HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502.
HOFMANN, K. A. & HÖCHTLEN, F. (1903). *Ber. dtsch chem. Ges.* **36**, 3090.
International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
JONES, P. E. & KATZ, L. (1967). *Chem. Comm.* p. 842.
KÖPF, H., BLOCK, B. & SCHMIDT, M. (1968). *Chem. Ber.* **101**, 272.
PREWITT, C. T. (1965). *ACACA*, E. I. Dupont de Nemours & Company, Wilmington, Delaware.
THOMAS, L. H. & UMEDA, K. (1957). *J. Chem. Phys.* **26**, 239.
WUENSCH, B. J. & PREWITT, C. T. (1965). *Z. Kristallogr.* **122**, 24.

Acta Cryst. (1969). B**25**, 753

The Crystal Structure of the 2:1 Complex between Antimony Trichloride and Naphthalene

BY R. HULME*
Department of Chemistry, King's College, London, W.C.2, England

AND J. T. SZYMAŃSKI
Department of Chemistry, University of Toronto, Toronto 5, Canada

(*Received* 13 *May* 1968)

Crystals of the 2:1 complex between antimony trichloride and naphthalene are monoclinic, $a=9{\cdot}154$, $b=9{\cdot}368$, $c=11{\cdot}989$ Å, $\beta=122°40'$, space group $P2_1/c$. The structure consists of $SbCl_3$ molecules stacked in the *bc* plane alternating with layers of naphthalene molecules in the plane $x=\frac{1}{2}$. The antimony atoms exist in a 4-coordinate, distorted sp^3d trigonal bipyramidal environment, with two chlorine atoms occupying equatorial positions, 2·347 and 2·348 Å away while the third equatorial position is filled by the antimony lone electron pair. The third chlorine atom occupies one axial position 2·367 Å from antimony, while the other axial position is utilized in bonding to the π-system of the naphthalene molecule. The distances from the antimony atom to the carbon atoms of the ring vary from 3·36 to 3·74 Å, and the distance from the antimony atom to the plane of the ring is 3·2 Å. The structure has been refined using anisotropic full-matrix least-squares refinement, the final R index being 0·091 for all 2566 reflexions used, and 0·072 for the 2034 observed terms only.

Introduction

Complexes between antimony trichloride and aromatic hydrocarbons have been known for many years. In the earliest work (Smith & Davies, 1882), the complex between antimony trichloride and naphthalene was reported, and incorrectly analysed as a 3:2 adduct. Menshutkin (1911, 1912*a*, *b*), who studied the eutectic diagrams of antimony trihalides with a large number of organic compounds, defined the composition of these intermolecular compounds as maxima in the phase diagrams. He showed that a series of complexes of antimony trihalides with aromatic hydrocarbons or substituted hydrocarbons could be made, and that the most common ratios for these were 1:1, 2:1 or 4:1. He found that the melting point of the antimony trichloride–naphthalene 2:1 adduct (85°) was higher than that of antimony trichloride (73°) or of naphthalene (80°). To determine the nature of the bonding forces between the constituents, an extensive study of antimony trihalide–aromatic hydrocarbon complexes was undertaken. Preliminary reports on some of these compounds have already been published (Szymański, 1963; Hulme, 1966; Cushen, Hulme & Szymański, in press). A knowledge of the structure of these compounds is important in view of the known catalytic properties of antimony trichloride in Friedel–Crafts reactions (Olah, 1963), and the possible relation of these structures to the reaction intermediates. We here report a more detailed account of the 2:1 adduct between antimony trichloride and naphthalene, whose structure was previously solved in projection and described elsewhere (Szymański, 1963).

Experimental

Antimony trichloride and naphthalene in 2:1 molar ratio were dissolved in hot 60–80° petroleum spirit The solution on cooling deposited a mass of fine crystals, which were then left to stand in the mother liquor for several weeks in a sealed flask. From the mass a number of excellently formed acicular crystals grew. The forms exhibited were {011}, {100}, and less prominently {110}. Chemical analysis, and quantitative estimation of the naphthalene ultraviolet absorption

---

* Present address: Department of Chemistry, University of St. Andrews, Fife, Scotland.

peak at 311mμ, indicated the 2:1 molar ratio which was later confirmed by the crystal structure determination.

The crystal data are as follows:

$SbCl_3.\frac{1}{2}(C_{10}H_8)$ F.W. = 292·2

Monoclinic,

$a=9{\cdot}154$, $b=9{\cdot}368$, $c=11{\cdot}989$ Å, all $\pm0{\cdot}002$ Å
$\beta=122°40' \pm 1'$. (Cu $K\alpha_1=1{\cdot}54051$, $K\alpha_2=1{\cdot}54433$)
$D_m=2{\cdot}22\pm0{\cdot}02$ g.cm^{-3}, $D_c=2{\cdot}24$ g.cm^{-3}.
$Z=4$. $U=865{\cdot}5$ Å^3. μ(Mo $K\alpha$) = 45·5 cm^{-1}.

Space group $P2_1/c$ determined from systematic spectral absences.

The unit-cell dimensions were fitted by least-squares to the values of 4 $\sin^2\theta/\lambda^2$ of 37 $0kl$ and 43 $h0l$ reflexions with $56° \le \theta \le 83°$, with corrections for absorption and eccentricity (Buerger, 1942). The spacings were measured on Weissenberg photographs calibrated with aluminum powder lines ($a=4{\cdot}04934$ Å). The probable errors in the cell parameters were taken arbitrarily as three times the standard deviations calculated from the least-squares matrix. The density was determined by flotation in mixtures of methyl iodide and iodobenzene. As the crystals slowly dissolved with decomposition in this solvent, an accurate density could not be obtained.

An acicular crystal of square cross section (0·04 mm^2) was cut into several pieces in a dry box to give approximate cubes of side 0·2 mm. Two of these were mounted about the a axis (the needle axis) and a third about the b axis. Equi-inclination Weissenberg data were collected for the layers $0kl$ to $7kl$ and $h0l$ to $h10l$ using Zr filtered molybdenum $K\alpha$ radiation and the multiple film technique with sheets of tin foil, 0·001″ thick, between the films. Of the 2566 independant reflexions which were measured, 532 were considered unobservably weak. These were allotted a value of one third of the locally observable minimum intensity ($I_{\min}$) together with a standard deviation of $I_{\min}\sqrt{\frac{4}{45}}$ (Hamilton, 1955).

A standard deviation was estimated for each observed unscaled intensity by considering its magnitude relative to the standard intensity scale used, and from other contributory factors such as spot-extension effects, the presence of Laue streaks and the local background intensity of the film. The intensity data were then correlated and reduced to structure amplitudes with standard deviations using the least-squares procedure of Hamilton, Rollett & Sparks (1965). No absorption or extinction corrections were applied.

Solution of the structure

The structure was solved from the Patterson function by routine application of the heavy atom technique. Antimony trichloride molecules lie in general fourfold positions, while the centre of the naphthalene molecule occupies the $(\frac{1}{2},0,\frac{1}{2})$ special position. The programs used for the least-squares refinement and molecular geometry calculations were *ORFLS* and *ORFFE* (Busing, Martin & Levy, 1962*a*, *b*). Standard scattering factors were used for carbon (*International Tables for X-ray Crystallography*, 1962*a*), for Cl^0 (*International Tables*, 1962*b*) and for Sb^0 (*International Tables*, 1962*c*). The chlorine and antimony curves were adjusted for the real part of the anomalous dispersion correction: 0·1e was added to the Cl^0 curve over the whole $\sin\theta$ range while for antimony a correction which varied linearly from 0·6e at $\sin\theta/\lambda=0$ to 0·8e. at $\sin\theta/\lambda=0{\cdot}9$ was subtracted.

Two cycles of least-squares refinement using isotropic temperature factors, and two with anisotropic lowered the residual to 0·11. At this point, thirteen low angle reflexions which were calculated to be very much larger than they had been observed, and were suspected of being affected by extinction, were excluded from the refinement. Six further cycles of least-squares lowered the residual for all data to 0·097 and for observed reflexions only to 0·077. The data were examined and a further twelve reflexions were removed as being probably extinction affected. Three further cycles of least-squares refinement lowered the overall R to 0·091 and R for observed reflexions only to 0·072. The weighted residuals for all data, and observed data only were 0·084 and 0·081 respectively. In the last cycle, the suggested shifts in all parameters were less than 0·01 of a standard deviation. Despite this, an (F_o-F_c) synthesis calculated at the end of the refinement showed excursions which were above the level of significance. [$\sigma(\varrho_{xyz})$ was about 0·4 e.Å^{-3}]. A negative shell of depth 2·0 e.Å^{-3} and radius 0·4 Å was centred on the antimony position. In addition, some ripple was observed in the vicinity of the antimony position. All three chlorine atoms lay on negative holes of about 1·0 e.Å^{-3}. Carbon atom positions corresponded to featureless areas of the map. Despite the fact that the probable hydrogen atom positions were found to be in positive areas of the map, no significance was attached to these, and hydrogen atoms were not included in the refinement.

The final value of $[\Sigma w_{hkl}\Delta^2{}_{hkl}/(m-n)]^{1/2}$ was 1·10. The atomic coordinates, thermal parameters and all standard deviations as obtained from the least-squares refinement are listed in Tables 1 and 2. Observed and calculated structure factors are listed in Table 3.

Table 1. *The atomic positional fractional coordinates (with their standard deviations in parentheses)*

	$10^5x(10^5\sigma_x)$	$10^5y(10^5\sigma_y)$	$10^5z(10^5\sigma_z)$
Sb	04381 (06)	06638 (05)	32478 (05)
Cl(1)	−11029 (32)	16384 (24)	11042 (21)
Cl(2)	08961 (31)	29346 (23)	42098 (23)
Cl(3)	−20673 (31)	01884 (27)	32956 (29)
C(1)	37389 (128)	12487 (143)	28295 (116)
C(2)	44411 (107)	17142 (089)	40483 (109)
C(3)	50422 (097)	07480 (076)	51223 (084)
C(4)	57938 (106)	12059 (110)	64724 (097)
C(5)	64211 (130)	01452 (180)	74760 (099)

Description of the structure

The structure consists of layers of antimony trichloride molecules near the plane $x=0$, alternating with layers of naphthalene molecules near the plane $x=\frac{1}{2}$. The latter are nearly normal to the a axis (see Fig. 1). Two antimony–chlorine distances are equal within experimental error [Sb–Cl(1) = 2·348, Sb–Cl(2) = 2·347 ± 0·002 Å] while the third is significantly longer [Sb–Cl(3) = 2·367 ± 0·002 Å]. The angles Cl(1)–Sb–Cl(3) and Cl(2)–Sb–Cl(3) are also equal within experimental error (94·42°, 94·71°, $\sigma=0{\cdot}09°$) while the angle opposite the longer antimony–chlorine bond, Cl(1)–Sb–Cl(2) is considerably smaller (91·59°). The antimony atom, and atoms Cl(1) and Cl(2) of one $SbCl_3$ molecule lie in a plane which is only 6·8° away from the plane of the naphthalene molecule. The antimony atom is only 3·2 Å away from this latter plane. The distances from the nearer 6-membered ring to antimony range from 3·36 to 3·73 Å. As the centre of the naphthalene molecule lies on a centre of symmetry $(\frac{1}{2},0,\frac{1}{2})$, there is a second antimony trichloride molecule related by the same centre of symmetry on the other side of the naphthalene molecule. Atom Cl(3), which points away from one naphthalene molecule, is directed towards another naphthalene molecule related to the first by the translation $x=-1$. Its distance from the plane of this second molecule is 3·54 Å and though it points towards the C(4′)–C(5′) bond this longer distance indicates that this is merely a packing arrangement and not an interaction. The projection of the antimony trichloride molecule onto the plane of the first naphthalene is shown in Fig. 2(*a*) while the projection of the structure on the plane normal to the Cl(3)–Sb bond is shown in Fig. 2(*b*).

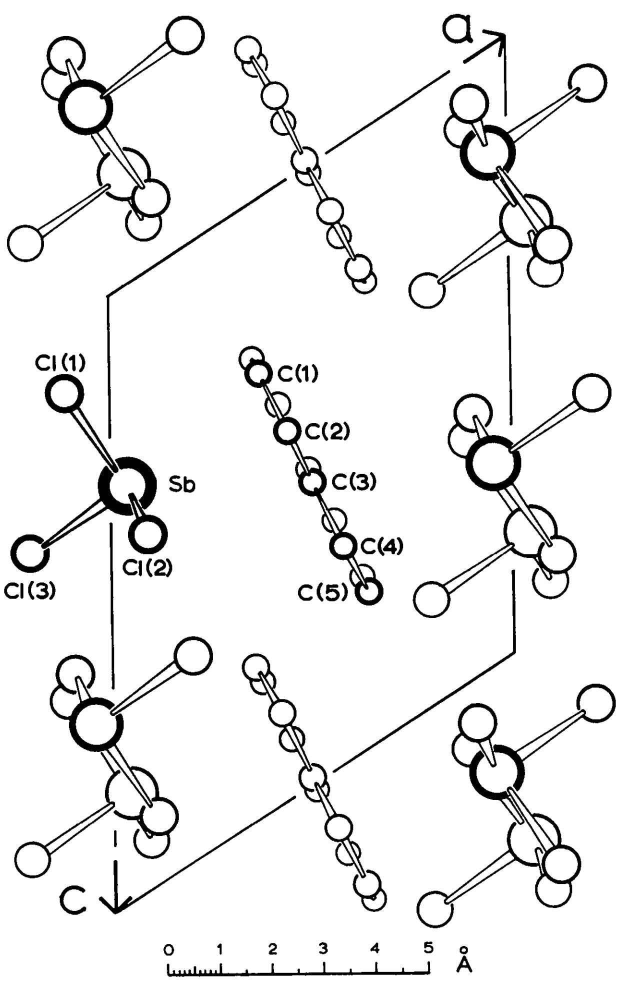

Fig. 1. The structure projected on the *ac* plane. The antimony trichloride molecule related to the asymmetric molecule by the symmetry operation $-x,\frac{1}{2}+y,\frac{1}{2}-z$ has been omitted to aid clarity.

The naphthalene bond distances and angles are considerably distorted from those in free naphthalene (Cruickshank, 1957), and are shown in Fig. 3 and Table 4. Fig. 3 also shows the method of numbering of the carbon atoms used. None of the bond lengths quoted have been corrected for rotational effects. The most significant change in the naphthalene bonding is that two adjacent bonds are very short, [C(2)–C(1) = 1·313 Å, C(1)–C(5′) = 1·343 Å], and these are the two bonds which are closest to the antimony atom. With increasing distance from antimony, the carbon–carbon bond lengths increase in a systematic manner up to the bond furthest away from antimony [C(3′)–C(4′) = 1·440 Å] which is the longest in the molecule. This pattern of bond lengths is completely different from that in free naphthalene, where there is an alternation of shorter and longer bonds. In addition, bonds pre-

Table 2. *Anisotropic temperature factors and their standard deviations* (*in parentheses*) *all* × 10^5

$$\mathrm{T.F.}=\exp-[h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23}].$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Sb	1164 (10)	0866 (07)	0872 (06)	0111 (05)	0611 (06)	0104 (05)
Cl(1)	2135 (43)	1369 (27)	0771 (21)	0239 (28)	0726 (26)	0096 (20)
Cl(2)	2111 (45)	1199 (25)	0975 (24)	−0069 (27)	0798 (28)	−0211 (20)
Cl(3)	1615 (41)	1590 (31)	1553 (34)	−0002 (29)	1053 (32)	0379 (27)
C(1)	1383 (167)	2280 (179)	1033 (119)	0082 (146)	0535 (116)	0492 (126)
C(2)	1279 (146)	1061 (099)	1615 (139)	0130 (095)	0868 (121)	0448 (096)
C(3)	0997 (111)	0948 (088)	0942 (086)	0056 (082)	0524 (082)	−0016 (070)
C(4)	1332 (149)	1693 (127)	1165 (115)	−0331 (115)	0838 (111)	−0610 (101)
C(5)	1330 (168)	4094 (295)	0703 (094)	−1044 (190)	0624 (109)	−0597 (146)

Table 3. *Observed and calculated structure factors*

The columns refer to the running index *l*, $10F_o$ and $10F_c$. Reflexions marked with an *X* were excluded from the refinement. Reflexions marked with an asterisk were unobserved, and are included here at the statistically probable value used in the refinement.

H = 0,K = 0		
2X	841	-1116
4X	831	-1008
6	703	733
8	482	-518
10	76	115
12	292	290
14	183	-176
H = 0,K = 1		
2X	588	679
3X	1154	1818
4	274	-304
5X	1104	-1286
6	402	402
7	225	-227
8	112	95
9	548	567
10	341	-366
11	250	-276
12	77	-25
H = 0,K = 2		
0X	761	1180
1X	899	-1511
2X	986	-1298
3	87	87
4	126	107
5	702	752
6X	921	1068
7	394	-409
8	333	-351
9	171	169
10	89	-90
11*	49	-3
12	150	155
13	148	-152
H = 0,K = 3		
1X	703	-892
2	787	899
3	693	766
4X	1128	-1408
5*	30	-6
6	233	226
7	57	31
8	405	412
9	83	85
10	299	-331
11	210	-198
12	143	140
H = 0,K = 4		
0	75	-57
1X	856	-1206
2	52	-46
3	189	198
4	160	160
5	526	583
6*	32	-28
7	740	-795
8	163	-153
9	303	308
10	92	-49
11	247	263
12*	48	39
13	143	-153
H = 0,K = 5		
1	507	568
2	978	1131
3	366	-377
4	693	-752
5	65	22
6*	38	46
7*	41	-10
8	203	230
9	121	-109
10	267	-295
11	90	114
12	156	139
H = 0,K = 6		
0	390	-392
1	444	-504
2	327	338
3	298	267
4*	35	-75
5	481	508
6	541	-564
7	403	-422
8	289	315
9*	53	25
10	98	97
11*	60	36
12	126	-138
H = 0,K = 7		
1	237	263
2	204	206
3	569	-598
4	212	-200
5	392	414
6	133	113
7	210	186
8	119	75
9	175	-182
10	140	-139
11	148	159
H = 0,K = 8		
0	638	-738
1	114	-120
2	202	220
3	225	-227
4	228	214
5	58	-65
6	306	-299
7	132	107
8	247	225
9	81	62
10*	50	-53
11*	53	-44
12	112	-114
H = 0,K = 9		
1	162	196
2	251	-282
3	266	-275
4	96	106
5	254	243
6*	45	-12
7*	41	-35
8*	52	-31
9	175	-189
10	112	122
11	94	114
H = 0,K = 10		
0	159	-168
1	332	337
2	129	144
3*	31	55
4*	37	50
5	167	-180
6	147	-149
7	129	141
8*	46	48
9	85	-69
10	90	-33
H = 0,K = 11		
1*	62	-3
2	153	-142
3*	63	-56
4	322	307
H = 0,K = 12		
0	81	85
1	181	204
2*	67	-39
3*	67	-35
4*	68	-43
5	155	-125
H = 0,K = 14		
0	165	144
H = 1,K = 0		
14	163	-119
12	242	225
10*	48	46
8	643	-665
6	561	597
4	650	-713
2*	23	23
0X	1104	2144
-2X	1056	-1545
-4X	1173	-1335
-6	732	786
-8	512	-535
-10	167	179
-12	191	185
-14	240	-212
H = 1,K = 1		
11	166	-165
10	238	-235
9	473	456
8	387	362
7	198	-193
6	243	242
5	792	-845
4	456	-439
3X	1237	1565
2	476	512
1	268	-228
0	219	-206
-1	56	-87
-2	587	-528
-3	1010	1049
-4	246	215
-5	818	-881
-6	305	-304
-7*	38	-28
-8*	42	-9
-9	598	650
-10	360	383
-11	347	-363
-12*	47	-63
-13*	50	71
-14	91	-101
H = 1,K = 2		
14	106	-72
13	183	-175
12*	55	80
11*	52	78
10*	49	-18
9	100	76
8	279	-266
7	409	-424
6	749	792
5	692	751
4	328	-322
3	268	-243
2X	989	-1249
1X	823	-935
0	579	549
-1X	1153	1426
-2	407	-387
-3	431	-410
-4	79	-69
-5	818	-821
-6	836	844
-7	565	592
-8	503	-533
-9*	41	-14
-10*	44	-34
-11*	47	26
-12	87	99
-13	93	117
-14	127	-111
-15	134	-133
H = 1,K = 3		
12	131	107
11	146	-147
10	283	-268
9*	58	48
8	451	465
7	262	270
6*	46	-16
5*	42	-45
4	977	-1134
3	420	411
2X	996	1189
1	772	-859
0	64	-52
-1	847	-945
-2	706	-722
-3	1010	1095
-4	1083	1244
-5	330	315
-6	520	-484
-7	189	-176
-8	415	-431
-9*	52	-4
-10	293	332
-11	113	-132
-12	171	-184
H = 1,K = 4		
13	123	-121
12*	52	-35
11	245	238
10*	46	-4
9	75	107
8	90	-94
7	668	-709
6*	40	12
5	536	574
4	324	315
3	57	-33
2	309	-304
1	1016	-1152
0	135	138
-1X	1089	1320
-2	482	478
-3	140	-123
-4	260	-252
-5	317	-313
-6	68	-73
-7	680	711
-8*	42	-36
-9	466	-507
-10*	42	-4
-11	156	-148
-12*	48	35
-13	243	242
H = 1,K = 5		
12	115	110
11	84	90
10	251	-252
9	215	-214
8	180	166
7	65	49
6	133	120
5	197	-182
4	460	-466
3	327	-306
2	900	987
1	790	890
0	278	-275
-1*	29	-11
-2	968	-1047
-3	587	-625
-4	812	844
-5	127	131
-6	206	-208
-7	234	-212
-8	148	-121
-9	87	-86
-10	213	230
-11	223	204
-12	213	-189
H = 1,K = 6		
10	167	169
9*	50	19
8	160	156
7	264	-257
6	532	-534
5	420	400
4	400	411
3	310	285
2	313	315
1	544	-532
0	534	-527
-1	286	320
-2	560	602
-3	349	-377
-4	230	-225
-5	240	-240
-6	300	-302
-7	452	460
-8	537	548
-9	78	-81
-10*	48	-53
-11*	51	14
-12	122	-155
H = 1,K = 7		
11	108	119
10	79	-81
9	191	-204
8*	40	59
7	235	245
6	159	153
5	128	135
4	251	-226
3	776	-785
2*	24	2
1	470	475
0	102	110
-1*	14	-14
-2	74	-79
-3	664	-724
-4	119	117
-5	600	616
-6	135	-131
-7	57	-12
-8*	36	31
-9	173	-197
-10	93	85
-11	140	150
-12*	47	-38
-13	111	-124
H = 1,K = 8		
9	85	95
8	205	184
7	74	52
6	341	-321
5	218	-226
4	361	348
3	58	-41
2	138	130
1	81	54
0	627	-694
-1	64	62
-2	624	618
-3	206	226
-4	106	107
-5*	35	-27
-6	291	-307
-7	220	-217
-8	286	291
-9*	48	29
-10	114	-111
H = 1,K = 9		
10	92	90
9	235	-225
8	105	-101
7	62	84
6*	40	10
5	212	224
4	75	60
3	376	-354
2	309	-317
1	240	248
0	187	219
-1	43	43
-2	198	239
-3	272	-314
-4	245	-266
-5	305	319
-6	112	112
-7	166	-163
-8*	42	19
-9	144	-162
-10	154	-135
-11	147	151
H = 1,K = 10		
8	83	60
7	174	146
6	186	-150
5	208	-179
4	109	97
3	168	143
2	103	102
1	190	191
0	137	-171
-1	359	-446
-2	156	206
-3	135	159
-4	50	-61
-5	147	147
-6	164	-156
-7	220	-231
-8	97	92
-9	105	120
H = 1,K = 11		
-4	300	-318
H = 2,K = 0		
12	260	223
10	125	-118
8	323	-310
6	409	-398
4	334	-333
2	646	644
0X	859	1694
-2X	1051	-1351
-4	567	-556
-6	744	713
-8	450	-421
-10	426	422
-12*	46	38
-14	160	-170
-16	115	139
H = 2,K = 1		
11	102	-100
10	82	-82
9	321	299
8	271	264
7	474	-463
6*	44	-7
5	215	-208
4	236	-234
3	817	833
2	500	457
1	421	-403
0	295	-247
-1	875	-722
-2	577	-578
-3	538	460
-4	214	186
-5	1038	-1084
-6	397	-362
-7	496	488
-8	363	338
-9	216	219
-10	421	413
-11	410	-379
-12	226	-240
-13	108	111
H = 2,K = 2		
11	125	115
10*	59	-47
9*	55	58
8*	45	-10
7	153	-141
6	498	513
5	481	487
4	390	-416
3	436	-435
2	452	-451
1	390	-384
0	303	296
-1	997	1161
-2	181	-162
-3	864	-924
-4	683	683
-5	444	-401
-6	459	435
-7	737	790
-8	531	-525
-9	302	-300
-10	143	126
-11*	46	44
-12*	49	35
-13*	52	51
-14	95	-101
-15	130	-172
H = 2,K = 3		
11	120	-115
10	191	-181
9	151	137
8	326	306
7	179	159
6	282	-274
5*	44	8
4	660	-636
3	436	-393
2	925	1013
1	535	-544
0	433	-413
-1X	408	393
-2	150	-163
-3	711	719
-4	1026	1174
-5	144	-124
-6	853	-902
-7	144	-126
-8	131	-113
-9	178	-159
-10	408	414
-11*	55	6
-12	286	-289
H = 2,K = 4		
11	225	240
10*	57	18
9*	54	-45
8*	50	-30
7	470	-485
6	65	-71
5	604	630
4	221	209
3	234	-229
2	64	49
1	502	-451
0	433	-408
-1	1046	1194
-2	401	337
-3	582	-631
-4*	28	-12
-5	108	-104
-6	392	-404
-7	431	467
-8	77	83
-9	650	-691
-10*	59	38
-11*	51	53
-12*	54	43
-13	254	260
-14*	52	35
-15	95	-102
H = 2,K = 5		
10	251	-225
9	244	-216
8	222	212
7	130	108
6*	44	38
5	137	113
4	324	-299
3	262	-231
2	685	689
1	898	884
0	726	-750
-1	62	-61
-2	372	-363
-3	781	-875
-4	871	899
-5	389	429
-6	345	-331
-7*	38	-1
-8	60	-19
-9	65	-79
-10	199	177
-11	247	258
-12	194	-186
H = 2,K = 6		
10	134	148
9*	60	35
8*	57	44
7	156	-147
6	559	-564
5	437	430
4	433	409
3*	39	-30
2	228	175
1	435	-418
0	714	-712
-1	201	202
-2	597	625
-3	389	-379
-4	349	-357
-5	102	98
-6	369	-388
-7	339	356
-8	545	587
-9	159	-143
-10	216	-202
-11*	57	16
-12	114	-173
H = 2,K = 7		
11	87	115
10*	48	-43
9	169	-121
8*	50	45
7	232	232
6	68	66
5	110	105
4	177	-169
3	629	-623
2	163	114
1	709	694
0	89	84
-1	134	-129
-2	33	-24
-3	511	-517
-4	110	-102
-5	761	909
-6	84	-87
-7	111	-106
-8	152	142
-9	239	-261
-10	69	50
-11	202	196
-12*	64	-78
-13	153	-155
H = 2,K = 8		
9	101	86
8	125	130
7*	54	-5
6	322	-302
5	265	-260
4	328	333
3*	39	31
2	115	-93
1	172	176
0	637	-668
-1*	25	-5
-2	658	696
-3	183	168
-4	123	-131
-5	53	-39
-6	332	-335
-7	228	-234
-8	359	357
-9*	51	76
-10	229	-198
H = 2,K = 9		
10	97	88
9	126	-147
8	111	-131
7	130	142
6	73	51
5	159	145
4	74	81
3	387	-359
2	343	-317
1	389	371
0	292	284
-1	39	25
-2	204	215
-3	248	-261
-4	306	-341
-5	328	322
-6	89	113
-7	246	-258
-8*	48	3
-9	76	-85
-10	124	-136
-11	242	244
-12	92	93
H = 2,K = 10		
7	107	120
6	139	-128
5	247	-227
4	129	125
3	190	180
2	81	76
1	168	159
0	177	-176
-1	515	-477
-2	198	216
-3	220	217
-4	147	-121
-5	128	142
-6	156	-139
-7	229	-222
-8	142	144
-9	170	162
H = 2,K = 11		
4	257	241
3*	71	-58
2	332	-326
1*	69	24
0*	68	76
-1*	68	-18
-2*	68	80
-3*	68	-56
-4	289	-275
-5*	70	56
-6	277	258
H = 2,K = 12		
-1	281	-270
-2*	73	-52
-3	163	172
H = 2,K = 13		
-4	174	-164
H = 3,K = 0		
12	177	147
10	260	-240
8	183	-159
6	370	358
4	462	-483
2	862	915
0	455	442
-2X	1818	-3266
-4	998	1007
-6	481	446
-8	618	-629
-10	466	456
-12*	46	-40
-14	230	-227
-16	156	155
H = 3,K = 1		
9	187	191
8	265	262
7	422	-406
6	59	-57
5	231	205
4	249	-221
3	788	876
2	419	420
1	1135	-1255
-1	1041	1021
-2	354	-327
-3	328	315
-4	410	388
-5	1043	-1148
-6	114	-138
-7	948	1022
-8	446	442
-9	104	104
-10	127	112
-11	456	-476
-12	376	-361
-13	304	281
H = 3,K = 2		
11	128	131
10	94	-102
9*	51	-12
8	134	111
7	235	-220
6	238	241
5	423	399
4	734	-796
3	434	-446
2*	34	-40
1	83	-78
0	560	536
-1	924	990
-2	688	-675
-3	1127	-1299
-4	1004	1119
-5	151	91
-6	229	-197
-7	749	775
-8	699	-736
-9	348	-317
-10	308	297
-11	82	95
-12*	52	5
-13*	55	-11
-14	146	-153
-15	158	-137
-16	110	116
H = 3,K = 3		
10	143	-125
9	146	150
8	309	289
7*	52	-14
6	353	-322
5*	45	5
4	298	-291
3	195	-172
2	977	1093
1	496	-465
0	670	-668
-1	757	756
-2	62	46
-3	332	298
-4	799	831
-5	394	-368
-6	1063	-1178
-7	110	94
-8	130	123
-9	99	-84
-10	358	341
-11	65	-60
-12	330	-310
-13	144	141
-14	136	134
-15	82	-69
H = 3,K = 4		
11	99	128
10*	58	-19
9	162	-160
8	92	68
7	266	-252
6*	46	-15
5	547	592
4	56	76
3	530	-532
2*	55	35
1	261	-238
0	133	-117
-1	991	1120
-2	284	259
-3	782	-804
-4*	25	-15
-5	164	156
-6	407	-403
-7	410	413
-8	166	165
-9	694	-769
-10	179	178
-11	283	294
-12*	51	-2
-13	268	277
-14*	57	-13
-15	135	-144
H = 3,K = 5		
10	120	-132
9	163	-150
8	259	253
7	72	75
6	123	-125
5	65	56
4	66	-68
3	176	-170
2	542	526
1	506	504
0	817	-848
-1	316	-292
-2	48	37
-3	771	-749
-4	852	904
-5	424	418
-6	665	-735
-7	55	-56
-8	59	60
-9	114	-100
-10	281	250
-11	204	203
-12	247	-253
-13*	62	-76
-14	161	118
H = 3,K = 6		
10	108	104
9*	65	-26
8*	62	-34
7*	59	-15
6	333	-326
5	336	328
4	548	541
3	338	-317
2	160	142
1	223	-183
0	589	-568
-1	331	303
-2	522	490
-3	511	-488
-4	171	-153
-5	278	252
-6	104	-103
-7	439	450
-8	527	574
-9	344	-348
-10	283	-293
-11*	54	71
-12	120	-124
-13*	60	21
-14	172	165
-15	87	-86
H = 3,K = 7		
9	174	-166
8	79	72
7	203	191
6	69	-50
5*	44	-43
4*	41	-34
3	522	-513
2	123	110
1	512	480
0	167	-149
-1	199	-173
-2	40	19
-3	426	-395
-4	87	95
-5	712	726
-6	160	-156
-7	317	-303
-8	204	185
-9	284	-280
-10*	46	28
-11	175	191
-12	141	-151
-13	140	-152
H = 3,K = 8		
10	102	91
9*	69	45
8	94	93
7*	45	-43
6	259	-235
5	148	-163
4	285	273
3	64	74
2	113	-99
1	293	264
0	399	-359
-1*	32	5
-2	724	709
-3	88	-76
-4	132	-114
-5	90	-78
-6	301	-290
-7	118	-114
-8	356	343
-9*	49	55
-10	229	-214
-11*	55	58
-12*	58	3
-13*	61	-55
-14	106	125
H = 3,K = 9		
9	123	-116
8	111	-121
7	128	136
6*	54	25
5*	51	61
4	71	50
3	286	-269
2	177	-177
1	316	309
0	266	259
-1	89	-74
-2	107	107
-3	326	-303
-4	339	-313
-5	307	266
-6	99	97
-7	238	-218
-8*	47	30
-9*	50	-4
-10	79	-85
-11	197	215
-12	115	114
-13	113	-105
H = 3,K = 10		
7	110	95
6	83	-87
5	158	-147
4	89	107
3	174	149
2*	43	30
1*	39	16
0	156	-136
-1	397	-373
-2	171	178
-3	297	289
-4	154	-150
-5	108	103
-6	136	-122
-7	231	-230
-8	203	181
-9	149	144
H = 3,K = 11		
8	91	-101
7*	73	-24
6	111	108
5*	48	22
4	106	114
3*	46	-36
2	320	-305
1*	45	43
0	119	120
-1*	62	16
-2*	61	56
-3*	62	-22
-4	240	-205
-5*	63	78
-6	285	271
-7*	65	-18
-8*	66	-13
-9*	68	-6
-10	140	-132
H = 3,K = 12		
5	144	-127
4*	70	-67
3	97	96
2*	68	28
1	82	76
0*	66	4
-1	254	-218
-2*	65	-74
-3	207	180
-4*	66	50
-5*	66	11
-6*	67	55
-7	153	-152
-8*	70	-56
-9	113	115
H = 4,K = 0		
10	257	-215
8*	44	-27
6	561	570
4	512	-562
2	775	752
0	151	141
-2X	1333	-1748
-4	1157	1293
-6	769	731
-8	907	-910
-10	711	678
-12	88	-97
-14	211	-176
-16	186	164
H = 4,K = 1		
9	160	153
8	151	163
7	405	-428
6	224	-240
5	253	250
4*	40	-66
3	582	627
2	386	382
0	619	-586
1	1180	-1365
-1	629	554
-2	85	-77
-3	210	184
-4	587	471
-5	1075	-1167
-6	159	-107
-7	940	931
-8	398	316
-9*	38	-31
-10*	41	38
-11	635	-581
-12	439	-383
-13	429	384
-14*	57	46
-15	95	-69
-16*	64	96
-17	106	-74
H = 4,K = 2		
9	73	-91
8*	56	50
7	91	-65
6	223	248
5	398	417
4	651	-755
3	479	-535
2	376	399
1	117	104
0	690	724
-1	601	611
-2	800	-744
-3	1124	-1165
-4	763	773
-5	476	401
-6	152	98
-7	817	817
-8	822	-819
-9	627	-589
-10	563	523
-11*	46	-32
-12*	49	-57
-13*	52	18
-14	117	-111
-15	145	-110
-16	179	126
-17	166	115
H = 4,K = 3		
10	106	-85
9	129	136
8	222	221
7	80	-89
6	360	-398
5	132	-136
4*	44	-38
3*	41	10
2	788	914
1	482	-487
0	842	-934
-1	853	852
-2	163	159
-3	520	472
-4	756	754
-5	914	-925
-6	1046	-1136
-7	192	127
-8	382	315
-9	78	64
-10	458	419
-11	122	-107
-12	370	-325
-13	138	117
-14	198	179
-15	76	-62
H = 4,K = 4		
11	127	113
10*	54	23
9	166	-161
8*	54	1
7	75	-101
6*	48	38
5	484	526
4	60	-76
3	502	-565
2*	37	30
1	49	63
0	191	142
-1	1009	1118
-2	337	-305
-3	1021	-1097
-4	139	-117
-5	54	48
-6*	34	30
-7	275	287
-8*	38	23
-9	615	-689
-10	171	160
-11	450	445
-12*	49	-42
-13	204	189
-14*	55	-37
-15	258	-237
H = 4,K = 5		
9	89	-95
8	214	229
7	134	126
6	148	-140
5*	46	44
4	138	-137
3	128	120
2	442	437
1	197	201
0	750	-829
-1	421	-401
-2	276	256
-3	236	-203
-4	864	859
-5	376	337
-6	815	-831
-7*	37	30
-8	240	209
-9	85	94
-10	257	224
-11	141	121
-12	297	-237
-13	137	-124
-14	154	141
H = 4,K = 6		
6	120	-138
5	252	255
4	332	355
3	307	-332
2	219	-230
1	217	-216
0	364	-367
-1	458	433
-2	438	415
-3	483	-464
-4	304	-263
-5	373	335
-6*	39	5
-7	353	312
-8	257	231
-9	426	-430
-10	395	-371
-11*	52	79
-12*	56	7
-13*	57	-7
-14	152	144
H = 4,K = 7		
9	87	-95
8	83	48
7	162	176
6*	50	-53
5	89	-116
4*	44	-76
3	202	-207
2	216	222
1	490	518
0	126	-116
-1	259	-249
-2	58	-84
-3	47	-44
-4	130	119
-5	661	662
-6	146	-141
-7	429	-412
-8	155	147
-9	163	-131
-10*	45	-16
-11	169	177
-12	135	-119
-13	138	-106
H = 4,K = 8		
7	91	-68
6	152	-160
5*	52	-54
4	237	230
3	150	140
2	235	-217
1	63	79
0	210	-202
-1*	38	-23
-2	562	559
-3	102	-88
-4	398	-388
-5	96	-91
-6	187	-174
-7*	43	12
-8	290	286
-9	73	84
-10	231	-225
-11*	48	-14
-12*	50	36
-13*	53	-16
-14	97	83
H = 4,K = 9		
9	102	-51
8	97	-78
7	155	151
6*	57	63
5*	54	-40
4	63	23
3	199	-181
2	68	-76
1	310	315
0	217	208
-1	150	-132
-2	76	-81
-3	161	-151
-4	218	-188
-5	247	247
-6	159	155
-7	217	-215
-8	70	-72
-9	71	91
-10*	52	-26
-11	193	173
-12	102	103
-13	128	-124
H = 4,K = 10		
5	119	-132
4	122	111
3	118	116
2*	48	-11
1	76	-52
0	113	-95
-1	191	-182
-2	126	120
-3	303	302
-4	171	-164
-5*	41	-78
-6*	57	-9
-7	132	-126
-8	185	171
-9	190	186
H = 4,K = 11		
6	129	113
5*	67	19
4*	65	27
3*	64	-20
2	220	-233
1*	61	22
0	165	151
-6	253	234
-7*	62	-12
-8	140	-137
-9*	64	-4
-10	93	-80
H = 4,K = 12		
5	111	-96
-1	141	-145
-2*	63	-29
-3	199	189
-4	100	106
H = 4,K = 13		
0	126	119
-4	105	-114
-5	106	-98
H = 5,K = 0		
10	217	-195
8	80	72
6	335	319
4	432	-424
2	269	247
0	85	82
-2	1435	-1608
-4	852	986
-6	499	459
-8	833	-820
-10	509	490
-12	125	-149
-14	186	-152
-16	138	122
H = 5,K = 1		
9	170	115
8*	51	80
7	225	-261
6	178	-132
5	320	306
4*	48	29
3	381	356
2	191	174
1	833	-889
0	529	-506
-1	485	450
-2*	34	55
-3	773	740
-4	566	531
-5	965	-925
-6	71	-87
-7	822	841
-8	263	247
-9	122	-138
-10	279	-249
-11	312	-305
-12	335	-350
-13	303	332
-14	187	150
H = 5,K = 2		
6	111	85
5	201	215
4	513	-508
3	367	-367
2	211	219
1	125	132
0	590	573
-1	399	359
-2	695	-641
-3	828	-810
-4	447	373
-5	700	628
-6	123	-108
-7	478	423
-8	657	-609
-9	683	-660
-10	385	374
-11	180	183
-12	81	-45
H = 5,K = 3		
9	138	112
8	155	162
7	125	-149
6	270	-258
5	87	-125
4	106	115
3	173	149
2	583	593
1*	47	25
0	741	-736
-1	310	304
-2	273	237
-3	67	72
-4	415	-938
-5	688	-650
-6	991	-981
-7	241	217
-8	523	517
-9	133	143
-10	185	179
-11*	71	-80
-12	356	-331
-13*	75	30
-14	176	214
H = 5,K = 4		
9	122	-139
8*	52	-35
7*	49	-21
6*	47	63
5	378	379
4*	49	27
3	522	-522
2	119	-117
1	168	160
0	107	129
-1	675	621
-2*	37	-5
-3	940	-938
-4	275	-239
-5	243	241
-6	190	166
-7	233	224
-8	181	176
-9	398	-416
-10*	46	-7
-11	474	516
-12*	44	-3
-13*	47	28
-14*	49	-72
-15	201	-227
-16*	54	-7
-17	99	79
H = 5,K = 5		
8	200	203
7	139	133
6	233	-195
5	99	-79
4	93	-81
3*	40	-1
2	383	355
1	123	130
0	579	-549
-1	642	-615
-2	404	385
-3	97	89
-4	436	414
-5	421	409
-6	750	-747
-7	261	-271
-8	220	222
-9	83	95
-10	159	171
-11*	48	55
-12	199	-210
-13	180	-177
-14	155	104
H = 5,K = 6		
5	216	187
4	402	382
3	346	-352
2	326	-314
1	71	-47
0*	46	-60
-1	402	386
-2	493	478
-3	275	-262
-4	412	-410
-5	216	215
-6	254	257
-7	150	161
-8	205	224
-9	278	-308
-10	351	-397
-11*	48	57
-12	160	165
-13*	55	6
-14	97	119
H = 5,K = 7		
7	137	153
6*	46	-36
5	219	-184
4*	42	-47
3	152	-131
2	165	181
1	433	402
0	168	-171
-1	466	-467
-2	155	-123
-3	166	170
-4	182	180
-5	358	356
-6*	36	34

Table 3 (*cont.*)

-7	620	-669
-8•	35	-2
-9•	37	41
-10	68	-50
-11	185	187
-12	97	-71
H = 5,K = 8		
6	119	-101
5•	55	26
4	239	224
3	185	163
2	239	-247
1•	47	-40
0	67	59
-1•	42	-40
-2	511	484
-3•	40	-3
-4	456	-456
-5	179	-170
-6	74	87
-7	67	60
-8	223	226
-9	95	110
-10	276	-272
-11•	48	-67
-12	100	141
H = 5,K = 9		
7	127	101
6•	54	86
5	89	-92
4•	49	-52
3•	46	-84
2•	44	-41
1	295	278
0	262	231
-1	317	-286
-2	214	-206
-3	81	-71
-4	139	-113
-5	202	179
-6	226	217
-7	216	-197
-8	131	-125
-9	185	190
-10•	46	23
-11•	48	68
-12	114	117
-13	170	-183
H = 5,K =10		
5	74	-87
3	164	160
4	87	109
2	78	-65
1	148	-139
0	70	-60
-1	123	-114
-2	119	99
-3	372	338
-4	185	-166
-5	223	-175
-6	78	79
-7	81	-80
-8	135	122
-9	174	183
-10	106	-87
-11	141	-133
H = 6,K = 0		
8	156	134
6	224	192
4	285	-270
2	251	229
0	298	-274
-2	406	-392
-4	1349	1335
-6	348	-311
-8	354	-316
-10	499	469
-12	218	-212
-14	87	-81
-16	133	142
-18	80	-100
H = 6,K = 1		
8	121	42
7	116	-141
6	110	-148
5	179	184
3•	42	12
4•	63	27
2	126	110
1	582	-588
0	378	-373
-1	581	578
-2	371	338
-3	351	-290
-4	365	335
-5	354	-331
-6	212	-201
-7	552	530
-8	37	57
-9	400	-385
-10	299	-285
-11	142	-146
-12	100	-103
-13	312	319
-14	225	245
-15	205	-198
-16	72	34
H = 6,K = 2		
5	223	220
4	192	-190
3	333	-323
2	363	369
1	185	169
0	234	212
-1	148	139
-2	339	-319
-3	567	-571
-4	283	279
-5	748	745
-6	351	-339
-7	39	13
-8	71	-63
-9	605	-575
-10	375	349
-11	192	186
-12	146	-121
-13•	49	-22
-14•	52	-3
-15•	56	-10
-16	118	130
H = 6,K = 3		
7	71	-90
6	239	-225
5•	52	-49
4	178	165
3	72	86
2	308	305
1	48	53
0	658	-695
-1	119	113
-2	372	367
-3	159	-137
-4	96	87
-5	197	-159
-6	668	-637
-7	51	54
-8	673	671
-9•	39	26
-10•	42	7
-11•	45	-34
-12	233	-221
-13•	51	47
-14	237	227
-15	69	-39
H = 6,K = 4		
7	91	58
6•	50	-18
5	197	194
4	79	67
3	37	-39
2	149	-142
1	347	327
0	47	67
-1	329	316
-2•	35	19
-3	776	-801
-4	362	-337
-5	362	350
-6	267	236
-7•	35	-15
-8	248	246
-9	282	-271
-10	198	-201
-11	488	521
-12•	47	-56
-13	159	-175
-14•	53	-1
-15	226	-213
-16•	54	-10
-17	98	106
H = 6,K = 5		
8	95	109
7	86	125
6	177	-182
5	95	-83
4•	49	30
3•	47	30
2	150	143
1	171	175
0	360	-344
-1	483	-488
-2	398	395
-3	265	246
-4	79	77
-5	491	464
-6	549	-556
-7	385	-376
-8	466	486
-9	208	204
-10•	43	-4
-11	63	85
-12	197	-215
-13	196	-206
-14	168	157
H = 6,K = 6		
5	70	53
4	242	240
3	211	-203
2	441	-446
1	75	85
0•	43	-24
-1	165	163
-2	448	460
-3	170	-184
-4	498	-486
-5	307	284
-6	166	156
-7	71	-67
-8	104	103
-9	298	-292
-10	422	-445
-11	162	174
-12	221	250
-13	68	-66
-14	71	73
-15	61	-16
-16	143	-143
H = 6,K = 7		
7	141	140
6•	56	-27
5	185	-177
4•	51	-2
3•	48	-2
2•	46	40
1	428	417
0	68	-48
-1	378	-364
-2•	40	17
-3	187	188
-4	75	59
-5	345	324
-6•	39	-21
-7	574	-619
-8•	42	30
-9	227	239
-10	133	-127
-11	188	204
-12•	50	8
-13	135	-164
-14	95	97
-15	141	131
H = 6,K = 8		
4	212	193
3	156	134
2	224	-235
1	81	-100
0•	47	55
-1	159	-132
-2	330	317
-3•	44	72
-4	536	-546
-5•	44	-46
-6	99	103
-7•	43	17
-8	227	212
-9	124	114
-10	291	-296
-11•	52	-50
-12	188	180
H = 6,K = 9		
1	222	221
0	194	181
-1	249	-268
-2	212	-216
-3•	46	38
-4	96	-83
-5	248	251
-6	262	252
-7	225	-216
-8	127	-136
-9	186	180
-10•	47	-11
-11•	54	10
-12	93	111
-13	177	-166
H = 6,K =10		
4	102	91
3	112	123
2	96	-73
1	133	-126
0•	51	-42
-1•	50	-27
-2	91	93
-3	301	314
-4	154	-153
-5	251	-242
-6	128	134
-7•	56	-60
-8	90	104
-9	211	220
-10	159	-142
-11	151	-144
H = 6,K =11		
0	237	230
-1•	60	-32
-2	133	-124
-3•	42	-5
-4•	59	-49
-5•	59	15
-6	187	211
-7•	60	-61
-8	235	-215
H = 6,K =12		
-3	177	182
-4•	62	62
-5	153	-147
-6•	63	-32
-7•	63	-12
-8•	64	-63
-9	146	136
H = 7,K = 0		
8	166	134
6•	48	9
4	340	-327
2	361	343
0	487	-458
-2	93	-93
-4	806	795
-6	766	-694
-8	217	-195
-10	597	577
-12	506	-510
-14•	44	39
-16	110	103
-18	124	-95
H = 7,K = 1		
5	249	265
4	116	124
3	173	-163
2•	39	-13
1	262	-277
0	264	-259
-1	677	697
-2	397	388
-3	354	-363
-4	104	111
-5•	36	-49
-6	273	-277
-7	691	641
-8	101	79
-9	580	-565
-10	244	-228
-11	115	96
-12•	39	-58
-13	328	319
-14	264	263
-15	285	-272
-16•	50	-35
-17	84	55
H = 7,K = 2		
4	144	-147
3	302	-300
2	387	403
1	290	291
0	269	-259
-1•	31	-15
-2	215	-215
-3	378	-331
-4	397	369
-5	676	698
-6	536	-511
-7	293	-268
-8•	28	-20
-9	529	-538
-10	413	412
-11	437	438
-12	326	-326
-13•	43	11
-14	103	107
-15•	48	-41
-16	94	73
-17	84	90
-18	89	-94
H = 7,K = 3		
7	85	-74
6	154	-152
5•	50	45
4	251	240
3•	44	46
2	63	69
1	82	70
0	514	-540
-1	209	210
-2	526	557
-3	468	-458
-4	149	-145
-5	68	-57
-6	556	-528
-7	381	366
-8	707	735
-9	214	-189
-10	180	-182
-11•	39	43
-12	281	-274
-13	74	75
-14	249	254
-15	59	-49
-16	114	-144
H = 7,K = 4		
3	300	-305
2•	42	-6
1	362	378
0	83	-109
-1•	35	-36
-2	101	72
-3	714	-726
-4	80	57
-5	657	657
-6	46	47
-7	71	-43
-8	141	131
-9	180	-177
-10	91	-107
-11	481	498
-12	69	-97
-13	287	-302
-14	77	62
-15	129	-138
-16•	53	-4
-17	180	176
H = 7,K = 5		
7	88	83
6	144	-150
5	80	-91
4	76	79
3	66	-59
2	112	117
1•	43	29
0	295	-296
-1	248	-249
-2	469	483
-3	155	143
-4	176	-171
-5	289	296
-6	464	-469
-7	298	-295
-8	552	561
-9•	38	19
-10	178	-190
-11•	42	23
-12	181	-184
-13	150	-170
-14	166	184
-15	89	76
-16	107	-94
H = 7,K = 6		
4	136	135
3	176	-168
2	226	-239
1	167	166
0	111	121
-1	89	109
-2	366	365
-3	218	-216
-4	395	-412
-5	335	336
-6	149	149
-7	243	-222
-8	154	153
-9	214	-198
-10	240	-241
-11	244	234
-12	230	237
-13	121	-86
-14•	50	17
-15•	52	17
-16	123	-122
H = 7,K = 7		
7	93	76
6•	56	-13
5	112	-143
4•	51	14
3•	48	25
2•	44	32
1	221	229
0	123	-119
-1	357	-364
-2	76	79
-3	198	140
-4•	39	9
-5	145	150
-6•	39	-30
-7	516	-566
-8	65	74
-9	248	268
-10	131	-132
-11	137	154
-12•	47	38
-13	147	-174
-14	82	87
-15	86	94
H = 7,K = 8		
4	121	136
3•	52	60
2	164	-174
1	76	-89
0	76	109
-1•	45	-60
-2	225	236
-3	75	69
-4	431	-426
-5•	43	2
-6	258	243
-7•	43	-25
-8	172	172
-9	72	57
-10	272	-271
-11•	49	-23
-12	178	180
H = 7,K = 9		
1	160	148
0	125	114
-1	266	-258
-2	141	-143
-3	63	64
-4•	45	-18
-5	174	174
-6	183	169
-7	211	-216
-8	100	-107
-9	161	160
-10•	49	30
-11•	51	-24
-12•	52	64
-13	147	-154
H = 7,K =10		
3	127	116
2	87	-77
1	85	-89
0•	52	-25
-1•	51	-17
-2	80	89
-3	168	179
-4	106	-107
-5	196	-197
-6	120	120
-7•	46	16
-8	66	41
-9	125	130
-10	173	-151
-11	131	-148
H = 7,K =11		
0	195	190
-1•	54	-14
-2	119	-112
-3•	53	8
-4•	52	-23
-5•	52	40
-6	143	131
-7•	53	-41
-8	177	-164
H = 8,K = 0		
8	123	129
6•	51	-52
4	183	-181
2	371	346
0	319	-276
-2	152	-128
-4	1028	1064
-6	760	-735
-8	64	-84
-10	619	591
-12	498	-470
-14	78	56
-16	154	157
H = 8,K = 1		
5	209	183
4•	52	53
3	272	-213
2•	48	-34
1	252	-223
0	125	-117
-1	583	634
-2	376	362
-3	501	-498
-4•	40	-2
-5	350	-323
-6	375	-331
-7	685	727
-8•	40	81
-9	643	-651
-10	134	-162
-11•	44	93
-12	102	115
-13	236	236
-14	220	226
-15	271	-262
H = 8,K = 2		
3	170	-179
2	326	359
1	269	239
0	214	-189
-1•	46	-32
-2	279	-267
-3	259	-258
-4	529	530
-5	503	510
-6	452	-483
-7	384	-369
-8•	44	73
-9	316	-299
-10	450	442
-11	436	450
-12	252	-281
-13	114	-137
H = 8,K = 3		
4	189	175
3•	57	9
2•	55	-33
1•	53	-91
0	390	-391
-1	111	111
-2	473	470
-3	257	-254
-4	206	-169
-5•	46	-2
-6	349	-320
-7	384	370
-8	660	686
-9	282	-300
-10	259	-279
-11•	51	13
-12	166	-144
-13	94	128
-14	253	245
-15•	59	-63
-16	137	-158
H = 8,K = 4		
3	227	-235
2•	49	-5
1	334	360
0•	45	-20
-1	98	-115
-2•	43	-32
-3	486	-473
-4•	41	75
-5	595	624
-6•	41	13
-7	129	-141
-8•	41	-7
-9	211	-214
-10•	43	-82
-11	299	355
-12•	46	-56
-13	264	-328
-14•	50	31
-15•	52	-53
-16•	54	34
-17	160	138
H = 8,K = 5		
4	139	144
3•	52	26
2	84	40
1•	47	35
0	283	-270
-1•	44	-89
-2	403	382
-3	227	243
-4	223	-199
-5•	40	56
-6	288	-296
-7	182	-179
-8	504	543
-9•	42	104
-10	167	-191
-11•	45	-7
-12	146	-176
-13	107	-80
-14	193	203
-15	90	84
H = 8,K = 6		
3	102	-141
2	216	-218
1	208	225
0	147	135
-1•	50	4
-2	109	82
-3	239	-239
-4	278	-300
-5	255	275
-6	161	171
-7	198	-193
-8•	47	-41
-9	217	-204
-10	157	-176
-11	229	240
-12	168	195
H = 8,K = 7		
3	111	92
2•	48	6
1	160	144
0	100	-109
-1	216	-228
-2	163	161
-3	193	203
-4•	42	43
-5•	42	-30
-6	155	-159
-7	239	-253
-8•	41	51
-9	295	356
-10•	43	-24
-11•	44	39
-12•	46	7
-13	116	-144
H = 8,K = 8		
2	156	-145
1•	53	-65
0	115	118
-1•	50	2
-2•	48	28
-3•	47	2
-4	313	-311
-5•	46	13
-6	231	212
-7•	46	62
-8•	47	9
-9•	48	-20
-10	209	-175
-11•	51	-3
-12	182	174
H = 8,K = 9		
-1	197	-181
-2	111	-128
-3	153	151
-4•	48	70
-5	105	108
-6•	47	67
-7	125	-138
H = 8,K =10		
-5	187	-183
-6	68	91
-7	84	79
-8•	49	-5
-9	71	60
-10	89	-101
-11	106	-115
H = 9,K = 0		
4	160	-153
2	246	239
0	219	-190
-2•	41	-37
-4	448	426
-6	695	-657
-8•	39	44
-10	359	333
-12	415	-411
-14	79	85
-16	156	128
H = 9,K = 1		
5	129	119
4•	55	79
3	120	-90
2•	52	-16
1	111	-103
0•	48	-70
-1	460	451
-2	268	254
-3	339	-355
-4	194	-160
-5•	43	19
-6	253	-232
-7	470	425
-8	136	132
-9	343	-366
-10•	45	-39
-11•	46	89
-12	105	102
-13	243	202
-14•	50	81
-15	260	-234
H = 9,K = 2		
4•	60	-42
3	129	-163
2	215	178
1	240	225
0	201	-198
-1•	50	-56
-2	219	-207
-3	151	-154
-4	333	332
-6	256	-261
-7	352	-365
-8•	46	15
-9•	47	-85
-10	240	239
-11	375	366
-12	293	-228
-13	117	-124
H = 9,K = 3		
4	156	156
3•	62	20
2•	59	-65
1•	57	-98
0	222	-240
-1•	54	121
-2	388	412
-3•	51	-89
-4	194	-193
-5	111	-88
-6	156	-168
-7	209	203
-8	431	453
-9•	50	-79
-10	282	-320
-11•	53	-33
-12•	54	-81
-13	112	126
-14	164	145
-15	104	-77
H = 9,K = 4		
3	122	-135
2•	53	-17
1	227	242
0•	49	67
-1	151	-167
-2•	46	-96
-3	322	-298
-4	126	116
-5	499	527
-6	138	138
-7	158	-143
-8	176	-200
-9	100	-85
-10•	46	-25
-11	209	232
-12	83	75
-13	248	-281
H = 9,K = 5		
0	189	-157
-1	82	-79
-2	244	254
-3	211	245
-4	140	-148
-5	138	-150
-6	138	-156
-7	138	-145
-8	325	351
-9	172	174
-10	227	-273
-11	147	-174
-12	67	-54
-13•	49	-34
-14	176	177
-15	118	112
H = 9,K = 6		
2	134	-120
1	130	122
0	251	252
-1•	55	2
-2•	53	-20
-3	116	-144
-4	197	-225
-5	159	176
-6	250	276
-7	112	-144
-8	87	-91
-9•	51	-71
-10	90	-44
-11	169	195
-12	190	227
-13•	57	-76
-14	101	-140
H = 9,K = 7		
-1	239	-235
-2•	46	49
-3	214	193
-4•	44	16
-5	97	-117
-6	86	-104
-7	236	-189
-8•	43	52
-9	279	333
-10•	45	8
-11	103	-129
H = 9,K = 8		
0	124	144
-1•	54	72
-2•	52	-13
-3•	51	15
-4	196	-187
-5•	50	-42
-6	314	294
-7•	50	63
-8•	50	-52
-9•	51	-20
-10	89	-117
-11•	53	-45
-12	172	176
H = 9,K = 9		
-1	123	-133
-2	120	-109
-3	139	153
-4	163	127
-5•	51	-9
-6•	51	25
-7	134	-149
-8	114	-125
-9	90	123
-10	91	111
H = 9,K =10		
-5	188	-170
-6•	52	83
-7	164	151
-8•	52	-65
-9•	53	10
-10•	54	-62
-11	110	-139
H =10,K = 0		
2	174	185
0	201	-165
-2	110	114
-4	166	176
-6	275	-270
-8	201	188
-10	135	125
-12	299	-308
-14	226	241
H =10,K = 1		
-1	159	159
-2	155	153
-3	263	-276
-4	182	-184
-5•	46	42
-6•	46	-81
-7	103	126
-8	103	140
-9	328	-301
-10•	47	-63
-11	185	182
-12•	49	106
-13•	50	-8
-14•	52	61
-15	119	-150
-16	123	-129
H =10,K = 2		
1	182	177
0	125	-142
-1	94	-131
-2•	53	47
-3•	52	-39
-4•	51	121
-5	224	182
-6	111	-147
-7	292	-331
-8	111	135
-9•	50	106
-10•	51	114
-11	305	284
-12	204	-164
-13	242	-251
-14	97	123
H =10,K = 3		
4	117	93
3•	66	-8
2•	64	-107
1•	62	-46
0	134	-81
-1•	58	-16
-2	253	281
-3•	55	-40
-4	243	-266
-5•	54	85
-6	119	44
-7•	53	42
-8	336	319
-9•	53	-97
-10	383	-356
-11•	55	59
-12•	56	68
-13•	58	2
-14	133	154
-15•	61	-45
-16	142	-156
H =10,K = 4		
1	122	169
0•	53	48
-1	165	-182
-2•	50	11
-3•	49	-33
-4•	48	-51
-5	351	390
-6•	47	58
-7	257	-302
-8	81	-104
-9•	47	-10
-10•	48	-48
-11	85	118
-12	87	85
-13	229	-267
-14•	53	-2
-15	121	137
H =10,K = 5		
-2	198	187
-3	218	211
-4	185	-202
-5	105	-104
-6•	47	35
-7	147	-167
-8	219	224
-9	211	210
-10	253	-260
H =10,K = 6		
0	104	145
-1•	59	-47
-2•	57	-94
-3•	56	-78
-4	213	-211
-5	94	106
-6	295	302
-7	131	-152
-8	132	-148
-9•	54	62
-10	110	-108
-11	97	97
-12	151	175
H =10,K = 7		
-1	159	-147
-2•	49	59
-3	226	237
-4•	47	-6
-5	104	-112
-6•	46	-29
-7	80	-91
-8•	46	-6
-9	313	358
-10•	47	4
-11	152	-174
H =10,K = 8		
-4	172	-159
-5•	54	-64
-6	266	258
-7•	53	66
-8	168	-163
-9•	54	18
-10•	54	-88
-11•	55	-62
-12	159	185
H =10,K = 9		
-2	99	-93
-3	178	165
-4	124	102
-5•	55	-43
-6•	54	13
-7	94	-99
-8	94	-116
-9	94	145
-10	78	97
H =10,K =10		
-5	125	-127
-6•	55	74
-7	124	130
H =11,K = 0		
0	176	-178
-2	187	157
-4•	46	54
-6	496	-481
-8	364	366
-10•	45	-64
-12	247	-269
-14	243	265
H =11,K = 1		
-1	94	92
-2•	53	69
-3	283	-278
-4	160	-184
-5	274	292
-6•	49	-5
-7	85	93
-8	155	155
-9	312	-329
-10•	52	-68
-11	318	333
-12	89	91
-13•	52	-64
-14•	53	-33
-15	95	-111
-16	126	-119
-17	133	145
H =11,K = 2		
0	104	-157
-1	101	-124
-2	99	87
-3•	56	5
-4•	55	59
-5	170	151
-6	213	-233
-7	270	-262
-8	254	273
-9	192	176
-10•	54	-75
-11	121	148
-12	123	-173
-13	239	-235
-14	225	234
H =11,K = 3		
-2	193	199
-3•	60	-40
-4	226	-240
-5	99	102
-6	127	94
-7•	57	-59
-8	179	189
-9•	57	-82
-10	362	-370
-11	100	162
-12	132	143
-13•	60	-60
-14•	62	69
-15•	63	-57
-16	145	-149
H =11,K = 4		
-1	191	-186
-2•	54	13
-3•	53	50
-4•	52	-34
-5	255	247
-6•	51	33
-7	297	-318
-8•	50	-3
-9	87	102
-10	88	-65
-11	89	129
-12	117	89
-13	206	-224
-14•	54	11
-15	148	171
H =11,K = 5		
-2	120	147
-3•	52	105
-4	199	-195
-5	88	-103
-6•	50	88
-7	111	-95
-8	141	141
-9	122	142
-10	298	-295
-11	74	-88
-12	104	132
H =11,K = 6		
-6	223	216
-7	128	-164
-8	99	-103
-9	99	92
-10•	58	-40
-11	101	110
-12	133	164
H =11,K = 7		
-3	146	149
-4•	51	-78
-5	87	-107
-6•	50	2
-7•	49	-49
-8•	49	64
-9	191	192
-10•	50	-40
-11	159	-188
H =11,K = 8		
-6	180	199
-7•	57	15
-8	98	-107
H =11,K =10		
-7	117	102
H =12,K = 0		
-2	179	198
-4	70	32
-6	260	-256
-8	365	361
-10	68	-57
-12	207	-207
-14	253	288
H =12,K = 1		
-3	303	-256
-4	122	-134
-5	240	237
-6	52	73
-7•	52	-46
-8	117	112
-9	286	-270
H =12,K = 2		
-6	198	-203
-7	196	-216
-8	178	222
-9	159	167
-10•	57	-16
-11•	57	74
-12	129	-163
-13	186	-203
-14	134	160
H =12,K = 3		
-2	131	137
-3	111	-114
-4	242	-228
-5	107	101
-6	106	114
-7•	61	77
-8	174	172
-9	135	-115
-10	284	-275
-11	136	97
H =12,K = 4		
1•	61	77
-1	156	-136
-2•	58	-1
-3•	56	79
-4•	55	19
-5	173	193
-6•	54	-73
-7	268	-301
-8•	53	-5
-9	92	94
-10•	54	47
-11•	54	74
-12•	55	-11
-13	175	-181
H =12,K = 5		
-4	155	-164
-5•	54	-66
-6•	54	73
-7•	53	20
-8	92	116
-9•	53	57
-10	206	-210
-11•	54	-54
-12	121	157
H =12,K = 6		
-7	136	-138
-8	105	-116
-9	105	90
H =12,K = 7		
-8•	74	59
-9	117	131
-10•	74	-61
-11	105	-140
H =13,K = 0		
-6	199	-197
-8	160	149
-10•	51	-35
-12	145	-133
-14	166	188
H =13,K = 3		
-10	143	-187
-11•	64	74
-12	145	152
H =13,K = 4		
-7	180	-199
H =13,K = 5		
-10	125	-131
H =14,K = 0		
-6	94	-56
-8	169	191
-10	92	-110
-12•	54	8
-14	122	116

viously equivalent in the D_{2h} symmetry, are now markedly different [C(1)–C(2) = 1·313 Å, C(4)–C(5) = 1·421 Å, C(1)–C(2) in naphthalene = 1·364 Å]. Although the standard deviations of these bond lengths are fairly large (0·012–0·018 Å) this is not unexpected in view of the small percentage of scattering from carbon atoms in the structure, especially at high Bragg angles. However, the distortion of the naphthalene molecule as a whole is very significant and leads directly to an interpretation of the intermolecular bonding in the structure.

Discussion of structure

The bonding in Group V trihalides is believed to involve sp^3-hybridization, though the interbond angles at the central atom are much less than the tetrahedral angle (Gillespie & Nyholm, 1957), because of lone-pair bond-pair electron repulsions. The crystal structure analysis of antimony trichloride (Lindqvist & Niggli, 1956) confirms this. The structure shows discrete $SbCl_3$ molecules located on mirror planes, with a mean Sb–Cl bond length of $2·36 \pm 0·03$ Å (two at 2·37 and one at 2·35 Å) and a mean interbond angle of 95·2°. The lone electron pair presumably occupies the fourth vertex of a distorted tetrahedron.

The present structure shows a similar $SbCl_3$ molecule again with approximate mirror symmetry [through Sb, Cl(3) and the bisector of angle Cl(1)–Sb–Cl(2)], though there are significant differences from Lindqvist & Niggli's result. The two mirror-related Sb–Cl bonds which lie in a plane nearly parallel to the naphthalene molecule, are significantly *shorter* than the Sb–Cl(3) bond.

As already mentioned above (see *Description of the structure*), the naphthalene molecule is considerably distorted from the D_{2h} symmetry found in free naphthalene. The two bonds closest to antimony have total bond orders of about 2·25 and 1·90 (Pauling, 1960), and the bond furthest from antimony has a bond order of only 1·25.

The longest bond in the $SbCl_3$ molecule, Cl(3)–Sb, points directly at this concentration of higher elec-

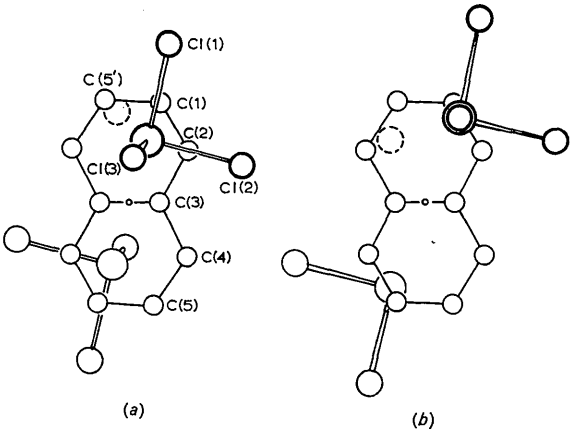

Fig. 2. (*a*) The projection of the antimony trichloride molecule onto the napthalene plane. (*b*) The structure projected onto the plane normal to the Cl(3)–Sb bond. In both drawings the position of Cl(3) related by the translation $x = 1$ is marked by the broken circle.

Table 4(*a*). *Interatomic distances and their standard deviations*

	Length l	$\sigma(l)$
Sb—Cl(1)	2·348 Å	0·002 Å
Sb—Cl(2)	2·347	0·002
Sb—Cl(3)	2·367	0·002
Sb—C(1)	3·364	0·010
Sb—C(2)	3·394	0·008
Sb—C(3)	3·549	0·008
Sb—C(3')	3·736	0·008
Sb—C(4')	2·719	0·008
Sb—C(5')	3·505	0·011
Cl(1)–Cl(2'')	3·506	0·003
C(1)–C(2)	1·313 Å	0·016 Å
C(2)–C(3)	1·420	0·012
C(3)–C(3')	1·425	0·014
C(3)–C(4)	1·440	0·012
C(4)–C(5)	1·421	0·017
C(1)–C(5')	1·343	0·018
Sb—Cl(1'')	3·832	0·002
Sb—Cl(2'')	3·581	0·002
Sb—Cl(3''')	3·656	0·002

Table 4(*b*). *Bond angles and their standard deviations*

	θ	$\sigma(\theta)$
Cl(1)–Sb—Cl(2)	$91·59_3$°	$0·08_4$°
Cl(2)–Sb—Cl(3)	$94·41_7$	$0·09_0$
Cl(1)–Sb—Cl(3)	$94·70_8$	$0·09_4$
C(1)–C(2)–C(3)	120·98	0·86
C(2)–C(3)–C(3')	119·27	0·98
C(2)–C(3)–C(4)	123·06°	0·79°
C(4)–C(3)–C(3')	117·64	0·97
C(3)–C(4)–C(5)	118·22	0·88
C(2)–C(1)–C(5')	122·84	0·99
C(4)–C(5)–C(1')	120·96	0·90

Code for symmetry related atoms.

Superscript	Position
none	$x, \quad y, \quad z$
'	$1-x, \quad -y, \quad 1-z$
''	$-x, \quad -\frac{1}{2}+y, \quad \frac{1}{2}-z$
'''	$-x, \quad -y, \quad 1-z$

tron density on the naphthalene molecule (as implied by the shorter bond lengths in this region), (see Figs. 2(*b*) and 4). The donation of electrons from the π-system to the antimony results in an sp^3d hybridization of the antimony electrons. The equatorial plane is then made up of Cl(1), Cl(2) and the antimony lone electron pair. Cl(3) and the electrons donated from the π-system fill the axial positions of a distorted trigonal bipyramid. The antimony lone electron pair points over the centre of the aromatic ring, and is ideally situated for back-bonding to the π-system.

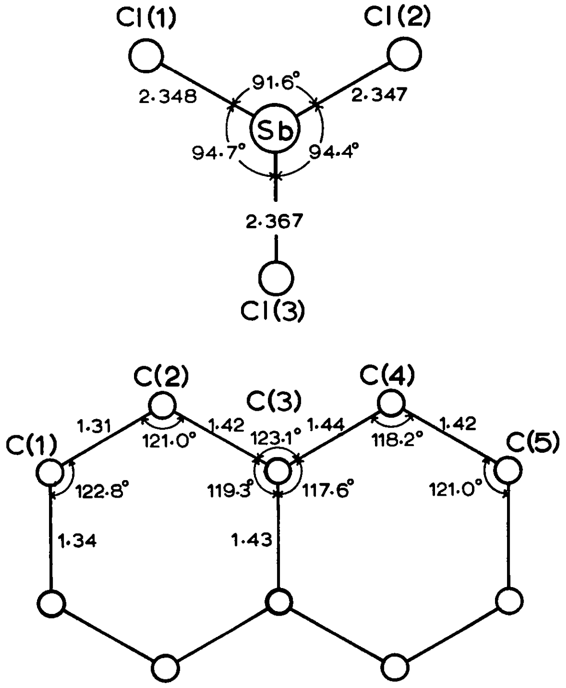

Fig. 3. Bond lengths and bond angles.

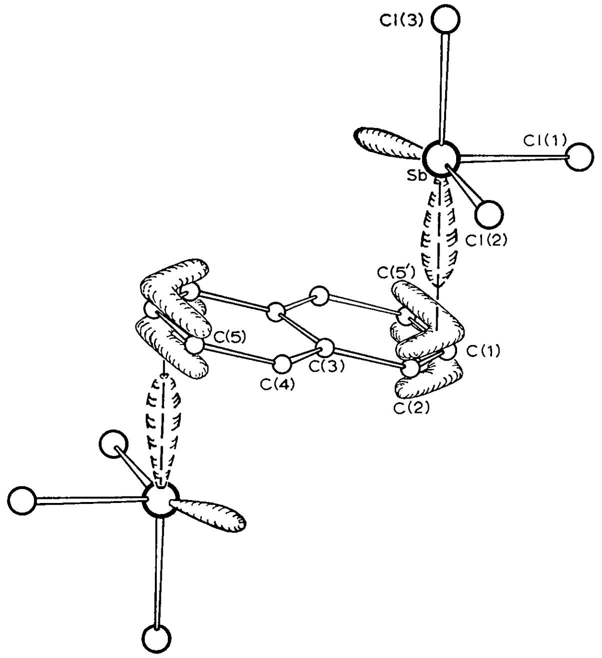

Fig. 4. Pictorial view of the structure illustrating the proposed bonding. The lobes on the napthalene molecule represent the areas of *highest* π-electron density. The broken lobe from antimony to naphthalene indicates the partially filled axial sp^3d orbital.

The structure can be described as a pseudo π-allene complex (Green & Nagy, 1964) in which C(2), C(1) and C(5′) behave as a π-allene-like complexing agent.

The best least-squares plane through all ten carbon atoms (plane 1), and the deviations of atoms from this plane, are given in Table 5. More significant, however, is the plane through atoms C(3), C(4), C(5), C(3′), C(4′), C(5′) (plane 2). These atoms are very nearly coplanar, (maximum deviation 0·003 Å), but C(1) and C(2) are significantly displaced from this plane in the direction *away* from the nearly antimony atom. The plane through C(1), C(2) and their respective adjacent carbon atoms C(5′) and C(3), (plane 3) is tilted 2·3° away from plane 2 in such a way as to increase the angle of tilt of this section to the vector Cl(3)–Sb, and hence presumably to the direction of the antimony d_{z^2} orbital. The angle of tilt of plane 3 to this direction (103·6°) is very similar to that found in a number of π-allyl-transition metal complexes (Churchill & Mason, 1967).

It should be pointed out that the proposed structure involves a very long bond of 3·2 Å from the π-system to antimony. This is much longer than is found in π-bond transition metal complexes, *e.g.* in the structure of naphthalenechromium tricarbonyl (Kunz & Nowacki, 1967) the distance from chromium to the plane of the naphthalene ring is 1·75 Å. This seems to indicate that in the present structure, the antimony–naphthalene bond is not a very strong interaction, though it is strong enough to produce considerable π-electron localization in naphthalene. The bond proposed is best envisaged as an only partial filling of the axial sp^3d antimony orbital from the naphthalene π-system.

Pauling (1960) gives 2·2 Å and 1·80 Å as the van der Waals radii of antimony and chlorine, and 1·70 Å as the half-thickness of the aromatic molecule. In the present structure there are three non-bonded antimony-chlorine contacts (3·58, 3·66, 3·83 Å) at less than the sum of the radii. The closest non-bonded chlorine-aromatic contact found here is 3·54 Å and the closest chlorine–chlorine distance is 3·51 Å. These latter distances are approximately consistent with Pauling's values for the van der Waals radius for chlorine, and the half-thickness of the aromatic ring. As there appears to be no simple explanation of the short antimony–chlorine contacts in terms of $SbCl_3$-dimer or -polymer formation, we must conclude that the van der Waals radius for antimony is not 2·2 but about 1·8 Å in this particular coordination. Based on this lower value, the expected non-bonded antimony–naphthalene distance would then be about 3·5 Å, and the fact that we find 3·2 Å in this structure is indicative of the presence of bonding.

It is worth noting that from a study of the Raman spectra of this complex and of the constituents in the crystalline state, Raskin (1958) claimed that the π-electron system of the naphthalene ring was involved in bonding to the antimony trichloride molecule. The spectrum of the complex differs from the spectra of the components by a 'significant redistribution of the intensities and a shift in the frequencies'. New bands were observed in some parts of the spectrum, and some previously single bands were found split. The decrease in C–C frequencies and the appearance of new lines in this region was attributed to the fact that these complexes are formed by means of the π-electrons. The new lines observed in the regions of the valence vibrations of $SbCl_3$ were attributed to the formation of bonds between the $SbCl_3$ molecule and the complexing agent.

These observations are in agreement with the structure proposed. The changes in frequency of the Sb–Cl bonds can be explained by the change in hybridization or coordination, and the changes in frequencies of C–C bonds are accounted for by the changes in π-bond order in the naphthalene bonds.

Comparison with related structures

The present structure is one member of a series of related antimony trihalide structures, (see Fig. 5). In free antimony trichloride, the three chlorine atoms and the antimony lone electron pair have a distorted tetrahedral distribution around antimony, [Fig. 5(*a*)]. In the presence of one electron donor source, (present structure), the distribution is now a distorted trigonal bipyramid [Fig. 5(*b*)]. The bond opposite the newly filled orbital is lengthened by 0·02 Å relative to the other antimony chlorine bonds. The structure of the 2:1 adduct between antimony triiodide and 1:4 dithiane (Bjorvatten, 1966), illustrates the situation when two electron donors approach an antimony trihalide molecule [Fig. 5(*c*)]. Here the distribution is distorted octahedral, and both antimony–iodine bonds opposite the newly filled orbitals are again longer than the third antimony iodine bond by about 0·02 Å. Finally, the $SbBr_6^{3-}$ ion in the structure of ammonium hexabromoantimonate (Lawton & Jacobson, 1966) affords an example of the extreme case of electron donation to an antimony trihalide system. The structure is a regular

Table 5. *Planes of best fit* (*least squares*)

(*a*) Coefficients.

Each plane is represented by $AX+BY+CZ=D$ where X, Y, Z are orthogonal coordinates obtained from the fractional coordinates x, y, z by the transformations:

$$X=ax+cz\cos\beta,\quad Y=by,\quad Z=cz\sin\beta\,.$$

Plane	Description	A	B	C	D
1	Whole naphthalene molecule	0·9920	0·0201	0·1246	1·9597
2	Part of naphthalene molecule	0·9922	0·0415	0·1179	1·9258
3	Rest of naphthalene molecule	0·9920	0·0022	0·1265	1·9587
4	Antimony and two chlorines	0·9962	0·0840	0·0246	−1·5612

(*b*) Deviations from planes (Å)

Atoms in parentheses were *not* included in the calculation of the plane.

Plane	1	2	3	4
Sb	(3·223)	(3·201)	(3·220)	0·0
Cl(1)	(3·500)	(3·441)	(3·515)	0·0
Cl(2)	(3·264)	(3·200)	(3·294)	0·0
Cl(3)				(2·352)
C(1)	0·001	(−0·039)	0·006	
C(2)	−0·016	(−0·057)	−0·006	
C(3)	0·011	−0·003	0·003	
C(4)	0·016	0·002		
C(5)	−0·015	−0·001		
C(5′)	0·015	0·001	−0·003	
Cl(3″)	(−3·546)	(−3·562)		

In the case planes 1 and 2, the carbon atoms related to the above by the centre of symmetry were included in the calculation. Cl(3″) is related to Cl(3) by the translation $x=1$.

(*c*) Angles between normals to planes (degrees)

Plane	1	2	3	4
1	0·0	1·29	1·03	6·81
2		0·0	2·30	5·88
3			0·0	7·49

(*d*) Angles between Cl(3)–Sb vector and normals to planes (degrees)

Plane	1	2	3	4
Angle	12·76	11·58	13·62	6·55

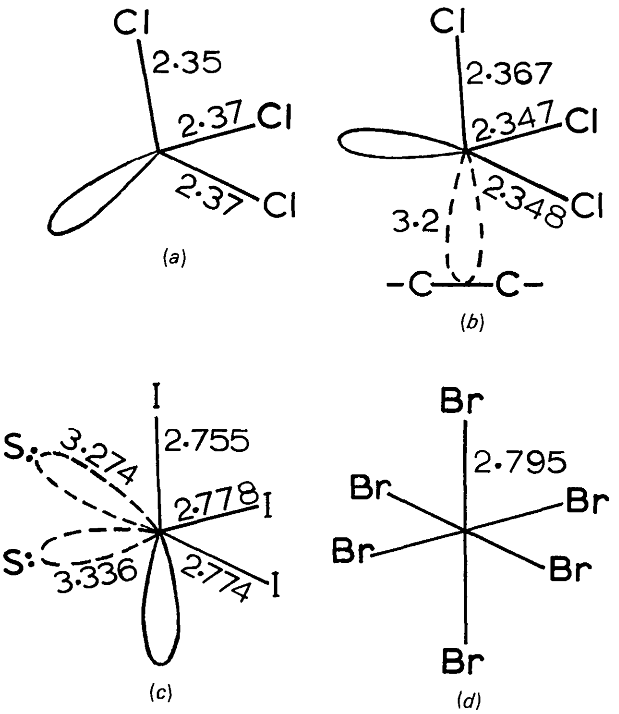

Fig. 5. Spatial distribution of bonds in some antimony trihalide complexes or ions. All bond lengths quoted are in Ångstrom units. (*a*) Free $SbCl_3$ (distorted tetrahedron). (*b*) $2SbCl_3.C_{10}H_8$, present structure (distorted trigonal bipyramid). (*c*) $2SbI_3.C_4H_8S_2$ (distorted octahedron). (*d*) $[SbBr_6]^{3-}$ (regular octahedron).

octahedron, and the presence of a triple negative charge and a lone electron pair in the molecule, result in a lengthening of the antimony–bromine bond to 2·795 Å from the mean value of 2·50 Å found in free $SbBr_3$ (Cushen & Hulme, 1962; 1963).

Electron donation into one of the hybrid orbitals of antimony has the effect of lengthening, and hence presumably weakening, the antimony–halogen bond opposite it. This can be compared with the well-known *trans* effect found in transition metal complexes.

We are indebted to Dr H. C. Freeman of the School of Chemistry, University of Sydney, Australia, in whose laboratory the data for this structure were recollected, and to Dr R. J. O'Brien of the Department of Chemistry, University of Toronto for helpful discussions.

References

Bjorvatten, T. (1966). *Acta Chem. Scand.* **20**, 1863.
Buerger, M. J. (1942). *X-ray Crystallography.* Chapter 20. New York: John Wiley.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962*a*). Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962*b*). Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee.
Churchill, M. R. & Mason, R. (1967). *Adv. Organometal. Chem.* **5**, 111.
Cruickshank, D. W. J. (1957). *Acta Cryst.* **10**, 504.
Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 747, 754.
Cushen, D. W., Hulme, R. & Szymański, J. T. (1968). *J. Chem. Soc.* In press.
Cushen, D. W. & Hulme, R. (1962). *J. Chem. Soc.* p. 2218.
Cushen, D. W. & Hulme, R. (1963). *J. Chem. Soc.* p. 4162.
Gillespie, R. J. & Nyholm, R. S. (1957). *Quarterly Rev.* **11**, 368.
Green, M. L. H. & Nagy, P. L. I. (1964). *Adv. Organometal. Chem.* **2**, 325.
Hamilton, W. C. (1955). *Acta Cryst.* **8**, 185.
Hamilton, W. C., Rollett, J. S. & Sparks, R. A. (1965). *Acta Cryst.* **18**, 129.
Hulme, R. (1966). *Acta Cryst.* **21**, A143.
International Tables for X-ray Crystallography (1962*a*). Vol. III. Ref. 12, p. 208. Birmingham: Kynoch Press.
International Tables for X-ray Crystallography (1962*b*). Vol. III. Ref. 69, p. 209. Birmingham: Kynoch Press.
International Tables for X-ray Crystallography (1962*c*). Vol. III. p. 211. Birmingham: Kynoch Press.
Kunz, V. & Nowacki, W. (1967). *Helvetica Chim. Acta.* **50**, 1052.
Lawton, S. L. & Jacobson, R. A. (1966). *Inorg. Chem.* **5**, 743.
Lindqvist, I. & Niggli, A. (1956). *J. Inorg. Nucl. Chem.* **2**, 345.
Menshutkin, B. N. (1911). *Chem. Zentr.* II, 751.
Menshutkin, B. N. (1912*a*). *Chem. Zentr.* I, 408, 807.
Menshutkin, N. B. (1912*b*). *Chem. Zentr.* II, 1436.
Olah, G. A. (1963). Editor, *Friedel-Crafts and Related Reactions.* Vol. I, p. 270. New York: Interscience.
Pauling, L. (1960). *The Nature of the Chemical Bond.* 3rd ed. Ithaca: Cornell Univ. Press.
Raskin, Sh. Sh. (1958). *Doklady Akad. Nauk SSSR*, **123**, 645.
Smith, W. & Davies, G. W. (1882). *J. Chem. Soc.* **41**, 411.
Szymański, J. T. (1963). Ph. D. Thesis, University of London.