

Fig. 2. A stereo view illustrating some features of the packing. Each of the five hydrogen bonds of the structure is shown once, as an arrow pointing from donor to acceptor. The b axis is directed out of the paper towards the viewer. Some of the oxygen and nitrogen atoms are numbered, to indicate the orientation of the individual molecules.

drogen bonds while the hydroxyl oxygen, O(1), accepts none. There is an intramolecular hydrogen bond in this structure which has not been reported in other galactopyranose structures. The C(6)–O(6) bond is rotated about the C(5)–C(6) bond to a position in which there are two unfavorable *gauche* interactions with adjacent ring bonds; however, this location allows the formation of a hydrogen bond between O(6) and the neighboring axial hydroxyl oxygen, O(4). The intermolecular hydrogen bonds connect the molecules in layers which extend parallel to the ab plane; no hydrogen bonds link these layers. A portion of this network is shown in Fig. 2.

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Lanthanum Tetraboride

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Abstract. LaB_4 , tetragonal, $P4/mbm$, $a=7.324(1)$, $c=4.181(1)$ Å, $Z=4$, $D_x=5.396$ g cm $^{-3}$. The crystals were prepared from a molten lanthanum metal flux. The structure, homotypic with that of CeB_4 , ThB_4 and UB_4 , was refined by the least-squares method based on 520 X-ray diffraction intensities. The B–B distances range from 1.75 to 1.85 Å, the La–B distances from 2.818 to 3.155 Å.

Introduction. The crystals were prepared according to the method described by Deacon & Hiscocks (1971). The unit-cell dimensions were obtained by the least-squares method based on the 2θ angles measured on a four-circle diffractometer (Rigaku) using monochromated Mo $K\alpha$ radiation ($\lambda=0.70926$ Å); they agree with the values reported by Fisk, Cooper,

Schmidt & Castellano (1972). The intensities were collected on the same diffractometer. In the range $2\theta \leq 90^\circ$, 2147 reflexions were measured of which 1863 were greater than zero. The specimen used was prismatic, $0.13 \times 0.10 \times 0.24$ mm in size and bounded predominantly by $\{110\}$. The observed intensities were corrected for absorption ($\mu=190$ cm $^{-1}$ for Mo $K\alpha$ radiation) and then reduced to a set of 520 independent reflexions by averaging the equivalent ones.

Evidently LaB_4 is isostructural with CeB_4 , ThB_4 and UB_4 ; the systematic absences $h0l$ with $h \neq 2n$ agree with those expected for the space group $P4/mbm$ in which these borides crystallize. The coordinates of the lanthanum atom were therefore taken from their structural data and refined by the full-matrix least-squares method with the computer program *ORFLS*

Table 1. Atomic coordinates and thermal parameters

The anisotropic temperature factor for the La atom was $\exp[-\{(h^2+k^2)\beta_{11}+l^2\beta_{33}+2hk\beta_{12}\}\times 10^{-5}]$.

	Position	x	y	z	B
La	4(g)	0.31661 (6)	$x+\frac{1}{2}$	0	—
B(1)	4(e)	0	0	0.2088 (30)	0.32 (12)
B(2)	4(h)	0.0884 (12)	$x+\frac{1}{2}$	$\frac{1}{2}$	0.41 (13)
B(3)	8(j)	0.1743 (12)	0.0394 (11)	$\frac{1}{2}$	0.29 (8)
		β_{11}	β_{33}	β_{12}	
La		67 (6)	500 (27)	25 (7)	

(Busing, Martin & Levy, 1962). By means of a difference synthesis the boron atoms were found in the positions determined by Zalkin & Templeton (1953) from spatial considerations for the above-mentioned tetraborides. In further least-squares refinement of the structure, the temperature factor of the lanthanum atom only was assumed to be anisotropic and the extinction correction of Zachariasen (1968) was applied. In practice the following simplified formula with \bar{g} as one of the parameters to be refined was used:

$$F_o = sF_c[1 + (\bar{g}/\sin 2\theta)(1 + \cos^4 2\theta) \times (1 + \cos^2 2\theta)^{-1} F_2^2]^{-1/4};$$

$$\bar{g} = 0.73 \times 10^{-4}.$$

The partial polarization of the incident X-rays through the monochromator was neglected. The reflexions $00l$ with small $|l|$ were still strongly affected by extinction even after this correction. An attempt to introduce an anisotropic extinction correction was not successful.

The final value of the conventional R was 0.035 based on the 520 reflexions;* the zero-reflexions were not used for the refinement. The atomic scattering factors were taken from Hanson, Herman, Lea & Skillman (1964).

Discussion. Table 1 lists the atomic coordinates and thermal parameters. The bond lengths and bond angles were calculated by means of the program *ORFFE* (Busing, Martin & Levy, 1964) and are indicated in Fig. 1, which shows the projection of the structure on (001). The structure of LaB_4 is more closely packed than that of LaB_6 whose density is calculated to be 4.714 g cm^{-3} based on the unit-cell dimension $a = 4.156 \text{ \AA}$ (Lundström, 1969), and thus has shorter La-B distances. Hence the relatively large lanthanum atom tends to expand the three-dimensional network of boron atoms in LaB_4 ; the mean intraoctahedral B-B distance, 1.81 \AA , and the interoctahedral B-B distance, 1.75 \AA , in this compound are consequently longer than the corresponding values of 1.76 and 1.66 \AA in LaB_6 [cited by Kunmann (1971)].

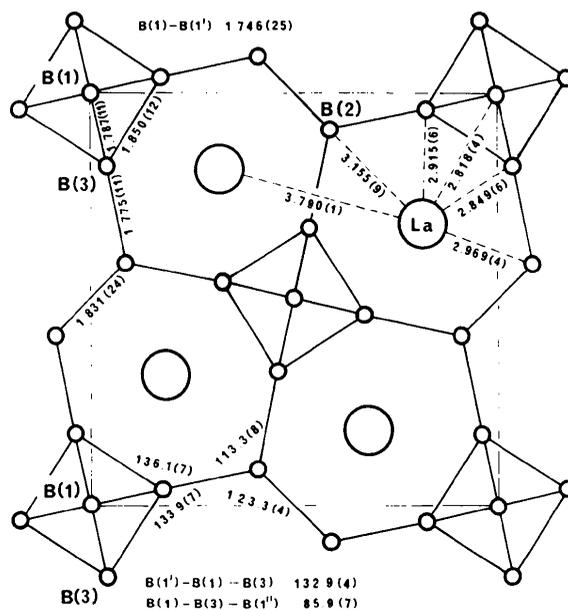


Fig. 1. Bond lengths (\AA) and bond angles ($^\circ$) indicated in the projection of the structure on (001).

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* A list of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30629 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.