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The crystal structure of β spodumene, $LiAlSi_2O_6$-II

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Auszug

Eine unabhängig von der Untersuchung von Li und Peacor ausgeführte Bestimmung der Struktur von β-Spodumen erbrachte genauere Werte der Strukturparameter.

Abstract

An independent determination of the structure of β spodumene by x-ray diffraction has yielded more precise values for the structural parameters.

Introduction

The recent paper by Chi-Tang Li and D. R. Peacor (1968) reported a structure determination of β spodueme. By chance we had independently determined the structure also by x-ray diffraction but using rather more data and found structural parameters which were substantially identical as far as the coordinates were concerned but differ somewhat in the thermal parameters. As a consequence of the use of more data (572 instead of 335 unique non-zero reflections) our results are more precise than those of the previous authors.

Experimental

The crystals were grown from a lithium-vanadate flux. The composition of the initial mixture was in the ratio 100 moles $LiVO_3$: 2.62 moles α spodumene. The melt was heated for 30 minutes at 1150 °C, then cooled to 980 °C. Further cooling was continued at 2.6 °C per day for 6 days, then 2 °C per day for 13 days. The mixture was then quenched and the crystals obtained by digestion with hydrochloric acid. The analysis of a bulk sample was 4.000 SiO_2, 1.000 Al_2O_3,

0.015 V_2O_5, 1.035 Li_2O. The vanadium and some of the slight excess of lithium was due to the inclusion of small quantities of the flux which was trapped inside some crystal aggregates.

To collect intensity data we selected a perfectly colourless clear crystal in the shape of an almost perfect octahedron of edge 0.36 mm, and mounted it to rotate about the c axis. We measured the intensities with a Hilger and Watts linear diffractometer using Mo$K\alpha$ radiation, balanced filters with a scintillation counter, and a pulse-height analyser. We collected a quadrant of the available reciprocal lattice, hkl, khl, $\bar{h}kl$ and $k\bar{h}l$ (i.e. half of each layer), measuring layers from $l = 0$ to $l = 12$. We took two pairs of measurements on each reflection, and averaged the results for equivalent reflections. In general the agreement between equivalent reflections was excellent except for the set 021, 201 and $\bar{2}01$ for which 021 differed from the other two by several standard deviations (7564 counts in a mean total count of 602,367 which is still less than $1\frac{1}{2}\%$). We collected a total of 572 unique reflections, of which only 7 were too weak to be properly measured. These seven were assigned the average measured value which was between 1 and 2 standard deviations above the background in each case, but they were omitted from the refinement. We did not apply absorption corrections since they were found to be negligible ($\mu R = 0.14$ for Mo$K\alpha$) both by calculation and experiment (method of NORTH, PHILLIPS and MATHEWS, 1968). Our values for the lattice constants were not significantly different from those of the previous authors whose values are to be preferred and were in fact used by us for our final calculation. We found $a = b = 7.534 \pm 0.005$, $c = 9.158 \pm 0.009$. The form-factor curves we used were taken from *International tables for x-ray crystallography*, Vol. III, Li^+, O and $\frac{1}{3}$(Al + 2Si).

Refinement and results

Our least-squares refinement proceeded much as for the previous workers. We also omitted the 040 and 021 reflections because of severe extinction. We had the same difficulties in locating the lithium atom, indeed it was not located unambiguously until we had introduced and refined individual anisotropic temperature factors for silicon atoms, as well as individual isotropic temperature factors on the oxygen atoms before calculating a difference synthesis at $R = 6.8\%$. Subsequent refinement with individual anisotropic temperature factors on all atoms including lithium reduced R to 3.69% over 563

Table 1. *Final atom parameters.* Coordinates and anisotropic temperature factors

Atom	x	y	z
Si(1)	0.3322 ± 1	0.1223 ± 1	0.2388 ± 1
Si(2)	0.4174 ± 1	(0.4174)	(0)
O(1)	0.4426 ± 4	0.1216 ± 4	0.3947 ± 3
O(2)	0.1243 ± 3	0.1153 ± 3	0.3001 ± 3
O(3)	0.3654_5 ± 4	0.3048 ± 4	0.1468_5 ± 3
Li^+	0.0826_5 ±24	0.1982 ±25	0.4944 ±17

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	0.0127 ± 3	0.0124 ± 3	0.0099 ± 3	−0.0004 ± 3	0.0003 ± 2	0.0001 ± 2
Si(2)	0.0111 ± 3	(0.0111)	0.0125 ± 4	−0.0007 ± 3	0.0003 ± 2	(−0.0003)
O(1)	0.0322 ±12	0.0343 ±13	0.0266 ±12	−0.0056 ± 9	−0.0162 ± 9	0.0014 ±10
O(2)	0.0153 ± 9	0.0308 ±12	0.0219 ± 9	0.0011 ± 7	−0.0005 ± 7	−0.0022 ± 8
O(3)	0.0483 ±16	0.0329 ±14	0.0339 ±15	−0.0035 ±11	0.0087 ±12	0.0145 ±11
Li^+	0.0555 ±90	0.0810 ±113	0.0227 ±76	−0.0238 ±76	−0.0059 ±62	−0.0063 ±81

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Si(1)	0.0044	0.0043	0.0023	−0.0005	0.0003	−0.0001
Si(2)	0.0039	0.0038	0.0030	−0.0010	0.0003	(−0.0003)
O(1)	0.0112	0.0119	0.0063	−0.0077	−0.0185	0.0016
O(2)	0.0053	0.0107	0.0052	0.0015	−0.0006	−0.0025
O(3)	0.0168	0.0114	0.0080	−0.0048	0.0010	0.0166
Li^+	0.0193	0.0282	0.0053	−0.0331	−0.0067	−0.0071

The temperature factor $q(hkl)$ is given by

$$q(hkl) = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$$

or

$$q(hkl) = \exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$$

reflections, not including the seven weak reflections, 040 or 021. ($R = 3.74\%$ over 570 reflections, 4.83% over all the measured 572 reflections.) The weighting scheme used for the final least-squares rounds with full-matrix calculations was

$$\sqrt{w} = \frac{1}{\sqrt{1 + \left(\frac{|F_o| - p_2}{p_1}\right)^2}}$$

where $p_1 = 8.2$ and $p_2 = 14.0$. The quantity minimized was

$$\Sigma w (|F_o| - |F_c|)^2.$$

The final atomic parameters and their standard deviations are given in Table 1. Both U and β values are given for the temperature factors to facilitate comparison with the previous results. The coordinates are not significantly different from those of the previous authors [the y coordinate of Si(1) was misprinted, it should have been 0.1221 which is within significance limits of our results]. The thermal

Table 2. *Thermal motion data*

Atom	Principal thermal axes	Root-mean-square displacement		Direction cosines of the principal axes in the xyz system of the crystal		
				α	β	γ
Si(1)	1	0.099	± 0.002 Å	− 0.1049	0.0031	0.9945
	2	0.110	± 0.002	0.5537	0.8308	0.0559
	3	0.114	± 0.002	0.8261	− 0.5565	0.0889
Si(2)	1	0.102	± 0.002	0.7071	0.7071	0
	2	0.108	± 0.002	0.6526	− 0.6526	− 0.3850
	3	0.111	± 0.002	0.2722	− 0.2722	0.9229
O(1)	1	0.112	± 0.004	0.6535	0.1192	0.7475
	2	0.181	± 0.004	− 0.1996	− 0.9254	0.3222
	3	0.219	± 0.004	− 0.7301	0.3597	0.5810
O(2)	1	0.123	± 0.004	0.9966	− 0.0627	0.0538
	2	0.146	± 0.003	− 0.0379	0.2318	0.9720
	3	0.177	± 0.004	− 0.0734	− 0.9707	0.2286
O(3)	1	0.128	± 0.004	− 0.2630	− 0.6710	0.6933
	2	0.213	± 0.004	− 0.5493	0.6949	0.4642
	3	0.231	± 0.004	0.7392	0.2587	0.5513
Li^+	1	0.140	± 0.025	0.2899	0.2071	0.9344
	2	0.309	± 0.023	− 0.8104	− 0.4663	0.3548
	3	0.309	± 0.021	0.5091	− 0.8601	0.0327

Table 3. *Observed and calculated structure factors*

h k l	A_c	B_c	F_c	F_o	h k l	A_c	B_c	F_c	F_o	h k l	A_c	B_c	F_c	F_o
0 2 0	1.35	0	1.35	1.01	1 3 2	15.09	-24.70	28.94	32.19	1 9 4	4.43	2.85	5.26	5.41
6	- 6.56	0	6.56	9.67	4	26.19	6.00	26.87	28.12	10	-20.99	0.39	21.00	20.75
8	6.44	0	6.44	6.90	5	-48.97	4.59	49.18	47.66	2 3 4	43.57	- 1.78	43.60	42.52
10	4.25	0	4.25	4.27	6	4.52	14.87	15.54	15.60	4	7.10	- 9.10	11.54	11.89
1 1 0	8.74	0	8.74	9.66	7	- 1.49	3.63	3.92	4.25	5	33.10	- 1.69	33.15	32.86
2	5.55	0	5.55	5.84	8	14.85	- 3.57	15.27	14.78	6	- 6.05	11.61	13.09	13.28
3	6.57	0	6.57	5.52	9	16.57	2.49	16.76	16.71	7	-29.14	1.32	29.17	29.87
4	-14.67	0	14.67	14.86	10	- 1.09	- 2.48	2.71	2.61	8	-12.62	4.86	13.52	13.18
5	-18.99	0	18.99	18.97	2 2 2	-44.11	0	44.11	41.31	9	- 4.71	1.10	4.84	4.90
6	40.00	0	40.00	38.05	3	-11.63	-29.25	31.48	32.54	3 3 4	-49.00	0	49.00	48.59
9	9.37	0	9.37	9.56	4	4.14	2.33	4.75	5.18	4	5.34	5.87	7.94	7.97
10	-28.26	0	28.26	28.03	5	3.96	- 4.89	6.29	5.41	5	- 4.75	- 3.53	5.92	6.54
2 2 0	11.49	0	11.49	12.49	6	4.88	1.95	5.25	5.50	6	- 8.01	- 1.44	8.14	8.12
3	14.11	0	14.11	13.21	7	15.21	4.03	15.73	15.41	7	16.79	- 4.39	17.36	17.69
4	6.27	0	6.27	6.35	8	- 0.62	- 1.19	1.34	1.81	8	- 3.58	1.78	4.00	3.78
5	12.39	0	12.39	12.70	9	5.34	- 5.20	7.46	7.96	9	3.58	2.36	4.29	4.26
6	-12.32	0	12.32	12.85	3 3 2	-22.03	0	22.03	22.33	4 4 4	36.38	0	36.38	35.89
7	-32.05	0	32.05	32.71	4	12.10	5.41	13.26	12.86	5	21.88	- 2.01	21.97	21.62
8	-11.38	0	11.38	11.03	5	-33.62	-17.57	37.94	37.53	6	- 7.84	- 5.25	9.43	9.71
9	- 8.40	0	8.40	7.26	6	15.79	0.29	15.79	15.50	7	5.62	4.04	6.92	7.49
3 3 0	- 2.25	0	2.25	3.39	7	16.83	3.60	17.21	17.87	8	- 2.50	0.10	2.51	2.11
4	9.39	0	9.39	9.55	8	-16.16	- 1.89	16.27	15.92	9	-16.29	- 2.45	16.47	16.21
5	- 1.99	0	1.99	2.67	9	14.44	2.39	14.63	14.98	5 5 4	32.38	0	32.38	32.10
6	26.91	0	26.91	26.28	4 4 2	-13.48	0	13.48	14.40	6	-15.23	- 2.40	15.42	15.65
7	9.33	0	9.33	9.14	5	-41.99	1.39	42.01	40.31	7	8.30	1.66	8.47	8.34
8	- 2.60	0	2.60	2.62	6	-19.20	2.90	19.42	18.91	8	-14.18	4.84	14.98	14.74
9	- 4.89	0	4.89	3.84	7	- 7.26	3.00	7.85	7.93	9	-10.83	- 1.28	10.90	11.47
4 4 0	44.67	0	44.67	42.64	8	8.03	1.49	8.16	8.65	6 6 4	11.06	0	11.06	10.99
5	27.54	0	27.54	27.73	9	20.16	- 0.93	20.18	20.01	7	10.29	- 1.11	10.35	10.30
6	- 6.83	0	6.83	7.74	5 5 2	21.90	0	21.90	22.16	7 7 4	3.94	0	3.94	3.37
7	0.70	0	0.70	2.04	6	- 4.61	-10.63	11.59	11.38	0 2 5	-47.86	-47.86	67.69	62.50
8	3.93	0	3.93	4.36	7	8.72	- 6.54	10.90	10.86	3	-32.96	32.96	46.61	45.43
9	-18.59	0	18.59	18.38	8	1.49	1.67	2.24	2.41	4	1.09	1.09	1.54	2.94
5 5 0	33.70	0	33.70	33.40	9	- 0.45	- 2.05	2.10	2.56	5	- 6.71	6.71	9.48	11.35
6	-23.13	0	23.13	23.33	6 6 2	6.09	0	6.09	6.03	6	2.49	2.49	3.52	3.08
7	11.08	0	11.08	10.92	7	-20.98	1.55	21.04	20.20	7	14.95	-14.95	21.14	21.85
8	-15.31	0	15.31	15.50	7 7 2	-15.53	0	15.53	15.80	8	0.87	0.87	1.23	2.29
9	-18.38	0	18.38	18.26	0 2 3	1.91	- 1.91	2.70	2.19	10	8.01	8.01	11.33	11.75
6 6 0	14.62	0	14.62	15.09	3	45.43	45.43	64.25	61.29	1 2 5	8.78	-15.22	17.57	18.12
7	2.77	0	2.77	3.37	4	5.17	- 5.17	7.31	7.70	3	0.40	-29.67	29.68	30.21
7 7 0	5.90	0	5.90	7.09	5	19.34	19.34	27.35	27.14	4	4.27	9.04	10.00	10.57
0 1 1	- 9.26	9.26	13.10	14.27	6	- 0.49	0.49	0.70	2.81	5	-14.92	- 2.92	15.20	14.93
3	- 6.16	6.16	8.71	7.87	7	-23.68	-23.68	33.48	33.56	6	- 3.92	12.36	12.97	12.91
5	- 4.87	4.87	6.88	7.34	8	- 3.34	3.34	4.73	5.35	7	3.32	21.80	22.05	21.38
6	22.08	22.08	31.22	31.16	9	- 1.47	- 1.47	2.07	1.74	8	-13.35	-15.69	20.60	20.56
7	19.42	-19.42	27.46	28.26	10	- 6.28	6.28	8.88	8.85	9	2.24	7.56	7.89	7.81
8	1.97	1.97	2.79	2.85	1 2 3	20.26	22.22	30.07	30.47	10	10.31	-12.53	16.23	16.14
9	6.45	- 6.45	9.12	8.94	3	-27.47	-45.94	53.52	52.89	2 2 5	0	- 4.65	4.65	4.48
10	3.35	3.35	4.73	5.01	4	- 3.96	6.81	7.88	7.89	3	19.39	20.79	28.44	29.16
1 1 1	0	32.15	32.15	32.83	5	25.99	-16.57	30.82	30.21	4	-41.67	35.69	54.87	52.09
2	-30.99	18.30	35.99	35.93	6	- 6.11	- 5.52	8.24	8.39	5	-16.68	0.72	16.70	17.24
3	- 1.03	-14.56	14.59	13.58	7	9.95	18.63	21.12	21.16	6	- 7.77	0.99	7.83	7.33
4	- 2.28	6.15	6.56	6.72	8	8.18	- 9.86	12.81	12.29	7	-14.19	- 9.45	17.05	17.10
5	-18.26	-26.62	32.28	32.40	9	- 7.85	10.90	13.43	13.46	8	12.26	- 8.61	14.98	14.57
6	- 8.48	22.62	24.16	23.21	10	- 8.47	- 9.36	12.62	12.46	9	13.10	1.28	13.16	13.37
7	- 5.74	18.64	19.50	19.84	2 2 3	0	9.37	9.37	9.87	3 3 5	0	-15.11	15.11	15.52
8	-10.25	-13.31	16.80	16.19	3	- 2.74	11.78	12.09	12.55	4	-16.89	- 4.30	17.43	17.37
9	10.60	7.93	13.24	13.28	4	14.66	10.55	18.06	19.05	5	- 4.40	24.37	24.76	24.57
10	10.97	-16.03	19.43	19.23	5	36.36	-22.66	42.84	41.54	6	9.64	-10.02	13.91	13.66
2 2 1	0	2.40	2.40	2.85	6	3.51	-12.62	13.10	13.22	7	-10.38	5.76	11.87	11.59
3	0.63	5.59	5.62	5.63	7	5.57	- 4.02	6.87	6.88	8	- 5.27	-14.54	15.47	15.53
4	-33.74	20.23	39.34	37.91	8	2.22	5.32	5.77	6.61	9	0.40	- 4.07	4.09	4.55
5	-38.24	-19.61	42.97	42.04	9	-16.78	6.44	17.98	18.16	4 4 5	0	-13.11	13.11	13.81
6	- 6.00	- 4.67	7.60	7.68	3 3 3	0	-39.06	39.06	40.35	5	7.12	-12.23	14.15	13.87
7	- 5.33	- 3.01	6.12	6.09	4	34.03	-17.02	38.05	37.60	6	-11.45	0.93	11.49	11.47
8	0.13	7.87	7.87	7.79	5	- 7.37	38.76	39.46	39.14	7	-13.86	- 3.57	14.31	14.30
9	18.09	5.66	18.96	18.99	6	2.67	- 7.13	7.61	7.15	8	- 7.22	12.55	14.47	14.45
3 3 1	0	5.88	5.88	6.62	7	5.09	19.34	20.00	20.24	9	- 3.58	- 2.27	4.24	4.39
4	-10.04	9.09	13.54	13.58	8	- 6.15	- 4.79	7.79	7.83	5 5 5	0	6.51	6.51	7.46
5	- 3.71	23.22	23.52	23.45	9	3.60	- 9.24	9.92	10.41	6	-13.39	- 9.41	16.36	16.39
6	17.60	-21.36	27.67	27.26	4 4 3	0	- 6.03	6.03	6.27	7	-12.26	-12.72	17.67	16.65
7	-11.24	5.70	12.60	12.76	5	-13.50	-13.14	18.84	18.79	8	5.78	10.73	12.18	12.36
8	- 5.51	-15.03	16.01	15.65	6	8.92	2.96	9.39	9.75	6 6 5	0	8.58	8.58	8.52
9	5.12	- 9.44	10.74	10.78	7	27.46	4.78	27.87	27.39	7	6.44	- 0.44	6.45	6.46
4 4 1	0	-10.91	10.91	10.68	8	7.66	10.30	12.83	12.62	7 7 5	0	1.09	1.09	2.00
5	0.86	-10.74	10.77	10.28	9	4.93	- 6.04	7.80	8.22	0 2 6	0	4.05	4.05	3.86
6	-12.16	2.72	12.46	12.97	5 5 3	0	11.49	11.49	11.77	3	3.55	0	3.55	3.05
7	-23.14	- 0.32	23.14	22.70	6	20.05	0.54	20.05	19.18	4	0	-20.08	20.08	19.24
8	- 7.30	11.81	13.89	13.40	7	5.44	-12.53	13.66	14.00	5	38.73	0	38.73	39.15
9	- 6.35	1.12	6.45	6.71	8	- 4.49	6.80	8.15	7.82	6	0	- 5.72	5.72	7.00
5 5 1	0	25.10	25.10	24.54	9	1.44	- 3.97	4.22	4.01	7	7.60	0	7.60	7.04
6	-12.51	-12.47	17.67	17.67	6 6 3	0	21.70	21.70	21.05	8	0	17.19	17.19	16.56
7	- 9.67	-13.84	16.88	17.02	7	- 0.36	- 1.32	1.37	2.19	9	-19.79	0	19.79	20.08
8	2.79	6.14	6.75	6.74	7 7 3	0	- 6.71	6.71	6.40	10	0	1.58	1.58	2.01
9	- 4.91	- 4.65	6.76	6.82	0 2 4	2.14	0	2.14	3.64	1 2 6	- 2.25	-18.51	18.64	19.28
6 6 1	0	15.43	15.43	15.67	3	0	17.52	17.52	17.87	3	1.96	3.10	3.66	3.77
7	7.45	4.46	8.68	9.21	4	-47.55	0	47.55	45.62	4	0.37	4.28	4.30	4.97
7 7 1	0	1.24	1.24	2.30	5	0	12.17	12.17	11.11	5	-21.17	7.01	22.30	22.25
0 1 2	-68.75	0	68.75	58.48	6	- 4.95	0	4.95	4.64	6	2.18	1.08	2.43	2.32
2	0	3.23	3.23	5.51	7	0	- 9.50	9.50	9.57	7	3.26	1.13	3.45	2.04
3	- 6.98	0	6.98	6.22	8	1.42	0	1.42	1.73	8	23.25	- 2.33	23.36	23.29
4	0	26.16	26.16	29.08	9	0	- 0.60	0.60	3.54	9	10.59	- 8.06	13.31	12.99
5	73.72	0	73.72	67.85	10	3.32	0	3.32	4.23	10	0.24	- 1.82	1.83	1.45
6	0	- 4.15	4.15	4.27	1 2 4	-23.98	- 1.46	24.02	26.55	2 2 6	-87.02	0	87.02	77.55
7	10.94	0	10.94	10.72	3	31.20	- 3.97	31.45	31.45	3	-10.38	18.63	21.33	21.56
8	0	- 8.37	8.37	8.18	4	- 4.63	- 8.21	9.43	9.20	4	- 0.16	3.23	3.23	3.80
9	-29.23	0	29.23	29.12	5	-30.39	- 3.55	30.59	29.82	5	3.25	- 9.23	9.78	10.12
10	0	0.61	0.61	2.62	6	16.34	1.73	16.43	16.35	6	15.40	- 0.99	15.43	15.33
1 1 2	61.62	0	61.62	55.93	7	-23.63	- 4.62	24.07	24.22	7	12.41	1.44	12.50	12.34
2	- 6.23	0.05	6.23	6.91	8	3.59	- 2.95	4.65	4.91	8	2.38	- 1.87	3.03	3.07

Table 3. *(Continued)*

h k l	A_c	B_c	F_c	F_o
2 9 6	3.59	3.20	4.81	4.89
3 3 6	-15.53	0	15.53	16.26
4	11.33	5.44	12.57	12.62
5	-18.85	- 4.26	19.32	19.10
6	13.35	0.67	13.37	13.57
7	14.85	- 1.32	14.91	14.88
8	-18.00	- 4.62	18.58	18.56
9	11.19	7.49	13.46	13.21
4 4 6	-13.11	0	13.11	13.51
5	-15.06	5.04	15.88	15.63
6	-12.58	4.70	13.43	13.55
7	- 7.60	4.04	8.61	8.39
8	7.62	- 7.70	10.83	10.94
9	11.59	- 2.92	11.95	11.75
5 5 6	6.26	0	6.26	5.78
6	- 4.08	1.01	4.21	3.57
7	3.20	- 3.20	4.53	4.71
8	- 9.04	2.36	9.34	9.78
6 6 6	3.84	0	3.84	4.86
7	-17.11	- 6.30	18.23	18.37
7 7 6	-15.60	0	15.60	16.10
0 2 7	39.34	-39.34	55.64	52.81
3	20.69	20.69	29.25	29.61
4	4.99	- 4.99	7.06	6.53
5	4.02	4.02	5.69	6.07
6	- 7.31	7.31	10.34	10.08
7	-20.89	-20.89	29.54	29.52
8	- 3.74	3.74	5.30	4.79
9	- 1.82	- 1.82	2.57	2.17
10	- 1.68	1.68	2.37	2.62
1 2 7	21.20	21.44	30.15	31.06
3	-17.27	- 6.40	18.41	18.75
4	- 4.65	3.32	5.72	5.38
5	13.65	- 7.79	15.72	15.77
6	3.46	2.36	4.19	3.71
7	12.42	6.98	14.25	14.54
8	11.64	- 9.40	14.96	14.91
9	-11.08	4.68	12.03	11.95
2 2 7	0	6.33	6.33	6.26
3	19.10	- 8.41	20.87	20.78
4	15.63	6.69	17.00	17.32
5	21.81	-13.83	25.83	26.16
6	1.63	- 9.16	9.30	9.25
7	- 3.51	- 0.16	3.52	3.61
8	5.87	11.11	12.56	12.02
9	-12.77	6.48	14.32	14.15
3 3 7	0	-28.24	28.24	28.16
4	15.34	2.02	15.47	15.56
5	- 0.77	13.60	13.63	13.61
6	5.21	- 9.04	10.43	9.91
7	4.80	19.92	20.49	20.96
8	0.64	-10.34	10.36	10.60
9	0.53	- 7.57	7.59	7.61
4 5 7	- 5.56	- 6.95	8.90	8.79
6	3.87	7.70	8.62	8.79
7	22.79	3.06	22.99	22.52
8	4.87	4.28	6.49	6.45
9	4.03	- 2.62	4.81	5.04
5 5 7	0	4.84	4.84	4.80
6	5.32	- 5.22	7.46	7.60
7	4.51	- 6.55	7.96	7.94
8	- 5.37	4.28	6.87	7.80
6 6 7	0	16.19	16.19	16.19
7	2.00	4.03	4.50	4.36
7 7 7	0	- 9.68	9.68	10.32
0 2 8	0.95	0	0.95	1.39
3	0	18.72	18.72	19.02
4	-25.80	0	25.80	26.06
5	0	- 8.01	8.01	8.34
6	- 2.19	0	2.19	2.36
7	0	- 7.01	7.01	7.65
8	- 6.75	0	6.75	7.56
9	0	0.85	0.85	1.67
1 2 8	14.06	- 4.08	14.64	14.52
3	21.37	12.60	24.81	25.05
4	- 3.00	- 5.91	6.63	6.44
5	- 5.00	1.66	5.27	5.32
6	9.91	6.17	11.67	11.02
7	-20.96	-10.40	23.39	23.92
8	2.18	1.81	2.83	3.37
9	0.43	4.61	4.63	4.54
2 2 8	4.99	0	4.99	5.37
3	32.69	- 2.96	32.82	33.04
4	3.58	-10.82	11.39	11.17
5	4.04	- 3.57	5.39	5.57
6	- 6.98	- 0.66	7.01	7.20
7	-29.28	1.89	29.34	29.47
8	- 7.34	6.89	10.06	9.57
3 3 8	-19.77	0	19.77	20.39
4	8.81	16.55	18.75	18.21
5	- 8.85	10.54	13.76	14.12
6	- 0.04	- 4.20	4.20	4.71
7	10.94	- 2.69	11.26	11.81
8	- 4.00	- 7.69	8.67	8.61
9	1.40	0.07	1.40	2.63
4 4 8	5.47	0	5.47	6.41
5	13.97	5.20	14.91	15.01
6	- 5.25	- 7.19	8.90	8.93
7	- 1.16	5.95	6.07	6.08
8	9.13	- 0.92	9.17	8.88
9	-10.88	- 1.30	10.95	10.60
5 5 8	14.94	0	14.94	14.92
6	- 7.67	- 5.25	9.30	9.46
7	10.71	9.00	13.99	14.48
8	- 8.30	4.86	9.62	9.52
6 6 8	8.67	0	8.67	8.87
7	10.04	- 2.60	10.37	10.14
7 7 8	1.28	0	1.28	1.76
0 2 9	-22.79	-22.79	32.24	31.07
3	-10.76	10.76	15.22	15.79
4	4.56	4.56	6.45	6.67
6	6.42	6.42	9.08	8.40
7	9.64	- 9.64	13.63	13.55
8	- 2.44	- 2.44	3.45	3.97
9	2.60	- 2.60	3.67	4.10
1 2 9	- 5.29	- 0.03	5.29	5.13
3	- 1.46	-17.48	17.54	17.65
4	- 8.89	1.56	9.03	9.00
5	- 9.00	-14.88	17.39	17.81
6	- 1.53	9.64	9.76	9.22
7	- 1.49	16.36	16.43	16.73
8	- 1.58	- 6.06	6.26	6.17
9	0.34	5.22	5.23	5.00
2 2 9	0	- 8.59	8.59	8.75
3	15.78	16.16	22.58	22.72
4	-13.61	7.51	15.55	15.33
5	-19.73	- 9.29	21.81	22.43
6	- 6.13	5.96	8.55	8.53
7	-10.85	- 7.25	13.05	13.54
8	3.91	0.77	3.98	3.83
9	8.68	0.98	8.74	9.18
3 3 9	0	5.42	5.42	7.52
4	- 7.33	- 2.96	7.91	7.98
5	- 2.33	15.90	16.07	16.88
6	13.29	-12.67	18.36	17.89
7	- 9.14	- 0.55	9.16	9.30
8	- 3.28	- 7.04	7.77	7.33
9	3.50	- 1.38	3.76	3.63
4 4 9	0	-13.53	13.53	13.58
5	- 4.49	- 4.64	6.46	6.03
6	- 6.09	0.98	6.17	6.50
7	-10.60	0.38	10.60	11.22
8	- 3.96	11.87	12.52	14.23
5 5 9	0	16.73	16.73	16.86
6	-11.84	- 2.92	12.19	12.42
7	- 4.76	-10.27	11.32	11.65
8	- 1.47	2.28	2.72	2.40
6 6 9	0	1.18	1.18	1.94
7	7.34	2.48	7.75	8.04
0 2 10	0	7.54	7.54	7.21
3	-19.91	0	19.91	20.27
4	0	- 1.66	1.66	2.78
5	19.63	0	19.63	20.19
6	0	- 9.55	9.55	9.80
7	14.01	0	14.01	14.56
8	0	8.67	8.67	9.50
9	- 4.70	0	4.70	5.22
1 2 10	- 2.11	-16.72	16.85	16.06
3	- 7.76	- 5.74	9.66	9.41
4	0.94	5.94	6.01	6.24
5	-16.35	5.09	17.12	17.29
6	2.06	8.53	8.78	8.55
7	5.43	- 0.38	5.44	5.62
8	9.92	- 2.90	10.33	10.12
9	5.37	- 4.07	6.74	6.85
2 2 10	-33.66	0	33.66	33.30
3	- 4.96	4.25	6.53	6.76
4	- 1.95	5.66	5.99	5.95
5	0.97	-12.22	12.26	12.24
6	13.97	0.95	14.00	13.11
7	7.38	- 0.50	7.40	7.29
8	2.44	- 3.15	3.99	3.65
9	3.34	0.92	3.46	3.88
3 3 10	-24.67	0	24.67	24.95
4	15.23	6.19	16.44	17.12
5	- 8.90	- 9.55	13.05	12.96
6	7.56	- 0.11	7.56	7.50
7	14.30	3.53	14.73	15.21
8	-10.33	- 2.76	10.69	10.53
9	4.27	6.03	7.39	7.15
4 4 10	- 6.33	0	6.33	6.72
5	-11.30	3.64	11.87	11.88
6	- 7.16	7.22	10.17	10.34
7	- 8.52	4.67	9.72	10.31
8	4.30	- 6.06	7.43	7.86
5 5 10	7.69	0	7.69	8.19
6	- 1.10	- 6.73	6.82	6.37
7	1.15	- 3.69	3.86	3.56
8	- 2.68	1.71	3.18	3.46
6 6 10	- 7.85	0	7.85	8.14
6 7 10	-11.26	- 3.01	11.65	11.34
0 3 11	3.93	3.93	5.55	5.60
4	5.88	- 5.88	8.32	7.87
5	12.29	12.29	17.37	16.89
6	-11.05	11.05	15.63	15.25
7	- 5.03	- 5.03	7.12	7.06
8	- 4.09	4.09	5.79	5.51
9	- 2.78	- 2.78	3.93	4.59
1 2 11	10.80	13.62	17.38	16.48
3	- 2.52	-13.34	13.57	12.86
4	- 8.76	5.86	10.54	10.97
5	11.44	- 0.25	11.44	11.33
6	0.61	- 2.91	2.98	2.81
7	3.94	4.43	5.93	5.89
8	5.47	- 4.40	7.02	7.12
9	- 6.61	0.57	6.63	6.72
2 2 11	0	4.79	4.79	5.05
3	5.04	0.90	5.12	5.25
4	8.27	5.51	9.93	9.80
5	12.65	- 9.27	15.68	16.06
6	1.63	- 6.13	6.35	6.24
7	0.45	- 1.92	1.97	2.00
8	4.90	6.86	8.43	9.12
9	- 3.61	0.21	3.62	3.68
3 3 11	0	- 9.84	9.84	9.67
4	8.27	2.53	8.64	8.79
5	2.24	15.21	15.38	15.66
6	3.02	- 5.34	6.13	6.12
7	0.36	9.20	9.20	8.91
8	- 2.14	- 4.39	4.88	4.55
4 4 11	0	3.53	3.53	3.57
5	- 9.58	- 5.45	11.02	11.21
6	9.64	- 3.75	10.35	10.23
7	11.27	- 1.91	11.43	10.99
8	3.79	1.10	3.95	3.76
5 6 11	3.85	- 1.96	4.32	4.21
7	3.22	- 3.50	4.75	5.66
6 6 11	0	10.20	10.20	10.12
7	0.85	2.00	2.17	2.23
0 3 12	0	15.52	15.52	15.07
4	-13.88	0	13.88	13.94
5	0	6.30	6.30	6.98
6	- 3.19	0	3.19	3.45
7	0	- 7.18	7.18	6.32
8	- 4.77	0	4.77	5.38
1 2 12	6.11	- 2.79	6.72	6.63
3	10.66	3.22	11.13	10.72
4	- 0.19	- 8.45	8.46	8.80
5	- 4.22	- 0.47	4.24	3.84
6	2.20	4.16	4.71	4.78
7	-10.21	- 8.11	13.04	12.86
9	- 0.70	1.44	1.60	2.04
2 2 12	1.67	0	1.67	2.07
3	18.84	- 2.70	19.04	19.06
4	1.94	- 6.57	6.86	6.65
5	3.15	- 2.78	4.21	3.95
6	- 3.23	11.47	11.92	11.41
7	-12.79	2.18	12.98	13.42
8	- 4.81	4.25	6.42	6.00
3 3 12	-12.64	0	12.64	12.09
4	4.04	7.76	8.75	9.05
5	- 5.06	2.22	5.52	5.78
6	- 2.88	- 3.45	4.50	4.61
7	5.27	- 5.19	7.39	7.01
8	- 5.29	- 0.48	3.33	3.06
4 4 12	7.41	0	7.41	7.50
5	7.64	- 0.34	7.65	7.98
6	- 1.99	- 4.42	4.84	4.76
7	0.37	2.83	2.85	3.16
5 5 12	6.02	0	6.02	6.28
6	- 1.95	- 4.72	5.11	5.23
7	5.99	5.58	8.19	7.98
6 6 12	5.66	0	5.66	5.46

Reflections not used in final refinement cycles

4 4 7 *	0		0.07	0.60
0 5 9 *	- 0.66	0.66	0.93	1.06
5 5 11*	0	0.13	0.13	1.40
0 9 12*	0	1.61	1.61	1.28
1 8 12*	- 0.26	- 0.17	0.31	0.96
0 4 1 *	- 1.03	- 1.03	1.46	0.85
0 9 5 *	0.08	- 0.08	0.12	1.32
0 4 0**	-118.82	0	118.82	89.22
0 2 1**	-98.32	-98.32	139.05	79.01

* Accidentally absent. Statistical value used for F_o

** Subject to extinction

parameters are notably different however, they are probably the result of our use of Mo$K\alpha$ radiation with a smaller crystal for which absorption corrections were unnecessary. Thermal motion data based on our results is given in Table 2. Copies of tables of interatomic distances and angles may be obtained by application to us; observed and calculated structure factors are given in Table 3.

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Thermal-vibration analysis	K. W. MUIR

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A. C. T. NORTH, D. C. PHILLIPS and F. SCOTT MATHEWS (1968), A semi-empirial method of absorption correction. Acta Crystallogr. A **24**, 352—359.