Zeitschrift für Kristallographie. Bd. 122, S. 433—456 (1965)

The crystal structure of rathite-I*

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(Received May 10, 1965)

Auszug

Die Kristallstruktur von Rathit-I wurde mittels dreidimensionaler Intensitätsdaten bestimmt. Vier Formeleinheiten $(Pb,Tl)_3As_4(As,Ag)S_{10}$ sind in der Einheitszelle der Symmetrie $P2_1/a$ mit $a = 25{,}16$ Å, $b = 7{,}94$ Å, $c = 8{,}47$ Å, $\beta = 100°28'$ enthalten. Die wahre Symmetrie von Rathit-I ist möglicherweise triklin. Die Lösung lieferten die Ähnlichkeit der Struktur mit derjenigen von Rathit-III und spezielle Verhältnisse der Röntgendiagramme.

Von drei unabhängigen Pb(Tl)-Atomen sind zwei von neun S-Atomen umgeben, das andere von sieben. Die As-Atome weisen trigonal-pyramidale Koordination durch die S-Atome auf. Von einem As-Atom wird angenommen, daß es statistisch von zwei verschiedenen trigonal-pyramidalen S-Koordinationen umgeben wird. Ein anderes As-Atom ist teilweise durch Ag ersetzt.

Die Struktur besteht aus zweierlei Schichten parallel zu (100). Die erste Art hat die Zusammensetzung $(Pb,Tl)S_3$ und besteht aus den Koordinationspolyedern um die Pb(Tl)-Atome mit Neuner-Koordination. Die zweite Art ist aus Pb(Tl)-, As(Ag)- und S-Atomen zusammengesetzt, welche ein deformiertes PbS-Gitter bilden. Trigonale As-S_3-Pyramiden sind zu Ketten endlicher Länge vereinigt.

Abstract

The crystal structure of rathite-I has been determined with the use of three-dimensional intensity data. Four chemical units of $(Pb,Tl)_3As_4(As,Ag)S_{10}$ are contained in the unit-cell of the symmetry $P2_1/a$ with $a = 25.16$ Å, $b = 7.94$ Å, $c = 8.47$ Å, $\beta = 100°28'$. The true symmetry of rathite-I may be triclinic. The solution was obtained from the similarity of the crystal structure to that of rathite-III and from a peculiar feature of the x-ray diagrams.

Among three independent Pb(Tl) atoms two are surrounded by nine S atoms and the other is surrounded by seven S atoms. As atoms have trigonal-pyramidal coordinations by S atoms. One As atom, however, is believed to occupy statistically two different trigonal-pyramidal S coordinations. Another As atom is partially replaced by Ag.

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* Contribution No. 155. — Part 19 on sulfides and sulfosalts.

The structure is composed of two kinds of layers parallel to (100). Layers of the first kind have the composition (Pb,Tl)S_3, and consist of coordination polyhedra around the Pb(Tl) atoms which are coordinated by nine S atoms. The layers of the second kind are composed of Pb(Tl), As(Ag) and S atoms, having a deformed PbS-type structure. Trigonal As-S_3 pyramids are linked into strings of finite length.

1. Introduction

Rathite-I, $(Pb,Tl)_3As_4(As,Ag)S_{10}$, is a mineral of a sulfosalt group, to which rathite-II, rathite-III, rathite-IV, dufrenoysite, baumhauerite and scleroclase belong. A characteristic feature of these minerals is that they have periods of 8.4 Å and 7.9 Å along two mutually perpendicular directions. Though most of the structures have already been investigated, no precise structure has yet been revealed owing to the large absorption effects and the large unit-cell dimensions. Some of the structures reported contain unreasonable features such as, for example, infinite chains of As-S_3 pyramids along the 8.4 Å axes which, as was pointed out by Y. Iitaka and W. Nowacki (1961), cannot exist.

The structure determination of rathite-I was carried out in order to obtain precise information concerning the structural principles of this group of minerals. It was also desired to clarify the relationship of rathite-III and rathite-I, which are dimorphous if the small amount of Tl and Ag in the latter plays no significant role in the formation of the mineral and can be replaced by Pb and by As respectively.

Rathite-III (Le Bihan, 1962) has hitherto not been found by us in the Lengenbach quarry. It is important to mention that the rathite-I of Le Bihan (1962) is almost identical with dufrenoysite and was called rathite-I*a* by us (Nowacki *et al.*, 1964). Rathite-II was first described by Berry (1953). The lattice constants, space group and chemical composition are:

Mineral	Formula	*a*	*b*	*c*	β	Space group
Rathite-I	$(Pb,Tl)_3As_4(As,Ag)S_{10}$	25.16	7.94	8.47	100°28′	$P2_1/a$ ($P\bar{1}$)
Rathite-III	$Pb_3As_5S_{10}$	24.52	7.91	8.43	90°	$P2_1$
Rathite-II	$Pb_9As_{13}S_{28}$	8.43	70.9	7.91	90°	$P2_1$

Thus, rathite-I and -III form two modifications of a single species and should perhaps have a name different from rathite-II; it is not, however, possible for us to introduce one. In the Lengenbach quarry rathite-II is frequently found, whereas rathite-I occurs rarely, and then usually polysynthetically twinned. The microprobe analysis (NOWACKI und BAHEZRE, 1963) yielded the composition $Pb = 41.2 \pm 1$, $As = 27.0 \pm 0.5$, $S = 28 \pm (1-2)$, $Tl = 3.6 \pm 1$, $\Sigma = 99.7\,{}^0/_0$.

2. Experimental

We looked through a large number of specimens from Lengenbach for a suitable rathite-I crystal as described by PEACOCK and BERRY (1940), but could not find one untwinned. Finally, through the kindness of Dr. L. G. BERRY (Queens University, Kingston, Canada) we obtained a good crystal (also from Lengenbach) for intensity measurements.

The unit-cell dimensions obtained from Weissenberg photographs are,

$$a = 25.16 \pm 0.02\ \text{Å},\quad b = 7.94 \pm 0.01\ \text{Å},\quad c = 8.47 \pm 0.01\ \text{Å},$$
$$\alpha = 90^\circ \pm 10',\quad \beta = 100^\circ 28' \pm 10',\quad \gamma = 90^\circ \pm 10'.$$

Although the space group of rathite-I was reported as $P2_1/a$, the Weissenberg photographs showed small discrepancies between the intensities of hkl and $\bar{h}k\bar{l}$ reflections, indicating triclinic symmetry for this crystal. Moreover, several weak reflections with $h =$ odd were observed among the $h0l$ reflections. The true space group must, therefore, be $P1$ or $P\bar{1}$. However, it is difficult to say whether these small deviations from monoclinic symmetry are common to all rathite-I crystals or whether they are only a special characteristic of the crystal examined, caused by a small content of Tl and Ag. For the structure determination the space group $P2_1/a$ was assumed, and the average intensities of the hkl and $\bar{h}k\bar{l}$ reflections were used, the difference being very small.

A sphere with a radius of 0.06 mm was prepared for the intensity measurement from a piece of the crystal. The integrated Weissenberg photographs were taken with Cu$K\alpha$ radiation up to the 7-th layer around the b axis and up to the second layer around the c axis. The intensities were measured with a Joyce-Loebl microdensitometer, and corrected for the Lorentz-polarization and absorption effects with the programme of Y. IITAKA for the Bull Γ AET electronic computer. The linear absorption coefficient of the crystal is 855 cm^{-1} for Cu$K\alpha$ and the absorption-correction factors for the sphere range between 180 at $\theta = 0^\circ$ and 14 at $\theta = 90^\circ$.

The chemical analysis of the crystal was carried out by W. NOWACKI and C. BAHEZRE (1963) with a Castaing x-ray microanalyser.

The unit-cell content calculated from the result, assuming 5.37 g/cm^3 (DANA's system of mineralogy, Vol. I, 1944) for the density, is $Pb_{10.7}Tl_{0.9}As_{19.3}S_{40.1}$, or approximately $Pb_{11}Tl_1As_{20}S_{40}$. In the actual structure-factor calculations, the Tl atoms were taken as Pb atoms, since the differences between the atomic scattering factors of these two elements are quite small, and since the number of Tl atoms in the unit cell is less than the value required by the space group $P2_1/a$.

3. Structure analysis

Since the *hk*0 x-ray diffraction diagram of rathite-I is almost identical with that of rathite-III (M.-TH. LE BIHAN, 1962), the *c* axis projection of the structure should have the same atomic arrangement as that of rathite-III. Actually the values $a \sin\beta$, b and c for rathite-I (24.75 Å, 7.94 Å, 8.47 Å) are nearly equal to the values found for rathite-III (24.52 Å, 7.91 Å, 8.43 Å) and the chemical contents of their unit cells are identical if the Tl atoms in rathite-I are replaced by Pb atoms ($Pb_{12}As_{20}S_{40}$). Calculation of the *hk*0 structure factors were, therefore, carried out with a programme by Y. IITAKA for Bull *Γ* AET utilizing the atomic coordinates of rathite-III; fairly good agreement between the observed and the calculated structure factors was obtained, the *R*-factor being 0.38. This projection was refined by difference Fourier syntheses until the *R*-value was reduced to 0.16.

The *z* coordinates were obtained from a special feature of the *h*0*l* x-ray diagram. Since the *h*0*l* intensity distribution along the *c* axis direction in reciprocal space is periodic to a fairly good approximation with the period 4, all atoms should lie nearly on a set of equally spaced planes perpendicular to the *c* axis, the interplaner spacing being *c*/4. There are two possible sets of planes which satisfy both this condition and the symmetry requirement for $P2_1/a$:

$$z = \frac{1}{8} + \frac{x}{2} = \frac{1}{8} - x\cos\beta,\ z = \frac{3}{8} + \frac{x}{2},\ z = \frac{5}{8} + \frac{x}{2},\ z = \frac{7}{8} + \frac{x}{2},$$

and

$$z = \frac{x}{2},\ z = \frac{1}{4} + \frac{x}{2},\ z = \frac{2}{4} + \frac{x}{2},\ z = \frac{3}{4} + \frac{x}{2}.$$

The structural similarity to rathite-III as well as crystallochemical considerations suggested that the correct set should be the former, and furnished two probable models of the structure. The true structure was found after several cycles of refinements of these models tested with the *h*0*l* difference Fourier projection. The *R* value of the correct model was reduced from the initial value 0.49 to 0.19 for the *h*0*l* reflections during the refinement.

4. Refinement

During the preliminary study with two-dimensional data, it was found from the Fourier projections that the As(5) atom has a lower electron density than the other As atoms and that there is a peak at a position about 0.6 Å apart from the position postulated for As(5). The agreement between the observed and the calculated structure factors becomes worse if As(5) is put at this peak. Therefore it was suspected that the As(5) atom statistically occupies both positions.

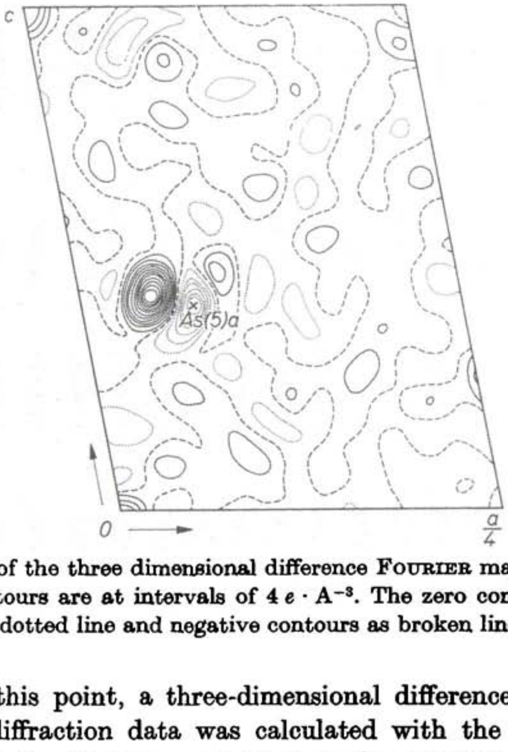

Fig. 1. A section of the three dimensional difference FOURIER map through the As(5) atom. Contours are at intervals of $4\,e \cdot A^{-3}$. The zero contour is shown as a dotted line and negative contours as broken lines

To clarify this point, a three-dimensional difference Fourier including 3477 diffraction data was calculated with the O. S. MILLS' programme for the Mercury computer at the calculating center of Oxford University. A part of the section through the As(5) atom is shown in Fig.1, in which a negative region at the postulated As(5) position and the peak near it is clearly observed, suggesting a statistical distribution of the As(5) atom between the two positions.

Three-dimensional least-squares refinements using equal weights for all reflections and assuming the statisticall distribution of the As(5) atom were then carried out with the programme written by C. T. PREWITT for the I.B.M. 7090 computer. Anisotropic temperature

Table 1. *The final positional coordinates and the populations of the As (5) atoms*

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	population	$\sigma(w)$
Pb(1)	0.79493	0.00005	0.24587	0.00016	0.52425	0.00013		
Pb(2)	0.29663	0.00006	0.25004	0.00020	0.02117	0.00015		
Pb(3)	0.07201	0.00005	0.08812	0.00017	0.90401	0.00014		
As(1)	0.65908	0.00010	0.14956	0.00036	0.70133	0.00030		
As(2)	0.64775	0.00011	0.16774	0.00037	0.24983	0.00034		
As(3)	0.45735	0.00013	0.15358	0.00044	0.31332	0.00043		
As(4)	0.44043	0.00010	0.16767	0.00037	0.86116	0.00030		
As(5) *a*	0.07434	0.00024	0.0154	0.00080	0.4164	0.00065	0.807	0.015
As(5) *b*	0.05005	0.00031	0.0316	0.00112	0.4270	0.00093	0.402	0.013
S(1)	0.26381	0.00025	0.0017	0.00082	0.2580	0.00073		
S(2)	0.72350	0.00023	0.0211	0.00081	0.2494	0.00071		
S(3)	0.17470	0.00024	0.1793	0.00077	0.4949	0.00068		
S(4)	0.18042	0.00025	0.1696	0.00081	0.9055	0.00074		
S(5)	0.88749	0.00024	0.1612	0.00078	0.3051	0.00069		
S(6)	0.87973	0.00025	0.1200	0.00080	0.8307	0.00072		
S(7)	0.40154	0.00024	0.0184	0.00082	0.6328	0.00068		
S(8)	0.40741	0.00030	0.0078	0.00087	0.0405	0.00078		
S(9)	0.01249	0.00032	0.1999	0.00100	0.5872	0.00082		
S(10)	0.00907	0.00025	0.1833	0.00089	0.1798	0.00074		

Table 2. *Temperature factors*

The values are the coefficients in the expression exp $[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{23}kl + 2\beta_{13}lh)]$

atom	$\beta_{11}\cdot 10^5$	$\sigma(\beta_{11})\cdot 10^5$	$\beta_{22}\cdot 10^5$	$\sigma(\beta_{22})\cdot 10^5$	$\beta_{33}\cdot 10^5$	$\sigma(\beta_{33})\cdot 10^5$	$\beta_{12}\cdot 10^5$	$\sigma(\beta_{12})\cdot 10^5$	$\beta_{23}\cdot 10^5$	$\sigma(\beta_{23})\cdot 10^5$	$\beta_{13}\cdot 10^5$	$\sigma(\beta_{13})\cdot 10^5$
Pb(1)	158	2	1394	43	1032	17	—11	5	72	15	74	4
Pb(2)	216	3	2104	47	1225	22	—172	7	526	20	2	6
Pb(3)	153	2	1644	44	1229	18	23	5	284	16	127	5
As(1)	101	4	1126	61	781	36	5	11	—12	33	87	9
As(2)	103	4	1026	64	1132	39	54	12	184	36	99	10
As(3)	149	6	1391	75	1729	49	80	15	252	46	259	13
As(4)	98	4	1178	61	775	36	—14	11	—9	33	66	9
As(5)*a*	153	12	2030	117	1412	86	8	27	230	72	90	23
As(5)*b*	50	11	1299	160	930	116	23	30	—111	96	80	26
S(1)	107	9	821	98	847	76	47	23	103	70	71	20
S(2)	86	8	889	94	804	73	38	22	3	67	62	19
S(3)	105	8	676	93	704	71	—18	21	—111	65	70	19
S(4)	108	10	802	127	905	90	—42	28	—139	84	129	24
S(5)	94	8	753	96	767	74	—33	22	65	68	56	20
S(6)	111	9	711	99	828	77	33	23	74	70	37	21
S(7)	102	8	945	94	650	73	—25	22	49	67	85	19
S(8)	172	11	837	134	862	95	11	30	110	90	164	26
S(9)	174	12	1252	143	896	104	55	32	0	97	64	28
S(10)	97	9	1133	99	856	77	22	23	—169	70	73	21

Table 3. *The calculated and the observed structure amplitudes*

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
4 0 0	18	34	-20 0 3	96	111	-6 0 7	100	114	8 1 1	67	-64	-18 1 2	361	357
6	154	-144	-22	72	80	-8	98	-96	9	155	-174	-19	89	-95
8	695	-650	-24	38	35	-10	178	-177	10	0	19	-20	225	-226
10	198	191	-26	228	211	-12	29	-36	11	24	-29	-21	0	-1
12	174	-158	-28	75	-85	-14	45	-35	12	132	133	-22	62	-49
14	442	415	-30	33	-39	-16	129	123	13	0	1	-23	67	56
16	31	28	0 0 4	202	228	-18	273	269	14	57	53	-24	38	38
18	0	5	2	80	-92	-20	136	123	15	104	110	-25	77	79
20	130	136	4	150	156	-22	71	-73	16	19	9	-26	67	-74
22	240	-231	6	576	577	-24	45	-45	17	56	60	-27	47	45
24	58	58	8	102	-99	-26	36	37	18	63	56	-28	100	113
26	50	35	10	120	113	-28	95	-115	19	0	3	-29	31	-26
28	34	24	12	325	-304	0 0 8	83	92	20	67	-59	-30	18	-26
30	29	29	14	48	-46	2	104	-102	21	59	-53	-31	38	-38
0 0 1	78	133	16	94	-82	4	217	-228	22	39	-32	0 1 3	310	338
2	121	173	18	124	-111	6	61	56	23	0	6	1	127	-98
4	0	-9	20	270	242	8	142	-137	24	48	41	2	132	127
6	93	-120	22	67	-62	10	140	133	25	43	-36	3	122	164
8	90	-134	24	26	-10	12	54	56	26	14	-17	4	0	11
10	103	-124	26	36	46	14	24	32	27	29	-25	5	119	143
12	151	138	-2 0 4	1112	-1181	16	31	46	28	40	-42	6	0	-5
14	216	201	-4	102	110	-2 0 8	138	-152	29	45	47	7	40	34
16	81	85	-6	48	-30	-4	315	325	30	21	-22	8	66	-72
18	20	-13	-8	160	165	-6	114	-118	-1 1 1	50	23	9	51	-64
20	109	-108	-10	360	346	-8	0	2	-2	0	5	10	0	-14
22	132	-122	-12	213	-209	-10	109	-117	-3	124	-127	11	155	-154
24	193	-176	-14	200	184	-12	178	-168	-4	36	52	12	149	131
26	57	64	-16	345	-336	-14	92	99	-5	66	-102	13	0	0
28	27	25	-18	0	-26	-16	44	-46	-6	54	-46	14	125	115
30	39	49	-20	92	94	-18	184	183	-7	47	7	15	46	40
-2 0 1	27	50	-22	0	0	-20	19	-10	-8	263	-289	16	41	37
-4	150	-166	-24	198	201	-22	51	-52	-9	138	120	17	106	98
-6	334	-365	-26	68	46	-24	76	-71	-10	23	-14	18	0	14
-8	204	-226	-28	64	-62	0 0 9	0	-1	-11	58	68	19	72	60
-10	129	134	-30	61	-63	2	104	-115	-12	112	96	20	76	-75
-12	48	68	0 0 5	235	-276	4	14	0	-13	147	160	21	37	16
-14	78	85	2	38	10	6	59	-68	-14	51	57	22	33	-29
-16	251	254	4	194	221	8	68	-78	-15	50	-34	23	58	-61
-18	72	-84	6	38	56	10	30	20	-16	160	157	24	0	-22
-20	85	-80	8	103	120	12	28	32	-17	69	-73	25	54	-54
-22	65	56	10	87	90	-2 0 9	164	181	-18	54	44	26	0	-8
-24	46	40	12	21	-15	-4	16	29	-19	109	-108	27	25	-14
-26	0	-10	14	21	-33	-6	28	-5	-20	99	-99	28	65	76
-28	29	19	16	55	-39	-8	51	-55	-21	92	-86	-1 1 3	240	-248
-30	17	-23	18	47	42	-10	69	-77	-22	146	-152	-2	126	129
-32	0	-1	20	82	75	-12	90	-88	-23	75	-68	-3	0	-60
0 0 2	334	342	22	102	95	-14	35	36	-24	45	-41	-4	54	-51
2	89	-98	24	21	2	-16	62	57	-25	82	92	-5	92	-116
4	246	252	-2 0 5	0	-37	-18	54	-62	-26	53	-54	-6	114	-117
6	98	88	-4	49	-41	-20	64	57	-27	129	133	-7	0	-29
8	937	-876	-6	72	74	-22	0	-6	-28	37	-33	-8	117	-114
10	286	274	-8	332	343	0 0 10	56	65	-29	22	16	-9	113	128
12	270	-256	-10	226	242	2	60	75	-30	89	95	-10	63	-57
14	196	172	-12	144	-136	4	123	-122	-31	22	22	-11	69	91
16	321	300	-14	53	-52	6	60	87	-32	12	49	-12	115	-114
18	141	-127	-16	57	42	-2 0 10	32	-43	0 1 2	119	113	-13	40	-36
20	24	-1	-18	196	-179	-4	98	100	1	367	346	-14	122	129
22	215	-203	-20	50	44	-6	35	-38	2	518	514	-15	89	69
24	67	65	-22	63	55	-8	0	4	3	178	164	-16	121	115
26	141	-144	-24	51	-50	-10	54	-64	4	726	-774	-17	77	-76
28	19	-15	-26	64	-78	-12	42	-51	5	370	344	-18	71	-64
30	89	113	-28	26	-9	-14	44	47	6	68	60	-19	84	-89
-2 0 2	510	-527	-30	14	20	-16	22	-37	7	73	64	-20	94	90
-4	206	190	0 0 6	31	39	1 1 0	172	159	8	376	-370	-21	52	-34
-6	275	-273	2	140	-153	2	145	126	9	288	-284	-22	0	-5
-8	154	-148	4	98	-103	3	159	155	10	81	-80	-23	42	37
-10	972	922	6	348	355	4	292	-290	11	408	-404	-24	89	-85
-12	205	-192	8	172	-183	5	164	-154	12	39	38	-25	0	-6
-14	220	209	10	246	240	6	591	567	13	0	4	-26	39	33
-16	152	-144	12	170	-165	7	187	-185	14	174	-151	-27	25	18
-18	235	-236	14	238	-218	8	492	-498	15	0	9	-28	19	9
-20	95	87	16	102	100	9	99	-93	16	475	442	-29	0	16
-22	53	-62	18	60	-54	10	330	323	17	106	102	-30	68	-69
-24	212	207	20	117	117	11	181	177	18	230	-226	-31	51	-56
-26	45	-50	22	29	-31	12	187	190	19	64	65	0 1 4	190	-186
-28	58	63	-2 0 6	177	-177	13	43	47	20	0	-2	1	95	-101
-30	23	-7	-4	249	272	14	528	-510	21	28	-11	2	215	233
-32	74	-90	-6	73	-78	15	276	264	22	81	85	3	174	174
0 0 3	29	-10	-8	171	164	16	93	97	23	76	-76	4	332	-352
2	55	72	-10	145	143	17	86	93	24	131	-129	5	125	120
4	69	-34	-12	334	-330	18	291	-280	25	46	-41	6	350	357
6	42	32	-14	71	71	19	135	-138	26	99	102	7	45	40
8	30	27	-16	188	-185	20	75	72	27	23	20	8	271	-259
10	87	-99	-18	161	146	21	190	-181	28	14	-17	9	100	-83
12	62	-61	-20	147	125	22	0	1	29	23	21	10	126	-116
14	62	-51	-22	75	-77	23	0	-5	-1 1 2	51	44	11	0	-1
16	38	21	-24	71	84	24	44	-47	-2	20	8	12	439	421
18	73	65	-26	131	-128	25	46	41	-3	349	-332	13	223	-199
20	60	57	-28	19	29	26	111	120	-4	525	509	14	153	-142
22	0	-4	0 0 7	88	85	27	65	65	-5	208	-203	15	45	-44
24	0	2	2	67	55	28	111	-120	-6	619	-620	16	220	202
26	41	-33	4	36	-32	29	40	43	-7	349	-318	17	101	96
28	25	25	6	50	-40	30	15	8	-8	67	53	18	52	-43
-2 0 3	137	-177	8	52	59	31	41	-47	-9	17	-12	19	142	131
-4	54	-111	10	41	-30	0 1 1	59	59	-10	388	-368	20	53	-40
-6	55	50	12	51	-51	1	111	133	-11	200	200	21	0	22
-8	117	127	14	48	32	2	42	-34	-12	23	17	22	0	7
-10	71	96	16	29	-28	3	66	103	-13	357	339	23	0	-3
-12	48	57	18	51	-59	4	141	-147	-14	57	67	24	92	-94
-14	275	-263	20	24	17	5	21	-21	-15	47	-42	25	59	-55
-16	439	-437	-2 0 7	37	20	6	105	-120	-16	218	-213	26	80	86
-18	177	-180	-4	53	64	7	92	-111	-17	98	-96	27	46	-53

Table 3. *(Continued)*

h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c
-1 1 4	202	-208	15 1 6	74	- 64	-1 1 8	26	35	18 2 0	44	50	26 2 2	29	28
-2	34	25	16	122	116	-2	117	121	19	98	-104	27	44	47
-3	101	- 89	17	56	- 53	-3	113	117	20	237	-235	28	35	40
-4	103	100	18	38	- 32	-4	0	- 20	21	55	61	29	16	- 5
-5	77	- 67	19	25	- 24	-5	64	62	22	158	163	-1 2 2	343	357
-6	341	-361	20	27	- 26	-6	76	- 77	23	45	- 37	-2	408	399
-7	174	173	21	36	30	-7	0	9	24	91	-101	-3	111	- 87
-8	400	412	22	89	98	-8	188	203	25	0	- 19	-4	775	-791
-9	190	185	-1 1 6	164	-155	-9	144	-141	26	59	60	-5	437	-418
-10	254	-240	-2	74	- 82	-10	157	-159	27	44	49	-6	81	- 65
-11	81	81	-3	0	- 19	-11	61	- 58	28	20	13	-7	86	- 93
-12	192	187	-4	0	- 16	-12	102	105	29	16	- 19	-8	173	169
-13	137	-128	-5	171	173	-13	0	- 9	30	15	- 8	-9	55	- 67
-14	194	183	-6	266	-265	-14	72	- 77	31	45	- 59	-10	271	-269
-15	70	- 70	-7	140	137	-15	38	32	0 2 1	43	59	-11	40	- 36
-16	246	-242	-8	317	330	-16	93	- 87	1	24	5	-12	441	448
-17	150	-146	-9	102	89	-17	30	18	2	37	- 2	-13	170	170
-18	95	98	-10	144	-141	-18	89	91	3	142	-161	-14	208	-204
-19	0	- 12	-11	104	-106	-19	21	22	4	22	21	-15	117	130
-20	170	-178	-12	127	125	-20	95	-107	5	143	-173	-16	29	29
-21	64	61	-13	73	- 79	-21	45	- 47	6	32	1	-17	42	35
-22	90	91	-14	42	- 39	-22	33	36	7	74	- 89	-18	45	- 32
-23	88	82	-15	112	-100	-23	24	- 11	8	47	30	-19	54	- 49
-24	0	13	-16	161	-162	-24	33	- 41	9	129	133	-20	106	-118
-25	0	- 2	-17	114	99	-25	21	- 2	10	0	- 11	-21	93	- 96
-26	47	- 50	-18	90	91	0 1 9	25	- 23	11	96	105	-22	72	80
-27	61	- 53	-19	89	89	1	54	54	12	67	- 71	-23	76	- 84
-28	52	58	-20	128	-124	2	0	24	13	55	72	-24	82	- 92
-29	55	- 46	-21	52	50	3	0	15	14	35	37	-25	32	- 27
-30	67	- 77	-22	117	121	4	0	3	15	0	8	-26	82	89
-31	20	- 15	-23	0	7	5	46	- 47	16	27	28	-27	81	74
0 1 5	64	- 62	-24	49	38	6	42	- 34	17	176	-169	-28	0	- 5
1	35	- 45	-25	56	- 49	7	0	- 14	18	45	37	-29	37	36
2	111	112	-26	40	40	8	26	22	19	190	-178	-30	50	- 57
3	54	- 44	-27	54	- 51	9	0	1	20	37	- 32	-31	16	- 19
4	0	2	-28	27	34	10	27	22	21	36	- 27	0 2 3	63	55
5	0	7	-29	21	- 20	11	0	4	22	0	8	1	195	208
6	0	- 11	0 1 7	56	- 52	12	34	- 37	23	56	58	2	0	19
7	131	145	1	103	-118	-1 1 9	0	18	24	22	9	3	61	75
8	0	17	2	44	- 49	-2	30	32	25	71	72	4	40	55
9	54	54	3	76	- 96	-3	0	2	26	22	- 3	5	100	- 91
10	69	- 66	4	0	6	-4	83	87	27	85	87	6	0	13
11	47	- 44	5	67	- 63	-5	30	- 25	28	0	- 3	7	46	- 54
12	35	- 34	6	109	95	-6	68	- 74	29	29	25	8	81	- 78
13	61	- 58	7	0	0	-7	84	- 93	30	31	31	9	96	- 87
14	0	14	8	0	17	-8	47	- 50	-1 2 1	230	242	10	41	19
15	0	- 8	9	97	101	-9	78	- 81	-2	0	6	11	0	13
16	0	- 8	10	86	- 75	-10	29	26	-3	178	204	12	0	- 3
17	0	5	11	55	52	-11	23	18	-4	31	- 37	13	110	106
18	0	19	12	39	- 38	-12	120	-125	-5	55	66	14	0	- 19
19	71	69	13	28	- 21	-13	84	86	-6	34	3	15	83	84
20	32	- 27	14	45	- 41	-14	41	- 47	-7	58	- 64	16	0	11
21	0	13	15	59	- 58	-15	0	- 1	-8	19	7	17	52	49
22	44	- 38	16	70	- 70	-16	39	44	-9	218	-231	18	26	22
23	0	- 2	17	29	- 24	-17	70	75	-10	94	91	19	25	25
24	0	7	18	37	34	-18	55	57	-11	266	-272	20	78	67
25	0	4	19	22	- 14	-19	0	10	-12	120	135	21	40	- 38
-1 1 5	0	- 37	20	30	30	-20	42	41	-13	67	- 66	22	33	- 40
-2	99	-108	-1 1 7	80	72	-21	26	- 34	-14	39	52	23	21	- 14
-3	125	96	-2	166	-186	-22	11	- 9	-15	106	117	24	21	- 18
-4	196	197	-3	158	162	0 1 10	88	-107	-16	128	-133	25	36	31
-5	199	194	-4	128	-131	1	60	64	-17	58	61	26	70	- 73
-6	67	52	-5	0	- 7	2	55	68	-18	78	- 78	27	18	7
-7	86	106	-6	0	5	3	24	- 27	-19	48	- 44	28	16	- 9
-8	0	12	-7	53	57	4	38	- 40	-20	39	- 37	-1 2 3	28	35
-9	98	- 77	-8	95	95	5	40	- 44	-21	73	70	-2	0	- 11
-10	331	333	-9	34	41	6	19	- 18	-22	114	-107	-3	39	- 38
-11	177	-161	-10	44	34	-1 1 10	74	70	-23	0	- 5	-4	0	- 37
-12	50	49	-11	52	- 58	-2	125	141	-24	71	77	-5	31	- 30
-13	0	- 3	-12	0	- 24	-3	38	37	-25	69	- 65	-6	62	- 80
-14	108	-108	-13	71	- 74	-4	0	10	-26	0	- 2	-7	157	-166
-15	143	-137	-14	137	131	-5	19	- 22	-27	67	64	-8	105	103
-16	40	- 45	-15	0	36	-6	36	30	-28	56	52	-9	38	14
-17	0	- 19	-16	63	- 56	-7	75	- 80	-29	22	19	-10	128	128
-18	117	-105	-17	28	- 11	-8	87	94	-30	13	- 5	-11	370	364
-19	67	73	-18	110	- 98	-9	31	- 31	-31	18	- 24	-12	69	73
-20	0	- 19	-19	38	37	-10	136	-167	0 2 2	365	-366	-13	85	92
-21	101	96	-20	83	71	-11	0	5	1	131	-116	-14	63	58
-22	43	48	-21	41	31	-12	72	88	2	738	753	-15	0	3
-23	71	68	-22	47	- 41	-13	63	74	3	312	-299	-16	54	- 30
-24	146	141	-23	34	20	-14	0	- 9	4	114	93	-17	49	40
-25	74	71	-24	0	- 9	-15	32	34	5	0	13	-18	38	28
-26	56	56	-25	36	- 39	-16	14	18	6	102	- 98	-19	221	-220
-27	59	- 61	-26	31	29	-17	16	13	7	73	- 84	-20	42	- 34
-28	0	18	-27	0	- 7	0 2 0	479	-468	8	231	229	-21	233	-231
-29	101	-112	0 1 8	167	-171	1	0	- 13	9	46	- 45	-22	82	88
-30	42	47	1	116	-112	2	447	462	10	503	-509	-23	46	- 40
0 1 6	302	-309	2	146	149	3	326	-330	11	255	250	-24	23	19
1	136	-132	3	92	- 90	4	246	-231	12	216	210	-25	64	67
2	382	412	4	92	- 98	5	563	-560	13	83	86	-26	54	- 58
3	162	-167	5	37	- 26	6	547	-520	14	34	- 34	-27	65	63
4	124	-125	6	118	117	7	241	-211	15	45	- 39	-28	31	16
5	32	24	7	49	43	8	193	211	16	0	- 4	-29	45	49
6	149	151	8	80	72	9	258	269	17	0	- 11	-30	40	36
7	163	161	9	0	1	10	326	-322	18	141	140	-31	28	30
8	72	73	10	136	-147	11	151	158	19	121	-116	0 2 4	376	-376
9	192	189	11	95	87	12	395	404	20	46	- 43	1	292	290
10	87	- 78	12	87	90	13	42	50	21	126	-131	2	138	142
11	30	- 30	13	0	- 12	14	121	-121	22	109	117	3	354	373
12	56	52	14	98	- 92	15	95	94	23	0	- 8	4	322	305
13	0	- 2	15	21	- 25	16	36	19	24	69	- 68	5	67	60
14	246	-246	16	42	48	17	166	-177	25	106	105	6	186	-196

Table 3. *(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
7 2 4	147	140	-28 2 5	36	33	1 2 8	135	-135	9 3 0	426	-433	20 3 2	119	-119
8	306	286	-29	41	-43	2	69	-59	10	123	137	21	33	-22
9	165	-151	-30	37	-40	3	0	-7	11	223	-238	22	74	-79
10	321	-311	0 2 6	321	-318	4	102	112	12	97	107	23	32	-28
11	155	-151	1	38	26	5	42	42	13	59	59	24	96	101
12	64	64	2	36	-14	6	176	-177	14	171	190	25	0	4
13	53	-52	3	38	26	7	79	78	15	74	74	26	27	-40
14	55	-45	4	38	17	8	172	170	16	318	-345	27	38	36
15	164	150	5	43	37	9	103	108	17	85	-91	28	60	75
16	0	-20	6	118	-119	10	27	-28	18	82	94	-1 3 2	24	-23
17	68	61	7	0	6	11	0	5	19	66	102	-2	371	376
18	209	199	8	316	320	12	40	39	20	106	-119	-3	199	208
19	42	-31	9	121	-120	13	57	-55	21	36	38	-4	195	-200
20	154	-149	10	107	-105	14	35	36	22	42	41	-5	32	5
21	79	74	11	45	-41	15	0	2	23	63	-64	-6	270	249
22	110	108	12	87	83	16	92	-112	24	129	142	-7	0	8
23	23	28	13	0	10	-1 2 8	57	-63	25	38	37	-8	509	-519
24	56	-49	14	24	8	-2	151	152	26	26	-33	-9	182	178
25	65	-65	15	35	-26	-3	38	32	27	0	6	-10	107	101
26	23	-24	16	133	-127	-4	100	-105	28	52	66	-11	90	-89
-1 2 4	30	-20	17	78	76	-5	0	-1	29	32	-33	-12	27	33
-2	296	289	18	61	54	-6	140	136	30	41	-59	-13	73	-74
-3	0	-13	19	91	96	-7	95	92	0 3 1	122	132	-14	55	-54
-4	305	-283	20	51	-56	-8	0	-5	1	129	-116	-15	23	-8
-5	136	-139	21	21	-17	-9	49	50	2	48	-57	-16	343	356
-6	121	122	22	39	40	-10	23	-18	3	65	-63	-17	23	21
-7	293	-276	-1 2 6	0	-20	-11	35	-37	4	239	-270	-18	168	-175
-8	235	216	-2	239	240	-12	58	62	5	35	36	-19	119	-123
-9	129	-106	-3	171	-160	-13	72	-70	6	109	-130	-20	32	33
-10	138	-126	-4	257	-255	-14	129	-139	7	96	89	-21	0	-11
-11	195	193	-5	74	-72	-15	99	-99	8	23	-14	-22	109	-111
-12	275	271	-6	331	328	-16	133	130	9	65	63	-23	23	15
-13	141	139	-7	172	150	-17	28	-24	10	132	142	-24	48	-56
-14	325	-308	-8	0	19	-18	18	-13	11	103	104	-25	0	12
-15	0	-6	-9	76	87	-19	69	75	12	137	147	-26	106	111
-16	96	83	-10	90	-86	-20	24	33	13	34	32	-27	47	-44
-17	27	-23	-11	43	30	-21	84	87	14	36	42	-28	15	-22
-18	0	-19	-12	117	110	-22	0	1	15	37	38	-29	51	-51
-19	137	-130	-13	0	16	-23	0	4	16	0	-24	-30	62	75
-20	54	-58	-14	258	-254	-24	79	-94	17	56	-63	0 3 3	125	134
-21	43	-40	-15	57	-55	0 2 9	16	14	18	107	-120	1	46	-33
-22	183	178	-16	121	120	1	78	-83	19	30	21	2	94	106
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-26	44	59	-20	59	62	5	17	6	23	81	-86	6	109	-116
-27	65	68	-21	0	2	6	33	26	24	41	44	7	27	1
-28	63	-73	-22	85	93	7	83	86	25	0	7	8	166	-170
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1	113	120	-26	0	8	11	0	2	29	0	2	12	32	29
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3	130	135	-28	58	-75	-2	31	-23	-2	157	186	14	86	79
4	115	-112	0 2 7	40	-45	-3	32	-30	-3	36	39	15	44	-33
5	143	152	1	48	-46	-4	0	11	-4	54	69	16	78	80
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7	24	21	3	74	75	-6	24	-20	-6	0	-7	18	43	-44
8	68	-53	4	0	-12	-7	116	124	-7	28	-39	19	95	-89
9	150	-147	5	85	-76	-8	41	-47	-8	84	-103	20	73	-71
10	0	-5	6	29	30	-9	25	18	-9	0	-8	21	18	2
11	105	-100	7	0	-12	-10	28	-21	-10	142	-177	22	35	-29
12	0	6	8	49	-44	-11	39	-35	-11	79	-86	23	40	43
13	0	14	9	37	23	-12	38	40	-12	30	30	24	24	-23
14	22	-16	10	24	8	-13	32	-23	-13	119	-121	25	24	23
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18	31	-19	14	0	5	-17	30	-31	-17	153	164	-3	63	35
19	59	53	15	0	-8	-18	40	41	-18	103	106	-4	150	-135
20	25	-18	16	0	10	-19	51	58	-19	33	29	-5	411	354
21	36	-41	17	0	1	-20	29	-34	-20	51	-53	-6	266	-264
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23	43	-45	19	19	22	0 2 10	32	-36	-22	77	-81	-8	75	-74
24	10	-6	-1 2 7	149	-152	1	45	-45	-23	0	5	-9	223	-195
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-4	0	-8	-5	99	96	-1 2 10	0	6	-27	84	-87	-13	58	-42
-5	212	-215	-6	23	-14	-2	93	100	-28	28	30	-14	116	119
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-7	43	-41	-8	45	54	-4	108	-118	-30	23	29	-16	30	30
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-9	59	62	-10	51	-51	-6	85	100	1	229	-244	-18	0	1
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-12	100	-90	-13	244	-238	-9	20	-18	4	271	260	-21	37	-31
-13	201	196	-14	0	-26	-10	15	-4	5	65	-73	-22	36	-29
-14	132	-132	-15	103	-101	-11	0	-2	6	546	-541	-23	34	40
-15	127	119	-16	21	-14	-12	18	19	7	229	-222	-24	32	-28
-16	87	-76	-17	0	8	-13	19	-13	8	98	91	-25	143	141
-17	106	-100	-18	29	23	-14	0	-10	9	241	249	-26	58	59
-18	144	134	-19	28	-24	-15	10	-15	10	0	23	-27	41	44
-19	34	25	-20	55	-47	-16	85	113	11	91	94	-28	76	81
-20	97	98	-21	88	80	1 3 0	106	97	12	76	-68	-29	0	-9
-21	138	137	-22	38	31	2	410	-411	13	45	-24	-30	0	-5
-22	0	5	-23	119	125	3	55	53	14	380	387	0 3 4	301	309
-23	45	-41	-24	28	-41	4	233	222	15	55	62	1	0	-11
-24	74	68	-25	47	47	5	74	-63	16	126	-137	2	169	-158
-25	26	-23	-26	21	-23	6	520	-537	17	178	181	3	0	-12
-26	44	-48	-27	45	-57	7	0	33	18	78	81	4	412	415
-27	74	71	0 2 8	44	-46	8	363	384	19	38	-20	5	55	50

Table 3. *(Continued)*

h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c
6 3 4	261	-239	0 3 6	143	134	9 3 8	34	34	28 4 0	27	-37	-15 4 2	138	140
7	347	312	1	50	-38	10	77	74	29	17	-21	-16	43	-32
8	73	-65	2	140	-138	11	0	-6	0 4 1	167	-169	-17	51	55
9	69	69	3	77	85	12	124	-131	1	70	80	-18	80	79
10	80	-75	4	322	331	13	0	10	2	179	-192	-19	34	29
11	71	-62	5	115	113	14	54	49	3	25	-34	-20	157	166
12	168	-161	6	63	-58	-1 3 8	41	37	4	60	-61	-21	55	-60
13	53	-37	7	123	-124	-2	160	-150	5	102	-122	-22	151	-162
14	268	262	8	24	-17	-3	62	54	6	40	45	-23	40	-44
15	0	5	9	0	-17	-4	29	17	7	95	-109	-24	84	98
16	92	-89	10	54	57	-5	49	43	8	103	109	-25	33	27
17	135	-142	11	0	-6	-6	169	175	9	35	28	-26	73	-81
18	106	104	12	243	-234	-7	0	2	10	74	81	-27	15	6
19	23	-15	13	0	-16	-8	68	-70	11	91	100	-28	0	-12
20	31	-12	14	83	82	-9	0	-4	12	0	-5	-29	21	22
21	52	47	15	79	-73	-10	169	169	13	63	65	0 4 3	0	13
22	95	-108	16	67	-68	-11	29	30	14	140	-147	1	35	43
23	22	-22	17	33	49	-12	144	-152	15	0	-7	2	78	-62
24	28	29	18	82	78	-13	0	-11	16	48	-51	3	32	-25
25	20	19	19	14	12	-14	48	-42	17	51	51	4	128	-125
-1 3 4	24	-12	20	56	56	-15	120	117	18	45	48	5	44	-45
-2	0	-17	21	14	18	-16	0	2	19	32	-30	6	111	-114
-3	184	-163	-1 3 6	0	18	-17	0	-7	20	0	6	7	97	-97
-4	372	-341	-2	204	-201	-18	28	-30	21	49	-45	8	89	75
-5	80	-70	-3	0	13	-19	0	-3	22	101	102	9	114	-122
-6	160	150	-4	175	-166	-20	117	127	23	0	15	10	145	134
-7	78	61	-5	26	16	-21	0	13	24	0	13	11	32	-17
-8	400	-382	-6	158	154	-22	24	-40	25	22	24	12	98	94
-9	91	-80	-7	41	-43	-23	28	-33	26	11	13	13	39	47
-10	320	313	-8	121	-112	0 3 9	44	-49	27	16	10	14	0	5
-11	208	189	-9	0	3	1	19	20	28	41	-48	15	102	102
-12	131	124	-10	306	300	2	40	-42	-1 4 1	66	76	16	0	-13
-13	52	48	-11	67	-54	3	0	-10	-2	34	33	17	0	-7
-14	68	59	-12	86	-80	4	26	-26	-3	80	97	18	68	-69
-15	0	-6	-13	0	2	5	23	20	-4	71	69	19	81	-86
-16	116	107	-14	50	-44	6	13	13	-5	0	11	20	0	0
-17	45	-33	-15	59	42	7	51	-49	-6	214	227	21	62	-60
-18	274	-264	-16	30	29	8	42	45	-7	137	-149	22	27	22
-19	77	64	-17	0	-6	9	18	-23	-8	110	106	23	69	-67
-20	73	72	-18	192	-189	10	17	18	-9	22	-28	24	12	1
-21	42	29	-19	80	-82	-1 3 9	55	64	-10	84	-90	25	25	-21
-22	86	-90	-20	130	128	-2	60	60	-11	33	-17	26	0	-1
-23	21	-10	-21	26	-19	-3	0	9	-12	26	-23	-1 4 3	145	151
-24	41	38	-22	20	9	-4	47	48	-13	97	102	-2	137	128
-25	20	10	-23	67	-58	-5	0	0	-14	89	-90	-3	28	29
-26	96	99	-24	64	67	-6	56	52	-15	141	147	-4	39	40
-27	0	6	-25	22	20	-7	0	19	-16	87	-92	-5	192	-192
-28	23	-34	-26	24	24	-8	24	19	-17	132	145	-6	43	-49
-29	0	6	-27	17	24	-9	98	93	-18	47	-49	-7	124	-119
0 3 5	66	-59	0 3 7	67	-69	-10	37	-34	-19	60	56	-8	56	-63
1	146	-145	1	192	176	-11	55	-61	-20	80	82	-9	99	-98
2	118	124	2	0	-26	-12	32	-32	-21	147	-158	-10	36	-32
3	142	-127	3	0	-17	-13	53	-53	-22	0	3	-11	104	-96
4	89	97	4	54	53	-14	82	-78	-23	89	-91	-12	30	-32
5	0	26	5	27	-19	-15	24	25	-24	34	-20	-13	162	154
6	34	32	6	84	87	-16	23	24	-25	18	-8	-14	0	-7
7	53	-50	7	0	7	-17	28	-28	-26	0	1	-15	125	111
8	74	-73	8	65	63	-18	44	52	-27	39	-35	-16	183	176
9	25	-27	9	56	-42	0 3 10	43	55	-28	0	0	-17	99	-84
10	102	-101	10	0	3	1	31	-38	-29	29	30	-18	56	58
11	39	37	11	47	-43	2	90	-135	0 4 2	453	453	-19	69	64
12	0	-29	12	41	-36	-1 3 10	0	10	1	320	331	-20	67	-64
13	95	-91	13	33	26	-2	40	-52	2	31	-48	-21	0	19
14	28	21	14	52	-48	-3	21	25	3	142	140	-22	31	28
15	28	-22	15	18	14	-4	70	78	4	39	-42	-23	21	-16
16	81	79	16	14	14	-5	17	12	5	295	-306	-24	111	-104
17	18	20	17	33	34	-6	40	37	6	191	189	-25	27	29
18	41	37	18	36	41	-7	30	-33	7	162	-160	-26	36	-29
19	16	-5	-1 3 7	78	59	-8	71	-84	8	210	-203	-27	14	22
20	32	39	-2	54	-55	-9	17	16	9	0	-12	-28	0	-19
21	13	-11	-3	84	-80	-10	20	31	10	250	266	-29	29	29
22	25	-23	-4	43	-39	-11	0	4	11	62	-58	0 4 4	289	304
23	0	1	-5	35	32	-12	84	-115	12	201	-204	1	58	50
-1 3 5	45	33	-6	87	84	-13	16	21	13	159	161	2	247	-236
-2	110	-109	-7	198	-194	0 4 0	211	185	14	47	42	3	27	-27
-3	0	-15	-8	138	135	1	104	-110	15	123	128	4	0	-2
-4	130	-130	-9	237	-229	2	417	-417	16	85	-91	5	0	14
-5	45	-53	-10	0	5	3	144	-151	17	0	7	6	78	59
-6	47	-27	-11	106	93	4	280	290	18	152	-165	7	108	-102
-7	166	-133	-12	0	4	5	52	58	19	23	-22	8	314	-320
-8	56	45	-13	32	30	6	56	-42	20	198	210	9	113	-109
-9	63	71	-14	85	-78	7	134	143	21	0	-3	10	218	207
-10	65	58	-15	0	11	8	134	-107	22	82	-92	11	32	-2
-11	95	80	-16	76	-65	9	69	77	23	0	-10	12	114	-103
-12	143	131	-17	29	-11	10	398	436	24	82.	92	13	104	93
-13	0	11	-18	0	-1	11	135	149	25	32	-34	14	53	51
-14	56	-49	-19	55	59	12	208	-240	26	24	31	15	0	18
-15	142	123	-20	0	2	13	88	80	27	0	1	16	124	124
-16	117	-106	-21	27	-19	14	100	101	-1 4 2	220	215	17	0	14
-17	41	43	-22	84	78	15	157	-160	-2	365	-362	18	97	-98
-18	103	-97	-23	0	-6	16	81	-86	-3	214	218	19	44	38
-19	213	-196	-24	11	10	17	0	-12	-4	48	67	20	18	9
-20	111	-94	-25	0	-3	18	93	-105	-5	188	171	21	99	-101
-21	34	-32	0 3 8	69	69	19	34	35	-6	71	73	22	61	-58
-22	46	44	1	0	11	20	123	132	-7	247	-242	23	53	-61
-23	0	2	2	201	-209	21	44	-47	-8	171	-169	24	43	54
-24	51	56	3	0	-11	22	27	-33	-9	154	-158	-1 4 4	85	76
-25	21	-32	4	92	95	23	99	102	-10	130	125	-2	191	-158
-26	37	37	5	94	-97	24	59	64	-11	0	12	-3	92	-81
-27	56	57	6	29	24	25	70	83	-12	292	-301	-4	334	331
-28	27	21	7	44	38	26	66	-79	-13	0	17	-5	151	-141
-29	54	74	8	42	32	27	23	0	-14	181	191	-6	236	-222

Table 3. *(Continued)*

h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c	h k l	$\lvert F_o \rvert$	F_c
-7 4 4	22	-20	-10 4 6	131	117	-2 4 9	78	-88	5 5 2	61	36	1 5 4	61	59
-8	0	-18	-11	119	108	-3	80	83	6	300	317	2	400	418
-9	140	122	-12	74	-73	-4	64	-62	7	60	63	3	63	-61
-10	162	136	-13	0	4	-5	64	63	8	197	-201	4	153	-158
-11	42	45	-14	189	182	-6	32	30	9	74	74	5	121	-132
-12	265	-246	-15	47	-40	-7	39	-44	10	22	27	6	58	56
-13	58	54	-16	99	-99	-8	26	-27	11	51	-42	7	54	-53
-14	218	209	-17	107	-100	-9	18	-18	12	231	274	8	29	-16
-15	59	52	-18	0	16	-10	65	64	13	36	-30	9	41	-43
-16	76	-70	-19	0	-9	-11	67	-66	14	125	-136	10	134	-124
-17	96	-74	-20	0	-2	-12	33	36	15	111	-124	11	76	76
-18	64	51	-21	25	28	-13	35	-39	16	47	45	12	65	-52
-19	40	-37	-22	63	-58	-14	24	-23	17	49	-42	13	29	-7
-20	46	36	-23	74	71	-15	11	5	18	78	-81	14	160	-164
-21	0	-6	-24	63	71	-16	21	24	19	0	-6	15	37	36
-22	115	-114	-25	32	35	-17	59	79	20	0	-7	16	179	174
-23	0	1	-26	56	-86	1 5 0	117	-140	21	0	-7	17	26	-17
-24	26	28	0 4 7	0	-7	2	146	149	22	35	-37	18	25	-32
-25	51	48	1	0	1	3	38	29	23	16	14	19	19	-12
-26	45	-50	2	41	30	4	627	-566	24	0	-12	20	24	21
-27	11	15	3	37	-30	5	191	196	25	0	10	21	43	-39
-28	40	48	4	104	96	6	105	115	-1 5 2	0	14	22	49	59
0 4 5	222	208	5	131	123	7	139	157	-2	60	-66	23	5	18
1	34	-19	6	38	-30	8	84	-89	-3	14	24	-1 5 4	71	75
2	77	70	7	101	103	9	49	54	-4	155	151	-2	29	-13
3	46	54	8	86	-87	10	41	31	-5	140	154	-3	134	133
4	81	-69	9	0	3	11	55	55	-6	308	-314	-4	101	87
5	36	33	10	36	-32	12	114	119	-7	29	-27	-5	38	-33
6	124	-105	11	27	25	13	91	-108	-8	303	306	-6	355	-340
7	83	-73	12	31	28	14	35	15	-9	30	-43	-7	144	-135
8	102	-89	13	52	-49	15	29	-25	-10	181	-181	-8	108	103
9	47	28	14	26	22	16	193	212	-11	136	-139	-9	101	-99
10	33	-31	15	19	-12	17	0	6	-12	0	8	-10	100	-98
11	0	4	16	40	39	18	173	-189	-13	75	67	-11	31	-31
12	108	98	-1 4 7	0	-7	19	53	49	-14	271	277	-12	61	56
13	46	-44	-2	0	-12	20	32	37	-15	44	50	-13	0	-9
14	77	74	-3	103	-106	21	0	3	-16	82	-87	-14	40	35
15	28	24	-4	48	-39	22	26	30	-17	74	77	-15	76	79
16	20	-9	-5	34	-29	23	70	69	-18	63	70	-16	97	-101
17	36	39	-6	0	-8	24	33	-35	-19	46	40	-17	83	74
18	20	-17	-7	148	140	25	0	-6	-20	81	-90	-18	124	128
19	0	10	-8	39	43	26	48	62	-21	16	21	-19	43	33
20	60	-59	-9	101	92	27	27	-29	-22	30	31	-20	129	-134
21	26	30	-10	81	69	0 5 1	35	35	-23	0	2	-21	91	-93
22	10	14	-11	0	15	1	186	-175	-24	0	10	-22	35	38
-1 4 5	140	-129	-12	28	14	2	0	20	-25	43	-38	-23	27	-26
-2	137	109	-13	65	55	3	43	-43	-26	14	-18	-24	38	36
-3	80	-75	-14	30	25	4	0	-1	-27	0	-4	-25	47	-40
-4	45	-46	-15	67	-52	5	41	-37	-28	36	54	-26	9	-11
-5	0	-6	-16	33	26	6	37	-35	0 5 3	52	-40	-27	0	-9
-6	28	23	-17	79	-64	7	140	153	1	70	-70	0 5 5	41	27
-7	0	-6	-18	131	-123	8	78	81	2	0	0	1	80	72
-8	180	-162	-19	52	55	9	141	148	3	130	-135	2	27	-25
-9	144	121	-20	47	-49	10	47	52	4	60	54	3	34	34
-10	90	-81	-21	0	2	11	24	20	5	128	-128	4	104	99
-11	58	59	-22	47	48	12	0	5	6	63	-68	5	117	-106
-12	43	40	-23	0	-11	13	84	-90	7	0	-7	6	54	48
-13	34	-27	-24	17	-25	14	0	-1	8	0	1	7	23	-17
-14	84	-67	0 4 8	106	105	15	110	-118	9	67	65	8	55	-43
-15	207	-185	1	0	13	16	43	-48	10	22	-26	9	0	1
-16	39	28	2	32	32	17	96	-98	11	51	45	10	25	-13
-17	136	-120	3	50	-51	18	58	-64	12	32	-36	11	24	22
-18	57	59	4	44	-36	19	38	33	13	76	78	12	36	-25
-19	81	-83	5	41	39	20	29	-28	14	53	46	13	99	94
-20	56	63	6	107	117	21	66	72	15	28	-25	14	0	4
-21	108	-102	7	39	33	22	22	24	16	23	-27	15	103	105
-22	78	-72	8	109	-112	23	68	65	17	0	-4	16	26	25
-23	112	118	9	0	-18	24	17	15	18	30	28	17	14	-6
-24	24	-14	10	59	60	25	17	-14	19	14	-17	18	16	15
-25	74	75	11	15	-20	26	15	21	20	15	-11	19	29	-26
-26	52	45	12	11	-10	-1 5 1	215	-229	21	27	-28	20	19	16
-27	26	-31	13	31	-34	-2	118	132	22	18	-12	-1 5 5	128	122
0 4 6	86	92	-1 4 8	58	-50	-3	19	18	23	26	26	-2	34	-23
1	0	-2	-2	175	-182	-4	29	-15	24	0	5	-3	241	207
2	55	53	-3	31	-32	-5	197	205	-1 5 3	56	58	-4	190	-181
3	132	120	-4	86	85	-6	0	-4	-2	108	-101	-5	24	-8
4	133	-131	-5	41	43	-7	150	155	-3	217	206	-6	0	5
5	135	123	-6	137	-143	-8	0	6	-4	0	0	-7	146	-130
6	163	150	-7	0	13	-9	37	34	-5	25	-18	-8	36	40
7	80	73	-8	146	141	-10	22	-25	-6	20	13	-9	124	-113
8	180	-171	-9	67	62	-11	127	-128	-7	35	-36	-10	72	-68
9	40	34	-10	47	49	-12	101	-112	-8	0	-6	-11	66	-53
10	132	123	-11	0	-5	-13	45	-51	-9	128	-124	-12	31	-24
11	103	-88	-12	73	-77	-14	0	1	-10	161	146	-13	186	164
12	27	-18	-13	39	-47	-15	85	-85	-11	228	-219	-14	117	115
13	90	-83	-14	70	74	-16	35	24	-12	117	117	-15	35	26
14	31	35	-15	0	10	-17	48	50	-13	54	-57	-16	0	14
15	0	4	-16	129	-139	-18	62	-61	-14	0	6	-17	36	28
16	65	68	-17	24	22	-19	63	59	-15	124	129	-18	35	-29
17	0	3	-18	28	39	-20	107	110	-16	53	-54	-19	39	-32
18	125	-139	-19	18	6	-21	28	31	-17	101	95	-20	58	52
19	12	-5	-20	16	-10	-22	17	12	-18	26	-25	-21	61	-55
20	31	82	-21	23	26	-23	0	-4	-19	30	23	-22	72	-66
-1 4 6	155	-139	0 4 9	0	-5	-24	25	-21	-20	83	-91	-23	29	-34
-2	264	-250	1	0	-2	-25	37	-31	-21	87	-80	-24	15	-11
-3	132	-123	2	50	50	-26	11	19	-22	86	-91	-25	15	19
-4	157	155	3	0	-4	-27	0	-2	-23	73	-79	-26	0	2
-5	0	20	4	37	35	0 5 2	104	-116	-24	47	53	0 5 6	110	-101
-6	136	-125	5	0	11	1	0	-4	-25	60	-60	1	58	60
-7	45	-30	6	29	31	2	155	151	-26	15	15	2	120	120
-8	87	-64	7	25	-28	3	175	-188	-27	19	16	3	45	-34
-9	115	97	-1 4 9	25	-19	4	210	-216	0 5 4	72	-73	4	143	-147

Table 3. *(Continued)*

h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c
5 5 6	0	12	-17 5 8	36	-43	6 6 2	155	-169	11 6 4	33	29	-11 6 6	0	-10
6	160	152	-18	55	71	7	79	-68	12	148	151	-12	169	164
7	48	-46	-19	48	-64	8	92	95	13	26	-8	-13	25	-14
8	38	-41	0 5 9	13	13	9	44	-39	14	51	47	-14	56	-63
9	35	-35	1	0	-2	10	22	-20	15	0	-5	-15	17	1
10	124	-130	2	54	-63	11	0	-1	16	115	-124	-16	63	73
11	0	-2	3	39	45	12	233	232	17	0	10	-17	31	31
12	127	127	-1 5 9	41	-43	13	38	-37	18	16	16	-18	70	-55
13	72	69	-2	19	-23	14	88	-93	19	14	21	-19	0	0
14	60	-56	-3	35	-37	15	0	-1	20	80	-85	-20	25	-42
15	23	18	-4	10	-6	16	20	-13	-1 6 4	26	-7	-21	13	16
16	44	50	-5	66	-74	17	0	1	-2	78	71	0 6 7	71	65
17	0	4	-6	55	79	18	0	9	-3	62	-42	1	54	-57
18	11	17	-7	38	28	19	0	10	-4	194	-173	2	155	149
-1 5 6	29	4	-8	0	8	20	161	-173	-5	90	-68	3	0	-13
-2	48	52	-9	44	50	21	54	61	-6	63	53	4	48	47
-3	18	-8	-10	24	-30	22	76	90	-7	96	83	5	40	-38
-4	28	28	-11	31	33	23	40	-17	-8	44	-29	6	23	-32
-5	46	-33	-12	7	5	-1 6 2	36	-39	-9	43	-24	7	18	-21
-6	111	-102	-13	27	33	-2	315	317	-10	197	-182	8	50	-48
-7	43	-43	0 6 0	60	43	-3	0	26	-11	32	-31	9	0	-11
-8	198	195	1	58	-33	-4	22	19	-12	187	167	10	70	-63
-9	21	12	2	161	157	-5	0	10	-13	35	30	11	15	14
-10	142	-133	3	73	-74	-6	113	117	-14	71	-68	-1 6 7	24	23
-11	21	-16	4	128	-119	-7	0	-14	-15	50	-43	-2	76	-72
-12	121	121	5	65	73	-8	81	78	-16	114	116	-3	16	1
-13	152	154	6	68	83	-9	31	-37	-17	57	45	-4	61	-62
-14	22	-24	7	0	-6	-10	97	-100	-18	17	10	-5	20	-16
-15	20	-10	8	137	173	-11	30	-18	-19	0	-3	-6	72	-58
-16	94	-93	9	26	-24	-12	53	49	-20	63	-75	-7	0	20
-17	0	-6	10	247	-254	-13	28	-18	-21	0	-10	-8	0	-8
-18	86	93	11	0	5	-14	218	-222	-22	13	19	-9	58	-52
-19	35	-29	12	134	137	-15	73	-66	-23	12	-19	-10	23	-26
-20	67	-81	13	74	-73	-16	115	121	-24	39	-86	-11	24	19
-21	30	-29	14	152	-173	-17	0	-10	0 6 5	28	8	-12	69	67
-22	57	67	15	33	23	-18	49	49	1	33	-10	-13	21	-18
-23	0	9	16	47	-51	-19	0	-11	2	80	74	-14	47	57
-24	28	-38	17	0	-14	-20	16	-25	3	54	-41	-15	0	11
0 5 7	0	-4	18	117	127	-21	20	13	4	48	52	-16	23	-31
1	48	43	19	0	-23	-22	113	119	5	71	-55	-17	11	6
2	58	-49	20	0	9	-23	51	60	6	67	-63	-18	34	77
3	86	81	21	24	-30	-24	76	-91	7	60	23	-19	0	13
4	0	7	22	113	136	-25	18	-18	8	86	-81	0 6 8	19	-21
5	20	-12	23	19	-13	0 6 3	92	83	9	64	59	1	0	9
6	36	-32	24	66	-84	1	56	-48	10	38	26	2	10	-10
7	46	-41	25	0	-10	2	81	-79	11	29	-35	3	0	2
8	13	12	0 6 1	87	-91	3	78	76	12	56	50	4	68	80
9	32	-23	1	35	-33	4	236	-226	13	26	18	5	15	-16
10	45	43	2	142	-134	5	53	44	14	88	88	6	61	-97
11	19	-18	3	0	-5	6	107	-96	15	14	-16	-1 6 8	0	6
12	40	-44	4	46	-47	7	59	53	16	13	3	-2	0	0
13	0	-3	5	87	76	8	66	67	17	16	-23	-3	0	5
14	14	12	6	49	42	9	55	53	18	26	-25	-4	96	-127
-1 5 7	39	26	7	42	33	10	121	111	-1 6 5	36	33	-5	0	-2
-2	36	38	8	74	82	11	20	19	-2	79	75	-6	59	84
-3	32	22	9	0	-8	12	47	43	-3	33	29	-7	21	29
-4	147	142	10	128	121	13	65	-52	-4	0	-10	-8	0	-10
-5	141	-125	11	40	-42	14	0	3	-5	73	58	-9	35	-51
-6	45	34	12	29	32	15	0	-14	-6	201	-170	-10	0	-7
-7	29	23	13	54	65	16	51	-55	-7	67	45	-11	19	22
-8	35	-33	14	0	-13	17	0	5	-8	53	41	-12	43	78
-9	43	44	15	0	-6	18	81	-82	-9	33	28	-13	17	30
-10	51	44	16	130	-126	19	30	22	-10	75	71	-14	42	-94
-11	78	71	17	35	-27	20	11	-13	-11	70	52	1 7 0	143	-159
-12	70	-66	18	31	-25	21	0	-6	-12	22	16	2	100	-102
-13	104	101	19	37	37	22	11	-14	-13	44	-38	3	0	9
-14	93	-104	20	37	30	-1 6 3	0	-17	-14	165	149	4	315	324
-15	0	15	21	21	-15	-2	76	71	-15	35	-34	5	0	-12
-16	23	-20	22	13	-8	-3	23	14	-16	46	44	6	117	-122
-17	64	-68	23	20	-13	-4	174	147	-17	56	51	7	71	75
-18	42	48	24	16	-23	-5	19	-11	-18	58	-74	8	30	22
-19	61	-56	-1 6 1	51	-51	-6	27	19	-19	22	-12	9	49	55
-20	14	11	-2	31	-20	-7	79	62	-20	44	-56	10	84	-84
-21	8	-6	-3	92	-89	-8	43	-37	-21	18	22	11	50	48
-22	32	47	-4	144	150	-9	35	-20	-22	34	-66	12	52	-57
0 5 8	149	-164	-5	0	16	-10	100	-94	-23	0	9	13	110	-113
1	16	-9	-6	73	72	-11	31	18	0 6 6	56	-46	14	43	42
2	86	99	-7	32	27	-12	133	-110	1	55	-55	15	0	8
3	41	36	-8	25	13	-13	30	-22	2	111	104	16	49	-91
4	59	-63	-9	0	-7	-14	0	-23	3	45	44	17	24	31
5	18	18	-10	30	-29	-15	30	35	4	114	113	18	72	80
6	21	19	-11	55	56	-16	108	-101	5	27	9	19	37	-33
7	34	33	-12	183	-184	-17	0	-6	6	108	-107	20	25	-31
8	53	54	-13	41	39	-18	74	69	7	18	16	21	0	0
9	26	-33	-14	30	-26	-19	61	-56	8	22	17	22	14	-6
10	14	-13	-15	80	-67	-20	58	-62	9	0	-1	0 7 1	107	-102
-1 5 8	68	-73	-16	89	98	-21	49	52	10	116	-113	1	89	-86
-2	22	24	-17	0	-1	-22	48	-56	11	0	-5	2	70	64
-3	40	-44	-18	52	51	-23	0	3	12	17	15	3	80	-73
-4	26	21	-19	27	-20	-24	14	-31	13	23	-17	4	67	70
-5	56	-54	-20	89	93	-25	15	-24	14	12	-11	5	37	40
-6	14	-9	-21	0	-10	0 6 4	63	-50	15	0	2	6	89	89
-7	0	3	-22	46	59	1	43	33	-1 6 6	35	21	7	45	41
-8	107	116	-23	19	-21	2	95	85	-2	240	223	8	72	-70
-9	0	2	-24	0	18	3	27	-29	-3	62	53	9	28	19
-10	90	-102	-25	13	-11	4	38	-35	-4	236	-216	10	55	-60
-11	51	50	0 6 2	373	-371	5	0	2	-5	39	-40	11	66	49
-12	17	21	1	0	-8	6	108	-104	-6	0	-1	12	42	-46
-13	24	25	2	54	36	7	40	30	-7	20	-19	13	69	-73
-14	31	-36	3	26	24	8	204	211	-8	67	-59	14	57	-54
-15	36	-33	4	141	-156	9	0	-5	-9	51	-35	15	37	-39
-16	13	4	5	73	-71	10	155	-148	-10	0	9	16	61	58

Table 3. *(Continued)*

h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c	h k l	$\lvert F_o\rvert$	F_c
17 7 1	0	- 13	11 7 3	28	23	14 7 5	36	- 31	13 8 0	0	8	4 9 0	20	- 11
18	20	21	12	58	50	-1 7 5	102	82	14	30	28	5	25	- 12
19	0	6	13	25	27	-2	54	40	15	72	80	6	93	92
20	46	48	14	31	- 23	-3	0	- 8	16	41	- 48	7	0	3
21	36	35	15	0	10	-4	145	133	17	38	34	8	82	- 79
-1 7 1	0	0	16	21	- 26	-5	57	47	18	27	- 36	9	45	- 55
-2	89	- 83	17	33	- 28	-6	61	53	19	21	26	10	19	- 6
-3	11	- 4	18	17	16	-7	105	90	0 8 1	42	- 32	11	66	- 69
-4	34	- 31	19	40	- 38	-8	52	36	1	35	- 27	12	30	45
-5	146	140	-1 7 3	20	7	-9	112	-112	2	23	4	13	28	26
-6	80	- 76	-2	95	- 85	-10	29	40	3	39	34	14	42	- 46
-7	109	115	-3	85	81	-11	23	- 26	4	38	- 35	0 9 1	26	- 23
-8	17	- 13	-4	30	29	-12	46	- 41	5	117	110	1	30	- 33
-9	27	32	-5	60	51	-13	57	62	6	33	31	2	0	- 7
-10	83	82	-6	92	84	-14	83	- 82	7	45	37	3	29	- 9
-11	58	- 62	-7	75	- 71	-15	50	42	8	94	86	4	58	37
-12	88	92	-8	113	94	-16	53	53	9	41	- 28	5	19	- 12
-13	64	- 59	-9	63	- 46	-17	16	- 10	10	0	- 4	6	46	41
-14	32	- 37	-10	70	- 61	-18	37	32	11	83	- 71	7	27	23
-15	25	- 27	-11	0	10	-19	0	3	12	17	- 20	8	42	32
-16	31	- 28	-12	93	- 89	-20	8	- 6	13	45	- 39	9	26	- 22
-17	13	- 11	-13	55	- 45	0 7 6	79	75	14	27	- 15	10	31	- 29
-18	0	- 2	-14	39	- 36	1	44	31	15	23	- 15	11	19	- 7
-19	29	27	-15	146	144	2	90	- 98	16	15	- 9	12	35	- 23
-20	27	- 27	-16	22	- 4	3	25	11	17	15	- 6	13	33	30
-21	37	31	-17	72	73	4	58	62	18	15	- 13	-1 9 1	42	46
-22	21	18	-18	47	34	5	0	- 15	-1 8 1	93	- 79	-2	57	- 45
-23	38	- 47	-19	0	5	6	50	- 53	-2	0	8	-3	26	28
0 7 2	39	31	-20	31	28	7	12	7	-3	65	- 53	-4	103	- 87
1	0	0	-21	18	- 19	8	39	33	-4	0	- 12	-5	62	- 53
2	95	-109	-22	56	66	9	36	42	-5	0	11	-6	69	63
3	65	- 68	0 7 4	64	59	10	73	83	-6	22	16	-7	35	- 36
4	153	165	1	33	- 22	11	45	- 56	-7	22	18	-8	29	33
5	26	29	2	240	-236	-1 7 6	23	13	-8	0	- 2	-9	0	6
6	148	-151	3	35	20	-2	44	- 41	-9	88	74	-10	64	54
7	101	113	4	117	107	-3	51	- 44	-10	0	6	-11	41	- 36
8	24	25	5	76	- 67	-4	27	- 24	-11	29	- 26	-12	48	51
9	0	11	6	63	- 56	-5	0	- 6	-12	60	57	-13	0	- 6
10	37	- 31	7	79	- 77	-6	53	50	-13	0	- 4	-14	32	- 23
11	57	- 63	8	52	47	-7	50	- 37	-14	82	- 73	0 9 2	59	- 54
12	148	-169	9	23	- 24	-8	116	-111	-15	30	- 32	1	45	- 48
13	40	40	10	70	72	-9	0	3	-16	13	- 2	2	79	89
14	77	88	11	101	86	-10	69	69	-17	80	- 70	3	0	6
15	43	- 50	12	20	- 12	-11	59	57	-18	15	15	4	66	- 67
16	24	- 23	13	21	9	-12	38	- 39	-19	0	- 6	5	0	5
17	16	- 20	14	45	50	-13	18	24	0 8 2	92	93	6	99	105
18	57	65	15	16	5	-14	13	- 9	1	101	-111	7	69	- 75
19	17	- 13	16	62	- 69	-15	19	12	2	43	- 44	8	0	- 2
20	8	5	17	36	35	-16	46	49	3	29	- 38	9	44	51
21	36	41	-1 7 4	102	92	-17	14	20	4	31	25	10	0	- 15
-1 7 2	37	33	-2	57	47	0 7 7	18	- 17	5	98	107	11	0	6
-2	0	- 3	-3	92	88	1	15	- 11	6	51	55	12	0	- 19
-3	14	5	-4	57	- 48	2	17	- 15	7	66	65	13	13	2
-4	84	- 90	-5	0	- 15	3	9	- 7	8	77	- 90	-1 9 2	0	- 3
-5	64	63	-6	218	206	4	41	- 43	9	25	35	-2	47	- 40
-6	197	207	-7	0	8	5	24	20	10	111	110	-3	66	68
-7	51	- 45	-8	89	- 89	6	13	- 5	11	25	35	-4	119	127
-8	170	-179	-9	59	- 51	-1 7 7	16	16	12	49	- 57	-5	0	12
-9	132	-142	-10	60	60	-2	26	6	13	0	- 25	-6	42	- 47
-10	21	3	-11	0	- 10	-3	43	- 26	14	71	72	-7	0	- 21
-11	43	- 46	-12	122	-105	-4	16	16	15	86	- 82	-8	110	115
-12	0	9	-13	45	- 34	-5	58	- 58	16	0	10	-9	73	73
-13	15	23	-14	42	- 34	-6	35	- 32	-1 8 2	53	35	-10	0	- 7
-14	139	-152	-15	93	88	-7	32	- 22	-2	65	- 66	-11	0	- 20
-15	19	43	-16	79	80	-8	22	- 29	-3	99	- 98	-12	17	- 16
-16	41	43	-17	0	- 8	-9	62	64	-4	0	19	-13	0	5
-17	39	41	-18	63	- 71	-10	59	- 60	-5	38	- 36	-14	24	- 24
-18	41	- 47	-19	28	- 32	-11	53	46	-6	45	- 33	0 10 0	43	- 52
-19	20	14	-20	80	92	-12	25	24	-7	52	64	1	21	33
-20	63	69	-21	0	- 8	-13	19	- 27	-8	27	- 30	2	51	59
-21	38	34	0 7 5	106	- 92	-14	19	- 27	-9	82	77	3	14	- 7
-22	9	- 11	1	58	54	0 8 0	117	105	-10	80	87	4	12	- 3
-23	31	- 35	2	14	- 15	1	43	29	-11	22	28	5	34	35
0 7 3	14	10	3	34	- 28	2	151	-141	-12	104	-105	0 10 1	45	43
1	38	- 33	4	97	- 91	3	0	19	-13	34	- 32	1	12	3
2	22	10	5	49	- 38	4	35	35	-14	73	82	2	15	- 13
3	59	- 60	6	30	27	5	65	- 66	-15	22	- 20	3	46	56
4	47	- 38	7	54	- 39	6	54	- 56	-16	56	- 60	-1 10 1	16	- 12
5	25	- 27	8	69	66	7	40	- 38	-17	76	- 66	-2	0	9
6	81	68	9	55	- 48	8	38	- 31	-18	15	- 22	-3	29	- 29
7	15	- 18	10	21	17	9	0	17	-19	25	26	-4	12	- 16
8	51	46	11	18	- 9	10	121	115	1 9 0	43	44			
9	58	50	12	30	26	11	63	- 67	2	101	98			
10	29	32	13	18	15	12	106	-114	3	20	5			

factors were introduced for the second cycle of the refinement. The atomic scattering factors given by B. Dawson, by A. J. Freeman and R. E. Watson and by L. H. Thomas, K. Umeda and K. King (International Table, Vol. III, 1962) were used for S, As and Pb, respectively. Using this programme, the atomic coordinates, temperature factors, layer-scale factors and the population of the As(5) atom at the two positions were refined. After three cycles of refinement, the R factor was reduced from the initial value of 0.23 to 0.102 for all 3477 reflections and 0.086 for the 3013 observed reflections. The experimentally determined relative layer-scale factors and the final values obtained by the least-squares refinement agree within 3%, except for those reflections with k larger than 7 which were obtained from the photographs around the c axis. The experimental layer-scale factor for these reflections was underestimated owing to the insufficient integration for $K\alpha_1$—$K\alpha_2$ splitting in the higher Bragg-angle regions.

The final positional coordinates and the temperature factors are given in Table 1 and Table 2, respectively, with the standard deviations calculated by the least-squares programme. Since the dispersion effect was not taken into account, the actual temperature factors of the Pb atoms should be smaller than the values given in Table 2. The calculated and the observed structure amplitudes are given in Table 3. For the calculation of the structure amplitudes, the population of the As(5) atoms at the two positions were assumed to be 0.668 and 0.332 respectively.

The maximum and the average coordinate shifts in the last cycle of the refinement expressed as fractions of the standard deviations are 2.01 and 0.66. Since we obtained a good convergence with the full-matrix least-squares programme, it is not expected that further refinement will cause significant changes in the atomic coordinates unless a new weighting scheme is employed.

5. Description of the structure

The atomic distances and the bond angles are given in Table 4. From the temperature factors the r.m.s. deviations of the atoms along the principal axes of the vibration ellipsoids were calculated and are given in Table 5 along with the direction cosines of two principal axes.

Pb(1) and Pb(2) are surrounded by nine S atoms in the manner shown in Fig.2. The coordination polyhedra around Pb(1) and Pb(2) are joined together by sharing the bases to form PbS_6 strings along the c axis direction. The strings are laterally combined by sharing triangular faces of the polyhedra and form PbS_3 layers parallel to (100). Pb(3) has seven nearest-neighbouring S atoms. The mean Pb(3)—S distance is somewhat shorter than the mean Pb(1), Pb(2)—S distances.

As(1), As(2) and As(4) are each coordinated by three S atoms forming trigonal pyramids with them, and these are joined into strings by sharing S atoms (Fig.3). The mean As—S distances agree

Table 4. *Interatomic distances and bond angles in rathite-I*

Pb(Tl)—S and As—S distances. The mean values of the shortest three distances are given for the As(1)—, As(2)—, As(4)— and As(5b)—S

	Pb(1)	Pb(2)	Pb(3)	As(1)	As(2)	As(3)	As(4)	As(5a)	As(5b)
S(1)	3.012 Å 3.231	3.032 Å 3.254		2.254 Å					
S(2)	2.998 3.212	3.118 3.423			2.234 Å				
S(3)	3.472 3.048			2.304	2.394			2.811 Å	3.299 Å
S(4)		3.419 2.979	2.801 Å	2.237	3.424				
S(5)	3.300	3.084	2.958			2.283 Å		2.762	2.944
S(6)	3.206	3.044	2.875				2.258 Å	2.784	3.277
S(7)	3.263			3.247	2.271	3.439	2.327		
S(8)		3.366	3.392	3.236	2.945	2.684	2.251		
S(9)			2.962			2.737	3.362	2.735 2.770	2.233 2.408
S(10)			3.143 2.964			2.277	3.158	2.701	2.473
Mean	3.194	3.191	3.014	2.265	2.300		2.279	2.761	2.371
σ		0.008				0.0085		0.010	0.012

Table 4. (Continued.) *S—S distances*
The asterisk means that the S—S bond is an edge of an As-S_3 pyramid
$\sigma = 0.011$ Å

	S(1)	S(2)	S(3)	S(4)	S(5)	S(6)	S(7)	S(8)	S(9)	S(10)
S(2)	4.130 Å 4.371 3.920 4.271									
S(3)	3.560 3.486*	3.430 Å 3.524*								
S(4)	3.580 3.398*	3.318 3.814	3.455* Å							
S(5)	4.069	4.216	3.686	3.449 Å						
S(6)	3.682	4.088	3.709	3.715	4.503 Å 4.001					
S(7)	4.255	3.483*	3.368*		3.734	3.419* Å				
S(8)	4.344	3.741		3.560	3.548*	3.454*	3.430* Å			
S(9)			4.296	4.587	3.601* 3.815	4.280	3.652 4.001	4.204 Å	3.510* Å	
S(10)			4.522		3.420*	3.705 4.011	3.655	3.585* 3.995	3.439* 3.720*	4.178 Å

Table 4. *(Continued)*

Bond angles			
S(1)—As(1)—S(3)	99.8°	S(5)—As(3)—S(9)	91.2°
S(1)—As(1)—S(4)	98.3	S(9)—As(3)—S(10)	86.1
S(3)—As(1)—S(4)	99.1	S(6)—As(4)—S(7)	96.4
S(2)—As(2)—S(3)	99.1	S(6)—As(4)—S(8)	100.0
S(2)—As(2)—S(7)	101.3	S(7)—As(4)—S(8)	97.0
S(3)—As(2)—S(7)	92.4	S(9)—As(5*b*)—S(9′)	98.2
S(5)—As(3)—S(8)	90.8	S(9)—As(5*b*)—S(10)	99.3
S(5)—As(3)—S(10)	97.2	S(9′)—As(5*b*)—S(10)	93.8
S(8)—As(3)—S(10)	92.2	$\sigma \fallingdotseq 0.44^\circ$	
As(1)—S(3)—As(2)	107.4	As(4)—S(8)—As(3)	99.5
As(2)—S(7)—As(4)	98.5	As(3)—S(10)—As(5*b*)	92.6
		$\sigma \fallingdotseq 0.40^\circ$	

well with the normal As—S covalent-bond distance. The S—As—S and As—S—As angles are in a good agreement with the values found in the structure of orpiment (N. MORIMOTO, 1954). As(3) is coordinated by S(5) and S(10) at distances of about 2.28 Å and by S(8) and S(9) at distances of about 2.7 Å. Although the former are in good agreement with the normal As—S covalent-bond distance, the distances of 2.7 Å are too long for As—S covalent bonds. The magnitude and anisotropy of the temperature motion of As(3) are very large in comparison to those of As(1), As(2) and As(4), which have a maximum r.m.s. deviation of 0.27 Å and a minimum deviation of 0.18 Å (Table 5). The As(3), S(8) and S(9) atoms are nearly on a straight line, and As(3) has the largest r.m.s. deviation nearly parallel to this line. Therefore, As(3) seems to form covalent bonds statistically with

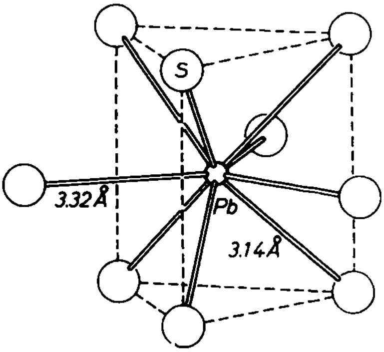


Fig. 2. The configuration of nine S atoms around a Pb atom

Table 5. *The r.m.s. deviations of the atomic positions along the principal axes of the vibration ellipsoids and the direction cosines of the axes refered to the orthogonal axes X // to a*, Y // to b and Z // to c*

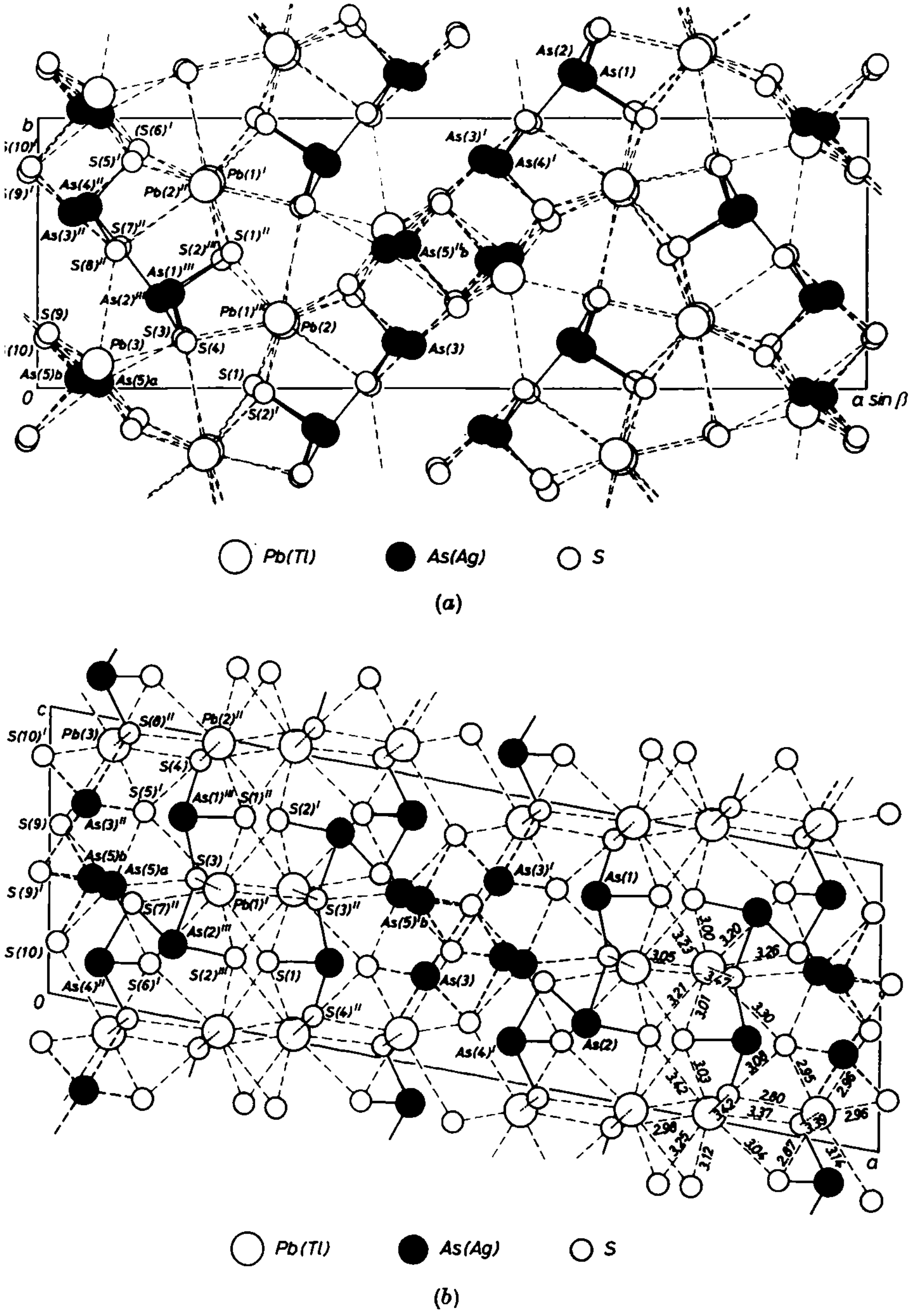
	r.m.s.d.	l	m	n
Pb(1)	0.223 Å	−0.946	0.285	0.143
	0.188	−0.060	0.271	−0.959
	0.212			
Pb(2)	0.309	−0.590	0.678	0.438
	0.183	−0.050	0.510	−0.858
	0.227			
Pb(3)	0.243	0.332	0.804	0.494
	0.188	0.217	0.444	−0.869
	0.216			
As(1)	0.190	0.081	0.996	−0.023
	0.156	−0.442	0.058	0.896
	0.182			
As(2)	0.212	0.465	0.514	0.721
	0.164	−0.719	0.694	−0.030
	0.176			
As(3)	0.271	0.603	0.342	0.721
	0.175	−0.791	0.153	0.588
	0.202			
As(4)	0.195	−0.192	0.981	−0.028
	0.162	0.317	0.035	−0.949
	0.175			
As(5*a*)	0.261	0.051	0.926	0.383
	0.215	0.280	0.354	−0.893
	0.218			
As(5*b*)	0.207	0.004	0.943	−0.333
	0.115	−0.938	0.124	0.324
	0.179			
S(1)	0.191	0.823	0.464	0.325
	0.153	−0.372	0.875	−0.310
	0.170			
S(2)	0.178	0.641	0.752	0.150
	0.152	−0.749	0.570	0.340
	0.168			
S(3)	0.182	−0.951	0.209	−0.221
	0.138	0.042	0.819	0.572
	0.160			

Table 5. *(Continued)*

	r.m.s.d.	*l*	*m*	*n*
S(4)	0.203	−0.746	0.347	−0.569
	0.151	−0.268	0.626	0.733
	0.155			
S(5)	0.176	−0.844	0.492	0.215
	0.145	0.361	0.817	−0.450
	0.167			
S(6)	0.190	0.930	0.185	−0.317
	0.145	−0.279	0.918	−0.283
	0.173			
S(7)	0.183	−0.832	0.542	−0.120
	0.140	0.307	0.258	−0.917
	0.173			
S(8)	0.235	0.963	0.070	0.259
	0.150	0.168	0.597	−0.785
	0.170			
S(9)	0.237	0.938	0.322	−0.136
	0.177	0.132	0.033	0.990
	0.195			
S(10)	0.200	0.082	0.869	−0.489
	0.155	−0.448	0.471	0.761
	0.178			

S(8) and S(9). If As(3) forms a covalent bond with S(8) the As(3)-S_3 trigonal pyramid is joined with the As(4)-S_3 pyramid.

As(5) was statistically distributed over two positions, (*a*) and (*b*), during the course of the refinement. The position (*a*) is surrounded octahedrally by six S atoms, while the position (*b*) has a trigonal pyramidal coordination of three S atoms, which is usual in crystal structures of arsenosulfides. It is suspected that the position (*a*) is not occupied by As but by a different kind of atom, since the distances from the position (*a*) to the surrounding S atoms are too long for As—S distances, and since the sum of the population factors for the positions (*a*) and (*b*), as obtained by the least-squares method, is much larger than one. Actually, a careful chemical analysis of the crystal used, carried out by G. BURRI with a CAMECA x-ray microanalyser, showed that the crystal contains a few weight percent of Ag. If the positions (*a*) are occupied by Ag atoms, the population factor for (*a*) becomes about 0.57 and the sum is nearly equal to one. Therefore,



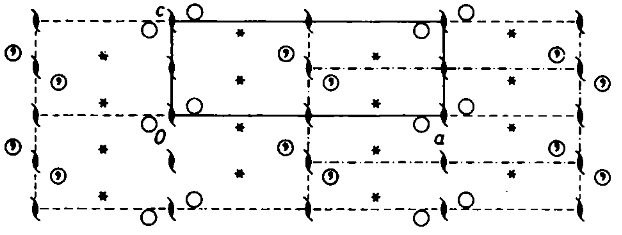
(a)

(b)

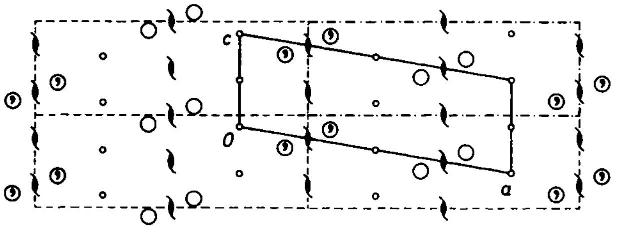
Fig. 3. The projection of the structure (a) along the c axis and (b) along the b axis

the position (*a*) is probably occupied by Ag instead of As. I is not to be expected from the crystallochemical point of view that the As(5) atoms occupy all the (*b*) positions, since two As(5)—S_3 trigonal pyramids around a center of symmetry should share two S atoms if it occurs.

The projections of the structure along the *b* and *c* axis are shown in Fig. 3(*a*) and (*b*). The structure is composed of two kinds of layers parallel to (100). The first kind are the PbS_3 layers. The second kind have a structure closely related to the PbS structure. It is derived from the PbS structure by dividing it into layers which have the thickness of *a*(PbS) and are parallel to (100) of PbS, and by mutually shifting the layers in the [011] direction of PbS by a distance amounting to $a(\text{PbS})/2\sqrt{2}$. The layers in the rathite-I structure correspond to a zone bounded by two planes perpendicular to the [223] direction in the deformed PbS structure. Although each metallic atom in the deformed PbS structure is coordinated by seven S atoms, the As atoms in the rathite-I structure are coordinated by less than seven S atoms, owing to the fairly large deviation from the ideal atomic configuration caused by the difference in chemical character of As and Pb.



Rathite - III



Rathite - I

Fig. 4. A comparison of the unit cells and the symmetries of rathite-I and rathite-III. The local centres of symmetry in rathite-III are represented by asteriks. Both of the structures are composed of identical units bounded by the dashed and dotted lines

The main difference in the structure of rathite-I as compared to that of rathite-III (M.-TH. LE BIHAN, 1962) lies in the relative positions of Pb(3) and As(5). They are made up of the same structural unit, which has the volume of one unit cell (Fig.4). In rathite-III, Pb(3) and As(5) are exchanged in the next structural unit along the a-axis direction whereby the centre of symmetry which exists in the rathite-I structure is destroyed.

The crystal structures of rathite-II (M.-TH. LE BIHAN, 1962), dufrenoysite (W. NOWACKI, F. MARUMO and Y. TAKÉUCHI, 1964), baumhauerite (M.-TH. LE BIHAN, 1962) and scleroclase (W. NOWACKI, Y. IITAKA, H. BÜRKI and V. KUNZ, 1961) are also composed of PbS_3 layers and layers which have the deformed PbS structure. The differences between these structures lie in the chemical composition and in the thickness of the second kind of layers.

Although infinite chains of $As\text{-}S_3$ pyramids have been described in the structures of rathite-II, rathite-III and baumhauerite, it is impossible to adapt such chains to the PbS_3 layers, as has been pointed out by Y. IITAKA and W. NOWACKI (1961) and by Y. TAKÉUCHI, S. GHOSE and W. NOWACKI (1965). In the structure of rathite-I the $As\text{-}S_3$ pyramids form chains with finite lengths. The length of the chain is not fixed since there are several possibilities for the coordinations around the As(3) and As(5) atoms as explained above. In the most favourable case, the chain can contain six $As\text{-}S_3$ pyramids, in the order of As(1)—As(2)—As(4′)—As(3′)—As(5″)—As(3).

Tl atoms are thought to be situated at the Pb position, replacing Pb atoms. It is not known whether the Tl atoms are in an ordered state or whether they are statistically distributed over several positions. Probable positions are the Pb(2) positions, since Pb(2) has a much larger anisotropic temperature factor than Pb(1) and Pb(3).

Acknowledgements

We thank Prof. L. G. BERRY (Kingston) for the untwinned rathite-I crystal, Prof. W. NEF and Dr. G. HÜSSER for the possibility of using the Bull Γ AET-computer, Dr. J. S. ROLLETT of Oxford University Computing Laboratory for calculations on the Mercury computer, the International Business Machines, Extension Suisse, and Cern (Geneva) for the IBM 7090-computer time, Dr. H. BÜRKI and Mr. V. KUNZ for their help in the x-ray experiments and in the calculations by the Bull Γ AET computer, Prof. B. J. WUENSCH (Cambridge, Mass.) for having introduced the Prewitt least-square refinement programme, Dr. N. D. JONES (Bern) for his help in improving the English of this paper and Mr. G. BURRI for an additional examination of the rathite-I crystal with the x-ray microanalyser. This investigation was sponsored by Schweizerischer Nationalfonds, Kommission zur Förderung der wissenschaftlichen Forschung and Stiftung Entwicklungsfonds Seltene Metalle.

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