International Tables for X-Ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.
KURLAND, R. J. & WILSON, E. B. (1957). *J. Chem. Phys.* **27**, 585.
LIDE, D. (1962). *Tetrahedron*, **17**, 125.
LIMINGA, R. & OLOVSSON, I. (1964). *Acta Polytech. Scand., Math. Computing Mach. Ser.* No 10.
LINDQVIST, I. (1963). *Inorganic Adduct Molecules of Oxo-compounds*. Berlin: Springer Verlag.
LINDQVIST, I. & ZACKRISSON, M. (1960). *Acta Chem. Scand.* **14**, 453.
NARDELLI, M., CAVALCA, L. & FAVA, G. (1957). *Gazz. chim. Ital.* **87**, 1232.
NARDELLI, M., COGHI, L. & AZZONI, G. (1958). *Gazz. chim. Ital.* **88**, 235.
ROSENHEIM, A. & STELLMANN, W. (1901). *Ber. dtsch chem. Ges.* **34**, 3377.
VILKOV, L. V., AKISHIN, P. A. & PRESNYAKOVA, V. M. (1962). *Zh. Strukt. Khim.* **3**, 5.
ZACKRISSON, M. (1961). *Acta Chem. Scand.* **15**, 1784.
ÅSBRINK, S. & BRÄNDÉN, C.-I. (1963). I.U.Cr. *World List of Crystallographic Computer Programs, No* 6023 *FACIT*.

Acta Cryst. (1966). **20**, 758

A Reinvestigation of the Crystal Structure of $LiIO_3$*

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The crystal structure of $LiIO_3$ has been reinvestigated with the use of single-crystal Mo $K\alpha$ diffractometer intensity data. Morphological measurements and diffraction photographs establish $P6_3$ to be the correct space group. The values for the lattice constants, $a_0 = 5{\cdot}1815$ and $c_0 = 5{\cdot}1709$, were determined by a least-squares fit of back-reflection powder lines. The structure consists of lithium ions, surrounded by six oxygen atoms in a distorted octahedral arrangement, and discrete trigonal iodate groups with a 1·817 Å iodine–oxygen bond distance and 98·65° O–I–O bond angle.

Introduction

As a result of discussions concerning naturally occurring iodates (Kaplan, 1966), we reviewed the literature on the configuration of iodate groups in crystalline solids. It was noted that the structure of $LiIO_3$ reported by Zachariasen & Barta (1931) (hereafter ZB) placed the iodine in an octahedral coordination with six equidistant oxygens, rather than in trigonal, pyramidal configuration as in other iodates. That the structure might merit reinvestigation was pointed out by Wyckoff (1960). This anomalous behavior of iodine in several alkali metal iodates has also been noted by Ibers (1956). On examination of the original paper of ZB, it became apparent that the structure was interpreted largely from powder diffraction data, and that the crystals available were extremely small. It is then not too surprising that an incorrect space group was chosen and erroneous positions were deduced for the lighter atoms. Since occasionally discrete molecules rearrange in the solid state when crystallized from gaseous or liquid state, (*e.g.* gaseous Al_2Cl_6 and infinite molecular sheets in crystalline $AlCl_3$), the structure of $LiIO_3$ merits reinvestigation.

Our structure results are reported here.

Experimental

Crystals of lithium iodate were prepared in the following manner: (1) stoichiometric quantities of reagent grade iodic acid and lithium carbonate were dissolved in water; (2) the solution was then warmed to remove any remaining carbon dioxide; (3) the solution was allowed to evaporate at room temperature until crystals began to form.

The crystals obtained are brilliant pseudo-prisms up to 0·5 mm long and 0·2 mm in diameter. Careful examination reveals that the apparent prisms are actually portions of extremely steep hexagonal pyramids. Morphological measurements made with a reflecting goniometer yielded results which permit the indexing of all but the very steep faces. The polar angle of these steep faces is so great that indexing becomes ambiguous. The approximate index of these faces, orienting them so that they taper toward the negative end of **c**, is $\{20\cdot0\cdot\overline{20}\cdot\bar{1}\}$. These are terminated by the forms $\{10\bar{1}1\}$, $\{10\bar{1}\bar{1}\}$, and very small faces of $\{12\bar{3}\bar{2}\}$. Mor-

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phological measurements are listed in Table 1. The morphology is illustrated in Fig. 1 and is characteristic of the point group 6 (C_6) rather than 622 (D_6) as reported by ZB.

Table 1. *Morphological data for* $LiIO_3$

Form	φ	ϱ
$\{10\bar{1}1\}$	30°	47°27′
$\{10\bar{1}\bar{1}\}$	30°	−47°27′
$\{20\ 0\ \bar{20}\ \bar{1}\}$	30°	−87°20′
$\{12\bar{3}\bar{2}\}$	49°05′	−55°13′

The polar character of the *c* axis is further established by the piezoelectric property of the crystals. Crystals which were held between two copper strips develop a positive polarity at the larger end upon compression.

The crystals of $LiIO_3$ dissolve slowly in water, and microscopic observation shows that the ends of the crystals dissolve at different rates and in different ways. The form $\{10\bar{1}1\}$ etches and rounds rapidly, while the form $\{10\bar{1}\bar{1}\}$ disappears and is replaced by sharp faces of $\{11\bar{2}\bar{1}\}$. The appearance of the crystal after about 15 seconds in water at 20°C is shown in Fig. 2. Broken fragments display similar behavior.

The room-temperature unit-cell dimensions for $LiIO_3$ were obtained by least-squares fit of 12 back-reflection powder lines obtained in a 114·5 mm Norelco powder camera with Cr $K\alpha$ radiation. These values (λ for Cr $K\alpha_1 = 2{\cdot}28962$ Å), $a_0 = 5{\cdot}4815 \pm 3$*, $c_0 = 5{\cdot}1709 \pm 4$, compare favorably with previous values of $a_0 = 5{\cdot}469 \pm 3$, $c_0 = 5{\cdot}155 \pm 3$ (ZB) and $a_0 = 5{\cdot}481$, $c_0 = 5{\cdot}172$ (NBS, 1957). Systematic absence of $000l$ reflections except for $l = 2n$, the morphology, and physical properties establish the space group $P6_3$.

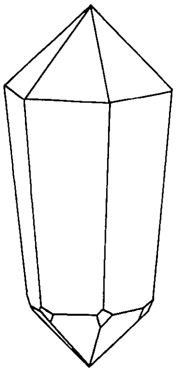

Fig. 1. Morphology of $LiIO_3$ grown from a saturated water solution.

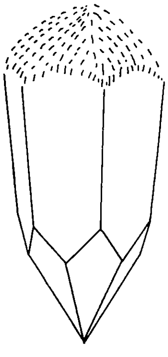

Fig. 2. The modified morphology of water-etched $LiIO_3$ crystals.

From typical crystal specimens described above, a pseudo-prism, approximately 0·08 mm maximum diameter and 0·32 mm length, was selected for intensity measurements and mounted on a Pyrex rod with the *c* axis parallel to the axis of the rod. A Picker diffractometer equipped with a Datex semiautomatic remote control system, an Electronics and Alloys full-circle crystal orienter, a scintillation counter and a Mo X-ray tube were used for the intensity measurements. Intensities were measured at room temperature by the stationary-crystal stationary-counter technique with balanced Zr and Y filters for 257 independent reflections, of which 14 were considered unobservable and assigned a minimum value (3 times the square root of the sum transmitted by the Zr and Y filters for the background count). A small correction was added to the background (Y filter) count which was obtained by measuring the 'band width' passed (Compton scattering, thermal diffuse scattering and parasitic scattering) by the filter pair for a particular 2θ value (Burbank, 1965). The data were corrected for Lorentz and polarization factors. The absorption coefficient for Mo $K\alpha$ is 117 cm^{-1}; hence absorption corrections are quite small. However, cylindrical absorption corrections approximating the pseudo-prism were applied; the values of the absorption factors ranged from 2·07 to 2·25. Structure factors were calculated with the use of the lithium, oxygen and iodine scattering factors given in *International Tables for X-ray Crystallography* (1962, Tables 3.3.1A and 3.3.1B). Iodine dispersion corrections ($\Delta f' = -0{\cdot}6$; $\Delta f'' = 2{\cdot}2$) were included by the method of Templeton (1955) in all later stages of refinement. Calculations were carried out on CDC 1604 and 3600 computers.

Structure determination

The distribution of intensities ($h\,h\,\bar{2h}\,l$ with $l = 2n+1$ very weak and $h0\bar{h}l$ all about equal except when $h = 3$) suggests that iodine atoms occupy (*b*) sites in space group $P6_3$. These positions are equivalent to those reported by ZB. A three-dimensional Fourier synthesis was calculated with the use of phases determined by the iodine atoms located in (*b*) sites with *z* arbitrarily set at zero in all calculations. This Fourier synthesis indicated locations for the oxygen atoms as well as a set of centrosymmetrically related 'ghosts'. Refinement of the three oxygen positional and two isotropic thermal parameters as well as the one scale factor by least-squares methods [unit weights were assigned all reflections except for unobserved reflections

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* Throughout this paper, the value of the error corresponds to the last significant digits in the function value.

with $F_{\text{obs}} > F_{\text{calc}}$ and the 110 reflection, which were assigned zero weights; the function minimized was $\Sigma\, w(|F_{\text{obs}}| - |F_{\text{calc}}|)^2]$ resulted in a discrepancy index, $R = \Sigma\, ||F_{\text{obs}}| - |F_{\text{calc}}|| / \Sigma\, |F_{\text{calc}}|$, of 0·057. A three-dimensional difference synthesis calculated with the above refined parameters suggested two possible locations for the lithium atom (in (*a*) sites with $z=0{\cdot}095$ or $0{\cdot}905$). It somewhat surprised the authors that the ghosts for the Li position should remain after inclusion of the oxygen atoms. Prior to any further attempt at refinement, the environment for a lithium atom at $z=0{\cdot}095$ (model *A*) would consist of 2 sets of 3 oxygen atoms at about 1·83 and 2·78 Å while that for a lithium atom at $z=0{\cdot}905$ (model *B*) would consist of 2 sets of 3 oxygen atoms at about 2·10 and 2·16 Å.

Least-squares refinement of positional and anisotropic thermal parameters for iodine and oxygen atoms for models *A* and *B* (an isotropic thermal parameter was used for the lithium atom in both cases) did not yield significantly different discrepancy indices. In both cases refinement of lithium atom positions resulted in

Table 2. *Positional and thermal parameters for* $LiIO_3$

	Li	I	O
x	0·0	$\frac{1}{3}$	0·09358 ± 0·00214*
y	0·0	$\frac{2}{3}$	0·34396 ± 0·00206
z	0·8907 ± 0·0123	0·0	0·16980 ± 0·00199
B or B_{11}	2·0	0·833 ± 0·05	1·190 ± 0·31
B_{22}		B_{11}	0·985 ± 0·30
B_{33}		0·853 ± 0·03	1·157 ± 0·27
B_{12}		$\frac{1}{2}B_{11}$	0·180 ± 0·26
B_{13}			0·541 ± 0·29
B_{23}			0·419 ± 0·26

* Standard deviations calculated from least-squares residuals.

Table 3. *Observed and calculated structure factors*

An asterisk denotes reflections assigned minimum observed value.

h	k	l		$10F_o$	$10F_c$	10A	10B
1	0	0		406	409	-409	0
2	0	0		459	475	-475	0
3	0	0		708	749	749	0
4	0	0		278	280	-280	0
5	0	0		241	246	-246	0
6	0	0		355	340	340	0
7	0	0		160	160	-160	0
8	0	0		96	97	-97	0
1	1	0		637	750	750	0
2	1	0		387	402	-402	0
3	1	0		352	352	-352	0
4	1	0		443	486	486	0
5	1	0		129	136	-136	0
6	1	0		145	152	-152	0
7	1	0		239	247	247	0
1	2	0		190	201	-201	0
2	2	0		633	671	671	0
3	2	0		249	266	-266	0
4	2	0		137	137	-137	0
5	2	0		350	369	369	0
6	2	0		151	147	-147	0
1	3	0		152	143	-143	0
2	3	0		242	256	-256	0
3	3	0		371	397	397	0
4	3	0		158	175	-175	0
5	3	0		154	158	-158	0
6	3	0		221	215	215	0
1	4	0		480	461	461	0
2	4	0		194	217	-217	0
3	4	0		157	174	-174	0
4	4	0		254	265	265	0
5	4	0		100	97	-97	0
1	5	0		173	186	-186	0
2	5	0		354	392	392	0
3	5	0		111	119	-119	0
4	5	0		114	114	-114	0
1	6	0		137	135	-135	0
2	6	0		101	110	-110	0
3	6	0		207	207	207	0
1	7	0		218	224	224	0
1	0	1		889	838	-81	835
2	0	1		632	613	-156	-593
3	0	1		14	11	10	-3
4	0	1		476	461	1	461
5	0	1		368	345	-56	-340
6	0	1		35	18	17	-7
7	0	1		226	221	32	218
8	0	1		191	184	-2	-184
1	1	1		224	185	-161	91
2	1	1		628	632	-45	630
3	1	1		575	570	112	-559
4	1	1		35	28	24	-13
5	1	1		346	328	3	328
6	1	1		274	275	19	-275
7	1	1	*	23	5	-5	1
1	2	1		591	595	-10	-595
2	2	1		49	42	36	-22
3	2	1		413	417	10	417
4	2	1		352	346	-9	-346
5	2	1		24	5	-5	2
6	2	1		241	242	-31	240
1	3	1		485	500	-2	500
2	3	1		418	401	-55	-397
3	3	1		68	60	-53	29
4	3	1		281	286	17	285
5	3	1		236	237	5	-237
6	3	1	*	24	11	-9	7
1	4	1		43	43	39	-18
2	4	1		341	366	-24	365
3	4	1		292	298	11	-298
4	4	1		45	41	36	-20
5	4	1		194	189	8	189
1	5	1		327	349	33	-347
2	5	1		30	3	3	-2
3	5	1		225	235	-1	235
4	5	1		197	189	-14	-188
1	6	1		257	268	-13	268
2	6	1		222	222	-2	-222
3	6	1		27	26	-22	13
1	7	1	*	23	14	12	-6
0	0	2		730	711	667	246
1	0	2		428	405	-405	-6
2	0	2		343	320	-307	-89
3	0	2		622	585	584	21
4	0	2		263	233	-231	-30
5	0	2		209	190	-186	-39
6	0	2		370	338	337	-19
7	0	2		127	120	-117	-24
1	1	2		772	801	793	-114
2	1	2		320	296	-287	-70
3	1	2		261	241	-230	-71
4	1	2		500	446	446	-19
5	1	2		225	208	-206	28
6	1	2		148	143	-143	-7
7	1	2		242	228	227	-1
1	2	2		407	379	-374	63
2	2	2		579	533	533	10
3	2	2		234	219	-217	-34
4	2	2		237	220	-217	38
5	2	2		322	300	299	22
6	2	2		115	114	-112	-18
1	3	2		362	332	-322	79
2	3	2		245	221	-220	-26
3	3	2		446	411	410	-32
4	3	2		161	152	-152	-11
5	3	2		125	120	-117	-22
1	4	2		475	465	464	-34
2	4	2		181	184	-182	-26
3	4	2		167	159	-159	-15
4	4	2		287	276	275	-21
5	4	2		115	117	-116	8
1	5	2		181	178	-178	-8
2	5	2		288	285	282	38
3	5	2		139	139	-139	8
4	5	2		104	104	-104	-4
1	6	2		144	147	-147	9
2	6	2		131	130	-129	13
1	7	2		253	242	241	-19
1	0	3		571	535	6	535
2	0	3		652	640	10	-640
3	0	3		25	8	-5	7
4	0	3		432	391	-1	391
5	0	3		403	376	0	-376
6	0	3		24	17	-7	16
7	0	3		244	243	-1	243
1	1	3		118	110	3	-110
2	1	3		472	453	-3	453
3	1	3		348	326	-7	-326
4	1	3		26	22	-2	22
5	1	3		304	290	-2	290
6	1	3		229	213	4	-213
7	1	3	*	24	7	6	-4
1	2	3		538	496	-2	-496
2	2	3		39	31	2	30
3	2	3		416	377	8	377
4	2	3		338	312	3	-312
5	2	3	*	21	5	1	-5
6	2	3		174	169	4	169
1	3	3		462	417	-1	417
2	3	3		441	415	4	-415
3	3	3		60	50	3	-50
4	3	3		291	273	-5	273
5	3	3		209	206	-5	-206
1	4	3		39	35	-9	34
2	4	3		304	281	-5	280
3	4	3		271	248	-2	-248
4	4	3		40	37	0	37
1	5	3		273	255	-1	-255
2	5	3	*	21	3	0	3
3	5	3		220	210	5	210
1	6	3		230	221	4	221
2	6	3		210	204	-2	-204
1	7	3	*	24	12	-1	12
0	0	4		561	516	494	-150
1	0	4		320	307	-306	-18
2	0	4		264	260	-258	32
3	0	4		456	433	432	-28
4	0	4		212	206	-206	13
5	0	4		166	158	-157	20
6	0	4		280	276	275	10
7	0	4		106	104	-103	17
1	1	4		575	545	544	43
2	1	4		250	239	-237	24
3	1	4		204	206	-203	36
4	1	4		379	360	360	-1
5	1	4		170	167	-163	-37
6	1	4		123	123	-123	0
1	2	4		307	283	-276	-64
2	2	4		440	417	416	-28
3	2	4		195	186	-186	13
4	2	4		190	187	-183	-39
5	2	4		250	241	240	-21
6	2	4		95	104	-103	14
1	3	4		288	266	-257	-70
2	3	4		194	191	-191	9
3	3	4		339	323	322	20
4	3	4		148	141	-140	9
5	3	4		105	106	-105	17
1	4	4		395	358	358	18
2	4	4		162	152	-151	9
3	4	4		139	129	-129	2
4	4	4		228	232	231	12
1	5	4		159	153	-153	-1
2	5	4		256	245	241	-42
3	5	4		122	117	-116	-13
1	6	4		144	136	-136	-8
2	6	4		124	119	-119	-11
1	0	5		437	420	19	420
2	0	5		365	343	46	-339
3	0	5		19	6	-1	-6
4	0	5		294	294	1	294
5	0	5		229	234	34	-231
6	0	5		27	15	-6	-14
1	1	5		59	58	49	31
2	1	5		373	362	19	361
3	1	5		343	353	-48	-350
4	1	5	*	20	16	-13	-10
5	1	5		218	223	-1	223
6	1	5		175	191	-17	-190
1	2	5		368	352	5	352
2	2	5	*	19	20	-18	-8
3	2	5		286	283	-12	283
4	2	5		230	232	3	-232
5	2	5	*	23	4	3	3
1	3	5		331	312	1	312
2	3	5		263	259	28	-258
3	3	5		42	38	31	23
4	3	5		191	193	-6	193
1	4	5		31	26	-15	-21
2	4	5		254	243	19	242
3	4	5		205	211	-6	-211
1	5	5		246	241	-21	-240
2	5	5		26	3	-2	-1
1	6	5		191	195	5	195
0	0	6		463	464	463	17
1	0	6		195	187	-187	6
2	0	6		213	206	-206	3
3	0	6		326	341	341	1
4	0	6		157	156	-156	-4
5	0	6		136	145	-145	4
1	1	6		332	331	331	4
2	1	6		194	193	-193	6
3	1	6		176	183	-183	0
4	1	6		246	257	257	9
5	1	6		86	89	-88	12
1	2	6		151	137	-136	15
2	2	6		312	320	320	11
3	2	6		158	151	-151	2
4	2	6		90	88	-88	3
1	3	6		117	105	-105	6
2	3	6		153	146	-146	0
3	3	6		204	221	220	-3
1	4	6		246	247	247	-3
2	4	6		131	131	-130	6
1	5	6		117	113	-113	2
1	0	7		263	270	-14	270
2	0	7		231	244	-34	-242
3	0	7	*	22	5	4	3
4	0	7		185	207	1	207
1	1	7		44	34	-31	15
2	1	7		238	248	-10	248
3	1	7		215	233	38	-230
4	1	7	*	24	11	11	-4
1	2	7		228	237	-2	-237
2	2	7	*	22	13	10	-8
3	2	7		177	185	1	185
1	3	7		209	218	-1	218
2	3	7		167	190	-23	-188
1	4	7	*	24	18	18	1
0	0	8		224	226	223	40
1	0	8		120	123	-123	6
2	0	8		111	106	-106	-9
3	0	8		189	215	215	13
1	1	8		236	252	251	-13
2	1	8		102	104	-104	-10
1	2	8		124	129	-128	17
2	2	8		202	199	199	6

a shortening of the shorter Li–O separations given above. Refinement of model A results in a lithium parameter of z equal to 0·1286 (with Li–O values of 1·71 and 2·78 Å); fractional positional parameters for oxygen differed by 1×10^{-5} and B_{ij} (oxygen) differed by a maximum of 0·1σ from values for model B listed in Table 2. However, one may rule out model A by consideration of the sum of ionic radii (using 0·60 Å for Li^+ and 1·32 to 1·40 Å for O^{2-}; Pauling, 1960).

The final positional and thermal parameters (Table 2) result in a discrepancy index of 0·049. Anisotropic thermal parameters, B_{ijn}, are of the form $\exp\left(-\frac{1}{4}\sum_{i=1}^{3}\sum_{j=1}^{3} B_{ijn}h_ih_ja^*_ia^*_j\right)$. Table 3 lists the observed and calculated structure factors scaled by 10 times as indicated. Table 4 lists the interatomic separations and bond angles. Other reported I–O bond lengths and O–I–O angles have been summarized by Ibers (1956), and range from acceptable values of 1·77 to 1·86 (average 1·82) found in $NaIO_3$ (van Eck and MacGillavry, 1943), $Ce(IO_3)_4$ (Cromer & Larson, 1956), $Ce(IO_3)_4 . H_2O$ (Ibers, 1956), and HIO_3 (Rogers & Helmholz, 1941) to improbable values of 2·08 Å (Naray-Szabo & Neugebauer, 1947) or larger (suggested by octahedral iodine in the iodates of cesium, rubidium and ammonium ion).

Table 4. *Interatomic separations in* $LiIO_3$

	Separations	Standard deviation
Li–O	2·038, 2·220 Å	0·06 Å
I–O (bonded)	1·817	0·017
I–O (next neighbor)	2·873	0·017
O–O (intra IO_3^- groups)	2·756	0·024
O–O (inter IO_3^- groups)	2·925, 3·085	0·024
O–I–O	98·65°	0·6°

It is unfortunate in a crystal whose space group is unambiguously established that the iodine atoms are related to each other by a point of inversion, and hence one is not able to utilize their dispersion to resolve the absolute configuration of the packing.

References

Burbank, R. D. (1965). *Acta Cryst.* **18**, 88.

Cromer, D. T. & Larson, A. C. (1956). *Acta Cryst.* **9**, 1015.

Eck, P. van & MacGillavry, C. H. (1943). *Rec. Trav. chim. Pays-Bas*, **62**, 729.

Ibers, J. A. (1956). *Acta Cryst.* **9**, 225.

International Tables for X-Ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

Kaplan, S. (1966). Doctoral dissertation, University of New Mexico.

Naray-Szabo, I. & Neugebauer, J. (1947). *J. Amer. Chem. Soc.* **69**, 1280.

NBS (1957). Circular 539, **7**, 26.

Pauling, L. (1960). *The Nature of the Chemical Bond,* 3rd ed. p. 514 f. Ithaca: Cornell Univ. Press.

Rogers, M. T. & Helmholz, L. (1941). *J. Amer. Chem. Soc.* **63**, 278.

Templeton, D. H. (1955). *Acta Cryst.* **8**, 842.

Wyckoff, R. W. G. (1960). *Crystal Structures,* Ch. VII, paragraph *a*12. New York: Interscience Publishers.

Zachariasen, W. H. & Barta, F. A. (1931). *Phys. Rev.* **37**, 1626.

Acta Cryst. (1966). **20**, 761

Cation Distribution and Oxygen Parameter in Magnesium Gallate, $MgGa_2O_4$

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Magnesium gallate, $MgGa_2O_4$, a partially inverted spinel, belongs to space group $Fd3m$ with $a_0 = 8{\cdot}286 \pm 0{\cdot}003$ Å. The distribution of the di- and trivalent cations on two possible sites, 8(*a*) and 16(*d*), has been determined by the least-squares method. The data used in the refinement were obtained from single crystals by film and counter techniques; the counter data were collected manually and by CCXD, a computer-controlled X-ray diffractometer.

The magnesium ions are distributed between the two sites as follows: 16 ± 2 % in the tetrahedral A site and 81 ± 1 % in the remaining octahedral B site. The oxygen parameter as refined in the present study is 0·3824 ± 0·0003, indicating the usual shift for the inverted spinel from the ideal value of 3/8.

Introduction

Magnesium gallate, $MgGa_2O_4$, is a partially inverted spinel with space group $Fd3m$. The metal ions fall on sets of special positions 8(*a*) and 16(*d*), frequently referred to as the A and B sites respectively. The oxygen ions occupy a set of special positions 32(*e*) with one positional parameter to be determined.

Interest in this compound developed through electron spin resonance studies of Cu^{2+} conducted at these Laboratories (Overmeyer, 1963) using magnesium gallate as a host lattice. A substantial broadening is ob-