hedra are in the range $2.31-2.40$ Å (mean 2.36 Å). The value of $\theta$ (angle between the erbium-oxygen bonds and the pseudo eightfold rotation inversion axis) is in the range $53.7-59.7°$ (mean $56.7°$), in agreement with that found in erbium glycolate (Table 7 in Ref. 8). The distances along the edges of the square planes are in the range $2.78-2.80$ Å, and the distance between these planes 2.59 Å. They are almost parallel with the $a$-axis and tilted $0.5°$ to each other.

The distances and angles within the ligand molecules are not far from those found in the other lanthanoid oxydiacetate compounds. The two halves of the ligand containing $O(1)-O(3)$ and $C(1)$, $C(2)$ are planar and the angle between them is $5°$. In the other ligand the half containing $O(7)$ and $O(8)$ is planar while the other is more twisted (dihedral angles $O(6)-C(5)-C(6)-O(8)$: $-179°$, $O(6)-C(4)-C(3)-O(5)$: $-151°$). Apparently, $O(5)$ has refined to a somewhat erroneous location.

The $OW(3)$ water oxygen atom is not coordinated to erbium. It is within hydrogen bond distance from $O(5)$ (2.74 Å), $O(4)$ (2.80 Å), and $OW(1)$ (2.74 Å).

As this structure contains both eight- and nine-coordinated erbium ions, erbium seems to have a preference for eight-coordination when it is not forced by the ligands to adopt nine-coordination as in the tris(oxydiacetato)-erbate complex.

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# Preparation and Structure of Stoichiometric $\delta$-NbN

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The cubic phase of niobium nitride, $\delta$-NbN, can be obtained in a reaction between niobium and nitrogen. A rod pressed isostatically from niobium powder was heated by induction in pure nitrogen gas at 2.0 MPa in an ADL MP crystal growth furnace. For experimental details see Ref. 1. The sample was 15 cm long and 10 mm in diameter. When the sample was red-hot, a violent and fast exothermic reaction between niobium and nitrogen started. The sample became incandescent and measurements with a disappearing filament pyrometer indicated that the temperature was at least 1800 °C. After approximately 20 s, the whole volume of the sample had reacted with nitrogen and the temperature of the specimen dropped to the level determined by the power of the induction heater.

A Guinier powder pattern obtained with a Guinier de Wolff camera with $CuK\alpha_1$ radiation ($\lambda = 1.54051$ Å) and germanium ($a_{Ge} = 5.6576$ Å) as an internal standard showed that a sample treated as described above was cubic $\delta$-NbN with $a = 4.394(3)$ Å. It was recently reported,[1] that zone melting of a specimen of $\delta$-NbN at pressures up to 2 MPa resulted in a sample containing $\gamma$-NbN with the composition $NbN_{0.84(1)}$, and $\beta$-$Nb_2N$ with the composition $NbN_{0.46(1)}$. A determination of the composition of $\delta$-NbN is reported below.

A neutron diffraction powder pattern of $\delta$-NbN was measured with a double axis neutron spectrometer at the DR3 reactor at Risø. The pattern was obtained with an incident neutron wave length of 0.998 Å, reflected from a (002) plane of a Be crystal. The collimations in front of and behind the sample were defined by Soller slits to be 21 and 30', respectively. The pattern was obtained for scattering angles ($2\theta$) from 20 to 102° in steps of 0.2° and could be indexed with a cubic unit cell with $a = 4.39$ Å.

The structure of the compound was refined using the Rietveld refinement programs [2,3] for powder profile intensities. The scattering lengths [4] (in $10^{-12}$ cm) were: $b_{Nb} = 0.711$, and $b_N = 0.940$.

The intensities of the diffraction pattern had contributions from 18 reflections. Two of these, (400) and (440), had an additional scattering contribution from an unidentified impurity.

$\delta$-NbN is isostructural with NaCl, space group $Fm3m$, No. 225, with niobium in site $4a$, and nitrogen in site $4b$. The variable parameters used in the refinements are: Two isotropic temperature factors, an occupancy factor for

*Table 1.* Results from neutron diffraction on a sample of $\delta$-NbN.

|  | Site | Occupancy | $B$ (Å²) |
|---|---|---|---|
| **18 reflections; $R = 9.4$ %** | | | |
| Nb | 4a | 1.0 | 0.6(2) |
| N | 4b | 1.00(4) | 0.3(1) |
| **16 reflections; $R = 3.7$ %** | | | |
| Nb | 4a | 1.0 | 0.77(9) |
| N | 4b | 0.98(2) | 0.26(5) |

*Table 2.* Observed and calculated intensities for $\delta$-NbN. Reading from left to right, the columns are: $h\ k\ l\ I_{obs}\ I_{calc}$.

| h | k | l | $I_{obs}$ | $I_{calc}$ | h | k | l | $I_{obs}$ | $I_{calc}$ |
|---|---|---|---|---|---|---|---|---|---|
| 1 | 1 | 1 | 6594 | 5971 | 5 | 1 | 1 | 8665 | 3144 |
| 2 | 0 | 0 | 163434 | 162989 | 3 | 3 | 3 | 2888 | 1048 |
| 2 | 2 | 0 | 157627 | 160406 | 4 | 4 | 0 | 65090 | 38216 |
| 3 | 1 | 1 | 5464 | 5734 | 5 | 3 | 1 | 2120 | 2811 |
| 2 | 2 | 2 | 67621 | 70289 | 6 | 0 | 0 | 15914 | 17045 |
| 4 | 0 | 0 | 71704 | 39061 | 4 | 4 | 2 | 63658 | 68178 |
| 3 | 3 | 1 | 9083 | 3860 | 6 | 2 | 0 | 60591 | 61886 |
| 4 | 2 | 0 | 120513 | 123773 | 5 | 3 | 3 | 2693 | 2674 |
| 4 | 2 | 2 | 102576 | 102404 | 6 | 2 | 2 | 60143 | 57092 |

nitrogen, a scale factor, three half-width parameters, a zero point parameter, and a unit cell parameter, giving a total of nine parameters. The results of the refinement including the 18 reflections are listed in Table 1. When the two reflections (400) and (440) were omitted from the refinements, the $R$-factor dropped from 9.4 to 3.7 % (see Table 1). The values of the occupancy factor and the two isotropic temperature factors obtained in the refinement with 16 reflections are not significantly different from the values obtained in the refinement with the 18 reflections. In Table 2 are listed the observed and calculated intensities from the refinement with the 18 reflections.

The investigation shows that the composition of the sample is $NbN_{0.98(2)}$, or that the sample is stoichiometric within experimental errors. It is interesting to note that the cubic phase, $\delta$-NbN, with a stoichiometric composition thus can be made by direct reaction of a rod made from niobium powder and pure nitrogen at a pressure of 2 MPa. The temperature at the synthesis is mainly determined by the heat of reaction for the exothermic process and the preparation is completed in a period of time less than 1 min.

When a rod of stoichiometric $\delta$-NbN was zone melted in an ambient nitrogen gas pressure of 2 MPa, it was observed that the molten zone expanded or boiled in the moment it was formed. This is because nitrogen gas is released from the melt. The composition of the zone melted material is between $NbN_{0.5}$ and $NbN_{0.6}$,[1] when the material is zone melted at an ambient nitrogen gas pressure of 2 MPa. In order to prepare crystals of $\delta$-NbN by zone melting it is most likely necessary to increase the ambient nitrogen gas pressure to 10 or 20 MPa.

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