	O_7	110
O_7	O_1^b	O_3^b	81
Te	O_2^b	O_5^a	119
Te	O_3^b	O_6^a	133
Te	O_3^b	O_7	112
Te	O_3^b	O_1^b	134
O_1^b	O_3^b	O_6^a	94
O_6^a	O_3^b	O_7	86
O_1^b	O_3^b	O_7	91
Te	O_4^a	O_7	126
Te	O_5^a	O_7	112
O_2^b	O_5^a	O_7	87
O_1^b	O_7	O_3^b	114
O_1^b	O_7	O_4^a	120
O_1^b	O_7	O_5^a	99
O_3^b	O_7	O_4^a	124
O_3^b	O_7	O_5^a	103
O_4^a	O_7	O_5^a	82

[a] The standard deviations in lengths are between 0.01 and 0.03 Å., while those in the bond angles are between 1 and 3°. [b] The numbering of the atoms is the same as in Fig. 1.

Acknowledgment.—The author wishes to acknowledge the use of the local modifications of the Busing–Levy least-squares and function and error programs for the IBM 7090 computer, the Zalkin–Fourier program, the Bauer bond distances, and precession LP factor programs. He is also grateful to Dr. Walter Hamilton and Dr. James Ibers for helpful discussions and assistance in the preparation of the manuscript.