Table 3. *Hydrogen-bond parameters* (Å, °)

D—H···*A*	*D*—H···*A*	*D*···*A*	H···*A*	*D*—H
N(1)—H(N11)···O(8^{i})	170 (3)	2.849 (3)	2.04 (3)	0.82 (3)
N(1)—H(N12)···O(1)	167 (3)	2.977 (3)	2.10 (3)	0.89 (3)
N(1)—H(N13)···O(6^{ii})	166 (3)	3.143 (3)	2.30 (3)	0.86 (3)
N(2)—H(N21)···O(3)	173 (3)	2.949 (3)	2.14 (3)	0.81 (3)
N(2)—H(N22)···O(2^{i})	154 (2)	3.108 (3)	2.33 (3)	0.84 (3)
N(3)—H(N31)···O(3^{i})	169 (2)	2.952 (3)	2.02 (3)	0.94 (3)
N(3)—H(N32)···O(1)	172 (3)	3.026 (3)	2.20 (3)	0.84 (3)
N(3)—H(N33)···O(2^{ii})	169 (3)	3.163 (3)	2.36 (3)	0.81 (3)
N(4)—H(N41)···O(6^{i})	161 (3)	2.995 (3)	2.13 (3)	0.90 (3)
N(4)—H(N43)···O(9^{iii})	171 (3)	3.148 (3)	2.30 (3)	0.86 (3)
N(5)—H(N51)···O(11)	160 (2)	2.985 (3)	2.10 (3)	0.92 (3)
N(5)—H(N52)···O(2^{i})	167 (3)	2.946 (2)	2.16 (3)	0.80 (3)
N(5)—H(N53)···O(9^{i})	173 (2)	3.156 (3)	2.31 (3)	0.85 (3)
O(10)—H(O101)···O(1^{ii})	176 (3)	2.825 (3)	1.77 (3)	1.06 (3)
O(9)—H(O91)···O(7^{iv})	164 (3)	2.768 (3)	1.98 (4)	0.81 (3)
O(12)—H(O121)···Cl	172 (3)	3.403 (2)	2.35 (4)	1.06 (4)
O(10)—H(O102)···O(4^{v})	165 (3)	2.803 (3)	1.77 (3)	1.05 (3)
O(11)—H(O111)···O(4^{v})	151 (4)	2.834 (3)	2.16 (4)	0.75 (4)
O(12)—H(O122)···O(7^{iii})	150 (3)	2.815 (3)	1.88 (4)	1.02 (4)
O(9)—H(O92)···O(6^{vi})	160 (4)	2.863 (3)	2.21 (4)	0.68 (3)
O(11)—H(O112)···O(2^{ii})	170 (5)	2.812 (3)	2.19 (4)	0.63 (4)

Symmetry code: (i) $1-x, 1-y, -z$; (ii) $1-x, 1-y, 1-z$; (iii) $x, y-1, z$; (iv) $x+1, y, z$; (v) $x-1, y, z$; (vi) $1-x, 2-y, 1-z$.

The $(NH_3)_5RuCl^{2+}$ cations, bisulfate counterions and lattice water molecules are linked by an extensive hydrogen-bonding network involving the chloride ion, the five ammine N atoms, the four water O atoms and seven of the eight bisulfate O atoms. Details of the hydrogen-bonding network are given in Table 3. In addition to these parameters, there are several other short non-bonding contacts in the structure involving the bisulfate O atoms, one or more of which could correspond to hydrogen bonds involving the bisulfate H atoms which were not located. The small but significant differences in Ru—N bond lengths in the title complex do not depend in any obvious way on the number or strength of the hydrogen bonds associated with a given ammonia ligand.

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A New Type of Borate Double Salt: Structure of $LiBa_2B_5O_{10}$

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Abstract. Dibarium lithium pentaborate, $LiBa_2B_5O_{10}$, $M_r = 495.67$, monoclinic, $P2_1/m$, $a = 4.413$ (2), $b = 14.585$ (9), $c = 6.700$ (4) Å, $\beta = 104.30$ (4)°, $V = 417.9$ (7) Å^3, $Z = 2$, $D_m = 3.90$ (by specific gravity bottle in toluene), $D_x = 3.94$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 94.1$ cm^{-1}, $F(000) = 440$, $T = 296$ K. Full-matrix least-squares refinement gave a final R of 0.018 and wR of 0.022, for 1015 $[I \geq 8\sigma(I)]$ observed reflections. The structure contains a new type of $[B_5O_{10}]^{5-}$ moiety composed of three BO_3 triangles and two BO_4 tetrahedra.

Introduction. The relationship between crystal structure and optical properties of nonlinear optical

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(NLO) materials, and particularly of the alkaline and alkaline-earth metal borates and double borates, has been the subject of extensive research in recent years since the discovery of the excellent NLO crystals β-BaB_2O_4 and LiB_3O_5 (Chen, Wu, Jiang & You, 1984; Chen, Wu, Jiang, Wu, You, Li & Lin, 1989), and the calculation of the macroscopic second-harmonic generation (SHG) coefficients of the various B—O anionic groups. The new compound $LiBa_2B_5O_{10}$ was found in the system $Li_2B_2O_4$–BaB_2O_4 (Huang, Wang & Liang, 1984) and its structure is described herein.

Table 1. *Atom coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Ba	0.33625 (5)	0.08851 (2)	0.69725 (4)	0.680 (3)
O1	0.7206 (6)	0.0862 (2)	1.1024 (4)	0.68 (4)
O2	−0.1688 (7)	−0.0124 (2)	0.6912 (5)	0.87 (4)
O3	0.6201 (9)	0.2500	0.0819 (6)	0.58 (6)
O4	0.6680 (7)	0.1676 (2)	0.4037 (4)	0.89 (5)
O5	−0.2200 (6)	−0.1451 (2)	0.8950 (4)	0.85 (5)
O6	−0.049 (1)	0.2500	0.6953 (7)	1.36 (8)
B1	−0.0479 (9)	−0.0790 (3)	0.8218 (6)	0.59 (6)
B2	0.769 (1)	0.2500	0.5014 (9)	0.8 (1)
B3	0.5592 (9)	0.1635 (3)	1.1772 (6)	0.50 (6)
Li	−0.013 (2)	0.2500	0.977 (2)	1.0 (2)

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Ba—O1*A*	2.823 (2)	Li—O5	1.924 (6)
Ba—O1*A′*	2.919 (3)	B1—O1*A*	1.412 (5)
Ba—O2	2.663 (3)	B1—O2	1.328 (5)
Ba—O2*B*	2.642 (3)	B1—O5	1.390 (5)
Ba—O2*A*	2.757 (4)	B2—O4	1.387 (5)
Ba—O4	2.961 (3)	B2—O6	1.350 (7)
Ba—O6	2.902 (3)	B3—O1	1.485 (5)
Ba—O5	3.017 (4)	B3—O3	1.469 (5)
Li—O3	1.92 (1)	B3—O4	1.475 (5)
Li—O6	1.86 (1)	B3—O5	1.480 (5)
O1—Ba—O1	69.32 (8)	O2—Ba—O5	119.50 (8)
O1—Ba—O2	50.07 (8)	O2—Ba—O5	154.0 (1)
O1—Ba—O2	71.71 (9)	O4—Ba—O6	93.8 (2)
O1—Ba—O2	72.87 (9)	O4—Ba—O5	135.86 (8)
O1—Ba—O2	92.82 (8)	O6—Ba—O5	63.4 (2)
O1—Ba—O2	108.04 (9)	O1—B1—O2	120.0 (4)
O1—Ba—O2	149.30 (9)	O1—B1—O5	114.9 (3)
O1—Ba—O4	111.42 (8)	O2—B1—O5	125.0 (3)
O1—Ba—O4	139.32 (8)	O4—B2—O4	119.9 (4)
O1—Ba—O6	103.1 (2)	O4—B2—O6	120.0 (2)
O1—Ba—O6	126.4 (2)	O1—B3—O3	110.6 (3)
O1—Ba—O5	47.84 (8)	O1—B3—O4	108.5 (3)
O1—Ba—O5	76.66 (8)	O1—B3—O5	106.4 (3)
O2—Ba—O2	74.6 (1)	O3—B3—O4	111.3 (3)
O2—Ba—O2	112.58 (9)	O3—B3—O5	107.0 (3)
O2—Ba—O4	65.75 (9)	O4—B3—O5	112.9 (3)
O2—Ba—O4	70.16 (9)	O3—Li—O6	120.5 (5)
O2—Ba—O4	139.00 (9)	O3—Li—O5	104.2 (4)
O2—Ba—O6	87.9 (2)	O6—Li—O5	110.7 (4)
O2—Ba—O6	107.6 (2)	O5—Li—O5	105.4 (4)
O2—Ba—O6	159.6 (2)	Ba—Li—Ba	82.1 (2)
O2—Ba—O5	80.56 (9)		

Experimental. According to the phase diagram for $Li_2B_2O_4$–BaB_2O_4 (Huang, Wang & Liang, 1984), the crystals of the title compound were obtained by thoroughly mixing $BaCO_3$, H_3BO_3 and Li_2CO_3 (analytically pure) in the appropriate ratio (corresponding to $0.8BaB_2O_4 + 0.2Li_2B_2O_4$). The mixture was then heated in a Pt crucible at 1253 K (completely melted) and the melt cooled slowly at a rate of 3 K d^{-1} from 1173 to 1143 K. Some clear needle-shaped crystals about 0.2 × 0.3 × 5.0 mm in size were grown on a Pt wire which was placed on the surface of the melt during the growth. A needle-shaped crystal with an average diameter of about 0.1 mm and a length of 0.45 mm was selected for X-ray diffraction and randomly mounted on a glass fibre. Data were collected at 296 K on a Rigaku AFC-5*R* single-crystal diffractometer with graphite-monochromated Mo *Kα* radiation ($\lambda = 0.71069$ Å). The lattice constants were obtained by the least-squares refinement of the setting angles of 20 reflections with $11 < 2\theta < 20°$. A total of 3797 reflections (index ranges $-7 \le h \le 7$, $0 \le k \le 24$, $-11 \le l \le 11$) were obtained by using the ω–2θ scan technique in the range $3 \le 2\theta \le 70°$, with scan speeds from 2 to 7° min^{-1} and scan width $(1.155 + 0.35\tan\theta)°$. These reduced to 1912 independent reflections with $R_{int} = 0.018$. Three standard reflections were measured during data collection and only random deviations were observed. The data were corrected for Lorentz–polarization effects and an empirical absorption correction was made from ψ scans (maximum transmission 1.0000 and minimum 0.9666, with average value of 0.9862). Only 1015 reflections with $I \ge 8\sigma(I)$ were used for the structure analysis.

Ba-atom positions were obtained by direct methods and the remaining atoms from a difference Fourier synthesis. The structure was refined by full-matrix least squares and a further correction for absorption effects was made using *DIFABS* (Walker & Stuart, 1983) (coefficients 0.96–1.071, with average of 1.002). Isotropic correction for secondary extinction was required (Zachariasen, 1963) with a final value of $x = 9 \times 10^{-7}$. A total of 89 parameters, with all atoms with anisotropic temperature factors, were refined. Final $R = 0.018$, $wR = 0.022$, $S = 1.005$, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{max} = 0.00$, maximum and minimum heights in final difference Fourier synthesis 0.6 and −1.98 e Å^{-3} respectively. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). All calculations (Frenz, 1978) were performed on a VAX 785 computer.

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Relevant interatomic distances and angles are given in Table 2. A drawing of the $[B_5O_{10}]^{5-}$ moiety, and

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* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54856 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0114]

drawings of the coordination around the Ba and Li cations are shown in Fig. 1. Fig. 2 shows the overall molecular packing.

The crystal structure is comprised of Li and Ba cations, and $[B_5O_{10}]^{5-}$ anions of a type not previously reported. The anion is bisected by the crystallographic mirror plane and atoms B2, O3 and O6 lie on that plane. One triangular BO_3 group at B2 and two tetrahedra at B3 share corners to give a six-membered ring with alternating B and O atoms. Two further triangular BO_3 groups involving B1 are attached to the tetrahedra through O5. In addition, atoms O1 are each shared by one BO_3 (B1) and one BO_4 (B3) and linked to the next unit cell to give a polymeric structure parallel to the *a* axis of the unit cell. The six-membered rings and chain-type packing are similar to features observed in BaB_2O_4 (Lu, Ho & Huang, 1982; Fröhlich, 1984) and $LiBO_2$ (Zachariasen, 1964).

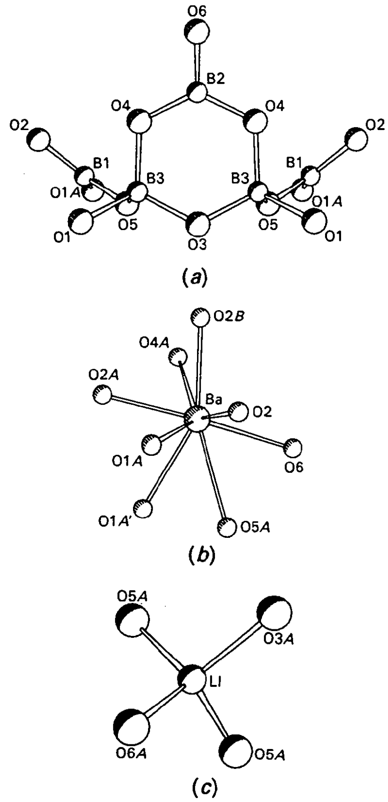

Fig. 1. Drawings of (*a*) the $[B_5O_{10}]^{5-}$ ring, (*b*) the coordination around the Ba cations, and (*c*) the coordination around the Li cations.

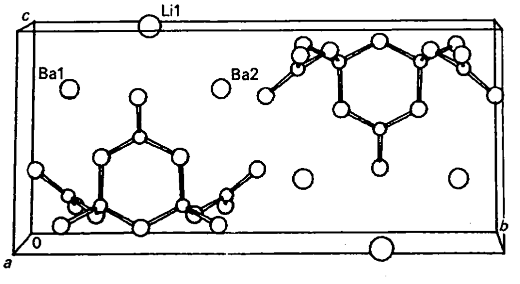

Fig. 2. A view of the molecular packing of $LiBa_2B_5O_{10}$.

The Ba cation is eight-coordinated in an irregular polyhedron: Ba—O distances vary from 2.642 (3) to 3.017 (3) Å. On the other hand, the Li cations lie on the crystallographic mirror planes with a distorted tetrahedral coordination: Li—O distances vary from 1.86 (1) to 1.92 (1) Å. These cation environments are similar to those found in other borate salts.

The novel anion found in the present compound suggests that further novel B—O ring and chain structures can be synthesized by suitable combinations of various borates.

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