and only traces of this compound are identifiable.

We conclude that one of the spontaneous decomposition reactions of 2-acetolactic acid, produces, also in fermentation solutions, diacetyl together with formic acid.

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Received October 19, 1970.

On the Crystal Structure of Nd$_4$Re$_2$O$_{11}$

KARL-AXEL WILHELMI and
EVA LAGERVALL

Institute of Inorganic and Physical Chemistry, University of Stockholm, Box 6801, S-113 86 Stockholm, Sweden

and

OLAF MULLER

Materials Research Laboratory, The Pennsylvania State University, University Park, Pennsylvania 16802, USA

Several rare earth-rhenium oxides have been described.[1-4] Only one of these compounds, the complex oxide La$_4$Re$_6$O$_{19}$, has been characterized by a crystal structure determination.[5-6]

One of the present authors[2,3] recently made a study comprising several compounds near the Ln_3ReO$_8$ stoichiometry (Ln=rare earth metal, Nd, Sm, Gd, Dy, Er, Yb, Y). Well developed crystals of an Nd compound were prepared by using a starting mixture of Ln_2O$_3$ and ReO$_3$ with ~20 % excess ReO$_3$ over the stoichiometric requirement. The mixtures were heated for several days at 1100—1300°C in closed Pt-10Rh capsules. The analysis of several samples indicated that the compound obtained presumably is richer in oxygen than Nd$_3$ReO$_8$ and the formula β-Nd$_3$ReO$_{8+x}$ ($x \approx 0.3$) was suggested. To establish the composition unambiguously a structure determination of the product has now been performed. This article is a preliminary report of this work.

The unit cell dimensions of the tetragonal cell, calculated from a powder pattern, obtained with a Hägg-Guinier camera, are in excellent agreement with those reported earlier.[2,3]

$$a=12.676(\pm2) \text{ Å}, \quad c=5.601(\pm1) \text{ Å},$$
$$V=899.9 \text{ Å}^3$$

The previously observed density, 8.14 g cm^{-3}, indicated a cell content of eight formula units Nd$_3$ReO$_8$ (calc. 8.18 g cm^{-3}). Rotation and Weissenberg photographs[2,3] of a single crystal along the a and c axes had revealed the Laue symmetry 4/m and shown the following extinctions: $h+k=2n+1$ for the $hk0$ reflections and $l=2n+1$ for $00l$ reflections. These observations were confirmed

Table 1. Coordinates, isotropic temperature factors and standard deviations for $Nd_4Re_2O_{11}$.
Space group $P4_2/n$ (No. 86).

$2(a) \quad \pm(\frac{1}{4},\frac{1}{4},\frac{1}{4})$
$2(b) \quad \pm(\frac{1}{4},\frac{1}{4},\frac{3}{4})$
$8(g) \quad \pm(x,y,z); \quad \pm(\frac{1}{2}-x,\frac{1}{2}-y,z); \quad \pm(\bar{y},\frac{1}{2}+x,\frac{1}{4}+z); \quad \pm(\frac{1}{2}+y,\bar{x},\frac{1}{4}+z)$

	x		y		z		B Å^2	
Nd(1) in 8(g)	0.1840	(1)	0.1182	(1)	0.9979	(2)	0.44	(2)
Nd(2) in 8(g)	0.1107	(1)	0.8064	(1)	0.7030	(2)	0.53	(2)
Re(1) in 8(g)	0.0247	(1)	0.9130	(1)	0.5707	(1)	0.35	(2)
O(1) in 8(g)	0.0301	(10)	0.1938	(9)	0.2009	(21)	0.41	(18)
O(2) in 8(g)	0.0013	(9)	0.4086	(9)	0.1937	(22)	0.32	(18)
O(3) in 8(g)	0.0415	(10)	0.6291	(9)	0.2905	(22)	0.45	(18)
O(4) in 8(g)	0.1484	(10)	0.8032	(11)	0.4943	(26)	1.03	(22)
O(5) in 8(g)	0.4559	(10)	0.3400	(10)	0.6868	(24)	0.75	(19)
O(6) in 2(a)	1/4		1/4		1/4		0.25	(36)
O(7) in 2(b)	1/4		1/4		3/4		0.92	(43)

in the present study. This establishes the space group as $P4_2/n$ (No. 86).

Two sets of three-dimensional X-ray data were collected, *viz.* one set with film technique and Cu$K\alpha$ radiation, and one set with a Siemens automatic diffractometer with Nb-filtered Mo$K\alpha$ radiation and scintillation counter with pulse height discrimination. The data sets were corrected for Lorentz and polarization effects as well as for absorption.

From the film data, the sixteen neodymium atoms and eight rhenium atoms were located from a three-dimensional Patterson synthesis in

$8(g)$: $\pm(x,y,z)$; $\pm(\frac{1}{2}-x,\frac{1}{2}-y,z)$;
$\pm(\bar{y},\frac{1}{2}+x,\frac{1}{4}+z)$; $\pm(\frac{1}{2}+y,\bar{x},\frac{1}{4}+z)$

(alternative origin at $\bar{1}$ at $\frac{1}{4},\frac{1}{4},\frac{1}{4}$ from $\bar{4}$).

The metal atom positions were refined using a full-matrix least-squares program. The diffractometer data (738 independent reflections) were now introduced in the calculations and tentative oxygen positions (in $5 \times 8(g)$, $2(a)$ and $2(b)$) were found from a three-dimensional $(F_o - F_{metal})$ synthesis. The positions of all atoms were then refined with a program, allowing for secondary extinction correction. Corrections were made for the real and imaginary anomalous dispersion for rhenium and neodymium. At the present stage of refinement the R factor is 2.6 %. The positional and isotropic thermal parameters together with their standard deviations are shown in Table 1. The result shows that the formula of the compound is $Nd_4Re_2O_{11}$.

The structure can be visualized as composed of ReO_6 octahedra occurring in edge-shared pairs, the octahedra of these clusters being related by an inversion axis;
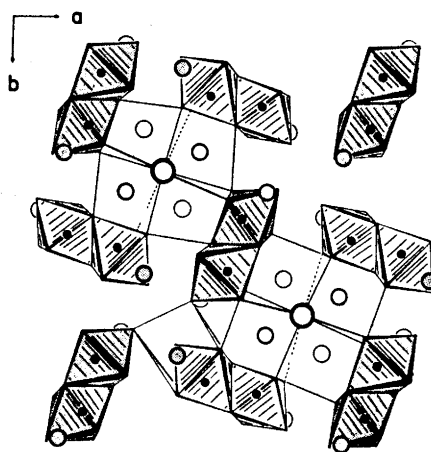

Fig. 1. The crystal structure of $Nd_4Re_2O_{11}$, projected on (001), visualized by the coupling of ReO_6 octahedra forming isolated Re_2O_{10} clusters. The medium-sized open circles represent the Nd(1) atoms. The Nd(2) atoms are hatched. The eight coordination around the Nd(2) atoms is indicated only as a trigonal prism surrounding one of the Nd(2) atoms and extending above the set of atoms included in the figure. Two concentric circles indicate 2 oxygen atoms at $z=\frac{1}{4}$ and $z=\frac{3}{4}$.

see Fig. 1. The Re_2O_{10} clusters are joined together by Nd atoms in two non-equivalent positions to form a three-dimensional framework. The Nd(1) forms ONd_4 tetrahedra which are joined together by sharing edges along c forming strings with the

Table 2. Interatomic distances (in Å) in $Nd_4Re_2O_{11}$. Standard deviations within parentheses.

Rhenium-oxygen distances, < 3.1 Å

Re(1)—O(3)	1.894 (12)
—O(5)	1.918 (13)
—O(2)	1.951 (11)
—O(1)	1.984 (12)
—O(2)	2.035 (12)
—O(4)	2.138 (14)

Neodymium-oxygen distances, < 3.3 Å

Nd(1)—O(6)	2.323 (1)
—O(7)	2.337 (1)
—O(5)	2.428 (13)
—O(1)	2.437 (12)
—O(3)	2.443 (12)
—O(1)	2.528 (12)
—O(2)	2.539 (12)
—O(5)	2.552 (13)
Nd(2)—O(4)	2.321 (14)
—O(1)	2.349 (12)
—O(4)	2.400 (14)
—O(5)	2.413 (13)
—O(3)	2.500 (12)
—O(4)	2.541 (14)
—O(3)	2.698 (12)
—O(2)	2.801 (12)
—O(4)	3.266 (14)

composition $\frac{1}{\infty}(ONd_2)$. The environment of the oxygen atoms around Nd(1) is eightfold. The arrangement of neodymium and oxygen can be described as a somewhat deformed fluorite arrangement similar to the one present in rhombohedral NdOF.[7] Nd(2) is surrounded by eight nearest oxygen atoms at distances of 2.321 — 2.801 Å with an additional very long Nd(2)—O distance at 3.266 Å. Six of the eight nearest oxygen atoms form a trigonal prism, indicated in Fig. 1.

The rhenium atoms in the clusters are only 2.421 ± 1 Å apart, which is exactly the same value as the corresponding distance observed in $La_4Re_6O_{19}$.[5,6] This is appreciably closer than the distance observed [8] in rhenium metal (2.741 and 2.760 Å) and is intermediate between the very short distance found in the $Re_2Cl_8^{2-}$ ion,[9] 2.24 Å, and the average Re—Re distance of 2.48 Å

found in Re_3Cl_9 groups (see Ref. 9). The formal oxidation state of rhenium in $Nd_4Re_2O_{11}$ is +5, *i.e.* higher than the corresponding value for $La_4Re_6O_{19}$. The latter structure is made up of pairs of ReO_6 octahedra formed by edge-sharing with a short Re—Re distance of 2.42 Å. These clusters are again joined by corner sharing to form a three-dimensional network $\frac{3}{\infty}(Re_2O_6)$. Within the voids there are La_4O tetrahedra. The coordination around lanthanium is tenfold and can be described as a trigonal prism which is capped on all three rectangular faces and one triangular face. The oxide can be formulated as $La_4O \cdot (Re_2O_6)_3$. A similar formulation can be used for the $Nd_4Re_2O_{11}$, *viz.* $Nd_2(ONd_2)Re_2O_{10}$.

$Nd_4Re_2O_{11}$ is not the only compound with the structure described above, as isotypic Sm and Gd analogues are also known. In a preliminary study,[3] these compounds had been tentatively labeled as β-$Nd_2ReO_{5.25}$, β-$Sm_2ReO_{5.25}$ and β-$Gd_2ReO_{5.75}$. In view of the present study, they should be re-formulated as $Nd_4Re_2O_{11}$, $Sm_4Re_2O_{11}$ and $Gd_4Re_2O_{11}$.

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