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The Molecular and Crystal Structure of Antimony Hydrogen Bis(thioglycollate)

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The molecular and crystal structure of $Sb(SCH_2COO)_2H$ has been determined and refined on the basis of three-dimensional X-ray data. The crystals are monoclinic, space group $I2/a$ and the unit-cell dimensions are $a = 12.41$, $b = 12.10$, $c = 12.05$ Å, $\beta = 109.50°$.

The compound has been shown to be a chelate, in which the organic part forms two five-membered rings with the antimony. The antimony atom has two oxygen and two sulphur atom neighbours in a one-sided fourfold coordination.

The presence of two very short intermolecular oxygen-oxygen distances (2.43 and 2.54 Å) suggests symmetrical hydrogen bonds linking the molecules into endless chains.

The determination of the structure of antimony hydrogen bis(thioglycollate) forms part of a series of investigations being carried out at this Institute to examine the coordination of the metal atom in compounds containing arsenic, antimony or bismuth.

EXPERIMENTAL

Antimony trioxide, Sb_2O_3, was dissolved in a hot solution of thioglycollic acid in water (1:1 vol/vol) [1] and allowed to cool slowly. The crystals formed were small colourless plates.

Crystal data.

Antimony hydrogen bis(thioglycollate), $Sb(SCH_2COO)_2H$

Monoclinic:

$a = 12.414 \pm 0.009$, $b = 12.097 \pm 0.007$, $c = 12.048 \pm 0.008$ Å,

$\beta = 109.50 \pm 0.02°$ $D_m = 2.45$ g·cm^{-1}, $Z = 8$, $D_x = 2.38$ g·cm^{-1}

Absent reflections: when $h+k+l$ odd, $h0l$ when h odd and l odd.

Possible space groups: $I2/a$ and Ia

Linear absorption coefficient (CuK), $\mu = 313$ cm^{-1}.

The unit-cell dimensions were obtained from a powder photograph taken in a Guinier focusing camera ($d = 80$ mm) using Cu$K\alpha_1$ radiation and potassium chloride ($a = 6.2930$ Å) as an internal standard.

A single crystal about $0.02 \times 0.08 \times 0.06$ mm in size was used to take multiple-film photographs with CuK radiation. The layer lines $hk0$–$hk5$, $h0l$–$h7l$, and $0kl$–$7kl$

were registered representing a total number of 1037 observed independent reflections. The intensities were estimated by visual comparison with a calibrated film strip and the Lorentz and polarisation corrections were applied. Absorption corrections were made using a program by Werner.[2]

The centrosymmetric space group $I2/a$ with the general position $(0,0,0;\ \frac{1}{2},\frac{1}{2},\frac{1}{2})+$

$$x,y,z;\ \tfrac{1}{2}+x,\bar{y},z;\ \bar{x},\bar{y},\bar{z};\ \tfrac{1}{2}-x,y,\bar{z}$$

was tentatively adopted for the structure analysis.

The structure thus obtained, which does not include the hydrogen atoms, confirmed the correctness of this choice.

DETERMINATION OF THE STRUCTURE

In order to locate the antimony atoms in the unit-cell, the Patterson projections $P(uvp)$ and $P(upw)$ and the sections $P(uv0)$ and $P(u0w)$ were calculated. From the appearance of the Patterson function it was concluded that the antimony atoms occupy a general point position. The positional parameters thus obtained and also the temperature factor and preliminary scale-factors were subjected to a partial refinement by the methods of least squares.

In order to locate the sulphur atoms, electron density sections along the b axis were calculated, where the signs of the structure factors were determined from the contribution of the antimony atoms to the intensities. Apart from the eight-fold very high antimony peaks, there were found high peaks, belonging to two eight-fold point positions. These were assumed to be sulphur peaks.

Then a three-dimensional difference synthesis was calculated, where the contributions of the antimony and sulphur atoms had been subtracted from the F values. From this synthesis the parameters for the oxygen and carbon atoms were found. Patterson projections and electron density sections were calculated using a program by Liminga and Olovsson.[3]

All the positional parameters found by the procedure described above were then, together with the individual isotropic temperature factors and the scale factors, finally refined by the method of least squares.

The function $\sum w(|F_o|-|F_c|)^2$ was minimized by means of a block-diagonal least-squares program.[4] The weighting factor used was $w = (a + |F_o| + c|F_c|^2)^{-1}$ (Cruickshank[5]). The values of the constants a and c applied for all the calculations were $a = 2|F_o|_{min}$, $c = 2(|F_o|_{max})^{-1}$. The scattering factors for antimony, sulphur, and oxygen were taken from *International Tables for X-ray Crystallography*[6] and for carbon from Hanson, Herman, Lea and Skillman.[7]

The final discrepancy factor, $R = \sum(|F_o|-|F_c|)(\sum F_o)^{-1} \times 100$, was 12.2. Only the observed reflections were considered. The largest coordinate shift in the last cycle of refinement was 0.00012 Å which is 0.006 σ. The largest shift in the temperature factors was 0.07 σ.

The final three-dimensional electron density map is illustrated in Fig. 1. The sections are taken through the centre of each atom. The electron density sections through the oxygen and carbon atoms are calculated from the difference syntheses where the antimony and the sulphur atoms are not included.

The observed and calculated structure factors are listed in Table 1, the final values of the coordinates and temperature factors together with their standard deviations in Table 2.

Table 1. Observed and calculated structure factors.

h	k	l	F_o	F_c
0	0	0	–	1112
0	0	2	8	-9
0	0	4	436	-357
0	0	6	92	80
0	0	8	48	53
0	0	10	58	-65
0	0	12	68	-60
0	0	14	34	37
0	1	1	166	-163
0	1	3	92	64
0	1	5	176	158
0	1	7	80	-84
0	1	9	34	-32
0	1	11	114	103
0	2	0	106	-98
0	2	4	32	24
0	2	6	40	-32
0	2	8	22	16
0	3	1	110	116
0	3	3	24	-9
0	3	5	198	-176
0	3	7	58	60
0	3	9	54	50
0	3	11	106	-94
0	4	0	424	-547
0	4	4	254	251
0	4	6	62	-54
0	4	8	102	-101
0	4	10	60	48
0	4	12	74	72
0	5	1	182	186
0	5	3	198	-178
0	5	5	102	-92
0	5	7	100	104
0	5	11	78	-70
0	6	6	70	60
0	7	1	110	-118
0	7	3	118	119
0	7	5	90	98
0	7	7	58	-61
0	7	11	50	41
0	8	0	180	161
0	8	4	128	-136
0	8	8	98	105
0	8	12	52	-61
0	9	1	116	-110
0	9	3	102	99
0	9	5	66	59
0	9	7	82	-81
0	9	11	46	53
0	10	0	72	85
0	10	8	28	-31
0	11	1	56	56
0	11	3	102	-111
0	11	7	60	64
0	12	0	90	-91
0	12	4	74	79
0	12	8	50	-58
0	13	1	60	63
0	13	3	20	-22
0	13	5	42	-49
0	13	7	26	25
0	14	0	42	-44
0	15	1	20	-23
0	15	3	26	42
1	1	-12	60	56
1	1	-10	50	-58
1	1	-8	132	-123
1	1	-6	150	147
1	1	-4	110	104
1	1	-2	300	-309
1	1	0	86	-98
1	1	2	274	284
1	1	4	34	30
1	1	6	248	-206
1	1	8	32	38
1	1	10	86	88
1	2	-13	112	-103
1	2	-9	130	131
1	2	-7	46	-43
1	2	-5	232	-220
1	2	-3	124	113
1	2	-1	210	325
1	2	1	216	-228
1	2	3	276	-266
1	2	5	86	80
1	2	7	142	146
1	2	9	44	-41
1	2	11	60	-59
1	3	-14	34	37
1	3	-12	62	62
1	3	-10	54	-54
1	3	-8	134	-125
1	3	-6	100	89
1	3	-4	60	-51
1	3	-2	238	-262
1	3	0	154	172
1	3	2	248	251
1	3	4	134	112
1	3	6	160	-156
1	3	8	66	-66
1	3	10	84	82
1	4	-9	28	25
1	4	-7	66	61
1	4	-5	24	-18
1	4	-3	58	-46
1	4	-1	110	114
1	4	1	64	-59
1	4	3	118	-109
1	4	5	42	36
1	4	7	36	-31
1	4	11	54	44
1	5	-12	58	-45
1	5	-10	60	55
1	5	-8	88	82
1	5	-6	110	-103
1	5	-4	84	-75
1	5	-2	206	202
1	5	0	102	102
1	5	2	212	-232
1	5	4	56	-23
1	5	6	150	148
1	5	10	62	-60
1	6	-13	66	74
1	6	-9	120	-123
1	6	-5	160	165
1	6	-3	52	-45
1	6	-1	246	-259
1	6	1	192	178
1	6	3	256	265
1	6	5	146	-130
1	6	7	102	-91
1	6	9	66	66
1	7	-12	46	-43
1	7	-10	62	63
1	7	-8	82	79
1	7	-6	72	-69
1	7	-4	44	44
1	7	-2	130	132
1	7	0	92	-90
1	7	2	138	-137
1	7	4	104	-99
1	7	6	82	82
1	7	8	62	69
1	7	10	58	-60
1	8	-7	42	-41
1	8	-1	60	-50
1	8	1	60	55
1	8	3	62	59
1	9	-10	62	-55
1	9	-8	38	-34
1	9	-6	60	62
1	9	-4	26	24
1	9	-2	100	-92
1	9	0	34	-36
1	9	2	106	98
1	9	4	36	31
1	9	6	60	-57
1	9	10	48	39
1	10	-9	76	80
1	10	-7	24	14
1	10	-5	96	-95
1	10	-3	52	47
1	10	-1	154	146
1	10	1	138	-131
1	10	3	154	-165
1	10	5	98	100
1	10	7	70	67
1	10	9	46	-46
1	11	-8	44	-40
1	11	-6	62	52
1	11	-2	74	73
1	11	2	104	90
1	11	4	38	39
1	11	6	60	-61
1	13	-6	38	-40
1	13	-2	44	36
1	13	2	32	-30
1	13	6	26	20
1	14	-5	48	56
1	14	-3	32	-29
1	14	-1	60	-61
1	14	1	40	43
1	14	3	52	54
1	14	5	26	-36
1	15	-2	40	46
1	15	2	42	-62
2	0	-14	46	48
2	0	-12	70	-62
2	0	-10	202	-190
2	0	-8	122	108
2	0	-6	398	319
2	0	-4	34	-21
2	0	-2	66	-106
2	0	0	248	301
2	0	2	136	123
2	0	4	384	-429
2	0	6	86	-80
2	0	8	196	195
2	1	-11	110	94
2	1	-9	30	20
2	1	-7	204	-136
2	1	-5	24	11
2	1	-3	156	172
2	1	-1	110	-139
2	1	1	130	-136
2	1	3	88	88
2	1	5	218	237
2	1	7	78	15
2	1	9	108	-98
2	2	-10	58	13
2	2	-6	50	-51
2	2	0	66	-37
2	2	4	48	42
2	2	4	46	37
2	3	-11	102	-95
2	3	-9	26	20
2	3	-7	150	142
2	3	-5	116	12
2	3	-3	130	-122
2	3	-1	32	39
2	3	1	110	116
2	3	3	96	-86
2	3	5	214	-132
2	3	7	54	-23
2	3	9	88	84
2	3	13	30	-37
2	4	-14	38	-46
2	4	-12	76	65
2	4	-10	120	126
2	4	-8	110	-106
2	4	-6	206	-199
2	4	-4	86	81
2	4	-2	150	159
2	4	0	220	-256
2	4	2	154	-154
2	4	4	260	278
2	4	6	66	65
2	4	8	138	-138
2	5	-11	76	-72
2	5	-9	86	-78
2	5	-7	186	188
2	5	-5	24	3
2	5	-3	210	-196
2	5	-1	138	137
2	5	1	178	174
2	5	3	92	-81
2	5	5	184	-194
2	5	7	42	36
2	5	9	88	86
2	5	11	36	-29
2	6	-6	36	39
2	6	-4	54	-55
2	6	-2	30	-32
2	6	0	72	75
2	7	-11	56	67
2	7	-7	88	-93
2	7	-3	116	115
2	7	1	100	-101
2	7	3	64	62
2	7	5	102	108
2	7	7	36	-34
2	7	9	58	-60
2	8	-12	60	-57
2	8	-10	70	-63
2	8	-8	78	75
2	8	-6	74	75
2	8	-4	86	-80
2	8	-2	108	-98
2	8	0	162	158
2	8	2	104	96
2	8	4	124	-131
2	8	6	48	-55
2	8	8	76	78
2	9	-11	44	47
2	9	-9	44	39
2	9	-7	114	-108
2	9	-3	136	134
2	9	-1	56	-54
2	9	1	132	-130
2	9	3	56	53
2	9	5	94	109
2	9	9	54	-46
2	10	-4	26	36
2	10	4	44	-49
2	11	-7	64	57
2	11	-3	114	-110
2	11	1	100	94
2	11	5	54	-51
2	11	7	38	42
2	12	-8	34	-40
2	12	-6	52	-49
2	12	-4	56	53
2	12	-2	54	43
2	12	0	80	-84
2	12	2	34	-39
2	12	4	74	74
2	12	6	36	38
2	13	-7	44	50
2	13	-3	44	-36
2	13	1	52	49
2	13	5	48	-57
3	1	-12	40	47
3	1	-8	182	-180
3	1	-6	18	16
3	1	-4	250	290
3	1	-2	196	-253
3	1	0	244	-274
3	1	2	262	234
3	1	4	108	96
3	1	6	112	-112
3	1	8	50	-46
3	1	10	50	50
3	2	-13	90	-94
3	2	-11	40	-42
3	2	-9	192	195
3	2	-7	18	16
3	2	-5	144	-156
3	2	-3	114	-100
3	2	-1	74	78
3	2	1	26	24
3	2	3	328	-300
3	2	5	38	30
3	2	7	204	207
3	2	13	38	5
3	3	-12	54	52
3	3	-10	86	-80
3	3	-8	174	-170
3	3	-6	68	71
3	3	-4	214	238
3	3	-2	38	-17
3	3	0	122	-136
3	3	2	104	100
3	3	4	78	63
3	3	6	132	-123
3	3	8	90	-80
3	3	10	58	51
3	3	12	34	35
3	4	-13	26	29
3	4	-7	54	49
3	4	-5	40	-30
3	4	-3	80	-73
3	4	-1	50	49
3	4	1	72	-57
3	4	3	66	-55
3	4	5	98	85
3	4	7	34	31
3	5	-12	40	-48
3	5	-8	146	143
3	5	-4	180	-173
3	5	-2	146	143
3	5	0	142	141
3	5	2	154	-137
3	5	4	124	-72
3	5	6	118	109
3	5	8	58	44
3	5	10	60	-58
3	6	-13	42	42
3	6	-11	38	38
3	6	-9	122	-120
3	6	-7	80	-74
3	6	-5	188	214
3	6	-3	152	125
3	6	-1	214	-202
3	6	1	50	26
3	6	3	230	226
3	6	5	64	-53
3	6	7	166	-153
3	7	-12	44	-40
3	7	-10	70	79
3	7	-8	96	103
3	7	-6	72	-81
3	7	-4	112	-126
3	7	0	50	44
3	7	2	50	-40
3	7	4	84	-68
3	7	6	92	81
3	7	8	94	86
3	7	10	46	-35
3	8	-3	32	39
3	8	5	48	-55
3	8	7	42	-36
3	9	-12	36	39
3	9	-10	36	-31
3	9	-8	68	-63
3	9	-6	58	44
3	9	-4	54	46
3	9	-2	42	-40
3	9	0	62	-49
3	9	4	76	57
3	9	6	68	-61
3	9	8	62	-48
3	9	10	38	39
3	10	-11	24	-26
3	10	-9	66	76
3	10	-7	72	75
3	10	-5	154	-146
3	10	-3	92	-94
3	10	-1	150	139
3	10	3	120	-126
3	10	5	56	47
3	10	7	88	92
3	11	-10	20	-22
3	11	-8	56	-64
3	11	-6	34	33
3	11	-4	82	81
3	11	-2	32	-36
3	11	0	32	-35
3	11	2	58	53
3	11	4	42	40
3	11	6	46	-47
3	11	8	40	-45
3	12	-9	16	21
3	12	-5	26	15
3	12	-1	28	-26
3	12	7	16	17
3	13	-8	16	21
3	13	-6	26	-30
3	13	0	32	34
3	13	4	36	-39
3	13	6	14	19
3	14	-5	42	59
3	14	-3	18	23
3	14	-1	46	-68
3	14	3	42	61
3	15	-2	14	22
3	15	0	20	25
4	0	-14	46	39
4	0	-10	86	-98
4	0	-8	58	52
4	0	-6	314	303
4	0	-4	16	-8
4	0	-2	390	-550
4	0	0	28	27
4	0	2	300	293
4	0	4	218	-186
4	0	6	92	-92
4	0	8	154	150
4	1	-11	64	60
4	1	-9	40	47
4	1	-7	158	-158
4	1	-5	144	-158
4	1	-3	170	193
4	1	-1	126	145
4	1	1	58	-60
4	1	3	18	11
4	1	5	100	93
4	1	7	50	13
4	1	9	114	-99
4	2	-10	26	-7
4	2	-8	22	19
4	2	-6	28	-23
4	2	-4	44	-43
4	2	-2	86	88
4	2	0	48	38
4	2	2	32	-26
4	2	4	36	32
4	2	6	48	-36
4	2	10	100	20
4	3	-13	44	48
4	3	-11	54	-54
4	3	-9	42	-32
4	3	-7	150	164
4	3	-5	50	49
4	3	-3	194	-178
4	3	-1	66	-66
4	3	1	22	15
4	3	3	52	37
4	3	5	92	-81
4	3	7	52	-47
4	3	9	100	94
4	3	11	28	24
4	4	-14	38	-48
4	4	-10	112	109
4	4	-8	48	-40
4	4	-6	226	-225
4	4	-4	46	45
4	4	-2	266	302
4	4	0	58	-59
4	4	2	248	-213
4	4	4	144	121
4	4	6	136	121
4	4	8	120	-101

Table 1. Continued.

h	k	l	F_o	F_c
4	4	10	88	-80
4	4	12	24	30
4	5	-13	40	23
4	5	-9	58	-66
4	5	-7	98	107
4	5	-5	190	215
4	5	-3	154	-155
4	5	-1	164	-171
4	5	1	158	159
4	5	3	28	-25
4	5	5	142	-119
4	5	7	54	56
4	5	9	82	72
4	6	-6	54	-48
4	6	-4	34	33
4	6	-2	55	-43
4	6	2	50	[illegible]
4	6	4	42	-36
4	7	-9	40	44
4	7	-7	86	-98
4	7	-5	72	-65
4	7	-3	110	118
4	7	-1	66	68
4	7	1	96	-99
4	7	3	54	-40
4	7	5	94	92
4	7	9	54	-51
4	8	-10	91	99
4	8	-8	40	41
4	8	-6	156	138
4	8	-4	42	-41
4	8	-2	120	-109
4	8	0	26	22
4	8	2	102	106
4	8	6	100	-96
4	8	8	62	47
4	9	-9	60	51
4	9	-7	78	-70
4	9	-5	108	-107
4	9	-3	108	106
4	9	-1	82	77
4	9	1	116	-108
4	9	5	104	94
4	9	9	56	-54
4	11	-9	32	-47
4	11	-5	52	47
4	11	-3	70	-68
4	11	-1	32	-28
4	11	1	152	108
4	11	5	87	-79
4	12	-6	62	-78
4	12	-2	78	76
4	12	2	64	-62
4	12	6	40	44
4	13	-7	40	56
4	13	-3	50	-58
4	13	1	34	24
4	13	5	30	-33
5	1	-14	30	-38
5	1	-12	32	44
5	1	-10	52	54
5	1	-8	120	-142
5	1	-6	78	-72
5	1	-4	194	209
5	1	-2	22	15
5	1	0	196	-205
5	1	2	46	37
5	1	4	166	146
5	1	6	98	-89
5	1	8	106	-89
5	1	10	50	43
5	2	-15	46	61
5	2	-11	74	-71
5	2	-9	106	105
5	2	-7	108	119
5	2	-5	218	-247
5	2	-3	238	-263
5	2	-1	140	147
5	2	1	182	171
5	2	3	112	-90
5	2	5	112	-97
5	2	7	102	63
5	2	9	38	42
5	3	-14	32	-35
5	3	-12	40	40
5	3	-8	64	-89
5	3	-4	200	222
5	3	-2	32	-34
5	3	0	202	-197
5	3	2	38	-30
5	3	4	92	74
5	3	6	44	-31
5	3	8	106	-70
5	4	-9	36	-26
5	4	-7	26	23
5	4	-5	40	-40
5	4	-3	58	-47
5	4	-1	62	50
5	4	1	78	77
5	4	5	30	13
5	5	-8	98	115
5	5	-6	38	39
5	5	-4	166	-179
5	5	0	190	202
5	5	4	128	-107
5	5	6	46	47
5	5	8	76	57
5	6	-11	82	88
5	6	-9	50	-47
5	6	-7	94	-101
5	6	-5	132	147
5	6	-3	174	186
5	6	-1	146	-151
5	6	1	174	-165
5	6	3	142	123
5	6	5	64	55
5	6	7	132	-103
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5	7	-8	56	56
5	7	-6	40	-37
5	7	-4	124	-140
5	7	-2	46	21
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5	7	4	50	-36
5	7	8	70	54
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5	8	-3	52	47
5	8	-1	34	-37
5	8	1	56	-54
5	9	-8	58	-61
5	9	-4	82	89
5	9	-2	56	-10
5	9	0	116	-107
5	9	4	64	45
5	10	-11	38	-55
5	10	-7	62	66
5	10	-5	78	-84
5	10	-3	110	-121
5	10	-1	104	99
5	10	1	116	115
5	10	3	96	-92
5	10	5	38	-30
5	11	-8	44	-47
5	11	-4	80	85
5	11	0	74	-73
5	11	4	36	41
5	13	0	36	35
5	14	-5	22	31
5	14	-3	34	48
5	14	-1	42	-40
5	14	1	32	-38
6	0	-14	40	40
6	0	-12	106	106
6	0	-10	64	-76
6	0	-8	146	-151
6	0	-6	168	163
6	0	-4	38	35
6	0	-2	312	-355
6	0	0	110	-98
6	0	2	306	290
6	0	4	70	59
6	0	6	116	-107
6	0	10	50	51
6	1	-13	66	-77
6	1	-9	82	77
6	1	-7	62	-63
6	1	-5	92	-79
6	1	-3	106	138
6	1	-1	128	157
6	1	1	90	-85
6	1	3	182	-160
6	1	5	64	33
6	1	7	72	59
6	1	9	48	-42
6	2	-10	40	-19
6	2	-8	46	35
6	2	-6	48	2
6	2	-4	18	13
6	2	-2	54	57
6	2	0	54	-44
6	2	2	52	-42
6	2	4	48	-3
6	3	-13	82	79
6	3	-9	72	-71
6	3	-7	48	48
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6	3	7	64	-55
6	4	-14	44	-49
6	4	-12	78	-74
6	4	-10	82	90
6	4	-8	82	90
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6	4	6	108	97
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6	4	10	62	-59
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6	5	-11	48	48
6	5	-9	68	-72
6	5	-7	34	40
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6	5	-3	160	-154
6	5	-1	182	-175
6	5	1	106	101
6	5	3	122	125
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6	5	7	56	-44
6	5	9	58	44
6	6	-8	66	-45
6	6	-6	56	3
6	6	-2	30	-33
6	6	0	44	41
6	6	2	34	25
6	6	4	36	-20
6	7	-9	42	57
6	7	-5	66	-109
6	7	-3	46	52
6	7	-1	106	102
6	7	1	70	-63
6	7	3	74	-66
6	7	7	54	41
6	8	-10	58	-72
6	8	-6	84	94
6	8	-4	36	22
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6	8	0	82	-65
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6	9	-9	56	59
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6	9	-1	114	121
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6	9	3	62	-67
6	11	-9	40	-49
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6	12	-6	44	-56
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6	12	2	60	-68
6	13	-1	46	-55
7	1	-10	88	87
7	1	-8	48	-35
7	1	-6	150	-143
7	1	-4	156	172
7	1	-2	120	152
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7	1	4	114	95
7	1	8	50	-46
7	2	-11	126	-140
7	2	-7	94	93
7	2	-5	54	-53
7	2	-3	102	-109
7	2	-1	38	36
7	2	1	134	194
7	2	3	42	-37
7	2	5	178	-151
7	3	-10	90	91
7	3	-8	66	-52
7	3	-6	78	-73
7	3	-4	100	102
7	3	-2	40	36
7	3	0	182	-177
7	3	2	102	-82
7	3	4	116	93
7	4	-7	66	6
7	4	-5	62	73
7	4	-3	36	-28
7	4	-1	42	32
7	4	1	66	59
7	4	3	62	-61
7	5	-10	68	-76
7	5	-6	100	103
7	5	-4	96	-101
7	5	-2	86	-76
7	5	0	160	155
7	5	2	62	50
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7	5	6	64	-7
7	5	8	68	51
7	6	-11	52	95
7	6	-9	38	37
7	6	-7	106	-125
7	6	-3	156	160
7	6	-1	60	-51
7	6	1	184	-178
7	6	3	58	60
7	6	5	134	112
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7	7	-4	62	-59
7	7	0	96	90
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7	7	4	70	-56
7	7	6	80	-67
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7	8	1	46	-39
7	8	3	50	29
7	9	0	58	-46
7	9	4	68	49
7	10	-7	86	89
7	10	-3	92	-109
7	10	-1	58	46
7	10	1	114	115
7	10	3	54	-49
7	11	-4	54	49
7	11	0	74	-66
8	0	-12	94	96
8	0	-10	44	-49
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8	0	-4	156	158
8	0	-2	92	-94
8	0	0	144	-131
8	0	2	250	205
8	0	4	122	100
8	0	6	166	-140
8	0	8	64	-46
8	1	-9	[illegible]	101
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8	1	-5	132	-134
8	1	-3	44	-38
8	1	-1	100	97
8	1	1	58	-45
8	1	3	98	-89
8	1	7	76	65
8	3	-13	42	54
8	3	-11	24	12
8	3	-9	86	-95
8	3	-5	102	116
8	3	-1	62	-57
8	3	1	26	24
8	3	3	84	75
8	3	7	72	-65
8	4	-12	62	-83
8	4	-10	38	46
8	4	-8	112	146
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8	4	-2	100	106
8	4	0	104	115
8	4	2	154	-135
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8	5	-7	88	-111
8	5	-5	82	98
8	5	-3	42	48
8	5	-1	128	-136
8	5	1	58	45
8	5	3	116	114
8	5	7	56	-52
8	6	-4	62	52
8	6	-2	42	1
8	6	0	56	-32
8	7	-5	54	-68
8	7	-1	66	72
8	7	3	90	-85
8	7	7	38	47
8	8	-2	66	-68
8	8	0	74	-62
8	8	2	52	54
8	9	-1	110	101
8	9	3	90	-84
8	11	3	54	62
8	13	-1	28	-30
9	1	-10	84	97
9	1	-6	124	-140
9	1	-2	118	110
9	1	0	98	-89
9	1	2	99	-94
9	1	4	[illegible]	77
9	1	[illegible]	86	56
9	2	-11	92	[illegible]
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9	2	-7	132	154
9	2	-5	80	92
9	2	-3	84	-89
9	2	-1	56	-45
9	2	1	106	90
9	[illegible]	[illegible]	64	[illegible]
9	2	5	114	-113
9	3	-10	80	81
9	[illegible]	[illegible]	[illegible]	[illegible]
9	[illegible]	[illegible]	116	-140
9	[illegible]	-4	74	[illegible]
9	[illegible]	[illegible]	65	95
9	[illegible]	[illegible]	44	-39
9	[illegible]	2	36	[illegible]
9	[illegible]	4	72	-53
9	[illegible]	[illegible]	44	45
9	[illegible]	[illegible]	54	-31
9	[illegible]	[illegible]	[illegible]	55
9	[illegible]	[illegible]	54	[illegible]
9	4	-1	50	-15
9	4	[illegible]	54	[illegible]
9	[illegible]	-10	[illegible]	-77
9	[illegible]	[illegible]	94	110
9	[illegible]	[illegible]	112	-105
9	[illegible]	[illegible]	54	50
9	[illegible]	[illegible]	[illegible]	[illegible]
9	[illegible]	4	[illegible]	-51
9	[illegible]	[illegible]	59	-37
9	[illegible]	-11	46	56
9	[illegible]	-9	58	[illegible]
9	6	[illegible]	98	-118
9	6	[illegible]	74	-80
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9	6	-1	74	[illegible]
9	6	[illegible]	[illegible]	[illegible]
9	6	5	126	102
9	[illegible]	-6	66	[illegible]
9	7	-2	74	-61
9	7	4	42	-38
9	7	6	32	-40
9	9	-2	68	61
9	10	-1	76	-42
9	10	1	84	66
9	11	-2	38	41
10	0	-14	32	-37
10	0	-12	42	46
10	0	-10	24	34
10	0	-8	172	-175
10	0	-4	228	227
10	0	-2	24	22
10	0	0	170	-145
10	0	4	72	64
10	0	6	58	-55
10	1	-9	50	55
10	1	-7	56	43
10	1	-5	72	-65
10	1	-3	100	-98
10	1	-1	56	49
10	1	1	66	52
10	1	3	88	-64
10	3	-11	28	35
10	3	-9	50	-53
10	3	-5	54	61
10	3	-3	76	57
10	3	-1	48	-29
10	3	1	50	-51
10	3	3	40	40
10	3	5	22	25
10	3	7	34	-32
10	4	-12	50	-5
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10	4	-4	140	-149
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10	4	4	38	-71
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10	5	-5	46	65
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10	5	-1	72	-70
10	5	3	94	75
10	6	-4	48	15
10	6	0	48	-39
10	7	-5	48	-59
10	7	-3	76	-67
10	8	0	76	-55
10	9	-3	78	-76
10	9	-1	64	48
10	9	3	54	-54
11	1	-12	42	-25
11	1	-8	46	41
11	1	-6	80	-90
11	1	-4	74	-78
11	1	-2	112	127
11	1	0	58	36
11	1	2	118	-81
11	2	-9	62	-72
11	2	-7	86	85
11	2	-5	94	113
11	2	-3	72	-61
11	2	-1	92	-86
11	2	3	46	51
11	3	-10	30	39
11	3	-6	64	-75
11	3	-4	28	-24
11	3	-2	102	114
11	3	0	58	50
11	3	2	56	-58
11	3	6	30	29
11	5	-6	56	73
11	5	-4	42	39
11	5	-2	104	-93
11	5	2	100	62
11	6	-9	50	72
11	6	-5	90	-108
11	6	-3	52	53
11	6	-1	94	94
11	6	1	58	-41
11	7	-2	108	-63
11	10	-3	48	-43
11	10	-1	72	-73
11	10	1	36	38
12	0	-10	78	85
12	0	-8	29	-31
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12	0	-4	104	113
12	0	-2	46	40
12	0	0	164	-135
12	0	2	52	-46
12	0	4	74	67
12	1	-7	42	52
12	1	-3	88	-79
12	1	1	82	69
12	3	-11	34	51
12	3	-9	28	[illegible]
12	3	-7	40	-49
12	3	-3	54	52
12	3	[illegible]	70	-48
12	4	-10	44	-37
12	4	-8	36	45

Table 1. Continued.

h	k	l	F_o	F_c	h	k	l	F_o	F_c	h	k	l	F_o	F_c	h	k	l	F_o	F_c	h	k	l	F_o	F_c
					12	5	-3	82	92	13	1	0	93	59	13	5	0	56	-42	14	0	-2	86	77
					12	5	1	100	-83	13	2	3	82	67	13	6	-5	42	-75	14	0	0	68	-48
12	4	-6	50	58	12	7	1	52	44	13	3	-8	40	52	13	6	-1	104	75	14	1	1	72	39
12	4	-4	108	-100	12	8	0	66	-46	13	3	-2	58	34	13	7	0	32	-26	14	4	-10	38	-64
12	4	-2	48	-45	13	1	-8	68	66	13	3	0	50	36	14	0	-10	76	78	14	4	-6	66	68
12	4	0	124	93	13	1	-4	36	-48	13	5	-8	42	-51	14	0	-6	108	-101	14	4	-2	58	-57
12	5	-7	42	-45	13	1	-2	64	65	13	5	-2	46	-50										

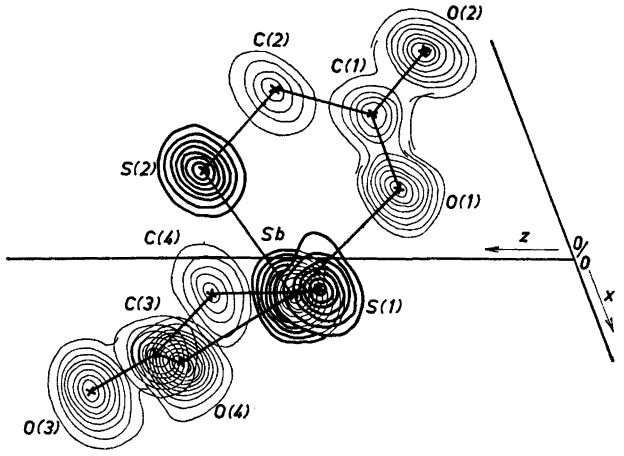

Fig. 1. A composite drawing of the final electron density map, viewed along the *b*-axis. Contours are at intervals of about 1 $eÅ^{-3}$ for carbon and oxygen, 2 $eÅ^{-3}$ for sulphur, and 6 $eÅ^{-3}$ for antimony. Zero contours are omitted. The crosses indicate final x and z coordinates.

The calculations were performed with the aid of the digital computers BESK and FACIT EDB of the *Computer Division of the National Swedish Rationalization Agency.*

Table 2. Final positional and thermal parameters and their standard deviations.

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	B Å²	$\sigma(B)$ Å²
Sb	0.04850	0.00015	0.12399	0.00015	0.36918	0.00015	3.03	0.03
S(1)	0.0435	0.0006	0.3227	0.0007	0.3407	0.0006	3.37	0.13
S(2)	−0.1140	0.0007	0.1105	0.0006	0.4382	0.0007	3.56	0.13
O(1)	−0.0956	0.0018	0.1097	0.0018	0.1901	0.0018	4.23	0.42
O(2)	−0.2776	0.0018	0.0903	0.0018	0.0924	0.0018	4.03	0.40
O(3)	0.1816	0.0017	0.3144	0.0018	0.6865	0.0017	3.95	0.39
O(4)	0.1383	0.0015	0.1826	0.0016	0.5549	0.0015	3.11	0.33
C(1)	−0.1957	0.0023	0.0963	0.0023	0.1891	0.0024	3.29	0.50
C(2)	−0.2299	0.0031	0.0828	0.0033	0.2991	0.0031	5.21	0.72
C(3)	0.1311	0.0021	0.2851	0.0021	0.5802	0.0020	2.62	0.42
C(4)	0.0505	0.0026	0.3601	0.0026	0.4843	0.0026	4.10	0.59

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The structure of one molecule is shown in Fig. 2 and the bond distances and angles and their standard deviations are given in Fig. 3 and Table 3.

The molecule is a chelate, where the organic part forms two five-membered rings with the antimony atom. The antimony atom has two oxygen and two sulphur atom neighbours in a one-sided four-fold coordination (Fig. 4). The coordination can be described as a distorted trigonal bipyramid where the two axial apices are occupied by oxygen atoms and two of the equatorial apices are occupied by sulphur atoms. The third equatorial position can be assumed to belong to an unshared electron pair.[8] This coordination can be looked upon as being of the $TeCl_4$-type.[9] The deformation of the bipyramid is

Table 3a. Bond distances with standard deviations.

	d Å	$\sigma(d)$ Å
Sb—S(1)	2.426	0.008
Sb—S(2)	2.431	0.008
Sb—O(1)	2.304	0.022
Sb—O(4)	2.255	0.019
S(1)—C(4)	1.763	0.033
S(2)—C(2)	1.838	0.039
C(1)—O(1)	1.250	0.036
C(1)—O(2)	1.267	0.036
C(3)—O(4)	1.287	0.031
C(3)—O(3)	1.273	0.033
C(1)—C(2)	1.529	0.048
C(3)—C(4)	1.545	0.041
O(2)—O(2′)	2.539	0.044
O(3)—O(3′)	2.433	0.042

Table 3b. Bond angles with standard deviations.

	Angle	σ(°)
S(2)—Sb—O(1)	80.9°	0.6°
Sb—O(1)—C(1)	118.5	1.9
O(1)—C(1)—C(2)	124.6	2.7
C(1)—C(2)—S(2)	114.3	2.4
C(2)—S(2)—Sb	100.7	1.2
O(1)—C(1)—O(2)	120.4	2.6
C(1)—O(2)—O(2′)	116.0	1.9
S(1)—Sb—O(4)	79.1	0.5
Sb—O(4)—C(3)	119.1	1.6
O(4)—C(3)—C(4)	117.9	2.2
C(3)—C(4)—S(1)	113.0	1.9
C(4)—S(1)—Sb	97.2	1.1
O(4)—C(3)—O(3)	117.1	2.3
C(3)—O(3)—O(3′)	117.4	1.8

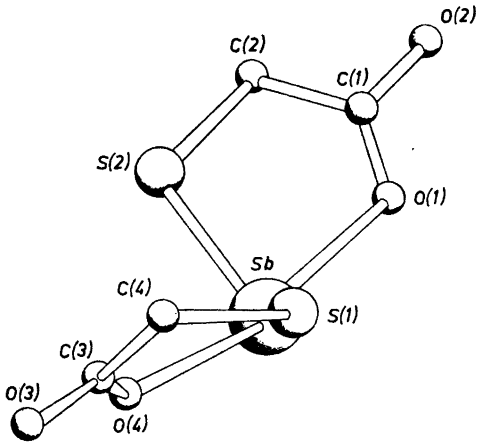

Fig. 2. The structure of one molecule viewed along the *b*-axis.

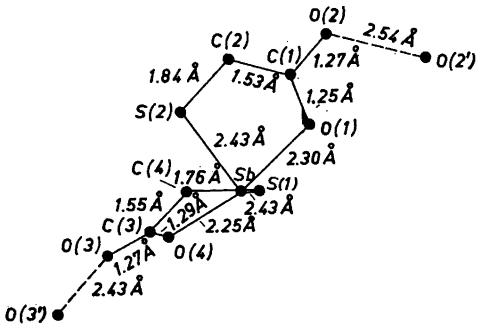

Fig. 3a. Bond distances.

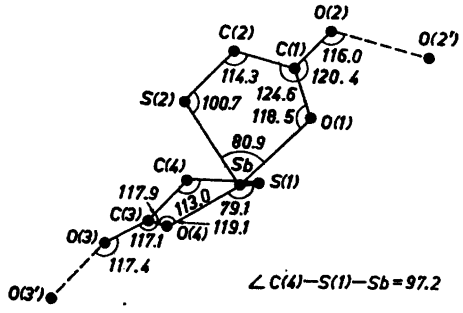

Fig. 3b. Bond angles.

in accordance with the repelling effect of the unshared electron pair on the sulphur and oxygen atoms. An analogous arrangement with four oxygen atoms linked to trivalent antimony was observed by Edstrand [10] in $Sb_4O_5Cl_2$, SbOCl,[11] $[Sb_8O_8(OH)_4][(OH)_{2-x}(H_2O)_{1+x}]Cl_{2+x}$,[12] and $Sb_8O_{10}(OH)_2I_2$,[13] and has later been found in β-Sb_2O_4 [14] and $SbNbO_4$.[15] It is also reported to exist in racemic ammonium antimonyl tartrate [16] and racemic potassium antimonyl tartrate.[17] A one-sided four-fold coordination for the metal atom has also been found for arsenic in potassium di-*o*-phenylenedioxyarsenate(III) [18] and for bismuth, *e.g.* in $BiSbO_4$.[19]

The shortest oxygen-oxygen distances between two molecules are 2.43 and 2.54 Å, respectively. Distances as short as these indicate hydrogen bonding. The two oxygen atoms are related by a centre of symmetry and by a twofold axis, respectively, these symmetry elements being present in the arrangement of the non-hydrogen atoms deduced above.

The method used in the present investigation does not allow a direct determination of the hydrogen atom positions to be made. It then seems to be of interest to find out if neutron diffraction or spectroscopic data reported in literature may help in deciding upon the probability of the hydrogen atom arrangement possessing *I*2/*a* or *Ia* symmetry. The latter case requires the hydrogen bridge to be asymmetric O—H···O. Such bonds are generally considerably longer than the ones found in the present case. On the other hand several authors have suggested symmetric bonding to be present in the shortest

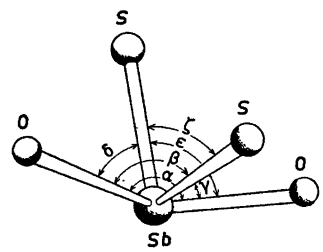

Fig. 4. The coordination around an antimony atom.
$\alpha = 157.6°$ $\beta = 88.1°$ $\gamma = 79.1°$
$\delta = 80.9°$ $\varepsilon = 82.7°$ $\zeta = 97.5°$.

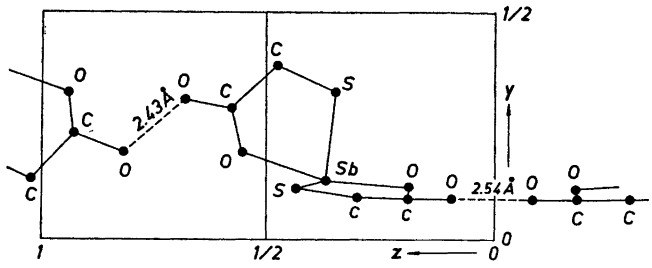

Fig. 5. Projection onto the *bc* plane of one molecular chain, showing the hydrogen bonds.

hydrogen bridges (2.4—2.5 Å). A symmetric bonding may either contain a hydrogen atom located at the centre or it may be that the hydrogen atom alternately occupies two equivalent points on either side of the centre position. The following structures may serve to illustrate the two kinds of symmetric hydrogen bonds.

Potassium hydrogen bisphenylacetate was the first compound in which a crystallographic symmetrical O···H···O bond was found.[20] The O···H···O distance which was first found to be 2.55 Å has later been redetermined to be 2.445 ± 0.004 Å.[21]

Neutron diffraction studies on this substance at room temperature[22] and at 120°K[23] make it possible to assume that in the hydrogen bond the hydrogen atom is located at the centre of the bond and not statistically on either side of the centre.

Potassium hydrogen maleate with O···H···O distance 2.44 Å[24] and potassium hydrogen chloromaleate with O···H···O distance 2.40 Å[25] have been studied by neutron diffraction. The hydrogen bonds were found to be at least statistically symmetric.

Blink, Hadži and Novak[26] have shown by spectroscopic methods that hydrogen bonds with O···H···O distances between 2.40 and 2.60 Å can have a potential curve of either a symmetric single minimum type or of a double minimum type. *E.g.*, in potassium hydrogen maleate and potassium hydrogen bis-phenylacetate they found the potential curve to be of the symmetric single minimum type. It is very plausible that also in antimony hydrogen bis-thioglycollate the hydrogen bonds are of one of the two symmetric types. But only neutron diffraction studies or spectroscopic measurements can confirm it.

Every molecule of antimony hydrogen bis-thioglycollate is joined to two other molecules by the hydrogen bonds forming endless zig-zag chains running perpendicular to the *b*-axis. The arrangement of the hydrogen bonds is given in Fig. 5. The interatomic distances and angles some of which are given in Table 3, are all plausible.

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REFERENCES

1. Klason, P. and Carlson, T. *Ber.* **39** (1906) 732.
2. Werner, P.-E. *IUCr World List of Crystallographic Computer Programs*, 2nd Ed., (1966), No. 6019.
3. Liminga, R. and Olovsson, I. *IUCr World List of Crystallographic Computer Programs*, 2nd Ed., (1966), Nos. 6014, 6015.
4. Åsbrink, S. and Brändén, C.-I. *IUCr World List of Crystallographic Computer Programs*, 2nd Ed., (1966), No. 6023.
5. Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. and Truter, M. R. *In Computing Methods and The Phase Problem in X-ray Crystal Analysis*, Oxford 1961, p. 32.
6. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1962, Vol. III.
7. Hanson, H. P., Herman, F., Lea, J. D. and Skillman, S. *Acta Cryst.* **17** (1964) 1040.
8. Pauling, L. *The Nature of the Chemical Bond*, Cornell Univ. Press, Ithaca, New York 1960, p. 181.
9. Stevenson, D. P. and Schomaker, V. *J. Am. Chem. Soc.* **62** (1940) 1267.
10. Edstrand, M. *Acta Chem. Scand.* **1** (1947) 178.
11. Edstrand, M. *Arkiv Kemi* **6** (1953) 89.
12. Edstrand, M. *Arkiv Kemi* **8** (1955) 257.
13. Edstrand, M. *Arkiv Kemi* **8** (1955) 279.
14. Rogers, D. and Skapski, A. C. *Proc. Chem. Soc.* **1964** 400.
15. Skapski, A. C. and Rogers, D. *Chem. Commun.* **1965** 611.
16. Kiosse, G. A., Golovastikov, N. J. and Belov, N. V. *Dokl. Akad. Nauk SSSR* **155** (1964) 545.
17. Grdenić, D. and Kamenar, B. *Acta Cryst.* **19** (1965) 197.
18. Skapski, A. C. *Chem. Commun.* **1966** 10.
19. Aurivillius, B. *Arkiv Kemi* **3** (1951) 153.
20. Speakman, J. C. *Nature* **162** (1948) 695.
21. Speakman, J. C. *Chem. Commun.* **1967** 32.
22. Bacon, G. E. and Curry, N. A. *Acta Cryst.* **10** (1957) 524.
23. Bacon, G. E. and Curry, N. A. *Acta Cryst.* **13** (1960) 717.
24. Peterson, S. W. and Levy, H. A. *J. Chem. Phys.* **29** (1958) 948.
25. Ellison, R. D. and Levy, H. A. *Acta Cryst.* **19** (1965) 260.
26. Blinc, R., Hadži, D. and Novak, A. *Z. Electrochem.* **64** (1960) 567.

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