

crystallographically symmetry related. This is not the case in the present compound, which also shows an apparent difference between the two hydrogen-bridged formate groups. As mentioned previously, the distances C(2)-O(3) and C(2)-O(4) differ significantly in contrast to C(1)-O(1) and C(1)-O(2) which are of about the same length (*cf.* Table 4). Furthermore, the number of ionic contacts to potassium is different: one formate group has 4+1 whereas the other has 3+0 potassium-oxygen contacts.

According to the facts given above there may be a tendency towards less ionization in one of the groups than in the other. This has important bearings on the symmetry character of the hydrogen bond in the dimer.

As mentioned earlier the positions of the hydrogen atoms have not been located experimentally. Naturally nothing definite can be said about the character of the hydrogen bond from the X-ray data. Attempts will be made to attack this problem with other methods.

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The Crystal Structure of $\text{La}_2\text{Be}_2\text{O}_5$ *

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The crystal structure of $\text{La}_2\text{Be}_2\text{O}_5$ has been derived and refined from Mo $K\alpha$ X-ray diffraction data. The *C*-centered monoclinic unit cell with $a_0=7.536$, $b_0=7.348$, $c_0=7.439$ Å, $\beta=91^\circ 33'$ contains four formula weights. The atomic arrangement in this equilibrium phase consists of a three-dimensional framework of cornersharing beryllium-oxygen tetrahedra with lanthanum atoms irregularly coordinated to ten oxygen atoms. The structure is compared with those of other oxide compounds containing beryllium and, in particular, with the structures of the recently reported nonequilibrium phases $\text{Ca}_{12}\text{Be}_{17}\text{O}_{29}$ and Y_2BeO_4 .

Introduction

The work described in this paper is part of a continuing study of binary oxide compounds that contain beryllia as a member. We have previously reported the crystal

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structures of the compounds $\text{Ca}_{12}\text{Be}_{17}\text{O}_{29}$ and Y_2BeO_4 (Harris & Yakel, 1966, 1967). Unusual trigonal beryllium-oxygen coordinations appeared in these probably metastable crystals which we could form only by quenching from the liquid state.

In both $\text{Ca}_{12}\text{Be}_{17}\text{O}_{29}$ and Y_2BeO_4 , the heavy cations have a large radius (0.99 and 0.97 Å, respectively) relative to those in the known equilibrium compounds

of BeO with Al₂O₃, SiO₂, and Cr₂O₃ ($R_{\text{cat}}=0.51, 0.42,$ and 0.69 \AA , respectively) where regular, or nearly regular, tetrahedral beryllium-oxygen coordinations are found. We were thus prompted to investigate the crystal structure of an apparent equilibrium phase at the 1:2 composition of the La₂O₃-BeO system in which the heavy cation radius is still larger (1.14 \AA).

The compound La₂Be₂O₅ was first reported by Weir & Van Valkenburg (1960) who established its formula on the basis of solid-state synthesis and listed optical and powder-pattern X-ray diffraction data. An equilibrium phase diagram for the La₂O₃-BeO system presented by Levin, Robbins & McMurdie (1961) shows a low-melting eutectic near the 1:2 composition and suggests that the La₂Be₂O₅ phase itself may form peritectically just to the BeO-rich side of the eutectic. A 1:2 phase was also observed in this system by Kuo & Yen (1964). They postulated an orthorhombic unit cell for their compound, with $a_0=3.81$, $b_0=9.95$, $c_0=11.07 \text{ \AA}$, $Z=4$ formula weights per cell, and gave refractive indices $n_x=1.980$ and $n_y=2.035$. Bragg spacings computed from these cell parameters are not in good agreement with the powder pattern for La₂Be₂O₅ listed by Weir & Van Valkenburg (1960) although the optical data show fair correspondence with the earlier results.

Experimental

We grew lath-like crystals of La₂Be₂O₅ by cooling melts near the eutectic of the La₂O₃-BeO system. Our experiments confirmed the equilibrium nature of this

phase. Powder patterns from crushed crystals were in excellent agreement with the La₂Be₂O₅ pattern reported by Weir & Van Valkenburg (1960). The crystal selected for the X-ray diffraction investigation had a shape roughly approximating a right triangular prism with an altitude of 0.17 mm , a base width of 0.08 mm , and a uniform thickness of 0.03 mm . Later results showed that the triangular faces were parallel to $\{101\}$ planes; smaller faces seemed to be parallel to $\{11\bar{1}\}$ planes, but most were too uneven for positive identification. The **b** axis was approximately parallel to the long triangular edge of the crystal.

Preliminary diffraction film data provided estimates of crystal orientation and lattice parameters. The crystal system was monoclinic and systematic reflection absences occurred consistent with space groups $C2/c$ or Cc . The crystal was transferred to a computer-controlled four-circle diffractometer (Busing, Ellison & Levy, 1964, 1965) for refinement of lattice parameters and collection of intensity data. The **b** axis was offset about 5° from the φ axis of the diffractometer and zirconium- or niobium-filtered Mo $K\alpha$ X-radiation ($\lambda K\alpha_1=0.70926 \text{ \AA}$) was used for all experiments. A least-squares fit to measurements of scattering angles of 12 high-angle reflections gave the following lattice parameters:

$$\begin{aligned} a_0 &= 7.5356 \pm 0.0006 \text{ \AA} & \beta &= 91^\circ 33' \pm 1' \\ b_0 &= 7.3476 \pm 0.0017 & U &= 411.7 \pm 0.2 \text{ \AA}^3 \\ c_0 &= 7.4387 \pm 0.0006 & \rho_x &= 6.061 \pm 0.003 \text{ g.cm}^{-3}, \\ & & & \text{assuming } Z=4 \text{ formula} \\ & & & \text{weights per cell.} \end{aligned}$$

Table 1. Atomic parameters for La₂Be₂O₅

Least-squares standard errors in the last significant figure are given in parentheses.
Fractional position parameters.

Positions	Atom	<i>x</i>	<i>y</i>	<i>z</i>
8(<i>f</i>)	La	0.23777 (2)	0.03494 (2)	0.19645 (2)
8(<i>f</i>)	Be	0.4693 (5)	0.2183 (5)	-0.4697 (5)
8(<i>f</i>)	O(1)	0.0987 (3)	0.1569 (3)	-0.0845 (2)
4(<i>d</i>)	O(2)	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{2}$
4(<i>e</i>)	O(3)	0	0.2493 (4)	$\frac{1}{4}$
4(<i>b</i>)	O(4)	0	$\frac{1}{2}$	0

Thermal vibration parameters*.

<i>B</i> ₁₁	<i>B</i> ₂₂	<i>B</i> ₃₃	<i>B</i> ₁₂	<i>B</i> ₁₃	<i>B</i> ₂₃
0.00214 (1)	0.00179 (1)	0.00171 (1)	0.00052 (1)	0.00052 (1)	0.00011 (1)
0.0021 (4)	0.0017 (4)	0.0018 (3)	-0.0002 (3)	-0.0000 (3)	-0.0002 (3)
0.0030 (2)	0.0029 (2)	0.0019 (2)	0.0011 (2)	0.0005 (1)	-0.0003 (1)
0.0010 (2)	0.0031 (3)	0.0033 (3)	-0.0004 (2)	-0.0003 (2)	0.0005 (2)
0.0019 (2)	0.0029 (3)	0.0011 (2)	0	-0.0002 (2)	0
0.0033 (3)	0.0015 (2)	0.0055 (4)	0.0001 (2)	0.0021 (3)	0.0010 (3)

R.M.S. thermal displacements (\AA) along principal axes.

Positions	Atom	Axis 1	Axis 2	Axis 3
8(<i>f</i>)	La	0.0598 (3)	0.0673 (3)	0.0876 (3)
8(<i>f</i>)	Be	0.064 (8)	0.073 (7)	0.079 (7)
8(<i>f</i>)	O(1)	0.060 (4)	0.081 (3)	0.107 (3)
4(<i>d</i>)	O(2)	0.051 (6)	0.086 (4)	0.103 (4)
4(<i>e</i>)	O(3)	0.054 (5)	0.075 (5)	0.089 (4)
4(<i>b</i>)	O(4)	0.056 (6)	0.079 (5)	0.138 (4)

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

We can find no relationship between this unit cell and that reported by Kuo & Yen (1964).

θ - 2θ scans were used to measure intensities of reflections from $2\theta=28^\circ$ to 130° ; low-angle data were observed in an ω -scanning mode. A total of 3605 independent reflections allowed by the possible space groups were examined. Replicate measurements brought the total number of observations, not including standards, to 3974.

The observed data were corrected for Lorentz-polarization and absorption effects. The latter corrections were made by approximating the crystal shape with six bounding planes and using the ORABS computer program of Wehe, Busing & Levy (1962). Replicate measurements were then averaged and relative F^2 values placed on an approximate absolute scale by Wilson's (1942) method. Statistical tests suggested a centrosymmetric intensity distribution. After a satisfactory trial structure had been derived, secondary extinction corrections were applied to all data following the procedure of Zachariasen (1963).

Results

Peaks on the Harker sections of a three-dimensional Patterson function synthesized from the 3605 corrected and scaled independent F^2 values gave initial parameters for lanthanum atoms in the $8(f)$ general positions of the space group $C2/c$. Tentative parameters for sixteen oxygen atoms, eight in $8(f)$, four in $4(e)$, and four in $4(d)$, were also deduced. Standard Fourier methods confirmed these tentative locations and showed the remaining four oxygen atoms [in $4(b)$ positions] and eight beryllium atoms [in $8(f)$ positions].

Iterative structure-factor, least-squares calculations (Busing, Martin & Levy, 1962) were employed to refine the trial structure. The least-squares procedure minimized residuals in weighted F^2 . We assumed the X-ray

scattering factors for La^{3+} and Be^{2+} given by Cromer & Waber (1965) and those for O^{-1} given in *International Tables for X-ray Crystallography* (1962). Anomalous dispersion corrections for the scattering of Mo $K\alpha$ X-rays by lanthanum atoms were also taken from the latter source. The real part of this correction was applied by drawing a smooth curve through the given values of $\Delta f'$ as a function of $\sin \theta/\lambda$ and making an appropriate adjustment in each entry of the La^{3+} scattering-factor table used by the least-squares program. An average value of +2.8 electrons was assumed for the imaginary part of the dispersion correction. The observations were weighted in inverse proportion to a variance defined as

$$\sigma^2(F^2) = s(A^{-1} \text{Lp})^2 [\sigma_{st}^2 + (0.04N)^2] + [0.10(F_{\text{corr}}^2 - F_{\text{uncorr}}^2)]^2,$$

where s is a scale factor, A^{-1} is an absorption correction, Lp is a Lorentz-polarization correction, σ_{st}^2 is the statistical variance of N (the net count), and F_{uncorr}^2 and F_{corr}^2 are the observed values of F^2 before and after correction for secondary extinction. The final term in the above equation is an attempt to allow for uncertainties in determining the constants governing the secondary extinction corrections.

Four least-squares cycles, the last three with anisotropic thermal vibration parameters, produced the following measures of agreement between observed and calculated $|F|$ and F^2 values:

$$R_1 [\equiv \Sigma (|F|_o - s|F|_c) / \Sigma |F|_o] = 0.066$$

$$R_2 [\equiv \Sigma (|F_o^2 - sF_c^2|) / \Sigma F_o^2] = 0.056$$

$$\sigma_1 [\equiv \sqrt{\Sigma w(F_o^2 - sF_c^2)^2} / \sqrt{n-m}] = 1.106,$$

where w is a weight [$\equiv 1/\sigma^2(F_o^2)$], n is the number of observations, and m is the number of variable parameters. The sums and averages were taken over all 3605 independent reflections.

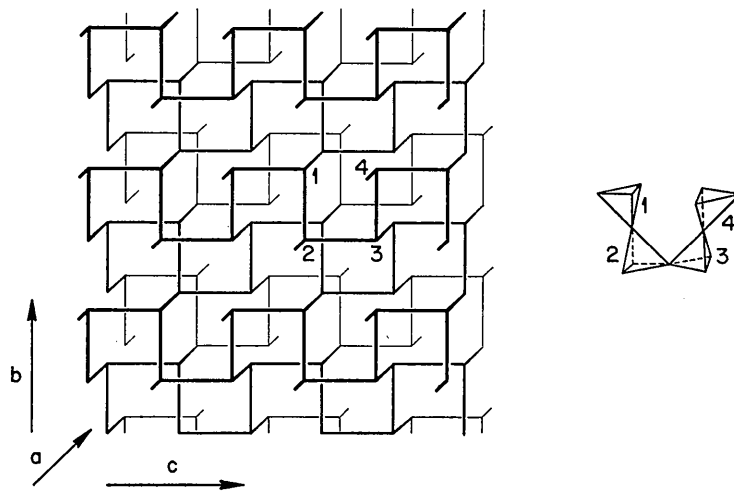


Fig. 1. A schematic representation of the tetrahedral framework in $\text{La}_2\text{Be}_2\text{O}_5$. Tetrahedra have been replaced by sets of three orthogonal Be-O vectors, as described in the text. Unit-cell vectors are shown at the lower left, and two units of a 'square-wave' string of tetrahedra are shown at the right.

Table 2. Comparison of observed and calculated $|F|$ values

$FC = 10 \cdot |F|_{\text{calc}}$, with the sign of A_{calc} . $FO = 10 \cdot |F|_{\text{obs}}$. $SIG = 10 \cdot \sigma(|F|_{\text{obs}})$, where $\sigma(|F|_{\text{obs}})$ is defined as $\sigma(F^2_{\text{obs}})/2|F|_{\text{obs}}$ unless F^2_{obs} is less than 1 (indicated by a W to the right of the entry for SIG in the table), in which case $SIG = \sigma(F^2_{\text{obs}})$. All F^2_{obs} have been scaled with the least-squares scale factor; all $|F|_{\text{obs}}$ have been scaled with the square root of that factor.

L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG																																																																																																																																																																									
***	0	0	L	***	10	0	L	***	1	1	L	***	5	1	L	***	11	1	L	***	19	1	L	***	4	2	L	***	10	2	L	***	16	2	L	***																																																																																																																																																																												
2	-2379	2461	51	-16	79	0	278	W	1	-389	407	11	17	-13	24	183	1	222	251	19	-3	-41	0	198	W	7	-589	579	16	-11	-411	475	31	-6	-498	498	28	-5	309	263	52	-4	694	691	87	-3	-300	261	52	-2	-588	608	28	-1	276	242	55	0	221	221	27	-1	31	42	151	-1	429	411	32	-8	490	509	27	-7	9	0	215	W	-6	652	669	22	-5	-425	429	25	-4	-1057	1060	25	-3	-31	42	151	-2	283	301	23	-1	218	243	25	-1	-498	590	19	1	523	251	23	6	574	584	18	7	-245	280	18	8	-424	428	18	9	137	153	28																																																																																		
4	1149	1160	24	-14	-625	612	30	2	-2524	2542	49	3	1309	1318	27	4	1047	999	24	5	164	202	25	6	234	252	23	7	27	103	44	8	320	327	22	9	-237	254	23	10	-742	741	20	11	0	2910	3135	71	12	-241	419	18	13	-26	69	79	14	-235	182	32	15	178	203	27	16	529	530	18	17	-271	290	21	18	-545	547	0	19	1	356	344	16	20	362	347	17	21	362	347	17	22	162	151	33	23	703	683	19	24	5407	428	17	25	-407	282	17	26	-174	193	30	27	6	936	966	22	28	7	441	441	18	29	8	922	915	22	30	-414	422	18	31	-373	305	45	32	-373	305	45	33	-373	305	45	34	-373	305	45	35	-373	305	45	36	-373	305	45	37	-373	305	45	38	-373	305	45	39	-373	305	45	40	-373	305	45	41	-373	305	45	42	-373	305	45	43	-373	305	45	44	-373	305	45	45	-373	305	45	46	-373	305	45	47	-373	305	45	48	-373	305	45	49	-373	305	45	50	-373	305	45

Table 2 (cont.)

Table with multiple columns of numerical data, organized into groups by row headers (e.g., '*** 2 6 L ***', '*** 12 6 L ***'). Each group contains several columns of values, some with associated signs (+/-) and some with 'SIG' labels. The data is presented in a dense, grid-like format across the page.

Table 2 (cont.)

L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG																														
***	3	9	L	***	9	9	L	***	0	10	L	***	6	10	L	***	12	10	L	***	3	11	L	***	11	11	L	***	2	12	L	***	10	12	L																						
7	-570	587	19	1	-930	938	24	8	635	614	20	0	880	860	19	8	372	371	19	13	-408	416	19	-11	-148	0	244	W	8	-853	824	23	-11	-339	220	50																					
8	-145	300	26	2	389	384	23	9	989	976	24	1	678	722	20	9	299	284	20	14	483	516	17	-10	-245	181	76	9	-468	466	21	-10	-580	562	25																						
9	-102	247	23	3	1127	1123	26	10	-620	630	19	2	-186	211	11	-2	-168	202	12	15	326	320	19	-9	403	376	42	10	70	667	21	-9	154	0	359	W																					
10	161	134	48	4	-425	461	18	11	-650	637	21	3	-57	118	51	4	-45	100	59	***	5	11	L	***	-8	-594	596	20	11	285	233	33	-8	-243	0	256	W																				
11	658	608	21	5	-656	648	21	12	253	184	42	4	-45	100	59	***	5	11	L	***	-7	-548	572	31	12	-307	261	32	-7	72	0	345	W																								
12	-311	300	25	6	230	197	32	13	204	247	27	5	-552	537	20	-8	-105	93	86	-14	495	483	18	-6	574	561	33	13	-25	0	117	W																									
13	-846	835	22	7	87	124	46	14	-24	0	127	W	6	549	522	20	-8	-105	93	86	-14	495	483	18	-6	574	561	33	13	-25	0	117	W																								
14	266	286	21	8	-51	99	69	15	186	335	24	7	848	833	22	-7	234	212	42	-13	300	309	21	-4	-436	497	30	***	4	12	L	***	-3	93	72	210																					
15	548	535	18	9	427	447	20	16	-239	232	22	8	-603	588	20	-6	-144	179	49	-12	-352	308	24	-3	93	72	210	***	4	12	L	***	-3	93	72	210																					
16	-215	195	27	10	-272	257	30	***	2	10	L	9	-710	700	21	-5	-527	503	25	-11	-57	0	127	W	-2	-111	187	80	-2	-111	187	80	***	4	12	L	***	-3	93	72	210																
				11	-658	590	18	***	2	10	L	10	526	519	19	-4	430	410	29	-10	-145	177	34	-1	-299	332	46	-14	-187	152	37	-14	-187	152	37	***	4	12	L	***	-3	93	72	210													
				12	210	284	15	***	2	10	L	11	306	333	18	-3	575	540	34	-9	-340	341	23	0	387	388	23	-13	-217	212	27	0	671	661	21	0	671	661	21	***	4	12	L	***	-3	93	72	210									
				13	531	540	17	***	2	10	L	12	-100	167	30	-2	-324	286	57	-8	504	499	19	1	652	670	23	-12	622	616	20	1	96	160	47	1	96	160	47	***	4	12	L	***	-3	93	72	210									
				-16	-292	279	21	***	11	9	L	-15	119	119	46	-1	-117	119	46	-1	-117	119	46	2	-668	681	22	-11	448	443	21	-2	-160	200	37	-2	-160	200	37	***	4	12	L	***	-3	93	72	210									
				-15	-683	684	19	***	11	9	L	-14	168	162	41	14	-40	0	101	W	0	324	315	23	-6	-939	913	23	-3	-530	540	21	-3	161	125	66	-3	161	125	66	***	4	12	L	***	-3	93	72	210								
				-14	317	340	18	***	11	9	L	-13	345	323	25	1	29	0	132	W	-5	-862	858	21	4	521	566	20	-9	-374	357	23	-4	-300	346	23	-4	-300	346	23	***	4	12	L	***	-3	93	72	210								
				-13	660	667	21	***	11	9	L	-12	28	216	50	***	8	10	L	***	2	44	79	72	-4	749	738	20	-5	263	315	20	-8	800	805	21	-5	-342	358	23	-5	-342	358	23	***	4	12	L	***	-3	93	72	210				
				-12	-150	77	91	***	11	9	L	-11	-247	201	57	***	8	10	L	***	3	326	364	20	-3	410	419	17	6	-215	268	25	-7	224	260	23	-6	510	513	20	-6	510	513	20	***	4	12	L	***	-3	93	72	210				
				-11	-194	205	29	***	11	9	L	-10	84	0	241	W	***	8	10	L	***	4	-251	248	23	-2	-342	340	20	7	113	111	51	-6	-64	0	120	W	7	371	389	20	7	371	389	20	***	4	12	L	***	-3	93	72	210		
				-10	-12	179	24	***	11	9	L	-9	706	690	27	-9	877	877	22	-13	-505	518	18	5	510	524	18	-1	-13	141	28	8	-133	183	26	-5	135	189	26	8	-582	615	18	8	-582	615	18	***	4	12	L	***	-3	93	72	210	
				-9	-507	535	18	***	11	9	L	-8	-341	344	44	-8	-619	619	19	-12	411	410	20	6	391	394	19	0	-336	306	18	9	-241	252	24	-4	-392	379	21	9	-167	177	32	9	-167	177	32	***	4	12	L	***	-3	93	72	210	
				-8	-305	346	19	***	11	9	L	-7	-903	909	27	-7	-701	677	20	-11	697	684	21	7	399	395	18	1	-554	573	20	10	350	370	18	-3	-483	501	18	10	285	301	18	10	285	301	18	***	4	12	L	***	-3	93	72	210	
				-7	1101	1087	25	***	11	9	L	-6	-420	425	37	-6	-399	428	16	-10	-451	485	18	-10	-451	485	18	2	798	794	23	-2	979	984	23	-2	979	984	23	***	4	12	L	***	-3	93	72	210									
				-6	-650	654	18	***	11	9	L	-5	642	663	27	-5	-136	134	35	-9	-572	584	19	***	16	10	L	***	3	835	850	23	***	13	11	L	***	-1	640	678	18	***	13	11	L	***	-1	640	678	18							
				-5	-1182	1183	26	***	11	9	L	-4	-220	290	42	-4	459	460	17	-8	355	395	18	***	16	10	L	***	4	-914	904	23	***	13	11	L	***	0	-994	975	22	***	13	11	L	***	0	-994	975	22							
				-4	431	439	16	***	11	9	L	-3	227	188	152	-3	802	805	19	-7	113	105	61	-3	-473	367	35	5	-609	624	21	-8	477	357	39	1	-378	390	25	-8	266	273	43	-8	266	273	43	***	4	12	L	***	-3	93	72	210	
				-3	855	864	20	***	11	9	L	-2	26	81	174	-2	-570	614	16	-6	86	169	32	-2	362	219	56	6	584	592	20	-7	413	384	37	2	632	681	20	-7	-166	293	38	-7	-166	293	38	***	4	12	L	***	-3	93	72	210	
				-2	-474	267	18	***	11	9	L	-1	-532	509	33	-1	-1289	1301	27	8	451	459	21	-1	252	0	240	W	-6	-522	493	33	3	122	56	131	-6	290	355	29	-6	290	355	29	***	4	12	L	***	-3	93	72	210				
				-1	206	188	26	***	11	9	L	0	184	202	33	0	995	981	24	9	574	589	19	-8	399	382	21	-5	-862	858	21	4	174	183	47	-5	114	34	23	-5	114	34	23	***	4	12	L	***	-3	93	72	210					
				0	-179	200	15	***	11	9	L	1	805	832	33	1	1160	1188	26	-3	-927	947	23	1	44	143	31	9	156	71	103	-4	234	152	103	5	256	235	32	-4	-529	536	26	-4	-529	536	26	***	4	12	L	***	-3	93	72	210	
				1	-1028	1012	24	***	11	9	L	2	-454	447	23	2	-560	569	19	-2	676	688	20	2	9	0	97	W	10	-325	259	32	-3	-90	0	337	W	-3	-358	443	32	-3	-358	443	32	***	4	12	L	***	-3	93	72	210			
				2	432	442	19	***	11	9	L	3	-809	801	22	3	-476	506	18	-1	884	877	22	-1	884	877	22	11	-542	541	19	-2	78	0	345	W	-2	682	706	25	-2	682	706	25	***	4	12	L	***	-3	93	72	210				
				3	1419	1408	30	***	11	9	L	4	317	324	23	4	115	69	86	0	-674	671	17	***	1	11	L	***	12	511	532	18	-1	360	267	65	8	874	862	23	-1	291	285	49	-1	291	285	49	***	4	12	L	***	-3	93	72	210
				4	-714	715	20	***	11	9	L	5	383	326	25	5	-343	358	19	1	-588	601	21	-15	-397	394	18	13	476	458	19	0	-481	498	18	9	368	310	28	0	-354	347	22	0	-354</												

Table 2 (cont.)

L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG	L	FC	FO	SIG					
***	3	13	L	***	7	13	L	***	0	14	L	***	4	14	L	***	10	14	L	***	3	15	L	***	11	15	L	***	6	16	L	***	3	17	L	***												
-5	282	292	24	4	763	760	23	0	-1037	1050	26	10	-591	567	20	4	-335	338	25	10	306	286	23	-3	133	129	72	-8	-335	342	20	1	-207	177	61													
-4	-1036	1064	25	5	69	172	32	1	-39	0	147	W	11	11	74	66	5	-97	0	133	W	5	-97	0	133	W	-2	-143	247	39	-7	142	162	35														
-2	-288	260	27	6	-481	488	21	2	912	904	23	3	92	0	150	W	6	651	651	19	***	5	15	L	***	-1	-54	158	60	-6	-61	133	41	3	387	350	30											
-3	360	353	22	7	24	85	75	3	92	0	150	W	***	6	14	L	***	7	20	0	101	W	7	20	0	101	W	0	445	452	18	-5	54	100	60	4	554	573	20									
-1	-38	70	82	8	-25	0	139	W	4	-234	213	35	-11	-41	0	119	W	***	12	14	L	***	-10	-114	63	94	1	20	0	129	W	-4	463	475	20	5	-427	388	24									
0	164	188	25	9	-195	214	27	5	48	76	95	5	48	76	95	-10	750	748	20	-9	-50	0	112	W	2	-659	700	20	-3	-195	182	38	6	-440	462	18												
2	-858	789	25	11	218	143	43	7	-84	65	104	-9	-13	0	123	W	-4	-636	580	25	-7	-93	0	139	W	3	163	199	26	-2	-723	722	21	7	215	233	26											
3	-316	306	29	***	9	13	L	***	8	744	758	21	-8	-479	472	20	-3	-61	168	71	-6	-862	894	22	***	0	16	L	***	0	689	678	19	***	5	17	L	***										
4	1031	997	26	***	9	13	L	***	9	13	108	56	-7	-18	49	122	-2	651	654	24	-5	132	0	146	W	0	-802	801	24	2	-263	320	26	-6	-607	610	18											
5	220	198	44	***	9	13	L	***	10	-727	732	21	-6	81	143	40	-1	-58	83	131	-4	635	644	20	1	297	305	28	3	97	140	52	-5	382	409	19												
6	-786	760	23	-10	-321	301	41	11	-46	0	124	W	-4	610	608	21	1	67	99	58	-2	-260	250	31	2	610	582	22	4	-177	228	29	-4	429	390	23												
7	-53	113	59	-9	-142	213	59	12	406	414	17	-3	120	188	30	2	18	0	127	W	1	47	112	58	3	-176	146	52	5	75	114	57	-3	-325	272	30												
8	128	174	38	-7	603	589	28	***	2	14	L	***	-2	-830	858	22	3	-114	112	51	0	-339	354	21	4	-85	0	161	W	6	440	428	21	-2	-154	178	38											
9	-105	158	38	-6	-239	290	42	-12	-499	484	20	-1	-17	126	45	0	867	875	21	1	-25	26	386	***	1	15	L	***	3	-188	224	36	7	179	185	40	-1	34	0	141	W							
10	246	290	23	-5	-837	840	24	-11	-79	150	36	2	-294	287	34	2	-294	287	34	2	-294	287	34	***	8	16	L	***	6	-260	283	22	1	224	177	61	2	507	490	25								
11	207	223	28	-4	512	483	32	-10	847	828	21	3	32	0	154	W	-11	44	0	120	W	11	44	0	120	W	5	135	265	24	9	-242	173	47	-6	179	196	31										
12	-614	624	19	-3	-49	131	102	-8	-688	694	20	4	-138	139	57	5	-154	129	55	-9	33	0	131	W	6	454	468	21	***	2	16	L	***	-4	-452	459	18	5	365	345	25							
13	-217	245	20	-2	7	73	185	-7	12	88	75	5	-154	129	55	-9	33	0	131	W	7	-137	152	41	7	-137	152	41	8	-66	163	33	9	67	0	120	W	8	-66	163	33							
-12	-403	419	18	1	-275	300	30	-5	17	0	131	W	7	72	136	47	-7	-45	123	49	9	67	0	120	W	10	-360	381	18	-9	-227	251	25	-8	-544	541	19	-1	-269	278	26							
-11	-92	0	122	W	2	742	743	24	-4	400	420	20	8	-682	657	21	-6	-797	785	23	-5	107	0	151	W	***	7	15	L	***	-6	206	164	46	1	211	182	52	-4	-10	387	21						
-10	-161	174	35	4	-772	787	22	-2	-792	809	22	-1	-139	90	69	11	-12	0	88	W	-2	-552	576	22	-9	-44	0	109	W	-4	359	341	23	3	-92	135	49	-2	6	135	40							
-9	-126	150	42	5	-82	0	147	W	6	262	283	25	1	7	0	191	W	***	8	14	L	***	-1	-38	0	126	W	-1	-38	0	126	W	-3	-184	174	43	4	343	350	24	-1	-39	135	42				
-8	641	651	20	7	-39	115	47	0	1126	1102	24	3	28	186	39	-10	-639	669	19	1	41	0	208	W	0	-38	170	30	-7	101	141	43	-2	-690	698	22	5	-90	133	42								
-7	220	196	35	8	113	165	30	1	7	0	191	W	4	147	183	45	-9	34	276	22	2	580	574	23	-5	-144	108	59	0	784	796	20	1	-281	301	35	***	10	16	L	***							
-6	-914	927	22	9	162	147	39	2	-551	550	24	3	28	186	39	-10	-639	669	19	1	41	0	208	W	4	-224	224	37	-4	-626	611	20	2	-608	653	23	-1	260	0	270	W							
-5	-236	232	29	10	-418	420	17	5	-10	0	164	W	6	617	601	23	-7	-85	206	23	4	-924	912	24	5	161	161	50	-2	38	0	327	W	3	147	59	176	0	388	363	22	***	0	18	L	***		
-4	819	801	21	***	11	13	L	***	7	111	145	48	8	-784	787	23	-5	58	153	31	6	724	709	22	-1	10	0	343	W	4	21	73	116	***	1	17	L	***	0	-456	467	25						
-3	-396	388	20	***	11	13	L	***	8	-784	787	23	-5	58	153	31	6	724	709	22	-1	10	0	343	W	4	21	73	116	***	1	17	L	***	5	32	135	53	***	1	17	L	***					
-2	177	221	48	***	11	13	L	***	9	-67	62	104	-4	-627	659	20	8	-279	250	34	1	55	161	48	6	397	411	23	7	-198	179	41	-8	244	211	32	2	424	415	24								
1	277	221	48	***	11	13	L	***	10	-639	669	19	1	41	0	208	W	1	41	0	208	W	6	717	712	20	-5	-144	108	59	0	784	796	20	1	-281	301	35	***	7	17	L	***					
2	786	768	25	***	11	13	L	***	11	-12	0	88	W	-3	-133	155	40	-2	-552	576	22	-9	-44	0	109	W	-4	359	341	23	3	-92	135	49	-2	6	135	40	-1	-39	135	42						
3	341	343	29	***	11	13	L	***	12	-321	294	22	0	-723	732	19	1	84	42	204	***	3	15	L	***	8	-103	134	37	-9	239	239	24	-2	-386	386	24	-5	4	0	128	W						
4	-962	969	24	***	11	13	L	***	1	84	42	204	***	3	15	L	***	3	-99	111	67	4	269	294	26	-11	-32	0	105	W	-8	507	461	21	-1	187	215	33	-4	197	179	43						
5	-212	255	30	***	11	13	L	***	2	255	251	34	3	-99	111	67	4	269	294	26	-11	-32	0	105	W	-10	-31	175	28	-7	-135	125	49	0	-101	127	49	-3	-296	301	24							
6	619	626	21	***	11	13	L	***	3	-99	111	67	4	269	294	26	-11	-32	0	105	W	-10	-31	175	28	-7	-135	125	49	0	-101	127	49	-3	-296	301	24	-2	-357	362	23							
7	104	146	44	***	11	13	L	***	4	269	294	26	-11	-32	0	105	W	-10	-31	175	28	-7	-135	125	49	0	-101	127	49	-3	-296	301	24	-2	-357	362	23	-1	527	524	20							
8	-86	0	147	W	5	-10	0	164	W	5	-10	0	164	W	6	617	601	23	-7	-85	206	23	4	-924	912	24	5	161	161	50	-2	38	0	327	W	3	147	59	176	0	388	363	22	***	0	18	L	***
9	155	227	26	***	11	13	L	***	6	617	601	23	-7	-85	206	23	4	-924	912	24	5	161	161	50	-2	38	0	327	W	3	147	59	176	0	388	363	22	***	0	18	L	***						
10	-436	437	20	***	11	13	L	***	7	111	145	48	8	-784	787	23	-5	58	153	31	6	724	709	22	-1	10	0	343	W	4	21	73	116	***	1	17	L	***	5	32	135	53	***	1	17	L	***	
11	-127	128	44	***	11	13	L	***	8	-784	787	23	-5	58	153	31	6	724	709	22	-1	10	0	343	W	4	21	73																				

Atomic position and thermal vibration parameters from the final least-squares cycle are listed in Table 1. A comparison of observed and computed $|F|$ values is given in Table 2. A final difference Fourier synthesis with all atoms removed showed no positive or negative peak containing more than 0.5 electron. The largest excursions on this map were associated with the subtracted lanthanum atom.

Description of the structure

The *ORFFE* program of Busing, Martin & Levy (1964) was used with the parameters of Table 1 to compute interatomic distances and angles. A selection of the more important of these appears in Table 3. Several drawings of the structure were prepared with the *ORTEP* program written by Johnson (1965).

Table 3. *Interatomic distances and angles in $\text{La}_2\text{Be}_2\text{O}_5$*

Distances		Standard deviation
Be-O(1)	1.602 Å	0.004 Å
Be-O(2)	1.637	0.004
Be-O(3)	1.661	0.003
Be-O(4)	1.678	0.004
La-O(1)	2.415	0.002
La-O(3)	2.426	0.002
La-O(1)	2.480	0.002
La-O(4)	2.502 ₃	0.002 ₂
La-O(2)	2.556 ₃	0.000 ₄
La-O(1)	2.719	0.002
La-O(2)	2.755 ₅	0.000 ₃
La-O(3)	2.903	0.002
La-O(4)	2.971 ₄	0.000 ₃
La-O(1)	2.999	0.002
Angles		Standard deviation
O(1)-Be-O(4)	113.4°	0.2
O(1)-Be-O(3)	111.8	0.2
O(1)-Be-O(2)	117.4	0.2
O(2)-Be-O(4)	105.1	0.2
O(2)-Be-O(3)	102.9	0.2
O(3)-Be-O(4)	105.0	0.2

From the results listed in Table 3, we see at once that the beryllium-oxygen coordination polyhedron in $\text{La}_2\text{Be}_2\text{O}_5$ closely approximates a regular tetrahedron with interatomic distances comparable to those found in BeO (Smith, Newkirk & Kahn, 1964) and chrysoberyl, Al_2BeO_4 (Farrell, Fang & Newnham, 1963). Further consideration shows that each beryllium-oxygen tetrahedron shares three of its four corners with three other beryllium-oxygen tetrahedra. The linked tetrahedra form a space-filling framework* that is an important feature of the structure.

To describe the inter-tetrahedral linkages, we note that each tetrahedron is oriented so that three of its

* We used the word 'framework' for want of a better term. It does not conform to the framework concept used in silicate structures since only three of the four corners of each tetrahedron are shared. The name 'interrupted framework' might be a more correct description.

Be-O vectors are roughly parallel to the unit-cell axes. The oxygen atoms at the ends of these vectors are the ones shared with adjacent tetrahedra. In an idealized representation, we may neglect the departures of the monoclinic angle and of the O-Be-O angles from 90° and replace each tetrahedron by three orthogonal vectors based at the central beryllium atom and directed along the idealized bonds to the shared oxygen atoms. The result of doing this is shown in Fig. 1.

Depending on the direction of view, one may reconstruct the three-dimensional pattern by considering strings of corner-sharing tetrahedra to be extended through the structure, repeated by axial translations into two-dimensional arrays, and cross-linked by corner-sharing with tetrahedra in adjacent related arrays. Viewed in the *a* direction, one may see strings with a 'squarewave' appearance (Fig. 1) resembling those found in petalite, $\text{LiAlSi}_4\text{O}_{10}$ (Liebau, 1961*c*), extending in [001] directions. Arrays on 200 planes are formed by the *b* lattice repetition and arrays on adjacent 200 planes are related by the *C*-centering operation. Viewed in the *c* direction, one may see strings resembling those found in diopside, $\text{CaMg}(\text{SiO}_3)_2$ (Warren & Bragg, 1928), extending in [110] or $[\bar{1}10]$ directions. Arrays on 002 planes are formed by the *a* (or *b*) lattice repetition and arrays on adjacent 002 planes are related by the two-fold axes half-way between them. Viewed in the *b* direction, one may see (Fig. 2) strings that are a variation of those found in wollastonite, CaSiO_3 (Mamedov & Belov, 1956), extending in [101] directions. Arrays on 020 planes are formed by the *c* (or *a*) lattice repetition and arrays on adjacent 020 planes are related by the *c*-glide operation.

The consequence of this linking together of tetrahedral strings is the formation of cube-like cages of tetrahedra whose outline is easily seen in Figs. 1 and 2. We note that six edges of a cage follow Be-O-Be bonds while the remaining six are only suggested by opposed tetrahedra faces. Each cage provides a location for a lanthanum atom, but the lanthanum-oxygen coordination polyhedron is rather irregular with five close La-O contacts, two longer contacts, and three still longer contacts in which interactions must be relatively weak (see Table 3). A detailed view of such a lanthanum atom environment is given in Fig. 3.

A few additional points are noteworthy. As shown in Fig. 3, the thermal motion ellipsoids of the oxygen atoms have principal axes normal and parallel to Be-O bonds - a further evidence of the structural importance of the tetrahedral framework. The thermal vibration of the O(4) atom is unusual in that it is large and directed along the line of two long (2.97 Å) contacts with lanthanum atoms. One might imagine that this reflects a static displacement of the O(4) atom favored by the resultant shortening of one of the 2.97 Å lanthanum contacts. Such a displacement would also produce a nonlinear Be-O-Be sequence which might be energetically favored, in analogy with the behavior of corner-sharing (SiO_4) tetrahedra (Liebau, 1961*d*). To

this point, note that the Be–O(3)–Be angle is $163.5 \pm 0.3^\circ$, but that the Be–O(2)–Be angle is required to be 180° and the thermal vibration parameters of the O(2) atom do not appear abnormal. We did not test structure models in which either the center of symmetry was removed or a pair of partial atoms replaced the O(4) atom.

Finally, the Be–O(1) bond is significantly shorter than the other three beryllium–oxygen bonds, and O(1) is the oxygen atom not shared with an adjacent tetrahedron.* A similar reduction in bond length with decreasing degree of oxygen association between tetrahedra has been reported in structures containing connected PO_4 , SiO_4 and BO_4 tetrahedra (de Decker, 1941; Grund, 1954; Prewitt & Shannon, 1967, respectively).

Discussion

Beryllium oxide (Smith, Newkirk & Kahn, 1964), Al_2BeO_4 (Bragg & Brown, 1926), and Cr_2BeO_4 (Weir & Van Valkenburg, 1960) have structures which are described in terms of close-packed layers of oxygen atoms with small metal atoms regularly occupying certain interstitial sites. The structure of beryl, $\text{Be}_3\text{Al}_2\text{Si}_6\text{O}_{18}$, is usually classified as a ring silicate in which beryllium atoms act as an inter-ring link while preserving their tetrahedral environment (Bragg & West, 1926). Phenakite, Be_2SiO_4 , has a three-dimensional arrangement of corner-sharing (BeO_4) and (SiO_4) tetrahedra in which each oxygen atom is bonded to two beryllium atoms and one silicon atom (Bragg & Zachariasen, 1930).

In $\text{La}_2\text{Be}_2\text{O}_5$, we see a structure that must be described as a three-dimensional framework formed exclusively of linked (BeO_4) tetrahedra. Although close contacts occur between oxygen atoms of a given BeO_4

tetrahedron, there are no extensive parts of this framework in which the oxygen or oxygen–lanthanum atom arrangement is close-packed. The structure of $\text{La}_2\text{Be}_2\text{O}_5$ may in this sense be compared with silicate structures; some associations with known silicate string configurations were made in the preceding section.

Silicates of general formula $\text{A}_2\text{Si}_2\text{O}_5$ are known and structures for compounds with $\text{A} = \text{Li}, \text{Na}$, and (0.25 Li, 0.25 Al, 0.5 vacancy) have been reported (Liebau, 1961*a, b, c*; Grund, 1954). In all studied to date, the silicate tetrahedra combine into sheets with A cations lying between the sheets. Structures of alkali titanates of general formula $\text{A}_2\text{Ti}_2\text{O}_5$ have also been given (Barblan, 1943; Andersson & Wadsley, 1961). Here too, sheet-like combinations of TiO_4 tetrahedra* are found. The structures of V_2O_5 * and the stable orthorhombic forms of P_2O_5 contain corner-sharing XO_4 tetrahedra; V_2O_5 * (Ketelaar, 1936) and the so-called third form of P_2O_5 (MacGillavry, de Decker & Nyland, 1949) have sheet-like configurations, but the second form of P_2O_5 has a three-dimensional framework with spirals of PO_4 tetrahedra about 2_1 screw axes (de Decker, 1941).

The BeO_4 tetrahedra in $\text{La}_2\text{Be}_2\text{O}_5$ do not join to form sheets, perhaps because of the high surface charge that a sheet of net composition Be_2O_5 would have. Rather, they form a unique three-dimensional framework of corner-sharing tetrahedra with cage-like sites for the large lanthanum cations. The second form of P_2O_5 is the only other example of an X_2O_5 framework, but the motifs are not alike and the effect of charge neutralization is absent in P_2O_5 .

It is important to contrast the $\text{La}_2\text{Be}_2\text{O}_5$ structure with the metastable $\text{Ca}_{12}\text{Be}_{17}\text{O}_{29}$ and Y_2BeO_4 structures (Harris & Yakel, 1966, 1967). In the latter, linear or

* All oxygen atoms have four lanthanum atom near neighbors at distances from 2.415 to 2.999 Å.

* The situation is complicated in these structures owing to the approach of a fifth oxygen atom to the XO_4 tetrahedra to give trigonal bipyramidal coordination (see also Byström, Wilhelm & Brotzen, 1950).

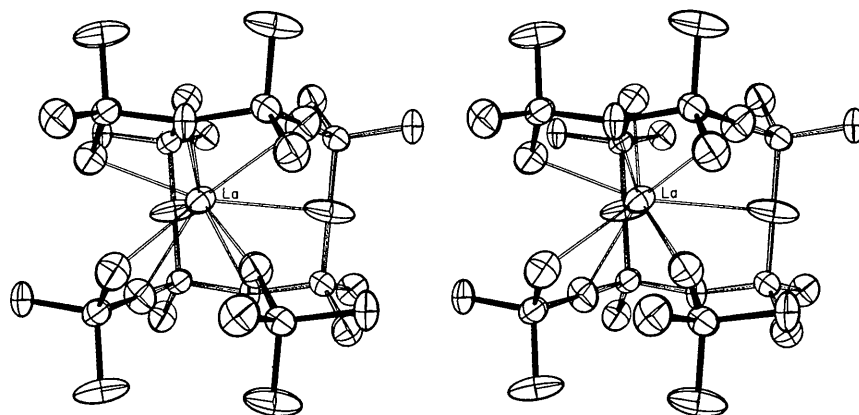


Fig. 3. Stereoscopic drawings of the lanthanum atom environment in $\text{La}_2\text{Be}_2\text{O}_5$. The unit-cell origin is half-way between the O(4) atoms (elongated ellipsoids) at the left of the drawing. The $+b$ axis is vertical and directed upward in the drawing plane. The $+c$ axis emerges up and to the right from the paper at an angle of 25° to the drawing plane, and the $+a$ axis emerges behind the paper in a right-handed relation to b and c . Atoms are represented by thermal displacement ellipsoids including 99.9% probability ($4 \times \text{r.m.s. displacement}$). Bounding and principal ellipses only are shown for lanthanum and beryllium atoms; forward principal axes are added for oxygen atoms. Thin bonds between lanthanum and oxygen atoms indicate important short contacts.

planar groupings of relatively normal calcium–oxygen and yttrium–oxygen coordination polyhedra were found, but beryllium atoms were not normally coordinated. Oxygen environments about beryllium atoms ranging from tetrahedral to trigonal were reported in $\text{Ca}_{12}\text{Be}_{17}\text{O}_{29}$ and trigonal environments exclusively were reported in Y_2BeO_4 . The principle underlying both structures seems to be the maintenance of normal heavy cation–oxygen coordination, albeit in a metastable phase, with beryllium atoms fitting in where best they can. The situation is reversed in $\text{La}_2\text{Be}_2\text{O}_5$. Here the principle underlying the structure seems to be the maintenance of a framework of normal BeO_4 tetrahedra with lanthanum atoms fitting in where best they can.

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