

requires about two bonds from tetrahedrally or trigonally coordinated lithium to saturate it. I have not succeeded in finding a satisfactory arrangement for the lithium atoms compatible with the assigned space group; perhaps the space-group assignment is wrong or the lithium atoms occupy a larger number of equivalent positions with some randomness. Thus in the plane $z = 0$ there are ten fluorine tetrahedra that might be occupied by lithium atoms; possibly Li(1) atoms occupy two of them, as in the reported structure, with Li(2) atoms in half of the other eight. The total electrostatic bond strengths would then be 0.94 for F(1), 1.07 for F(2), and 0.89 for F(3), in satisfactory agreement with the rule.

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Acta Cryst. (1979). B35, 1536

(+)-Lupanine perchlorate monohydrate: erratum. By H. MAŁUSZYŃSKA, A. HOSER and Z. KAŁUSKI, *Institute of Chemistry, A. Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland*

(Received 18 April 1979)

Abstract

In Table 2 of Małuszyńska, Hoser & Kałuski [*Acta Cryst.* (1979), B35, 970–973] the coefficient of Y in the equation for plane 3 should read 0.9657.

All relevant information is contained in the *Abstract*.

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Acta Cryst. (1979). B35, 1536

The ethyl carbonate of 1-isoquinolyl(phenyl)methanol. By EWA SKRZYPCZAK-JANKUN, and ZYGMUNT KAŁUSKI, *Chemistry Department, A. Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland*

(Received 18 April 1979)

Abstract

A printer's error is corrected. In Skrzypczak-Jankun & Kałuski [*Acta Cryst.* (1977), B33, 3921–3923] the first two lines in the right-hand column of text on p. 3922 should read: 'isoquinolyl)pyrazole (King & Reimlinger, 1971), and 3-methylisoquinoline (Ribár, Divjaković, Janić, Argay,'

All relevant information is contained in the *Abstract*.

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Acta Cryst. (1979). B35, 1536–1537

The crystal structure of BaCaLu₂F₁₀. By A. VÉDRINE and D. TROTTIER, *Laboratoire de Chimie des Solides, ENSCCF et UER Sciences exactes et naturelles, Université de Clermont-Ferrand II, BP 45, 63170 Aubière, France* and R. CHEVALIER, *Laboratoire de Cristallographie et Physico-Chimie des matériaux, UER Sciences exactes et naturelles, Université de Clermont-Ferrand II, BP 45, 63170 Aubière, France*

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Abstract

The compound BaCaLu₂F₁₀ is isostructural with KY₃F₁₀. The unit cell is cubic with $a = 11.366$ (2) Å. The space group is $Fm\bar{3}m$, $D_m = 6.40$ (5), $D_x = 6.45$ Mg m⁻³, $Z = 8$. The

structure was solved by conventional methods; of the 617 reflections recorded using an automated four-circle diffractometer 163 which had positive peak intensities were used in the refinement of the model to an R value of 4.1%. There is no ordering between the Ca and Lu atoms, which are statistically distributed in sites 24(e).

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Introduction

In a recent paper we emphasized the connections between the structure and the luminescence properties of the compound $\text{BaCaLu}_2\text{F}_{10}$ doped with Eu^{2+} (Valon, Cousseins, Védrine, Gacon, Boulon & Fong, 1976). Powder diffraction patterns were identical to those of KY_3F_{10} , studied by Pierce & Hong (1973). Preliminary X-ray powder intensity calculations show that the atoms are placed as follows in the space group $Fm\bar{3}m$: Ba in 8(c), Ca and Lu randomly distributed in 24(e), F in 48(i) and 32(f). Moreover the Eu^{3+} ion introduced as a structural probe (Valon, Gacon, Védrine & Boulon, 1977) in $\text{BaCaLu}_2\text{F}_{10}$ may indicate the possible existence of ordering between the Ca^{2+} and Lu^{3+} ions. Consequently it seems necessary to examine the crystal structure of $\text{BaCaLu}_2\text{F}_{10}$.

Experimental

Single crystals were prepared by growth in the solid state from the fluorides BaF_2 , CaF_2 and LuF_3 heated in stoichiometric proportions, at 1173 K for 8 h in a sealed nickel tube. The crystals were colourless octahedra or tetrahedra. Their identity was confirmed by comparison with the X-ray powder diffraction data.

Systematic absences in the X-ray diffraction patterns, as observed on Weissenberg and precession photographs, indicated three possible space groups: $Fm\bar{3}m$, $F\bar{4}3m$ and $F432$.

The experimental density was measured by picnometry in *o*-xylene.

For the intensity measurements, a crystal in the shape of a tetrahedron with the (010), (111), ($\bar{1}\bar{1}\bar{1}$) and ($3\bar{2}\bar{1}$) planes as faces was selected. The volume of the crystal was $0.166 \times 10^{-3} \text{ mm}^3$.

Data were collected at room temperature using an automated four-circle diffractometer (CAD-4 Nonius). For the data collection reflected-beam graphite-monochromated radiation was used ($\lambda = 0.7107 \text{ \AA}$). Within a 2θ sphere of 120° , 617 reflections were recorded using an ω - 2θ scan method (scanning = $1^\circ + 0.3 \tan \theta^\circ$). Of the 617 reflections examined, 163 had positive peak intensities. The intensities of three standard reflections were checked hourly and the orientation was checked after every hundred reflections (standard reflections: 800, 080 and 008). The relative standard deviation was found to be 0.01.

Intensity data were corrected for Lorentz-polarization and absorption effects using de Meulenaer & Tompa's (1965) analytical method with the *AGNOST* program (linear absorption coefficient $\mu = 33.05 \text{ mm}^{-1}$).

Structure determination and refinement

As the possible space groups and the powder diffraction patterns are identical to those of KY_3F_{10} , we placed the Ba^{2+} ions in the 8(c) ($\frac{1}{4}, \frac{1}{4}, \frac{1}{4}; \frac{3}{4}, \frac{3}{4}, \frac{3}{4}$) sites of $Fm\bar{3}m$. Approximate locations of all Ca, Lu and F atoms were found from difference Fourier maps. After several cycles of least-squares refinement, using isotropic and then anisotropic thermal parameters, the agreement factor converged to $R = 4.1\%$.

Another possibility in the space group $F\bar{4}3m$ is: eight Ba^{2+} atoms on sites 4(c) and 4(d), 48 F atoms on 48(h) sites and two sets of 16 F atoms on 16(e) sites. The final value of R is

Table 1. Positional and thermal parameters

The positional parameters and the anisotropic thermal parameters are multiplied by 10^4 .

	Sites	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å ²)		
Ba	8(c)	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	1.6 (0.1)		
Ca, Lu	24(e)	2428 (2)	0	0	0.7		
F	32(f)	1130 (8)	1130 (8)	1130 (8)	0.9		
F	48(i)	$\frac{1}{2}$	1733 (13)	1733 (13)	2.2		
		β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ca, Lu	21 (1)	10 (0)	10 (0)	0	0	0	0
F(32f)	18 (4)	18 (4)	18 (4)	4 (4)	4 (4)	4 (4)	4 (4)
F(48i)	67 (15)	31 (7)	31 (7)	0	0	0	15 (9)

Table 2. Distances (Å) and angles (°)

Ba-F(32f)	2.700 (12) × 4	Ca, Lu-F(32f)	2.340 (5) × 4
Ba-F(48i)	3.097 (8) × 12	Ca, Lu-F(48i)	2.188 (7) × 4
F(48i)-F(48i)	2.790 (20)	F(32f)-F(32f)	2.570 (20)
	2.460 (40)	F(48i)-F(48i)	2.790 (20)
F(32f)-F(48i)	2.831 (9)		
F(48i)-F(32f)-F(48i)	58.9 (7)	F(48i)-F(32f)-F(48i)	59.0 (7)
	51.6 (8)	F(32f)-F(48i)-F(32f)	54.0 (5)

4%.* The *x* parameters at site 16(e) are 0.1086 and 0.8845, slightly different from the value of 0.1130 for the space group $Fm\bar{3}m$; the *z* parameter at 48(h) is 0.49 which is significantly different from the value of 0.50 at 48(i) in the space group $Fm\bar{3}m$. Because the isotropic thermal parameters of the F atoms at sites 16(e) did not converge during the refinement cycles, the space group $F\bar{4}3m$ was rejected. Moreover this assumption is consistent with the result of the non-linear optical test.

Assignment to the non-centrosymmetric space group $F432$ leads to atomic positions identical to those of $Fm\bar{3}m$ and we finally chose this latter group.

We must add that in order to detect local ordering at 24(e) sites, we recorded a section of reciprocal space with long exposure. From the Bragg reflection no modulation of the intensity was observed which is in good agreement with the assumption of total disorder between the Ca and Lu atoms.

Final atomic parameters are listed in Table 1 and significant interatomic distances and angles in Table 2.

The authors wish to express their gratitude to Professor J. C. Cousseins for his continued interest in this work.

* Lists of structure factors and of components and orientation of anisotropic thermal ellipsoids have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34334 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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