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Crystal Structure of the 2:1 Addition Compound Antimony Triiodide-1,4-Dithiane

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The crystal structure of the 2:1 addition compound antimony triiodide-1,4-dithiane has been determined by three-dimensional X-ray crystallographic methods. The crystals have a melting point of 165°C and the density calculated from the lattice constants is 3.83 g cm^{-3}. The space group is $C2/c$ and the monoclinic unit cell containing four formula units has the lattice constants a = 13.578 (0.003) Å, b = 9.181 (0.006) Å, c = 16.820 (0.003) Å, β = 111.47 (0.02)°. The dithiane molecules are situated in centres of symmetry, the antimony triiodide molecules occupy general positions. Each antimony atom forms charge transfer bonds with two sulphur atoms of different molecules and two such "antimony bridges" link neighbouring dithiane molecules together, the result being the formation of endless chains of antimony triiodide and dithiane molecules in the crystal. The Sb—S bond distances are 3.274 (0.007) Å and 3.336 (0.005) Å. The intramolecular Sb—I distances after correcting for librational motion are 2.778 (0.0019) Å, 2.746 (0.0028) Å, and 2.767 (0.0031) Å. These distances are significantly longer than the value 2.719 (0.0015) Å found in electron diffraction investigation of antimony triiodide and supports the view that antimony in the triiodide molecule may act as an acceptor towards n donors.

When antimony triiodide forms addition compounds with n donor molecules, two types of bond may result; either iodine or antimony may be coordinated to the donor atom of the partner molecule. An example of the first type is found in the solid addition compound with sulphur (S_8) as the donor molecule.[1] Here all three iodine atoms belonging to a particular antimony triiodide molecule are linked to sulphur atoms. Rather short intermolecular I···Sb distances are, however, also observed and it appears very probable that the great stability of the sulphur compound depends not only on the I···S bonds but also to some extent on the interaction between antimony and iodine of neighbouring molecules. It would actually appear possible to isolate solid complexes of n donors with antimony triiodide in which the antimony atom is the centre of coordination of the acceptor molecule. For this reason the structure study has been extended to other addition compounds

containing antimony triiodide and in the present study 1,4-dithiane was chosen as the *n* donor molecule. Antimony triiodide was found to form a 1:1 and a 2:1 compound with dithiane, and in the present study a detailed X-ray examination of the latter compound has been undertaken. It might, however, be added that the 1:1 compound is not isostructural with the 1:1 compound between iodoform and dithiane which exhibit I—S bonds.[2] (The space group of the 1:1 compound antimony triiodide-dithiane derived from the X-ray extinctions can either be $P2_1$ or $P2_1/m$ and the monoclinic unit cell containing four formula units has the lattice constants $a = 7.21$ Å, $b = 15.63$ Å, $c = 14.14$ Å, $\beta = 93.0°$.)

EXPERIMENTAL

Crystals of the 2:1 compound were prepared by evaporating the solvent from a solution of antimony triiodide and dithiane in carbon disulphide. Since the crystals are somewhat volatile, the specimens selected for study were sealed in thin-walled glass capillaries. Oscillation and Weissenberg photographs indicated a monoclinic unit cell with the crystallographic *b*-axis along the needle axis. The unit cell dimensions were determined by means of zero-level *h0l* and *0kl* Weissenberg photographs prepared with unfiltered Cu*K*-radiation. Reflection patterns of BaF_2 were superimposed on these films for calibration purpose. The 2θ values (based on $a = 6.2001$ Å for BaF_2) for a total of 107 *h0l* and *0kl* reflections were used in a least squares refinement of the lattice parameters. With the wavelengths for Cu*K*-radiation taken as $\alpha_1 = 1.54050$ Å, $\alpha_2 = 1.54434$ Å, $\alpha = 1.5418$ Å, and $\beta = 1.39217$ Å the following results were obtained (e.s.d. in parentheses):

$a = 13.578$ (0.003) Å, $b = 9.181$ (0.006) Å,
$c = 16.820$ (0.003) Å, $\beta = 111.47$ (0.02)°.

The melting point was found equal to 165°C and the density measured using the flotation method was 3.70 g cm^{-3} leading to the value $Z = 4$ for the number of molecules in the unit cell. The density calculated from the lattice constants is 3.83 g cm^{-3}. The space group derived from the X-ray extinctions can either be *Cc* or *C2/c*. The latter was assumed to be the correct one and this was eventually confirmed in the course of the structure determination. The X-ray material collected consisted of integrated zero layer Weissenberg diagrams for the *h0l* and *0kl* zones and a set of equi-inclination integrated Weissenberg diagrams with rotation about the *b*-axis ($k = 1$ to 8). Mo*K*α-radiation was employed and the cross section of the crystals selected for the X-ray work were approximately 0.08×0.08 mm^2. The multiple film method was used with four films (Ilford Industrial G) separated by tin-foils. The intensities of the reflections were measured photometrically. The intensities were corrected for Lorentz and polarization effects in the usual way. No general corrections for absorption or secondary extinction were applied. 1197 independent reflections were obtained. The number of reflections compatible with the actual experimental conditions is 1947.

DETERMINATION AND REFINEMENT OF THE STRUCTURE

Approximate coordinates of the iodine and antimony atoms were derived from Patterson projections along the *b*- and *a*-axis. Fourier maps based on the observed structure factors with signs calculated from contribution of the heavy atoms were worked out and the coordinates refined in the usual way until no further change of signs occurred. The coordinates thus obtained were used in difference syntheses in which the contributions from the heavy atoms were subtracted. In the resulting Fourier maps peaks belonging to the carbon and sulphur atoms were present. The positional parameters resulting from the

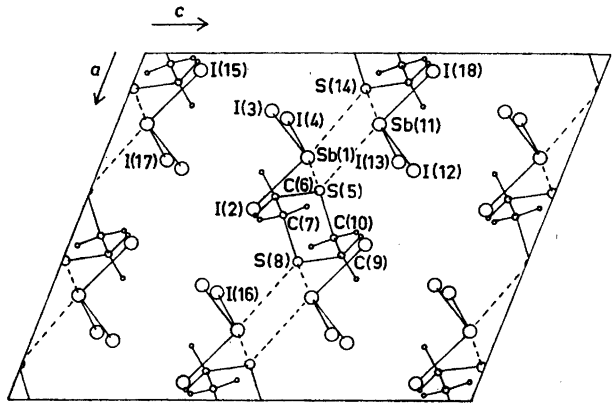

Fig. 1. A view of the structure along the *b*-axis.

two-dimensional analysis were then refined by three-dimensional least squares procedure with the full set of intensity data. The least squares refinement was made on the UNIVAC 1107 by use of ACA Computer Program No. 317 (UCLALS-1) written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood. This program minimizes the weighted sum of the squares of the quantity $(K \cdot F_o - G \cdot F_c)$ by a full-matrix routine were K and G are scale factors. G is one of the adjustable parameters. The weighting scheme used was that of Hughes [3] with $4 \cdot F_o$ (min) = 90. The program calculates the standard deviations from the inverse matrix of the normal equations. The isotropic temperature factors are of the form $\exp(-B \sin^2\theta/a^2)$ and the anisotropic of the form $\exp-(h^2B_{11} + k^2B_{22} + l^2B_{33} + hkB_{12} + hlB_{13} + klB_{23})$. Isotropic tempera-

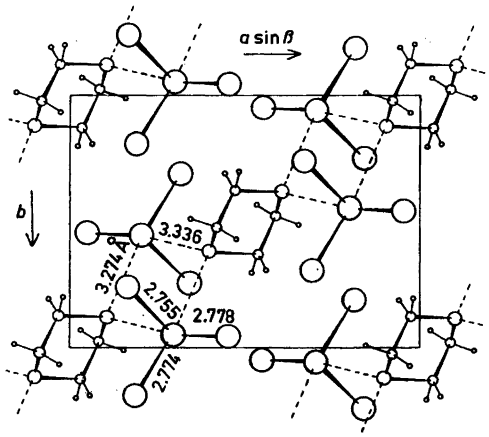

Fig. 2. A view of the structure along the c-axis. For simplicity only the chains in the direction [1$\bar{1}$0] are shown in the figure. The chains in the [110] direction are related to these by glide planes.

ture factors were originally assigned to all the atoms and were refined with the positional parameters in two cycles. In these cycles the R-factor was reduced from 0.19 to 0.15. Six more cycles of least squares refinements were then carried out after introducing anisotropic temperature factors for Sb, I, and S. The B values of C were refined isotropically. In the last two cycles the hydrogen atoms were included in the structure factor calculations, but hydrogen parameters were not refined. Positions of the hydrogen atoms were calculated from the position of the carbon and sulphur atoms with an assumed C—H distance of 1.08 Å. The B values of hydrogen were set equal to 2.5 Å^2. Unobserved reflections were not included in the R value or the least squares refinements. The interlayer scalings were adjusted by use of the ratios $\sum|F_c|/\sum|F_o|$ for the various layers before the refinements and this was repeated after the fourth and sixth cycle of least squares refinements. In the last cycle no parameter shift exceeded one tenth of the e.s.d.

Table 1 contains the final positional parameters together with their e.s.d. values. The B values with their e.s.d. are given in Table 2. The observed structure factors are compared in Table 3 with those computed on basis of the parameters listed in Tables 1 and 2. The final R value is 0.081. The atomic scattering factors used were those given by H. P. Hanson *et al.*[4] for iodine, antimony, sulphur, and hydrogen. For carbon the C (valence) values [5]

Table 1. Final atomic coordinates and their e.s.d.'s. The hydrogen atoms are given the number of the atom to which they are attached + 20.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Sb(1)	0.29629	0.00014	0.94553	0.00022	0.43587	0.00012
I(2)	0.45050	0.00015	0.95728	0.00022	0.36423	0.00014
I(3)	0.16249	0.00016	0.75766	0.00025	0.31872	0.00014
I(4)	0.18708	0.00019	1.19384	0.00027	0.35926	0.00020
S(5)	0.3981	0.0005	0.6211	0.0008	0.4923	0.0004
C(6)	0.4150	0.0019	0.5232	0.0029	0.4036	0.0015
C(7)	0.4683	0.0022	0.3719	0.0032	0.4363	0.0017
H(27)	0.441		0.309		0.479	
H(27)	0.484		0.300		0.391	
H(26)	0.344		0.488		0.352	
H(26)	0.470		0.568		0.377	

Table 2. Final thermal parameters. The numbers below each parameter is its e.s.d. Anisotropic values are multiplied by 10^5; isotropic are in Å^2.

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	Atom	B
Sb(1)	250	630	273	− 92	174	− 11	C(6)	1.5
	10	29	8	27	13	22		0.4
I(2)	316	703	426	33	395	73	C(7)	2.0
	12	29	10	29	17	25		0.4
I(3)	259	1130	325	−244	124	−366		
	11	37	9	31	14	26		
I(4)	441	835	707	415	626	529		
	15	36	16	34	25	34		
S(5)	241	512	250	206	208	70		
	36	93	26	90	49	72		

Table 3. Observed and calculated structure factors. The data are separated into groups having common values of h and k. The three columns in each group list values of l, F_o and F_c in that order. Unobserved reflections are indicated by an asterisk and the values of F_o given correspond to the minimum observable intensities.

H= 0,K=0

2	32*	47
4	580	-662
6	293	341
8	84	-101
10	227	-234
12	41*	7
14	160	159
16	249	238
18	53*	-11
20	58*	-37
22	62*	32
24	66	-51

H= 2,K=0

-24	64*	32
-22	76	65
-20	104	95
-18	61	-57
-16	48*	8
-14	44*	-18
-12	285	-299
-10	195	219
-8	251	301
-6	27*	37
-4	529	599
-2	62	-113
0	413	-348
2	76	65
4	552	-555
6	79	73
8	433	452
10	39*	-54
12	93	98
14	47*	21
16	157	-161
18	56*	-43
20	60*	-39
22	64*	1

H= 4,K=0

-24	63*	49
-22	62	-56
-20	172	-145
-18	51*	12
-16	85	79
-14	121	127
-12	35*	-33
-10	47	54
-8	421	412
-6	757	-818
-4	843	-875
-2	861	864
0	61	-35
2	571	-486
4	551	495
6	169	157
8	234	-214
10	285	-297
12	171	-187
14	90	95
16	54*	-24
18	58*	-13
20	96	88

H= 6,K=0

-24	63*	-25
-22	59*	31
-20	55*	-27
-18	101	97
-16	115	124
-14	253	-271
-12	177	-178
-10	205	-199
-8	302	-283
-6	395	329
-4	60	51
-2	116	92
0	453	384
2	593	-537
4	392	-339
6	100	86
8	230	-222
10	80	72
12	172	175
14	54*	23
16	58*	38
18	61*	-60
20	65*	-52

H= 8,K=0

-26	74	56
-24	63*	27
-22	59*	-56
-20	55*	-29
-18	51*	-46
-16	48*	-50
-14	201	-198
-12	42*	-9
-10	678	653
-8	59	-51
-6	529	-485
-4	548	478
-2	163	-141
0	687	-626
2	196	181
4	329	291
6	230	205
8	47*	30
10	66	-68
12	89	96
14	70	-71
16	83	-90
18	65*	70

H=10,K=0

-24	63*	-8
-22	84	-79
-20	56*	28
-18	181	186
-16	50*	38
-14	109	97
-12	45*	42
-10	150	-146
-8	41*	47
-6	266	-235
-4	40*	-6
-2	615	572
0	42*	24
2	44*	22
4	232	220
6	204	-204
8	109	-108
10	55*	32
12	58*	20
14	102	106
16	65*	23

H=12,K=0

-24	64*	16
-22	61*	66
-20	71	86
-18	71	67
-16	180	-172
-14	237	-219
-12	306	284
-10	46*	32
-8	229	-221
-6	526	499
-4	238	224
-2	349	-328
0	112	-102
2	73	-76
4	51*	7
6	53*	3
8	56*	-8
10	169	180
12	62*	15
14	80	-103

H=14,K=0

-24	66*	-41
-22	63*	-53
-20	60*	10
-18	57*	-67
-16	55*	3
-14	77	77
-12	73	70
-10	146	148
-8	236	-229
-6	262	-262
-4	122	129
-2	155	-159
0	74	-85
2	224	229
4	56*	0
6	58*	-34
8	61*	-38
10	78	-94

H=16,K=0

-22	65*	17
-20	91	89
-18	60*	-20
-16	124	-131
-14	96	100
-12	76	-85
-10	342	-353
-8	86	100
-6	165	176
-4	55*	-38
-2	56*	41
0	57*	-29
2	59*	-28
4	71	-91
6	118	-133
8	66*	79

H=18,K=0

-20	66*	-44
-18	64	-73
-16	62*	-65
-14	61*	-1
-12	151	161
-10	59*	-1
-8	59*	-37
-6	148	170
-4	90	-101
-2	157	-179
0	62*	52
2	64*	18
4	66*	57

H=20,K=0

-16	123	142
-14	75	92
-12	106	-137
-10	64*	-3
-8	64*	37
-6	64*	-46
-4	65*	30
-2	66*	45

H= 1,K=1

-20	82*	-9
-19	79*	24
-18	76*	-22
-17	114	-101
-16	99	-93
-15	92	87
-14	64*	26
-13	61*	-41
-12	292	341
-11	243	-256
-10	242	271
-9	292	374
-8	121	-150
-7	88	108
-6	39*	-43
-5	242	-361
-4	101	104
-3	133	154
-2	227	-399
-1	100	-97
0	197	-259
1	196	-104
2	433	498
3	200	-195
4	386	479
5	210	-219
6	42*	40
7	328	392
8	49*	-19
9	52*	-44
10	55*	61
11	210	-225
12	101	-104
13	183	163
14	187	-184
15	96	-83
16	73*	-7
17	118	-112
18	102	91
19	82*	44

H= 3,K=1

-20	80*	71
-19	77*	19
-18	126	116
-17	71*	32
-16	68*	23
-15	179	-164
-14	63*	31
-13	177	192
-12	57*	72
-11	102	105
-10	170	-214
-9	289	-358
-8	363	-472
-7	42*	-2
-6	39*	-14
-5	110	-108
-4	508	565
-3	183	-149
-2	236	204
-1	58	-48
0	131	-94
1	239	-210
2	267	247
3	439	427
4	72	-59
5	44*	40
6	491	-528
7	402	-410
8	240	-215
9	163	139
10	143	137
11	62*	-47
12	65*	70
13	165	-160
14	70*	1
15	107	94
16	76*	59
17	79*	-4
18	82*	28

H= 5,K=1

-21	82*	20
-20	79*	37
-19	76*	-56
-18	104	-102
-17	115	107
-16	199	-195
-15	65*	-13
-14	62*	-25
-13	277	-298
-12	57*	62
-11	149	165
-10	109	-115
-9	50*	42
-8	104	107
-7	308	-297
-6	426	427
-5	209	185
-4	91	-77
-3	160	137
-2	549	-545
-1	161	134
0	307	-266
1	122	-105
2	44*	-37
3	321	-299
4	118	-105
5	293	283
6	53*	3
7	56*	-35
8	247	227
9	208	-207
10	121	118
11	243	245
12	116	-116
13	72*	35
14	112	-109
15	78*	-60
16	81*	-36

H= 7,K=1

-21	82*	24
-20	79*	-13
-19	77*	28
-18	74*	-52
-17	159	-149
-16	69*	20
-15	94	105
-14	214	221
-13	139	143
-12	128	116
-11	119	-111
-10	282	-280
-9	169	170
-8	281	-266
-7	83	77
-6	80	66
-5	48*	-6
-4	157	-144
-3	47*	-19
-2	306	-270
-1	281	-251
0	360	324
1	324	302
2	469	432
3	173	173
4	91	-75
5	215	-201
6	182	-174
7	254	242
8	65*	-20
9	67*	57
10	70*	-65
11	140	-126
12	100	-95
13	78*	69
14	80*	42

H=19,K=1

-20	80*	70
-19	100	109
-18	75*	-20
-17	81	84
-16	71*	1
-15	135	-124
-14	66*	-10
-13	179	175
-12	238	-236
-11	103	89
-10	173	-154
-9	259	-246
-8	294	285
-7	56*	9
-6	327	303
-5	77	60
-4	54*	46
-3	196	191
-2	55*	58
-1	72	60
0	85	81
1	175	-158
2	158	-134
3	283	266
4	244	-229
5	65*	-21
6	67*	10
7	285	-270
8	171	163
9	132	125
10	94	91
11	78*	18
12	81*	57

H=11,K=1

-20	82*	-57
-19	80*	-81
-18	119	-112
-17	76*	28
-16	74*	49
-15	72*	30
-14	195	172
-13	146	-145
-12	67*	42
-11	65*	47
-10	64*	47
-9	63*	38
-8	266	257
-7	62*	50
-6	61*	1
-5	61*	60
-4	398	-389
-3	217	-210
-2	93	-94
-1	63*	83
0	231	220
1	65*	14
2	67*	61
3	269	-266
4	70*	18
5	109	110
6	153	143
7	76*	49
8	78*	70
9	80*	-83
10	82*	-93

H=13,K=1

-18	81*	61
-17	121	-125
-16	157	139
-15	76*	83
-14	74*	-21
-13	100	98
-12	181	-177
-11	123	-128
-10	70*	-62
-9	69*	-55
-8	69*	-3
-7	68*	-70
-6	68*	-71
-5	68*	20
-4	69*	31
-3	69*	-46
-2	214	207
-1	184	-183
0	124	121
1	192	194
2	110	-117
3	76*	61
4	145	-134
5	160	-184
6	81*	-45

H=15,K=1

-16	82*	-8
-15	81*	-87
-14	106	-104
-13	78*	-15
-12	110	-106
-11	77*	-46
-10	170	162
-9	76*	-14
-8	188	182
-7	75*	66
-6	106	-108
-5	75*	-74
-4	101	-104
-3	102	111
-2	77*	16
-1	78*	78
0	105	-116
1	160	-165
2	156	-158

H=17,K=1

-10	83*	16
-9	82*	27
-8	82*	-102
-7	82*	83
-6	82*	-145

H= 0,K=2

0	53*	-7
1	98	83
2	435	438
3	207	195
4	31*	-32
5	203	209
6	372	-423
7	42*	30
8	84	102
9	106	-118
10	148	-171
11	85	-103
12	89	-96
13	118	-134
14	217	221
15	66*	-66
16	69*	22
17	71*	49
18	74*	14
19	77*	27

H= 2,K=2

-20	78*	-45
-19	75*	38
-18	72*	13
-17	69*	-10
-16	66*	8
-15	64*	18
-14	143	-128
-13	80	76
-12	119	-118
-11	198	-237
-10	77	-130
-9	290	-361
-8	365	470
-7	105	124
-6	282	342
-5	135	150
-4	512	-650
-3	261	-253
-2	440	416
-1	24*	24
0	454	390
1	371	337
2	755	-1002
3	242	-220
4	151	-145
5	503	-520
6	379	375
7	46*	-69
8	92	87
9	52*	62
10	103	107
11	103	-106
12	60*	-38
13	63*	17
14	66*	36
15	83	74
16	72*	-12
17	75*	-9
18	167	-145

H= 4,K=2

-20	88	-75
-19	104	-78
-18	113	-102
-17	88	-82

-16	162	157
-15	86	-74
-14	60*	42
-13	57*	-61
-12	55*	17
-11	52*	50
-10	245	275
-9	66	70
-8	214	-227
-7	42*	40
-6	194	-189
-5	37*	-20
-4	67	-50
-3	252	-214
-2	437	-391
-1	345	-293
0	392	343
1	159	-145
2	247	220
3	66	59
4	137	-129
5	70	73
6	351	341
7	61	69
8	84	-88
9	106	115
10	260	-271
11	62*	-32
12	64*	27
13	95	-98
14	70*	-56
15	73*	-15
16	76*	25
17	78*	-13

H= 6,K=2

-20	76*	37
-19	73*	-46
-18	111	112
-17	68*	28
-16	66*	53
-15	206	205
-14	61*	-68
-13	58*	39
-12	322	-328
-11	177	-182
-10	98	87
-9	49*	42
-8	208	191
-7	46*	37
-6	541	-518
-5	341	-310
-4	441	387
-3	93	-73
-2	736	726
-1	524	478
0	486	-454
1	320	282
2	244	-221
3	127	-112
4	50*	61
5	52*	62
6	122	-113
7	57*	70
8	59*	-9
9	139	-144
10	64*	-57
11	67*	-47
12	139	137
13	87	82
14	127	140
15	77*	80

H= 8,K=2

-20	105	-93
-19	74*	46
-18	72*	36
-17	69*	24
-16	77	-74
-15	65*	-24
-14	116	-122
-13	60*	-27
-12	179	186
-11	57*	-41
-10	80	73
-9	73	66
-8	193	172
-7	273	234
-6	102	97
-5	200	186
-4	417	-394
-3	73	57
-2	49*	38
-1	50*	24
0	51*	-46
1	52*	14
2	295	-278
3	102	-99
4	334	330
5	59*	-70
6	157	159
7	124	127
8	65*	-74
9	93	99
10	70*	71
11	72*	24
12	75*	-77
13	77*	52

H=10,K=2

-20	78*	27
-19	76*	-56
-18	88	90
-17	71*	83
-16	95	90
-15	67*	66
-14	136	-122
-13	64*	-33
-12	129	114
-11	156	151
-10	165	161
-9	208	189
-8	557	-544
-7	175	-170
-6	89	-86
-5	269	-263
-4	457	437
-3	56*	65
-2	113	-113
-1	81	81
0	58*	55
1	110	-107
2	167	186
3	62*	62
4	64*	-11
5	151	161
6	67*	-54
7	69*	-47
8	195	-205
9	74*	-93
10	76*	-7
11	78*	-49

H=12,K=2

-19	76*	50
-18	76*	6
-17	74*	43
-16	147	-140
-15	71*	16
-14	69*	-41
-13	106	-106
-12	67*	-74
-11	162	-149
-10	110	102
-9	64*	-56
-8	169	159
-7	65*	-36
-6	116	-111
-5	62*	-31
-4	189	200
-3	63*	66
-2	64*	55
-1	64*	112
0	348	-342
1	66*	-56
2	68*	22
3	160	-168
4	71*	21
5	72*	-58
6	74*	-61
7	76*	-47
8	78*	119

H=14,K=2

-17	78*	-38
-16	102	-102
-15	130	-142
-14	107	114
-13	73*	-27
-12	266	281
-11	71*	123
-10	203	-205
-9	70*	13
-8	162	-136
-7	69*	-89
-6	130	144
-5	69*	37
-4	154	-172
-3	70*	10
-2	71*	-87
-1	162	-182
0	72*	86
1	74*	-83
2	75*	92
3	76*	71
4	78*	115

H=16,K=2

-13	78*	-6
-12	77*	-15
-11	77*	25
-10	76*	-19
-9	76*	-24
-8	162	-168
-7	76*	-94
-6	149	150
-5	76*	-23
-4	171	190
-3	77*	106
-2	78*	-107
-1	79*	76

H= 1,K=3

-19	75*	-28
-18	72*	-19
-17	83	73
-16	66*	-67
-15	200	-188
-14	81	-80
-13	162	-166
-12	92	105
-11	202	228
-10	159	181
-9	46*	13
-8	43*	-52
-7	84	104
-6	231	-316
-5	135	187
-4	40	-46
-3	67	-75
-2	84	82
-1	17*	-7
0	457	-458
1	550	-631
2	122	-116
3	294	-316
4	397	461
5	380	466
6	40*	34
7	94	-98
8	158	-181
9	49*	-23
10	52*	26
11	223	243
12	58*	1
13	123	-123
14	128	-117
15	119	-112
16	69*	-65
17	74*	-61
18	75*	63

H= 3,K=3

-20	76*	42
-19	73*	-78
-18	70*	2
-17	68*	18
-16	65*	-25
-15	140	144
-14	59*	60
-13	107	-110
-12	67	66
-11	118	145
-10	206	-241
-9	170	198
-8	206	-229
-7	497	-631
-6	259	280
-5	287	-302
-4	98	92
-3	146	105
-2	175	-156
-1	144	-125
0	324	275
1	203	173
2	197	176
3	82	58
4	217	-193
5	162	159
6	165	-161
7	90	77
8	50*	29
9	401	-438
10	113	94
11	92	-91
12	61*	3
13	91	89
14	67*	32
15	96	-93
16	87	73
17	94	75

H= 5,K=3

-20	75*	40
-19	167	146
-18	70*	-19
-17	67*	-22
-16	118	-116
-15	129	-137
-14	59*	-11
-13	57*	17
-12	137	152
-11	185	-209
-10	93	88
-9	109	-118
-8	86	-76
-7	73	66
-6	239	226
-5	132	124
-4	399	361
-3	501	475
-2	382	-348
-1	166	-142
0	368	-332
1	329	-296
2	311	283
3	256	230
4	119	110
5	306	-274
6	78	-64
7	205	-188
8	155	144
9	255	246
10	173	156
11	63*	66
12	66*	-33
13	91	88
14	95	-95
15	74*	42

H= 7,K=3

-20	75*	-14
-19	73*	-30
-18	70*	-25
-17	68*	27
-16	65*	74
-15	180	-183
-14	176	187
-13	115	111
-12	56*	-1
-11	412	438
-10	135	-134
-9	82	-79
-8	132	121
-7	47*	-26
-6	46*	-31
-5	131	111
-4	281	-262
-3	208	-190
-2	103	89
-1	95	-83
0	251	227
1	130	-126
2	90	77
3	254	229
4	53*	-2
5	416	400
6	57*	-13
7	123	-120
8	61*	-15
9	64*	21
10	93	-95
11	99	104
12	71*	-52
13	153	-155
14	76*	39

H= 9,K=3

-20	76*	51
-19	74*	49
-18	71*	-42
-17	69*	33
-16	67*	-45
-15	165	171
-14	63*	31
-13	61*	8
-12	59*	-47
-11	58*	-53
-10	245	-227
-9	148	-141
-8	54*	43
-7	212	-196
-6	381	356
-5	291	263
-4	110	-93
-3	85	67
-2	256	-247
-1	53*	-12
0	149	137
1	419	404
2	56*	-8
3	56*	-64
4	264	-237
5	268	-236
6	63*	-63
7	65*	3
8	112	101
9	101	-96
10	72*	36
11	74*	31
12	76*	-45

H=11,K=3

-19	76*	72
-18	74*	-66
-17	182	-176
-16	76*	33
-15	122	-112
-14	66*	3
-13	141	140
-12	63*	-49
-11	97	-102
-10	128	125
-9	60*	-8
-8	59*	41
-7	217	209
-6	199	-206
-5	58*	76
-4	127	-123
-3	59*	-4
-2	59*	-32
-1	309	-298
0	61*	-10
1	135	-130
2	63*	18
3	135	129
4	66*	36
5	159	-162
6	70*	61
7	72*	25
8	74*	-20
9	187	182

H=13,K=3

-17	75*	36
-16	73*	86
-15	72*	36
-14	70*	55
-13	101	94
-12	184	-168
-11	67*	2
-10	110	-105
-9	188	-189
-8	158	155
-7	65*	14
-6	65*	-44
-5	145	-161
-4	115	-132
-3	224	-221
-2	180	192
-1	190	196
0	152	158
1	69*	69
2	94	-93
3	72*	-26
4	97	-92
5	75*	73
6	76*	24

H=15,K=3

-15	76*	80
-14	75*	-53
-13	121	-121
-12	73*	58
-11	153	-157
-10	96	100
-9	72*	3
-8	71*	22
-7	92	87
-6	71*	34
-5	160	167
-4	72*	16
-3	72*	-62
-2	73*	-14
-1	74*	-13
0	75*	-28
1	104	115

H= 0,K=4

0	469	438
1	458	464
2	245	-233
3	494	510
4	294	-294
5	181	-184
6	123	130
7	43*	-29
8	133	-136
9	220	238
10	53*	-33
11	194	-206
12	59*	-27
13	250	-266
14	75	-78
15	68*	37
16	88	73
17	92	79
18	77*	-35

H= 2,K=4

-19	77*	39
-18	105	-98
-17	71*	48
-16	69*	-15
-15	85	87
-14	63*	32
-13	75	-72
-12	192	-212
-11	150	-175
-10	64	75
-9	136	-168
-8	83	100
-7	84	-93
-6	145	-184
-5	155	178
-4	133	140
-3	269	262
-2	114	-102
-1	199	186
0	156	-129
1	42	35
2	106	96
3	170	-151
4	258	-260
5	215	-210
6	106	100
7	207	-216
8	186	197
9	53*	7
10	122	-138
11	123	129
12	83	80
13	65*	39
14	68*	36
15	88	84
16	92	-81
17	77*	38

H= 4,K=4

-19	76*	-61
-18	73*	46
-17	192	-169
-16	68*	-27
-15	65*	24
-14	62*	49
-13	81	84
-12	56*	48
-11	159	-184
-10	51*	-34
-9	159	172
-8	232	250
-7	495	551
-6	141	-143
-5	114	-101
-4	264	-237
-3	592	-583
-2	458	424
-1	80	-62
0	36*	-25
1	198	176
2	134	-118
3	310	-285
4	157	268
5	47*	-36
6	50*	32
7	458	465
8	114	79
9	113	109
10	61*	2
11	174	-162
12	86	-88
13	69*	-44
14	110	111
15	75*	-32
16	78*	17

H= 6,K=4

-19	76*	-23
-18	73*	50
-17	88	84
-16	113	124
-15	99	111
-14	102	-100
-13	100	97
-12	58*	54
-11	55*	-31
-10	106	104
-9	201	-193
-8	77	-74
-7	128	-124
-6	213	195
-5	139	-98
-4	44*	22
-3	43*	-36
-2	43*	27
-1	205	175
0	252	223
1	313	281
2	212	-192
3	151	146
4	52*	39
5	146	-137
6	178	172
7	119	-113
8	141	-141
9	64*	-44
10	66*	63
11	108	-114
12	72*	67
13	74*	31
14	77*	-50

H= 8,K=4

-19	76*	-39
-18	74*	-23
-17	71*	40
-16	69*	19
-15	67*	29
-14	64*	-3
-13	287	-304
-12	60*	-61
-11	168	-152
-10	249	237
-9	364	343
-8	54*	-25
-7	219	193
-6	264	-243
-5	159	-142
-4	227	207
-3	145	126
-2	85	-81
-1	277	256
0	234	-206
1	341	-317
2	108	97
3	384	-371

4	59*	-23
5	154	142
6	63*	65
7	132	119
8	67*	-12
9	70*	-4
10	146	-136
11	86	82
12	77*	52

H=10,K=4

-19	78*	-66
-18	76*	67
-17	74*	-28
-16	89	-82
-15	90	78
-14	68*	-12
-13	103	100
-12	64*	22
-11	63*	50
-10	122	-119
-9	104	100
-8	59*	10
-7	58*	-31
-6	148	-140
-5	269	-245
-4	107	-94
-3	198	-189
-2	156	148
-1	59*	44
0	154	-153
1	156	140
2	63*	-25
3	64*	27
4	96	98
5	90	88
6	170	-172
7	92	98
8	74*	-25
9	110	-112
10	78*	15

H=12,K=4

-17	158	159
-16	75*	-47
-15	73*	-79
-14	128	-119
-13	190	-172
-12	114	112
-11	68*	37
-10	66*	-1
-9	66*	-20
-8	184	-178
-7	220	-215
-6	144	138
-5	64*	48
-4	65*	9
-3	380	371
-2	122	-118
-1	66*	53
0	67*	30
1	187	-194
2	70*	-60
3	71*	5
4	73*	52
5	75*	-32
6	77*	30
7	105	-114

H=14,K=4

-15	78*	-50
-14	76*	64
-13	75*	-62
-12	74*	6
-11	109	109
-10	72*	77
-9	182	173
-8	71*	-60
-7	71*	18
-6	71*	-55
-5	71*	-81
-4	141	141
-3	72*	-38
-2	73*	-52
-1	74*	-39
0	75*	18
1	136	-135
2	145	145

H=16,K=4

-9	165	-173
-8	78*	84
-7	154	-152
-6	78*	68
-5	78*	102

H= 1,K=5

-18	72*	-30
-17	69*	-50
-16	66*	-58
-15	63*	-45
-14	60*	3
-13	185	-195
-12	55*	3
-11	52*	-44
-10	80	-91
-9	326	424
-8	123	-150
-7	40*	-18
-6	96	126
-5	39	-47
-4	61	76
-3	311	456
-2	247	-305
-1	244	-262
0	39	41
1	413	-444
2	157	159
3	29*	-10
4	184	-207
5	37*	34
6	112	-120
7	216	270
8	46*	45
9	104	119
10	52*	55
11	55*	16
12	57*	-20
13	122	137
14	63*	-43
15	103	-104
16	69*	30
17	119	-111

H= 3,K=5

-18	70*	9
-17	87	90
-16	64*	-55
-15	62*	-14
-14	59*	-44
-13	147	161
-12	54*	47
-11	127	158
-10	84	97
-9	46*	-24
-8	43*	-38
-7	95	-105
-6	140	-156
-5	347	-358
-4	200	195
-3	143	130
-2	109	102
-1	336	304
0	347	-313
1	260	-235
2	62	50
3	374	355
4	225	223
5	397	413
6	45*	-17
7	293	-295
8	50*	-14
9	121	-131
10	64	57
11	58*	1
12	61*	55
13	64*	-45
14	66*	-34
15	69*	54
16	72*	-24

H= 5,K=5

-19	83	74
-18	69*	-15
-17	120	115
-16	64*	48
-15	114	-118
-14	74	82
-13	185	-202
-12	54*	13
-11	132	141
-10	97	-90
-9	157	-154
-8	128	-121
-7	148	-148
-6	156	142
-5	527	518
-4	206	-188
-3	207	192
-2	84	75
-1	107	-93
0	284	253
1	84	-69
2	42*	-32
3	274	-243
4	46*	-22
5	48*	-20
6	75	63
7	81	-84
8	85	58
9	88	-62
10	60*	-12
11	237	244
12	65*	-36
13	68*	51
14	71*	51

H= 7,K=5

-19	72*	-2
-18	70*	0
-17	136	-145
-16	65*	-22
-15	62*	-55
-14	60*	24
-13	64	68
-12	79	80
-11	157	158
-10	52*	-7
-9	177	170
-8	106	-111
-7	230	-211
-6	198	191
-5	45*	-23
-4	175	164
-3	45*	30
-2	258	-240
-1	577	-554
0	46*	-29
1	48*	-33
2	160	145
3	390	373
4	53*	-40
5	55*	-46
6	73	-66
7	59*	26
8	61*	12
9	63*	45
10	66*	58
11	91	-84
12	70*	-24

H= 9,K=5

-18	71*	-4
-17	69*	5
-16	67*	26
-15	68	-64
-14	63*	1
-13	169	172
-12	86	-80
-11	127	-119
-10	56*	58
-9	375	-367
-8	93	83
-7	53*	28
-6	209	-203
-5	52*	24
-4	92	-79
-3	67	52
-2	90	81
-1	154	148
0	80	-73
1	55*	-8
2	56*	-50
3	112	99
4	59*	13
5	120	-117
6	63*	19
7	261	-252
8	67*	-36
9	69*	66
10	71*	-72

H=11,K=5

-17	89	-91
-16	69*	-15
-15	103	-100
-14	66*	31
-13	64*	-7
-12	63*	-39
-11	111	108
-10	166	-162
-9	80	-70
-8	59*	43
-7	183	173
-6	114	96
-5	332	321
-4	175	-171
-3	284	-267
-2	59*	-41
-1	178	-175
0	118	115
1	105	101
2	63*	-18
3	130	-119
4	91	-85
5	68*	13
6	69*	-37
7	134	127

H=13,K=5

-15	198	196
-14	70*	-56
-13	139	142
-12	68*	36
-11	86	-75
-10	66*	93
-9	75	22
-8	75	-74
-7	74	-60
-6	64*	-19
-5	74	-74
-4	65*	80
-3	65*	-11
-2	66*	-22
-1	67*	-29
0	67*	-23
1	219	219
2	70*	14
3	128	128

H=15,K=5

-11	189	-188
-10	72*	42
-9	71*	-25
-8	71*	95
-7	242	253
-6	71*	-79
-5	71*	-16
-4	71*	-20
-3	72*	-12

H= 0,K=6

0	178	-178
1	139	146
2	25*	13
3	160	161
4	48	-54
5	101	108
6	44*	-24
7	85	84
8	207	218
9	55*	-37
10	59*	-40
11	62*	-50
12	65*	-49
13	88	-94
14	72*	55
15	75*	-24
16	98	-92

H= 2,K=6

-17	79*	4
-16	76*	2
-15	73*	56
-14	69*	-34
-13	136	130
-12	152	166
-11	207	-239
-10	57*	-51
-9	225	-279
-8	139	-164
-7	228	292
-6	43*	25
-5	218	266
-4	105	-112
-3	80	-77
-2	42	34
-1	155	140
0	109	94
1	423	412
2	35*	22
3	63	-57
4	185	180
5	410	-447
6	105	-107
7	53*	48
8	112	-117
9	206	225
10	86	96
11	66*	-32
12	100	-100
13	72*	66
14	75*	-21
15	123	128

H= 4,K=6

-18	81*	-32
-17	78*	-41
-16	75*	70
-15	72*	-15
-14	69*	-62
-13	66*	9
-12	127	-130
-11	93	96
-10	87	91
-9	109	120
-8	51*	-36
-7	219	226
-6	45*	36
-5	123	112
-4	205	183
-3	207	-186
-2	39*	-20
-1	160	-139
0	40*	48
1	74	-68
2	130	-114
3	47*	20
4	162	-152
5	131	113
6	141	133
7	123	115
8	61*	-37
9	138	140
10	67*	-1
11	71*	-29
12	137	139
13	102	-104
14	80*	-40

H= 6,K=6

-18	81*	-31
-17	78*	33
-16	75*	-74
-15	233	219
-14	113	121
-13	67*	-14
-12	64*	34
-11	206	-218
-10	59*	-22
-9	56*	70
-8	135	120
-7	52*	30
-6	51*	-65
-5	344	-323
-4	48*	-61
-3	190	-164
-2	64	-66
-1	395	361
0	126	-92
1	260	230
2	193	170
3	281	-256
4	57*	6
5	60*	-50
6	62*	-20
7	65*	65
8	155	148
9	194	-194
10	104	-97
11	114	-110
12	91	-82

H= 8,K=6

-17	79*	-36
-16	76*	21
-15	74*	-83
-14	71*	-59
-13	115	-116
-12	67*	54
-11	148	-153
-10	98	-97
-9	61*	49
-8	112	-92
-7	206	175
-6	57*	56
-5	56*	8
-4	56*	-15
-3	56*	-31
-2	137	123
-1	57*	-65
0	58*	25
1	154	-129
2	91	-79
3	198	-172
4	81	83
5	114	-118
6	125	-117
7	105	100
8	99	-101
9	77*	71
10	96	88

H=10,K=6

-16	79*	19
-15	77*	14
-14	75*	-46
-13	94	-85
-12	71*	-63
-11	155	147
-10	132	100
-9	203	187
-8	65*	32
-7	188	-171
-6	64*	43
-5	331	-311
-4	64*	-9
-3	64*	43
-2	136	-120
-1	145	142
0	67*	40
1	134	-131
2	69*	-77
3	71*	59
4	73*	-71
5	219	231
6	138	148
7	79*	-7

H=12,K=6

-15	81*	46
-14	102	93
-13	103	-97
-12	76*	10
-11	75*	-59
-10	74*	-38
-9	73*	46
-8	72*	-2
-7	72*	-6
-6	111	-94
-5	71*	48
-4	71*	14
-3	166	149
-2	73*	12
-1	162	151
0	75*	-11
1	76*	-1
2	150	146
3	105	-114
4	81*	-17

H=14,K=6

-11	210	201
-10	80*	-26
-9	79*	88
-8	79*	27
-7	79*	-76
-6	79*	54
-5	79*	49
-4	79*	0
-3	80*	82
-2	80*	95

H= 1,K=7

-16	72*	46
-15	92	-79
-14	66*	-28
-13	94	-100
-12	60*	-51
-11	57*	39
-10	62	-63
-9	63	68
-8	48*	33
-7	137	178
-6	41*	1
-5	37*	-22
-4	56	65
-3	52	-61
-2	230	318
-1	204	258
0	60	65
1	230	-255
2	153	-165
3	292	-323
4	64	-61
5	285	348
6	44*	-42
7	115	135
8	51*	-10
9	65	-64
10	57*	-1
11	134	130
12	81	83
13	66*	36
14	110	110
15	72*	-33

H= 3,K=7

-16	71*	6
-15	68*	47
-14	65*	11
-13	62*	-13
-12	59*	-3
-11	181	212
-10	53*	66
-9	50*	59
-8	110	120
-7	271	-337
-6	89	88
-5	105	112
-4	179	-191
-3	177	171
-2	177	-169
-1	197	-192
0	70	65
1	183	164
2	84	-76
3	230	222
4	43*	-23
5	46*	43
6	130	135
7	52*	14
8	78	72
9	130	-131
10	61*	-6
11	64*	-10
12	92	-92
13	70*	49
14	73*	-51

H= 5,K=7

-17	73*	35
-16	70*	38
-15	67*	-61
-14	65*	38
-13	62*	-56
-12	59*	-29
-11	57*	-50
-10	54*	-41
-9	94	87
-8	157	-166
-7	66	-54
-6	182	-183
-5	43*	-23
-4	117	113
-3	455	422
-2	114	88
-1	42*	-44
0	63*	-7
1	387	-375
2	74	61
3	152	135
4	51*	-2
5	53*	-5
6	83	-73
7	193	-192
8	161	-154

9 63* 55	-9 185 -175	-8 71* 88
10 66* -69	-8 114 -88	-7 135 123
11 69* 80	-7 249 -240	-6 70* 21
12 72* 60	-6 57* 5	-5 71* 41
	-5 258 245	-4 71* -82
H= 7,K=7	-4 56* -46	-3 189 -191
-16 71* -25	-3 84 76	-2 72* -71
-15 125 -131	-2 95 -80	-1 119 108
-14 108 -110	-1 216 -202	
-13 106 108	0 59* 59	H= 0,K=8
-12 102 -101	1 194 179	0 176 -200
-11 154 150	2 139 117	1 99 107
-10 57* 19	3 94 78	2 63 -75
-9 168 -164	4 134 115	3 152 174
-8 163 153	5 191 -181	4 188 204
-7 81 59	6 69* -11	5 63 -68
-6 51* 7	7 71* -60	6 103 107
-5 72 62		7 67 -70
-4 49* -19	H=11,K=7	8 46* -46
-3 310 -300	-14 72* -55	9 108 134
-2 119 106	-13 81 75	10 172 198
-1 129 -100	-12 69* -44	11 55* -33
0 125 -120	-11 98 -97	12 73 84
1 52* 4	-10 66* 36	13 100 -108
2 203 -185	-9 82 78	14 121 -142
3 56* 20	-8 65* -45	
4 58* -24	-7 189 181	H= 2,K=8
5 125 106	-6 64* -8	-15 65* 42
6 62* 47	-5 64* -30	-14 120 132
7 64* -60	-4 201 200	-13 59* 3
8 92 75	-3 64* 5	-12 56* 9
9 69* -12	-2 112 95	-11 53* -38
10 72* 15	-1 65* -53	-10 51* 45
	0 66* -36	-9 84 -90
H= 9,K=7	1 71 -67	-8 45* -45
-16 73* 21	2 69* -32	-7 42* 5
-15 71* -14	3 70* 65	-6 192 -193
-14 69* 37	4 72* -37	-5 136 137
-13 67* -18		-4 32* 2
-12 110 103	H=13,K=7	-3 38 -31
-11 63* 47	-10 72* 22	-2 57 47
-10 61* 31	-9 168 -165	-1 45 31

0 130 114	11 78 -86	-2 51* 24
1 143 119		-1 125 126
2 240 195	H= 6,K=8	0 201 199
3 104 -85	-15 64* 44	1 56 -51
4 38* -3	-14 62* -48	2 88 83
5 72 -64	-13 59* 31	3 180 -180
6 44* 0	-12 78 84	4 220 -223
7 47* 0	-11 66 -65	5 60* 61
8 58 -62	-10 80 79	6 62* -31
9 53* 14	-9 50* -48	7 64* 69
10 143 -168	-8 101 -91	
11 59* 27	-7 62 58	H=10,K=8
12 61* 12	-6 45* -39	-13 65* -10
13 64* -11	-5 112 -99	-12 63* 57
	-4 143 -125	-11 62* -1
H= 4,K=8	-3 64 -56	-10 60* 17
-15 64* 51	-2 193 -162	-9 86 92
-14 61* -32	-1 144 121	-8 152 147
-13 59* 31	0 45* -21	-7 58* -52
-12 56* 23	1 46* 31	-6 122 122
-11 96 -108	2 48* -15	-5 102 -104
-10 183 -205	3 49* 0	-4 57* -4
-9 77 84	4 128 121	-3 57* 22
-8 45* -46	5 54* -29	-2 58* -37
-7 179 177	6 91 87	-1 58* 21
-6 248 218	7 58* -36	0 99 -105
-5 53 -46	8 90 -90	1 61* 34
-4 158 129	9 63* -14	2 62* 19
-3 193 -157		3 63* 13
-2 73 -60	H= 8,K=8	4 65* 68
-1 48 -38	-14 92 99	
0 202 -156	-13 111 -130	H=12,K=8
1 157 125	-12 126 -138	-9 65* 22
2 40* -2	-11 58* -29	-8 64* -4
3 67 -57	-10 152 -162	-7 64* -53
4 120 -109	-9 115 117	-6 64* -75
5 99 -94	-8 53* 4	-5 64* -28
6 266 -264	-7 52* 44	-4 64* -113
7 164 161	-6 59 55	-3 64* 154
8 100 93	-5 82 -73	-2 65* 100
9 58* 48	-4 50* 36	
10 124 145	-3 50* -32	

were used. As a final check a three-dimensional difference Fourier synthesis was calculated with all atoms removed. The highest peak was 2.2 e·Å^{-3} and

Table 4. Interatomic distances and angles and their e.s.d.'s Numbers in parentheses are after correction due to libration.

	Distance	e.s.d.		Angle	e.s.d.
Sb(1)—I(2)	2.774 Å	0.0019 Å	I(2)—Sb(1)—I(3)	96.84°	0.07°
	(2.778)			(96.80)	
Sb(1)—I(3)	2.746	0.0028	I(3)—Sb(1)—I(4)	94.72	0.10
	(2.755)			(94.70)	
Sb(1)—I(4)	2.767	0.0031	I(2)—Sb(1)—I(4)	97.28	0.08
	(2.774)			(97.18)	
I(2)—I(3)	4.129	0.0021	S(5)—C(6)—C(7)	109	2
	(4.137)		S(8)—C(7)—C(6)	116	2
I(3)—I(4)	4.055	0.0034	C(7)—S(8)—C(9)	100	2
	(4.066)		I(4)—Sb(1)—S(5)	169.0	1.6
I(2)—I(4)	4.159	0.0023	I(18)—Sb(11)—S(5)	170.8	1.3
	(4.164)		I(2)—Sb(1)—S(5)	81.7	0.10
S(5)—C(6)	1.83	0.024	I(3)—Sb(1)—S(5)	74.6	0.15
S(8)—C(7)	1.77	0.023	I(12)—Sb(11)—S(5)	75.0	0.13
C(6)—C(7)	1.57	0.038	I(13)—Sb(11)—S(5)	87.7	0.13
S(5)—S(8)	3.49	0.011	Sb(1)—S(5)—S(8)	147.3	0.8
S(5)—Sb(1)	3.274	0.007	Sb(11)—S(5)—S(8)	124.6	0.3
S(5)—Sb(11)	3.336	0.005	Sb(1)—S(5)—Sb(11)	88.0	0.16
Sb(1)—Sb(11)	4.594	0.0037	S(5)—Sb(11)—S(14)	92.0	0.15
S(5)—I(2)	3.974	0.007			
S(5)—I(12)	3.732	0.007			
I(3)—I(15)	3.996	0.0031			
I(2)—I(16)	4.046	0.0024			
I(3)—I(17)	4.237	0.0029			
I(2)—I(17)	4.281	0.0036			

the lowest minimum -3.4 e·Å^{-3}, both in the region of the antimony triiodide molecule. In the region of the dithiane molecule the maximum deviation from zero was about ± 0.8 e·Å^{-3}.

DESCRIPTION OF THE STRUCTURE

The structure represents a molecular complex of discrete antimony triiodide and dithiane molecules in the crystal. The latter molecules are situated in centres of symmetry, whereas the former molecules occupy general positions. Each antimony atom forms bonds with two sulphur atoms, and two bridging antimony triiodide molecules link neighbouring dithiane molecules together forming endless chains of antimony triiodide and dithiane molecules in the crystal. The direction of the chains alternates between [110] and [1$\bar{1}$0] in layers parallel to the XY plane. The Sb—S bond distances, 3.274 and 3.336 Å, are about 0.70 Å shorter than that expected for a van der Waals' contact. There is a significant difference in bond lengths of the two types of Sb—S bonds, the difference being about 7.0 times the standard deviation. Some atomic distances from the plane through the antimony atoms of the chain were calculated with the results given in Table 5. This plane is very

Table 5. Some atomic distances from the plane through the antimony atoms of the chain. This plane also contains the main chain axis through ($\frac{1}{4}$, $\frac{1}{4}$, $\frac{1}{2}$) and ($\frac{1}{4}$, $\frac{3}{4}$, $\frac{1}{2}$).

Atom	Distance	Atom	Distance
S(5)	0.032 Å	I(2)	0.347 Å
C(6)	1.42	I(3)	2.543
C(7)	1.33	I(4)	0.499

nearly a mirror plane of the dithiane carbon atoms. The sulphur atoms are, however, displaced out of the plane by about 0.032 Å which is 5.0 times the e.s.d. value of the atomic position, indicating that the dithiane mirror plane deviates significantly from the plane through the antimony atoms. The angles between the Sb—S bonds and the antimony plane are about 0.6°. The Sb—S bonds are nearly in the equatorial and axial directions of the ring system. The equatorial and axial angles S···S—Sb are 147.3° and 124.6°, the corresponding "ideal" values calculated for the free dithiane molecule [6] are 146° and 105°. This indicates that all lone pair electrons belonging to a particular sulphur atom take part in the bond formation with antimony atoms. It is interesting to note that the direction of the shorter Sb—S bond approaches closely the "ideal" equatorial angle, whereas the direction of the other Sb—S bond, which is 0.06 Å longer, deviates about 20° from the axial direction. The distances and angles in the dithiane molecule agree with those found in the free molecule.[6] Of interest are also one rather short S···I approach of 3.732 Å and two intermolecular I···I separations of 3.996 Å and 4.046 Å, listed in Table 4. Apart from these short distances, all intermolecular distances are equal to or longer than that expected for van der Waals' contacts.

Rigid body analysis of the antimony triiodide molecule was carried out by the method of Cruickshank [7] using a program by Gantzel, Coulter and Trueblood.[8] The program was revised to include the refinement of the three coordinates of the centre of libration by a method based on trial and error.[9] The coordinates were altered by predetermined steps after finding which direction of alternation would minimize the sum $\sum(U(\text{obs}) - U(\text{calc}))^2$. When the value of the coordinates oscillated, the steps were reduced and the process continued. The mass centre of the molecule was chosen as the initial origin of the libration. In the course of refinements the standard deviation obtained from $\sigma(U) = [\sum(U(\text{obs}) - U(\text{calc}))^2/(n - s)]^{\frac{1}{2}}$ (n = number of observations, s = number of parameters) was reduced from 0.0073 Å^2 to 0.0023 Å^2. The latter value is about the same as that obtained from the structure factor refinements. At the same time the centre of libration moved 1.6 Å away from the mass centre and towards the main chain axis. The final results are given in Table 6. The translational motion of the antimony triiodide molecule indicated by this analysis is not markedly anisotropic. However, the r.m.s. amplitude of libration varies from 4.5° about one principal axis to 2.5° and 1.1° about the two other axes. The direction of maximum libration is within 20° of the direction of the chain.

Table 6. Rigid-body thermal parameters of antimony triiodide referred to the directions of the orthogonal axes a, b, c^*. The coordinates (monoclinic) of centre of libration are (0.336, 0.831, 0.425). Numbers below U_{ij} are differences ($\times 10^4$) of U_{ij} derived from B_{ij} and those calculated from rigid body parameters.

	$U_{ij} \times 10^4$					
	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sb(1)	197	267	356	− 21	− 44	− 22
	15	2	− 18	− 7	5	18
I(2)	224	307	501	− 20	17	38
	− 15	− 7	28	20	− 12	− 12
I(3)	248	474	411	1	−117	−126
	4	8	− 8	− 26	25	− 7
I(4)	286	360	879	42	11	191
	− 3	− 3	− 2	13	− 19	1

$$\mathbf{T} = \begin{pmatrix} 193 & -22 & -50 \\ & 264 & -17 \\ & & 298 \end{pmatrix} \times 10^{-4}\ \text{Å}^2 \qquad \omega = \begin{pmatrix} 188 & -33 & -23 \\ & 72 & 9 \\ & & 15 \end{pmatrix} \times 10^{-1}(^\circ)^2$$

$$\sigma(\mathbf{T}) = \begin{pmatrix} 13 & 11 & 12 \\ & 12 & 12 \\ & & 16 \end{pmatrix} \times 10^{-4}\ \text{Å}^2 \qquad \sigma(\omega) = \begin{pmatrix} 12 & 9 & 6 \\ & 15 & 9 \\ & & 8 \end{pmatrix} \times 10^{-1}(^\circ)^2$$

Principal axes	Eigenvalues	Direction cosines of eigenvectors		
	0.032 Å^2	0.345	0.144	−0.928
T	0.027	0.292	−0.956	−0.040
	0.017	0.892	0.257	0.371
	20 (°)^2	−0.957	0.259	0.132
ω	6	−0.269	−0.962	−0.058
	1.2	−0.112	0.091	−0.990

The Sb—I bond distances after correcting for librational motion [10] are 0.059, 0.055, and 0.036 Å greater than the value 2.719 Å found in electron diffraction investigation of the vapour.[11] The e.s.d. value in these differences is 0.005 Å. The mean value of the I—Sb—I angles was found equal to 96.2°, which is three degrees smaller than the value found in the free molecule. Similar changes in the antimony triiodide molecule also take place when iodine is the acceptor atom.[1] The lengthening of the Sb—I bonds give substantial support to the view that antimony in the triiodide molecule may act as an acceptor towards n donors.

The dithiane compound contains two different types of nearly linear arrangements S···Sb—I, the S—Sb—I angles being 169.0° and 170.8°. Antimony is thus surrounded by three iodine and two sulphur atoms at the corners of a deformed octahedron, in which the sixth corner is unoccupied. It is interesting to note that the Sb—I distance opposite the empty corner seems a few hundredths of an Å shorter than the two others.

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