SHORT COMMUNICATION

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Single-crystal refinement of the structure of LaOCl.

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Abstract

LaOCl crystallizes in the tetragonal *P4/nmm* structure of PbFCl with $a = 4{\cdot}1209\ (2)$ and $c = 6{\cdot}8840\ (7)$ Å; $Z = 2$. Single crystals of LaOCl were grown from a $BaCl_2$ flux. Least-squares single-crystal refinement based on 81 independent reflections led to an R value of 0·011. Powder data based on Guinier refinements are also reported. No significant differences over the previous powder work [Sillén & Nylander (1941). *Sven. Kem. Tidskr.* **53**, 367] are found. (JCPDS Diffraction File No. 34–1494.)

Introduction

The structural parameters of LaOCl have been refined using single-crystal data. Except for the greater precision, the results are the same as those obtained by Sillén & Nylander (1941) from powder diffraction data.

Experimental

Single crystals of LaOCl were grown by slowly cooling a 1:1 mixture of LaOCl powder and $BaCl_2$ flux from 1373 K to 773 K at a rate of 5 Kh^{-1}. The LaOCl powder precursor was obtained by blending high-purity La_2O_3 (99·999% from Research Chemicals Co.) with an equal weight of NH_4Cl (Fisher Analytical) and heating it in an Al_2O_3 container to 573, 773 and 1173 K for 2–4 h each.

A single crystal (0·04 × 0·11 × 0·11 mm) was used for the intensity measurements on an Enraf–Nonius CAD-4 automatic diffractometer. 87 independent reflections were recorded using the 2θ–θ technique ($2\theta < 55°$; Mo $K\alpha$ radiation; $\lambda = 0{\cdot}71069$ Å). An analytical absorption correction based on the crystal shape was applied; transmission coefficients ranged from 0·189 to 0·708 ($\mu = 191{\cdot}6\ cm^{-1}$).

The structure was refined using 81 reflections with $I > 2\sigma(I)$. In the least-squares refinement, the function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F_o)$. The standard deviations $\sigma(F_o)$ were based on counting statistics and an 'ignorance factor' p, of 0·02 (Corfield, Doedens & Ibers, 1967). The refinement converged at $R = 0{\cdot}011$ and $R_w = 0{\cdot}015$ (8 variables: La with anisotropic thermal parameters, O and Cl with isotropic parameters, scale, extinction parameter $3{\cdot}0\ (1) \times 10^{-6}$, shift/error essentially zero). Computational and mathematical details can be found elsewhere (Frenz, 1978; Nugent & Harlow, 1979).

The final positional and thermal parameters are listed in Table 1.* The interatomic distances are listed in Table 2, and Table 3 gives a comparison of the single-crystal lattice parameters with Sillén & Nylander's (1941) data.

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Table 1. *Positional and thermal* (Å^2) *parameters and their standard deviations*

	x	y	z	B_{11}/B_{iso}	B^*_{33}
La	0·25	0·25	0·17552 (6)	0·64 (2)	0·59 (2)
O	0·25	0·75	0·00	0·72 (9)	
Cl	0·25	0·25	0·6298 (3)	1·18 (3)	

* The form of the anisotropic temperature factor expression is: $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + \cdots + 2\,B_{23}klb^*c^*)]$.

Table 2. *Interatomic distances for* LaOCl *in* Å

La–O	2·387 (1) (× 4)	La–Cl	3·126 (2) (× 4)
O–O	2·911 (1) (× 4)	La–Cl	3·205 (1) (× 1)
O–Cl	3·279 (1) (× 4)	Cl–Cl	3·415 (2) (× 4)

Table 3. *Comparison of single-crystal cell parameters and z coordinates with previous data*

a(Å)	c(Å)	V(Å^3)	La	Cl	Source
4·109	6·865	115·9	0·178 (6)	0·635 (15)	Sillén & Nylander (1941)
4·117 (1)	6·881 (1)	116·63	0·17552 (6)	0·6298 (3)	Present work on single crystal
4·1209 (2)	6·8840 (7)	116·90 (1)	—	—	Powder data

* Tables of structure factors and X-ray powder diffraction data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38586 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU. England.

Powder diffraction patterns were obtained with a Guinier–Hägg-type focusing camera ($r = 40$ mm). The radiation was monochromatic Cu $K\alpha_1$ ($\lambda = 1{\cdot}5405$ Å), and KCl ($a = 6{\cdot}2931$ Å) was used as an internal standard. The lattice parameters were refined by least-squares procedure, and are listed in Table 3. The densities are: $D_x = 5{\cdot}407\ (5)$, $D_m = 5{\cdot}39\ g\ cm^{-3}$.

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