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The crystal structure of vrbaite $Hg_3Tl_4As_8Sb_2S_{20}$*

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Auszug

Die Kristallstruktur von Vrbait wurde bestimmt. Vier Formeleinheiten $Hg_3Tl_4As_8Sb_2S_{20}$ sind in der rhombischen Zelle, $a = 13,399$, $b = 23,389$, $c = 11,287$ Å, Raumgruppe $C2ca—C_{2v}^{17}$, enthalten. Die Struktur wurde mittels einer Art Superpositionsmethode auf Grund einer dreidimensionalen Pattersonfunktion gefunden; $R = 4,8\,^0/_0$.

Die Vrbaitstruktur ist durch unendliche As_2SbS_5-Ketten parallel c und durch $Hg_3As_4S_{10}$-Schichten normal zu b charakterisiert. Die ersteren umgeben („sandwich") die Tl-Atome und bilden Schichtbereiche („slabs") normal zur b-Axe. Diese Schichtbereiche und die $Hg_3As_4S_{10}$-Schichten, welche die anderen Tl-Atome umgeben, wechseln in der b-Richtung ab. Die gefundene Struktur erklärt gut die gute Spaltbarkeit parallel (010).

Vrbait ist unseres Wissens die erste Struktur mit gemischten (As,Sb)-Ketten.

Abstract

The crystal structure of vrbaite has been determined. Four chemical units of $Hg_3Tl_4As_8Sb_2S_{20}$ are contained in the orthorhombic unit cell: $a = 13.399$, $b = 23.389$, $c = 11.287$ Å, symmetry $C2ca—C_{2v}^{17}$. The structure was solved through a kind of superposition method using the three-dimensional Patterson function. The final discrepancy index R is $4.8\,^0/_0$.

The structure of vrbaite is characterized by infinite As_2SbS_5 chains parallel to c and by $Hg_3As_4S_{10}$ sheets perpendicular to b. The former sandwich the Tl atoms and make slabs perpendicular to the b axis. These slabs and the $Hg_3As_4S_{10}$ sheets, sandwiching the other Tl atoms, are arranged alternately along the b direction. The good cleavage parallel to (010) can be well explained by the structure. Vrbaite is the first structure with mixed (As,Sb) chains.

Introduction

Vrbaite is a very rare sulfosalt, found by JEŽEK (1912) in a specimen from Allchar, Macedonia. JEŽEK made morphological studies and

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[*] Communication no. 208a.-Part 58 on sulfides and sulfosalts. Part of this work was presented at the 7th IMA meeting (31st August 1970, Kyoto, Japan) and at the Annual Meeting of the Swiss Society for Crystallography, 17th October 1970, Basel.

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described its point symmetry as $2/m\ 2/m\ 2/m$. Křehlík (1912) made a chemical analysis of this material and showed its formula to be $TlAs_2SbS_5$. The cell dimensions of vrbaite were determined by Frondel (1941). He proposed *Cmca* as the probable space group, taking account of x-ray and morphological data. Recently Caye, Picot, Pierrot and Permingeat (1967) reexamined the chemical composition of vrbaite with the aid of electron-probe microanalysis, and obtained a new formula, $Hg_3Tl_4As_8Sb_2S_{20}$. This formula was confirmed by Nowacki (1968).

The present investigation was undertaken to elucidate the coordinations of the sulfur atoms around the thallium atoms which show a quite irregular feature in the structures of sulfosalts, and to examine whether the antimony atoms and the arsenic atoms are in an ordered state or not.

Experimental

A specimen from Allchar, Macedonia (Naturhistorisches Museum, Wien), half of which was being used for chemical analysis (Nowacki, 1968), was used for the present studies. A spherical crystal with radius 0.106 mm was prepared by Bond's (1951) method for the determination of the cell dimensions and intensities. The lattice constants were obtained from back-reflection Weissenberg photographs calibrated by powder patterns of silicon. A least-squares best fit of the lattice parameters was calculated with the aid of an IBM-1620 program written by N. D. Jones (unpublished). The results agree well with the values obtained by Frondel (1941).

Present determination	Frondel[1]
$a = 13.399 \pm 0.001$ Å	13.38 ± 0.05 Å
$b = 23.389 \pm 0.001$	23.37 ± 0.05
$c = 11.287 \pm 0.001$	11.25 ± 0.05

The number of formula units, four, was calculated from the cell dimensions obtained and the measured specific gravity, 5.30 (Palache, Berman and Frondel, 1944).

The diffraction symbol for vrbaite is $mmmC{*}ca$, which permits *Cmca* and *C2ca* as possible space groups. Since a piezoelectric test showed an acentric feature, $C2ca$—C_{2v}^{17} was selected as the correct space group.

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[1] Frondel's values are converted to Ångstrom from kX units.

Three-dimensional intensity data (1772 independent reflections) were collected by an automated Weissenberg counter-diffractometer (Supper-Pace) using Cu$K\alpha$ radiation. One hundred and twenty-four of these reflections [with $I < 2.33\,\sigma\,(I)$] were assigned to unobserved reflections.

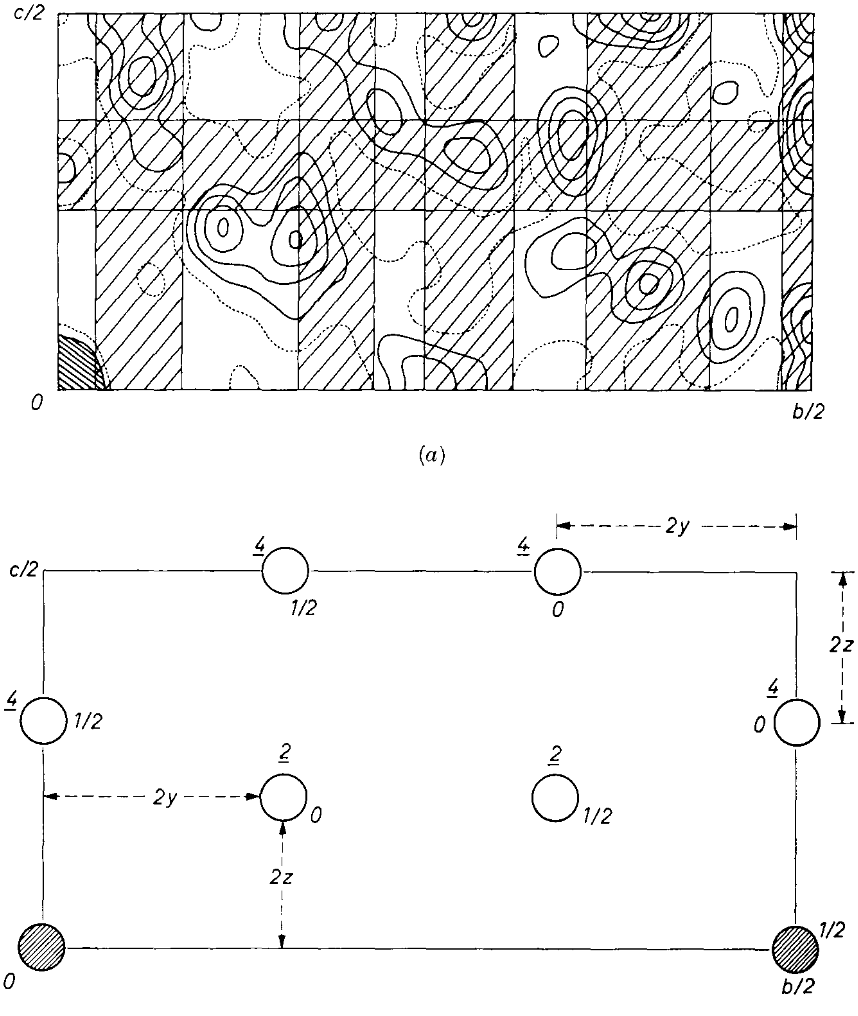

Fig. 1. (*a*) Patterson section $P(0,v,w)$. Rotation peaks should not exist in the shaded area. Contours are equal but in arbitrary intervals. (*b*) The vector set for equipoints 8*b*. The underlined values indicate weights of points

Determination of the structure and refinement

The Patterson function $P(u,v,w)$, evaluated with the three-dimensional data, showed that all peaks are distributed on the sections $u = n/8$ ($n = 0$, 1 and 2). All heavy peaks which correspond to the vectors between heavy atoms[2] are on the sections $u = 0$ and $1/4$. These features of the diagram suggest that all atoms are arranged nearly on planes at intervals of $1/8$ along the a direction, and all heavy atoms on every other plane. The only equipoints of the space group $C2ca$ are $4a$ (on twofold axes) and $8b$ (points in the general position)[3].

Since there are twelve mercury atoms in the cell and since this number is not divisible by eight, at least four mercury atoms should occupy the special positions. A possibility of statistical distribution of mercury atoms was omitted, because no anomaly of background intensities was observed on the films. As the space group $C2ca$ has no symmetry element to fix the x coordinate of points, the origin of the cell can be set at one mercury atom of $4a$ and the positions of at least three heavy atoms (one Hg and two Tl atoms) must be found.

The vector set of equipoint $8b$ is illustrated in Fig. 1*b*. These relations of equipoints and the heights of peaks which are expected for the vectors between heavy atoms, limit the probable positions of rotation peaks in the Patterson diagram $P(0,v,w)$, Fig. 1*a*. It was, however, impossible to find uniquely the rotation peaks of heavy atoms and another attempt was made to obtain the heavy-atom positions, because almost all peaks of the Patterson maps have broadened shapes and overlap each other.

If one component atom of a structure occupies the origin of the cell, the Patterson diagram contains the image of the true structure and the origin of the diagram coincides with that of the cell. If this crystal has axial symmetry, the image of the structure, being concentrated in one section, will also be obtained from the Harker section (the implication diagram). Therefore when we superpose the implication diagram on each section of the Patterson diagram in such a way that the origin of the former coincides with one of the symmetry axes in the cell, and when we note the peaks common in both diagrams, the image of the true structure should be found in the resultant map. In the present case, the Patterson section $P(0,v,w)$, which was drawn on a scale of one

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[2] The word "heavy atoms" will be used for mercury and thallium atoms hereafter, because the difference of their atomic scattering factors is quite small.

[3] $(0,0,0;\ \tfrac{1}{2},\tfrac{1}{2},0) + 8b\colon\quad x, y, z;\ x,\bar{y},\bar{z};\quad x,\tfrac{1}{2}+y,\ \tfrac{1}{2}-z;\quad x,\tfrac{1}{2}-y,\ \tfrac{1}{2}+z.$
$4a\colon\quad x,0,0;\quad x,\tfrac{1}{2},\tfrac{1}{2}.$

to two, was superposed on $P(0,v,w)$ and $P(\frac{1}{4},v,w)$. The result, however, showed still too many candidates for the heavy atom positions.

In order to eliminate some of the candidates, the relations of the cross vectors between the origin and the points in the general positions were derived. The end points of vectors show special relations as indicated in Fig. 2: these points are related to each other by three twofold axes. One of them designated as I is a component of the symmetry elements in the Patterson space group *Cmmm*, while the others, II and III, being equivalent, are additional ones. When two sets of Patterson diagrams are superposed according to the additional operations, the cross vector peaks between the atom of the origin and the atoms of the general positions can be obtained. This result also includes the image of the actual structure.

The above procedure eliminated most of the peaks in the Patterson section. The implication diagram was also superposed on the resultant maps, and five peaks common in both diagrams were adopted as candidates (Figs. 3*a* and 3*b*). In order to eliminate the number of candidates and to obtain the relative positions of each of the heavy atoms, five sets of minimum functions were evaluated using the origin atom, each of the candidates and their symmetrically equivalent positions. Four minimum-function diagrams drawn with four points I, II, III and IV (Figs. 3*a* and 3*b*) were compatible with each other, and these peaks were considered to correspond to one mercury, two thallium and one antimony atoms. Position I was regarded as the antimony atom, because the peak height in the minimum-function diagram is lower. The relative positions of these peaks, having been impossible to derive by the operation in Fig. 2, are found from minimum-function diagrams. The structure factors were calculated with the coordinates obtained for the reflections which are in the range of $\sin\theta \leq 0.7$. The discrepancy index R was about $43\,^0/_0$. The atomic scattering factor of mercury was used for both mercury and thallium atoms at this stage. The remaining atoms were found by successive three-dimensional Fourier and difference Fourier syntheses. In the course of this procedure, the antimony atom and one of three heavy atoms (denoted II in Fig. 3*a*) revealed an anomaly in the difference map; that is, a deep depression was found at the position of the latter and an elevation at the position of the former. Besides, the coordination of sulfur atoms around them was quite strange: atom I, which was regarded as Sb, has two nearest sulfur atoms and the coordination of atom II was a flat trigonal pyramid. Therefore the Sb atom (I) and atom II were

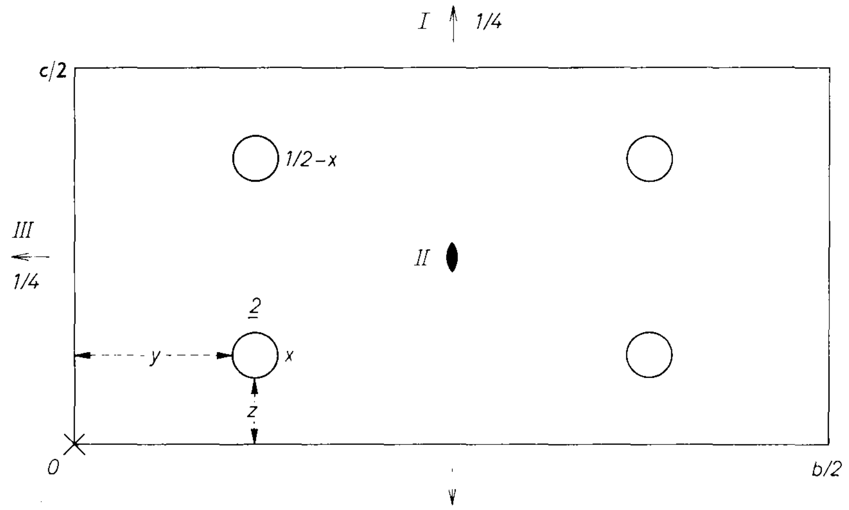

Fig. 2. The relations of cross vectors between the origin and the points in the general positions. The underlined values indicate weights of points. I, II and III are directions of the three twofold axes

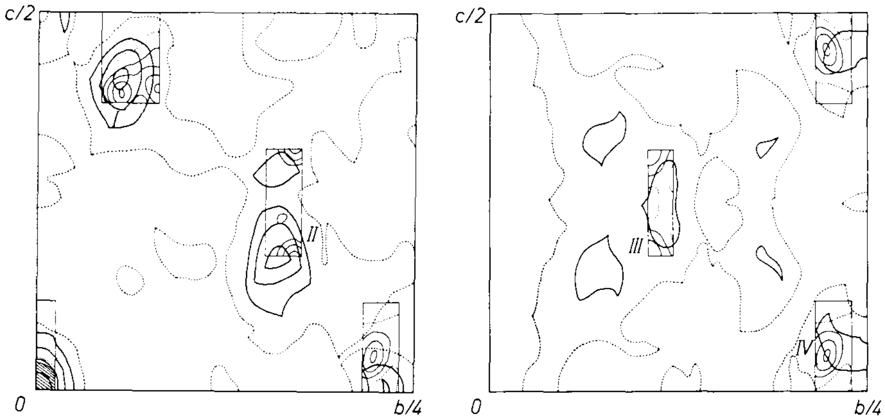

Fig. 3. Candidates for heavy atoms. The same diagram as in Fig. 1*a* was used as the implication diagram. All candidates should be in the area within the rectangular sections. (*a*) $x = 0$. (*b*) $x = 0.24 \approx \frac{1}{4}$. Independent candidates in this section are only III and IV

interchanged in position. Four arsenic and ten sulfur atoms were found in the further process. The *R* index was about 22% at this stage and no anomaly was found in the three-dimensional difference synthesis. The mercury atom was distinguished from thallium atoms by the difference in the coordinations around them.

Table 1. *Comparison of observed and calculated structure factors for vrbaite*

h	k	l	$\|F_o\|$	$\|F_c\|$
0	2	0	28*	79
	4		338	289
	6		259	267
	8		163	164
	10		160	155
	12		259	243
	14		271	283
	16		244	269
	18		1152	1229
	20		663	669
	22		93	54
	24		115	97
	26		355	352
	28		441	459
0	2	1	116	32
	4		378	376
	6		450	489
	8		831	918
	10		587	637
	12		705	762
	14		174	191
	16		285	298
	18		171	168
	20		32*	8
	22		131	139
	24		105	110
	26		257	256
	28		14*	10
0	0	2	344	389
	2		196	193
	4		928	1046
	6		234	271
	8		717	781
	10		114*	82
	12		152	171
	14		453	475
	16		49*	27
	18		93	50
	20		175	181
	22		500	514
	24		69	9
	26		255	255
	28		150	149
0	2	3	366	378
	4		1360	1640
	6		178	172
	8		621	678
	10		298	322
	12		87*	85
	14		822	881
	16		489	495
	18		20*	9
	20		235	238
	22		253	251
	24		450	461
	26		68	69
	28		27*	8
0	0	4	242	255
	2		24*	59
	4		687	766
	6		353	388
	8		823	919
	10		327	356
	12		672	713
	14		37*	11
	16		214	227
	18		173	166
	20		238	253
	22		89	80
	24		86	92
	26		226	240
0	2	5	109	119
	4		276	302
	6		32*	68
	8		521	577
	10		540	583
	12		96	88
	14		306	330
	16		86	98
	18		170	170
	20		390	398
	22		7*	20
	24		49	45
	26		138	142
0	0	6	594	633
	2		182	196
	4		25*	7
	6		482	524
	8		682	780
	10		537	595
	12		814	899
	14		61*	80
	16		143	154
	18		126	118
	20		92	99
	22		106	107
	24		213	222
0	2	7	84	70
	4		663	743
	6		105	76
0	8	7	123	120
	10		118	134
	12		80	88
	14		225	226
	16		416	441
	18		75	73
	20		122	129
	22		18*	4
	24		314	330
0	0	8	297	324
	2		268	285
	4		194	214
	6		347	371
	8		112.	118
	10		264	297
	12		52*	43
	14		55*	25
	16		31*	12
	18		290	298
	20		194	198
	22		45	41
0	2	9	49*	25
	4		361	389
	6		264	287
	8		292	318
	10		180	191
	12		426	451
	14		67	66
	16		33*	37
	18		123	123
	20		53	54
0	0	10	667	699
	2		249	254
	4		43*	27
	6		200	203
	8		186	193
	10		61	74
	12		65	67
	14		56	52
	16		96	99
	18		194	201
0	2	11	24*	1
	4		206	213
	6		58	54
	8		324	334
	10		60	53
	12		336	352
	14		387	389
	16		141	142
0	0	12	166	162
	2		243	239
	4		297	297
	6		61	52
	8		36	9
	10		33	13
	12		41	15
	14		111	111
0	2	13	61	59
	4		203	216
	6		63	65
	8		107	111
	10		204	209
1	1	1	98*	136
	3		116*	104
	5		388	404
	7		835	909
	9		701	738
	11		526	543
	13		526	552
	15		253	257
	17		139	121
	19		260	261
	21		182	188
	23		260	254
	25		255	254
	27		141	143
1	1	2	155	127
	3		519	546
	5		600	638
	7		166	185
	9		41*	58
	11		190	205
	13		396	402
	15		305	297
	17		122	131
	19		176	171
	21		281	283
	23		156	163
	25		190	196
	27		166	171
1	1	3	494	511
	3		460	499
	5		297	306
	7		145	160
	9		673	726
	11		325	348
	13		157	169
	15		187	200
	17		272	271
	19		180	170
1	21	3	210	216
	23		275	275
	25		32*	44
	27		214	220
1	1	4	865	943
	3		404	418
	5		283	294
	7		656	711
	9		295	310
	11		538	557
	13		278	281
	15		99	81
	17		407	400
	19		387	387
	21		305	300
	23		72	62
	25		255	262
	27		205	219
1	1	5	723	764
	3		241	244
	5		247	258
	7		299	300
	9		457	483
	11		123	121
	13		235	248
	15		109	101
	17		450	467
	19		373	378
	21		260	266
	23		200	200
	25		180	185
1	1	6	241	255
	3		454	476
	5		351	373
	7		239	237
	9		56*	55
	11		339	341
	13		147	154
	15		445	451
	17		249	252
	19		82	76
	21		193	206
	23		259	256
	25		97	107
1	1	7	395	417
	3		440	456
	5		499	528
	7		414	439
	9		124	142
	11		296	312
	13		425	442
	15		395	399
	17		320	320
	19		179	167
	21		263	272
	23		120	119
1	1	8	207	212
	3		257	268
	5		399	428
	7		190	195
	9		99	91
	11		148	144
	13		151	164
	15		174	170
	17		128	147
	19		155	156
	21		185	187
	23		155	158
1	1	9	358	365
	3		241	242
	5		154	166
	7		193	203
	9		264	281
	11		301	312
	13		248	256
	15		302	302
	17		172	164
	19		324	322
	21		76	79
1	1	10	96	98
	3		310	310
	5		114	110
	7		214	217
	9		45*	38
	11		307	306
	13		[illegible]3*	23
	15		85	91
	17		151	150
	19		127	120
1	1	11	46*	19
	3		181	177
	5		145	144
	7		136	127
	9		229	232
	11		126	127
	13		145	155
	15		71	68
	17		57	46
1	1	12	157	152
	3		229	234
1	5	12	179	178
	7		150	150
	9		104	105
	11		81	73
	13		78	78
1	1	13	86	89
	3		62	56
	5		22*	12
	7		193	186
	9		147	143
2	0	0	402	170
	2		248	210
	4		245	228
	6		1110	1219
	8		762	795
	10		95*	76
	12		536	539
	14		797	822
	16		117	129
	18		384	386
	20		605	614
	22		216	216
	24		308	300
	26		198	202
	28		78	71
2	2	1	573	551
	4		64*	82
	6		464	483
	8		49*	53
	10		136	100
	12		132	100
	14		464	476
	16		551	556
	18		113	81
	20		382	388
	22		89	80
	24		316	315
	26		131	131
	28		182	193
2	0	2	372	372
	2		841	884
	4		273	267
	6		122*	93
	8		196	194
	10		754	780
	12		252	255
	14		237	206
	16		674	704
	18		257	260
	20		254	250
	22		236	234
	24		115	110
	26		88	89
	28		223	228
2	2	3	369	365
	4		305	303
	6		199	176
	8		200	204
	10		200	206
	12		663	684
	14		312	302
	16		138	130
	18		541	548
	20		313	308
	22		73	62
	24		326	330
	26		248	257
2	0	4	89	89
	2		492	514
	4		250	231
	6		412	422
	8		113	116
	10		553	567
	12		520	534
	14		215	206
	16		62*	49
	18		199	186
	20		159	155
	22		385	385
	24		93	102
	26		23*	20
2	2	5	362	369
	4		104	101
	6		517	548
	8		232	245
	10		95	85
	12		431	444
	14		196	185
	16		348	357
	18		92	85
	20		197	193
	22		127	131
	24		40*	39
	26		45	43
2	0	6	98	89
	2		98	92
	4		347	363
	6		103	43
	8		557	574
	10		55*	25
2	12	6	307	317
	14		67*	57
	16		88	74
	18		315	312
	20		103	99
	22		298	307
	24		39	25
2	2	7	586	623
	4		166	160
	6		335	346
	8		194	210
	10		261	266
	12		147	148
	14		343	342
	16		63	61
	18		146	142
	20		121	112
	22		83	69
	24		22*	26
2	0	8	263	262
	2		421	417
	4		286	276
	6		69	49
	8		529	558
	10		474	508
	12		58*	35
	14		165	157
	16		97	91
	18		61	20
	20		166	169
	22		150	152
2	2	9	352	349
	4		126	139
	6		239	241
	8		120	114
	10		184	194
	12		201	197
	14		97	105
	16		240	248
	18		173	166
	20		187	189
2	0	10	267	255
	2		422	423
	4		314	317
	6		282	295
	8		68	64
	10		288	287
	12		187	182
	14		301	305
	16		212	207
	18		64	52
2	2	11	106	96
	4		163	164
	6		703	714
	8		173	171
	10		36*	42
	12		286	291
	14		178	179
	16		74	78
2	0	12	243	228
	2		193	193
	4		101	92
	6		105	109
	8		54	49
	10		193	187
	12		153	161
2	2	13	436	427
	4		110	110
	6		149	149
	8		47	51
3	1	1	171	157
	3		237	222
	5		688	688
	7		649	649
	9		676	676
	11		551	536
	13		651	651
	15		138	104
	17		308	303
	19		39*	70
	21		100	74
	23		213	213
	25		229	226
	27		213	219
3	1	2	267	275
	3		784	792
	5		831	842
	7		174	147
	9		181	192
	11		147	151
	13		424	430
	15		319	310
	17		147	133
	19		121	122
	21		316	305
	23		260	257
	25		246	245
	27		203	208
3	1	3	219	202
	3		684	681

Table 1. *(Continued)*

h	k	l	$\|F_o\|$	$\|F_c\|$
3	5	3	141	125
	7		206	199
	9		492	479
	11		488	487
	13		125	116
	15		410	398
	17		368	356
	19		39*	34
	21		291	291
	23		283	271
	25		82	76
	27		162	159
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	3		494	504
	5		260	247
	7		591	583
	9		160	151
	11		592	586
	13		210	199
	15		117	102
	17		418	402
	19		297	291
	21		293	286
	23		84	79
	25		246	248
	27		98	104
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	3		281	284
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	7		346	350
	9		654	659
	11		162	159
	13		312	310
	15		217	222
	17		381	384
	19		365	368
	21		295	300
	23		254	255
	25		179	178
3	1	6	251	254
	3		601	600
	5		482	489
	7		128	124
	9		159	167
	11		188	204
	13		136	126
	15		346	337
	17		262	262
	19		40*	17
	21		300	300
	23		326	327
	25		35	23
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	3		468	476
	5		359	348
	7		466	462
	9		100	92
	11		221	217
	13		444	443
	15		452	451
	17		296	291
	19		224	231
	21		301	305
	23		124	127
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	3		199	196
	5		372	368
	7		241	248
	9		51*	54
	11		205	199
	13		312	312
	15		214	220
	17		69	51
	19		162	156
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	3		40*	43
	5		197	185
	7		157	157
	9		350	343
	11		90	77
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	15		132	139
	17		144	141
	19		259	256
	21		74	67
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	3		257	254
	5		51	62
	7		227	219
	9		176	178
	11		320	305
	13		164	169
	15		125	130
	17		174	177
	19		172	170
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	3		193	182
	5		42	54
	7		208	209
3	9	11	242	249
	11		178	170
	13		158	157
	15		158	150
3	1	12	150	139
	3		87	92
	5		49	48
	7		52	43
	9		34	43
	11		80	74
	13		80	73
3	1	13	61	63
	3		89	93
	5		21*	15
	7		263	254
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	2		118*	103
	4		72*	66
	6		380	364
	8		308	299
	10		320	313
	12		1065	1039
	14		150	134
	16		409	404
	18		907	895
	20		467	438
	22		233	230
	24		106	99
	26		147	145
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	4		535	540
	6		308	288
	8		1023	1009
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	12		428	416
	14		51*	29
	16		0*	29
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	20		196	189
	22		54*	52
	24		267	263
	26		197	197
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	8		249	244
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	4		1506	1565
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	12		178	115
	14		888	830
	16		487	472
	18		85*	64
	20		94	96
	22		281	270
	24		441	439
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	2		100*	101
	4		236	225
	6		408	397
	8		1022	1033
	10		363	335
	12		408	396
	14		60*	76
	16		157	134
	18		209	200
	20		293	287
	22		78	61
	24		29*	20
	26		309	325
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	4		393	388
	6		123	30
	8		229	223
	10		387	370
	12		0*	16
	14		307	310
	16		30*	34
	18		64*	52
	20		152	147
	22		66	59
	24		70	70
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	4		196	200
	6		473	467
	8		450	446
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	14		59*	32
	16		96	91
	18		277	272
	20		233	237
	22		120	123
	24		100	99
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	8		156	148
	10		164	171
	12		363	351
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	18		143	138
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	22		61	62
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	6		402	403
	8		235	218
	10		316	312
	12		62	42
	14		152	147
	16		285	287
	18		141	133
	20		208	205
	22		155	155
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	4		251	250
	6		194	195
	8		579	584
	10		341	328
	12		372	368
	14		53	37
	16		6*	13
	18		222	217
	20		185	180
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	2		252	239
	4		84	22
	6		250	245
	8		258	239
	10		74	69
	12		182	174
	14		48	59
	16		150	145
	18		184	190
4	2	11	134	120
	4		70	61
	6		79	71
	8		139	137
	10		41	40
	12		291	292
	14		297	290
4	0	12	30*	21
	2		200	189
	4		170	160
	6		42	32
	8		135	129
	10		66	60
	12		124	116
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	4		367	353
	6		52	47
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	3		82*	100
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	7		628	601
	9		544	523
	11		395	381
	13		526	495
	15		149	137
	17		206	201
	19		161	156
	21		47*	37
	23		140	136
	25		223	227
	27		214	223
5	1	2	227	227
	3		551	543
	5		604	593
	7		69*	52
	9		197	196
	11		249	239
	13		285	272
	15		270	256
	17		107	94
	19		128	133
	21		223	220
	23		194	188
	25		154	153
	27		203	208
5	1	3	158	147
	3		634	622
	5		251	232
	7		248	241
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	11		389	374
	13		118	134
	15		386	370
	17		299	275
	19		114	90
	21		272	267
	23		208	196
	25		99	92
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	3		340	341
	5		278	255
	7		578	555
	9		153	153
	11		543	520
	13		180	160
	15		169	165
	17		298	283
	19		349	338
	21		235	232
	23		66	61
	25		270	274
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	3		322	321
	5		414	418
	7		301	294
	9		427	421
	11		125	120
	13		235	225
	15		316	299
	17		342	335
	19		298	298
	21		184	182
	23		271	271
	25		188	186
5	1	6	208	216
	3		581	567
	5		398	394
	7		203	188
	9		146	134
	11		303	295
	13		169	156
	15		419	411
	17		187	182
	19		87	74
	21		163	163
	23		264	262
5	1	7	324	325
	3		483	473
	5		378	358
	7		393	377
	9		91	80
	11		188	194
	13		350	345
	15		489	488
	17		259	251
	19		254	253
	21		276	277
	23		157	161
5	1	8	180	189
	3		135	118
	5		253	243
	7		197	189
	9		11*	35
	11		276	261
	13		250	238
	15		174	169
	17		16*	29
	19		157	153
	21		100	103
5	1	9	395	379
	3		140	130
	5		207	210
	7		79	82
	9		336	330
	11		127	115
	13		228	222
	15		198	199
	17		168	158
	19		310	307
5	1	10	239	227
	3		204	195
	5		164	153
	7		227	236
	9		138	143
	11		320	308
	13		143	137
	15		118	118
	17		136	140
5	1	11	97	87
	3		190	183
	5		66	71
	7		178	169
	9		204	201
	11		119	120
	13		103	105
5	1	12	198	185
	3		88	89
	5		120	123
	7		88	79
5	9	12	38*	45
	11		101	97
5	1	13	98	97
	3		95	88
6	0	0	111*	98
	2		347	302
	4		220	203
	6		940	910
	8		738	707
	10		76*	63
	12		346	318
	14		734	699
	16		244	225
	18		415	390
	20		570	552
	22		191	190
	24		263	255
	26		251	255
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	4		161	114
	6		435	414
	8		133	121
	10		111	55
	12		59*	47
	14		366	349
	16		457	427
	18		82	65
	20		373	365
	22		28*	40
	24		256	241
	26		163	167
6	0	2	303	309
	2		743	718
	4		137	133
	6		157	152
	8		233	215
	10		715	681
	12		260	247
	14		185	156
	16		702	672
	18		251	231
	20		227	219
	22		160	167
	24		64	63
	26		85	87
6	2	3	373	351
	4		260	258
	6		222	172
	8		197	190
	10		246	235
	12		651	611
	14		219	208
	16		153	141
	18		537	517
	20		316	296
	22		27*	31
	24		289	287
6	0	4	70*	75
	2		458	453
	4		283	275
	6		294	284
	8		168	159
	10		450	430
	12		448	420
	14		201	181
	16		68*	78
	18		136	131
	20		140	134
	22		394	385
	24		65	69
6	2	5	223	214
	4		64*	41
	6		518	520
	8		236	226
	10		172	150
	12		441	430
	14		128	115
	16		346	335
	18		103	93
	20		149	150
	22		156	151
	24		39	32
6	0	6	96	90
	2		137	129
	4		283	277
	6		74	69
	8		488	476
	10		97	63
	12		212	217
	14		84	77
	16		75	62
	18		253	243
	20		114	107
	22		259	257
6	2	7	410	406
	4		136	128
	6		302	302
	8		149	141
	10		276	264
	12		100	91

Table 1. *(Continued)*

h	k	l	$\lvert F_o\rvert$	$\lvert F_c\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_c\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_c\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_c\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_c\rvert$
6	14	7	441	430	7	9	5	547	521	8	4	3	1147	1142	9	11	2	257	231	10	14	1	376	355
	16		93	73		11		95	78		6		116	112		13		198	188		16		420	405
	18		112	111		13		197	192		8		543	506		15		200	184		18		109	118
	20		133	128		15		187	188		10		276	257		17		97	107		20		205	205
	22		68	65		17		429	412		12		90	105		19		123	134		22		84	87
6	0	8	264	252		19		365	371		14		701	650		21		185	185	10	0	2	351	352
	2		393	386		21		274	278		16		408	382		23		104	110		2		507	510
	4		334	322		23		142	145		18		52*	4	9	1	3	302	300		4		224	227
	6		81	75	7	1	6	220	229		20		197	186		3		338	333		6		67*	78
	8		539	534		3		424	424		22		197	197		5		127	102		8		65*	42
	10		453	445		5		228	223		24		377	381		7		132	122		10		459	439
	12		45*	33		7		170	158	8	0	4	115	115		9		446	410		12		140	113
	14		159	152		9		79	37		2		71*	66		11		361	327		14		124	109
	16		65	47		11		214	201		4		465	459		13		72	73		16		437	427
	18		26*	24		13		91	90		6		313	294		15		164	161		18		238	227
	20		152	148		15		289	279		8		721	691		17		167	158		20		207	205
6	2	9	449	406		17		247	240		10		267	248		19		175	173		22		223	234
	4		172	168		19		77	74		12		557	525		21		173	173	10	2	3	162	168
	6		250	240		21		217	225		14		27*	27		23		224	228		4		220	219
	8		121	127	7	1	7	324	329		16		176	171	9	1	4	447	446		6		125	87
	10		99	89		3		389	386		18		167	154		3		197	194		8		108	98
	12		159	152		5		433	427		20		213	218		5		315	312		10		128	127
	14		102	100		7		326	317		22		57	59		7		498	484		12		502	474
	16		214	211		9		209	192	8	2	5	86	81		9		155	140		14		235	228
	18		80	73		11		275	261		4		260	255		11		431	416		16		148	140
6	0	10	229	214		13		450	437		6		84	48		13		161	155		18		344	341
	2		387	371		15		369	362		8		419	406		15		90	84		20		212	211
	4		317	300		17		343	337		10		422	416		17		195	190	10	0	4	133	135
	6		279	273		19		109	113		12		90	72		19		271	275		2		328	331
	8		60	56		21		208	209		14		285	272		21		197	202		4		114	104
	10		256	254	7	1	8	201	184		16		55*	75	9	1	5	483	500		6		327	314
	12		146	135		3		265	263		18		158	160		3		173	172		8		120	104
	14		271	267		5		471	462		20		305	305		5		133	136		10		370	352
	16		173	169		7		32*	18		22		27*	28		7		273	272		12		430	408
6	2	11	61	64		9		36*	30	8	0	6	496	511		9		245	236		14		141	126
	4		159	147		11		48*	11		2		164	161		11		251	250		16		0*	33
	6		615	599		13		231	229		4		62*	51		13		187	176		18		165	161
	8		161	156		15		181	174		6		450	437		15		142	136		20		101	98
	10		97	94		17		179	174		8		630	614		17		292	295	10	2	5	271	283
	12		237	228		19		195	186		10		477	455		19		236	247		4		117	114
6	0	12	165	159	7	1	9	200	197		12		722	697		21		153	155		6		337	335
	2		185	176		3		132	137		14		61	80	9	1	6	188	190		8		150	137
	4		79	72		5		174	184		16		92	98		3		284	299		10		85	54
	6		70	70		7		255	250		18		109	96		5		301	307		12		277	275
	8		29*	36		9		225	217		20		79	82		7		161	150		14		157	158
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	3		123	86		13		259	255		4		553	553		11		274	273		18		74	77
	5		435	402		15		251	247		6		88	69		13		115	110		20		146	151
	7		857	823		17		202	196		8		152	110		15		375	375	10	0	6	137	142
	9		673	630	7	1	10	74	69		10		128	129		17		143	142		2		0*	23
	11		515	468		3		245	250		12		74	77		19		41	38		4		245	264
	13		495	456		5		86	81		14		156	154	9	1	7	336	346		6		58*	43
	15		129	116		7		171	179		16		351	352		3		312	323		8		461	454
	17		176	166		9		49	51		18		59	65		5		311	318		10		55*	15
	19		228	210		11		267	260		20		107	106		7		331	329		12		310	309
	21		214	207		13		32	34	8	0	8	245	253		9		108	98		14		51	35
	23		175	179	7	1	11	80	88		2		222	218		11		209	209		16		100	105
	25		226	234		3		129	138		4		161	162		13		239	235		18		243	245
7	1	2	43*	73		5		109	119		6		319	310		15		290	282	10	2	7	473	488
	3		485	488		7		176	178		8		88	76		17		212	208		4		157	156
	5		631	608		9		218	217		10		248	246	9	1	8	97	100		6		226	235
	7		267	252	8	0	0	2521	2789		12		38*	40		3		138	138		8		173	176
	9		66*	26		2		122	97		14		47*	53		5		220	222		10		178	167
	11		114	101		4		323	306		16		24*	22		7		230	228		12		124	122
	13		468	435		6		163	148		18		239	230		9		121	128		14		125	121
	15		300	272		8		81*	76	8	2	9	83	85		11		215	212		16		52	50
	17		168	156		10		157	135		4		308	310		13		96	81	10	0	8	165	161
	19		168	157		12		328	305		6		231	219		15		117	114		2		263	266
	21		255	248		14		276	258		8		252	254	9	1	9	345	354		4		155	151
	23		181	193		16		348	334		10		[illegible]	148		3		232	235		6		87	84
	25		236	248		18		975	938		12		37[illegible]	359		5		111	113		8		354	342
7	1	3	339	322		20		555	537		14		47	56		7		74	82		10		356	361
	3		425	419		22		74	83	8	0	10	514	541		9		198	194		12		58	52
	5		283	277		24		86	83		2		204	198		11		169	168		14		128	129
	7		180	169	8	2	1	153	128		4		49	51		13		144	143	10	2	9	152	155
	9		656	586		4		320	310		6		170	174	9	1	10	74	72		4		144	140
	11		173	169		6		315	297		8		161	158		3		223	226		6		119	114
	13		230	207		8		695	625		10		84	79		5		134	133		8		45	45
	15		230	222		10		467	433		12		58	59		7		159	169		10		191	190
	17		339	326		12		552	530	8	2	11	74	72		9		60	68		12		186	190
	19		108	101		14		126	134		4		155	159	9	1	11	48	45	10	0	10	251	256
	21		180	175		16		262	245		6		37	40		3		149	154		2		260	271
	23		193	191		18		127	131		8		244	242	10	0	0	379	322		4		224	227
	25		63	58		20		69	68	9	1	1	129	131		2		161	118		6		175	183
7	1	4	789	799		22		114	114		3		132	107		4		218	215	11	1	1	172	179
	3		492	495		24		102	106		5		250	239		6		756	720		3		184	177
	5		197	187	8	0	2	292	280		7		445	423		8		479	448		5		367	356
	7		530	505		2		153	159		9		493	458		10		47*	28		7		433	425
	9		335	314		4		765	745		11		406	369		12		403	374		9		538	519
	11		408	374		6		221	232		13		394	366		14		575	539		11		450	441
	13		335	307		8		527	507		15		274	262		16		214	202		13		505	486
	15		123	110		10		223	199		17		49*	56		18		189	193		15		63	56
	17		456	431		12		108	88		19		198	186		20		467	463		17		245	241
	19		327	322		14		380	355		21		94	92		22		142	141		19		86	83
	21		289	282		16		72	32		23		242	249	10	2	1	316	308		21		95	94
	23		58	43		18		129	108	9	1	2	137	141		4		151	150	11	1	2	179	172
7	1	5	603	611		20		159	151		3		290	267		6		223	235		3		515	518
	3		224	227		22		390	390		5		351	341		8		136	125		5		578	567
	5		264	269		24		13*	11		7		56*	55		10		113	83		7		244	229
	7		155	145	8	2	3	281	260		9		76*	79		12		162	143		9		87	88

Table 1. *(Continued)*

h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$	h	k	l	$\|F_o\|$	$\|F_c\|$
11	11	2	125	102	11	9	8	40	38	12	4	5	225	239	13	5	4	214	223	14	0	4	114	125
	13		400	380		11		61	59		6		49*	21		7		290	291		2		269	271
	15		234	218	11	1	9	186	196		8		133	143		9		111	106		4		185	192
	17		147	148		3		93	98		10		222	220		11		246	247		6		125	127
	19		96	90		5		110	118		12		34*	31		13		113	112		8		172	172
11	1	3	218	205		7		148	148		14		219	224		15		118	114		10		148	152
	3		341	333		9		222	227		16		36	17	13	1	5	220	225		12		221	229
	5		100	82	12	0	0	1354	1337	12	0	6	729	766		3		200	207	14	2	5	57	59
	7		189	181		2		112	106		2		97	107		5		166	181		4		98	99
	9		272	261		4		110	105		4		134	136		7		219	224		6		303	318
	11		363	334		6		348	347		6		269	280		9		79	86		8		138	148
	13		61*	50		8		221	215		8		280	285		11		97	90		10		118	132
	15		278	253		10		226	215		10		275	278		13		116	120	14	0	6	145	147
	17		237	237		12		583	568		12		271	284	13	1	6	173	180		2		100	97
	19		80	78		14		97	110		14		54	49		3		274	289		4		123	124
11	1	4	375	379		16		221	219	12	2	7	102	101		5		167	177		6		48	35
	3		352	362		18		511	516		4		267	271		7		163	170		8		277	291
	5		233	229	12	2	1	124	132		6		96	92		9		60	58	14	2	7	156	157
	7		315	301		4		344	337		8		79	81		11		212	225	15	1	1	233	233
	9		115	99		6		137	138		10		100	101	13	1	7	211	221		3		64	60
	11		359	339		8		535	520		12		211	220		3		268	280		5		245	244
	13		210	205		10		379	367	12	0	8	66	56		5		225	233		7		466	471
	15		100	88		12		216	213		2		161	166		7		149	155		9		382	388
	17		309	317		14		62	68		4		157	159		9		103	107		11		296	300
	19		138	140		16		62	41		6		256	261	13	1	8	77	73	15	1	2	78	76
11	1	5	533	555		18		166	171		8		178	181		3		53	53		3		280	288
	3		204	200	12	0	2	315	316	12	2	9	94	93	14	0	0	120	88		5		371	376
	5		266	266		2		147	140	13	1	1	104	98		2		264	251		7		195	198
	7		303	312		4		843	855		3		95	79		4		159	159		9		34*	26
	9		479	482		6		102	89		5		281	272		6		429	419		11		115	116
	11		146	148		8		154	163		7		328	329		8		399	391	15	1	3	155	150
	13		234	236		10		42*	40		9		336	323		10		122	117		3		282	287
	15		88	97		12		153	153		11		174	175		12		50	44		5		144	149
	17		294	303		14		364	358		13		244	235		14		154	370		7		116	124
11	1	6	193	195		16		285	291		15		131	134	14	2	1	140	139		9		281	292
	3		340	354		18		164	167		17		80	79		4		150	151	15	1	4	402	422
	5		307	324	12	2	3	168	165	13	1	2	123	116		6		178	176		3		346	359
	7		145	145		4		828	838		3		173	166		8		148	149		5		144	145
	9		100	95		6		61*	55		5		215	211		10		17*	22		7		227	236
	11		45*	42		8		334	315		7		72	89		12		33*	24	15	1	5	330	346
	13		52	51		10		171	161		9		108	113		14		143	139		3		195	199
	15		161	163		12		164	158		11		196	197	14	0	2	227	239		5		148	152
	17		210	212		14		475	465		13		106	113		2		376	368	16	0	0	1141	1169
11	1	7	343	347		16		304	310		15		125	133		4		112	126		2		91	98
	3		260	270		18		31*	26		17		68	74		6		71	68		4		282	285
	5		210	216	12	0	4	103	82	13	1	3	70	79		8		132	128		6		29*	11
	7		290	301		2		135	134		3		325	320		10		346	345	16	2	1	100	97
	9		93	91		4		135	140		5		199	199		12		108	113		4		202	215
	11		157	155		6		276	271		7		226	220		14		34	36		6		103	103
	13		292	304		8		603	603		9		234	226	14	2	3	164	164	16	0	2	146	141
	15		247	251		10		243	242		11		174	176		4		186	179		2		102	100
11	1	8	114	115		12		310	298		13		112	113		6		105	97		4		362	373
	3		186	186		14		56	59		15		213	219		8		91	91					
	5		301	304		16		79	71	13	1	4	240	240		10		171	171					
	7		188	192	12	2	5	52*	28		3		115	118		12		358	375					

* unobserved reflection

Several cycles of least-squares refinement in which individual form factors were used for Hg and Tl atoms and in which isotropic temperature factors for each atom were varied, reduced R to $11^0/_0$. During these calculations, an equal weight was used for all reflections. Nonionized atomic form factors given by IBERS, THOMAS *et al.*, THOMAS and UMEDA, FREEMAN and WATSON, and DAWSON were employed for Hg, Tl, Sb, As and S respectively (*International tables*, 1962). Additional cycles of least-squares refinement in which anisotropic temperature factors were varied, reduced R to $4.8^0/_0$ for all 1772 reflections. In the course of these calculations, individual weights calculated by the modified formula of GABE (1966) was used for each of the reflections:

$$w = \frac{1}{\sigma^2(F)} = 4F_o{}^2 \Big/ \sum_{i=1}^{4} \left\{ \left(\frac{\partial F_o{}^2}{\partial q_i} \right)^2 \sigma^2\,(q_i) \right\},$$

where q_1 = peak count, q_2 = background count, $q_3 = (LP)^{-1}$ and q_4 = transmission. At the final stage, the effect of anomalous dis-