X-ray powder diffraction data for potassium erbium fluoride

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X-ray powder diffraction data, unit-cell parameters, and space group for potassium erbium fluoride, $K_{0.5}Er_{0.5}F_2$, are reported [$a = 5.6862(5)$ Å, unit-cell volume $V = 183.85$ Å^3, $Z = 4$, and space group Fm-3m]. Potassium erbium fluoride is isostructural with a large family of fluorides represented by the mineral fluorite CaF_2. All of the measured lines of the title compound were indexed and are consistent with the Fm-3m space group. No detectable impurities were observed. © *2013 International Centre for Diffraction Data*. [doi:10.1017/S0885715613000596]

Key words: X-ray powder diffraction, potassium erbium fluoride, $KErF_4$

I. INTRODUCTION

Potassium erbium fluoride ($K_{0.5}Er_{0.5}F_2$ or$KErF_4$) is an important substance having interesting luminescent properties in the vacuum ultraviolet (VUV) part of the spectral region (Gesland *et al.*, 1999), owing to the interesting properties of erbium connected especially with transition states of its f orbital. This can be used in many interesting luminescent applications. Several forms of mixed erbium–potassium fluorides exist as can be seen in the phase diagram of KF–ErF_3 (Aléonard *et al.*, 1973).

Ionic liquids are commonly used for preparation of various lanthanide fluorides (Zhang *et al.*, 2008; Bartůněk *et al.*, 2011, 2012, 2013). Ionic liquids are used as solvents persisting at higher temperatures and often as fluorination agents as well. In this work, cubic $K_{0.5}Er_{0.5}F_2$ was prepared by reaction of erbium nitrate with potassium hexafluorophosphate in ionic liquid 1-butyl-3-methylimidazolium chloride (bmim-Cl).

II. EXPERIMENTAL

A. Synthesis

Cubic $K_{0.5}Er_{0.5}F_2$ was prepared by the reaction of Er $(NO)_3.6H_2O$ with stoichiometric amount of KPF_6 in ionic liquid bmim-Cl acting as a solvent. Bmim-Cl was melted previously. The reaction was carried out in the sealed Teflon tube with bmim-Cl at 200 °C for 2 h. Subsequently, the reaction mixture was washed several times with methanol and water to remove the remaining bmim-Cl and other soluble products. The obtained white powder was dried in air and analyzed.

B. Specimen preparation

The sample was prepared by careful grinding in an agate mortar and front-loaded into a specimen holder.

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C. AAS analysis, diffraction data collection and reduction

AAS analysis was used to confirm the proposed stoichiometry $K_{0.5}Er_{0.5}F_2$. The atomic ratio of K:Er was obtained to be 1:1.10 with a cumulative error of measurements 6% supporting the well-proposed stoichiometry $K_{0.5}Er_{0.5}F_2$. The remaining small-crystal core consisting of erbium fluoride could explain the excess of erbium. The spectrometer Spectr AA880 was used for AAS analysis.

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO θ–θ powder diffractometer with parafocusing Bragg–Brentano geometry and Cu Kα radiation (Ni filter, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect X-ray powder diffraction (XRPD) data over the angular range from 5 to 90°2θ with a step size of 0.017°2θ and a counting time of 20.32 s per step.

The software package HighScore Plus V 3.0e (PANalytical, Almelo, the Netherlands) was used to smooth all of the data, to fit the background, to eliminate the $K\alpha_2$ component, and the top of the smoothed Cu Kα_1 ($\lambda = 1.5406$ Å) peaks were used to determine the peak positions and intensities of the diffraction peaks.

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 1. Automatic indexing results obtained by DICVOL04 (Boultif and Louër, 2004) show that the title compound is cubic with space group Fm-3m and the unit-cell parameters: $a = 5.6862(5)$ Å, unit-cell volume $V = 183.85$ Å^3, and $Z = 4$ (Table I). The figures of merits are – $F_{10} = 12.7$ (0.0262, 30) (Smith and Snyder, 1979) and $M_{10} = 46.0$ (de Wolff, 1968). All of the lines were indexed and are consistent with the Fm-3m space group.

Sodium, potassium, cerium, and erbium have got the following atomic radii – 2.23, 2.77, 2.70, and 2.45 Å respectively. There are two isostructural fluorides in PDF4+ (ICDD, 2012): sodium erbium fluoride ($Na_{0.5}Er_{0.5}F_2$, Card no. 04-013-7403) with $a = 5.465$ Å and potassium cerium fluoride ($K_{0.5}Ce_{0.5}F_2$, Card no. 04-007-9363) with $a = 5.906$ Å.

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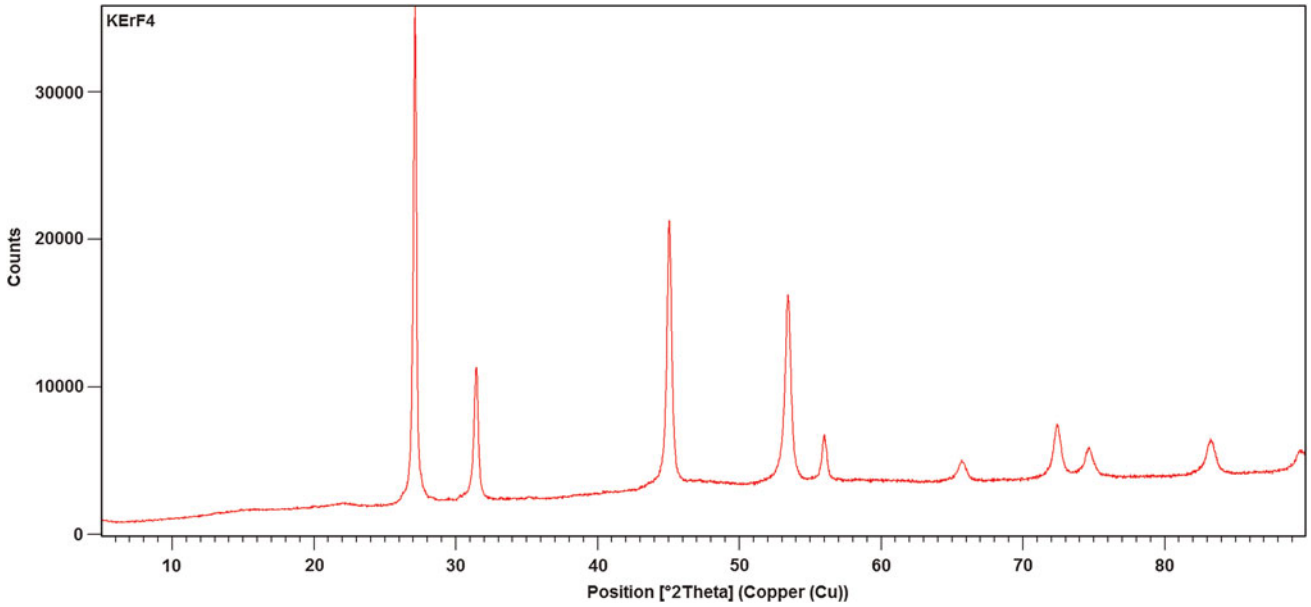


Figure 1. (Colour online) XRD pattern of $K_{0.5}Er_{0.5}F_2$.

TABLE I. Indexed X-ray powder diffraction data for the title compound, $K_{0.5}Er_{0.5}F_2$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 5.6862$ (5) Å, unit-cell volume $V = 183.85$ Å^3, and $Z = 4$, space group Fm-3m]. All of the lines were indexed and are consistent with the Fm-3m space group. The d-values were calculated using Cu Kα_1 radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$(°)
27.093	3.288	100	1	1	1	27.142	3.283	0.048
31.405	2.846	27	2	0	0	31.441	2.843	0.036
45.024	2.012	55	2	2	0	45.061	2.010	0.037
53.380	1.715	39	3	1	1	53.399	1.714	0.019
55.949	1.642	10	2	2	2	55.977	1.641	0.028
65.640	1.421	4	4	0	0	65.625	1.421	−0.015
72.361	1.305	11	3	3	1	72.387	1.304	0.026
74.589	1.271	6	4	2	0	74.581	1.271	−0.008
83.172	1.161	7	4	2	2	83.163	1.161	−0.009
89.513	1.094	4	3	3	3	89.488	1.094	−0.025

The present $K_{0.5}Er_{0.5}F_2$ has $a = 5.6862(5)$ Å, which with respect to the atomic radii is in good agreement with the unit-cell parameters of the two isostructural compounds above.

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