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Li_3NbO_4

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Abstract

A single crystal of trilithium niobate, Li_3NbO_4, was obtained for the first time by extraction from a LiCl flux. The structure comprises Nb_4O_{16} clusters, which form a body-centred cubic lattice, with Li ions located among the Nb_4O_{16} clusters. O ions coordinate to Nb and Li in an octahedral manner.

Comment

The structure of Li_3NbO_4 has been reported previously (Blasse, 1963). The structure determination, however, was carried out with a powder sample and the *R* factor was fairly large. Since a single crystal has been successfully obtained (Suzuki, Shishido, Ukei & Fukuda, 1993), the present study was carried out.

After submission of this manuscript, the authors were informed of a structural study of Li_3NbO_4 by neutron diffraction (Grenier & Bassi, 1965). The positional parameters from the earlier study are similar to those reported here, but the space group determined previously ($I23$) is incorrect.

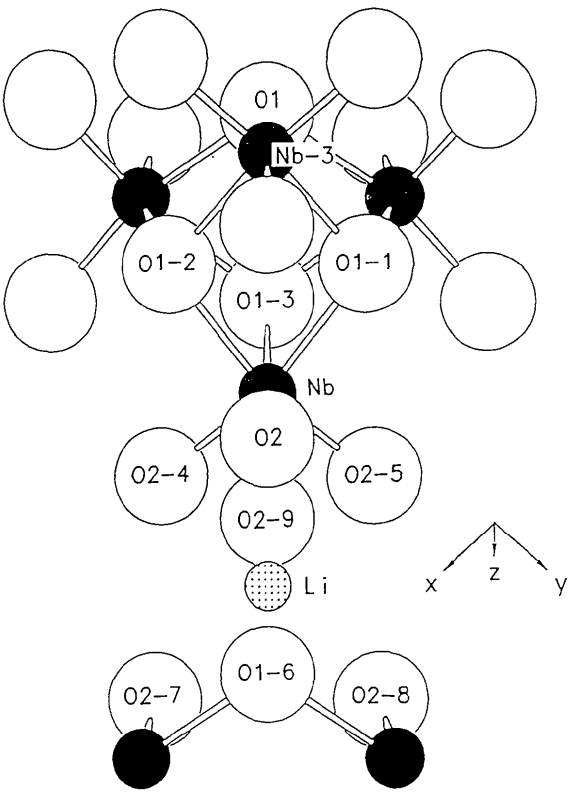

Fig. 1. A view of the Nb_4O_{16} cluster and a Li ion with coordinating O ions. Filled, dotted and open circles represent Nb, Li and O ions, respectively. Atomic nomenclature is abbreviated: for example, O1-2 represents O(1^{II}).

Experimental

Crystal data

Li_3NbO_4
M_r = 177.73
Cubic
$I\bar{4}3m$
a = 8.412 (2) Å
V = 595.2 (4) Å^3

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 19 reflections
θ = 30.4–31.3°
μ = 3.69 mm^{-1}

$Z = 8$
$D_x = 3.967$ Mg m^{-3}
Room temperature
Cube
0.25 × 0.22 × 0.22 mm
Colourless

Data collection

Rigaku AFC-6*A* diffractometer
2θ–ω scans
Absorption correction: by integration from crystal shape
$T_{min} = 0.453$, $T_{max} = 0.521$
4608 measured reflections
134 independent observed reflections

134 observed reflections [$|F_o| \geq 3\sigma(|F_o|)$]
$R_{int} = 0.019$
$\theta_{max} = 32.5°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 100 reflections
intensity variation: ≤1.4%

Refinement

Refinement on F
$R = 0.031$
$wR = 0.045$
$S = 9.85$
134 reflections
17 parameters
$w = 1/\sigma^2(|F_o|)$
$(\Delta/\sigma)_{max} = 0.44$
$\Delta\rho_{max} = 1.3$ e Å^{-3}
$\Delta\rho_{min} = -1.2$ e Å^{-3}

Extinction correction: Zachariasen (1968)
Extinction coefficient: 1.86 (4) × 10^{-4}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) and Tokonami (1965) for O atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	Wyckoff position	x	y	z	U_{eq}
Nb	8(*c*)	0.14028 (3)	x	x	0.0074
O(1)	8(*c*)	−0.1090 (4)	x	x	0.0084
O(2)	24(*g*)	0.1247 (3)	x	0.3601 (3)	0.0112
Li	24(*g*)	0.3782 (5)	x	0.1046 (6)	0.017 (1)

Table 2. *Selected geometric parameters* (Å, °)

Nb—Nb^{iii}	3.3381 (7)	Nb—Li	2.846 (4)
Nb—O(1^{i})	2.130 (4)	Nb—O(1^{ii})	2.130 (4)
Nb—O(1^{iii})	2.130 (4)	Nb—O(2)	1.858 (3)
Nb—O(2^{iv})	1.858 (3)	Nb—O(2^{v})	1.858 (3)
Li—O(2^{iv})	2.145 (5)	Li—O(2^{v})	2.145 (5)
Li—O(2^{ix})	2.057 (6)	Li—O(1^{vi})	2.414 (7)
Li—O(2^{vii})	2.095 (5)	Li—O(2^{viii})	2.095 (5)
O(1)—O(1^{iii})	2.593 (5)		
O(1^{i})—Nb—O(1^{ii})	75.0 (1)	O(1^{i})—Nb—O(2)	92.6 (1)
O(1^{i})—Nb—O(2^{iv})	164.2 (1)	O(1^{i})—Nb—O(2^{v})	92.6 (1)
O(2)—Nb—O(2^{iv})	97.8 (1)	O(2^{iv})—Li—O(2^{v})	81.5 (2)
O(2^{iv})—Li—O(2^{ix})	93.8 (2)	O(2^{iv})—Li—O(1^{vi})	88.2 (2)
O(2^{iv})—Li—O(2^{vii})	92.7 (2)	O(2^{iv})—Li—O(2^{viii})	166.5 (3)
O(2^{ix})—Li—O(1^{vi})	177.3 (2)	O(2^{ix})—Li—O(2^{vii})	98.8 (2)
O(2^{vii})—Li—O(1^{vi})	79.3 (2)	O(2^{vii})—Li—O(2^{viii})	90.2 (2)
Nb—O(1^{i})—Nb^{iii}	103.2 (2)		

Symmetry codes: (i) $x, -x, -x$; (ii) $-x, x, -x$; (iii) $-x, -x, x$; (iv) z, x, x; (v) x, z, x; (vi) $\frac{1}{2}+x, \frac{1}{2}+x, \frac{1}{2}+x$; (vii) $\frac{1}{2}+x, \frac{1}{2}-x, \frac{1}{2}-z$; (viii) $\frac{1}{2}-x, \frac{1}{2}+x, \frac{1}{2}-z$; (ix) $\frac{1}{2}-x, \frac{1}{2}-x, z-\frac{1}{2}$.

Among the 4608 measured reflections within the hemisphere, 2283 reflections were considered observed. Refinements by full-matrix least squares on these observed reflections were carried out with corrections for absorption and extinction. 134 reflections were obtained from the 2283 corrected reflections by averaging the equivalent reflections. The 134 independent reflections were used in the final refinement by full-matrix least squares on F. All calculations were carried out using the *UNICS*III program system (Sakurai & Kobayashi, 1979) and the full-matrix least-squares program *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979).

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$Bi_{12}(Bi^{III}_{0.5}Tl^{III}_{0.5})O_{19.50}$

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Abstract

The structure of the sillenite-type title compound has been determined at room temperature by single-