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The Crystal Structure of Tellurium Dimethanethiosulphonate

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The methanetiosulphonates, $S(S_2O_2CH_3)_2$, $Se(S_2O_2CH_3)_2$ and $Te(S_2O_2CH_3)_2$, were first prepared in 1950 [1]. They are derived from pentathionic, selenopentathionic and telluropentathionic acid, respectively, by substitution of methyl for the hydroxyl groups of the acids. The crystals of the methanethiosulphonates are isomorphous [1,2].

In the present article, the structure of tellurium dimethanethiosulphonate is described. A following article [3] reports the crystal structure of the ammonium salt of telluropentathionic acid. It will be shown that the S—S—Te—S—S chains in the two structures are closely analogous.

The structure of tellurium dimethanethiosulphonate, and subsequently of the isomorphous sulphur and selenium analogues, was solved by use of the heavy atom technique. The tellurium atom, in presence of four sulphur atoms, four oxygen atoms and two methyl groups, was found to dominate the signs of the reflections to a sufficient degree.

The structure is the first to be reported for a compound containing divalent tellurium bonded to sulphur.

X-RAY DATA

The crystals of tellurium dimethanethiosulphonate, $Te(S_2O_2CH_3)_2$, are monoclinic prismatic. They appear as needles or prisms, elongated along the b axis and in most cases flattened along the a axis.

The dimensions of the unit cell [1] are: $a = 11.43$ Å, $b = 5.29$ Å, $c = 16.32$ Å, $\beta = 91°$. There are four molecules per unit cell; density, calc. 2.36, found 2.35 g/cm^3. Absent reflections, $h0l$ when $h + l$ is odd, $0k0$ when k is odd. The space group is thus $C_{2h}{}^5$—$P2_1/n$.

For intensity measurements, zero layer Weissenberg photographs were taken about the b and a axes, using Cu$K\alpha$ radiation. The crystals employed were small, having cross-sections of about 0.05 × 0.05 mm and 0.1 × 0.1 mm, respectively, in the case of the b and a axis photographs. 145 of the 246 $h0l$

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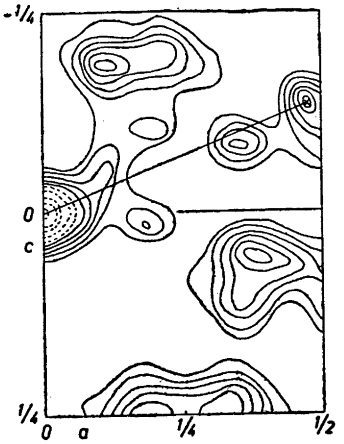

Fig. 1. Patterson projection along the b *axis. Contours at arbitrary but equal intervals, except at the origin peak, where the dotted contours represent intervals four times as large. The straight line is the Te-Te vector.*

reflections and 97 of the 114 0*kl* reflections theoretically observable with Cu$K\alpha$ radiation were recorded on the films with measurable intensities.

The intensities were estimated visually, and corrected for Lorentz and polarization factors. No correction for absorption was applied. The structure factors were eventually 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 carbon atoms of the methyl groups, the hydrogen atoms otherwise being ignored. A temperature factor exp $(-B \sin^2\Theta/\lambda^2)$ with $B = 5.1$ Å^2 for the *h*0*l* reflections and $B = 3.1$ Å^2 for the 0*kl* reflections was applied to the calculated structure factors. These values of B were found to give the best agreement with the observed structure factors.

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 [4].

DETERMINATION OF THE STRUCTURE

The Patterson projection along the *b* axis is shown in Fig. 1. In the asymmetric unit, comprising a quarter of the unit cell, one Te—Te vector should occur. There are, however, four high peaks in the map, of approximately the same height. The Te—Te vector was identified by making a Fourier synthesis for each of the possible positions, using signs corresponding to the tellurium contributions alone. One of the Fourier maps, based on the tellurium coordinates, $x = 0.234$ and $z = -0.066$, showed four well resolved sulphur atoms. The three other high peaks in the Patterson map were subsequently found to be due to overlapping Te—S vectors.

The *z* coordinates were later confirmed through Patterson and Fourier projections along the *a* axis. Also, the 0*kl* data showed that the origin first

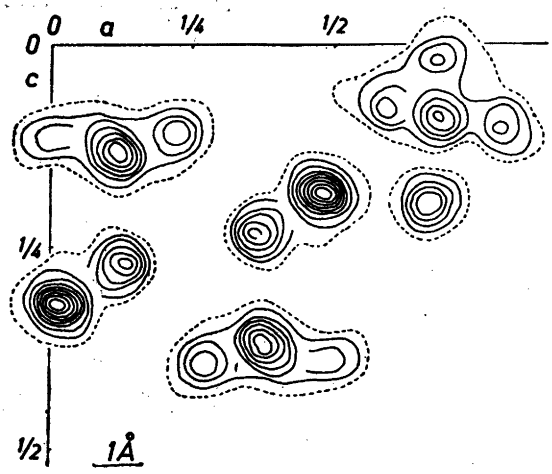

Fig. 2. Projection of Te $(S_2O_2CH_2)_2$ along the b *axis,* ϱ (xz). *The 2-electron line is dotted, and the first fully drawn line represents 6 e.Å^{-2} for tellurium and 4 e.Å^{-2} for the other atoms. Contour intervals thereafter: 8 e.Å^{-2} for tellurium, 4 e.Å^{-2} for sulphur, and 2 e.Å^{-2} for oxygen and carbon atoms.*

chosen in the case of the *b* axis projection, as reported in the preliminary note [2], corresponded to a twofold screw axis, located at $x = \frac{1}{4}$, $z = \frac{1}{4}$ from a centre of symmetry.

Refinement of the *h0l* projection was made by double Fourier series methods in the usual way. Peaks due to oxygen and carbon atoms appeared in the second Fourier map, after inclusion of the sulphur contributions in the calculated structure factors. The final electron density map, $\varrho(xz)$, and an explanatory diagram, are reproduced in Figs. 2 and 3.

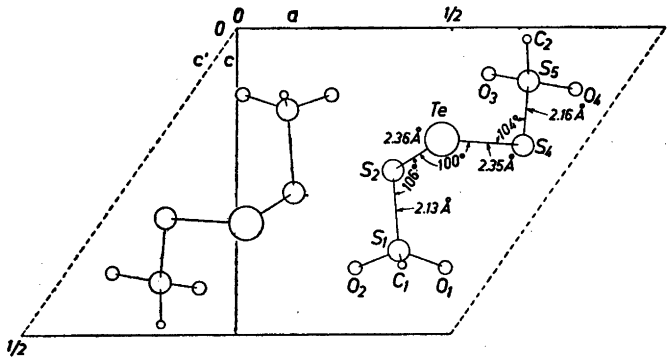

Fig. 3. Arrangement of the molecules as viewed along the b *axis. The dotted line,* c', *shows the direction of the* c *axis in the setting* $P2_1/c$. *The molecules are seen to be extended along the direction of translation of the glide plane.*

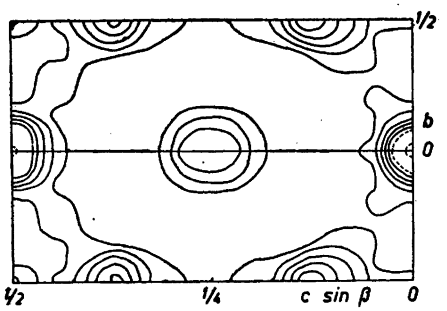

Fig. 4. Patterson projection along the a *axis. Contours at arbitrary but equal intervals, except for the dotted contours which represent intervals four times as large.*

The 0*kl* data were used to evaluate the y coordinates. Fig. 4 shows the Patterson projection along the a axis. The Fourier and Patterson symmetries being *pgg* and *pmm*, respectively, equivalent vectors, of double weight, occur in the Patterson map at $v = \frac{1}{2} + 2y$, $w = \frac{1}{2}$ and $v = \frac{1}{2}$, $w = \frac{1}{2} + 2z$, and equivalent vectors, of single weight, at $v = 2y$, $w = 2z$ and $v = -2y$, $w = 2z$. The high peak at $v = 0$, $w = \frac{1}{2}$ in Fig. 4 shows that the tellurium atom lies near the glide plane at $y = \frac{1}{4}$. Correspondingly, the vector at $v = 2y$, $w = 2z$, of single weight, occurs at $v = \frac{1}{2}$ and coincides with the vector at $v = -2y$, $w = 2z$, thus acquiring double weight. This peak, at $w = 0.372$, and that at $v = \frac{1}{2}$, $w = \frac{1}{2} + 2z$ both give the z coordinate of tellurium as 0.186, whereas the value derived from the b axis Patterson map, referred to a centre of symmetry as origin, is 0.184, and the final value, from both projections, 0.182.

In the plane group, *pgg*, atoms at $y = \frac{1}{4}$ do not contribute to 0*kl* reflections when l is odd, whereas for reflections with l even the contribution is large. Signs were therefore calculated for the 0*kl* reflections with l even on the basis of $y = \frac{1}{4}$ for the tellurium atom, and a Fourier synthesis was made using these even l terms only. Such a projection contains peaks corresponding to the atomic positions, and images of these atoms produced by the operation of a spurious centre of symmetry. The true peaks were identified partly from considerations of possible tellurium-sulphur and sulphur-sulphur bond lengths and angles, and ultimately by the use of Bragg-Lipson charts for some reflections the signs of which were difficult to determine. The last electron density map, $\varrho(yz)$, and a line diagram, are shown in Figs. 5 and 6.

The atomic coordinates are listed in Table 1. The z coordinates of the oxygen and carbon atoms are those determined from $\varrho(xz)$, since in $\varrho(yz)$ overlapping occurs for these atoms. Their y coordinates, except for C_2, were arrived at through trial and error calculations. For other atoms, the following z values, from $\varrho(xz)$ and $\varrho(yz)$, respectively, differed in the two projections: S_1, 0.363 and 0.361; S_2, 0.233 and 0.231; S_5, 0.086 and 0.088. These figures were used for the calculation of structure factors for the respective zones, whereas bond lengths and angles are based on the average z values.

In Table 4, observed and calculated values of structure factors are compared for all *h*0*l* and 0*kl* reflections within the range of Cu$K\alpha$ radiation. The reliability factor, $R = \Sigma \, || F_{obs} | - | F_{calc} || / \Sigma \, | F_{obs} |$, is 0.15 for the *h*0*l* reflections and 0.18 for the 0*kl* reflections, with an overall value of 0.16 for the two zones.

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

	x	y	z		x	y	z
S_1	0.383	−0.031	0.362	O_1	0.487	−0.140	0.393
S_2	0.371	−0.058	0.232	O_2	0.279	−0.142	0.393
Te	0.484	0.279	0.182	C_1	0.384	0.301	0.393
S_4	0.672	0.101	0.195	O_3	0.592	−0.317	0.078
S_5	0.686	−0.134	0.087	O_4	0.792	−0.271	0.099
				C_2	0.680	0.119	0.015

The unobserved reflections are included in R, as the difference between $|F_{calc}|$ and the lowest observable value of $|F_{obs}|$, only when $|F_{calc}|$ is greater than the smallest value of $|F_{obs}|$. They are not included in $\Sigma|F_{obs}|$.

DESCRIPTION OF THE MOLECULE

The S—S—Te—S—S chain of tellurium dimethanethiosulphonate is unbranched and non-planar. The terminal sulphur atoms are rotated out of the plane of the middle atoms of the chain, to opposite sides of the plane. This is the *trans* form of a pentathionic compound [5].

Bond lengths and angles, calculated on the basis of the coordinates of Table 1, are given below. The numbering of atoms is shown in Figs. 3 and 6. The data are estimated as reliable to within ± 0.03 Å for the average Te—S and S—S bond lengths, and ± 0.06 Å for S—O and S—C bond lengths. Angles involving tellurium and sulphur atoms only are probably correct to within ± 3°.

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

$S_1-S_2 = 2.13$ Å	$\angle S_1S_2Te = 106°$	$S_4-S_5 = 2.16$ Å
$S_2-Te = 2.36$	$\angle TeS_4S_5 = 104°$	$Te-S_4 = 2.35$
	$\angle S_2TeS_4 = 100°$	

Non-bonded distances

$S_1-Te = 3.57$ Å	$S_2-S_4 = 3.60$ Å	$Te-S_5 = 3.55$ Å
$S_1-S_4 = 4.38$	$S_1-S_5 = 5.74$	$S_2-S_5 = 4.36$

The dihedral angles, S_1S_2Te/S_2TeS_4 and S_2TeS_4/TeS_4S_5, are both 81°. These are the angles between not adjacent S—S and Te—S bonds, as seen projected on a plane normal to the intermediate S—Te bond.

The average S—SO_2CH_3 bond length, 2.15 Å, compares with corresponding bond lengths in analogous compounds of known structure, as follows: S—SO_2CH_3 = 2.10 Å in dimethanesulphonyl disulphide [6], S—SO_3 = 2.14 Å in barium pentathionate dihydrate [7], 2.12 Å in barium tetrathionate dihydrate [8], and 2.15 Å in potassium trithionate [9].

No Te—S bond lengths, directly measured, have hitherto been reported in literature. The average length, 2.36 Å, found in the present structure, is a little shorter than the Te—S single bond length, 2.41 Å, calculated on the basis of Pauling's covalent radii [10]. The difference, if real, is in accordance with the

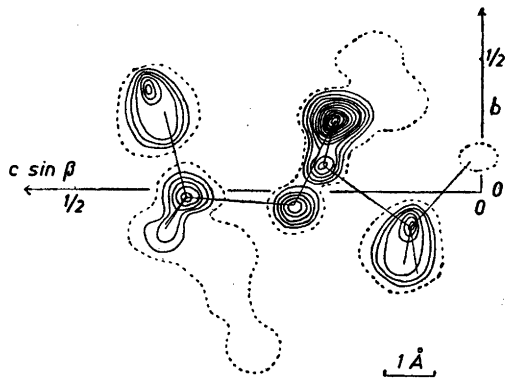

Fig. 5. Projection of Te $(S_2O_2CH_3)_2$ along the a axis, ϱ (yz). The 6-electron line is dotted. Contour intervals: Three times 4 e.Å^{-2} and thereafter 8 e.Å^{-2} for the tellurium atom, and 4 e.Å^{-2} for the other atoms.

shortening of the middle S—S bonds in barium pentathionate dihydrate [7], as compared with the external bonds.

The S—Te—S angle, 100°, is in fair agreement with the values, 102° and 98°, respectively, found in hexagonal tellurium [11] and tellurium dibromide [12].

Two oxygen atoms and a methyl group surround each of the terminal sulphur atoms, in approximately tetrahedral arrangement. The pertinent data are listed in Table 3. The average sulphur-oxygen bond length, 1.43 Å, lies in the range which is usual for such bonds [13,14], and the sulphur-carbon bond lengths, 1.83 Å and 1.78 Å, are close to the sum of the single radii [10], 1.81 Å.

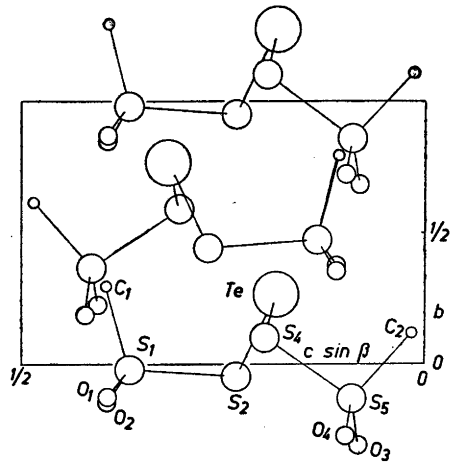

Fig. 6. Arrangement of the molecules as seen along the a axis.

Table 3. Dimensions of the sulphonyl groups. The right column represents non-bonded distances.

S_1-O_1	= 1.41 Å	$\angle S_2S_1O_1$	= 112°	O_1-O_2	= 2.33 Å
S_1-O_2	= 1.43	$\angle S_2S_1O_2$	= 107°	O_1-C_1	= 2.61
S_1-C_1	= 1.83	$\angle S_2S_1C_1$	= 110°	O_2-C_1	= 2.63
S_5-O_3	= 1.45	$\angle S_4S_5O_3$	= 114°	O_3-O_4	= 2.32
S_5-O_4	= 1.43	$\angle S_4S_5O_4$	= 105°	O_3-C_2	= 2.73
S_5-C_2	= 1.78	$\angle S_4S_5C_2$	= 96°	O_4-C_2	= 2.78

It was pointed out earlier [5] that a *trans* form of a pentathionic compound possesses a twofold axis of symmetry, provided that bond lengths and angles are the same in both halves of the molecule. In tellurium dimethanethiosulphonate, no molecular symmetry is crystallographically required. As appears from Table 2, however, the S—S—Te—S—S chain possesses, within the probable errors, a twofold axis of symmetry. The relative positions of the oxygen atoms and the methyl groups do not quite conform to the requirements of a twofold axis.

There appears to be no particularly short approaches between atoms belonging to different molecules. The shortest intermolecular S—S distance, 3.65 Å, is between S_4 and its equivalent along a screw axis.

SUMMARY

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

The molecule consists of an unbranched and non-planar S—S—Te—S—S chain, with two oxygen atoms and a methyl group attached to each of the terminal sulphur atoms. The chain occurs in a *trans* form, the terminal sulphur atoms being rotated an angle of 81° out of the plane of the S—Te—S atoms, to opposite sides of the plane.

The average Te—S and S—S bond lengths are 2.36 Å and 2.15 Å, respectively. The S—Te—S bond angle is 100°, and an average value of 105° was found for the Te—S—S bond angles.

The crystal structures of sulphur and selenium dimethanethiosulphonate, which are isomorphous with tellurium dimethanethiosulphonate, will be published later.

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Table 4. Observed and calculated values of structure factors for tellurium dimethanethiosulphonate.

l	F_{obs}	F_{calc}	l	F_{obs}	F_{calc}	l	F_{obs}	F_{calc}
	*h*0*l* zone			40*l*			80*l*	
	00*l*		0	40	+ 50	0	50	+ 54
			2	53	+ 27	2	< 15	− 12
2	117	− 135	4	112	− 94	4	23	− 48
4	118	− 118	6	137	+ 140	6	29	+ 37
6	34	+ 30	8	113	− 93	8	< 16	+ 10
8	69	− 73	10	16	+ 12	10	31	− 30
10	93	+ 89	12	55	+ 58	12	< 16	+ 8
12	31	+ 35	14	44	− 48	14	< 14	− 16
14	37	− 43	16	19	+ 20	16	< 10	+ 3
16	29	+ 24	18	< 13	+ 10			
18	16	− 14	20	< 7	− 10		90*l*	
20	< 10	− 7						
						1	55	− 55
	10*l*			50*l*		3	< 16	+ 4
						5	19	− 30
1	82	− 89	1	122	− 188	7	< 16	− 6
3	166	+ 184	3	102	+ 111	9	31	+ 50
5	146	− 156	5	50	− 64	11	< 15	− 9
7	147	+ 116	7	64	− 74	13	< 14	− 17
9	37	+ 8	9	104	+ 107	15	< 10	+ 12
11	79	− 79	11	69	− 68			
13	37	+ 29	13	< 16	+ 5		10,0,*l*	
15	29	+ 22	15	< 16	+ 2			
17	19	− 14	17	< 13	− 12	0	37	+ 46
19	16	+ 15	19	< 9	+ 16	2	25	+ 47
21	< 6	− 4				4	40	− 58
						6	31	+ 50
	20*l*			60*l*		8	19	− 19
						10	< 15	− 13
0	80	+ 80				12	16	+ 23
2	257	− 247	0	128	+ 131	14	< 9	− 4
4	57	− 81	2	< 11	− 13			
6	166	+ 167	4	47	− 45			
8	91	− 81	6	31	+ 48		11,0,*l*	
10	< 14	+ 5	8	16	− 30			
12	< 15	− 2	10	23	+ 15	1	55	− 68
14	< 16	− 26	12	23	+ 35	3	< 13	− 10
16	< 16	0	14	31	− 43	5	< 16	+ 16
18	< 14	+ 9	16	< 14	+ 15	7	< 15	− 14
20	< 9	− 10	18	< 10	− 1	9	< 14	+ 16
						11	11	− 21
	30*l*			70*l*		13	< 8	0
1	135	− 133					12,0,*l*	
3	152	+ 150	1	19	− 10			
5	19	− 20	3	91	+ 91			
7	59	− 46	5	44	− 44	0	< 15	+ 3
9	67	+ 66	7	25	− 28	2	< 15	+ 1
11	29	− 45	9	< 16	+ 35	4	< 15	− 22
13	29	+ 29	11	< 16	− 16	6	< 14	+ 22
15	< 16	+ 8	13	< 16	+ 2	8	< 13	− 7
17	23	− 24	15	16	+ 27	10	< 10	− 4
19	< 11	+ 10	17	< 10	− 13	12	< 6	+ 11

l	F_{obs}	F_{calc}
	13,0,l	
1	< 14	− 8
3	< 13	+ 10
5	< 11	+ 10
7	11	− 18
9	< 8	+ 10
	14,0,l	
0	< 10	− 4
2	< 10	+ 9
4	< 2	− 9
6	< 7	+ 1
	$\bar{1}0l$	
1	48	− 60
3	76	+ 84
5	266	− 244
7	80	+ 49
9	170	+ 137
11	108	− 92
13	< 16	+ 3
15	< 16	− 8
17	25	− 30
19	25	+ 28
21	< 6	− 7
	$\bar{2}0l$	
2	19	− 3
4	40	+ 40
6	61	+ 72
8	91	− 96
10	< 14	+ 2
12	29	+ 28
14	59	− 49
16	58	+ 56
18	< 14	− 5
20	< 10	− 11
	$\bar{3}0l$	
1	112	+ 100
3	117	+ 132
5	81	− 86
7	57	− 39
9	76	+ 72
11	34	− 34
13	47	+ 49
15	< 16	− 6
17	< 15	− 15
19	< 11	+ 7

l	F_{obs}	F_{calc}
	$\bar{4}0l$	
2	98	− 106
4	50	+ 30
6	40	+ 29
8	97	− 96
10	96	+ 84
12	< 16	− 16
14	44	− 42
16	< 15	+ 12
18	< 13	− 3
20	< 8	+ 1
	$\bar{5}0l$	
1	23	− 21
3	128	+ 116
5	123	− 128
7	50	+ 56
9	25	− 9
11	31	− 36
13	55	+ 48
15	31	− 28
17	14	+ 4
19	11	+ 14
	$\bar{6}0l$	
2	127	− 117
4	59	+ 59
6	68	+ 72
8	113	− 103
10	65	+ 69
12	< 16	− 3
14	< 12	− 8
16	< 14	+ 16
18	11	− 11
	$\bar{7}0l$	
1	41	− 28
3	25	+ 38
5	81	− 88
7	87	+ 69
9	< 16	0
11	37	− 36
13	31	+ 28
15	< 14	− 10
17	16	− 15
	$\bar{8}0l$	
2	101	− 96
4	25	+ 22
6	31	+ 18
8	< 16	− 10
10	< 16	+ 10
12	19	− 10

l	F_{obs}	F_{calc}
14	< 14	− 13
16	19	+ 18
18	< 5	− 6
	$\bar{9}0l$	
1	88	+ 98
3	< 12	+ 10
5	43	− 45
7	29	+ 26
9	< 16	− 3
11	< 16	− 9
13	16	+ 18
15	< 11	− 2
17	< 6	− 3
	$\bar{10}$,0,l	
2	29	− 33
4	< 16	+ 16
6	31	− 38
8	< 16	− 7
10	31	+ 37
12	< 14	− 10
14	< 10	− 8
16	< 2	+ 6
	$\bar{11}$,0,l	
1	< 16	+ 20
3	< 12	+ 8
5	19	− 27
7	25	+ 26
9	< 14	− 2
11	16	− 15
13	< 9	+ 12
	$\bar{12}$,0,l	
2	< 15	− 6
4	25	+ 27
6	< 14	+ 1
8	23	− 28
10	19	+ 21
12	< 8	− 8
	$\bar{13}$,0,l	
1	< 14	+ 5
3	< 13	+ 7
5	< 13	− 16
7	< 11	+ 8
9	< 9	− 4
	$\bar{14}$,0,l	
2	< 10	− 14
4	< 9	+ 13
6	< 8	− 3

l	F_{obs}	F_{calc}
	0*kl* zone	
	00*l*	
2	143	− 139
4	122	− 123
6	48	+ 40
8	79	− 86
10	94	+ 110
12	41	+ 44
14	46	− 61
16	37	+ 34
18	21	− 24
20	8	− 13
	01*l*	
1	8	− 10
2	98	− 98
3	49	+ 47
4	233	+ 230
5	57	+ 60
6	101	− 90
7	84	− 71
8	80	− 83
9	41	+ 36
10	75	+ 67
11	20	+ 8
12	86	− 86
13	21	+ 16
14	44	+ 50
15	16	− 14
16	17	+ 21
17	< 7	− 2
18	26	− 40
19	< 6	− 1
20	8	+ 15
21	< 3	+ 2
	02*l*	
0	134	− 122
1	86	+ 91
2	28	+ 29
3	42	+ 42
4	60	+ 50
5	< 6	− 24
6	69	− 58
7	38	− 48
8	109	+ 111
9	11	− 13
10	56	− 71
11	18	− 10
12	30	− 42
13	29	+ 30
14	59	+ 69
15	< 8	− 10
16	22	− 29
17	< 7	− 12
18	< 6	− 3
19	< 5	+ 8
20	< 3	+ 12
	03*l*	
1	8	+ 4
2	43	+ 26
3	35	− 41
4	80	− 77
5	49	+ 41
6	56	+ 58
7	32	− 28
8	< 7	+ 6
9	14	− 18
10	43	− 52
11	25	+ 29
12	20	+ 16
13	21	− 19
14	20	+ 13
15	15	− 17
16	26	− 30
17	20	+ 30
18	14	+ 29
19	< 2	− 8
	04*l*	
0	58	+ 26
1	35	− 34
2	50	− 48
3	11	+ 3
4	32	− 12
5	39	+ 36
6	51	+ 55
7	59	− 56
8	20	− 25
9	36	+ 53
10	11	− 16
11	12	− 19
12	< 7	+ 5
13	16	− 29
14	14	− 18
15	18	+ 30
16	12	+ 20
17	< 3	− 8
	05*l*	
1	56	− 38
2	42	− 42
3	65	+ 54
4	36	+ 24
5	35	− 44
6	10	− 4
7	40	+ 14
8	16	− 18
9	12	+ 20
10	32	+ 35
11	28	− 33
12	20	− 15
13	8	+ 12
14	< 3	+ 9
	06*l*	
0	30	− 19
1	49	+ 42
2	17	+ 24
3	26	− 28
4	< 6	− 12
5	20	− 21
6	< 6	− 16
7	26	+ 36
8	14	+ 17
9	6	− 4
10	< 3	− 8

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