Crystal structure of bis(μ_2-aqua)diaquasodium 4-azidobenzenesulfonate monohydrate, $Na(H_2O)_4[N_3C_6H_4SO_3] \cdot H_2O$

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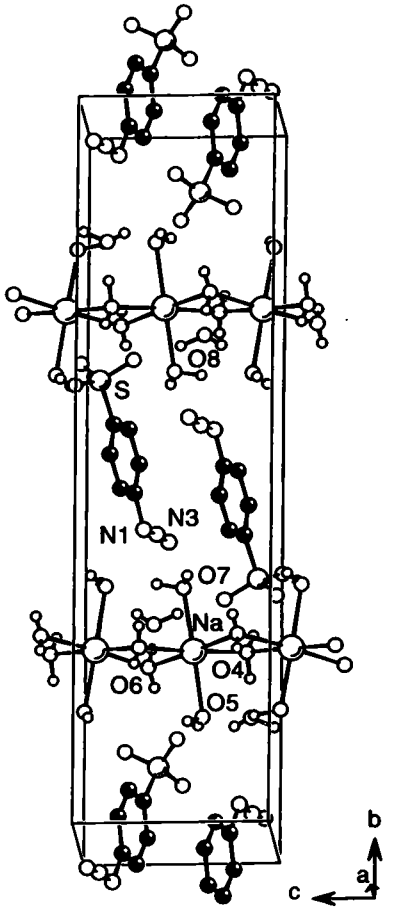

Abstract

$C_6H_{14}N_3NaO_8S$, monoclinic, $P12_1/c1$ (no. 14), $a = 7.018(1)$ Å, $b = 27.193(5)$ Å, $c = 7.089(1)$ Å, $\beta = 92.01(2)°$, $V = 1352.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.071$, $wR_{ref}(F^2) = 0.160$, $T = 193$ K.

Source of material

The compound was obtained along with sodium 4-pentazolylbenzenesulfonate from 4-diazoniumbenzenesulfonate and sodium azide in methanol at 223 K; for details see [1]. To 60 mL of the obtained methanolic solution 10 mL of water and 0.70 mL of hydrochloric acid (37 %) were added and then extracted first with 30 mL of dichloromethane and then with 30 mL of diethylether. From the methanol/water phase the title compound crystallized at 228 K in the course of 6 months.

Discussion

The azido group is nearly coplanar with the benzene ring. All bond lengths correspond to those commonly observed for similar compounds and correspond to those of the anhydrous compound [2]. Each Na^+ ion is coordinated by six O atoms of four symmetry-inequivalent water molecules in a nearly octahedral environment at distances of 2.373(4) Å to 2.450(4) Å. The edge-sharing octahedra form linear chains along [001]. There is an additional, not coordinated molecule of water (atom O8), which is joined by hydrogen bonds to two of the coordinated water molecules.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.06 × 0.15 × 0.60 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.09 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS-2, ω scans at $\varphi = 0°$ and $\varphi = 60°$, $\Delta\omega = 1°$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9943, 2363
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1741
$N(param)_{refined}$:	228
Program:	SHELX-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4e	0.890(8)	0.520(2)	0.895(7)	0.06(2)
H(3)	4e	0.705(6)	0.447(2)	0.803(6)	0.04(1)
H(5)	4e	0.241(9)	0.536(2)	0.655(8)	0.07(2)
H(6)	4e	0.416(7)	0.606(2)	0.735(6)	0.05(1)
H(4A)	4e	0.522(9)	0.785(3)	0.382(9)	0.10(3)
H(4B)	4e	0.573(8)	0.729(2)	0.365(7)	0.06(2)
H(5A)	4e	0.815(9)	0.149(3)	0.426(9)	0.10(3)
H(5B)	4e	0.671(9)	0.151(3)	0.382(9)	0.07(4)
H(6A)	4e	0.030(9)	0.283(3)	0.190(9)	0.07(2)
H(6B)	4e	0.069(9)	0.234(3)	0.165(9)	0.10(3)
H(7A)	4e	0.862(8)	0.347(2)	0.432(8)	0.04(2)
H(7B)	4e	0.763(9)	0.341(3)	0.59(1)	0.12(3)
H(8A)	4e	0.720(8)	0.672(2)	0.278(8)	0.05(2)
H(8B)	4e	0.736(9)	0.671(3)	0.483(9)	0.08(2)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Na	4*e*	0.7543(2)	0.24841(6)	0.4039(2)	0.0393(9)	0.0434(9)	0.0395(9)	–0.0007(7)	–0.0002(7)	–0.0005(7)
S	4*e*	0.7919(2)	0.62140(4)	0.8892(2)	0.0396(6)	0.0388(6)	0.0385(6)	–0.0020(5)	–0.0017(4)	0.0008(5)
O(1)	4*e*	0.9567(5)	0.6055(1)	1.0048(5)	0.044(2)	0.047(2)	0.056(2)	–0.001(2)	–0.012(2)	0.003(2)
O(2)	4*e*	0.8450(5)	0.6460(1)	0.7155(4)	0.053(2)	0.048(2)	0.041(2)	–0.008(2)	0.003(2)	0.005(2)
O(3)	4*e*	0.6597(5)	0.6522(1)	0.9926(4)	0.049(2)	0.040(2)	0.048(2)	0.003(2)	0.006(2)	–0.007(2)
C(1)	4*e*	0.6649(6)	0.5681(2)	0.8237(6)	0.039(2)	0.040(2)	0.034(2)	–0.001(2)	–0.001(2)	0.002(2)
C(2)	4*e*	0.7505(7)	0.5223(2)	0.8388(6)	0.043(3)	0.043(2)	0.041(2)	0.002(2)	0.000(2)	0.009(2)
C(3)	4*e*	0.6453(7)	0.4801(2)	0.7906(7)	0.049(3)	0.040(2)	0.048(3)	0.003(2)	0.004(2)	–0.002(2)
C(4)	4*e*	0.4577(6)	0.4846(2)	0.7298(6)	0.045(3)	0.044(2)	0.036(2)	–0.003(2)	0.004(2)	–0.001(2)
C(5)	4*e*	0.3704(7)	0.5301(2)	0.7122(6)	0.045(3)	0.047(3)	0.040(2)	0.001(2)	–0.002(2)	–0.002(2)
C(6)	4*e*	0.4751(7)	0.5723(2)	0.7592(6)	0.044(3)	0.043(3)	0.041(2)	0.002(2)	0.001(2)	0.001(2)
N(1)	4*e*	0.3610(6)	0.4396(1)	0.6865(6)	0.047(2)	0.042(2)	0.054(2)	–0.003(2)	0.002(2)	–0.003(2)
N(2)	4*e*	0.1905(7)	0.4429(2)	0.6298(5)	0.058(3)	0.037(2)	0.044(2)	–0.003(2)	0.006(2)	–0.005(2)
N(3)	4*e*	0.0360(7)	0.4407(2)	0.5769(6)	0.047(3)	0.059(3)	0.066(3)	–0.007(2)	–0.003(2)	–0.002(2)
O(4)	4*e*	0.5281(5)	0.2594(2)	0.1467(5)	0.040(2)	0.046(2)	0.047(2)	0.000(2)	–0.003(1)	0.003(2)
O(5)	4*e*	0.7288(8)	0.1594(2)	0.3563(7)	0.044(2)	0.047(2)	0.055(2)	0.003(2)	–0.003(2)	0.002(2)
O(6)	4*e*	0.9855(5)	0.2414(2)	0.6675(5)	0.040(2)	0.043(2)	0.049(2)	0.002(2)	0.001(1)	0.004(2)
O(7)	4*e*	0.7772(6)	0.3345(1)	0.4552(6)	0.049(2)	0.043(2)	0.059(2)	0.000(2)	0.002(2)	0.002(2)
O(8)	4*e*	0.7401(5)	0.6852(1)	0.3629(6)	0.056(2)	0.044(2)	0.042(2)	–0.002(2)	–0.006(2)	–0.002(2)

References

1. Biesemeier, F.; Harms, K.; Müller, U.: Pentazol-Derivate und daraus entstehende Azide. Z. Anorg. Allg. Chem. **630** (2004) in press.
2. Biesemeier, F.; Harms, K.; Müller, U.: Crystal structure of sodium 4-azidobenzenesulfonate, $Na[N_3C_6H_4SO_3]$. Z. Kristallogr. NCS **219** (2004) 37-38.
3. Sheldrick, G. M.: SHELX-97. A program package for crystal structure solution and refinement. University of Göttingen, Germany 1997.