Table 2. *Interatomic distances* (Å) *and angles* (°)

Cl–S(1)	2·218 (2)	N(3)–C(1)	1·337 (4)
S(1)–N(1)	1·579 (4)	N(4)–C(1)	1·323 (4)
S(1)–N(2)	1·575 (4)	C(1)–C(2)	1·506 (5)
S(2)–N(1)	1·636 (4)	C(2)–C(3)	1·508 (7)
S(2)–N(4)	1·605 (3)	C(2)–C(4)	1·527 (6)
S(3)–N(2)	1·633 (3)	C(2)–C(5)	1·501 (7)
S(3)–N(3)	1·596 (3)	S(2)–S(3)	2·378 (2)
Cl–S(1)–N(1)	103·7 (1)	N(3)–C(1)–N(4)	123·6 (3)
Cl–S(1)–N(2)	105·2 (2)	N(3)–C(1)–C(2)	117·1 (3)
N(1)–S(1)–N(2)	109·0 (2)	N(4)–C(1)–C(2)	119·1 (3)
N(1)–S(2)–N(4)	112·7 (2)	C(1)–C(2)–C(3)	110·9 (4)
N(2)–S(3)–N(3)	112·5 (2)	C(1)–C(2)–C(4)	106·1 (3)
S(1)–N(1)–S(2)	118·9 (2)	C(1)–C(2)–C(5)	110·9 (4)
S(1)–N(2)–S(3)	119·6 (2)	C(3)–C(2)–C(4)	109·2 (4)
S(3)–N(3)–C(1)	118·3 (3)	C(3)–C(2)–C(5)	110·3 (5)
S(2)–N(4)–C(1)	119·3 (3)	C(4)–C(2)–C(5)	109·3 (5)

Related literature. The molecular shape of ^{t}BuC(NSN)$_2$SCl may be compared to that of $(CH_3)_2NC(NSN)_2CN(CH_3)_2$ (Ernest, Holick, Rihs, Schomburg, Shoham, Wenkert & Woodward, 1981), with d(S–S) = 2·482 Å, and that of $S_4N_4Cl_2$, d(S–S) = 2·484 (1) Å (Zak, 1981). Similar folded structures are found for the following: $R_2P(NSN)_2PR_2$, R = Me, d(S–S) = 2·551 (2) Å (Burford, Chivers, Codding & Oakley, 1982); R = Ph, d(S–S) = 2·528 (1) Å (Burford, Chivers & Richardson, 1983); and $Ph_3PN(NSN)_2NPPh_3$, d(S–S) = 2·452 (2) Å (Bojes, Chivers, Cordes, Maclean & Oakley, 1981). For comparison, d(S–S) in S_4N_4 is 2·58 Å (Sharma & Donohue, 1963; De Lucia & Coppens, 1978.)

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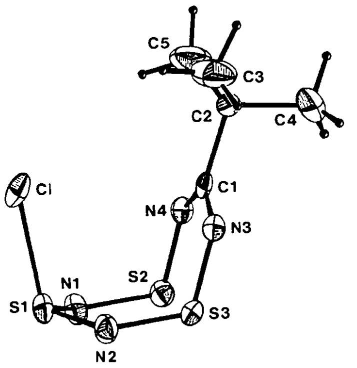

Fig. 1. *ORTEP*II illustration (Johnson, 1976) of ^{t}BuC(NSN)$_2$SCl.

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Structure of Ethyl {(1*S*,5*R*,8*R*)-8-Hydroxy-3-oxo-2-oxa-6-azabicyclo[3.3.0]oct-6-yl}acetate Hydrobromide

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Abstract. $C_{10}H_{16}NO_5^+.Br^-$, M_r = 310·15, orthorhombic, $P2_12_12_1$, a = 5·5495 (6), b = 12·3923 (15), c = 18·8252 (21) Å, V = 1294·6 (3) Å^3, Z = 4, D_x = 1·591 Mg m^{-3}, Mo $K\alpha$, λ = 0·7107 Å, μ = 3·372 mm^{-1}, $F(000)$ = 632, T = 294 K, final R = 0·047 for 1100 reflections. The hydrobromide of an intermediate in the enantioselective synthesis of the pyrrolizidine-triol crotanecine was examined to confirm its stereochemistry. There are no unusual bond distances or angles.

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Experimental. Colourless flat needles (cut), 0·1 × 0·2 × 0·4 mm; Enraf–Nonius CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares refinement of 25 reflections with $10{\cdot}4 \le \theta \le 19{\cdot}3°$; space group uniquely determined from absences; 1355 unique reflections collected, 750 considered observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics]; $\theta_{\max} = 25°$, $\omega/2\theta$ scans, scan range $1{\cdot}5\ (0{\cdot}60 + 0{\cdot}347\tan\theta)°$, scan speed 3·4 ranging to 0·74° min^{-1}; three standard reflections ($06\bar{3}$, $12\bar{5}$, $21\bar{1}$) measured every 1500 s of X-ray exposure time, max. variation 0·6%; data collected: $+h$ $+k$ $+l$ to max. indices of 6 14 22; data corrected for background, Lp, and absorption (Walker & Stuart, 1983), min. and max. absorption coefficients 0·64 and 1·28. Structure solved using Patterson function and difference Fourier maps, refined by full-matrix least squares based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$; w defined as $[\sigma^2(F_o) + 0{\cdot}00004(F_o^2)]^{-1}$; *XRAY76* system of computer programs (Stewart, 1976); H atoms included in calculated positions with isotropic thermal parameters set to 1·1 times $B_{\rm eq}$ of the bonded atom; H-atom parameters not refined; all non-H atoms refined with anisotropic thermal parameters. Model converged with: 1100 reflections [observed plus 350 for which $I_c > 3\sigma(I_o)$; 255 have $I_c < 3\sigma(I_o)$], 155 variables, $R = 0{\cdot}047$, $wR = 0{\cdot}038$, max. $(\Delta/\sigma) = 0{\cdot}045$, $S = 1{\cdot}03$, isotropic extinction parameter $8{\cdot}5\,(1{\cdot}1) \times 10^{-4}$, max. residual electron density 0·7 e Å^{-3} associated with Br. Scattering factors were those of Cromer & Mann (1968) for non-H atoms, and Stewart, Davidson & Simpson (1965) for H atoms. Anomalous-dispersion corrections included for non-H atoms (*International Tables for X-ray Crystallography,* 1974). Relative stereochemistry determined and absolute configuration fixed by known absolute configuration of starting material (Yadav, Rüeger & Benn, 1984). This is in agreement with the configuration determined for crotanecine (Richardson & Culvenor, 1985). Table 1 gives the atomic parameters and Table 2 bond lengths and angles.* Fig. 1 shows the numbering scheme, and Fig. 2 is an *ORTEP* plot.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (×10) *for the non-hydrogen atoms*

$B_{\rm eq}$ is $\frac{1}{3}$ the trace of the B_{ij} matrix.

	x	y	z	$B_{\rm eq}$(Å^2)
Br	1280·4 (18)	5214·5 (7)	2542·5 (8)	48·7 (5)
C(1)	6613 (19)	7234 (8)	3197 (5)	32 (5)
C(2)	6408 (23)	6238 (7)	3667 (5)	36 (5)
C(3)	6197 (24)	6698 (8)	4412 (6)	36 (6)
O(4)	6160 (17)	6207 (5)	4953 (4)	47 (4)
O(5)	6139 (15)	7776 (5)	4388 (3)	35 (3)
C(6)	6021 (25)	8198 (7)	3676 (6)	33 (6)
C(7)	3446 (20)	8540 (7)	3473 (5)	29 (5)
O(8)	1713 (14)	7844 (5)	3774 (4)	35 (4)
C(9)	3506 (19)	8370 (6)	2691 (5)	29 (5)
N(10)	4730 (12)	7301 (5)	2602 (5)	28 (3)
C(11)	5830 (21)	7067 (9)	1884 (5)	34 (6)
C(12)	3947 (27)	6978 (10)	1324 (6)	51 (7)
O(13)	1972 (20)	7450 (8)	1322 (5)	77 (6)
O(14)	4683 (17)	6313 (7)	821 (4)	69 (6)
C(15)	2886 (29)	6193 (12)	229 (7)	97 (11)
C(16)	3979 (40)	5603 (13)	−294 (7)	124 (13)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)–C(2)	1·52 (1)	C(7)–C(9)	1·49 (1)
C(1)–C(6)	1·53 (1)	C(9)–N(10)	1·50 (1)
C(1)–N(10)	1·53 (1)	N(10)–C(11)	1·51 (1)
C(2)–C(3)	1·52 (1)	C(11)–C(12)	1·49 (2)
C(3)–O(4)	1·19 (1)	C(12)–O(13)	1·24 (2)
C(3)–O(5)	1·34 (1)	C(12)–O(14)	1·32 (1)
O(5)–C(6)	1·44 (1)	O(14)–C(15)	1·50 (2)
C(6)–C(7)	1·54 (2)	C(15)–C(16)	1·37 (2)
C(7)–O(8)	1·41 (1)		

C(6)–C(1)–C(2)	105·8 (8)	C(6)–C(7)–C(9)	100·7 (8)
C(6)–C(1)–N(10)	104·0 (8)	O(8)–C(7)–C(9)	109·1 (7)
C(2)–C(1)–N(10)	114·6 (8)	C(7)–C(9)–N(10)	104·2 (7)
C(1)–C(2)–C(3)	103·8 (7)	C(9)–N(10)–C(11)	116·9 (7)
C(2)–C(3)–O(4)	127·0 (9)	C(9)–N(10)–C(1)	106·0 (7)
C(2)–C(3)–O(5)	110·2 (8)	C(1)–N(10)–C(11)	111·5 (7)
O(4)–C(3)–O(5)	122·8 (10)	N(10)–C(11)–C(12)	111·4 (9)
C(3)–O(5)–C(6)	113·2 (7)	C(11)–C(12)–O(13)	125·9 (11)
O(5)–C(6)–C(1)	104·8 (7)	C(11)–C(12)–O(14)	109·7 (11)
O(5)–C(6)–C(7)	111·9 (9)	O(13)–C(12)–O(14)	124·3 (12)
C(1)–C(6)–C(7)	105·5 (8)	C(12)–O(14)–C(15)	112·9 (10)
C(6)–C(7)–O(8)	111·4 (7)	O(14)–C(15)–C(16)	107·0 (14)

Hydrogen-bonding distances

N(10)···Br	3·219 (7)	H(N10)···Br	2·23

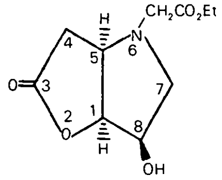

Fig. 1. Chemical numbering scheme.

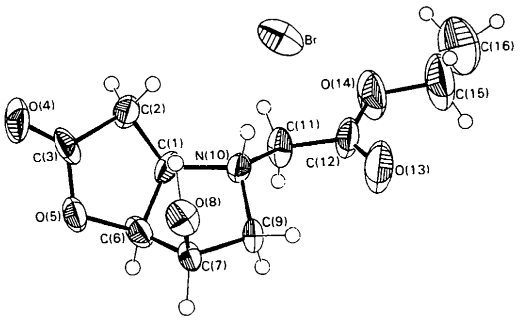

Fig. 2. *ORTEP* plot (Johnson, 1965) showing the crystallographic atomic labelling scheme. H atoms are included as spheres of radius 0·1 Å.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42392 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1985). C**41**, 1689–1690

New description of the $Ca_3(BO_3)_2$ structure.

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(*Received* 22 *February* 1985; *accepted* 29 *July* 1985)

Abstract

The structure of $Ca_3(BO_3)_2$ [and isostructural $Sr_3(BO_3)_2$] is difficult to describe using the traditional cation-centered polyhedra model. However, the structure can be described in a simple and more elegant way as anticorundum Ca_3X_2 ($X = BO_3$), by considering the alternative model of an anion-stuffed cation array [White & Hyde (1982). *Phys. Chem. Miner.* **8**, 55–63].

From a comparison of their powder patterns, $Ca_3(BO_3)_2$ and $Sr_3(BO_3)_2$ have been reported as isostructural (Richter & Müller, 1980). However, only the structure of the Ca compound has been determined from single-crystal data. It is trigonal, $R\bar{3}c$, with cell dimensions $a = 8{\cdot}6377\,(8)$, $c = 11{\cdot}849\,(2)$ Å and $Z = 6$. The structure was first solved by Schuckmann (1970), who observed a slight deviation from planarity, in the BO_3 group, which was confirmed in a redetermination carried out by Vegas, Cano & García-Blanco (1975).

In both cases, the structure was described as built up of cation-centered anion polyhedra, with eight-coordinated Ca at the center of a distorted square antiprism and BO_3 triangles, but the structure could not be related to any known structural type. It is our aim to offer an alternative and clearer description, regarding this structure as an anion-stuffed cation array, as proposed by White & Hyde (1982) and applied successfully to describe and relate the structures of the humite and leucophoenicite families (White & Hyde, 1983).

The structure, projected on the *ab* plane, is represented in Fig. 1. The cation array is clearly related to the corundum structure, *i.e.* Ca with slightly distorted h.c.p. and B occupying pairs of face-sharing octahedra. As does Al in corundum, the B atoms deviate towards the opposite faces of these octahedra. The model is represented in Fig. 2.

The structure should be stuffed with O atoms, occupying tetrahedral holes of the Ca h.c.p., but, if so, they could not bond to B atoms. So, the BO_3 groups, as a whole, are inserted into the Ca_6 octahedra and the structure is better described as anticorundum Ca_3X_2 ($X = BO_3$).

O atoms are pentacoordinated by four Ca [at $2{\cdot}347\,(1)$, $2{\cdot}431\,(1)$, $2{\cdot}440\,(1)$ and $2{\cdot}732\,(1)$ Å] and one B [$1{\cdot}384\,(1)$ Å], forming an irregular triangular bipyramid,

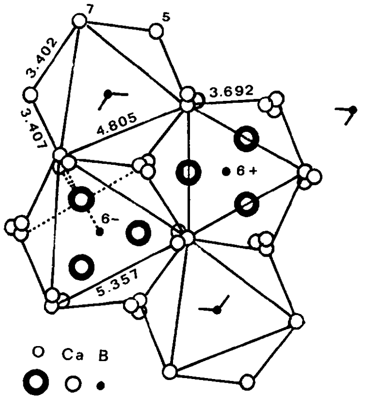

Fig. 1. Cation array of $Ca_3(BO_3)_2$ projected on the *ab* plane, showing a sheet of BCa_6 octahedra. Heights are expressed in twelfths (5 and 7 for Ca, 6+ and 6− for B). Ca—Ca distances are given in Å (e.s.d.'s ~0·001 Å).