Crystal structure of sodium adamantyl(oxyethylene)sulfate dihydrate, $NaC_{10}H_{15}OC_2H_4SO_3 \cdot 2H_2O$

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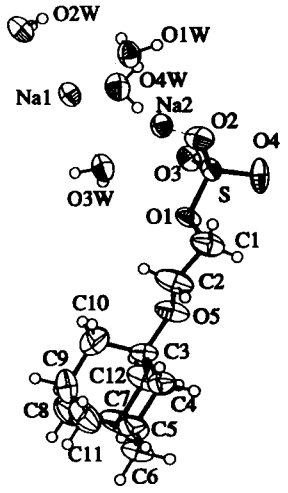

Fig. 1. Molecule plot.

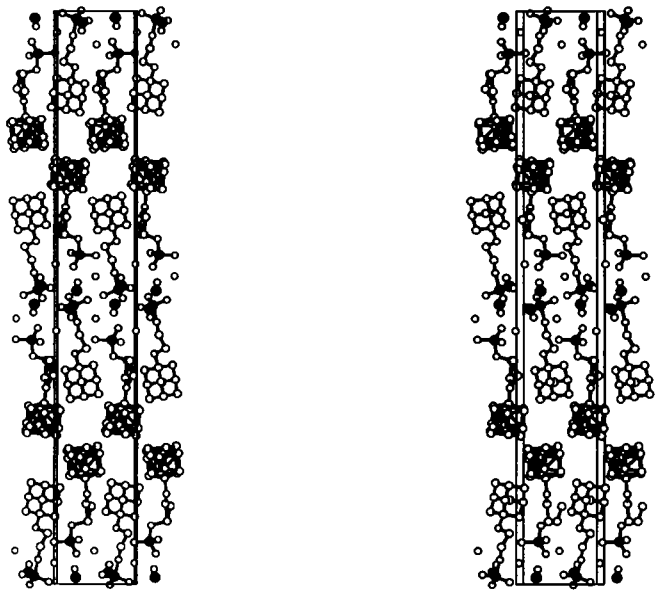

Fig. 2. Stereo plot of the packing of the molecules.

Source of material: Crystallization from an ethanol/water solution. There are two adamantyl(oxyethylene)sulfate anions, two sodium cations and four water molecules in the asymmetric unit. In one of the two adamantyl(oxyethylene)sulfate anions nearly all C atoms were found to be disordered. For C1 and C2 of the ethylene oxide chain two positions and population parameters were refined. For the adamantyl part of the molecule position and orientation of two rigid adamantane structures with the same population parameters as in the ethylene oxide chain were refined. Both parts of the disordered adamantane ring structures are rotated relative to each other by approximately 50° around the C3–C5 bond. The final population parameters were 0.33(1) and 0.67(1). The molecules form a bilayer structure.

$C_{12}H_{23}NaO_7S$, monoclinic, $P12_1/n1$ (No. 14), a =6.816(3) Å, b =48.73(3) Å, c =9.521(2) Å, β =91.15(3)°, V =3161.7 Å^3, Z =8, $R(F)$ =0.086, $R_w(F^2)$ =0.206.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless plate, size 0.03 x 0.16 x 0.58 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	2.60 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω/2θ
$T_{measurement}$:	300 K
$2\theta_{max}$:	50°
N(hkl)$_{unique}$:	5474
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
N($param$)$_{refined}$:	396
Programs:	SHELXS-86, SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	Occ.	x	y	z	U_{iso}
H(11W)	4e		0.24(1)	–0.040(2)	–0.108(7)	0.065
H(12W)	4e		0.29(1)	–0.048(1)	–0.223(7)	0.065
H(21W)	4e		0.407(8)	–0.066(2)	–0.520(8)	0.066
H(22W)	4e		0.50(1)	–0.062(2)	–0.412(3)	0.066
H(31W)	4e		0.142(8)	0.029(2)	–0.394(7)	0.066
H(32W)	4e		0.321(9)	0.030(2)	–0.394(7)	0.066
H(41W)	4e		0.01(1)	0.048(2)	–0.646(7)	0.073
H(42W)	4e		0.08(1)	0.027(2)	–0.60(1)	0.073
C(11_1)	4e	0.33(1)	0.421(3)	0.1102(5)	0.058(3)	0.048(8)
H(111_1)	4e	0.33(1)	0.502(3)	0.0951(5)	0.028(3)	0.063

Table 2. (Continued)

Atom	Site	Occ.	x	y	z	U_{iso}
H(112_1)	4e	0.33(1)	0.453(3)	0.1137(5)	0.157(3)	0.063
C(12_1)	4e	0.33(1)	0.469(4)	0.1362(4)	−0.030(3)	0.036(7)
H(121_1)	4e	0.33(1)	0.609(4)	0.1391(4)	−0.033(3)	0.047
H(122_1)	4e	0.33(1)	0.417(4)	0.1344(4)	−0.125(3)	0.047
C(11_2)	4e	0.67	0.393(3)	0.1099(4)	−0.026(3)	0.111(8)
H(111_2)	4e	0.67	0.412(3)	0.0979(4)	−0.106(3)	0.144
H(112_2)	4e	0.67	0.505(3)	0.1083(4)	0.037(3)	0.144
C(12_2)	4e	0.67	0.369(4)	0.1403(4)	−0.077(2)	0.131(9)
H(121_2)	4e	0.67	0.473(4)	0.1448(4)	−0.141(2)	0.170
H(122_2)	4e	0.67	0.244(4)	0.1426(4)	−0.126(2)	0.170
C(14_1)	4e	0.33(1)	0.551(2)	0.1970(5)	0.030(2)	0.11(2)
H(14A_1)	4e	0.33(1)	0.581(3)	0.1921(6)	0.127(3)	0.140
H(14B_1)	4e	0.33(1)	0.657(2)	0.1903(6)	−0.028(3)	0.140
C(15_1)	4e	0.33(1)	0.533(4)	0.2280(4)	0.016(3)	0.15(3)
H(15_1)	4e	0.33(1)	0.655(5)	0.2366(6)	0.046(5)	0.196
C(16_1)	4e	0.33(1)	0.368(6)	0.2382(4)	0.108(3)	0.14(3)
H(16A_1)	4e	0.33(1)	0.396(7)	0.2338(6)	0.206(3)	0.183
H(16B_1)	4e	0.33(1)	0.357(7)	0.2580(4)	0.101(4)	0.183
C(17_1)	4e	0.33(1)	0.175(4)	0.2249(5)	0.062(3)	0.15(3)
H(17_1)	4e	0.33(1)	0.070(5)	0.2314(7)	0.120(4)	0.190
C(18_1)	4e	0.33(1)	0.129(4)	0.2323(5)	−0.092(3)	0.12(2)
H(18A_1)	4e	0.33(1)	0.116(6)	0.2521(5)	−0.101(4)	0.161
H(18B_1)	4e	0.33(1)	0.006(4)	0.2240(7)	−0.121(4)	0.161
C(19_1)	4e	0.33(1)	0.295(4)	0.2221(5)	−0.184(2)	0.09(2)
H(19A_1)	4e	0.33(1)	0.266(6)	0.2269(7)	−0.281(2)	0.120
C(110_1)	4e	0.33(1)	0.312(3)	0.1910(4)	−0.171(1)	0.12(2)
H(11A_1)	4e	0.33(1)	0.190(4)	0.1822(6)	−0.201(2)	0.154
H(11B_1)	4e	0.33(1)	0.416(5)	0.1842(6)	−0.230(1)	0.154
C(111_1)	4e	0.33(1)	0.487(5)	0.2354(5)	−0.137(3)	0.14(2)
H(11C_1)	4e	0.33(1)	0.478(6)	0.2552(5)	−0.147(4)	0.178
H(11D_1)	4e	0.33(1)	0.593(5)	0.2292(7)	−0.196(4)	0.178
C(112_1)	4e	0.33(1)	0.192(3)	0.1938(5)	0.076(2)	0.14(2)
H(11E_1)	4e	0.33(1)	0.070(2)	0.1851(6)	0.047(3)	0.184
H(11F_1)	4e	0.33(1)	0.220(4)	0.1889(6)	0.173(2)	0.184

Table 2. (Continued)

Atom	Site	Occ.	x	y	z	U_{iso}
H(14A_2)	4e	0.67	0.653(1)	0.1907(3)	−0.053(2)	0.123
H(14B_2)	4e	0.67	0.532(2)	0.1825(2)	−0.189(1)	0.123
H(15_2)	4e	0.67	0.616(2)	0.2296(3)	−0.199(2)	0.134
H(16A_2)	4e	0.67	0.629(2)	0.2385(3)	0.045(2)	0.136
H(16B_2)	4e	0.67	0.493(2)	0.2602(2)	−0.031(2)	0.136
H(17_2)	4e	0.67	0.334(3)	0.2429(3)	0.168(1)	0.154
H(18A_2)	4e	0.67	0.127(2)	0.2556(2)	−0.022(2)	0.117
H(18B_2)	4e	0.67	0.033(2)	0.2309(3)	0.059(2)	0.117
H(19_2)	4e	0.67	0.021(2)	0.2221(3)	−0.185(2)	0.143
H(11A_2)	4e	0.67	0.056(1)	0.1830(3)	−0.039(2)	0.114
H(11B_2)	4e	0.67	0.164(2)	0.1779(2)	−0.181(2)	0.114
H(11C_2)	4e	0.67	0.301(3)	0.2473(3)	−0.249(1)	0.185
H(11D_2)	4e	0.67	0.315(3)	0.2175(3)	−0.3093(8)	0.185
H(11E_2)	4e	0.67	0.250(2)	0.1960(3)	0.179(1)	0.134
H(11F_2)	4e	0.67	0.479(2)	0.1989(3)	0.174(1)	0.134
H(211)	4e		−0.110(2)	0.0599(2)	−0.112(1)	0.086
H(212)	4e		−0.321(2)	0.0708(2)	−0.152(1)	0.086
H(221)	4e		−0.075(2)	0.1036(2)	−0.201(1)	0.099
H(222)	4e		0.011(2)	0.0831(2)	−0.312(1)	0.099
H(241)	4e		−0.057(2)	0.1442(2)	−0.291(1)	0.080
H(242)	4e		−0.284(2)	0.1499(2)	−0.301(1)	0.080
H(25)	4e		−0.114(2)	0.1885(2)	−0.387(1)	0.092
H(261)	4e		−0.279(2)	0.1930(2)	−0.602(1)	0.101
H(262)	4e		−0.418(2)	0.1798(2)	−0.491(1)	0.101
H(27)	4e		−0.439(2)	0.1550(2)	−0.703(1)	0.108
H(281)	4e		−0.120(2)	0.1626(3)	−0.783(1)	0.121
H(282)	4e		−0.159(2)	0.1308(3)	−0.779(1)	0.121
H(29)	4e		0.151(2)	0.1407(3)	−0.672(1)	0.115
H(21A)	4e		−0.026(2)	0.1021(2)	−0.590(1)	0.110
H(21B)	4e		0.100(2)	0.1146(2)	−0.466(1)	0.110
H(21C)	4e		0.080(2)	0.1841(3)	−0.585(1)	0.115
H(21D)	4e		0.164(2)	0.1655(3)	−0.463(1)	0.115
H(21E)	4e		−0.378(2)	0.1111(2)	−0.611(1)	0.104
H(21F)	4e		−0.484(2)	0.1293(2)	−0.499(1)	0.104

Table 3. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Na(1)	4e		0.3355(5)	−0.01989(7)	−0.4073(3)	0.048(2)	0.052(2)	0.039(2)	−0.004(2)	0.001(2)	0.007(2)
Na(2)	4e		0.2403(4)	0.01173(7)	−0.0893(3)	0.034(2)	0.050(2)	0.039(2)	−0.004(2)	0.000(2)	0.002(2)
O(1W)	4e		0.3171(9)	−0.0345(1)	−0.1714(7)	0.048(4)	0.052(4)	0.051(4)	−0.012(3)	0.005(3)	−0.002(3)
O(2W)	4e		0.5132(9)	−0.0584(1)	−0.4954(7)	0.054(4)	0.055(4)	0.044(4)	−0.016(3)	−0.009(3)	0.009(3)
O(3W)	4e		0.2356(9)	0.0266(2)	−0.3335(6)	0.034(4)	0.077(5)	0.042(4)	−0.001(4)	−0.003(3)	0.016(4)
O(4W)	4e		−0.0130(9)	0.0371(2)	−0.5789(6)	0.048(4)	0.079(5)	0.042(4)	0.005(4)	−0.006(3)	0.011(4)
S(1)	4e		0.1964(4)	0.07436(5)	0.1247(2)	0.053(2)	0.036(1)	0.044(1)	−0.005(1)	−0.001(1)	0.004(1)
O(11)	4e		0.217(1)	0.1024(1)	0.0447(9)	0.079(6)	0.055(4)	0.122(7)	0.000(4)	0.026(5)	0.034(5)
O(12)	4e		0.300(1)	0.0778(2)	0.2522(8)	0.124(8)	0.121(7)	0.077(6)	0.024(6)	−0.057(6)	−0.009(5)
O(13)	4e		−0.009(1)	0.0734(1)	0.1418(8)	0.062(5)	0.069(5)	0.104(6)	−0.014(4)	0.011(4)	0.009(4)
O(14)	4e		0.277(1)	0.0535(1)	0.0396(8)	0.117(7)	0.043(4)	0.087(5)	−0.010(4)	0.041(5)	−0.027(4)
O(15)	4e		0.378(2)	0.1574(2)	0.039(1)	0.20(1)	0.093(7)	0.14(1)	−0.013(7)	0.018(8)	0.011(7)
C(13)	4e		0.357(1)	0.1853(2)	−0.017(1)	0.10(1)	0.029(6)	0.11(1)	−0.002(6)	0.010(8)	0.021(6)
C(14_2)	4e	0.67	0.530(1)	0.1936(2)	−0.105(1)	0.07(1)	0.04(1)	0.17(2)	0.004(9)	0.07(1)	0.02(1)
C(15_2)	4e	0.67	0.507(2)	0.2239(2)	−0.142(1)	0.11(2)	0.06(1)	0.14(2)	−0.02(1)	0.07(2)	0.03(1)
C(16_2)	4e	0.67	0.506(2)	0.2409(2)	−0.007(2)	0.08(2)	0.06(1)	0.17(3)	−0.02(1)	−0.03(2)	−0.01(2)
C(17_2)	4e	0.67	0.335(2)	0.2320(2)	0.083(1)	0.15(3)	0.09(2)	0.11(2)	−0.01(2)	−0.01(2)	−0.08(2)
C(18_2)	4e	0.67	0.143(2)	0.2363(2)	0.001(2)	0.08(1)	0.06(1)	0.13(2)	0.02(1)	−0.00(1)	0.03(1)
C(19_2)	4e	0.67	0.143(1)	0.2193(2)	−0.133(1)	0.08(2)	0.12(2)	0.13(2)	0.01(1)	−0.04(2)	−0.03(2)
C(110_2)	4e	0.67	0.164(1)	0.1890(2)	−0.096(1)	0.09(1)	0.08(1)	0.10(2)	−0.04(1)	−0.01(1)	−0.02(1)
C(111_2)	4e	0.67	0.315(2)	0.2282(2)	−0.223(1)	0.19(3)	0.14(2)	0.09(2)	0.01(2)	−0.04(2)	0.06(2)
C(112_2)	4e	0.67	0.357(2)	0.2018(2)	0.1211(8)	0.10(2)	0.12(2)	0.10(2)	0.02(1)	0.02(1)	0.03(1)
S(2)	4e		−0.2710(3)	0.01372(4)	−0.2269(2)	0.026(1)	0.047(1)	0.034(1)	−0.004(1)	−0.0035(9)	0.008(1)
O(21)	4e		−0.2220(9)	0.0431(1)	−0.2872(5)	0.068(4)	0.036(3)	0.029(3)	0.000(3)	−0.005(3)	0.008(3)
O(22)	4e		−0.0949(8)	0.0040(1)	−0.1575(7)	0.031(3)	0.055(4)	0.078(5)	−0.001(3)	−0.015(3)	0.013(3)
O(23)	4e		−0.3245(8)	−0.0010(1)	−0.3530(6)	0.049(4)	0.052(4)	0.038(3)	−0.009(3)	−0.004(3)	0.002(3)
O(24)	4e		−0.4264(8)	0.0167(1)	−0.1295(6)	0.030(3)	0.104(6)	0.045(4)	−0.016(4)	0.011(3)	−0.006(4)
C(21)	4e		−0.196(2)	0.0655(2)	−0.190(1)	0.106(9)	0.050(6)	0.042(6)	−0.003(6)	−0.023(6)	0.004(5)
C(22)	4e		−0.108(2)	0.0889(2)	−0.266(1)	0.094(9)	0.058(7)	0.076(8)	−0.015(6)	−0.040(7)	0.024(6)
O(25)	4e		−0.2459(9)	0.0982(1)	−0.3670(7)	0.072(5)	0.037(4)	0.064(4)	−0.012(3)	−0.030(4)	0.011(3)

Table 3. (Continued)

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(23)	4*e*		–0.194(1)	0.1224(2)	–0.4470(9)	0.055(6)	0.027(5)	0.051(6)	0.000(4)	–0.014(5)	0.003(4)
C(24)	4*e*		–0.167(2)	0.1470(2)	–0.356(1)	0.080(8)	0.039(6)	0.067(7)	0.002(5)	0.008(6)	0.000(5)
C(25)	4*e*		–0.130(2)	0.1724(2)	–0.448(1)	0.10(1)	0.035(6)	0.077(8)	–0.021(6)	0.006(7)	–0.007(5)
C(26)	4*e*		–0.301(2)	0.1768(2)	–0.545(1)	0.10(1)	0.042(6)	0.090(9)	0.005(6)	0.007(8)	0.025(6)
C(27)	4*e*		–0.328(2)	0.1520(2)	–0.638(1)	0.085(9)	0.069(8)	0.09(1)	–0.014(7)	–0.042(8)	0.040(7)
C(28)	4*e*		–0.143(2)	0.1471(3)	–0.721(1)	0.14(1)	0.076(9)	0.060(8)	–0.011(9)	0.004(9)	0.000(7)
C(29)	4*e*		0.031(2)	0.1435(3)	–0.619(1)	0.077(9)	0.10(1)	0.09(1)	–0.004(8)	0.042(8)	0.017(8)
C(210)	4*e*		–0.010(2)	0.1180(2)	–0.530(1)	0.079(9)	0.083(9)	0.092(9)	0.030(7)	0.013(7)	0.006(7)
C(211)	4*e*		0.054(2)	0.1681(3)	–0.528(1)	0.075(9)	0.09(1)	0.10(1)	–0.034(8)	–0.005(8)	0.026(8)
C(212)	4*e*		–0.364(2)	0.1270(2)	–0.550(1)	0.079(8)	0.062(7)	0.098(9)	–0.017(6)	–0.035(7)	0.026(7)

References

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2. Sheldrick, G. M.: SHELXL-93, a program for refining crystal structures. University of Göttingen, Germany 1993.