Bis-(1,4-anhydroerythritol).NaI

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Abstract. $(C_4H_8O_3)_2$.NaI, monoclinic, space group $P2_1/n$, $a=6\cdot24$ (5), $b=21\cdot10$ (5), $c=9\cdot71$ (5) Å, $\beta=94\cdot3$ (5)°, $Z=4$, $D_m=1\cdot85$ (1), $D_x=1\cdot87$ g cm^{-3}. There is a polymeric, ladder-like network with rungs composed of pairs of 1,4-anhydroerythritol ligands connecting Na$^+$ ions. The rails of the ladder are composed of Na$^+$ ions connected by single ligands. The I$^-$ ions lie in holes along lines parallel to the rails.

Introduction. The observed 1:2 stoichiometry (Ballard, Haines, Norris & Wells, 1974) suggested the possibility of a non-polymeric structure in which the coordination sphere of Na$^+$ consists of a pair of ligands.

A crystal, $0\cdot38 \times 0\cdot15 \times 0\cdot09$ mm, was oscillated in a Lindemann tube about its longest dimension. Equi-inclination Weissenberg photographs ($0kl$ to $5kl$) were scanned by the S.R.C. Microdensitometer Service and the measured intensities scaled by visually estimated precession photograph ($hk0$) intensities. There was no correction for absorption. Cu $K\alpha$ radiation was used.

From a Patterson map the position of I$^-$ was found and all other non-hydrogen atoms were found in successive difference maps. The final $R'=\sum w^{1/2}\Delta/$

$(\sum w^{1/2}|F_o|)$ was 0·0776; R was 0·0754. Ionic and neutral scattering factors were employed (Cromer & Mann, 1968; Cromer & Liberman, 1970). The weighting scheme was $w^{-1}=863\cdot5+|F_o|+0\cdot125|F_o|^2$. Final atomic coordinates and temperature factors are given in Table 1; Table 2 gives the bond distances and angles.*

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31592 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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

Na—O(1)	2·50 (2)	O(4)–C(1)	1·42 (3)
Na—O(2)	2·37 (2)	C(2)–C(3)	1·49 (3)
Na—O(3)	2·39 (2)	O(4)–C(4)	1·42 (3)
Na—O(4)	2·38 (2)	O(5)–C(6)	1·42 (2)
Na—O(5)	2·47 (2)	O(6)–C(3)	1·44 (3)
Na—O(6)	2·38 (2)	C(1)–C(2)	1·52 (3)
O(1)–C(2)	1·43 (3)	C(3)–C(4)	1·50 (3)
O(2)–C(5)	1·39 (3)	C(5)–C(6)	1·53 (3)
O(3)–C(7)	1·42 (3)	C(5)–C(8)	1·52 (3)
O(3)–C(8)	1·40 (3)	C(6)–C(7)	1·52 (3)

O(2)–Na—O(1)	97·7 (7)
O(3)–Na—O(1)	150·3 (6)
O(3)–Na—O(2)	82·5 (7)
O(5)–Na—O(1)	97·9 (7)
O(5)–Na—O(2)	66·3 (7)
O(5)–Na—O(3)	109·0 (7)
O(6)–Na—O(1)	65·5 (7)
O(6)–Na—O(2)	116·9 (7)
O(6)–Na—O(3)	87·8 (8)
O(6)–Na—O(5)	163·1 (7)
C(2)–O(1)–Na	110·5 (12)
C(5)–O(2)–Na	117·7 (11)
C(7)–O(3)–Na	118·6 (13)
C(8)–O(3)–Na	129·6 (13)
C(8)–O(3)–C(7)	111·6 (17)
C(3)–C(2)–O(1)	114·3 (17)
C(3)–C(2)–C(1)	103·6 (17)
C(1)–C(2)–O(1)	109·4 (16)
C(2)–C(1)–O(4)	106·8 (17)
C(4)–O(4)–C(1)	108·2 (16)
C(6)–O(5)–Na	120·9 (12)
C(3)–O(6)–Na	124·1 (15)
C(2)–C(3)–O(6)	107·9 (18)
C(4)–C(3)–O(6)	113·1 (18)
C(4)–C(3)–C(2)	100·7 (18)
C(3)–C(4)–O(4)	106·7 (19)
C(6)–C(5)–O(2)	113·6 (15)
C(8)–C(5)–O(2)	110·2 (16)
C(8)–C(5)–C(6)	103·5 (16)
C(5)–C(6)–O(5)	107·9 (16)
C(7)–C(6)–O(5)	112·5 (15)
C(7)–C(6)–C(5)	100·5 (16)
C(6)–C(7)–O(3)	105·4 (18)
C(5)–C(8)–O(3)	106·0 (18)

Table 1. *Fractional coordinates* ($\times 10^4$) *and temperature factors*

	x	y	z	U
Na	1398 (11)	3705 (3)	9863 (6)	†
I	499 (2)	1228 (1)	185 (1)	†
O(1)	3688 (22)	4323 (6)	8337 (14)	261 (30)
O(2)	2865 (22)	2688 (5)	9479 (13)	197 (27)
O(3)	8239 (20)	3101 (5)	10178 (12)	183 (26)
O(4)	55 (22)	5592 (6)	8531 (13)	244 (29)
O(5)	4254 (21)	3388 (6)	11628 (13)	226 (28)
O(6)	−531 (26)	4202 (7)	7943 (16)	352 (35)
C(1)	2103 (32)	5323 (8)	8884 (20)	201 (40)
C(2)	2528 (30)	4853 (9)	7750 (19)	203 (38)
C(3)	338 (33)	4696 (8)	7125 (20)	214 (40)
C(4)	−775 (37)	5321 (9)	7259 (22)	292 (46)
C(5)	4950 (26)	2569 (7)	10026 (16)	104 (33)
C(6)	5401 (30)	2811 (7)	11503 (17)	125 (33)
C(7)	7824 (34)	2912 (9)	11541 (20)	248 (43)
C(8)	6556 (39)	2932 (10)	9219 (23)	346 (50)

† Anisotropic temperature factors (Å$^2 \times 10^3$)
$$T=\exp\left[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{13}hla^*c^*+2U_{12}hka^*b^*)\right].$$

	U_{11}	U_{22}	U_{33}
Na	103 (41)	273 (37)	106 (34)
I	255 (9)	331 (6)	103 (6)

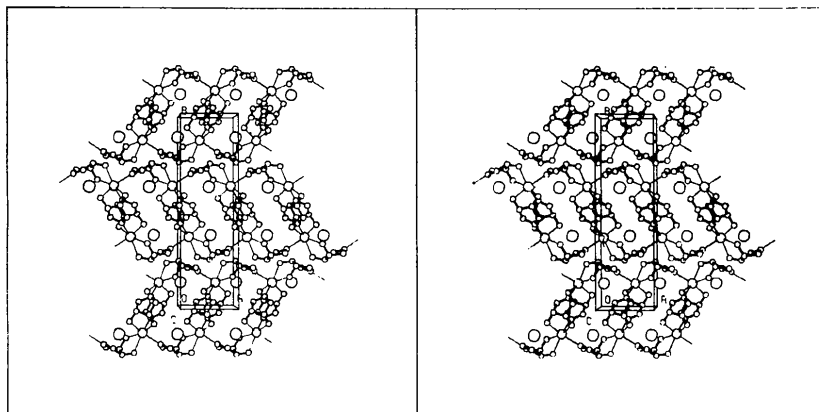|	U_{23}	U_{13}	U_{12}
Na	5 (26)	68 (29)	1 (29)
I	67 (5)	−23 (5)	−97 (7)

Fig. 1. Stereo drawing showing a view down the c axis.

Discussion. About Na$^+$ is a distorted octahedron of coordinated O atoms, four from the hydroxyl groups of two crystallographically distinct tetrahydrofuran rings and two from the ether ring O atoms of a space-group related pair of rings (Figs. 1 and 2). The structure is polymeric but, unlike the perchlorate (Ballard et al., 1974), is infinitely extended in one direction only. The rows of I$^-$ ions are parallel to those of Na$^+$, the nearest Na$\cdots$I separation being 5·38 Å compared with 3·24 Å in NaI.

A near-envelope conformation is adopted by the two crystallographically distinct tetrahydrofuran rings (which have enantiomeric counterparts by virtue of the space-group symmetry). The conformations of the two distinct rings are nearly the same. In both cases four of the ring atoms are nearly coplanar, C(4), O(4), C(1) and C(2) in one ring, C(5), C(8), O(3) and C(7) in the

Table 3. *Torsional angles of the furanoid rings*

The e.s.d. is about 1°.

O(4)–C(1)–C(2)–C(3)	−22·3°
C(1)–C(2)–C(3)–C(4)	34·5
C(2)–C(3)–C(4)–O(4)	−36·3
C(3)–C(4)–O(4)–C(1)	23·4
C(4)–O(4)–C(1)–C(2)	0·0
O(1)–C(2)–C(3)–O(6)	34·7
O(1)–C(2)–C(1)–O(4)	−144·5
O(6)–C(3)–C(4)–O(4)	78·6
O(3)–C(8)–C(5)–C(6)	24·4
C(8)–C(5)–C(6)–C(7)	−34·4
C(5)–C(6)–C(7)–O(3)	33·2
C(6)–C(7)–O(3)–C(8)	−19·6
C(7)–O(3)–C(8)–C(5)	−3·2
O(2)–C(5)–C(6)–O(5)	−35·8
O(2)–C(5)–C(8)–O(3)	146·1
O(5)–C(6)–C(7)–O(3)	−81·3

other. In each ring both hydroxyl O atoms are displaced to the same side of the ring as the remaining ring C atom, C(3) and C(6) respectively. The displace-
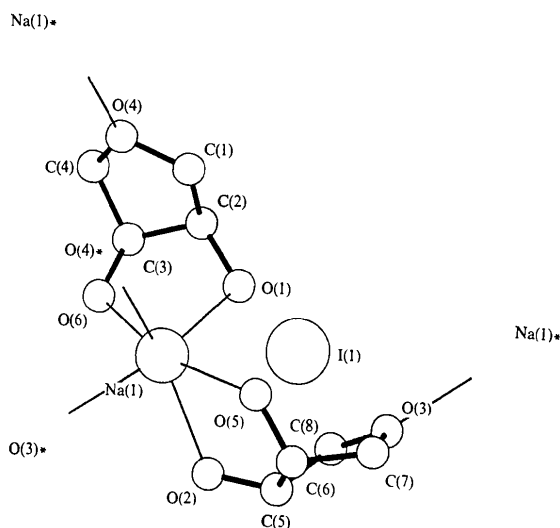

Fig. 2. The asymmetric unit showing the numbering of the atoms.

ments of the out-of-plane atoms are to that side from which the four-atom sequences in the orders given above appear clockwise. The conformation described here is different from that of the NaClO$_4$ complex (Ballard et al., 1974). Torsion angles are given in Table 3.

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References

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