# The Crystal Structure of LiNb$_3$O$_8$

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The structure of LiNb$_3$O$_8$ has been determined from single crystal
X-ray data and refined by least squares methods. The symmetry is
monoclinic (space group $P2_1/a$) and the unit cell dimensions
$a = 15.262$ Å, $b = 5.033$ Å, $c = 7.457$ Å, and $\beta = 107.34°$.
The oxygen atoms are approximately hexagonally close-packed
with the metal atoms in octahedral interstices. The occupied octahedra
form zig-zag strings of two types in the b axis direction. One type
contains niobium atoms and the other a regular alternation of niobium
and lithium atoms. The structure is a superstructure of the α-PbO$_2$
type.

A few years ago a gravimetric study of the thermal decomposition of the
compound LiNb$_6$O$_{15}$F was performed at this Institute.[1,2] It was demon-
strated that this process may be described by the formula

$$3\text{LiNb}_6\text{O}_{15}\text{F(s)} = \text{NbOF}_3\text{(g)} + 3\text{LiNb}_3\text{O}_8\text{(s)} + 4\text{Nb}_2\text{O}_5\text{(s)}$$
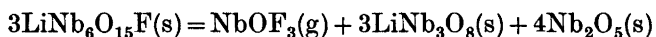
The niobium pentoxide obtained in this reaction was shown by a crystal
structure analysis to be a previously unknown polymorph of this material and
was named N $-$ Nb$_2$O$_5$.[3] This article will describe the result of an X-ray crys-
tallographic investigation of the other solid reaction product, the ternary
lithium niobium oxide LiNb$_3$O$_8$.

EXPERIMENTAL

LiNbO$_3$ was first prepared by heating of Li$_2$CO$_3$ (Baker Analyzed, 99.89 %) and
H $-$ Nb$_2$O$_5$ (Kawecki, 99.99 %) in the mol ratio 1 : 1 in a platinum crucible at 1100°C
for two days. Single crystals of LiNb$_3$O$_8$ were then obtained from a mixture of equivalent
amounts of LiNbO$_3$ and H $-$ Nb$_2$O$_5$, which was heated at 1000°C for two days. The
product consisted of rod-shaped, colourless crystals.
Single crystal X-ray studies showed the crystals to be of monoclinic symmetry. The
unit cell dimensions were derived from a Guinier powder photograph, taken with CuKα_1
radiation and with potassium chloride ($a = 6.2919$ Å at 20°C)[4] as an internal standard.
Accurate cell parameters were obtained by least squares refinement. The indexed powder
pattern and the cell constants are listed in Table 1 and Table 2, respectively.

Table 1. The Guinier powder pattern of LiNb$_3$O$_8$ (Cu$K\alpha_1$ radiation).

h k l	d_{obs}	$10^5 \times \sin^2\theta_{obs}$	$10^5 \times \sin^2\theta_{calc}$	I
2 0 0	7.291	1116	1118	m
$\bar{1}$ 1 1 ⎫	4.141	3460	⎧3451	s
2 1 0 ⎭			⎩3460	
0 1 1	4.111	3510	3513	s
$\bar{2}$ 1 1	3.875	3951	3949	m
$\bar{1}$ 0 2	3.724	4277	4281	m
$\bar{2}$ 0 2	3.658	4435	4437	s
4 0 0	3.642	4472	4472	s
0 0 2	3.559	4683	4683	m
2 1 1	3.342	5311	5313	m
$\bar{4}$ 0 2	3.038	6427	6428	m
$\bar{4}$ 1 1 ⎫	2.993	6622	⎧6621	w
$\bar{1}$ 1 2 ⎭			⎩6623	
$\bar{2}$ 1 2	2.959	6776	6779	s
4 1 0	2.949	6820	6815	s
0 1 2	2.909	7013	7025	w
2 0 2	2.878	7164	7165	s
$\bar{4}$ 1 2	2.600	8773	8770	w
0 2 0	2.516	9370	9368	s
2 1 2	2.498	9509	9507	s
6 0 0	2.429	10059	10063	w
$\bar{1}$ 2 1 ⎫	2.379	10481	⎧10478	v w
2 2 0 ⎭			⎩10486	
$\bar{6}$ 0 2	2.360	10654	10654	m
$\bar{2}$ 2 1	2.325	10973	10975	w
$\bar{6}$ 1 1	2.268	11531	11530	w
$\bar{1}$ 1 3	2.211	12137	12136	m
5 1 1	2.205	12207	12206	m
$\bar{3}$ 1 3 ⎫	2.192	12350	⎧12326	w
2 2 1 ⎭			⎩12339	
0 1 3	2.146	12887	12879	w

A crystal measuring $0.026 \times 0.074 \times 0.026$ mm^3 was selected by means of Weissenberg techniques for the collection of the X-ray diffraction data. The crystal was mounted with its longest edge as rotation axis. This direction coincided with the monoclinic axis. The intensity data were collected with a Siemens automatic diffractometer, using Nb-filtered Mo$K\alpha$ radiation and scintillation counting with pulse height discrimination. Each reflection was measured according to the 5-values technique. The scan range was given by the expressions

$$\Delta\theta(+) = k_1(+) + k_2(+)\tan\theta$$
$$\Delta\theta(-) = k_1(-) + k_2(-)\tan\theta$$

The constants were given the values $k_1(+) = 0.15$, $k_2(+) = 1.00$, $k_1(-) = 0.08$ and $k_2(-) = 1.00$ after a careful study of the widths of the peaks of some selected reflections evenly distributed over the θ-range. Measurements were made up to $\theta_{max} = 30°$. Three standard reflections were measured, with a period of 23 reflections.

The data were corrected by application of Lorentz and polarization factors. The shape of the crystal was somewhat irregular and the absorption correction applied was accordingly approximate. The small size of the crystal and comparatively low linear absorption coefficient, 59.5 cm^{-1},[5] combined to reduce the influence of this source of error.

All calculations were performed on the computers IBM 360/75 and IBM 1800. The programs used are listed in Table 6.

Table 2. Crystallographic data for LiNb$_3$O$_8$.

Unit cell dimensions:
  $a=$ 15.262 (2) Å
  $b=$ 5.033 (1) Å
  $c=$ 7.457 (1) Å
  $\beta=107.34$ (1)°
Unit cell content: 4 LiNb$_3$O$_8$
Density: $d_{obs}=4.975$ g cm^{-3}
       $d_{calc}=5.024$ g cm^{-3}

Space group: $P2_1/a$
Point positions:
x,y,z
$\bar{x},\bar{y},\bar{z}$
$\frac{1}{2}-x,\frac{1}{2}+y,\bar{z}$
$\frac{1}{2}+x,\frac{1}{2}-y,z$

Numbering of the atoms:
$(n\ 1)$
$(n\ 2)$
$(n\ 3)$
$(n\ 4)$

Atom (n)	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$	B	$\sigma(B)$
Nb(1)	0.16852	(5)	0.26959	(27)	0.00831	(10)	0.402	(19)
Nb(2)	0.07508	(5)	0.75486	(30)	0.24924	(11)	0.433	(17)
Nb(3)	0.16458	(5)	0.25757	(27)	0.51674	(10)	0.396	(17)
Li(1)	0.4201	(14)	0.2673	(80)	0.2597	(29)	2.27	(44)
O(1)	0.2817	(5)	0.4326	(15)	0.0846	(11)	0.84	(11)
O(2)	0.1558	(5)	0.4000	(15)	0.7354	(10)	0.77	(12)
O(3)	0.2157	(5)	0.5886	(16)	0.4148	(11)	0.92	(12)
O(4)	0.0389	(5)	0.1077	(15)	0.8551	(11)	0.75	(12)
O(5)	0.0989	(5)	0.9402	(15)	0.5005	(10)	0.58	(11)
O(6)	0.0485	(5)	0.4387	(15)	0.3528	(10)	0.62	(11)
O(7)	0.1006	(5)	0.5796	(15)	0.0375	(11)	0.71	(11)
O(8)	0.1551	(5)	0.0855	(15)	0.2225	(11)	0.72	(11)

STRUCTURE DETERMINATION

The Weissenberg photographs showed monoclinic symmetry with systematic absences $h0l$ for $h=2n+1$ and $0k0$ for $k=2n+1$. The only space group with these conditions is $P2_1/a$. If $0k0$ reflections with $k=2n+1$ are absent for reasons other than space group symmetry, the two space groups Pa and $P2/a$ are also possible. These space groups correspond to $P2_1/c$ (No.14), Pc (No. 7), and $P2/c$ (No. 13), respectively, in the *International Tables of X-Ray Crystallography*. The unconventional setting in the present study had to be chosen with respect to the computer programs applied.

The structure was solved by means of the Patterson function. The study was started with the assumption that $P2_1/a$ was the correct space group. The Patterson projection along [010] then gave approximate niobium coordinates. The oxygen positions were found from a three-dimensional Fourier synthesis based on this arrangement of the niobium atoms. All the atomic peaks thus identified could be assigned to fourfold point positions $4(e)$ in the space group $P2_1/a$.

The structure was refined by full matrix least squares techniques. Reflections with large relative errors, $\sigma(|F_{obs}|)/|F_{obs}| \geq 0.20$, were excluded from the calculations. The refinement then comprised 831 independent reflections. Atomic scattering factors for unionized atoms were used, with the application of the real and imaginary part of the dispersion correction.[6] Corrections were made for secondary extinction, which proved to be quite significant. The intensities of the most affected reflections had been reduced by as much as 54 %.

Table 3. Observed and calculated structure factors for LiNb₃O₈. The scale factor has been applied on the calculated F values.

H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c
0	0	2	52.57	48.99	2	1	1	30.89	30.87	3	5	4	18.67	19.11	6	2	0	13.15	13.65
0	0	4	101.91	100.49	2	1	2	73.53	70.35	3	5	6	17.41	15.47	5	0	1	10.55	10.55
0	0	6	22.00	21.35	2	1	3	25.66	25.71	3	6	-4	12.94	8.14	5	3	-1	9.42	7.88
0	0	8	70.16	69.60	2	1	4	19.41	18.77	3	6	2	11.14	9.43	5	3	0	12.39	12.14
0	1	1	58.01	52.55	2	1	5	12.90	12.37	4	0	-10	37.02	37.57	5	3	4	12.97	13.84
0	1	2	14.76	14.70	2	1	6	56.53	56.05	4	0	-9	32.62	32.29	5	4	-6	19.48	18.20
0	1	3	34.86	32.94	2	1	7	24.19	24.07	4	0	-8	65.06	63.25	5	4	-3	14.78	14.98
0	1	4	29.66	28.92	2	2	-8	16.15	17.74	4	0	-7	13.13	14.95	5	4	-2	20.35	20.03
0	1	6	31.84	31.55	2	2	-7	36.44	34.81	4	0	-6	44.24	42.43	5	4	-1	10.37	8.30
0	1	7	19.84	18.50	2	2	-6	34.88	34.11	4	0	-5	46.62	46.03	5	4	0	17.66	20.04
0	1	8	23.20	22.65	2	2	-5	37.10	36.38	4	0	-4	74.00	73.44	5	4	2	19.04	19.13
0	1	9	32.23	33.50	2	2	-4	10.89	7.93	4	0	-3	29.44	30.46	5	4	4	10.48	10.22
0	2	0	85.12	86.05	2	2	-3	44.07	43.31	4	0	-2	46.72	46.34	5	5	-4	10.40	10.60
0	2	2	33.45	34.26	2	2	-2	52.51	52.09	4	0	-1	52.82	52.63	5	5	0	15.93	16.52
0	2	4	91.59	91.67	2	2	-1	31.02	32.59	4	0	0	70.74	65.92	5	5	2	26.87	25.71
0	2	6	22.66	21.94	2	2	0	17.11	16.97	4	0	1	45.06	42.93	5	5	2	12.37	12.68
0	2	7	11.52	9.66	2	2	1	25.47	26.56	4	0	2	12.83	12.60	5	5	4	12.87	15.77
0	2	8	60.14	59.23	2	2	2	79.15	73.81	4	0	3	15.66	14.77	5	6	-4	19.07	19.61
0	3	1	39.08	38.84	2	2	3	33.93	33.84	4	0	4	65.34	63.22	5	6	-2	21.68	23.97
0	3	2	11.03	11.55	2	2	4	30.19	30.12	4	0	5	37.48	37.59	5	6	1	11.17	10.41
0	3	3	31.54	32.10	2	2	5	19.29	19.52	4	0	7	20.41	20.30	5	6	2	24.53	23.98
0	3	4	18.82	18.06	2	2	6	67.58	65.65	4	0	9	29.47	28.06	6	0	-10	46.77	47.66
0	3	5	34.17	33.08	2	2	7	27.56	28.20	4	1	-10	13.31	11.33	6	0	-9	20.59	19.51
0	3	6	18.71	18.84	2	2	8	30.57	30.30	4	1	-9	20.84	21.41	6	0	-8	19.74	16.88
0	3	7	15.10	17.54	2	3	-8	23.78	23.45	4	1	-7	27.36	27.30	6	0	-6	77.79	78.03
0	3	8	41.89	41.16	2	3	-7	18.32	19.24	4	1	-3	47.29	46.83	6	0	-5	19.11	19.23
0	4	-4	75.00	75.67	2	3	-6	78.98	77.13	4	1	-5	24.54	25.33	6	0	-4	35.21	35.64
0	4	0	76.66	76.52	2	3	-5	11.63	10.73	4	1	-4	53.79	96.55	6	0	-3	14.27	15.98
0	4	2	30.73	30.75	2	3	-4	33.87	33.51	4	1	-2	23.69	24.15	6	0	-2	60.59	66.43
0	4	4	19.77	19.07	2	3	-3	25.97	26.16	4	1	-1	18.34	19.42	6	0	-1	17.17	16.90
0	5	1	25.33	24.37	2	3	-2	86.59	90.23	4	1	0	93.92	95.68	6	0	-4	40.34	41.29
0	5	3	20.66	21.91	2	3	-1	18.17	18.41	4	1	1	13.34	12.71	6	0	1	10.36	10.97
0	5	4	17.45	17.08	2	3	0	31.79	32.27	4	1	2	34.89	35.04	6	0	2	102.04	101.01
0	5	5	31.56	31.31	2	3	1	23.50	23.68	4	1	3	10.25	11.08	6	0	3	13.19	12.71
0	5	6	20.11	18.89	2	3	2	104.76	103.93	4	1	4	96.29	94.68	6	0	4	38.22	38.00
0	6	0	77.24	78.20	2	3	3	22.11	22.30	4	1	5	10.60	13.68	6	0	6	69.07	66.97
0	6	-2	17.14	17.66	2	3	4	17.75	19.13	4	1	6	28.50	28.09	6	0	7	14.28	15.78
0	6	4	59.07	58.68	2	3	5	13.65	15.63	4	1	8	67.36	63.01	6	0	8	17.25	16.48
1	1	-3	57.96	54.19	2	3	6	51.65	50.26	4	2	-9	22.73	23.18	6	1	-10	32.99	29.71
1	1	-2	8.34	6.28	2	3	7	20.17	20.42	4	2	-8	50.78	49.05	6	1	-9	11.85	12.05
1	1	5	8.60	8.00	2	4	-8	18.72	17.67	4	2	-7	19.84	22.04	6	1	-8	33.54	34.60
1	2	-8	11.95	9.74	2	4	-7	30.65	31.51	4	2	-6	38.76	38.32	6	1	-7	37.90	38.08
1	2	-6	13.21	10.03	2	4	-6	25.37	25.01	4	2	-4	39.53	39.69	6	1	-6	15.98	14.22
1	2	-4	14.66	15.73	2	4	-5	11.76	14.04	4	2	-4	63.71	62.70	6	1	-5	22.42	23.08
1	2	-2	10.28	9.86	2	4	-3	29.76	29.35	4	2	-3	36.44	35.95	6	1	-4	34.96	35.49
1	2	0	14.27	14.52	2	4	-2	40.40	40.35	4	2	-2	35.73	36.65	6	1	-3	46.30	47.03
1	2	2	10.55	10.94	2	4	-1	22.07	22.12	4	2	-1	39.49	41.29	6	1	-1	28.27	29.18
1	2	3	9.79	10.07	2	4	0	12.70	10.05	4	2	0	51.33	52.86	6	1	0	14.04	14.06
1	2	4	12.39	12.99	2	4	1	31.16	30.01	4	2	1	35.69	35.05	6	1	1	44.24	44.09
1	2	5	20.23	20.74	2	4	2	57.12	56.47	4	2	2	16.58	16.14	6	1	3	29.77	28.71
1	3	-8	12.09	9.45	2	4	3	31.41	32.46	4	2	3	33.77	32.79	6	1	5	35.25	33.42
1	3	-6	13.26	12.25	2	4	4	19.43	21.10	4	2	5	9.27	7.38	6	1	5	25.65	24.48
1	3	-4	10.23	11.94	2	4	5	10.98	12.28	4	2	6	33.73	34.22	6	1	6	44.64	43.56
1	3	-2	18.90	17.70	2	4	5	53.42	52.47	4	2	6	11.74	9.10	6	1	7	26.40	25.93
1	3	2	12.01	14.07	2	4	7	23.63	23.47	4	2	7	16.21	16.08	6	1	8	18.42	19.82
1	4	-4	15.54	15.94	2	5	-6	56.64	57.62	4	3	-9	19.04	18.13	6	2	-9	13.94	11.97
1	4	-3	13.54	13.39	2	5	-5	16.56	16.13	4	3	-8	37.20	38.11	6	2	-8	16.32	14.35
1	4	-2	9.89	9.59	2	5	-4	22.65	22.70	4	3	-5	13.60	13.77	6	2	-6	78.68	79.71
1	4	0	21.14	20.99	2	5	-3	17.49	17.86	4	4	-9	44.07	42.86	6	2	-5	15.12	15.33
1	4	1	13.49	12.35	2	5	-2	63.69	65.52	4	3	-3	19.86	20.14	6	2	-3	33.32	33.38
1	4	2	20.95	21.15	2	5	-1	10.74	9.45	4	3	-2	12.43	11.93	6	2	-2	97.91	110.93
1	4	4	22.70	21.18	2	5	0	16.95	17.68	4	3	0	83.27	89.58	6	2	-1	8.52	9.89
1	4	5	29.00	27.77	2	5	1	21.08	19.03	4	3	1	13.77	13.29	6	2	0	44.77	44.95
1	4	6	15.00	15.64	2	5	2	49.03	48.19	4	3	1	32.48	33.17	6	2	1	12.93	14.44
1	5	-6	20.27	19.86	2	5	5	17.59	16.65	4	3	3	10.70	11.30	6	2	2	93.44	92.87
1	5	-2	14.40	10.59	2	5	5	13.56	13.70	4	3	3	77.94	77.08	6	2	4	27.79	26.71
1	5	-2	15.71	17.25	2	5	6	39.77	37.62	4	3	6	25.86	25.68	6	2	5	13.07	14.40
1	5	2	12.11	11.22	2	6	-5	15.18	12.64	4	3	8	60.79	58.20	6	2	7	72.75	70.59
1	6	-3	11.16	11.55	2	6	-3	21.00	19.46	4	4	-8	43.71	43.63	6	2	8	15.13	14.27
1	6	-2	23.29	23.20	2	6	-2	30.02	29.10	4	4	-7	16.18	17.10	6	3	-9	35.59	37.09
1	6	0	25.50	25.74	2	6	-1	18.76	21.25	4	4	-6	34.10	33.02	6	3	-7	40.62	40.30
1	6	2	16.55	18.05	2	6	1	19.38	16.97	4	4	-5	31.12	30.15	6	3	-6	27.08	24.80
1	6	4	21.58	22.57	2	6	2	39.70	38.76	4	4	-4	49.00	49.54	6	3	-4	31.23	31.32
2	0	-8	22.47	22.71	2	6	3	21.33	22.17	4	4	-4	13.89	15.26	6	3	-3	42.81	43.01
2	0	-7	32.46	31.89	2	6	4	13.66	15.11	4	4	-2	29.09	29.49	6	3	-1	26.38	27.23
2	0	-6	9.01	9.01	3	1	5	11.64	12.36	4	4	-1	37.79	39.78	6	3	0	13.60	13.58
2	0	-5	18.15	18.95	3	1	0	10.04	9.48	4	4	0	40.18	40.08	6	3	1	36.07	34.92
2	0	-3	40.65	39.90	3	1	4	11.51	10.91	4	4	1	27.94	27.71	6	3	2	17.43	16.80
2	0	-2	68.92	65.19	3	1	7	19.79	19.51	4	4	1	16.16	15.99	6	3	3	30.85	30.95
2	0	-1	40.89	38.81	3	2	-4	9.94	9.93	4	4	3	25.96	26.39	6	3	5	20.37	20.42
2	0	0	25.20	25.08	3	2	-11	52.81	52.54	4	4	5	24.56	24.11	6	3	6	13.40	14.07
2	0	1	45.65	42.74	3	2	-8	12.39	11.54	4	4	7	13.79	13.24	6	3	7	28.01	27.15
2	0	2	78.44	77.67	3	3	-5	15.14	15.14	4	5	-4	11.71	11.53	6	4	-6	65.03	64.84
2	0	3	32.37	31.83	3	3	-4	16.23	16.46	4	5	-4	63.00	61.99	6	4	-4	19.46	19.57
2	0	4	35.84	36.08	3	3	-2	11.94	12.47	4	5	-2	35.32	35.20	6	4	-2	22.25	89.52
2	0	5	27.35	27.30	3	3	-1	11.80	11.90	4	5	0	64.63	66.26	6	4	-1	9.53	9.58
2	0	6	67.44	65.72	3	3	0	27.39	27.97	4	5	1	11.07	12.29	6	4	0	24.23	24.78
2	0	7	20.63	21.20	3	3	2	23.15	21.52	4	5	1	17.44	18.81	6	4	1	14.12	13.77
2	0	8	24.48	23.28	3	3	3	11.81	10.69	4	5	4	67.06	65.27	6	4	2	77.39	76.60
2	0	0	16.22	16.24	3	3	4	12.97	14.74	4	6	-6	27.02	27.87	6	4	4	18.82	18.78
2	1	-9	14.90	17.82	3	3	6	12.21	10.65	4	6	-4	38.13	37.60	6	4	6	59.11	59.09
2	1	-8	32.68	30.04	3	4	-1	27.39	27.71	4	6	-3	11.21	13.35	6	5	-7	24.29	26.66
2	1	-7	14.29	14.26	3	4	6	16.09	15.40	4	6	-2	19.64	17.96	6	5	-5	16.05	15.04
2	1	-6	83.29	82.08	3	5	-5	20.68	18.94	4	6	3	31.78	30.94	6	5	-4	21.42	19.50
2	1	-5	20.35	19.20	3	5	-4	22.65	23.89	4	6	3	20.91	20.48	6	5	-3	34.73	35.75
2	1	-4	46.30	44.95	3	5	-2	24.15	25.16	4	6	1	10.51	8.81	6	5	1	24.86	24.54
2	1	-3	28.60	29.11	3	5	-1	11.56	11.75	5	1	-4	12.14	11.99	6	5	1	23.67	23.13
2	1	-2	93.86	95.68	3	5	0	20.01	20.84	5	2	-7	22.44	23.53	6	5	2	16.29	16.88
2	1	-1	28.91	29.37	3	5	2	20.74	18.62	5	2	-6	14.87	12.90	6	5	3	22.26	22.13
2	1	0	49.94	47.06	3	5	3	11.59	11.48	5	2	-2	19.90	20.13	6	5	5	18.39	18.26

Table 3. Continued.

H	K	L	Fo	Fc
6	6	-4	14.85	14.15
6	6	-2	43.24	46.49
6	6	0	14.29	17.27
6	6	1	12.30	12.30
6	6	2	58.97	57.86
7	1	-8	11.41	4.64
7	1	-5	28.15	28.97
7	1	-5	27.95	28.97
7	1	-1	10.23	10.50
7	1	2	8.22	6.08
7	2	-4	0.42	9.40
7	2	-2	11.49	11.40
7	2	-1	14.72	16.33
7	2	0	8.54	10.20
7	2	2	11.33	12.74
7	2	3	31.01	33.32
7	2	4	12.49	11.75
7	3	-8	12.84	14.73
7	3	-4	17.96	13.25
7	3	0	12.06	12.14
7	4	-5	10.40	9.39
7	4	-4	17.25	16.95
7	4	-2	19.99	20.77
7	4	0	15.64	16.88
7	4	2	19.54	17.62
7	4	4	18.18	17.30
7	4	6	17.31	16.83
7	5	-6	14.09	13.98
7	5	-5	17.43	17.55
7	5	-4	19.66	19.23
7	5	0	15.60	14.18
7	6	-2	11.32	14.58
7	6	-1	12.26	14.10
7	6	0	19.02	20.04
7	6	2	19.35	21.10
8	0	-10	27.52	28.45
8	0	-8	36.06	36.29
8	0	-8	32.42	32.91
8	0	-6	26.74	27.71
8	0	-5	40.21	41.19
8	0	-4	33.93	33.45
8	0	-2	12.72	11.09
8	0	-1	23.35	23.09
8	0	0	72.69	73.05
8	0	1	23.41	23.28
8	0	2	12.78	13.14
8	0	3	20.33	19.30
8	0	4	67.11	65.99
8	0	6	20.16	19.17
8	0	6	27.52	26.36
8	0	7	11.15	10.16
8	1	-10	25.87	23.92
8	1	-9	16.87	16.84
8	1	-8	64.23	64.53
8	1	-7	23.44	23.37
8	1	-6	40.63	39.93
8	1	-5	29.68	29.25
8	1	-4	84.37	87.71
8	1	-3	23.45	24.91
8	1	-2	43.74	46.12
8	1	-1	35.42	35.36
8	1	0	93.76	97.89
8	1	1	29.04	28.52
8	1	2	36.80	36.19
8	1	3	31.44	29.71
8	1	4	58.32	57.47
8	1	7	23.88	23.47
8	1	7	17.29	16.40
8	2	-9	25.46	24.68
8	2	-6	20.04	20.89
8	2	-5	30.85	30.07
8	2	-4	46.42	47.34
8	2	-3	10.53	12.32
8	2	-1	33.35	33.79
8	2	0	61.33	62.62
8	2	1	17.62	16.99
8	2	2	14.79	14.51
8	2	3	15.33	16.60
8	2	4	66.65	65.97
8	2	5	16.57	15.49
8	2	6	18.94	18.43
8	3	-9	15.37	14.53
8	3	-8	55.30	55.74
8	3	-7	20.53	20.77
8	3	-6	36.07	34.54
8	3	-5	24.71	25.12
8	3	-4	67.28	67.37
8	3	-3	23.43	22.50
8	3	-2	40.32	40.54
8	3	-1	40.00	34.25
8	3	0	45.79	44.35
8	3	1	22.30	21.73
8	3	2	24.03	23.89
8	3	3	22.84	27.16
8	3	4	52.14	50.51
8	3	5	17.80	20.65
8	3	6	15.60	16.29
8	4	-6	13.89	16.07
8	4	-5	26.49	26.64
8	4	-4	34.43	33.72
8	4	-4	9.81	9.34
8	4	-1	18.57	17.64
8	4	0	48.17	49.45
8	4	1	10.31	10.40
8	4	2	10.24	10.02
8	4	3	19.27	17.03
8	4	4	55.68	54.07
8	4	5	16.38	15.49
8	5	-6	26.17	27.11
8	5	-5	25.61	26.99
8	5	-3	14.46	15.01
8	5	-2	31.25	30.51
8	5	-1	26.83	25.88
8	5	0	62.35	63.09
8	5	2	26.71	25.26
8	5	3	23.68	22.97
8	5	4	38.34	39.49
8	6	-4	22.25	20.30
8	6	-1	12.90	14.80
8	6	0	38.31	37.42
8	6	1	12.63	14.42
9	1	-6	11.35	11.48
9	1	0	9.77	9.17
9	1	5	14.46	14.30
9	2	-3	27.44	28.59
9	3	-6	11.94	12.64
9	3	-4	10.77	9.95
9	3	-2	11.74	13.00
9	3	0	10.67	10.57
9	3	1	12.51	10.08
9	3	2	15.97	16.80
9	3	-4	13.49	12.80
9	3	5	11.84	7.17
9	4	-3	21.26	22.08
9	5	-6	12.61	15.61
9	5	-4	15.72	15.58
9	5	-2	25.54	25.20
9	5	0	21.15	21.17
9	5	1	14.16	14.54
9	5	2	17.65	21.31
10	0	-10	33.44	32.50
10	0	-9	21.32	23.46
10	0	-8	43.22	42.93
10	0	-7	33.06	33.94
10	0	-6	49.06	49.26
10	0	-5	21.07	22.93
10	0	-4	48.23	47.93
10	0	-3	41.81	42.79
10	0	-2	36.39	36.33
10	0	-1	31.85	30.55
10	0	0	40.13	40.67
10	0	1	42.86	41.74
10	0	3	28.95	27.19
10	0	5	22.24	20.92
10	0	7	26.53	24.56
10	1	-9	15.03	15.97
10	1	-7	12.75	12.34
10	1	-6	49.35	49.57
10	1	-5	12.82	12.15
10	1	-4	11.62	10.68
10	1	-3	8.97	9.58
10	1	-2	82.98	87.57
10	1	0	28.60	28.59
10	1	2	85.37	83.65
10	1	4	26.02	27.02
10	1	6	70.09	69.79
10	2	-9	12.44	13.63
10	2	-8	41.32	41.84
10	2	-7	33.79	33.89
10	2	-6	47.72	47.75
10	2	-5	15.70	16.89
10	2	-3	44.50	44.73
10	2	-2	48.34	49.58
10	2	-1	23.80	24.22
10	2	0	34.48	34.24
10	2	2	38.57	36.25
10	2	2	36.07	36.13
10	2	4	24.07	23.81
10	2	4	16.81	16.48
10	3	6	26.15	25.89
10	3	-7	19.49	18.14
10	3	-6	67.87	68.09
10	3	-5	13.56	11.07
10	3	-2	64.71	67.57
10	3	0	9.54	6.75
10	3	0	13.15	13.28
10	3	4	78.86	78.05
10	3	4	21.87	21.53
10	3	6	59.77	58.97
10	4	-7	32.83	32.71
10	4	-6	38.40	38.99
10	4	-5	19.95	19.52
10	4	-4	33.24	34.37
10	4	-2	31.68	32.55
10	4	-1	18.87	17.95
10	4	0	24.38	22.40
10	4	2	32.04	31.73
10	4	2	27.63	26.34
10	4	4	24.15	23.88
10	4	5	22.07	21.53
10	5	-4	34.67	35.29
10	5	-2	57.57	58.90
10	5	0	15.64	16.07
10	5	2	56.30	59.18
10	6	-3	18.87	19.97
10	6	-1	13.80	13.73
11	1	-5	11.20	11.18
11	1	-2	8.58	7.17
11	1	-1	32.52	33.23
11	2	-5	12.67	12.89
11	2	0	16.09	15.71
11	2	2	9.67	7.28
11	3	2	12.29	11.03
11	4	-4	24.01	23.33
11	4	-2	12.38	11.29
11	4	0	16.95	17.60
11	4	4	12.12	12.24
12	0	-8	61.64	62.22
12	0	-7	24.61	23.72
12	0	-6	23.52	22.63
12	0	-4	93.14	99.86
12	0	-3	23.86	24.18
12	0	-2	24.37	30.18
12	0	-1	21.18	20.46
12	0	0	78.84	80.40
12	0	1	19.07	18.95
12	0	2	25.69	26.35
12	0	3	20.19	19.93
12	0	4	72.57	72.09
12	0	6	23.23	22.09
12	1	-9	33.52	33.25
12	1	-8	20.44	18.48
12	1	-5	41.40	41.51
12	1	-5	40.50	41.18
12	1	-3	11.59	13.71
12	1	0	30.60	31.07
12	1	-1	36.01	36.97
12	1	0	15.39	14.10
12	1	1	17.05	15.84
12	1	3	25.90	25.45
12	1	4	23.45	22.59
12	1	5	19.15	19.13
12	2	-8	51.99	51.98
12	2	-6	20.63	21.66
12	2	-4	24.22	22.33
12	2	-4	16.23	14.10
12	2	-4	64.50	65.97
12	2	-4	20.35	21.29
12	2	-2	29.20	30.75
12	2	-1	16.50	17.39
12	2	0	70.88	81.71
12	2	1	15.26	14.99
12	2	2	36.09	35.31
12	2	2	12.33	13.50
12	2	5	62.66	62.07
12	2	6	16.07	15.12
12	3	-6	32.45	33.57
12	3	-5	33.84	33.84
12	3	-3	10.32	8.90
12	3	-2	24.55	25.59
12	3	-1	37.05	31.38
12	3	0	25.37	24.13
12	3	1	17.09	16.44
12	3	3	23.05	22.71
12	4	-7	44.35	41.58
12	4	-7	17.13	15.88
12	4	-5	20.21	19.86
12	4	-5	20.53	19.43
12	4	-4	14.21	12.48
12	4	-2	53.26	54.50
12	4	-2	25.48	27.88
12	4	-1	18.58	16.69
12	4	0	67.91	66.65
12	4	1	12.63	11.62
12	4	2	25.90	27.29
12	4	2	20.67	19.00
12	4	3	25.66	26.90
12	5	-2	22.42	22.34
12	5	0	31.03	30.14
12	5	1	11.35	8.72
12	5	1	15.61	12.65
13	1	-7	13.19	14.42
13	2	-2	9.89	11.30
13	2	1	11.62	12.63
13	3	-6	12.48	12.08
13	3	-2	10.99	9.21
13	4	-3	11.92	8.28
13	4	-2	37.80	17.56
13	4	0	17.13	16.02
13	4	1	27.23	23.95
14	0	-9	12.67	7.97
14	0	-8	24.68	25.18
14	0	-7	23.98	23.40
14	0	-6	36.68	37.93
14	0	-4	11.09	11.21
14	0	-3	21.89	22.34
14	0	-2	46.36	48.41
14	0	1	14.96	14.78
14	0	-9	63.71	64.93
14	1	-9	21.55	22.56
14	1	-8	35.36	35.62
14	1	-8	24.23	23.74
14	1	-6	54.04	55.19
14	1	-5	25.00	25.54
14	1	-4	45.71	45.97
14	1	-3	31.90	33.93
14	1	-2	52.92	52.81
14	1	-1	21.20	21.54
14	1	1	29.89	29.42
14	1	2	51.08	50.40
14	1	3	22.36	20.34
14	1	4	27.08	28.27
14	1	4	21.64	21.30
14	2	-8	28.22	27.55
14	2	-6	27.05	26.77
14	2	-6	21.25	20.77
14	2	-4	20.78	21.03
14	2	-3	19.90	21.38
14	2	-2	46.44	46.57
14	2	1	17.90	16.31
14	2	2	51.39	50.61
14	3	-7	19.73	22.38
14	3	-6	57.07	58.07
14	3	-5	18.31	18.51
14	3	-4	36.61	34.54
14	3	-3	25.37	26.06
14	3	-2	67.12	70.58
14	3	-1	19.58	19.58
14	3	0	37.98	37.11
14	3	1	30.16	30.77
14	3	3	17.00	17.41
14	4	-6	20.15	19.93
14	4	-4	14.52	14.45
14	4	-3	19.72	20.52
14	4	-2	28.10	28.39
14	4	2	44.63	44.01
14	5	-3	27.30	27.84
14	5	-1	11.79	15.30
15	1	-4	10.93	6.14
15	1	-1	11.55	12.15
15	1	3	15.56	14.99
15	2	-5	14.25	16.34
15	3	-2	17.92	16.26
15	3	-2	14.08	14.58
15	3	0	14.42	14.35
15	4	-1	11.25	8.07
16	0	-8	33.80	32.95
16	0	-7	14.05	15.24
16	0	-6	45.29	45.66
16	0	-4	34.08	35.78
16	0	-3	41.85	41.61
16	0	-3	12.18	11.56
16	0	-2	39.37	39.99
16	0	-1	32.92	32.00
16	0	0	38.03	36.76
16	0	1	18.04	16.62
16	0	2	28.26	29.54
16	0	3	30.52	30.73
16	1	-8	42.97	42.94
16	1	-4	22.67	23.76
16	1	-3	53.51	54.67
16	1	-1	20.06	19.56
16	1	1	10.99	9.28
16	2	-7	19.50	21.27
16	2	-7	14.16	14.16
16	2	-5	29.75	30.96
16	2	-3	24.09	24.42
16	2	-3	13.13	14.82
16	2	-2	39.25	38.60
16	2	1	16.03	15.68
16	2	2	21.16	21.15
16	2	2	24.77	22.89
16	3	-4	17.16	17.13
16	3	0	56.39	57.52
16	3	0	16.62	17.65
16	3	2	57.31	58.58
16	3	2	11.36	12.37
16	4	-4	32.93	32.21
16	4	-2	22.02	22.03
16	4	-2	32.58	35.86
16	4	5	29.05	29.29
17	1	-3	15.24	11.28
17	2	-6	12.72	11.76
18	0	-6	40.27	41.90
18	0	-4	23.03	23.34
18	0	-4	26.04	28.85
18	0	-2	16.63	15.40
18	0	-2	64.64	65.88
18	0	0	20.26	19.23
18	0	1	37.90	40.05
18	0	1	19.48	19.44
18	1	-7	52.26	52.84
18	1	-4	25.15	27.02
18	1	-4	34.84	34.51
18	1	-3	28.75	29.12
18	1	0	14.49	16.03
18	1	0	23.62	21.49
18	1	1	20.89	22.11
18	1	2	30.96	29.61
18	2	-6	53.52	52.54
18	2	-4	22.65	24.86
18	2	-4	28.06	30.86
18	2	-2	15.26	14.57
18	2	-2	54.72	55.47
18	2	0	19.66	20.67
18	2	0	25.00	26.23
18	3	-1	20.24	19.74
18	3	-4	30.78	30.51
18	3	-3	25.10	26.46
18	3	0	16.79	16.97
19	2	-1	15.64	15.49
20	0	-5	17.23	18.88
20	0	-3	30.85	30.86
20	0	-2	12.02	11.91
20	0	-1	33.56	33.60
20	1	-5	20.06	21.62
20	1	-4	45.57	46.96
20	1	-3	20.31	19.29
20	1	-1	37.33	39.18
20	1	-1	27.86	27.14
20	2	-3	14.71	12.99

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The structure factors were weighted according to the formula $w = (\sigma|F_{obs}|^2 + a|F_{obs}|^2 + b|F_{obs}|^3)^{-1}$ with $a = 0.30 \times 10^{-4}$, $b = 0.15 \times 10^{-4}$. In this way an R-value of 0.042 was reached.

At this stage of the investigation it was evident that in the niobium-oxygen structure there are screw axes along the b direction and that the space group $P2/a$ is incompatible with the structure. Thus it was concluded that $P2_1/a$ is the correct space group. Attempts to lower the symmetry to Pa were unsuccessful. A lithium in the structure was searched for in a three-dimensional difference synthesis, with the niobium and oxygen contributions subtracted. This function was found to contain a maximum which could be identified as being due to a lithium atom. When the lithium atoms were included in the refinement, the R-value dropped to 0.036.

The final values of the atomic parameters and their estimated standard deviations are presented in Table 2. The comparison between observed and calculated structure factors is given in Table 3. The weight analyses are shown in Table 4.

Table 4. Weight analysis used in the last cycle of the refinement of $LiNb_3O_8$. $w =$ weighting factor. $\Delta = ||F_{obs}| - |F_{calc}||$.

Interval F_{obs}	$\overline{w\Delta^2}$	Number of independent reflections	Interval $\sin \theta$	$\overline{w\Delta^2}$	Number of independent reflections
0.0 − 11.8	1.164	76	0.0 − 0.2321	1.422	103
11.8 − 14.0	1.126	82	0.2321 − 0.2924	0.861	114
14.0 − 17.0	0.873	86	0.2924 − 0.3347	1.002	102
17.0 − 19.7	1.019	77	0.3347 − 0.3684	0.920	88
19.7 − 23.3	0.936	95	0.3684 − 0.3969	1.135	89
23.3 − 26.5	0.945	70	0.3969 − 0.4217	0.779	85
26.5 − 32.3	1.003	84	0.4217 − 0.4440	0.861	95
32.3 − 40.0	0.840	93	0.4440 − 0.4642	1.075	65
40.0 − 58.0	0.917	89	0.4642 − 0.4827	0.948	69
58.0 − 110.0	1.234	79	0.4827 − 0.5000	0.903	21

DESCRIPTION AND DISCUSSION OF THE STRUCTURE

The structure of $LiNb_3O_8$ (*cf.* Fig. 1) thus derived may be described as built of oxygen octahedra containing niobium or lithium atoms. The octahedra share edges and form zig-zag strings along the b-axis direction. The strings are of two different types, A and B (*cf.* Fig. 2). The A-type consists exclusively of NbO_6 octahedra, as distinguished from the B-type where LiO_6 and NbO_6 octahedra alternate.

Parallel strings are packed together in the structure to form identical planar layers with an approximately constant distance between the strings $A - A$, $A - B$ or $B - B$ (see Fig. 2), which correspond to a close-packed arrangement of the oxygen atoms. The layers consist of an alternation of two A-type strings and two B-type strings. From one layer to the next, the arrangement is shifted by 1 1/2 distance between the strings in a direction perpendicular
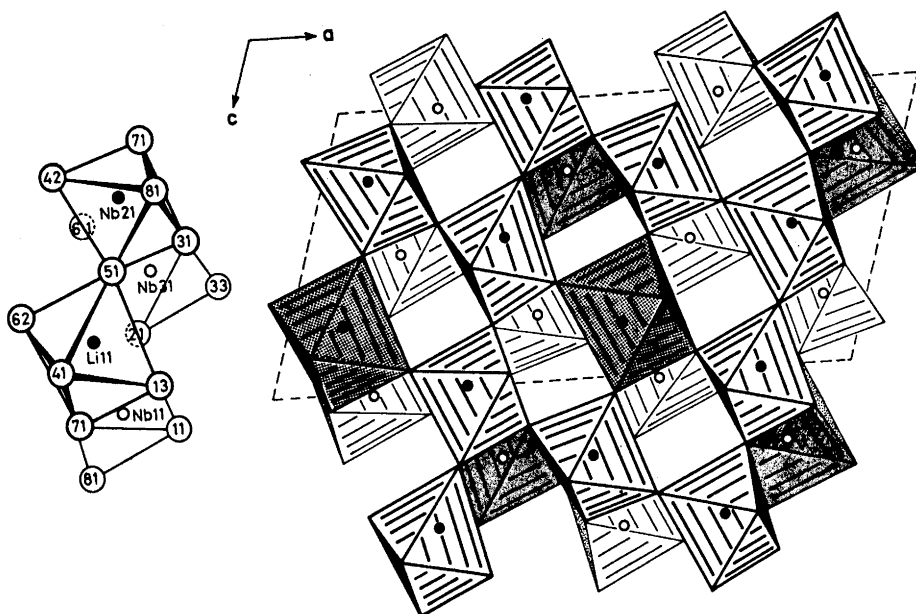
Fig. 1. The crystal structure of $LiNb_3O_8$ viewed along [010]. $\bigcirc$, metal atoms at approximately $y = 0.25$. $\bullet$, metal atoms at approximately $y = 0.75$. The shaded octahedra contain Li atoms.
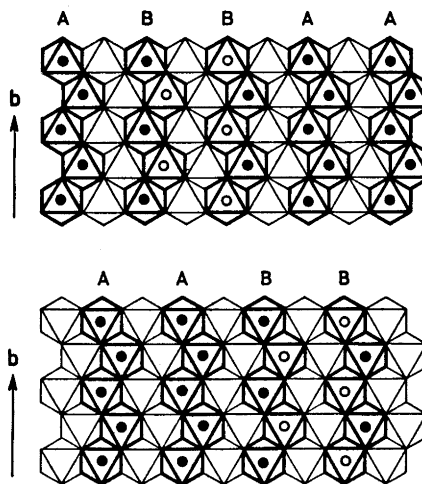
Fig. 2. Idealized crystal structure of $LiNb_3O_8$, based on *hcp* arrangement of O atoms and visualized as formed by a stacking of layers. The layers are composed of zig-zag strings of metal-oxygen octahedra. Filled and open rings represent Nb and Li atoms, respectively. Octahedra in adjacent layers are coupled by means of corner oxygen sharing.

to the strings. The strings in adjacent layers are connected by corner sharing to form a three-dimensional network.

It is possible to describe the structure of $LiNb_3O_8$ as a slightly distorted hexagonally close-packed array of oxygen atoms with lithium and niobium atoms occupying one half of the octahedral interstices in an ordered way.

The repeat unit along the unique direction of the basic *hcp* oxygen network involves eight layers, two of which are schematically shown in Fig. 2.

The shapes and sizes of the various metal-oxygen octahedra are rather different. In the A-type strings both of the two crystallographically different NbO_6 octahedra are similarly distorted, with five rather short $Nb-O$ distances $(1.81-2.11$ Å$)$ and one considerably longer $(2.29$ and 2.32 Å$)$ (*cf.* Table 5

Table 5. Interatomic distances in $LiNb_3O_8$. Standard deviations are given within paren theses. *a* indicates that the atom belongs to the next unit cell.

Metal-oxygen distances in Å			Mean value
Nb(11)a − O(11)a	1.844	(8)	
O(81)a	1.913	(7)	
O(71)a	1.922	(8)	
O(13)a	2.060	(8)	1.993
O(21)	2.091	(7)	
O(41)	2.129	(7)	
Nb(21) − O(42)a	1.818	(7)	
O(61)	1.866	(8)	
O(71)	1.945	(8)	
O(51)	2.027	(7)	2.009
O(81)	2.107	(8)	
O(31)	2.290	(8)	
Nb(31) − O(21)	1.823	(7)	
O(51)a	1.872	(8)	
O(33)	1.943	(8)	
O(61)	2.043	(7)	2.013
O(31)	2.077	(8)	
O(81)	2.320	(7)	
Li(11) − O(41)a	2.076	(36)	
O(51)	2.082	(27)	
O(62)a	2.159	(28)	2.188
O(21)	2.203	(37)	
O(13)	2.266	(26)	
O(71)a	2.343	(27)	

Metal-metal distances in Å		
Nb(11)a − Li(11)	3.248	(34)
Li(11)a	3.235	(34)
Nb(21) − Nb(31)	3.239	(2)
Nb(31)a	3.260	(2)
Nb(31) − Li(11)	3.527	(33)
Li(11)a	3.426	(32)

and Fig. 1 (detail)). An analogous kind of distortion of NbO_6 octahedra has been found in $CaNb_2O_6$ [7] with five shorter distances in the range $1.774-2.077$ Å (each with a standard deviation of ± 9 in the last digit) and one longer of 2.342 Å (± 8).

The NbO_6 octahedra of the B strings are more regular ($Nb-O$ distances ranging from 1.84 to 2.13 Å) than those of the A strings. The average $Nb-O$

Table 6. Computer programs used for the crystallographic calculations. All programs written in Fortran.

No. Program name and function. Computer.	Authors.
1 DATAP2. Lp- and absorption corrections. Preparative calculations for extinction correction according to Zachariasen's 1963-formula. IBM 360/75.	P. Coppens, L. Leiserowitz and D. Rabinovich, Rehovoth, Israel. Modified by O. Olofsson and M. Elfström, Uppsala, Sweden. Inclusion of calculations for extinction correction by B. G. Brandt and S. Åsbrink, Stockholm, Sweden. Further modifications by B. G. Brandt and A. G. Nord, Stockholm, Sweden.
2 DRF. Fourier summations and structure factor calculations. IBM 360/75.	A. Zalkin, Berkeley, USA. Modified by R. Liminga and J.-O. Lundgren, Uppsala, Sweden. Further modified by O. Lindgren, Göteborg, and by A. G. Nord and B. G. Brandt, Stockholm, Sweden.
3 LALS. Full matrix least squares refinement of positional and thermal parameters and of scale factors. IBM 360/75.	P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Los Angeles, USA. Modified by A. Zalkin, Berkeley, USA, and by J.-O. Lundgren, R. Liminga and C.-I. Brändén, Uppsala, Sweden. Further modified by O. Lindgren, Göteborg, and by B. G. Brandt, and A. G. Nord, Stockholm, Sweden.
4 DISTAN. Calculation of interatomic distances and bond angles with estimated standard deviations. IBM 360/75.	A. Zalkin, Berkeley, USA. Modified by A. G. Nord and B. G. Brandt, Stockholm, Sweden.
5 DATAPH. Lp- and absorption corrections. Preparative calculations for anisotropic extinction correction. IBM 360/75.	P. Coppens, L. Leiserowitz and D. Rabinowich, Rehovoth, Israel. Inclusion of calculations for anisotropic extinction correction by P. Coppens and W. C. Hamilton, New York, USA. Modified by I. Carlbom, Stockholm, Sweden.
6 LINUS. Full matrix least squares refinement of anisotropic extinction parameters, positional and thermal parameters, scale factors and partial occupancy factors. IBM 360/75.	W. R. Busing, K. O. Martin and H. A. Levy, Oak Ridge, USA (program ORFLS). Modified by W. C. Hamilton and J. A. Ibers, New York, USA. Further modified by I. Carlbom, Stockholm, Sweden.
7 LAZY. Calculation of sin$^2\Theta$- and *d*-values from the measured reflexion sites of a Guinier powder photograph after internal standard correction. IBM 360/75, IBM 1800.	A. G. Nord, Stockholm, Sweden.
8 SIP. Generation of steering paper tape for SIEMENS AED. IBM 360/75.	R. Norrestam, Stockholm, Sweden.
9 PIRUM. Indexing of powder photographs and least squares refinement of unit cell parameters. IBM 1800.	P.-E. Werner, Stockholm, Sweden.
10 VIP. Angle settings for three-circle diffractometers. IBM 1800.	R. Norrestam, Stockholm, Sweden.
11 SIMSA. Interpretation of output on paper tape from SIEMENS AED and evaluation of intensities. IBM 1800.	R. Norrestam, Stockholm, Sweden.

distance is, however, very nearly the same for all three types of NbO_6 octahedra (1.99 — 2.01 Å).

All the O — O distances are normal, with a minimum value of 2.57 Å, *viz.* for oxygen engaged in edge sharing between the NbO_6 octahedra of the *A* strings. The O — O distance values of the LiO_6 octahedra are slightly longer (average 2.90 Å) than those of the NbO_6 octahedra (averages 2.80, 2.81 and 2.84 Å) which reflects the fact that the Li atom is somewhat larger than Nb.

The crystal structure of $LiNb_3O_8$ represents a new type of superstructure based on the orthorhombic PbO_2 type.[8] The latter is actually a somewhat distorted version of the hypothetic "A" structure discussed by Pauling and Sturdivant.[9] This family of superstructures is essentially based on a (distorted) hexagonal close-packing of oxygen atoms with metal atoms occupying octahedral interstices so as to give rise to infinite zig-zag strings of octahedra joined by edge sharing. The stoichiometry of these compounds corresponds to an oxygen to metal ratio of two. Superstructures may be formed if two or more kinds of metal atoms are present in the compound. The resulting structural details depend on the way the metal atoms are distributed among different zig-zag strings, and within the strings of octahedra.

Another member of this structural family, which has been reported recently, is $LiFeW_2O_8$.[10] The zig-zag strings of this compound contain either exclusively WO_6 octahedra or LiO_6 and FeO_6 octahedra alternating in the same string. The Li — O mean distance of 2.13 Å is rather close to the value found in the present investigation (2.19 Å).

A discussion of this family of crystal structures will shortly be presented elsewhere.

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