

## SHORT COMMUNICATIONS

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**The refined structure of SrO · 6Al<sub>2</sub>O<sub>3</sub>.** By A. J. LINDOP,\* C. MATTHEWS† and D. W. GOODWIN, *Department of Physics, University of York, Heslington, York YO1 5DD, England*

A full refinement of the mixed oxide SrO · 6Al<sub>2</sub>O<sub>3</sub>, having the magnetoplumbite structure, has been performed. Data were collected on a computer-controlled four-circle X-ray diffractometer using Mo K $\alpha$  radiation. Final  $R=0.060$ .

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Continuing our study of the alkali-earth aluminates for the purpose of finding suitable hosts for the Nd<sup>3+</sup> ion, which provides the active element in a number of successful solid-state lasers, a number of single crystals of SrO · 6Al<sub>2</sub>O<sub>3</sub> containing various concentrations of both Nd<sup>3+</sup> and Cr<sup>3+</sup> have been grown with Czochralski techniques (Goodwin & Lindop, 1970). As an aid to the analysis of the optical properties of these crystals it is essential to know the exact environment of the dopant ions and so a full refinement of the crystal structure has been performed on a sample of Nd<sup>3+</sup> doped material. The structure was first examined by Adelsköld (1938) and found to be isomorphous with the structure of magnetoplumbite PbO · 6Fe<sub>2</sub>O<sub>3</sub>.

In order to minimize the effects of a high linear absorption coefficient for X-rays an approximately spherical crystal was cut and ground using techniques previously described for SrO · 2Al<sub>2</sub>O<sub>3</sub> (Lindop & Goodwin, 1972). The result was a sample having an absorption factor of  $\mu R=1.7$  for Mo K $\alpha$  X-radiation. Following an initial alignment using a precession camera and Cu K $\alpha$  radiation, a complete set of X-ray reflexion data was collected with Mo K $\alpha$  radiation on a Hilger and Watts four-circle computer-controlled diffractometer.

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The system provided an integrated intensity from a combined  $\omega$  and  $\theta$  step scan for each reflexion with a diffraction angle  $2\theta$  less than 60°. In all 5028 reflexions were measured. After correction for absorption, according to the factors for a sphere having  $\mu R=1.7$  as quoted in *International Tables for X-ray Crystallography* (1967), equivalents were combined to produce a total of 609 structure amplitudes.

The structure belongs to the hexagonal space group  $P6_3/mmc$ , and has been well described by Braun (1957) in terms of another isomorphous compound BaO · 6Fe<sub>2</sub>O<sub>3</sub> which was of interest for its magnetic properties. The unit-cell parameters for SrO · 6Al<sub>2</sub>O<sub>3</sub> were quoted as  $a=5.568$ ,  $c=21.99$  Å by Wyckoff (1965), which are not significantly different from the values in Table 1 for this work.

Table 1. Unit-cell parameters of SrO · 6Al<sub>2</sub>O<sub>3</sub>

$$\begin{aligned} a &= 5.562 \pm 0.002 \text{ \AA} \\ b &= 5.562 \pm 0.002 \\ c &= 21.972 \pm 0.005 \end{aligned}$$

The starting point for the refinement was chosen as the structure of PbO · 6Fe<sub>2</sub>O<sub>3</sub> (Wyckoff, 1965). Owing to an error in Wyckoff (1965) the parameters for Al(5) were taken from the related structure of  $\beta$ -alumina.

The refinement of the structure was achieved with a modified version of the least-squares program of Busing, Martin & Levy (1962), using ionic scattering factors for

Table 2. Positional and thermal ( $\times 10^6$ ) parameters of atoms in SrO · 6Al<sub>2</sub>O<sub>3</sub>

	$x$	$y$	$z$	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Sr	$\frac{2}{3}$	$\frac{1}{3}$	$\frac{1}{2}$	10245	10245	7823	5123	0	0
Al(1)	0	0	0	6884	6884	2917	3442	0	0
Al(2)	0	0	$\frac{1}{2}$	5950	5950	55515	2975	0	0
Al(3)	$\frac{1}{3}$	$\frac{2}{3}$	0.0276	6842	6842	2390	3421	0	0
Al(4)	0.1685	0.3370	-0.1082	6270	6426	3760	3213	175	350
Al(5)	$\frac{1}{3}$	$\frac{2}{3}$	0.1903	6789	6789	3976	3395	0	0
O(1)	0	0	0.1481	5110	5110	4221	2555	0	0
O(2)	$\frac{1}{3}$	$\frac{2}{3}$	-0.0538	6427	6427	377	3214	0	0
O(3)	0.1822	0.3644	$\frac{1}{2}$	10812	7742	2559	3871	0	0
O(4)	0.1552	0.3104	0.0523	6232	4701	1320	2351	587	1174
O(5)	0.5025	1.0050	0.1476	6045	6684	5550	3342	1114	2229
Average errors	$\pm 0.0005$	$\pm 0.0010$	$\pm 0.0002$	$\pm 1200$	$\pm 1200$	$\pm 1600$	$\pm 600$	$\pm 100\%$	$\pm 100\%$

O<sup>2-</sup> and Al from *International Tables for X-ray Crystallography* (1968) and for Sr from Cromer & Waber (1965). A correction for secondary extinction was applied in the refinement using the technique of Larson (1969).

Normal refinement using isotropic temperature factors produced a satisfactory convergence of the parameters. This was completed with anisotropic temperature factors until the calculated parameter alterations were exceeded by their standard deviations. At this point a minimum residual  $R=0.060$  was obtained. This is a particularly satisfactory low value of  $R$  in view of the uncertainty in the absorption correction due to deviations from a true spherical sample.\*

Table 3. *Variable parameters for the structure of CaO.6Al<sub>2</sub>O<sub>3</sub> after Kato & Saalfeld (1968)*

	<i>x</i>	<i>z</i>
Al(3)		0.0280
Al(4)	0.1687	-0.1089
Al(5)		0.1904
O(1)		0.1501
O(2)		-0.0555
O(3)	0.1806	
O(4)	0.1549	0.0524
O(5)	0.5036	0.1501
	$a = 5.564 \text{ \AA}$	
	$c = 21.892 \text{ \AA}$	

The final values of the positional and thermal parameters are shown in Table 2, where the thermal parameters have been multiplied by 10<sup>6</sup>. The deviations from the starting structure of PbO.6Fe<sub>2</sub>O<sub>3</sub> are quite significant and a great deal larger than the errors in the parameters. However, a comparison with the structure of the isomorphous compound CaO.6Al<sub>2</sub>O<sub>3</sub> measured by Kato & Saalfeld (1968), the variable parameters of which are summarized in Table

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31307 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

3, shows that the two aluminates have very similar parameters. The differences between the Sr and the Ca compounds are small but in some cases an order of magnitude greater than the errors in either set of parameters. Similarly if one compares the bond distances and angles between the two compounds one obtains only small differences with no systematic change as found for SrO.2Al<sub>2</sub>O<sub>3</sub> and CaO.2Al<sub>2</sub>O<sub>3</sub> (Lindop & Goodwin, 1972). The application of this refined structure to crystal field calculations for determining the e.s.r. and optical spectra of both Nd<sup>3+</sup> and Cr<sup>3+</sup> ions is in progress and will be reported elsewhere.

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**DL-Leucine: errata.** By BENEDETTO DI BLASIO, CARLO PEDONE and AUGUSTO SIRIGU, *Istituto Chimico, Università di Napoli, Via Mezzocannone 4, 80134 Napoli, Italy*

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In Table 1 of the paper by Di Blasio, Pedone & Sirigu [*Acta Cryst.* (1975). **B31**, 601-602] the positional and thermal parameters are referred to the unit cell where the  $b$  and  $c$  axes are interchanged.  $B_{33}$  for O(1), listed as 2860 (73), should be 2960 (73).

All information is contained in the abstract.