In order to explore just what might have happened in the original investigation, we then used the original NS parameters as a starting point in another refinement, keeping the value of B, however, at 3·33. The first cycle of this series gave $R=8\cdot3\%$ and $\Sigma w\varDelta^2 = 15\cdot49$. This series converged to a set of positional parameters which was not significantly different from the starting point, but, to our great surprise, was quite different from those obtained in our previous refinement. Moreover, the final values of R and $\Sigma w\varDelta^2$, viz. 8·7% and 14·34, are both startlingly greater than the values of 3·9% and 3·11 obtained above. The parameters and molecular quantities are presented in Tables 1 and 2.

It is thus apparent that when the coordinates I of Table 1 are used as a starting point for least squares, that procedure refines to a different answer than when coordinates III are used as a starting point.

As an after-the-fact analysis as to *how* this could have happened (as opposed to *why*, a very different question) we have examined the F_{calc} obtained from the two refinements. Of the 53 F_{calc}, 27 are centric and 26 are acentric; among the former, there is one sign change, that of 004, and among the latter there is one significant phase change, caused by a change in the sign of the imaginary part of 134. For the remainder of the acentric F's, the phase changes average about 8°. Moreover, the $\varDelta F$'s for what we call the incorrect structure do not, to us, contain a clue which might suggest that something was awry. The average $\varDelta F$ is

0·8, with no outstanding discrepancies, the largest value being 4·2. The largest percentage discrepancies occur in the case of 004, 034, and 134, which are 35%, 38%, and 43%, respectively. For the correct structure, the discrepancies are 1%, 22%, and 7%, but there is of course nothing to imply that the sign of 004 is incorrect, or that the sign of 034 is not incorrect. It might be suggested that if, in the final stages of a refinement, there remain F's for which the discrepancies are outside some externally estimated limit, then those F's should be omitted, and the refinement continued. This procedure seems to us a dangerous one, and we do not recommend it.

Meanwhile, we find the occurrence of a false minimum which is quite close to the true minimum rather disturbing.

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The Crystal Structure of Y2BeO4*

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The crystal structure of an yttrium beryllium oxide compound has been determined by three-dimensional Patterson and difference Fourier methods and refined by iterative least-squares calculations. The structure analysis showed the composition of the compound to be represented by Y_2BeO_4. The unit-cell dimensions are $a_0=3\cdot5315\pm0\cdot0005$, $b_0=9\cdot8989\pm0\cdot0010$, and $c_0=10\cdot4000\pm0\cdot0010$ Å; the cell contains four formula weights of Y_2BeO_4 with an X-ray density $\varrho=4\cdot582\pm0\cdot002$ g.cm^{-3}. Within the limits of a linear least-squares refinement of X-ray data the space group is *Pmcn*.

The structure of Y_2BeO_4 is described as consisting of ribbons of interconnected octahedra formed from two pseudo-close-packed layers of oxygen atoms between which yttrium atoms occupy octahedral interstices. The ribbons are five oxygen atoms (four octahedra) wide and extend without limit parallel to the **a** direction. The intersection of these ribbons forms channels which contain beryllium atoms trigonally coordinated to oxygen atoms in a coplanar group.

Introduction

Weir & Van Valkenburg (1960) reported the formation of a compound in the binary system Y_2O_3–BeO by

arc fusion. The composition of the compound was not determined since it could not be formed by solid state reactions. Potter (1960) also synthesized a compound in the same binary system by quenching a 1:2 molar mixture of Y_2O_3 and BeO from the molten state on a platinum strip heater. A comparison of optical and X-ray diffraction data for these two compounds showed them to be identical.

A composition $Y_2Be_2O_5$ was suggested in the latter work on the basis of the absence of a detectable second phase in 1:2 quenched mixtures and the presence of free Y_2O_3 in 1:1 quenched mixtures. A preliminary report by Harris, Potter & Yakel (1962) listed the 1:2 composition, but work by Bartram (1961) had indicated the formula Y_2BeO_4 for this phase. The source of the uncertainty as to composition may be understood in light of the tentative Y_2O_3–BeO phase diagram given by Bartram in which the compound in question is shown to form peritectically from solid Y_2O_3 and liquid. Thus any quench on composition would contain free Y_2O_3. Quantitative chemical analyses of the quenched mixtures are therefore suspect and since, as noted above, a solid-state synthesis could not be effected, the final definition of composition waited on the analysis of the crystal structure.

The purpose of the present paper is to report the structure of this phase. The results show that the composition is represented by the formula Y_2BeO_4 and the compound will be referred to by this formula in the following description, although in reality the question of composition was not settled until relatively late in the structure derivation.

Experimental

Single crystals of Y_2BeO_4 were extracted from the quenched $1Y_2O_3:2BeO$ mixtures; they were clear, colorless, and elongated in the [100] direction. X-ray diffraction data obtained from rotation, Weissenberg, and precession photographs recorded with Cu $K\alpha_1(\lambda = 1\cdot54051$ Å) and Mo $K\alpha_1(\lambda = 0\cdot70926$ Å) radiations con-

firmed the previously reported orthorhombic symmetry of the crystals and the diffraction symbol $mmmP\cdot cn$ (space group $Pmcn$ or $P2_1cn$). The lattice parameters were measured to be $a_0 = 3\cdot5315 \pm 0\cdot0005$, $b_0 = 9\cdot8989 \pm 0\cdot0010$, $c_0 = 10\cdot4000 \pm 0\cdot0010$ Å. An X-ray density of $4\cdot582 \pm 0\cdot002$ g.cm^{-3} was computed on the basis of 4 formula weights of Y_2BeO_4 in this cell. The agreement with the experimental density previously reported (Harris, Potter & Yakel, 1962) is not good; it is possible that the sample used to obtain the high value of $5\cdot1$ g.cm^{-3} was not pure.

All intensity data were collected from an irregularly shaped crystal which approximated a cylinder 0·003 cm in diameter. A Weissenberg goniometer equipped with a proportional counter was first used to measure the intensities of $0kl$, $1kl$, and $2kl$ reflections. A second more extensive set of data was obtained with a computer-controlled four-circle diffractometer (θ–2θ scan) (Busing, Ellison & Levy, 1964, 1965). Over 1000 individual reflection intensities were recorded in the first data set and over 3000 in the second. Nickel-filtered Cu $K\alpha$ radiation was used exclusively in collecting the first set and primarily in collecting the second; about 200 reflection intensities were also measured with Cu $K\beta$ radiation in the collection of the second set. Of the 495 allowed independent reflections accessible with Cu $K\alpha$ radiation, 491 were recorded.

The two sets of intensity data were independently corrected for Lorentz, polarization, and absorption effects. Absorption corrections for the first data set were made assuming cylindrical crystal shape and using Bond's (1959) tables. Absorption corrections for the second set were made by approximating the crystal

Table 1. Atomic parameters for Y_2BeO_4

Least-squares standard errors are given in parentheses

Atom	Fractional position parameter $\times 10^5$			Thermal parameter* $\times 10^5$			
	x	y	z	B_{11}	B_{22}	B_{33}	B_{23}†
Y(1)	25000	67664 (4)	9028 (4)	880 (52)	175 (6)	74 (5)	32 (2)
Y(2)	25000	8626 (4)	11738 (4)	851 (53)	171 (6)	89 (6)	−38 (2)
O(1)	25000	24361 (31)	27699 (34)	1402 (275)	152 (23)	102 (27)	−37 (22)
O(2)	75000	1841 (30)	25044 (33)	1849 (289)	72 (25)	113 (29)	12 (21)
O(3)	75000	11518 (31)	−1297 (34)	988 (291)	182 (29)	72 (30)	−1 (22)
O(4)	25000	35344 (42)	4453 (36)	1625 (327)	569 (37)	79 (33)	−41 (29)
Be	25000	37329 (58)	19369 (69)	2004 (559)	174 (52)	222 (57)	−135 (46)

RMS thermal displacements (Å) $\times 10^3$ along principal axes

Least-squares standard errors are given in parentheses

	Axis 1	Axis 2	Axis 3	Angle 3, b‡
Y(1)	59 (2)	75 (2)	96 (2)	162 (1)°
Y(2)	64 (2)	73 (2)	97 (1)	24 (2)
O(1)	66 (11)	94 (9)	94 (7)	31 (14)
O(2)	59 (11)	108 (8)	80 (10)	103 (22)
O(3)	63 (13)	79 (12)	95 (8)	179 (13)
O(4)	64 (14)	101 (10)	169 (5)	5 (4)
Be	56 (23)	113 (16)	133 (12)	52 (9)

* Coefficients in the expression $B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + 2B_{12}hk + 2B_{13}hl + 2B_{23}kl$.

† $B_{12} = B_{13} = 0$ for $4(c)$ positions of $Pmcn$.

‡ By symmetry one of the axes must be in the a direction; this is axis 2 in all cases. The angle is between axis 3 and the b axis, measured clockwise, looking down the positive a axis.

shape with seven bounding planes and using the ORABS computer program of Wehe, Busing & Levy (1962). The data sets were then collated and intensities of equivalent reflections averaged. Relative $|F|^2$ values were placed on an approximate absolute scale after the method of Wilson (1942). This calculation was performed with the computer program ORSTAT (Yakel, 1964) which also tested the statistical distribution of intensities in the general *hkl* data and in particular zones of data. The distribution in the general *hkl* set was centric. During the refinement of the trial structure, a correction for secondary extinction was made for all data using the method proposed by Zachariasen (1963).

The structure

Derivation

If, as the statistical tests suggest, one assumes the space group of Y_2BeO_4 to be *Pmcn*, then the mirror planes normal to **a** at $x=\frac{1}{4}$ and $\frac{3}{4}$ are only 1·766 Å apart. Considerations of atom size and environment make it most probable that the 8 yttrium and 20 oxygen atoms in each unit cell would lie on the mirrors. Barring disorder, the 4 beryllium atoms would also be constrained to the same planes. This arrangement was supported by the regular decrease in observed $|F|^2$ values for reflections with identical *k* and *l* and $h=0$, 2, and 4, or $h=1$ and 3.

Table 2. *Comparison of observed and calculated* $F^2(hkl)$
E signifies apparent extinction effect on F^2(obs.)
R signifies apparent Renninger effect on F^2(obs.)

	L	CALC	OBS	SIG
•••	0	0	L	•••
	2	330	332	15
E	4	1479	1202	54
	6	1916	1651	75
	8	595	563	27
	10	838	831	40
	12	0	1	0
•••	0	1	L	•••
	1	140	169	8
	2	123	131	6
	3	185	183	9
	4	251	241	12
	5	81	82	4
	6	82	83	4
	7	631	596	29
	8	535	510	25
	9	376	380	19
	10	374	391	19
	11	159	175	9
R	12	6	9	1
	13	306	323	16
•••	0	2	L	•••
	0	56	64	3
	1	1534	1560	71
	2	95	106	5
	3	2516	2339	106
	4	1	3	0
	5	166	170	8
	6	173	162	8
	7	2394	2103	95
	8	329	331	16
	9	163	160	8
	10	83	86	4
	11	200	212	11
	12	221	234	12
	13	109	111	5
•••	0	3	L	•••
	1	1194	1242	56
	2	2414	2370	107
	3	28	33	2
	4	43	43	2
	5	843	825	39
	6	695	658	31
	7	563	559	27
	8	358	350	17
	9	23	22	1
	10	357	376	18
	11	335	342	17
	12	104	116	6
	13	48	49	7
•••	0	4	L	•••
	0	1062	1103	50
	1	20	22	1
	2	745	817	38
	3	108	106	5
	4	642	639	30
	5	356	358	17
	6	9	9	1
	7	1	1	0
	8	53	53	3
	9	888	827	39
	10	188	192	10
	11	242	245	12
	12	33	36	2
	13	336	321	18
•••	0	5	L	•••
	1	2319	2256	101
	2	830	846	40
	3	254	254	12
	4	368	367	18
	5	1649	1540	70
	6	496	477	23
	7	0	1	0
	8	293	286	14
	9	833	782	37
R	10	6	10	1
	11	122	120	6
	12	136	138	7
•••	0	6	L	•••
	0	2	3	1
	1	127	131	7
	2	107	113	6
	3	37	41	2
•••	0	6	L	•••
	4	116	113	6
	5	90	85	4
	6	355	341	17
	7	4	3	1
	8	646	616	30
	9	20	20	1
	10	7	7	0
	11	0	1	1
	12	891	869	43
•••	0	7	L	•••
	1	421	447	22
	2	1633	1536	71
	3	165	172	9
	4	516	512	25
	5	347	352	17
	6	530	517	25
	7	23	23	1
	8	691	661	32
	9	150	148	7
	10	23	23	1
	11	15	14	1
•••	0	8	L	•••
	0	410	410	20
	1	61	62	3
	2	145	151	8
	3	74	75	4
	4	852	828	39
	5	235	221	11
	6	385	374	18
	7	56	55	3
	8	8	9	1
	9	183	188	9
	10	214	218	11
	11	317	283	16
•••	0	9	L	•••
	1	503	521	25
	2	114	117	6
R	3	0	3	1
	4	41	40	2
	5	359	385	19
	6	265	258	13
	7	71	69	3
	8	1	1	0
	9	78	80	4
•••	0	10	L	•••
	0	208	207	10
	1	415	405	20
	2	12	12	1
	3	1277	1219	57
	4	216	202	10
R	5	17	25	1
	6	19	21	1
	7	447	443	22
	8	163	172	8
	9	138	140	9
•••	0	11	L	•••
	1	0	1	0
	2	0	1	0
	3	81	81	4
	4	2	3	0
	5	9	8	1
	6	24	24	1
	7	228	228	14
•••	0	12	L	•••
	0	866	889	42
	1	110	106	5
	2	21	21	1
	3	188	193	9
	4	543	533	25
	5	14	14	5
•••	0	13	L	•••
	1	97	81	7
	2	16	16	4
•••	1	0	L	•••
E	2	6309	5540	291
	4	1862	1688	76
	6	1099	1042	49
	8	857	788	38
•••	1	0	L	•••
	10	47	49	3
	12	626	631	30
•••	1	1	L	•••
	0	22	23	1
	1	9	9	1
	2	419	416	20
	3	216	219	11
	4	26	27	1
	5	651	633	30
	6	319	300	15
	7	109	109	6
	8	156	151	8
	9	266	268	13
	10	173	171	9
	11	445	453	22
	12	786	790	38
	13	99	97	9
•••	1	2	L	•••
	1	1284	1377	62
R	2	3	5	0
	3	948	939	43
	4	312	318	15
	5	3185	2876	129
	6	34	34	2
	7	26	26	1
	8	62	58	3
	9	750	746	36
	10	391	396	19
	11	155	154	8
	12	9	9	1
	13	53	53	6
•••	1	3	L	•••
	0	1101	1181	54
	1	194	214	10
	2	96	104	5
	3	1629	1678	76
	4	2045	1956	88
	5	63	61	3
	6	150	150	7
	7	145	142	7
	8	310	318	16
	9	620	614	30
	10	165	163	8
R	11	0	2	0
	12	220	236	12
	13	144	131	9
•••	1	4	L	•••
	1	29	37	2
	2	1116	1200	55
	3	148	155	8
	4	105	114	6
	5	42	40	2
	6	52	52	3
	7	603	586	28
	8	135	138	7
	9	64	62	3
	10	116	114	6
	11	670	657	32
	12	77	86	4
•••	1	5	L	•••
	0	1105	1182	55
	1	1181	1217	56
	2	63	61	3
	3	1811	1795	82
	4	554	535	26
	5	9	9	1
	6	341	339	17
	7	1321	1246	58
	8	76	78	4
	9	17	17	1
	10	313	314	15
	11	249	255	13
	12	25	12	6
•••	1	6	L	•••
	1	178	196	10
	2	3	3	1
	3	15	15	1
	4	186	188	9
	5	5	7	1
	6	473	462	22
	7	21	21	1
	8	201	197	10
•••	1	6	L	•••
	9	1	2	0
	10	681	686	33
	11	58	60	3
	12	14	10	5
•••	1	7	L	•••
	0	2184	2063	94
	1	443	466	23
	2	8	10	1
	3	268	279	14
	4	842	825	39
	5	24	24	1
	6	1000	963	45
	7	261	268	13
	8	61	61	3
	9	26	25	1
	10	384	420	20
	11	132	117	9
•••	1	8	L	•••
	1	43	43	2
	2	708	701	34
	3	129	130	7
	4	206	199	10
	5	70	67	3
	6	195	189	9
	7	216	214	11
	8	478	469	23
	9	119	122	6
	10	2	0	5
•••	1	9	L	•••
	0	62	56	3
	1	133	137	7
	2	8	9	1
	3	553	565	27
	4	169	167	8
	5	30	30	2
	6	16	15	1
	7	169	169	8
	8	438	448	22
	9	161	155	11
•••	1	10	L	•••
	1	988	972	46
	2	230	222	11
	3	132	131	7
	4	4	4	0
	5	705	704	34
	6	140	138	7
	7	32	34	2
	8	69	74	7
•••	1	11	L	•••
	0	31	29	2
	1	4	4	0
	2	12	11	1
	3	2	2	0
	4	13	14	1
	5	180	184	9
	6	11	21	5
	7	0	0	4
•••	1	12	L	•••
	1	156	152	8
	2	674	664	31
	3	21	22	1
	4	192	179	12
	5	190	187	11
•••	2	0	L	•••
E	0	7829	6027	283
	2	216	200	10
	4	1223	1111	52
	6	1233	1116	52
	8	412	399	20
	10	662	659	32
	12	0	5	5
•••	2	1	L	•••
	1	77	78	4
	2	75	76	4
	3	127	122	6
	4	196	188	9
	5	60	62	3
	6	52	53	3
•••	2	1	L	•••
	7	421	407	20
	8	371	355	17
	9	277	275	14
	10	297	305	15
	11	126	137	7
	12	5	11	5
•••	2	2	L	•••
	0	27	31	2
	1	916	983	46
	2	50	54	3
	3	1664	1518	70
	4	2	2	1
	5	87	87	4
	6	125	121	6
	7	1624	1559	72
	8	230	243	12
	9	121	114	6
	10	62	61	3
	11	163	172	9
	12	181	171	11
•••	2	3	L	•••
	1	662	693	33
	2	1392	1358	63
	3	17	19	1
	4	35	32	2
	5	553	536	26
	6	511	492	24
	7	388	383	19
	8	255	260	13
	9	19	20	1
	10	277	286	14
	11	271	279	14
	12	85	74	7
•••	2	4	L	•••
	0	657	677	33
	1	8	10	1
	2	294	302	15
	3	69	67	3
	4	408	411	20
	5	264	265	13
	6	14	14	1
R	7	1	2	0
	8	45	45	2
	9	680	658	32
	10	145	153	8
	11	195	199	10
•••	2	5	L	•••
	1	1447	1444	67
	2	583	597	29
	3	185	184	9
	4	255	262	13
	5	1207	1176	55
	6	335	340	17
R	7	0	6	1
	8	226	232	12
	9	646	628	30
	10	4	6	1
	11	98	86	8
•••	2	6	L	•••
R	0	2	5	1
	1	71	76	4
	2	71	75	4
	3	27	29	2
	4	83	82	4
	5	54	53	3
	6	243	245	12
	7	4	3	0
	8	504	497	24
	9	17	18	1
	10	6	6	0
•••	2	7	L	•••
	1	295	311	15
	2	1196	1152	54
	3	124	133	7
	4	381	379	19
	5	260	262	13
	6	398	400	20
	7	19	19	1
	8	546	551	27
	9	122	123	6
	10	18	12	5
•••	2	8	L	•••
	0	342	349	17
	1	48	49	3
	2	104	113	6
	3	51	50	3
	4	625	634	31
	5	130	185	9
	6	303	301	15
	7	47	50	3
	8	7	7	1
	9	149	125	10
•••	2	9	L	•••
	1	378	390	19
	2	88	91	5
	3	0	1	0
	4	30	30	2
	5	307	309	15
	6	210	207	10
	7	58	57	3
	8	1	0	4
•••	2	10	L	•••
	0	166	165	8
	1	332	329	16
	2	9	9	1
	3	994	974	46
	4	171	163	8
	5	13	15	1
	6	15	7	5
•••	2	11	L	•••
R	1	0	1	0
	2	0	1	0
	3	64	62	3
	4	2	1	4
•••	3	0	L	•••
	2	2530	2210	101
	4	709	655	32
	6	606	555	27
	8	537	527	26
•••	3	1	L	•••
	0	32	40	2
	1	21	21	1
	2	134	131	7
	3	71	76	4
	4	8	8	1
	5	268	265	13
	6	190	183	9
	7	69	77	4
	8	101	103	5
	9	170	170	9
•••	3	2	L	•••
	1	738	726	35
	2	2	4	1
	3	337	324	16
	4	122	117	6
	5	1515	1383	65
	6	23	23	1
	7	14	16	1
	8	42	40	2
	9	457	472	23
•••	3	3	L	•••
	0	627	677	33
	1	115	125	6
	2	28	29	2
	3	646	632	31
	4	892	834	40
	5	37	35	2
	6	86	87	4
	7	101	97	5
	8	202	202	10
	9	371	381	18
•••	3	4	L	•••
	1	16	17	1
	2	483	489	24
	3	74	73	4
	4	49	47	3
	5	22	22	1
	6	45	45	2
	7	363	362	18
	8	83	84	4
•••	3	4	L	•••
	9	41	41	2
•••	3	5	L	•••
	0	560	632	31
	1	557	588	29
	2	40	40	2
	3	983	942	45
	4	303	304	15
	5	8	10	1
	6	205	212	11
	7	778	774	37
	8	45	49	3
•••	3	6	L	•••
	1	75	84	4
	2	3	5	1
	3	4	6	1
	4	106	106	5
	5	5	5	0
	6	269	271	13
	7	14	14	1
	8	123	110	9
•••	3	7	L	•••
	0	1193	1165	56
	1	227	244	12
	2	13	13	1
	3	163	171	9
	4	514	515	25
	5	19	19	1
	6	594	576	28
	7	165	178	12
•••	3	8	L	•••
	1	25	25	1
	2	427	430	21
	3	80	83	4
	4	130	129	6
	5	43	43	2
	6	121	115	9
•••	3	9	L	•••
	0	41	38	2
	1	84	83	4
	2	4	5	0
	3	339	348	17
	4	108	105	9
	5	19	18	5
•••	3	10	L	•••
	1	620	616	31
	2	148	137	9
•••	4	0	L	•••
	0	2515	2143	102
	2	92	84	4
	4	645	581	29
•••	4	1	L	•••
	1	30	31	2
	2	30	29	2
	3	57	59	3
	4	99	97	5
•••	4	2	L	•••
	0	10	12	1
	1	372	390	19
	2	18	21	1
	3	753	734	36
•••	4	3	L	•••
	1	260	281	14
	2	568	581	28
•••	4	4	L	•••
	0	286	316	16

Three-dimensional Patterson functions were computed in sections normal to the u axis. As expected, all distinguishable Patterson peaks were found in the $u=0$ and $\frac{1}{2}$ sections. Calculated interatomic vectors for two sets of yttrium atoms located in the $4(c)$ positions of $Pmcn$ were readily matched to the strongest of these peaks. Oxygen and beryllium atoms were found by difference-Fourier methods. All atoms were at $x=\frac{1}{4}$ or $\frac{3}{4}$, and no indications of possible deviations from these planes, with a consequent lowering of space group symmetry to $P2_1cn$, could be discovered.

Refinement

A structure factor calculation made with the scale and temperature factors given by Wilson's method and with the atomic positional parameters given by the Fourier functions showed an agreement index $R_1 = 0.14$, where $R_1 = \Sigma(|F|_o - |F|_c)/\Sigma|F|_o$. The atomic scattering factors for this and subsequent calculations were taken from Cromer & Waber (1964) (for Be, Be$^+$, and Be^{2+}) and from *International Tables for X-ray Crystallography* (1962) (for Y, Y^{3+}, O, and O$^-$). Anomalous dispersion corrections to the scattering by yttrium were included from the latter tables ($\Delta f'(\mathrm{Y}) = 0.7$ electron, $\Delta f''(\mathrm{Y}) = 2.2$ electrons).

The initial parameters were refined by iterative least-squares calculations (Busing, Martin & Levy, 1962). The observations were weighted in inverse proportion to the variance defined as

$$\sigma^2(F^2) = s(A^*/\mathrm{Lp})^2[\sigma_{st}^2 + (0.05\mathrm{N})^2]$$
$$+ 0.010\ (F_{corr}^2 - F_{uncorr}^2)^2$$

where s is a scale factor, A^* is an absorption correction, Lp is the Lorentz–polarization correction, σ_{st}^2 is the statistical variance of N, the net count, F_{uncorr}^2 and F_{corr}^2 are the observed values of F^2 before and after correction for secondary extinction. With the inclusion of anisotropic individual atom temperature factors, the final value of R_1 was 0.027 and that of R_2 was 0.060 $[R_2 = \Sigma(|F_o^2 - F_c^2|)/\Sigma|F_o^2|]$.

Atomic position and thermal parameters from the final least-squares cycle are presented in Table 1. A comparison of observed F^2 values with those computed from the final parameters is given in Table 2. The scattering factors used were for Y^{3+}, Be^0, and O$^-$. Results when other combinations of scattering factors (*e.g.* Y^0, Be^0, and O^0) were used were the same as far as atom parameters and R values were concerned; the Y^{3+}, Be^0, O$^-$ combinations gave the lowest value of $\Sigma_1(= \sqrt{\Sigma(w(F_o^2 - F_c^2)^2}/\sqrt{n-m}$, where w is a weight, n is the number of observations, and m the number of variable parameters). This value of Σ_1 was 1.47; the next lowest Σ_1 was computed at 1.52 for the scattering factor combination (Y^{3+}, Be^{2+}, O$^-$). The ratio is 1.034 which, by Hamilton's (1965) test is significant at the 0.5% level ($R_{3,\ 450,\ 0.005} = 1.009$).

Despite the absence of any evidence that it might be required, an attempt was made to refine the data with parameters based on the acentric space group $P2_1cn$.

The x parameters of two atoms were deliberately changed from $\frac{1}{4}$ and a least-squares cycle was computed. No significant lowering of the R values was obtained and the temperature factors of two oxygen atoms and the beryllium atom in the asymmetric unit became negative. One must conclude that, on the basis of X-ray diffraction data refined by linear least-squares methods, the atom arrangement in Y$_2$BeO$_4$ is adequate-

Table 3. *Interatomic distances and angles in* Y$_2$BeO$_4$

Distances

			Standard deviation
(2)	Y(1)–O(1)	2·337 Å	0·002 Å
	Y(1)–O(2)	2·280	0·003
	Y(1)–O(3)	2·212	0·003
(2)	Y(1)–O(4)	2·274	0·002
	Y(2)–O(1)	2·276	0·003
(2)	Y(2)–O(2)	2·342	0·002
(2)	Y(2)–O(3)	2·245	0·002
	Y(2)–O(3')	2·271	0·003
	Be–O(1)	1·549	0·007
	Be–O(2)	1·550	0·006
	Be–O(4)	1·564	0·008

Angles

			Standard deviation
	O(1)–Y(1)–O(1)	98·1 °	0·1 °
(2)	O(1)–Y(1)–O(2)	76·5	0·1
(2)	O(1)–Y(1)–O(3)	87·2	0·1
(2)	O(1)–Y(1)–O(4)	79·3	0·1
(2)	O(1)–Y(1)–O(4)	171·0	0·1
	O(2)–Y(1)–O(3)	154·7	0·1
(2)	O(2)–Y(1)–O(4)	111·0	0·1
(2)	O(3)–Y(1)–O(4)	84·1	0·1
	O(4)–Y(1)–O(4)	101·9	0·1
(2)	O(1)–Y(2)–O(2)	76·4	0·1
	O(1)–Y(2)–O(3')	161·7	0·1
(2)	O(1)–Y(2)–O(3)	110·7	0·1
	O(2)–Y(2)–O(2)	97·9	0·1
(2)	O(2)–Y(2)–O(3)	78·5	0·1
(2)	O(2)–Y(2)–O(3)	170·6	0·1
(2)	O(2)–Y(2)–O(3')	91·8	0·1
	O(3)–Y(2)–O(3)	103·8	0·1
(2)	O(3)–Y(2)–O(3')	79·8	0·1
	O(1)–Be–O(2)	124·0	0·5
	O(1)–Be–O(4)	116·8	0·4
	O(2)–Be–O(4)	119·2	0·5

Oxygen–oxygen separations in ribbon

In layers

(2)	O(1)–O(3)	3·719 ± 0·004 Å
(2)	O(1)–O(1)	3·5315 ± 0·0005
(2)	O(1)–O(3)	3·137 ± 0·004
(2)	O(2)–O(4)	3·753 ± 0·004
(2)	O(2)–O(2)	3·5315 ± 0·0005
(2)	O(2)–O(3)	3·312 ± 0·004

Between layers

	O(1)–O(4)	2·944 ± 0·005 Å
(2)	O(1)–O(2)	2·857 ± 0·004
	O(2)–O(3)	2·902 ± 0·005

Oxygen–oxygen separations between ribbons (about Be)

O(1)–O(2)	2·735 ± 0·005 Å
O(2)–O(4)	2·686 ± 0·005
O(1)–O(4)	2·651 ± 0·005

ly represented by positions in the space group *Pmcn*. A final three-dimensional difference Fourier synthesis with all atoms removed showed no peak containing more than 0·3 electron; this peak was associated with a diffraction ripple from an yttrium atom.

Description

Important interatomic distances and angles were computed from the parameters of Table 1 with the ORFFE computer program of Busing, Martin, & Levy (1964). They are listed in Table 3. The following paragraphs will refer the reader to Figs. 1–3, which were drawn with the ORTEP program (Johnson, 1965).

The structure of Y₂BeO₄ contains ribbons of edge-sharing octahedra formed from two pseudo-close-

packed layers of oxygen atoms between which yttrium atoms occupy octahedral interstices. The long direction of the ribbons is parallel to **a** and they are five oxygen atoms (four octahedra) wide. A given ribbon touches (and shares oxygen with) two other ribbons at either end of its width; the touching ribbons diverge at an angle of 52° leaving rather narrow channels running through the crystal in the **a** direction. These features are easily seen in Fig. 1.

Close yttrium-oxygen interatomic contacts vary from 2·21 to 2·34 Å (average 2·286 Å). They are comparable to the average separation of 2·297 Å reported for the more distorted octahedra in Y₂O₃ (Paton & Maslen, 1965). Oxygen–oxygen interatomic contacts in a pseudo-close-packed layer of a ribbon are signifi-
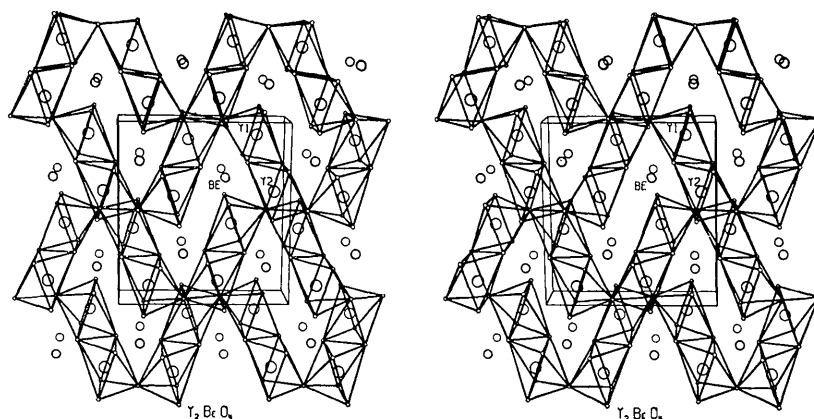
Fig. 1. Stereoscopic drawings of a section of the Y₂BeO₄ structure viewed along a direction 10° from −**a**. The horizontal direction is parallel to **b**. Atoms have been drawn as spheres of arbitrary radius: the largest are Y, next largest Be, and smallest O. Oxygen atoms in an octahedron about an yttrium atom have been connected along the octahedron edges. The unit-cell outline has its raised lower left hand origin at x=0·5, y=−0·5, z=0.
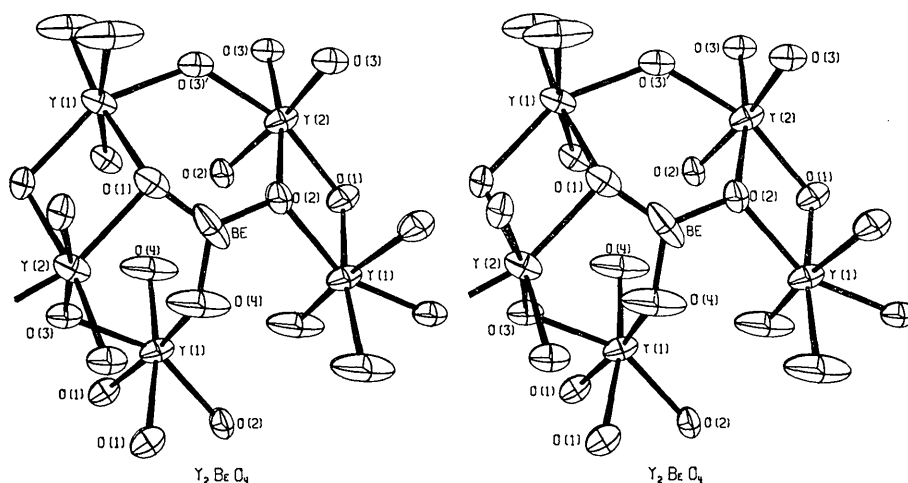

Fig. 2. Stereoscopic drawings of atom environments about Y(1), Y(2), and Be. The direction of view and structure orientation is the same as that of Fig. 1 except for a 16° clockwise rotation about the vertical drawing axis. The Be atom has the coordinates listed in Table 1. Atoms are represented by thermal displacement ellipsoids including 99·9 % probability (4 × r.m.s. displacement).

cantly greater than those between layers of the same ribbon. Oxygen atoms of a layer deviate from a plane (such as $0.831y - 0.403z = 1$) by no more than 0.2 Å.

An octahedron about a Y(2) atom shares edges with six octahedra in the same ribbon and shares one corner [as at O(3')] with an octahedron in a touching ribbon. An octahedron about a Y(1) atom shares edges with four octahedra in the same ribbon and shares one corner with an octahedron in a touching ribbon. Thus, an oxygen atom of a Y(2) octahedron is in close contact with 3 or 4 yttrium atoms, while an oxygen atom of a Y(1) octahedron has only 2 or 3 close yttrium atom neighbors. The oxygen atom with 2 such neighbors is O(4). As one may see in Fig. 2, this atom has an unusually large component of thermal vibration normal to the O(4)–Y(1) vector in a direction which crosses an inter-ribbon channel. The behavior of the O(4) atom probably accounts for much of the distortion of the octahedron about Y(1) and, to a lesser degree, about Y(2).

Along the channels, and lying at about the center of a triangle of oxygen, are beryllium atoms which act as a 'glue' holding three separate yttrium–oxygen ribbons together. The mirror plane of symmetry requires the atoms of this group to be coplanar. Beryllium–oxygen separations average 1.55 Å, or about 0.1 Å shorter than the characteristic tetrahedral separation. The thermal vibration of the beryllium atom is large in the **a** direction and in a direction along the inter-ribbon channel.

An interesting analogy is found between the structure of Y_2BeO_4 and that of malachite, $Cu_2(OH)_2(CO_3)$ (Wells, 1951). In a projection down its short **c** axis malachite is seen to contain ribbons of quite distorted Cu–O(OH) octahedra with carbon atoms tying three separate ribbons together (Fig. 3). The ribbons are only two octahedra wide in malachite and the inter-ribbon channels are open in the **a** direction rather than

closed, as in the **c** direction of Y_2BeO_4. While caution must be exercised in describing a BeO_3 group analogous to the CO_3 group, the similarity between the role of beryllium in Y_2BeO_4 and that of carbon in malachite is striking.

Discussion

The only previous observation of a threefold beryllium–oxygen coordination was in $Ca_{12}Be_{17}O_{29}$ (Harris & Yakel, 1966) where Be–O separations in trigonal groups averaged 1.58 Å and small displacements of beryllium atoms from the planes of the oxygen atoms were found. In that instance too, it was suggested that the trigonally coordinated beryllium atoms were acting as a 'glue' between more normally bound parts of the structure. Further similarities can be noted in the method of preparation and properties of Y_2BeO_4 and $Ca_{12}Be_{17}O_{29}$. The compounds were most readily formed by quenching from the liquid state; neither has been clearly shown to form in solid state reactions; both tend to dissociate into component oxides on heating.

One may speculate that the atomic environments in both compounds are more nearly representative of liquids than of equilibrium solids. If this be true, the occurrence of trigonally coordinated beryllium in both structures may reflect the occurrence of the same configuration in the liquid. The recently reported phase transition in pure BeO (Smith, Cline & Frechette, 1962) at $2100\,°C$ (the melting point is $2530\,°C$) has been shown to involve a decrease in the symmetry of the beryllium–oxygen coordination polyhedron (Smith, Cline & Austerman, 1965). This may also indicate that the oxygen coordination number or geometry of beryllium changes in the solid–liquid transition.
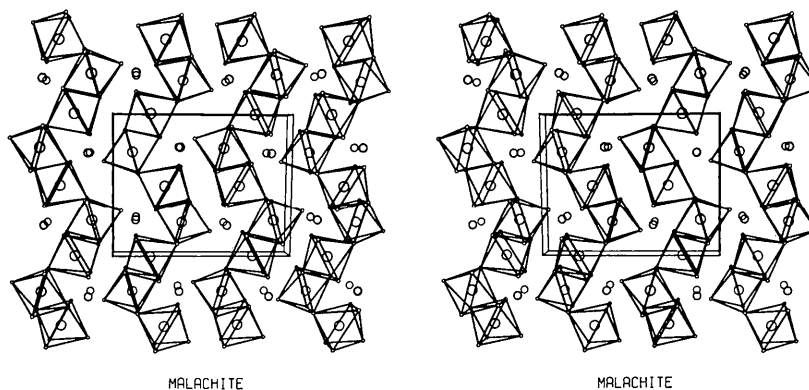
MALACHITE   MALACHITE

Fig. 3. Stereoscopic drawings of a section of the malachite, $Cu_2(OH)_2(CO_3)$, structure viewed along a direction $10°$ from the normal to the *ab* plane (*i.e.* $\sim +c$). The horizontal direction is parallel to **b**. Atoms have been drawn as spheres with the same radii used in Fig. 1: the largest are Cu, next largest C, and smallest O or OH. Oxygen atoms in a pseudo-octahedron about a copper atom have been connected along octahedron edges. The unit cell outline has its raised lower left hand origin at $x = 0$, $y = 0$, $z = 0$.

Laboratory, who made their automated diffractometer available for the collection of the data reported here. We are also indebted to R. A. Potter of the Metals and Ceramics Division, who participated in the preparation of the crystals.

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The Crystal Structure of Ce₅Mg₄₂

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The structure of the compound present in the Ce–Mg system at the approximate stoichiometry of CeMg₈ has been determined with the use of diffractometrically recorded single-crystal intensities. The unit cell, containing two formula units of Ce₅Mg₄₂, is body-centered tetragonal with $a = 14\cdot78$, $c = 10\cdot43$ Å. The calculated density is 2·51 g.cm⁻³, and the space group, as confirmed by the final structure, is $I4/m$.

Introduction

During a phase-relation study of the magnesium-rich region of the Ce–Mg system (Wood & Cramer, 1965), a new compound with a tentative composition CeMg₈.₂₅ was discovered. The composition and symmetry indicated this to be a new structure type; therefore, we have carried out a determination of this structure by conventional single-crystal X-ray diffraction techniques. Additional interest in this study resulted from the expectation that, in view of the similarity of cell constants, a simple relationship would be found to connect this structure with the other structures occurring in the magnesium-rich region (Savitskii, Terekhova, Burov, Markova & Naumkin, 1962; Evdokimenko & Kripyakevich, 1963; Johnson, Smith, Wood & Cramer, 1964; Johnson & Smith, 1966).

Experimental

The sample of Ce₅Mg₄₂ used in this investigation was kindly provided by D. Wood. Details concerning the preparation are given by Wood & Cramer (1965).

Single-crystal oscillation, Weissenberg, and precession photographs showed tetragonal symmetry. The observed extinctions $(h+k+l \neq 2n)$ and $4/m$ Laue