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The sodium chloride complex *catena*-poly[[{μ_3-2-[bis(2-hydroxyethyl)amino]ethan-1-ol}sodium] chloride], $N(CH_2CH_2OH)_3{\cdot}NaCl$

Christina Krabbe, Vinusuya Gock, Michael Lutter and Klaus Jurkschat*

Lehrstuhl für Anorganische Chemie II, Technische Universität Dortmund, 44221 Dortmund, Germany. *Correspondence e-mail: klaus.jurkschat@tu-dortmund.de

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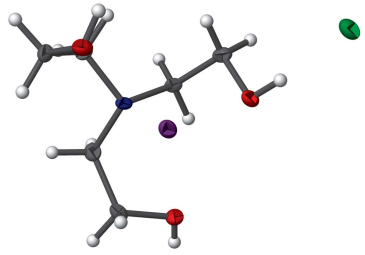
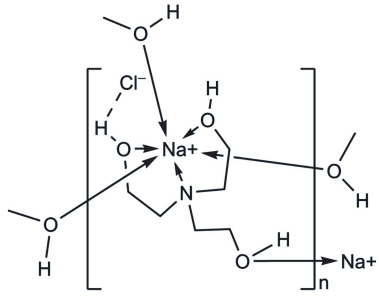
The reaction of sodium chloride with 2-[bis(2-hydroxyethyl)amino]ethan-1-ol results in the formation of the title salt $\{[Na\{N(CH_2CH_2OH)_3\}]Cl\}_n$. The polymeric structure is characterized by a sodium cation coordinated by one nitrogen and five oxygen atoms in a distorted octahedral environment. The resulting one-dimensional {—O—Na—O—Na—O}— coordination polymer extends parallel to [010] and is connected through the chloride counter-anion *via* O—H···Cl hydrogen bonding, giving rise to a two-dimensional supramolecular structure parallel to (001).

3D view



Chemical scheme



Structure description

In the context of our long-standing focus on tin derivatives of aminoalcohols, as for instance stannatranes (Glowacki *et al.*, 2016, 2017; Zöller *et al.*, 2011, 2012; Zöller & Jurkschat, 2013), we are also interested in the structures of selected starting materials such as salt complexes of the amino alcohol $N(CH_2CH_2OH)_3$. Sodium complexes of this alcohol with iodide (Voegele *et al.*, 1974) and perchlorate (Naiini *et al.*, 1994) counter-anions have been reported previously. In both these molecular structures, the three alcohol functional groups of one molecule coordinate the sodium cation in addition to the nitrogen atom. However, the title compound (Fig. 1) shows another coordination pattern. The sodium cation Na1 is six-coordinated by N1, O3, O5 of one molecule and by O1*A*, O2*A* and O3*B* of symmetry-related molecules at distances of 2.533 (3), 2.495 (3), 2.438 (3), 2.389 (3), 2.355 (4), and 2.463 (3) Å, respectively [Symmetry code: (*A*) $1-x$, $y-\frac{1}{2}$, $1-z$. (*B*) $1-x$, $y+\frac{1}{2}$, $1-z$.]. The sodium cation exhibits a distorted octahedral environment with O3, O5, O5*A* and O3*B* occupying the equatorial positions, and with N1 and O1*A* axial. The distortion from the ideal octahedral environment is expressed by the

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···Cl1	0.86 (4)	2.21 (4)	3.061 (3)	169 (4)
O2—H2···Cl1^{i}	0.72 (5)	2.44 (5)	3.141 (4)	165 (6)
O3—H3···Cl1^{ii}	0.83 (3)	2.33 (3)	3.124 (3)	161 (3)

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

trans angles [N1—Na1—O2*A* = 141.31 (12)°; O1—Na1—O1*A* = 165.04 (12)°; O3—Na1—O3*B* = 167.27 (11)°] deviating clearly from 180°. As a result of this coordination pattern, a one-dimensional polymer is formed along [010].

In the crystal structure three hydrogen bonds between chloride anions and oxygen atoms of the alcohol functional groups are present (Table 1, Fig. 1). A graph-set analysis according to Etter and Bernstein (Bernstein *et al.*, 1990, 1995; Etter *et al.*, 1990; Etter, 1990, 1991) gives the unitary graph set N_1 = DDD. These hydrogen bonds create a two-dimensional supramolecular network structure extending parallel to (001).

Synthesis and crystallization

After addition of $N(CH_2CH_2OH)_3$ (3.14 g, 0.02 mmol) to a solution of sodium chloride (2.46 g, 0.04 mmol) in THF, two thirds of the solvent were distilled off. The title compound crystallized from the solution as colourless plate-shaped crystals.

^{1}H-NMR: (400.13 MHz, DMSO-d_6) δ 2.57 (*s*, $\nu_{1/2}$ = 8 Hz, 6 H, NCH_2), 3.42 (*s*, $\nu_{1/2}$ = 8 Hz, 6 H, OCH_2), 4.38 (*s*, $\nu_{1/2}$ = 8 Hz, 3 H, O*H*).

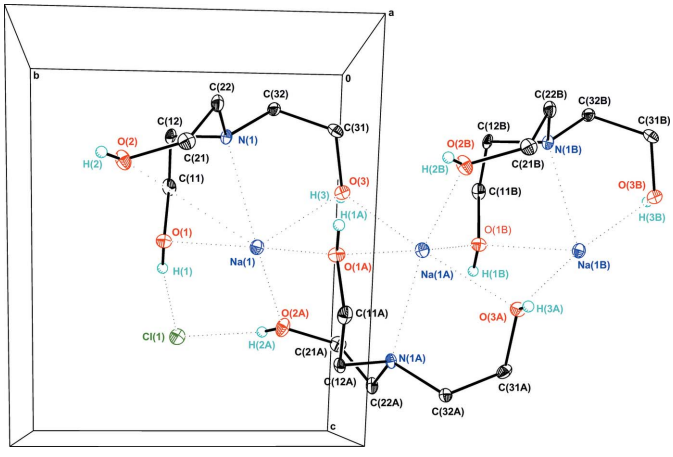


Figure 1
The crystal structure of $[Na(N(CH_2CH_2OH)_3)]Cl$, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen atoms bonded to carbon atoms have been omitted for clarity. Atoms Cl1*A* and Cl1*B* are not shown because they are located behind and in front of the *bc* plane. Hydrogen bonds are drawn as dashed lines. [Symmetry codes: (*A*) $1-x, y-\frac{1}{2}, 1-z$. (*B*) $1-x, y+\frac{1}{2}, 1-z$.]

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Na(C_6H_{15}NO_3)]\cdot Cl$
M_r	207.63
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	173
a, *b*, *c* (Å)	7.7981 (10), 7.2249 (8), 8.9956 (11)
β (°)	110.380 (14)
V (Å^3)	475.09 (11)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.42
Crystal size (mm)	0.32 × 0.06 × 0.03
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.903, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3110, 1719, 1201
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.605
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.043, 0.70
No. of reflections	1719
No. of parameters	121
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.18
Absolute structure	Flack *x* determined using 408 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.07 (7)

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. For the X-ray data collection, a strategy for centrosymmetric space groups was chosen. As a result of the oxygen atoms O1, O3, and O5 being crystallographically non-equivalent, the N1 atom is a stereogenic center, and thus the compound crystallizes in a non-centrosymmetric space-group type. Consequently, the number of collected data is less than expected for this symmetry and probably explains the goodness-of-fit parameter lying outside the usual range. However, the absolute structure was determined correctly (Table 2).

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x190238 [https://doi.org/10.1107/S2414314619002384]

The sodium chloride complex *catena*-poly[[{μ_3-2-[bis(2-hydroxyethyl)amino]-ethan-1-ol}sodium] chloride], $N(CH_2CH_2OH)_3{\cdot}NaCl$

Christina Krabbe, Vinusuya Gock, Michael Lutter and Klaus Jurkschat

***catena*-Poly[[{μ_3-2-[bis(2-hydroxyethyl)amino]ethan-1-ol}sodium] chloride]**

Crystal data

$[Na(C_6H_{15}NO_3)]{\cdot}Cl$
M_r = 207.63
Monoclinic, $P2_1$
a = 7.7981 (10) Å
b = 7.2249 (8) Å
c = 8.9956 (11) Å
β = 110.380 (14)°
V = 475.09 (11) Å^3
Z = 2
$F(000)$ = 220
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1217 reflections
θ = 2.4–28.9°
μ = 0.42 mm^{-1}
T = 173 K
Column, colourless
0.32 × 0.06 × 0.03 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer
Graphite monochromator
Detector resolution: 16.0560 pixels mm^{-1}
ω und ψ scan
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 2010)
T_{min} = 0.903, T_{max} = 1.000
3110 measured reflections
1719 independent reflections
1201 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.5°, θ_{min} = 2.4°
h = −4→9
k = −8→8
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.043
S = 0.70
1719 reflections
121 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0132P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 408 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.07 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The hydrogen atoms of the OH groups were located in a difference Fourier map and were refined with $U_{iso}(H) = 1.2U_{eq}(O)$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.11499 (13)	0.52160 (16)	0.72753 (10)	0.0212 (3)
Na1	0.4707 (2)	0.2903 (2)	0.49696 (18)	0.0163 (4)
N1	0.3494 (4)	0.3861 (4)	0.2080 (3)	0.0125 (8)
O1	0.2902 (4)	0.5714 (4)	0.4739 (3)	0.0169 (7)
H1	0.237 (5)	0.574 (6)	0.543 (4)	0.033 (14)*
O2	0.6300 (4)	0.6951 (4)	0.2967 (3)	0.0222 (8)
H2	0.697 (6)	0.756 (8)	0.285 (5)	0.05 (2)*
O3	0.3064 (3)	0.0176 (5)	0.3419 (3)	0.0170 (6)
H3	0.202 (4)	0.010 (7)	0.347 (4)	0.021 (12)*
C21	0.6635 (5)	0.5139 (7)	0.2528 (4)	0.0194 (10)
H21A	0.713380	0.439122	0.347642	0.023*
H21B	0.754537	0.520099	0.201930	0.023*
C22	0.4935 (5)	0.4210 (5)	0.1415 (4)	0.0180 (11)
H22A	0.443202	0.497838	0.047906	0.022*
H22B	0.528554	0.303870	0.107800	0.022*
C11	0.1473 (5)	0.5556 (6)	0.3212 (4)	0.0191 (10)
H11A	0.063574	0.659234	0.303972	0.023*
H11B	0.078900	0.442075	0.315703	0.023*
C12	0.2370 (4)	0.5544 (6)	0.1967 (4)	0.0141 (10)
H12A	0.143227	0.560173	0.092254	0.017*
H12B	0.313816	0.663093	0.209870	0.017*
C31	0.3015 (5)	0.0465 (6)	0.1825 (4)	0.0179 (10)
H31A	0.224855	−0.047398	0.113926	0.021*
H31B	0.423976	0.033351	0.179493	0.021*
C32	0.2286 (5)	0.2344 (5)	0.1218 (4)	0.0155 (10)
H32A	0.213121	0.242377	0.010263	0.019*
H32B	0.109213	0.249789	0.131188	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0187 (6)	0.0235 (6)	0.0250 (5)	0.0026 (6)	0.0121 (4)	0.0014 (6)
Na1	0.0148 (10)	0.0159 (9)	0.0183 (7)	0.0024 (8)	0.0057 (7)	0.0008 (7)
N1	0.0098 (19)	0.0107 (19)	0.0178 (18)	−0.0020 (14)	0.0056 (15)	0.0012 (14)
O1	0.0136 (16)	0.0215 (19)	0.0169 (14)	0.0010 (13)	0.0069 (14)	−0.0008 (12)
O2	0.021 (2)	0.019 (2)	0.0310 (18)	−0.0079 (15)	0.0150 (16)	−0.0067 (15)
O3	0.0136 (16)	0.0200 (16)	0.0171 (13)	−0.0014 (18)	0.0050 (12)	0.0013 (16)

C21	0.016 (2)	0.023 (3)	0.022 (2)	0.002 (3)	0.0100 (18)	0.000 (3)
C22	0.021 (3)	0.012 (3)	0.024 (2)	−0.001 (2)	0.011 (2)	0.0001 (18)
C11	0.013 (2)	0.017 (3)	0.025 (2)	−0.001 (2)	0.0027 (18)	−0.002 (2)
C12	0.010 (2)	0.010 (3)	0.0174 (19)	0.003 (2)	−0.0008 (16)	0.003 (2)
C31	0.019 (2)	0.020 (3)	0.0165 (19)	−0.004 (2)	0.0090 (17)	−0.006 (2)
C32	0.017 (2)	0.014 (3)	0.015 (2)	−0.0020 (18)	0.0048 (19)	0.0006 (17)

Geometric parameters (Å, º)

Na1—O2^{i}	2.355 (4)	C21—C22	1.512 (5)
Na1—O1^{i}	2.389 (3)	C21—H21A	0.9700
Na1—O1	2.438 (3)	C21—H21B	0.9700
Na1—O3^{ii}	2.463 (3)	C22—H22A	0.9700
Na1—O3	2.495 (3)	C22—H22B	0.9700
Na1—N1	2.533 (3)	C11—C12	1.513 (4)
Na1—C11	3.123 (4)	C11—H11A	0.9700
N1—C22	1.467 (4)	C11—H11B	0.9700
N1—C32	1.477 (4)	C12—H12A	0.9700
N1—C12	1.481 (4)	C12—H12B	0.9700
O1—C11	1.441 (4)	C31—C32	1.499 (5)
O1—H1	0.86 (4)	C31—H31A	0.9700
O2—C21	1.418 (5)	C31—H31B	0.9700
O2—H2	0.72 (5)	C32—H32A	0.9700
O3—C31	1.436 (4)	C32—H32B	0.9700
O3—H3	0.83 (3)		
O2^{i}—Na1—O1^{i}	100.48 (12)	Na1—O3—H3	110 (3)
O2^{i}—Na1—O1	88.25 (12)	O2—C21—C22	113.0 (3)
O1^{i}—Na1—O1	165.04 (12)	O2—C21—H21A	109.0
O2^{i}—Na1—O3^{ii}	95.27 (11)	C22—C21—H21A	109.0
O1^{i}—Na1—O3^{ii}	90.72 (10)	O2—C21—H21B	109.0
O1—Na1—O3^{ii}	76.30 (10)	C22—C21—H21B	109.0
O2^{i}—Na1—O3	87.85 (11)	H21A—C21—H21B	107.8
O1^{i}—Na1—O3	76.57 (10)	N1—C22—C21	115.2 (3)
O1—Na1—O3	116.20 (10)	N1—C22—H22A	108.5
O3^{ii}—Na1—O3	167.27 (11)	C21—C22—H22A	108.5
O2^{i}—Na1—N1	141.31 (12)	N1—C22—H22B	108.5
O1^{i}—Na1—N1	106.95 (12)	C21—C22—H22B	108.5
O1—Na1—N1	71.76 (11)	H22A—C22—H22B	107.5
O3^{ii}—Na1—N1	110.88 (11)	O1—C11—C12	107.6 (3)
O3—Na1—N1	72.95 (10)	O1—C11—Na1	49.17 (18)
O2^{i}—Na1—C11	98.40 (12)	C12—C11—Na1	82.6 (2)
O1^{i}—Na1—C11	157.45 (11)	O1—C11—H11A	110.2
O1—Na1—C11	26.55 (9)	C12—C11—H11A	110.2
O3^{ii}—Na1—C11	99.73 (11)	Na1—C11—H11A	159.2
O3—Na1—C11	91.99 (11)	O1—C11—H11B	110.2
N1—Na1—C11	50.65 (10)	C12—C11—H11B	110.2
C22—N1—C32	110.7 (3)	Na1—C11—H11B	80.8

C22—N1—C12	110.5 (3)	H11A—C11—H11B	108.5
C32—N1—C12	108.6 (3)	N1—C12—C11	111.7 (3)
C22—N1—Na1	113.6 (2)	N1—C12—H12A	109.3
C32—N1—Na1	106.1 (2)	C11—C12—H12A	109.3
C12—N1—Na1	107.1 (2)	N1—C12—H12B	109.3
C11—O1—Na1^{ii}	118.2 (2)	C11—C12—H12B	109.3
C11—O1—Na1	104.3 (2)	H12A—C12—H12B	107.9
Na1^{ii}—O1—Na1	97.85 (10)	O3—C31—C32	111.7 (3)
C11—O1—H1	106 (2)	O3—C31—H31A	109.3
Na1^{ii}—O1—H1	118 (3)	C32—C31—H31A	109.3
Na1—O1—H1	111 (3)	O3—C31—H31B	109.3
C21—O2—Na1^{ii}	129.3 (3)	C32—C31—H31B	109.3
C21—O2—H2	108 (4)	H31A—C31—H31B	107.9
Na1^{ii}—O2—H2	113 (4)	N1—C32—C31	112.9 (3)
C31—O3—Na1^{i}	116.9 (2)	N1—C32—H32A	109.0
C31—O3—Na1	105.8 (3)	C31—C32—H32A	109.0
Na1^{i}—O3—Na1	94.46 (8)	N1—C32—H32B	109.0
C31—O3—H3	112 (2)	C31—C32—H32B	109.0
Na1^{i}—O3—H3	115 (3)	H32A—C32—H32B	107.8
Na1^{ii}—O2—C21—C22	85.8 (3)	Na1—N1—C12—C11	−30.7 (3)
C32—N1—C22—C21	−156.9 (3)	O1—C11—C12—N1	66.4 (4)
C12—N1—C22—C21	82.8 (4)	Na1—C11—C12—N1	23.5 (3)
Na1—N1—C22—C21	−37.7 (4)	Na1^{i}—O3—C31—C32	−155.0 (2)
O2—C21—C22—N1	−64.2 (4)	Na1—O3—C31—C32	−51.4 (3)
Na1^{ii}—O1—C11—C12	44.2 (4)	C22—N1—C32—C31	84.7 (4)
Na1—O1—C11—C12	−63.1 (3)	C12—N1—C32—C31	−153.9 (3)
Na1^{ii}—O1—C11—Na1	107.2 (2)	Na1—N1—C32—C31	−39.0 (3)
C22—N1—C12—C11	−155.0 (3)	O3—C31—C32—N1	65.8 (4)
C32—N1—C12—C11	83.5 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	0.86 (4)	2.21 (4)	3.061 (3)	169 (4)
O2—H2···Cl1^{ii}	0.72 (5)	2.44 (5)	3.141 (4)	165 (6)
O3—H3···Cl1^{iii}	0.83 (3)	2.33 (3)	3.124 (3)	161 (3)

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