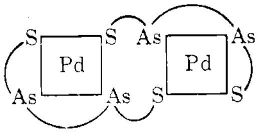
we assign dimeric structure IV to the C_4 complex as well. The complex undergoes S-alkylation[2,8,9] with



IV

methyl iodide but the isolated product did not provide satisfactory and reproducible analyses for the expected product, $Pd(C_4)I_2$. This observation is not surprising in view of the strong tendency toward formation of the species $Pd_2(C_4)I_4$.

Experimental Section

Synthesis of 1,4-Bis(phenyl-*o*-thiomethylphenylarsino)butane.—1,4-Butanebis(phenylarsonic acid) was prepared from phenyldichloroarsine (64 g) and 1,4-dibromobutane (32 g) following a procedure[2,3] analogous to those used for propanebis(phenylarsonic acid) and ethanebis(phenylarsonic acid). The crude material was recrystallized from hot 1:1 ethanol–water; yield, 30–34 g; mp 194–196°. *Anal.* Calcd for $C_{16}H_{20}As_2O_4$: C, 45.09; H, 4.73; As, 35.16; O, 15.02. Found: C, 45.28; H, 4.75; As, 35.30; O (by difference), 14.67.

1,4-Butanebis(phenylchloroarsine) was prepared from butanebis(phenylarsonic acid) by reduction in cold aqueous hydrochloric acid (~4 *M*) with sulfur dioxide gas for 2 hr. The yellow oily material was allowed to stand with the mother liquor for 2 days. The viscous material was then separated, washed with cold 4 *M* HCl, and dried over KOH and Drierite; yield, 28–30 g.

The final product, 1,4-bis(phenyl-*o*-thiomethylphenylarsino)butane was obtained as follows. *o*-Bromothioanisole (14 g) was allowed to react with *n*-butyllithium (1.6 *N*, 45 ml) in ether and then with butanebis(phenylchloroarsine) (15 g in 20 ml of dry tetrahydrofuran).[2] After hydrolysis with dilute HCl the white precipitate was collected and dried. The product was recrystallized from a 1:3 acetone–ethanol mixture; yield, 9–10 g, mp 73–75°. *Anal.* Calcd for $C_{30}H_{32}As_2S_2$: C, 59.40; H, 5.32; As, 24.70; S, 10.57. Found: C, 59.41; H, 5.28; As, 24.80; S, 10.67.

Preparation of Complexes.—A stock solution of $PdCl_4^{2-}$ was prepared as described earlier.[2] In order to prepare $Pd_2(C_4)Cl_4$ the C_4 ligand (0.3 g, 0.0005 mol) was dissolved in hot 1:3 acetone–ethanol and treated with the chloropalladate solution (2.5 ml, 0.0005 mol). The soft, fluffy precipitate was filtered, washed with acetone, and dried. *Anal.* Calcd for $C_{30}H_{32}As_2Cl_4Pd_2S_2$: C, 37.49; H, 3.36; Cl, 14.75. Found: C, 38.05; H, 3.58; Cl, 14.66.

$[Pd(C_4)Cl][B(C_6H_5)_4]$ was obtained from the filtrate from the above preparation by treating it with an ethanol solution of sodium tetraphenylborate. The resulting precipitate was collected, washed with ethanol, and dried. *Anal.* Calcd for $C_{54}H_{52}As_2BPdClS_2$: C, 60.75; H, 4.91; Cl, 3.32. Found: C, 60.53; H, 4.98; Cl, 3.42.

$[Pd(C_4)Br][B(C_6H_5)_4]$ was isolated from the filtrate from the preparation of $Pd_2(C_4)Cl_4$ by first treating with lithium bromide and then adding an ethanol solution of sodium tetraphenylborate. The precipitate was washed with ethanol and dried. *Anal.* Calcd for $C_{54}H_{52}As_2BBrPdS_2$: C, 58.32; H, 4.71; Br, 7.19. Found: C, 58.58; H, 4.64; Br, 7.35.

To produce $Pd_2(C_4)I_4$, freshly prepared $Pd_2(C_4)Cl_4$ was suspended in dichloromethane and stirred with an ethanol solution of lithium iodide. The resulting dark red solution was filtered and allowed to crystallize. *Anal.* Calcd for $C_{30}H_{32}As_2I_4Pd_2S_2$: C, 27.15; H, 2.43; I, 38.25. Found: C, 27.11; H, 2.37; I, 38.20.

Pd(demethylated C_4) was formed when a solution made up of C_4 ligand (0.6 g, 0.001 mol) in dimethylformamide (30 ml) and chloropalladate stock solution (5 ml, 0.001 mol) was heated under reflux for 6 hr. The crystals which separated were collected, washed with ethanol, and dried. *Anal.* Calcd for $C_{25}H_{26}As_2PdS_2$: C, 49.25; H, 3.84; S, 9.39. Found: C, 49.12; H, 3.88; S, 9.67.

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Crystal Structure of Potassium Pentafluorotellurate($KTeF_5$)[1]

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The crystal structure of potassium pentafluorotellurate, $KTeF_5$, has been determined by a three-dimensional single-crystal X-ray diffractometer study. A full-matrix least-squares refinement using 646 observed reflections resulted in a final weighted R factor of 2.18%. The structure was refined in the orthorhombic space group Pbcm. The cell constants are $a = 4.735\ (1)$ Å, $b = 9.209\ (2)$ Å, and $c = 11.227\ (2)$ Å. With four molecules per unit cell, the calculated density is 3.547 g/cm^3; the observed density is 3.55 g/cm^3. The structure contains isolated TeF_5^- ions which approximate a square pyramid but which in fact have only the C_s symmetry required by the space group. The apical Te–F distance is 1.862 (4) Å; the basal Te–F distances are 1.953 (3) and 1.952 (3) Å, respectively.

Introduction

Shortly after we had collected diffractometer data for a crystal of potassium pentafluorotellurate, the crystal structure was reported by Edwards and Mouty,[3] who used film methods and photometric techniques to collect their intensity data. However, since five-coordi-

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[1] This work was performed under the auspices of the U. S. Atomic Energy Commission.

[2] Los Alamos Scientific Laboratory, University of California, postdoctoral appointee.

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nate complexes are of considerable interest[4-13] and since no very accurate X-ray determination of a square-pyramidal anion containing a lone pair of electrons has been reported, we have completed refinement of our data and report the results here.

Experimental Section

The crystals of $KTeF_5$ used in this X-ray investigation were prepared by slow evaporation of a 2:1 (molar) mixture of potassium fluoride and tellurium tetrafluoride in 42% aqueous hydrofluoric acid. The large translucent crystals so obtained were easily cleaved to a size suitable for X-ray examination.

The crystal used in this study was approximately a rectangular parallelepiped of dimensions 0.165 × 0.253 × 0.155 mm in the *a*, *b*, and *c* directions, respectively. Precession photographs (Mo Kα radiation) showed the crystal to be orthorhombic, with systematic absences $0kl$, $k = 2n + 1$, and $h0l$, $l = 2n + 1$, indicating possible space groups Pbcm (D_{2h}^{11}, no. 57) or Pbc2_1, a nonstandard setting of Pca2_1 (C_{2v}^5, no. 21).

After alignment of the crystal on an automated Picker four-circle diffractometer, 12 reflections with 2θ in the range 50–56° were centered using the Mo Kα_1 component of the incident X-ray beam (takeoff angle 2°, 0.70926 Å); these reflections were used in a least-squares refinement of the cell parameters and the calculation of an orientation matrix. The room-temperature unit cell constants and their corresponding standard deviations are $a = 4.735$ (1),[14] $b = 9.209$ (2), and $c = 11.227$ (2) Å. The calculated density of 3.547 (1) g/cm³ for $Z = 4$ is in agreement with the measured density[3] of 3.55 g/cm³.

Intensity data were collected on the Picker diffractometer using Zr-filtered Mo Kα radiation (5° takeoff angle). A θ–2θ scan over a 2° range was used. The count was taken in 0.05° steps, for 2 sec at each step; a background was counted for 20 sec at each end of the scan. Intensities were collected for all hkl and $\bar{h}\bar{k}\bar{l}$ planes for which $2\theta \leq 60°$. Of the 1515 reflections measured, 1266 were observed according to the criterion $I \geq 2.5\sigma(I)$, where I is the intensity after background correction and $\sigma(I)$ is its standard deviation based on counting statistics.[15]

The intensity of a standard reflection, sampled after every 25 reflections, showed a small but significant increase, its final value being approximately 3.5% greater than its original value. The crystal was checked periodically for orientation and centering. Multiplicative correction factors for the data were derived by fitting these measurements as a function of time with a fourth-order polynomial. The order of the polynomial was determined from significance tests based on the sum of the squares of the residuals.[16]

Standard corrections were made for Lorentz and polarization effects. An absorption correction was made by the Busing and Levy method using Burnham's program, modified by Larson, Cromer, and Roof.[17] The linear absorption coefficient for Mo Kα radiation is 71.2 cm^{-1} and the calculated transmission factors varied from 0.194 to 0.357.

Refinement of the Structure

Refinement was carried out by full-matrix least-squares techniques[18] using Edwards and Mouty's structure as a starting model. Unobserved reflections were not included in the refinement. The function minimized was $\Sigma w^2(|F_o| - |F_c^*|)^2$, where w^2 is the weight defined as $1/\sigma^2(F_o)$, and $F_c^* = kF_c/\{1 + gLp[2(1 + \cos^4 2\theta)/(1 + \cos^2 2\theta)^2]F_c^2\}^{1/2}$ in which k is a scale constant, Lp is the Lorentz–polarization factor, g is the extinction coefficient,[19] and F_c is the structure factor calculated in the usual way. Structure factors were calculated using scattering factors for the neutral atoms potassium,[20] fluorine,[20] and tellurium,[21] with appropriate anomalous dispersion corrections[22] for potassium and tellurium.

The structure was refined in the centric space group Pbcm to a weighted R factor, $R = \Sigma w||F_o| - |F_c^*||/\Sigma w|F_o|$, of 2.18% with anisotropic temperature factors for all atoms. Hamilton's significance tests[16] on R', $R' = [\Sigma w^2(F_o - F_c^*)^2/\Sigma w^2 F_o^2]^{1/2}$, reject the hypotheses (1) that extinction is not present, (2) that only the heavy atoms vibrate anisotropically, and (3) that our model is not significantly different from the model of Edwards and Mouty, at better than the 0.005 confidence level. All acentric models tested gave physically unreasonable temperature factors, with no significant improvement in the R' factor. The various hypotheses tested in the centric space group and the residuals obtained are summarized in Table I.

TABLE I

R FACTORS FOR VARIOUS REFINEMENT MODELS IN THE CENTRIC SPACE GROUP

Hypothesis	R (×10²)	R' (×10²)
All atoms anisotropic; secondary extinction correction[a]	2.18	2.51
All atoms anisotropic; no extinction correction	2.70	3.08
Extinction correction; only K and Te atoms anisotropic	3.09	3.49
Model of Edwards and Mouty[b]	6.38	6.90

[a] Reference 19. [b] Reference 3; all parameters were held constant during refinement except the scale factor.

The final positional parameters, anisotropic temperature factors, and extinction coefficient g are given in Table II. The final calculated and observed structure factors are given in Table III.

A final difference Fourier map showed residuals centered around the tellurium atom positions; these ranged in value from −0.8 to 0.8 e/Å³. The largest residuals elsewhere were about 0.4 e/Å³ with $\sigma(\rho) = 0.4$ e/Å³.

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TABLE II

FINAL LEAST-SQUARES PARAMETERS FOR $KTeF_5$[a]

	x	y	z	β_{11}[b]	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
K	0.4973 (4)[c]	1/4	0	288 (10)	67 (2)	41 (1)	0	0	0
Te	0.1124 (2)	0.05563 (7)	1/4	312 (4)	86.6 (9)	34.1 (5)	−109 (4)	0	0
F_1	0.3700 (8)	−0.0146 (4)	0.1270 (3)	705 (28)	99 (5)	70 (4)	67 (25)	139 (22)	−56 (8)
F_2	−0.0337 (9)	0.1844 (4)	0.1265 (3)	394 (24)	246 (8)	72 (4)	62 (23)	−86 (21)	123 (11)
F_3	0.3799 (12)	0.2037 (5)	1/4	368 (30)	74 (7)	64 (5)	−78 (28)	0	0

[a] $g = 1.68\ (9) \times 10^{-6}$ (secondary extinction coefficient).[19] [b] Anisotropic temperature factors are given by $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$; β_{ij} values are $\times 10^4$. [c] Reference 14.

TABLE III

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR $KTeF_5$[a]

H= 0 K= 0		
0	0	462
2	146	143
4	135	134
6	103	102
8	182	185
10	68	69
12	66	69
14	34	35
H= 0 K= 2		
0	70	68
1	141	145
2	153	157
3	93	95
4	29	29
5	85	86
6	118	120
7	91	90
8	45	45
9	65	65
10	74	75
11	42	42
12	22	23
13	32	34
14	43	46
15	28	28
H= 0 K= 4		
0	118	117
1	77	79
2	12	11
3	130	133
4	37	39
5	120	121
6	15	15
7	69	70
8	56	56
9	54	55
10	*8	2
11	59	58
12	19	20
13	47	48
14	*10	6
15	31	31
H= 0 K= 6		
0	47	47
1	97	96
2	16	16
3	93	95
4	96	98
5	81	83
6	13	10
7	66	67
8	35	36
9	55	55
10	16	12
11	43	44
12	41	40
13	34	33
14	*10	5
H= 0 K= 8		
0	38	41
1	10	10
2	90	90
3	20	19
4	46	46
5	20	22
6	70	71
7	12	13
8	30	28
9	*9	5
10	52	51
11	11	9
12	22	21
H= 0 K= 10		
0	43	45
1	20	19
2	20	19
3	12	9
4	53	52
5	10	7
6	18	16
7	15	13
8	33	33
9	13	13
10	14	13
H= 0 K= 12		
0	*9	3
1	24	23
2	24	26
3	21	19
4	9	6
5	20	17
6	19	20
H= 1 K= 0		
0	68	70
2	160	157
4	38	33
6	124	121
8	48	45
10	73	75
12	23	24
14	44	46
H= 1 K= 1		
0	45	44
1	123	133
2	52	52
3	81	78
4	60	58
5	71	69
6	46	38
7	82	80
8	24	23
9	62	62
10	21	19
11	38	38
12	18	17
13	30	30
14	13	12
15	26	25
H= 1 K= 2		
0	99	97
1	98	99
2	48	47
3	37	39
4	183	181
5	36	37
6	48	46
7	61	60
8	64	64
9	47	47
10	21	21
11	25	26
12	61	60
13	20	21
14	18	16
15	17	20
H= 1 K= 3		
0	63	62
1	67	66
2	67	68
3	13	12
4	66	66
5	13	14
6	56	54
7	40	41
8	43	41
9	33	32
10	37	36
11	15	14
12	29	29
13	13	11
14	23	22
15	13	13
H= 1 K= 4		
0	63	63
1	96	95
2	51	52
3	115	115
4	7	9
5	99	97
6	37	33
7	66	65
8	27	28
9	55	54
10	24	26
11	50	49
12	10	6
13	38	36
14	15	13
H= 1 K= 5		
0	64	67
1	14	13
2	69	70
3	9	9
4	70	71
5	8	5
6	57	58
7	*7	7
8	44	44
9	*8	1
10	35	35
11	*9	5
12	31	30
13	*9	2
14	25	23
H= 1 K= 6		
0	15	14
1	66	63
2	73	72
3	41	41
4	9	8
5	33	32
6	54	53
7	42	40
8	9	8
9	39	40
10	40	39
11	26	26
12	*9	9
13	18	17
H= 1 K= 7		
0	28	29
1	36	36
2	37	37
3	47	46
4	41	43
5	40	40
6	31	33
7	26	26
8	20	20
9	23	22
10	21	19
11	23	21
12	20	19
13	17	16
H= 1 K= 8		
0	78	77
1	20	21
2	24	21
3	11	12
4	48	49
5	11	11
6	18	18
7	14	15
8	53	52
9	11	12
10	13	13
11	*10	6
12	28	25
H= 1 K= 9		
0	*7	4
1	29	28
2	*7	*0
3	33	34
4	*8	5
5	30	29
6	*8	1
7	22	21
8	*9	2
9	19	19
10	*9	*0
11	19	19
H= 1 K= 10		
0	19	19
1	15	12
2	45	45
3	17	14
4	15	13
5	17	14
6	40	39
7	12	11
8	13	13
9	11	7
H= 1 K= 11		
0	14	12
1	16	17
2	11	9
3	17	18
4	13	6
5	17	16
6	*9	7
7	12	12
8	14	9
H= 1 K= 12		
0	17	17
1	19	16
2	*9	5
3	22	19
4	10	16
5	20	18
H= 2 K= 0		
0	105	109
2	21	21
4	32	31
6	18	19
8	54	52
10	*8	6
12	18	19
14	*9	6
H= 2 K= 1		
0	17	12
1	90	89
2	27	26
3	141	136
4	34	32
5	126	120
6	26	24
7	74	71
8	15	16
9	57	58
10	16	17
11	57	59
12	16	15
13	46	46
14	12	12
15	30	30
H= 2 K= 2		
0	61	[illegible]
1	39	34
2	55	54
3	*5	1
4	9	7
5	*6	5
6	39	38
7	24	23
8	27	26
9	17	15
10	26	25
11	*9	3
12	*9	6
13	9	4
14	13	14
H= 2 K= 3		
0	128	124
1	64	62
2	110	111
3	79	79
4	93	92
5	69	67
6	82	79
7	44	47
8	75	74
9	36	35
10	57	57
11	33	33
12	42	42
13	25	24
14	30	30
H= 2 K= 4		
0	29	27
1	11	4
2	38	37
3	29	27
4	26	26
5	25	24
6	36	37
7	*7	6
8	17	15
9	*8	5
10	15	15
11	12	11
12	11	11
13	11	9
14	14	14
H= 2 K= 5		
0	104	103
1	45	45
2	92	90
3	13	11
4	71	70
5	8	8
6	71	70
7	25	25
8	65	65
9	23	21
10	53	51
11	*9	7
12	37	35
13	*10	4
H= 2 K= 6		
0	33	33
1	16	12
2	12	11
3	*4	1
4	43	43
5	*7	*0
6	8	9
7	8	7
8	24	23
9	16	8
10	*9	6
11	*9	2
12	22	19
13	*10	*0
H= 2 K= 7		
0	45	46
1	51	50
2	46	40
3	36	34
4	34	34
5	34	34
6	30	31
7	39	39
8	33	31
9	32	31
10	24	24
11	27	20
12	19	19
H= 2 K= 8		
0	*7	3
1	9	5
2	31	30
3	*7	8
4	16	9
5	*8	5
6	27	26
7	16	2
8	*9	2
9	*9	4
10	17	17
11	*10	5
H= 2 K= 9		
0	11	11
1	54	50
2	*8	7
3	41	42
4	*8	1
5	39	39
6	*9	2
7	40	38
8	*9	6
9	33	31
10	*10	5
H= 2 K= 10		
0	16	15
1	13	10
2	12	11
3	11	9
4	18	16
5	10	8
6	11	8
7	*9	8
8	13	10
H= 2 K= 11		
0	25	17
1	23	24
2	21	20
3	23	20
4	23	22
5	23	19
6	23	18
H= 2 K= 12		
0	12	13
1	11	5
2	*9	6
3	*9	5
H= 3 K= 0		
0	40	42
2	15	14
4	98	99
6	13	11
8	36	35
10	11	11
12	41	40
14	*10	6
H= 3 K= 1		
0	29	29
1	90	89
2	39	37
3	79	77
4	50	49
5	68	66
6	34	32
7	60	60
8	20	20
9	52	52
10	16	16
11	38	39
12	18	18
13	27	28
14	14	12
H= 3 K= 2		
0	20	18
1	17	18
2	78	79
3	33	31
4	15	18
5	26	25
6	60	58
7	13	13
8	12	11
9	15	14
10	41	41
11	16	15
12	10	7
13	*10	10
H= 3 K= 3		
0	67	64
1	43	42
2	65	65
3	14	15
4	56	57
5	9	11
6	52	52
7	26	27
8	43	43
9	26	26
10	37	37
11	13	13
12	28	28
13	*10	8
H= 3 K= 4		
0	32	30
1	58	55
2	14	13
3	33	33
4	42	43
5	29	30
6	9	8
7	40	39
8	20	22
9	33	32
10	*9	7
11	19	18
12	20	20
13	14	14
H= 3 K= 5		
0	65	64
1	10	9
2	57	57
3	13	12
4	54	55
5	11	11
6	48	50
7	11	8
8	42	44
9	11	8
10	33	31
11	*9	8
12	27	26
H= 3 K= 6		
0	39	39
1	29	30
2	14	14
3	28	28
4	17	17
5	28	26
6	9	8
7	22	23
8	25	25
9	18	19
10	11	10
11	17	16
12	13	9
H= 3 K= 7		
0	22	21
1	40	40
2	27	27
3	44	43
4	29	28
5	41	41
6	25	25
7	31	32
8	14	15
9	25	24
10	16	14
11	24	22
H= 3 K= 8		
0	*7	3
1	21	23
2	32	32
3	16	17
4	*8	2
5	16	14
6	28	28
7	16	16
8	*9	2
9	14	15
10	25	19
H= 3 K= 9		
0	11	9
1	30	29
2	11	9
3	33	32
4	*9	6
5	31	29
6	11	7
7	22	22
8	*10	5
H= 3 K= 10		
0	32	33
1	*9	3
2	*9	8
3	*9	1
4	26	27
5	*9	1
6	*9	7
7	*9	3
H= 3 K= 11		
0	19	17
1	11	12
2	16	15
3	15	13
4	15	13
H= 4 K= 0		
0	46	53
2	92	97
4	49	49
6	74	75
8	33	35
10	55	54
12	24	23
H= 4 K= 1		
0	*8	2
1	18	16
2	10	11
3	22	19
4	14	14
5	19	20
6	9	11
7	12	14
8	*9	1
9	11	10
10	*9	5
11	16	9
12	*10	6
H= 4 K= 2		
0	83	81
1	39	39
2	22	26
3	47	47
4	48	48
5	40	41
6	18	20
7	29	29
8	53	54
9	26	25
10	14	15
11	25	23
12	24	27
H= 4 K= 3		
0	27	24
1	9	10
2	26	21
3	12	2
4	20	20
5	*8	1
6	15	14
7	*9	7
8	17	16
9	*9	5
10	13	13
11	*9	1
12	12	11
H= 4 K= 4		
0	*7	3
1	52	52
2	32	33
3	42	44
4	16	8
5	46	42
6	27	28
7	41	41
8	*9	*0
9	33	33
10	18	18
11	24	25
H= 4 K= 5		
0	18	16
1	20	17
2	16	16
3	11	12
4	11	11
5	10	8
6	14	13
7	12	10
8	12	11
9	14	12
10	10	9
11	*10	8
H= 4 K= 6		
0	*7	1
1	39	41
2	33	33
3	43	45
4	*8	5
5	40	42
6	29	29
7	32	32
8	*9	1
9	25	25
10	19	19
H= 4 K= 7		
0	*8	4
1	18	17
2	*8	2
3	16	16
4	*8	3
5	15	15
6	9	2
7	13	13
8	*9	3
9	17	11
H= 4 K= 8		
0	39	40
1	12	11
2	19	20
3	13	13
4	43	44
5	12	12
6	17	16
7	11	9
8	29	28
H= 4 K= 9		
0	10	10
1	12	8
2	12	8
3	11	9
4	10	10
5	*9	7
6	10	9
H= 4 K= 10		
0	*9	11
1	11	5
2	27	29
3	*9	4
H= 5 K= 0		
0	43	50
2	21	25
4	54	56
6	17	21
8	34	36
10	14	16
H= 5 K= 1		
0	10	11
1	22	22
2	*9	8
3	15	13
4	*9	1
5	14	13
6	*9	5
7	18	17
8	*9	6
9	12	13
10	*10	5
H= 5 K= 2		
0	18	20
1	24	25
2	44	45
3	25	27
4	17	17
5	22	23
6	41	40
7	18	19
8	13	13
9	15	18
10	27	27
H= 5 K= 3		
0	10	9
1	10	10
2	8	7
3	14	13
4	13	12
5	16	13
6	11	8
7	12	9
8	11	6
9	*9	7
H= 5 K= 4		
0	12	13
1	41	41
2	13	11
3	35	36
4	17	16
5	32	33
6	*9	7
7	33	32
8	*10	10
9	27	26
H= 5 K= 5		
0	10	12
1	11	11
2	16	17
3	*8	5
4	16	16
5	*9	4
6	16	14
7	*9	8
8	11	8
H= 5 K= 6		
0	34	32
1	21	21
2	11	8
3	21	27
4	26	26
5	19	20
6	*9	7
7	17	16
H= 5 K= 7		
0	16	16
1	*9	3
2	13	13
3	10	5
4	13	14
5	*9	3
6	*10	11
H= 5 K= 8		
0	12	13
1	*9	6
2	29	30
3	*9	5
H= 6 K= 0		
0	*9	2
2	23	24
4	*9	5
6	19	19
H= 6 K= 1		
0	12	11
1	23	24
2	*9	5
3	23	23
4	*10	3
5	20	21
6	*10	4
H= 6 K= 2		
0	17	18
1	12	11
2	*9	2
3	13	11
4	12	15
5	12	10
6	*10	2
H= 6 K= 3		
0	18	20
1	11	13
2	22	21
3	17	19
4	19	19
5	16	16
H= 6 K= 4		
0	16	13
1	12	13
2	*9	5
3	11	10
4	12	11
H= 6 K= 5		
0	20	21
1	*9	1
2	19	20
3	*9	2

[a] Column headings are l, F_o, $|F_c{}^*|$; the F_o's have been scaled to $|F_c{}^*|$.

Discussion of the Structure

Table IV lists pertinent interatomic distances and angles from our model and, for comparison, the corresponding values from the model of Edwards and Mouty.[3] The structure contains isolated pentafluorotellurate ions.[23] The TeF_5^- ion is approximately square pyramidal, with the Te and apical F atoms on the crystallographic mirror at $y = 1/4$. The Te atom is displaced 0.378 Å below the plane of the basal fluorines. A view of the ion is shown in Figure 1. Unlike the E–M model in which the TeF_5^- ion has C_{4v} symmetry within experimental error, in our structure the basal plane of the TeF_5^- is lengthened approximately 0.12 Å in a direction perpendicular to the mirror plane, *i.e.*, 2.768 Å (average) for F---F′ *vs.* 2.648 Å for F_1---F_2. This distortion is probably due to the interaction of the lone pair of electrons on an adjacent tellurium atom with the $F_1F_1'F_3$ and $F_2F_2'F_3$ faces of the pyramid,[24] as shown in Figure 2. There is also a distortion of the TeF_5^- ion from C_{2v} symmetry, the Te---F_3 "axis" forming an angle of 86.7 (5)° with the basal fluorine plane. This dis-

TABLE IV

INTERATOMIC DISTANCES (Å) AND ANGLES (DEG) IN $KTeF_5$[a]

Atoms	Present work	E–M model[b]
Te–F_1	1.953 (4)[c]	1.96 (2)
Te–F_2	1.952 (4)	1.96 (2)
Te–F_3	1.862 (4)	1.84 (2)
F_1- - -F_2	2.648 (5)	2.69 (3)
F_1- - -F_3	2.439 (5)	2.41 (2)
F_2- - -F_3	2.408 (6)	2.41 (2)
F_1- - -F_1'	2.761 (7)	2.73 (3)
F_2- - -F_2'	2.774 (7)	2.76 (3)
F_1–Te–F_2	85.4 (2)	86.7 (1.7)
F_1–Te–F_3	79.4 (2)	78.7 (1.6)
F_2–Te–F_3	78.3 (2)	78.9 (1.6)
F_1–Te–F_1'	90.0 (2)	88.4 (1.7)
F_2–Te–F_2'	90.6 (2)	89.6 (1.7)
K–F_1	2.887 (3)	2.88 (2)
K–F_2	2.952 (4)	2.97 (2)
K–F_3	2.892 (1)	2.90 (2)
K–$F_2{}^{I}$	2.706 (4)	2.68 (2)
K–$F_1{}^{II}$	2.671 (3)	2.67 (2)

[a] The reference unit is at *x, y, z;* primed numerals refer to atoms at *x, y,* $1/2 - z$; the superscripts I and II refer to the positions $1 + x, y, z$ and $1 - x, -y, -z$, respectively. [b] Model of Edwards and Mouty.[3] [c] Reference 14.

TABLE V

BOND DISTANCES (Å) AND ANGLES (DEG) IN SQUARE-PYRAMIDAL IONS AND MOLECULES CONTAINING A LONE PAIR OF ELECTRONS

	TeF_5^-	SbF_5^{2-}[a]	$SbCl_5^{2-}$[b]	XeF_5^-[c]	BrF_5[d]
M–X (apical)	1.862 (4)	2.00 (9)	2.36	1.81 (8)	1.68
M–X (basal)	1.953 (3)		2.61		1.80
	1.952 (3)	2.04 (9)	2.62	1.88 (8)	1.81
					1.75
X (apical)–M–X (basal)	79.4 (2)	83 (2)	87	79 (4)	80.5
	78.3 (2)		83	80 (4)	86.5
					85.4

[a] The structure of K_2SbF_5 was refined by the authors from the data of Byström and Wilhelmi using least-squares techniques; the distances and angles given are based on the model determined from that refinement. [b] Reference 5. [c] Reference 10. [d] Reference 6.

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(23) All interionic Te- - -F distances are >3.00 Å.

(24) The interionic Te- - -F_2 (or Te- - -F_2') and Te- - -F_3 distances are 3.709 (5) and 3.992 (5) Å, respectively.

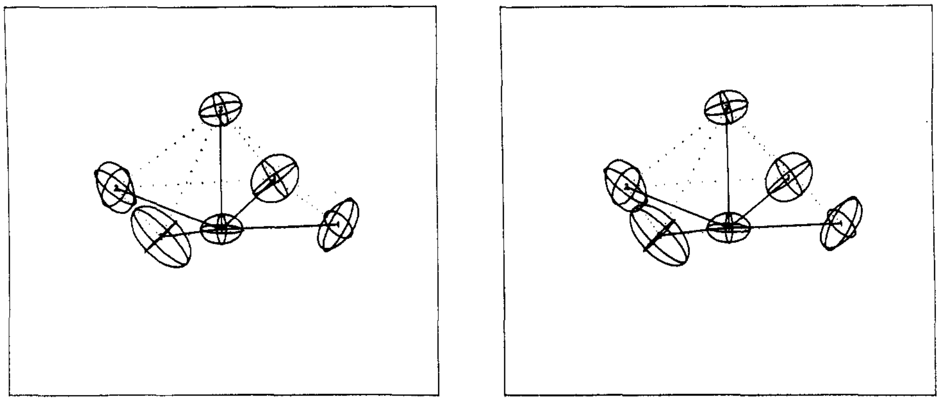

Figure 1.—Stereoview of the pentafluorotellurate ion.

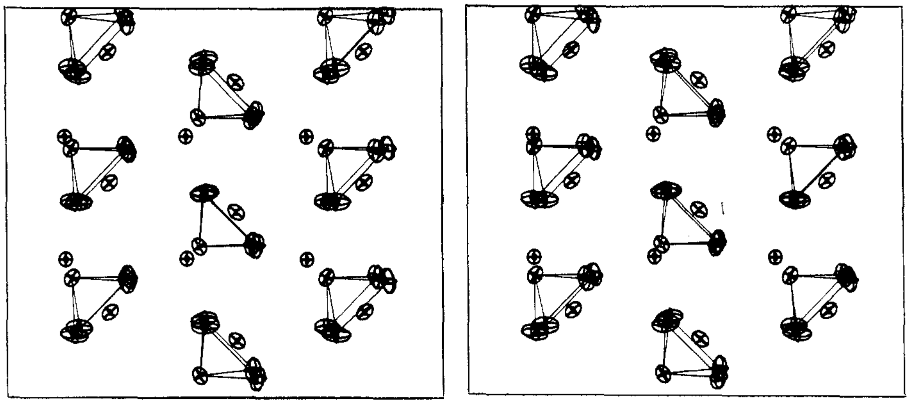

Figure 2.—Stereoview down [001] showing the packing of the K^+ and TeF_5^- ions in the *ab* plane. Only atoms with $0 \leq z < 1/2$ are shown.

tortion of the TeF_5^- ion from C_{4v} symmetry is consistent with the splitting of the ν_9 fundamental in the infrared spectrum of solid $KTeF_5$ observed by Greenwood, Sarma, and Straughan.[25]

For comparative purposes, Table V contains bond distances and angles which have been found in various square-pyramidal moieties containing a lone pair of electrons, including the ions SbF_5^{2-} and XeF_5^+ which are isoelectronic with TeF_5^-. The geometry of these ions is consistent with the model predicted by Gillespie[8] for AX_5E type molecules and from simple molecular orbital considerations.[26]

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