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The Crystal Structure of Ammonium Telluropentathionate

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In a preceding article[1], the crystal structure of tellurium dimethanethiosulphonate was described. The present work is concerned with the crystal structure of another thio derivative of divalent tellurium, *viz.*, ammonium telluropentathionate. The compounds, $Te(S_2O_2CH_3)_2$ and $(NH_4)_2Te(S_2O_3)_2$, both contain an unbranched and non-planar S—S—Te—S—S chain, the dimensions and configuration of which have been found to be almost exactly the same in the two crystals.

Telluropentathionic acid, the ammonium salt of which was first prepared in 1950[2], is derived from pentathionic acid by substitution of tellurium for the middle sulphur atom of the latter acid. The crystal structure of barium pentathionate dihydrate was published recently[3]. The telluropentathionate and pentathionate anions have analogous structures, except that they in these salts occur as rotational isomers of each other.

X-RAY DATA

The crystals of ammonium telluropentathionate, $(NH_4)_2Te(S_2O_3)_2$, are monoclinic prismatic, and occur as yellow prisms, elongated along the a axis and bounded by $\{011\}$.

From oscillation and Weissenberg photographs, taken with Cu$K\alpha$ radiation, $\lambda = 1.542$ Å, the following unit cell dimensions were derived: $a = 5.03$ Å, $b = 18.90$ Å, $c = 11.61$ Å, $\beta = 100°$. The axial lengths are believed to be accurate to ± 0.5 %. The values reported earlier[2] for the β angle and the c axis are in error.

There are four molecules per unit cell; density, calc. 2.35, found 2.34 g/cm^3. The systematic absences, $h0l$ when l is odd and $0k0$ when k is odd, determine the space group as C_{2h}^5—$P2_1/c$.

For intensity measurements, zero layer Weissenberg photographs were taken about the a and c axes, using a double film technique. The crystals employed had cross-sections of about 0.05×0.05 mm. 199 of the 282 $0kl$

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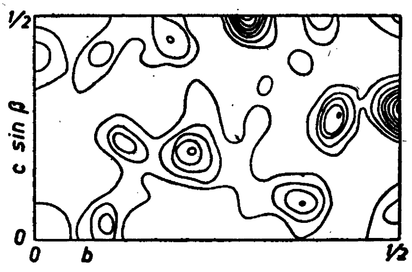

Fig. 1. Patterson projection along the a *axis. Contours at arbitrary but equal interval. The origin peak is not drawn.*

Fig. 2. Patterson projection along the c *axis. Contours as in Fig. 1.*

reflections and 103 of the 128 $hk0$ reflections attainable with Cu$K\alpha$ radiation were recorded on the films with measurable intensities. The intensities were estimated visually, and corrected for the Lorentz and polarization factors as described by Goldschmidt and Pitt [4]. No correction for absorption was applied. The structure factors were later placed on an approximately absolute scale by comparison with the calculated values.

The calculated structure factors were based on the Hartree atomic scattering curves for sulphur and oxygen, and the Thomas-Fermi curve for tellurium. The same curve was used for all sulphur atoms, and the oxygen curve was used for the nitrogen atom of the ammonium ions. A temperature factor exp ($-B \sin^2\Theta/\lambda^2$) with $B = 4.0$ Å^2 for the $0kl$ reflections and $B = 3.8$ Å^2 for the $hk0$ reflections was applied to the calculated structure factors. The values of B, and the scale factors, were determined from plots of log $|F_{\text{obs}}|/|F_{\text{calc}}|$ against $\sin^2 \Theta$.

The Patterson and Fourier summations were made at 6° intervals along all three axes, using Beevers-Lipson strips. Peaks were located by Booth's method [5].

DETERMINATION OF THE STRUCTURE

As in the case of tellurium dimethanethiosulphonate [1], the structure was solved by means of the heavy atom technique. The tellurium atom, accounting for 28 % of the electrons in the asymmetric unit, was found to dominate the signs of the reflections to a sufficient degree.

The position of the tellurium atom was located from Patterson projections along the a and c axes. The asymmetric parts of these projections are reproduced in Figs. 1 and 2. The Fourier symmetries of the plane groups being pgg and pgm, respectively, equivalent vectors, of double weight, occur at $v = \frac{1}{2}$, $w = \frac{1}{2} + 2z$ and $v = \frac{1}{2} + 2y$, $w = \frac{1}{2}$ in Fig. 1 and at $u = 0$, $v = \frac{1}{2} + 2y$ and $u = 2x$, $v = \frac{1}{2}$ in Fig. 2. The Te—Te vectors are readily recognized as the highest peaks along the appropriate lines in Figs. 1 and 2, and gave the following tellurium coordinates: $x = 0.132$, $y = 0.105$, $z = 0.102$. Only the x coordinate differs, by -0.002, from the final value.

Fourier projections along the a axis were subsequently carried out, starting with signs calculated from the tellurium contributions alone. The positions of the sulphur atoms were recognized in the first map, and peaks due to oxygen atoms and ammonium ions appeared during the refinement process. Essentially the same procedure was followed with regard to the projection along the c axis. Here, however, overlapping occurs to a large extent, and some of the atoms had to be placed by trial and error methods.

The positive direction of the a axis relative to that of the c axis was established by comparison of some observed and calculated $h0l$ and $\bar{h}0l$ structure factors.

The final electron density maps, $\varrho(yz)$ and $\varrho(xy)$, are reproduced in Figs. 3 and 5, and a diagram showing the outline of the ions as seen along the a axis is shown in Fig. 4.

The atomic coordinates are listed in Table 1. The y coordinates are those determined from $\varrho(yz)$, since the resolution is much better in this projection. For the same reasons, the x coordinates, particularly of S_2, O_3 and N_1, are probably less accurate than the y and z coordinates.

Errors in the y and z coordinates due to finite termination of Fourier series were estimated from a Fourier synthesis based on the calculated $0kl$ structure factors[6]. The tellurium coordinates and the y coordinates of the sulphur atoms remained unchanged, whereas average shifts of 0.025 Å occurred in the sulphur z coordinates, with a maximum shift of 0.04 Å in zS_1. The S_1 peak is subject to overlapping by O_2, and the significance of the shift may be difficult to judge. For the oxygen atoms and the ammonium ions, the average shifts in the y and z coordinates were 0.04 Å and 0.03 Å, respectively. The presence of the heavy tellurium atom introduces a further uncertainty in the coordinates of these lighter atoms.

Table 1. Atomic coordinates, in fractions of cell edges. Origin at centre of symmetry.

	x	y	z		x	y	z
S_1	0.434	-0.022	0.309	O_1	0.622	-0.077	0.320
S_2	0.419	0.005	0.132	O_2	0.151	-0.030	0.330
Te	0.134	0.105	0.102	O_3	0.565	0.034	0.375
S_4	0.432	0.199	0.161	O_4	0.746	0.288	0.052
S_5	0.539	0.236	0.003	O_5	0.317	0.270	-0.063
N_1	0.067	0.097	0.455	O_6	0.646	0.182	-0.063
N_2	0.033	0.334	0.270				

In Table 5, observed and calculated values of structure factors are compared for all $0kl$ and $hk0$ reflections within the range of Cu$K\alpha$ radiation. The reliability factor, $R = \Sigma||F_{\text{obs}}|-|F_{\text{calc}}||/\Sigma|F_{\text{obs}}|$, with unobserved reflections included when $|F_{\text{calc}}|$ is greater than the smallest observable value of $|F_{\text{obs}}|$, is 0.18 for the $0kl$ data and 0.15 for the $hk0$ data, with an overall value of 0.17 for the two zones.

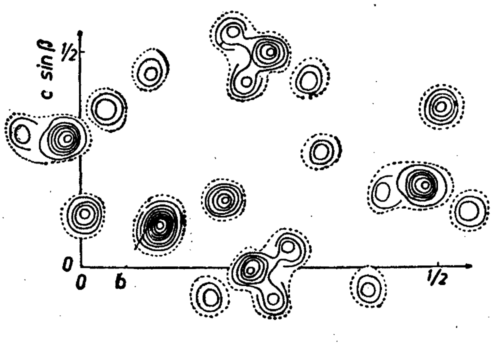

Fig. 3. Electron density projection of $(NH_4)_2Te(S_2O_3)_2$ *along the* a *axis,* $\varrho(yz)$*. The 4-electron line is dotted. Contour intervals: 8 e.Å*$^{-2}$ *for the tellurium atom, 4 e.Å*$^{-2}$ *for the sulphur atoms, and 2 e.Å*$^{-2}$ *for the oxygen atoms and ammonium ions.*

THE TELLUROPENTATHIONATE ION

Distances and angles, calculated on the basis of the coordinates of Table 1, are given below. The numbering of atoms is shown in Fig. 4. The average Te—S, S—S and S—O bond lengths are estimated as reliable to within ± 0.03 Å, ± 0.03 Å and ± 0.04 Å, respectively, whereas N—O distances may be in error by as much as ± 0.1 Å. Angles involving the heavier atoms, tellurium and sulphur, are probably correct to within ± 3°.

The dihedral angles, S_1S_2Te/S_2TeS_4 and S_2TeS_4/TeS_4S_5, are 86° and 95°, respectively.

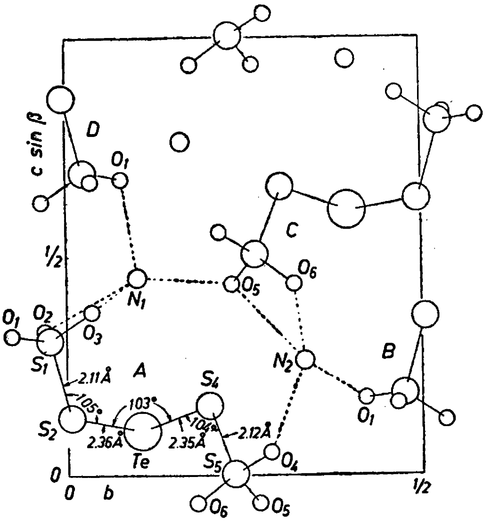

Fig. 4. Arrangement of the ions as seen along the a *axis. Half a unit cell (two asymmetric units) is shown.*

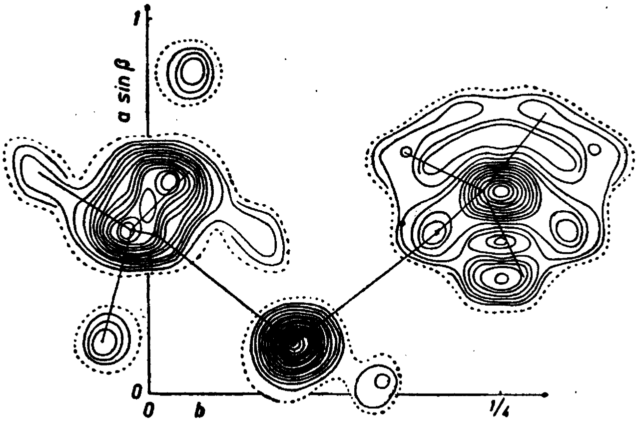

Fig. 5. Electron density projection of $(NH_4)_2Te(S_2O_3)_2$ *along the* c *axis,* $\varrho(xy)$*. The 6-electron line is dotted. Contour intervals as in Fig. 3. Lines are drawn to show the outline of one telluropentathionate ion. One of the ammonium ions is resolved, whereas the other coincides with the tellurium peak.*

Table 2. Sulphur-sulphur and tellurium-sulphur bond lengths and bond angles.

$S_1—S_2 = 2.11$ Å
$S_2—Te = 2.36$

$\angle\ S_1S_2Te = 105°$
$\angle\ TeS_4S_5 = 104°$
$\angle\ S_2TeS_4 = 103°$

$S_4—S_5 = 2.12$ Å
$Te—S_4 = 2.35$

Non-bonded distances

$S_1—Te = 3.57$ Å
$S_1—S_4 = 4.52$

$S_2—S_4 = 3.68$ Å
$S_1—S_5 = 6.11$

$Te—S_5 = 3.52$ Å
$S_2—S_5 = 4.69$

The S—S—Te—S—S chain of the telluropentathionate ion is unbranched and non-planar, and occurs in a *trans* form [7], as does the analogous chain of tellurium dimethanethiosulphonate [1]. In fact, the bond lengths and angles of Table 2 are, within the probable errors, the same as those found for tellurium dimethanethiosulphonate.

The only differences between the S—S—Te—S—S chains of the two compounds occur in the dihedral angles, these being susceptible to variations with rotations about the Te—S bonds. In tellurium dimethanethiosulphonate [1], the dihedral angles are both 81°. The larger values in the telluropentathionate ion, 86° and 95°, are in accordance with the larger $S_1—S_4$, $S_1—S_5$ and $S_2—S_5$ distances, 4.52 Å, 6.11 Å and 4.69 Å, as compared with 4.38 Å, 5.74 Å and 4.36 Å, respectively, in the methanethiosulphonate.

The terminal sulphur atoms are each surrounded by three oxygen atoms, at an average distance of 1.43 Å. Together with the sulphur atoms S_2 and S_4, respectively, each sulphonate group forms a thiosulphate group having the shape of a distorted tetrahedron. The dimensional data are listed in Table 3.

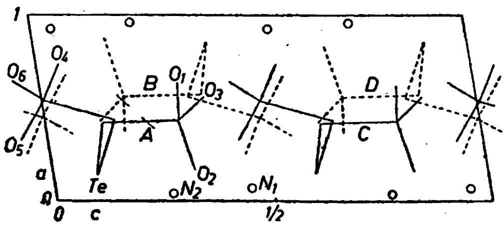

Fig. 6. Arrangement of the ions as viewed along the b *axis, showing the layer structure.*

Table 3. Sulphur-oxygen bond lengths and bond angles.

S_1-O_1 = 1.40 Å	$\angle S_2S_1O_1$ = 100°	$\angle O_1S_1O_2$ = 124°
S_1-O_2 = 1.48	$\angle S_2S_1O_2$ = 109°	$\angle O_1S_1O_3$ = 106°
S_1-O_3 = 1.40	$\angle S_2S_1O_3$ = 107°	$\angle O_2S_1O_3$ = 109°
S_5-O_4 = 1.47	$\angle S_4S_5O_4$ = 99°	$\angle O_4S_5O_5$ = 111°
S_5-O_5 = 1.40	$\angle S_4S_5O_5$ = 109°	$\angle O_4S_5O_6$ = 112°
S_5-O_6 = 1.43	$\angle S_4S_5O_6$ = 114°	$\angle O_5S_5O_6$ = 112°
Average: 1.43 Å	Average: 106°	Average: 112°

The average distance from the oxygen atoms to the nearest non-bonded sulphur atom is 2.86 Å, and the average non-bonded oxygen-oxygen distance within sulphonate groups is 2.37 Å, with individual values ranging from 2.73 to 3.00 Å and 2.23 to 2.54 Å, respectively. The average dimensions of the thiosulphate groups are the same, within the limits of error, as those found in barium pentathionate dihydrate [3] and barium tetrathionate dihydrate [8].

The telluropentathionate ion consists of two thiosulphate groups attached to divalent tellurium through covalent Te—S bonds. In displacement reactions with nucleophilic reagents [9,10] the tellurium atom acts as the electrophilic part of the ion. The telluropentathionate ion may thus be regarded as a thiosulphate complex of divalent tellurium.

THE IONIC ENVIRONMENT

The relative arrangement of oxygen atoms and ammonium ions is of interest in connection with the frequent occurrence of N—H···O hydrogen bonds in ammonium salts of oxy-acids and in organic crystals. The lengths of N—H···O bonds usually lie in the range [11,12] 2.8—3.0 Å.

In the present structure, each ammonium ion appears to form hydrogen bonds to four oxygen atoms, which in each case belong to four different telluropentathionate ions. The arrangement is a distorted tetrahedral one, with an average nitrogen-oxygen distance of 2.83 Å for N_1 and 2.88 Å for N_2. The data are collected in Table 4. The letters *A* to *D* refer to different telluropentathionate ions, as shown in Figs. 4 and 6. A prime denotes nearest corresponding atom in adjacent unit cell along the *a* axis.

Table 4. Distances, and angles between directions, from ammonium ions to oxygen atoms.

N_1-O_{1D}	= 2.83 Å	N_2-O_{1B}	= 2.75 Å
N_1-O_{2A}	= 2.87	$N_2-O_{4A'}$	= 2.82
$N_1-O_{3A'}$	= 2.80	N_2-O_{5C}	= 2.95
N_1-O_{5C}	= 2.83	$N_2-O_{6C'}$	= 2.99
$\angle O_{1D}-N_1-O_{2A}$	= 105°	$\angle O_{1B}-N_2-O_{4A'}$	= 96°
$\angle O_{1D}-N_1-O_{3A'}$	= 125°	$\angle O_{1B}-N_2-O_{5C}$	= 113°
$\angle O_{1D}-N_1-O_{5C}$	= 90°	$\angle O_{1B}-N_2-O_{6C'}$	= 147°
$\angle O_{2A}-N_1-O_{3A'}$	= 73°	$\angle O_{4A'}-N_2-O_{5C}$	= 120°
$\angle O_{2A}-N_1-O_{5C}$	= 127°	$\angle O_{4A'}-N_2-O_{6C'}$	= 123°
$\angle O_{3A'}-N_1-O_{5C}$	= 138°	$\angle O_{5C}-N_2-O_{6C'}$	= 79°

Other nitrogen-oxygen approaches shorter than 3.3 Å are N_1—$O_{4C'}$ = 3.04 Å, N_1—O_{3A} = 3.06 Å, N_1—$O_{2D'}$ = 3.16 Å, and N_2—$O_{2B'}$ = 2.91 Å. The latter has not been included in Table 4 because the angles, $O_{2B'}$—N_2—O_{1B} = 59° and $O_{2B'}$—N_2—O_{5C} = 159°, appear to deviate too much from the tetrahedral value for hydrogen bonding to occur.

According to the above picture, the six oxygen atoms of a telluropentathionate ion participate in hydrogen bonding to eight different ammonium ions. Two oxygen atoms, one from each sulphonate group, form two hydrogen bonds, whereas each of the other four oxygen atoms form one bond only.

From Fig. 6, which shows the contents of the unit cell as viewed along the *b* axis, it appears that the telluropentathionate ions are arranged in layers normal to the *a* axis, with intermediate layers of ammonium ions. Twelve N—H···O bonds pass across each *a* face and hold the layers together.

SUMMARY

The crystal structure of ammonium telluropentathionate has been determined from X-ray data, by two-dimensional Patterson and Fourier methods.

The telluropentathionate ion contains an unbranched S—S—Te—S—S chain, which is non-planar and has a *trans* configuration. The dihedral angles are 86° and 95°.

The average Te—S and S—S bond lengths are 2.36 Å and 2.12 Å, respectively. The Te—S—S bond angles are 105° and 104°, and the S—Te—S bond angle is 103°.

Three oxygen atoms, in approximately tetrahedral positions, surround each of the terminal sulphur atoms, at an average distance of 1.43 Å.

Hydrogen bonding occurs between the oxygen atoms and the nitrogen atoms of the ammonium ions.

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Table 5. Observed and calculated values of structure factors for ammonium telluropentathionate. The figures represent one fourth of the absolute values.

k	F_{obs}	F_{calc}
	0*kl* zone	
	0*k*0	
2	12	+13
4	10	+6
6	17	−16
8	27	+23
10	40	+39
12	5	+3
14	21	−19
16	6	−8
18	5	+6
20	5	+6
22	3	−3
24	4	−5
	0*k*1	
1	21	+22
2	21	−20
3	24	−25
4	2	−1
5	17	−17
6	18	+18
7	5	+3
8	27	+30
9	26	+24
10	10	−6
11	18	+17
12	11	−12
13	7	−6
14	10	+6
15	7	−7
16	9	+8
17	10	+10
18	6	+6
19	6	+7
20	< 3	−2
21	5	+6
22	6	−6
23	4	−4
24	< 2	+1
	0*k*2	
0	15	+17
1	30	−34
2	30	−28
3	37	−41
4	6	−6
5	3	−4
6	32	−30
7	24	+23
8	13	+13
9	11	+11
10	15	+5
11	34	−31
12	6	+2
13	17	−14
14	7	−6
15	< 4	0
16	< 4	0
17	< 4	+5
18	5	+6
19	< 4	+1
20	< 3	+3
21	11	−10
22	3	+3
23	< 2	−4
24	< 1	−4
	0*k*3	
1	6	−2
2	43	−46
3	28	+25
4	26	−27
5	7	+8
6	17	+21
7	< 2	+1
8	19	+18
9	16	−15
10	17	−14
11	14	−16
12	23	−21
13	11	+6
14	11	−10
15	10	−7
16	9	+11
17	6	−1
18	4	+5
19	13	−12
20	5	−5
21	< 3	−3
22	6	−6
23	< 2	−1
	0*k*4	
0	30	−39
1	8	+3
2	26	−32
3	8	−10
4	20	+17
5	14	+14
6	< 3	+3
7	25	+26
8	12	−10
9	8	+5
10	27	−29
11	4	0
12	9	+4
13	< 4	0
14	7	+8
15	7	+6
16	< 4	+2
17	11	+11
18	7	−5
19	< 3	−1
20	7	−7
21	< 3	−3
22	< 2	+2
23	< 2	−2
	0*k*5	
1	37	−39
2	11	+6
3	8	−4
4	17	−19
5	27	+29
6	7	+8
7	10	−8
8	< 3	+2
9	22	−21
10	< 3	−3
11	20	−19
12	8	+5
13	13	+12
14	8	−6
15	8	+8
16	< 4	+3
17	< 4	+1
18	< 4	−1
19	9	−8
20	3	+2
21	< 3	+1
22	< 2	+1
	0*k*6	
0	2	−2
1	14	+15
2	7	0
3	12	+14
4	28	+32
5	15	−10
6	10	+11
7	21	−18
8	3	−6
9	8	−10
10	9	−10
11	8	+5
12	< 4	−1

k	F_{obs}	F_{calc}
13	6	+ 8
14	9	+ 9
15	9	− 5
16	5	+ 4
17	6	− 6
18	8	− 9
19	3	+ 1
20	< 3	− 3
21	4	+ 5
22	< 1	0
	0*k*7	
1	7	+ 1
2	20	+ 22
3	5	+ 5
4	19	+ 20
5	18	+ 17
6	9	− 8
7	< 3	− 1
8	5	− 6
9	14	+ 10
10	11	+ 10
11	5	− 3
12	18	+ 15
13	< 4	+ 7
14	6	+ 6
15	4	+ 4
16	5	− 5
17	4	+ 2
18	< 3	− 2
19	< 3	− 2
20	5	+ 4
21	< 2	+ 2
	0*k*8	
0	5	+ 5
1	< 3	+ 3
2	< 3	+ 2
3	19	+ 20
4	5	− 3
5	< 4	− 4
6	5	− 5
7	16	− 21
8	13	+ 10
9	< 4	− 2
10	< 4	+ 2
11	5	+ 8
12	< 4	+ 4
13	9	+ 8
14	4	− 2
15	3	− 4
16	5	+ 2
17	8	− 8
18	6	+ 4
19	< 2	0
20	< 2	+ 3
	0*k*9	
1	12	+ 17
2	< 4	+ 4
3	< 4	− 2
4	< 4	0
5	12	− 12
6	9	− 12
7	< 4	− 1
8	10	− 9
9	15	+ 14
10	6	− 2
11	< 4	+ 2
12	< 4	0
13	3	+ 2
14	< 3	− 2
15	9	− 9
16	4	− 5
17	7	+ 3
18	5	− 3
19	1	+ 3
	0,*k*,10	
0	11	+ 10
1	< 4	+ 2
2	6	+ 5
3	< 4	+ 3
4	5	− 9
5	< 4	+ 2
6	9	− 11
7	7	+ 5
8	11	+ 10
9	< 4	0
10	< 4	+ 4
11	< 4	+ 2
12	< 3	+ 1
13	< 3	0
14	8	− 8
15	3	+ 1
16	3	− 3
17	< 2	+ 1
	0,*k*,11	
1	< 4	+ 2
2	4	− 7
3	8	− 9
4	4	− 6
5	8	− 10
6	9	+ 5
7	9	− 6
8	3	+ 4
9	4	+ 7
10	< 3	− 1
11	< 3	− 1
12	< 3	− 4
13	< 3	− 3
14	< 2	− 2
15	5	− 5
16	4	+ 4
	0,*k*,12	
0	4	+ 3
1	5	− 7
2	7	− 5
3	6	− 8
4	< 3	− 1
5	< 3	0
6	6	− 4
7	4	+ 6
8	3	− 1
9	< 3	0
10	< 3	0
11	2	− 4
12	< 2	− 1
13	5	− 5
14	< 1	− 1
	0,*k*,13	
1	< 3	+ 1
2	4	− 6
3	< 3	0
4	< 3	0
5	6	+ 6
6	< 3	+ 4
7	2	− 1
8	5	+ 6
9	< 2	− 1
10	< 2	0
11	< 2	− 1
	0,*k*,14	
0	< 2	− 4
1	< 2	− 3
2	3	− 3
3	< 2	− 1
4	5	+ 5
5	< 2	0
6	< 2	+ 3
7	< 1	+ 2
	*hk*0 zone	
	0*k*0	
2	12	+ 13
4	9	+ 7
6	14	− 16
8	26	+ 22
10	38	+ 40
12	4	+ 3
14	20	− 20

k	F_{obs}	F_{calc}
16	6	−8
18	4	+6
20	5	+7
22	3	−3
24	4	−5

1k0

k	F_{obs}	F_{calc}
0	20	−19
1	19	−18
2	5	+6
3	31	−32
4	56	−64
5	9	+7
6	22	−23
7	37	+38
8	3	+4
9	13	+15
10	8	+10
11	12	−11
12	5	−2
13	6	−9
14	13	−15
15	5	+5
16	7	−9
17	7	+8
18	8	+9
19	< 3	0
20	3	+5
21	5	−7
22	< 2	−1
23	1	−3
24	< 1	−2

2k0

k	F_{obs}	F_{calc}
0	27	+22
1	21	−19
2	7	+5
3	30	−33
4	13	+13
5	7	+4
6	8	+8
7	25	+29
8	< 2	+4
9	< 2	+1
10	< 3	0
11	13	−17
12	6	+5
13	15	−18
14	< 3	−1
15	7	+8
16	2	+1
17	9	+12
18	3	−3
19	2	+1
20	< 2	+1
21	4	−5
22	< 2	−1
23	2	−3

3k0

k	F_{obs}	F_{calc}
0	29	−22
1	10	−11
2	9	−9
3	14	−13
4	8	+8
5	9	+7
6	13	+13
7	13	+15
8	13	−13
9	15	+18
10	12	−11
11	8	−10
12	3	+4
13	< 3	0
14	10	+11
15	< 3	+2
16	2	+4
17	5	+4
18	3	−3
19	2	+1
20	3	−4
21	3	−5

4k0

k	F_{obs}	F_{calc}
0	15	−18
1	< 3	+1
2	11	−11
3	5	+1
4	14	+13
5	8	−10
6	5	+7
7	< 3	+1
8	7	−6
9	12	−10
10	13	−13
11	3	+2
12	< 3	0
13	2	−2
14	7	+7
15	< 3	−2
16	< 2	+2
17	< 2	0
18	3	−3
19	< 1	0

5k0

k	F_{obs}	F_{calc}
0	< 3	−1
1	8	+7
2	3	+4
3	6	+7
4	5	+6
5	2	+3
6	7	+7
7	10	−9
8	< 3	−2
9	3	+2
10	< 2	−1
11	4	+3
12	< 2	+1
13	5	+5
14	2	+3
15	2	−1

6k0

k	F_{obs}	F_{calc}
0	< 2	−2
1	2	+3
2	< 2	−2
3	6	+4
4	5	−4
5	3	−4
6	3	−3
7	4	−4
8	< 2	0

REFERENCES

1. Foss, O. and Vihovde, E. H. *Acta Chem. Scand.* **8** (1954) 1032.
2. Foss, O. and Jahr, J. *Acta Chem. Scand.* **4** (1950) 1560.
3. Foss, O. and Zachariasen, H. *Acta Chem. Scand.* **8** (1954) 473.
4. Goldschmidt, G. H. and Pitt, G. J. *J. Sci. Instr.* **25** (1948) 397.
5. Booth, A. D. *Fourier techniques in organic structure analysis* Cambridge 1948, pp. 62–65.

6. Booth, A. D. *Proc. Roy. Soc.* **A 188** (1946) 77.
7. Foss, O. *Acta Chem. Scand.* **7** (1953) 1221.
8. Foss, O., Furberg, S. and Zachariasen, H. *Acta Chem. Scand.* **8** (1954) 459.
9. Foss, O. *Acta Chem. Scand.* **3** (1949) 1385.
10. Foss, O. *Acta Chem. Scand.* **6** (1952) 521.
11. Donohue, J. *J. Phys. Chem.* **56** (1952) 502.
12. Romers, C., Ketelaar, J. A. A. and MacGillavry, C. H. *Acta Cryst.* **4** (1951) 114.

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