

The measured parameters and the two sigma errors in Å are

Sm ₂ O ₃		Eu ₂ O ₃	
a =	14.1767 ± 0.0057	a =	14.1088 ± 0.0058
b =	3.6262 ± 0.0014	b =	3.6020 ± 0.0013
c =	8.8502 ± 0.0033	c =	8.8051 ± 0.0027
β =	100.056 ± 0.034°	β =	100.063 ± 0.0031
Xaλρ =	7.758 ± 0.005 g cm ⁻³	Xaλρ =	7.958 ± 0.005 g cm ⁻³
Gd ₂ O ₃		irradiated Eu ₂ O ₃	
a =	14.0972 ± 0.0028	a =	14.1083 ± 0.0073
b =	3.5740 ± 0.0006	b =	3.6018 ± 0.0013
c =	8.7642 ± 0.0021	c =	8.7988 ± 0.0042
β =	100.041 ± 0.020	β =	100.042 ± 0.041
Xaλρ =	8.306 ± 0.003 g cm ⁻³		

Now monoclinic europia swells when it is irradiated under these conditions by ~ 5%. Since the crystal lattice parameters do not change it is clear that the swelling owes its origin entirely to vacancy clustering as already observed (Pearce J H, in UKAEA AERE Report 10116 pp 42-43).

The lattice parameters for the unirradiated oxides should be compared with other determinations for the samaria-gadolinia binary system (Ferguson I F, Acta Cryst. A31 (S.3) (1975) S.69).

It is also interesting to compare these results with those obtained for MoO₃-stabilised face centred cubic europia irradiated under the same conditions. There the lattice parameter expanded from 5.3704 ± 0.0008 to 5.3745 ± 0.0010 corresponding to an increase in length of 0.07% (Huyton A, in UKAEA AERE Report 10116 p 44).

08.2-13 THE AVERAGE STRUCTURE OF $\square_{0.5}K_{1.5}[Al_{1.5}Si_{0.5}O_4]$, AN ALKALI-DEFICIENT COMPOSITION BASED ON A FRAMEWORK WITH CRISTOBALITE TOPOLOGY. By M. Gregorkiewicz, Instituto de Físico-Química Mineral, CSIC, Madrid, Spain.

Clear octahedral crystals with composition close to $\square_{0.5}K_{1.5}[Al_{1.5}Si_{0.5}O_4]$ were grown at 850-1000°C from a mixture of the oxides in fused KF.

Powder diagrams including back-reflections and internal Si standard, as well as Weissenberg photographs (CuKα, spot and integrated reflections) for 11 crystals from 4 different syntheses led invariably to the cubic diffraction symmetry $m\bar{3}mFd$, corresponding to space group $Fd\bar{3}m$ with $a=765(2)$ pm and $Z=4$, the observed density being 2.603(5) g/cm³. A very small deviation from cubic symmetry ($\Delta a/a \approx 0.1\%$), as suggested from the optical behaviour and peak widths in 2θ with occasional splitting of back-reflections, occurs possibly within twin domains and was not resolved in the present study.

For three samples, about 350 X-ray intensities were collected on an automatic single crystal diffractometer, using MoKα radiation and up to 2θ=83°. After LP correction and data reduction, a total of 87 unique structure amplitudes was obtained, with internal consistency errors of $R_i=0.006(0.010,0.010)$ for the assumed symmetry and without appreciable differences for the three samples.

Symbolic addition gave the positional parameters for all atoms, with T=Al,Si at 8a, K statistically at 8b, and O near to 16c, plus a further peak at 32e with $x=0.41$, which accounts for the existence of 4n,4n,4n+2 reflections and has been interpreted as an additional site for potassium. During refinement, several constraints have been applied concerning the repartition of potassium and the representation of oxygen either by anisotropic thermal motion or precessing about the T-T line.

The final model ($R=0.048$ for 5 free parameters) contains a tetrahedral framework $[Al_{1.5}Si_{0.5}O_4]$ with cristobalite topology and random distribution of Al and Si. The oxygen atoms lie statistically on one of six sites at 41.2 pm off the T-T line, which corresponds to a tetrahedral rotation of $\phi=17.0^\circ$ and leads to a distance (Al,Si)-O = 170.7(2) pm and an angle T-O-T=152.0(5)°. Potassium ions occupy statistically 2/3 of the framework cavities, most of them at 32e, slightly off the cavity's center at 8b. This position is discussed in terms of electrostatic repulsion between potassium ions in neighbouring cavities, since it permits non-bonding distances K-K(trans)=364 pm and K-K(cis)=359 pm, which are considerably longer than for the ideal 8b site with K-K=331.3 pm. An ordered model based on all-trans configuration between neighbouring potassium pairs is proposed. It gives an interesting explanation for the observed stoichiometry and provides a possible twin mechanism which might account for a domain structure.

08.2-14 K- AND Rb-EXCHANGED NEPHELINE HYDRATE I. By Staffan Hansen and Lars Fälth, Inorganic Chemistry 2, Chemical Center, University of Lund, P.O.Box 740, S-220 07 Lund, Sweden

The crystal structures of the K⁺- and Rb⁺-exchanged forms of the microporous framework silicate nepheline hydrate I (NHI) were refined in space group $P\bar{1}m2_1$ to $R = 0.032$ and $R = 0.048$, respectively, using single crystal X-ray diffraction data. Lattice constants: $a = 8.113(3)$, $b = 15.223(2)$, $c = 5.1817(7)$ Å for KNHI, and $a = 8.0802(8)$, $b = 15.259(2)$, $c = 5.1584(5)$ Å for RbNHI (subcell). Compositions of $K_{1.1}Na_{1.9}Al_3Si_3O_{12} \cdot H_2O$ and $RbNa_2Al_3Si_3O_{12} \cdot H_2O$, $Z = 2$ were indicated by the least-squares refinements.

Both structures contained two Na⁺ ions which were but little affected by the ion-exchange process, a behaviour that is also exhibited by CsNHI (Hansen and Fälth, Z. Kristallogr. (1983), 164, 79). The two Na atoms coordinated by 7 framework O atoms were situated in a void built of 6-rings.

In the 8-ring channels the exchange was complete, K⁺ and Rb⁺ occupied similar sites in the vicinity of an elongated 8-ring. In KNHI the coordination sphere was completed by two symmetry-related water molecules, a situation which was probably paralleled in RbNHI.

The main difference between the two structures was that KNHI had a second cation in the 8-ring tunnel. This K⁺ ion provided the necessary charge compensation for the 10% vacancy in one of the Na sites.