amplitudes of the Si—O framework resulted in these peaks being 'refined out'. A possible additional factor causing the mismatch may be residual preferred orientation in the sample, although great care was taken to eliminate it as far as possible. To establish the structural parameters of this novel zeolite with greater accuracy Rietveld analysis will be undertaken using X-ray data and neutron data, the latter in conjunction with Dr A. K. Cheetham (Oxford).

Theta-l is the first reported example of the new, topologically distinct, structure type designated TON in accordance with IUPAC recommendations (Barrer, 1978). The unidimensional 10-*T*-ring channel system, unique for a high-silica zeolite, and the lack of channel intersections and associated 'cage' volumes (Derouane, 1980) should give theta-1 valuable structure-specific properties.

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Structure of Calcium Sodium Pentaborate

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Abstract. $CaNaB_5O_9$, $M_r = 261\cdot1$, monoclinic, $P2_1/c$, $a = 6\cdot463$ (5), $b = 13\cdot932$ (11), $c = 7\cdot858$ (6) Å, $\beta = 109\cdot55$ (5)°, $V = 666\cdot8$ Å^3, $Z = 4$, $D_x = 2\cdot601$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot009$ mm^{-1}, $F(000) = 512\cdot0$, room temperature, final $R = 0\cdot064$ for 594 unique observed reflections; crystal selected from synthetic material. The structure consists of complex metaborate sheets with a B_5O_9 building block. The B_5O_9 unit contains BO_4 and BO_3 groups in the ratio 2:3 in two rings. Na and Ca are partially ordered in sites in channels between the metaborate sheets.

Introduction. The preparation of $CaNaB_5O_9$ was first described in the course of investigations of the fluxing action of boric oxide in ceramic bodies (Mellor, 1980). Powder X-ray data reported for the phase are sufficient to equate it with the phase identified by Lawson (1981) as $Na_2O.CaO.4B_2O_3$. Lawson studied phase relations in the system Na_2O–CaO–B_2O_3, and his liquidus phase equilibrium data disclosed compositional areas suitable for crystal growth of this phase from the melt. It melts incongruently, so crystals were grown from mixes containing an excess of Na_2O and B_2O_3. The melts were cooled very slowly: this, combined with some evaporation of Na_2O and B_2O_3, enabled relatively large (*ca* 0·5 mm) crystals to be grown. The remaining melt persisted as glass and crystals were hand-picked under a petrographic microscope from the crushed mixture.

Experimental. Equant crystal ~0·2 mm diameter; Nicolet *P*3 diffractometer with Mo *Kα* radiation and $\omega/2\theta$ scan method; cell refined from 14 reflections with $2\theta > 20°$; no absorption correction applied; $\sin\theta/\lambda \le 0\cdot481$ Å^{-1}; indices in ranges $0 \le h \le 6$, $0 \le k \le 13$, $0 \le |l| \le 7$; no significant variation in intensities of standard reflections 161 and 014; 608 unique reflections, 14 classed as unobserved $[I < 2\sigma(I)]$ and not used in any of the calculations. Structure solved using *MULTAN*80 (Main *et al.*, 1980) and refined by block-diagonal least squares to minimize $\sum w(|F_o| - |F_c|)^2$ where w was calculated as $\{1 + [(|F_o| - 36)/17]^2\}^{-1}$. In refinement Ca and Na treated as vibrating anisotropically, O isotropically and B as fixed isotropic. $R = 0\cdot064$, $wR = 0\cdot070$. At end of refinement max. Δ/σ 0·07. Max. $\Delta\rho$ in final difference map $<0{\cdot}7\ \mathrm{e}\ \mathrm{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed on the Honeywell 66/80 of the Computing Centre of the University of Aberdeen using *NRC* programs (Ahmed, Hall, Pippy & Huber, 1966). Atomic parameters and selected bond lengths and angles are given in Tables 1 and 2.*

Table 1. *Atomic parameters of* $\mathrm{CaNaB_5O_9}$

	x	y	z	$B_{eq}/B_{iso}(\mathrm{Å}^2)$
$\mathrm{Ca_{0.83}Na_{0.17}}$	0·0950 (4)	0·2379 (2)	0·5695 (3)	1·354*
$\mathrm{Na_{0.83}Ca_{0.17}}$	0·4738 (6)	0·5892 (3)	0·6632 (5)	0·929*
O(1)	0·8382 (9)	0·6516 (5)	0·6448 (8)	1·7 (1)
O(2)	1·1326 (9)	0·5392 (4)	0·7018 (7)	0·7 (1)
O(3)	1·4383 (9)	0·4427 (4)	0·8059 (7)	0·6 (1)
O(4)	1·0881 (9)	0·3775 (4)	0·7842 (7)	1·0 (1)
O(5)	0·7710 (9)	0·4842 (4)	0·6594 (8)	1·0 (1)
O(6)	0·7986 (9)	0·3712 (4)	0·9039 (7)	0·6 (1)
O(7)	0·7458 (9)	0·3138 (4)	0·6024 (7)	0·9 (1)
O(8)	0·5257 (9)	0·1696 (4)	0·5445 (7)	0·8 (1)
O(9)	0·4831 (9)	0·2766 (4)	0·7488 (8)	0·7 (1)
B(1)	0·9110 (17)	0·5610 (8)	0·6705 (14)	0·229 (2)
B(2)	1·2213 (18)	0·4514 (8)	0·7692 (14)	0·229 (2)
B(3)	0·8459 (18)	0·3900 (8)	0·7410 (14)	0·229 (2)
B(4)	0·5906 (17)	0·2543 (8)	0·6296 (15)	0·229 (2)
B(5)	0·5666 (17)	0·1426 (8)	0·3790 (14)	0·229 (2)

* Calculated for the predominant ion from the corresponding anisotropic thermal vibration parameters where the correction is of the form $\exp\,[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{23}kl + B_{12}hk + B_{13}hl)]$ as $B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + ac\cos\beta B_{13})$.

Table 2. *Bond lengths* (Å) *and angles* (°) *for the anion* $(\mathrm{B_5O_9})_n^{3n-}$

Average e.s.d. for B–O distance is 0·012 Å and for bond angles 0·8°.

B(1)–O(1)	1·34	O(1)–B(1)–O(2)	121
O(2)	1·40	O(5)	122
O(5)	1·39	O(2)–B(1)–O(5)	117
B(2)–O(2)	1·38	O(2)–B(2)–O(3)	116
O(3)	1·34	O(4)	120
O(4)	1·37	O(3)–B(2)–O(4)	124
B(3)–O(4)	1·50	O(4)–B(3)–O(5)	112
O(5)	1·47	O(6)	107
O(6)	1·44	O(7)	104
O(7)	1·50	O(5)–B(3)–O(6)	114
		O(7)	108
		O(6)–B(3)–O(7)	110
B(4)–O(7)	1·37	O(7)–B(4)–O(8)	125
O(8)	1·35	O(9)	122
O(9)	1·38	O(8)–B(4)–O(9)	113
B(5)–O(3)	1·45	O(3)–B(5)–O(6)	112
O(6)	1·46	O(8)	108
O(8)	1·46	O(9)	109
O(9)	1·50	O(6)–B(5)–O(8)	113
		O(9)	108
		O(8)–B(5)–O(9)	106
B(1)–O(2)–B(2)	122	B(3)–O(6)–B(5)	115
B(2)–O(3)–B(5)	125	B(3)–O(7)–B(4)	118
B(2)–O(4)–B(3)	122	B(4)–O(8)–B(5)	123
B(1)–O(5)–B(3)	123	B(4)–O(9)–B(5)	119

Discussion. The structure is built of a complex sheet of B and O atoms. The basic building block of the sheet is a $\mathrm{B_5O_9}^{3-}$ unit. Its structure is shown in Fig. 1 as unit (I): it consists of two rings composed of $\mathrm{BO_3}$ triangular units but linked by a $\mathrm{BO_4}$ tetrahedral unit. This unit propagates by translation along the *a* axis of the structure by sharing of O between B(2)–B(5′). Infinite chains are thus produced extending in the **a** direction. These chains are shown in Figs. 2 and 3: they lie close to the **c**-glide directions and propagate along the **c** direction by sharing of O atoms between B(4)–B(5′).

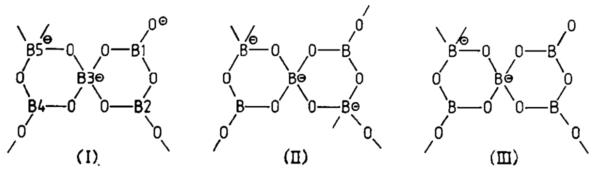

Fig. 1. Structure of some borate polyanions.

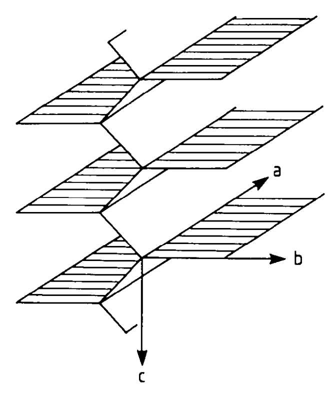

Fig. 2. Sheet structure development of the boron–oxygen portions of $\mathrm{CaNaB_5O_9}$.

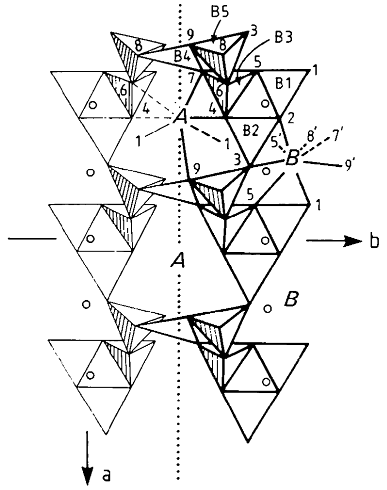

Fig. 3. Portions of the $\mathrm{CaNaB_5O_9}$ structure showing the relationship of cation sites to boron–oxygen sheets.

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* Lists of structure factors and vibrational parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42321 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

As a result of this sharing, thick anionic sheets $(B_5O_9)_n^{3n-}$ are produced. All O atoms in the polyanion except O(1), which is part of the triangular unit about B(1), are bonded to two B atoms: O(1) is bonded to one B. The separate anion sheets corresponding to contiguous glide planes, which are symmetry-related by the operation of the centre of symmetry lying between sheets, do not share any O atoms. This anion unit appears to be a new type. The basic unit shown in Fig. 1, (I), is, however, similar to the borate anions in $Ca_2B_5O_9Br$ (Lloyd, Levasseur & Fouassier, 1973) and in $Na_2B_4O_7$ (Krogh-Moe, 1974), shown in Fig. 1 as units (II) and (III) respectively.

The structure contains two sets of cation sites for *M* ions (Fig. 3). Those of one type (*A*) lie in the middle of sheets, while those of the other (*B*) lie between sheets, close to centres of symmetry. Positions *A* can be regarded, together with associated O atoms, as forming channels parallel to the *c* axis: each cation is coordinated to seven O atoms, five belonging to the sheet which nearly envelops it and two underbonded O(1) atoms of adjacent sheets. These cations are separated in the **c** direction by 3·94 Å (~*c*/2), sharing the atoms O(1) and O(4) between two *M* atoms and so forming polyhedral chains sharing edges $(MO_5)_n$. The distances *M*(*A*)—O range from 2·43 to 2·75 Å, averaging 2·53 Å. The occupancy of position *A* is $Ca^{2+}{}_{0.83}Na^{+}{}_{0.17}$ while that of position *B* is complementary: $Na^{+}_{0.83}Ca^{2+}_{0.17}$. Positions *B* form centrosymmetric dimers, M_2O_{14}, where $M—M' = 3·67$ Å in which each cation is coordinated to eight O atoms, two of which [O(5) and O(5′)] are shared. The *M*(*B*)—O distances range from 2·38 to 2·70 Å, with an average of 2·53 Å. The mean sizes of the holes at *A* and *B* are virtually identical, in accord with the similar sizes of Na and Ca (Vainshtein, Fridlein & Indentov, 1982).

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Structure of Calcium Trisodium Pentaborate

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Abstract. $CaNa_3B_5O_{10}$, $M_r = 323·1$, triclinic, $P\bar{1}$, $a = 7·460$ (3), $b = 7·445$ (4), $c = 11·124$ (6) Å, $\alpha = 120·58$ (4), $\beta = 61·94$ (3), $\gamma = 120·17$ (3)°, $V = 440·3$ Å^3, $Z = 2$, $D_x = 2·437$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·879$ mm^{-1}, $F(000) = 316$, room temperature, final $R = 0·069$ for 2487 unique observed reflections; crystal selected from synthetic material. The structure contains discrete $(B_5O_{10})^{5-}$ anions in the form of a pair of six-membered rings. Each ring contains two triangular BO_3 units. The rings are connected by a shared tetrahedral BO_4 unit.

Introduction. In the course of phase equilibrium studies of the system $Na_2O—CaO—B_2O_3$, three ternary phases were encountered (Lawson, 1981). The synthesis of two of these phases, $CaNaB_5O_9$ and $CaNaBO_3$, had been reported in the literature and the structure of one, $CaNaB_5O_9$, has been determined (Fayos, Howie & Glasser, 1985). However, the composition of a third phase was not definitely established as a result of the phase studies: the mole ratio $3Na_2O.6CaO.11B_2O_3$ was suggested. In order to resolve these doubts as well as to understand the principles of borate polyanion formation