As a result of the higher precision of the present analysis, the differences between the individual S—S distances appear to be statistically significant. However, correction for libration improves the agreement between individual S—S distances, the mean corrected value of 2·055 (2) Å and the mean S—S—S angle of 108·2 (6)° agreeing well with the best previous values (Donohue, 1982) of 2·060 (3) Å and 108·0 (7)°. As before, the diffraction data are fitted well by a rigid-body model (r.m.s. $\Delta U_{ij} = 0{\cdot}0010$ Å^2), but independent internal modes are likely to be present (r.m.s. $\sigma U_{ij} = 0{\cdot}0002$ Å^2). A detailed discussion of these points has been given by Pawley & Rinaldi (1972), which includes discussion of the marked deviation of the solid-state structure from the $\bar{8}2m$ (D_{4d}) symmetry of the free-state S_8 molecule.

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Trineodymium Ruthenate(V). A Rietveld Refinement of Neutron Powder Diffraction Data

BY W. A. GROEN, F. P. F. VAN BERKEL AND D. J. W. IJDO

Gorlaeus Laboratories, Leiden University, PO Box 9502, 2300 *RA Leiden, The Netherlands*

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Abstract. Nd_3RuO_7, $M_r = 575{\cdot}82$, orthorhombic, *Cmcm*, $a = 10{\cdot}9042$ (6), $b = 7{\cdot}3827$ (5), $c = 7{\cdot}4963$ (4) Å, $V = 603{\cdot}47$ (9) Å^3, $Z = 4$, $D_x = 3{\cdot}82$ Mg m^{-3}, $\mu R = 0{\cdot}56$. The structure has been refined by Rietveld analysis of neutron powder diffraction data recorded at room temperature [$\lambda = 2{\cdot}5804$ (1) Å, $R_I = 4{\cdot}88$, $R_p = 8{\cdot}83$, $R_{wp} = 9{\cdot}09\%$]. The compound adopts an orthorhombic fluorite-related structure of the La_3NbO_7 type. One third of the Nd ions are eight-coordinated and lie in rows in the [001] direction which alternate with parallel rows of corner-linked RuO_6 octahedra. The remaining two thirds of the Nd ions are seven-coordinated and lie between the slabs of the NdO_8 and RuO_6 polyhedra.

Introduction. In a programme to investigate ceramic materials for their use as potential host lattices for the disposal of nuclear waste (Ringwood, 1978), we studied the equilibrium at 1573 K in the system BaO–RuO_2–Nd_2O_3.

In this system $Nd_2Ru_2O_7$ with the pyrochlore structure is known to exist (Bertaut, Forratana & Montmory 1959). Dixon, Marr, Lachowski, Gard & Glasser (1980) reported the presence of the orthorhombic phase Nd_2RuO_5 also. We were unable to prepare this compound but found a new orthorhombic phase with a higher neodymium content: Nd_3RuO_7. van Berkel & IJdo (1986) describe this compound with space group *Cmcm* as a superstructure of the cubic fluorite structure with $a_{\rm orth} \simeq 2a_c$, $b_{\rm orth} \simeq c_{\rm orth} \simeq \sqrt{2}a_c$ and suggest the same structure as that found for La_3NbO_7, determined from X-ray powder diffraction (Rossell, 1979).

A very precise structure determination is necessary for an interpretation of the magnetic properties of Nd_3RuO_7.

Experimental. The system was prepared from 99·9% Nd_2O_3, ignited at 1273 K; RuO_2 was prepared from fine metal (99·99%) at 1073 K in air. The mixture was heated in an alumina crucible in a vacuum-sealed quartz tube according to:

$$Nd_2Ru_2O_7 + 2Nd_2O_3 + \tfrac{1}{2}O_2 \rightarrow 2Nd_3RuO_7.$$

The oxygen was added as $KClO_3$ in a separate crucible. X-ray powder diffraction patterns were obtained with a Philips PW 1050 diffractometer.

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The systematic absences indicate space group *Cmcm*, *Cmc*2_1 or *C*2*cm*. Electron diffraction patterns (Siemens Elmiscop 102, 100 kV) confirm this result.

Since no single crystals were available, Rietveld's (1969) method was used for refinement of neutron powder diffraction data on the powder diffractometer at the Petten High-Flux Reactor; $5 < 2\theta < 163°$ in steps of 0·1°; neutrons at 295 K from the (111) planes of a Cu crystal; pyrolytic graphite with a total thickness of 120 mm as a second-order filter; Soller slits, horizontal divergence 0·5°, placed between the reactor and the monochromator and in front of the four ^{3}He counters; sample holder ($\varnothing = 14·46$ mm) consisted of a V tube, closed with Cu plugs with O rings. No precautions to avoid preferred orientation were taken. Maximum absorption correction 5%, $\mu R = 0·56$ (Weber, 1967). The background was determined from parts in the diagram which did not contain any contribution from reflections, and interpolation between these points. Statistically expected value of R_p 5·66%. The proposed structure of Nd_3NbO_7 (Rossell, 1979) was used as a trial model: Ru in 4(*b*) $(0,\frac{1}{2},0)$, Nd1 in 4(*a*) (0,0,0), Nd2 in 8(*g*) $(x,y,\frac{1}{4})$, O1 in 16(*h*) (x,y,z), O2 in 8(*g*) $(x,y,\frac{1}{4})$ and O3 in 4(*c*) $(0,y,\frac{1}{4})$.

The coherent scattering lengths used were: Nd 7·69, Ru 7·21 and O 5·81 fm (Koester, Rauch, Herkens & Schroeder, 1981). 23 parameters in the refinement: a scale factor, three halfwidth parameters defining the Gaussian line shape of the reflections, the counter zero error, an asymmetry parameter, the unit-cell parameters, the atomic position parameters and the isotropic thermal parameters.

The Rietveld program minimizes the function $\chi^2 = \sum_i w_i[y_i(\text{obs.}) - (1/c)y_i(\text{calc.})]^2$, where $y_i(\text{obs.})$ and $y_i(\text{calc.})$ are the observed and calculated data points, w_i is the statistical weight $[1/y_i(\text{obs.})]$ allotted to each data point and c is the scale factor. The following R factors were calculated: $R_I = 100\sum|I_i(\text{obs.}) - (1/c)I_i(\text{calc.})|/\sum I_i(\text{obs.})$, $R_p = 100\sum|y_i(\text{obs.}) - (1/c)y_i(\text{calc.})|/\sum y_i(\text{obs.})$, $R_{wp} = 100[\sum w_i|y_i(\text{obs.}) - (1/c)y_i(\text{calc.})|^2/\sum w_i|I_i(\text{obs.})|^2]^{1/2}$, where $I_i(\text{obs.})$ and $I_i(\text{calc.})$ are the observed and calculated integrated intensities of each reflection. $\Delta/\sigma < 0·3$ in final cycle. Largest correlation matrix element for structural parameters 0·48.

Discussion. Atomic parameters are given in Table 1 and selected atomic distances and angles are shown in Table 2. The agreement between the observed and the calculated profile at $T = 293$ K is shown in Fig. 1.* Fig. 2 shows the structure of Nd_3RuO_7.

The structure is basically the same as that of La_3NbO_7 (Rossell, 1979). One third of the Nd ions are eight-coordinated and lie in rows in the [001] direction which alternate with parallel rows of corner-linked RuO_6 octahedra as can be seen in Fig. 2. The remaining two thirds of the Nd ions are seven-coordinated and lie between the slabs of the NdO_8 and the RuO_6 polyhedra. The RuO_6 octahedra are corner-linked through the O3 atoms. Successive O3 atoms along a [001] row lie in the zigzag sequence $(0,y,\frac{1}{4})$, $(0,-y,\frac{3}{4})$....., with $y = 0·4168$, which gives rise to a corresponding sequence of tilts to the octahedra.

The eight-coordinated Nd1 atoms lie in very distorted cubes of four O2 atoms and four O1 atoms as can

Table 1. *Fractional atomic coordinates and isotropic thermal parameters of* Nd_3RuO_7 *at* 295 K

	x	*y*	*z*	*B*(Å^2)
Nd1	0	0	0	1·1 (1)
Nd2	0·2226 (3)	0·3093 (4)	0·25	0·9 (1)
Ru	0	0·5	0	1·3 (1)
O1	0·1257 (3)	0·3169 (4)	−0·0412 (4)	1·3 (1)
O2	0·1309 (4)	0·0278 (6)	0·25	0·9 (1)
O3	0	0·4168 (7)	0·25	0·9 (1)

Table 2. *Atomic distances* (Å) *and angles* (°) *at room temperature*

NdO_8 polyhedron		NdO_7 polyhedron	
Nd1–O1	2·729 (3) 4×	Nd2–O1	2·461 (4) 2×
Nd1–O2	2·365 (3) 4×	Nd2–O1	2·426 (3) 2×
		Nd2–O2	2·306 (5) 1×
RuO_6 octahedron		Nd2–O2	2·270 (5) 1×
Ru–O1	1·950 (3) 4×	Nd2–O3	2·554 (4) 1×
Ru–O3	1·972 (2) 2×	Nd2–O3	4·189 (5) 1×
Ru–O3–Ru	143·7 (3)		
O1–Ru–O3	86·3 (1)		
O1–Ru–O1	89·34 (9)		

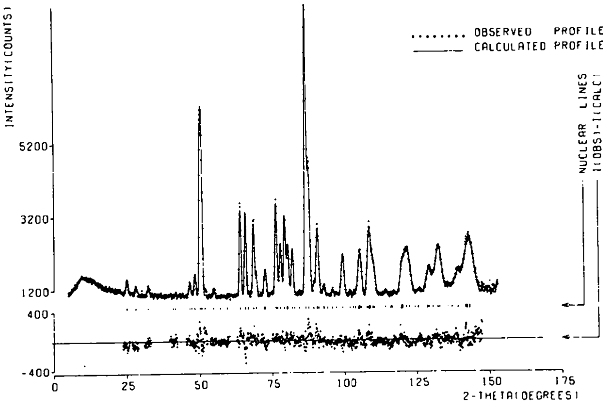

Fig. 1. Observed (dots) and calculated (full line) neutron diffraction profile of Nd_3RuO_7 at room temperature; a difference (observed − calculated) curve appears at the bottom of the figure. Thick marks below the profile indicate the positions of the Bragg reflections included in the calculation.

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* A list of numerical values corresponding to the data in Fig. 1, structure factors and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44270 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

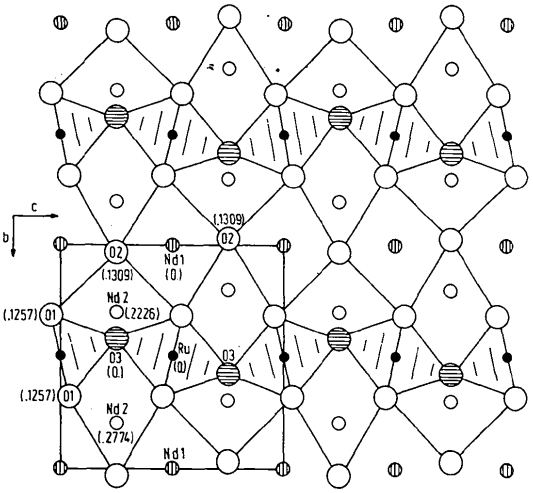

Fig. 2. Part of the structure between $x = 0$ and $x = 0{\cdot}25$ of Nd_3RuO_7 projected along the *a* axis. Fractional coordinates of the atoms along the *a* axis are given.

be seen in Fig. 2 and from the Nd1—O1 and Nd1—O2 distances in Table 2.

The Nd2 atoms are surrounded by seven O atoms; four Nd2—O1, two Nd2—O2 and one Nd2—O3 distance. One O3 atom lies too far away for eight-coordination, because of the tilting of the RuO_6 octahedra.

This Ln_3RuO_7 system exists only for Ln = Nd, Sm and Eu (van Berkel & IJdo, 1986).

The magnetic behaviour of this compound is under investigation.

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Synthèse et Structure du Diborouranate de Magnésium, MgB_2UO_7

PAR MADELEINE GASPERIN

Laboratoire de Minéralogie–Cristallographie associé au CNRS, Université P. et M. Curie, 4 place Jussieu, 75252 Paris CEDEX 05, France

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Abstract. $M_r = 396{\cdot}03$, orthorhombic, *Pcam*, $a = 9{\cdot}747\,(3)$, $b = 7{\cdot}315\,(2)$, $c = 7{\cdot}911\,(2)$ Å, $V = 564{\cdot}05$ Å^3, $Z = 4$, $D_x = 4{\cdot}7$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 27{\cdot}4$ mm^{-1}, $F(000) = 680$, $T = 290$ K; 1997 contributing reflections, $R = 0{\cdot}034$, $wR = 0{\cdot}043$. All the atoms, except O(3), are located in sheets perpendicular to [00*z*]. U^{6+} and Mg^{2+} are surrounded by octahedra, with, for U^{6+}, a uranyl bond [O(3)—U—O(3) = 1·80 Å] along the *c* axis. B atoms are in triangular B_2O_5 groups. In the [00*z*] direction, the UO_6 and MgO_6 polyhedra are joined by corners and form Mg—O—U chains. U—O distances vary between 1·79 and 2·35 Å, Mg—O between 2·0 and 2·17 Å and B—O between 1·35 and 1·43 Å.

Introduction. Ce travail s'insère dans l'étude des borouranates d'alcalino terreux et fait suite à la description de $CaB_2U_2O_{10}$ publiée récemment (Gasperin, 1987). En effet, si plusieurs phases oxydées contenant du bore, de l'uranium et un alcalin ont été signalées (Hoekstra, 1967), aucun composé de ce type n'avait jusqu'ici été mis en évidence avec un ion bivalent.

Partie expérimentale. Chauffage à l'air à 1473 K pendant 15 heures de U_3O_8, $MgCO_3$, B_2O_3 avec 1U+4Mg+18B. Après lavage à l'eau bouillante, plaquettes transparentes incolores.

Symétrie orthorhombique *Pcam* ou *Pca*2_1 mise en évidence sur film. Plaquette (001) allongée selon [100]. Dimension maximale: 140 µm; dimension minimale: 30 µm. Diffractomètre Philips PW 1100, monochromateur en graphite, méthode d'intégration $\omega/2\theta$ avec $0{,}02°\,\theta\,s^{-1}$. Paramètres affinés par moindres carrés à