Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Poly[[μ-1,4-anhydroerythritolato-di-μ-aqua-sodium(I)] monohydrate]

Tobias Kerscher, Patrick Zeller, Peter Mayer and Peter Klüfers*

Ludwig-Maximilians-Universität, Department Chemie, Butenandtstrasse 5–13, 81377 München, Germany
Correspondence e-mail: kluef@cup.uni-muenchen.de

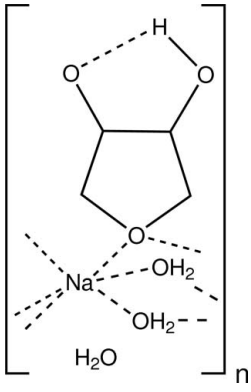
Received 31 October 2008; accepted 25 November 2008

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.086; data-to-parameter ratio = 14.4.

In the title compound, $\{[Na(C_4H_7O_3)(H_2O)_2]\cdot H_2O\}_n$, the sodium ion is octahedrally coordinated by two bridging 1,4-anhydroerythritolate ligands, unexpectedly coordinated by the ring oxygen and four water ligands. This bonding pattern leads to one-dimensional antitactical polymeric chains along [010]. One of the exocyclic O atoms of the anhydroerythritolate group is an acceptor in four hydrogen bonds, giving further evidence that it is deprotonated.

Related literature

For the neutral 1,4-anhydroerythritole as a coordination ligand on sodium with either the hydoxyl groups coordinating sodium or a mixed coordination by both the ring oxygen and the hydroxyl groups, see: Ballard *et al.* (1974, 1976). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$[Na(C_4H_7O_3)(H_2O)_2]\cdot H_2O$ M_r = 180.13
Monoclinic, $C2/c$
a = 23.155 (6) Å
b = 6.0900 (16) Å
c = 14.543 (5) Å
β = 127.678 (17)°
V = 1623.1 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 200 (2) K
0.23 × 0.20 × 0.10 mm

Data collection

Oxford Diffraction XCalibur diffractometer
Absorption correction: none
6310 measured reflections
1699 independent reflections
1088 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 0.95
1699 reflections
118 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3	0.82	1.85	2.4367 (17)	127
O4—H41···O3^{i}	0.839 (14)	1.939 (15)	2.7706 (17)	170.9 (17)
O4—H42···O3^{ii}	0.883 (14)	1.827 (14)	2.7078 (19)	174.5 (18)
O6—H61···O2^{ii}	0.877 (14)	1.940 (14)	2.813 (2)	173.1 (18)
O6—H62···O5^{iii}	0.859 (14)	1.838 (14)	2.6903 (18)	171.0 (18)
O5—H51···O3^{i}	0.842 (15)	1.825 (15)	2.6653 (18)	176 (2)
O5—H52···O2^{iv}	0.828 (14)	2.131 (15)	2.958 (2)	177 (2)

Symmetry codes: (i) $x, -y, z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x, -y, -z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

TK is indebted to the Hanns-Seidel Stiftung for a PhD grant funded by the German Bundesministerium für Bildung und Forschung.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2067).

References

Ballard, R. E., Haines, A. H., Norris, E. K. & Wells, A. G. (1974). *Acta Cryst.* B**30**, 1590–1593.
Ballard, R. E., Haines, A. H., Norris, E. H. & Wells, A. G. (1976). *Acta Cryst.* B**32**, 1577–1578.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2008). E**64**, m1637 [**doi:10.1107/S1600536808039640**]

Poly[[μ-1,4-anhydroerythritolato-di-μ-aqua-sodium(I)] monohydrate]

T. Kerscher, P. Zeller, P. Mayer and P. Klüfers

Comment

The title compound was obtained as a byproduct in a reaction involving sodium hydroxide, anhydroerytritole and iron(II) chloride. .

Sodium is octahedrically coordinated by bridging anhydroerythritolate and water ligands. The anhydroerythritolate coordinates the sodium unexpectedly by its ring oxygen atom while the alkoxide group is stabilized by intramolecular hydrogen bonding from the hydroxyl group. The anhydroerythitolate contains a five-membered ring containing O1, C1, C2, C3 and C4 which can be described according to Cremer & Pople (1975) by the puckering parameters q_2 = 0.3903 Å and Φ_2 = 180.9772. The closest pucker descriptor is an envelope E_{O1}.

The bridging ligands lead to a linear chain-like structure along [010] which resembles an antitactical polymer well known from organic chemistry.

Experimental

The title compound was obtained as a byproduct by the reaction of 40 mMol anhydroerytritol with 160 mMol sodiumhydoxide in 15 mL water at room temperature.

Upon standing for about 14 days at room temperature, colorless platelets of the title compound crystalized from the solution.

Refinement

Carbon hydrogen atoms and hydoxide hydrogen atoms were calculated in ideal geometry with U(H)=1.2*U(C) for all C-bound hydrogen atoms and U(H)=1.5*U(O) for the hydroxide hydrogen atom. The water-bound hydrogen atoms were found from the difference map, the O-H distances were fixed to 0.84 Å and the H-H distances within the water molecules were fixed to 1.36 Å.

Figures

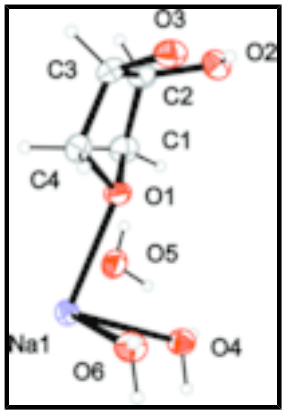


Fig. 1. The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

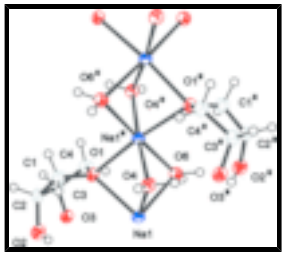


Fig. 2. Part of the antitactical polymer-like chain structure of the title compound along [010]. Symmetry code: (iii) -*x* + 1/2, *y* - 1/2, -*z* + 1/2.

Poly[[μ-1,4-anhydroerythritolato-di-μ-aqua-sodium(I)] monohydrate]

Crystal data

$[Na(C_4H_7O_3)(H_2O)_2]\cdot H_2O_1$	F_{000} = 768
M_r = 180.13	D_x = 1.474 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2600 reflections
a = 23.155 (6) Å	θ = 3.8–26.5º
b = 6.0900 (16) Å	μ = 0.18 mm^{-1}
c = 14.543 (5) Å	*T* = 200 (2) K
β = 127.678 (17)º	Platelet, colourless
V = 1623.1 (9) Å^3	0.23 × 0.20 × 0.10 mm
Z = 8	

Data collection

Oxford Diffraction KappaCCD diffractometer	1088 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.044
Monochromator: graphite	θ_{max} = 26.5º
T = 200(2) K	θ_{min} = 4.2º
ω scans	*h* = −21→28
Absorption correction: none	*k* = −7→6

6310 measured reflections
1699 independent reflections

$l = -17 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.086$
$S = 0.95$
1699 reflections
118 parameters
9 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: none

Special details

Refinement. Carbon hydrogen atoms and hydroxide hydrogen atoms were calculated in ideal geometry with U(H)=1.2*U(C) for all C-bound hydrogen atoms and U(H)=1.5*U(O) for the hydroxide hydrogen atom. The water-bound hydrogen atoms were found from the difference map, the O-H distances were restrained to 0.84 Å and the H-H distances within the water molecules were restrained to 1.36 Å.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Na1	0.24809 (3)	0.43051 (10)	0.24000 (5)	0.0238 (2)
O1	0.16736 (6)	0.16529 (17)	0.09168 (9)	0.0238 (3)
O2	0.05729 (6)	−0.21567 (19)	−0.03931 (10)	0.0322 (3)
H2	0.0846	−0.2907	−0.0445	0.048*
O3	0.14255 (6)	−0.19459 (18)	−0.08460 (9)	0.0295 (3)
O4	0.24091 (7)	0.19528 (18)	0.36644 (10)	0.0261 (3)
H41	0.2075 (8)	0.196 (3)	0.3733 (15)	0.039*
H42	0.2796 (8)	0.222 (3)	0.4385 (13)	0.039*
O6	0.34457 (6)	0.18090 (18)	0.30154 (10)	0.0285 (3)
H61	0.3781 (9)	0.212 (3)	0.3752 (12)	0.043*
H62	0.3710 (10)	0.123 (3)	0.2850 (14)	0.043*
C1	0.09012 (9)	0.1596 (3)	0.03391 (15)	0.0288 (4)
H11	0.0686	0.3080	0.0067	0.035*
H12	0.0801	0.1041	0.0868	0.035*
C2	0.05911 (9)	0.0052 (3)	−0.06810 (14)	0.0282 (4)
H1	0.0097	0.0547	−0.1367	0.034*
C3	0.11567 (9)	0.0160 (3)	−0.09473 (14)	0.0272 (4)
H3	0.0922	0.0765	−0.1742	0.033*
C4	0.17244 (9)	0.1743 (3)	−0.00250 (14)	0.0284 (4)

H43	0.2218	0.1304	0.0251	0.034*
H44	0.1626	0.3251	−0.0343	0.034*
O5	0.06903 (7)	0.4653 (2)	0.23361 (12)	0.0439 (4)
H51	0.0915 (10)	0.384 (3)	0.2926 (14)	0.066*
H52	0.0326 (9)	0.400 (3)	0.1783 (15)	0.066*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Na1	0.0278 (4)	0.0183 (4)	0.0225 (4)	−0.0002 (3)	0.0140 (3)	−0.0003 (3)
O1	0.0232 (7)	0.0270 (6)	0.0190 (6)	−0.0019 (5)	0.0116 (5)	−0.0015 (5)
O2	0.0308 (7)	0.0304 (7)	0.0336 (7)	−0.0053 (5)	0.0188 (6)	−0.0022 (5)
O3	0.0292 (7)	0.0322 (7)	0.0242 (7)	0.0002 (5)	0.0149 (6)	−0.0048 (5)
O4	0.0268 (7)	0.0304 (7)	0.0229 (7)	−0.0016 (6)	0.0160 (6)	−0.0025 (5)
O6	0.0269 (7)	0.0299 (7)	0.0281 (7)	0.0016 (5)	0.0165 (6)	−0.0036 (5)
C1	0.0238 (10)	0.0310 (10)	0.0292 (10)	0.0037 (7)	0.0149 (8)	0.0005 (8)
C2	0.0202 (9)	0.0307 (10)	0.0232 (9)	0.0007 (8)	0.0079 (8)	0.0013 (8)
C3	0.0282 (9)	0.0310 (10)	0.0180 (9)	0.0006 (8)	0.0119 (8)	0.0017 (7)
C4	0.0332 (10)	0.0290 (10)	0.0248 (10)	−0.0031 (8)	0.0186 (9)	0.0014 (7)
O5	0.0361 (8)	0.0447 (9)	0.0349 (8)	−0.0085 (6)	0.0136 (7)	0.0117 (6)

Geometric parameters (Å, °)

Na1—O4^{i}	2.3544 (13)	O4—H42	0.883 (14)
Na1—O6	2.3787 (14)	O6—Na1^{ii}	2.3893 (14)
Na1—O6^{i}	2.3893 (14)	O6—H61	0.877 (14)
Na1—O1	2.4139 (13)	O6—H62	0.859 (14)
Na1—O4	2.4155 (14)	C1—C2	1.516 (2)
Na1—O1^{i}	2.4517 (14)	C1—H11	0.9900
Na1—Na1^{i}	3.0550 (8)	C1—H12	0.9900
Na1—Na1^{ii}	3.0550 (8)	C2—C3	1.578 (2)
O1—C1	1.438 (2)	C2—H1	1.0000
O1—C4	1.445 (2)	C3—C4	1.517 (2)
O1—Na1^{ii}	2.4517 (14)	C3—H3	1.0000
O2—C2	1.417 (2)	C4—H43	0.9900
O2—H2	0.8200	C4—H44	0.9900
O3—C3	1.394 (2)	O5—H51	0.842 (15)
O4—Na1^{ii}	2.3544 (13)	O5—H52	0.828 (14)
O4—H41	0.839 (14)		
?···?	?		
O4^{i}—Na1—O6	103.35 (5)	Na1^{ii}—O4—H41	124.1 (12)
O4^{i}—Na1—O6^{i}	80.67 (5)	Na1—O4—H41	125.9 (13)
O6—Na1—O6^{i}	174.07 (3)	Na1^{ii}—O4—H42	117.9 (12)
O4^{i}—Na1—O1	101.92 (5)	Na1—O4—H42	107.6 (12)
O6—Na1—O1	86.84 (5)	H41—O4—H42	101.5 (14)
O6^{i}—Na1—O1	96.65 (5)	Na1—O6—Na1^{ii}	79.69 (4)

O4^{i}—Na1—O4	172.80 (5)	Na1—O6—H61	104.5 (12)
O6—Na1—O4	79.65 (5)	Na1^{ii}—O6—H61	115.6 (13)
O6^{i}—Na1—O4	95.86 (5)	Na1—O6—H62	145.5 (12)
O1—Na1—O4	84.69 (5)	Na1^{ii}—O6—H62	110.5 (13)
O4^{i}—Na1—O1^{i}	85.17 (5)	H61—O6—H62	100.4 (15)
O6—Na1—O1^{i}	90.22 (5)	O1—C1—C2	105.23 (13)
O6^{i}—Na1—O1^{i}	85.75 (5)	O1—C1—H11	110.7
O1—Na1—O1^{i}	172.78 (4)	C2—C1—H11	110.7
O4—Na1—O1^{i}	88.30 (5)	O1—C1—H12	110.7
O4^{i}—Na1—Na1^{i}	51.06 (3)	C2—C1—H12	110.7
O6—Na1—Na1^{i}	129.44 (5)	H11—C1—H12	108.8
O6^{i}—Na1—Na1^{i}	50.00 (3)	O2—C2—C1	112.42 (14)
O1—Na1—Na1^{i}	135.52 (4)	O2—C2—C3	106.92 (13)
O4—Na1—Na1^{i}	122.01 (4)	C1—C2—C3	104.28 (13)
O1^{i}—Na1—Na1^{i}	50.56 (3)	O2—C2—H1	111.0
O4^{i}—Na1—Na1^{ii}	137.46 (4)	C1—C2—H1	111.0
O6—Na1—Na1^{ii}	50.31 (3)	C3—C2—H1	111.0
O6^{i}—Na1—Na1^{ii}	129.01 (4)	O3—C3—C4	113.69 (14)
O1—Na1—Na1^{ii}	51.66 (3)	O3—C3—C2	108.65 (13)
O4—Na1—Na1^{ii}	49.30 (3)	C4—C3—C2	102.13 (13)
O1^{i}—Na1—Na1^{ii}	121.82 (4)	O3—C3—H3	110.7
Na1^{i}—Na1—Na1^{ii}	170.73 (4)	C4—C3—H3	110.7
C1—O1—C4	103.87 (12)	C2—C3—H3	110.7
C1—O1—Na1	123.05 (9)	O1—C4—C3	106.58 (13)
C4—O1—Na1	110.75 (9)	O1—C4—H43	110.4
C1—O1—Na1^{ii}	121.20 (10)	C3—C4—H43	110.4
C4—O1—Na1^{ii}	119.37 (9)	O1—C4—H44	110.4
Na1—O1—Na1^{ii}	77.78 (4)	C3—C4—H44	110.4
C2—O2—H2	109.7	H43—C4—H44	108.6
Na1^{ii}—O4—Na1	79.64 (4)	H51—O5—H52	109.6 (19)
O4^{i}—Na1—O1—C1	−98.09 (11)	Na1^{i}—Na1—O4—Na1^{ii}	−175.97 (5)
O6—Na1—O1—C1	158.92 (11)	O4^{i}—Na1—O6—Na1^{ii}	−141.91 (5)
O6^{i}—Na1—O1—C1	−16.27 (12)	O1—Na1—O6—Na1^{ii}	−40.40 (4)
O4—Na1—O1—C1	79.03 (12)	O4—Na1—O6—Na1^{ii}	44.77 (4)
Na1^{i}—Na1—O1—C1	−52.67 (13)	O1^{i}—Na1—O6—Na1^{ii}	133.00 (5)
Na1^{ii}—Na1—O1—C1	119.44 (12)	Na1^{i}—Na1—O6—Na1^{ii}	167.97 (6)
O4^{i}—Na1—O1—C4	25.43 (10)	C4—O1—C1—C2	41.42 (15)
O6—Na1—O1—C4	−77.55 (10)	Na1—O1—C1—C2	168.00 (9)
O6^{i}—Na1—O1—C4	107.26 (10)	Na1^{ii}—O1—C1—C2	−96.32 (14)
O4—Na1—O1—C4	−157.45 (10)	O1—C1—C2—O2	90.07 (16)
Na1^{i}—Na1—O1—C4	70.85 (11)	O1—C1—C2—C3	−25.39 (17)

Na1^{ii}—Na1—O1—C4	−117.04 (10)	O2—C2—C3—O3	1.87 (17)
O4^{i}—Na1—O1—Na1^{ii}	142.47 (5)	C1—C2—C3—O3	121.13 (14)
O6—Na1—O1—Na1^{ii}	39.48 (4)	O2—C2—C3—C4	−118.55 (14)
O6^{i}—Na1—O1—Na1^{ii}	−135.70 (5)	C1—C2—C3—C4	0.71 (16)
O4—Na1—O1—Na1^{ii}	−40.41 (4)	C1—O1—C4—C3	−41.55 (15)
Na1^{i}—Na1—O1—Na1^{ii}	−172.11 (6)	Na1—O1—C4—C3	−175.52 (10)
O6—Na1—O4—Na1^{ii}	−45.63 (4)	Na1^{ii}—O1—C4—C3	97.14 (13)
O6^{i}—Na1—O4—Na1^{ii}	138.29 (5)	O3—C3—C4—O1	−92.61 (16)
O1—Na1—O4—Na1^{ii}	42.12 (4)	C2—C3—C4—O1	24.23 (16)
O1^{i}—Na1—O4—Na1^{ii}	−136.17 (5)		

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3	0.82	1.85	2.4367 (17)	127
O4—H41···O3^{iii}	0.839 (14)	1.939 (15)	2.7706 (17)	170.9 (17)
O4—H42···O3^{i}	0.883 (14)	1.827 (14)	2.7078 (19)	174.5 (18)
O6—H61···O2^{i}	0.877 (14)	1.940 (14)	2.813 (2)	173.1 (18)
O6—H62···O5^{ii}	0.859 (14)	1.838 (14)	2.6903 (18)	171.0 (18)
O5—H51···O3^{iii}	0.842 (15)	1.825 (15)	2.6653 (18)	176 (2)
O5—H52···O2^{iv}	0.828 (14)	2.131 (15)	2.958 (2)	177 (2)

Symmetry codes: (iii) *x*, −*y*, *z*+1/2; (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iv) −*x*, −*y*, −*z*.

Fig. 1

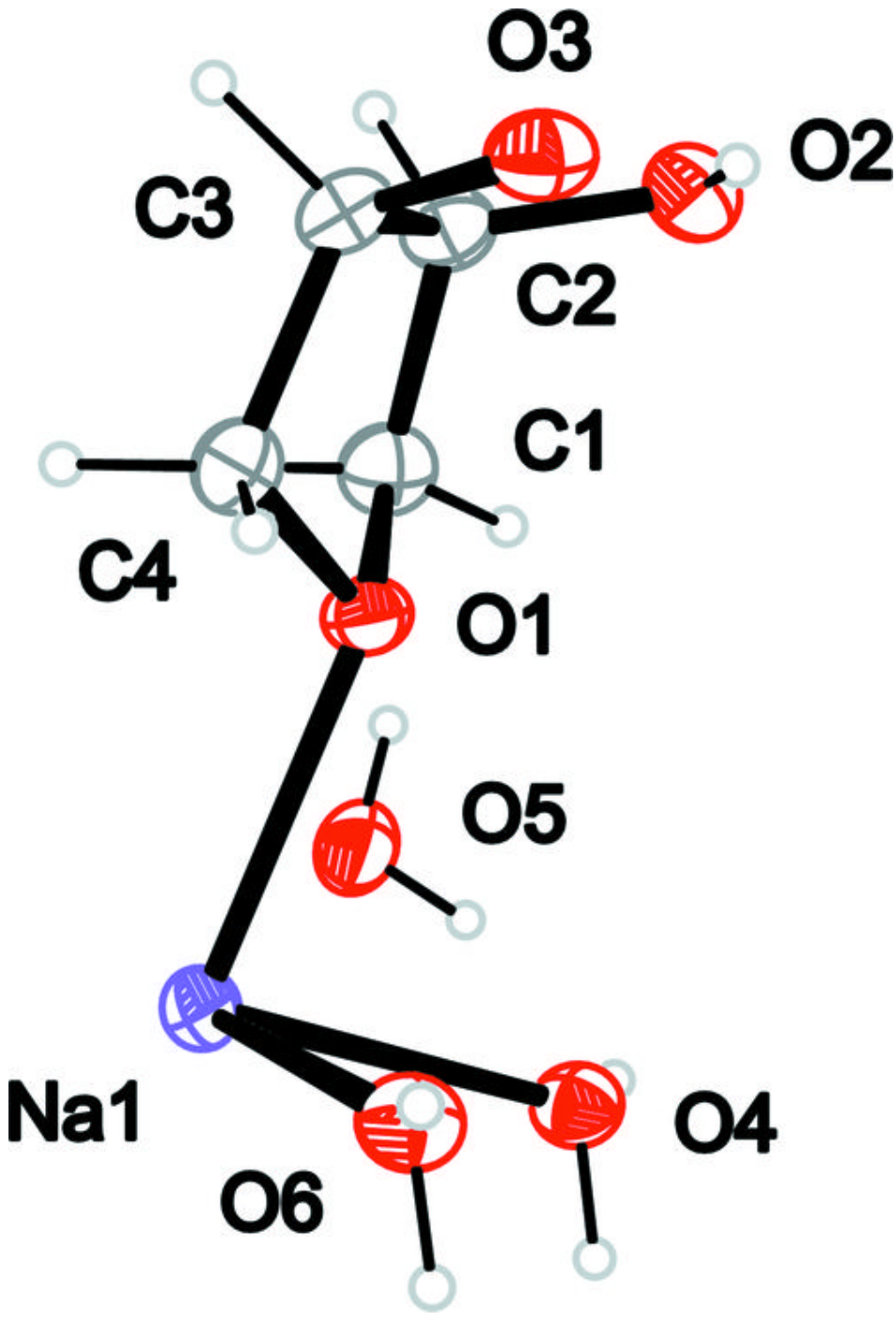
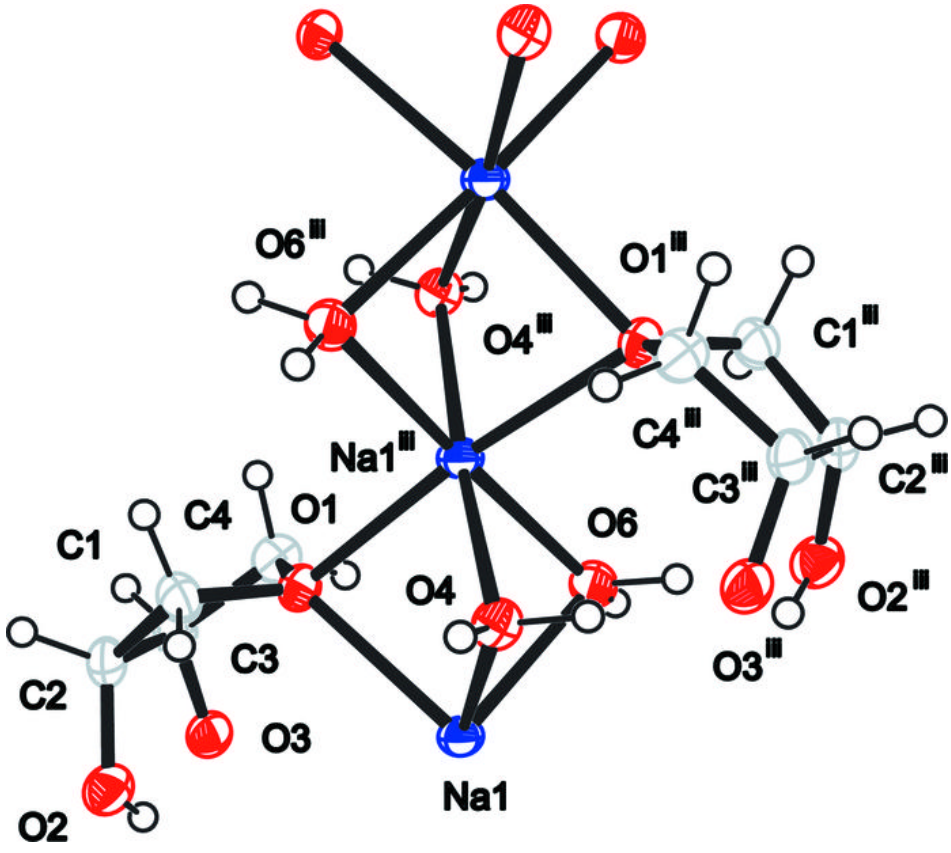


Fig. 2


O6III
O1III
O4III
C1III
C4III
Na1III
C3III
C2III
C1
C4
O1
O6
O4
O2III
C3
O3III
C2
O3
O2
Na1