Crystal structure of sodium 4-azido-1,2,3-triazolide monohydrate, $Na(N_3C_2HN_3) \cdot H_2O$

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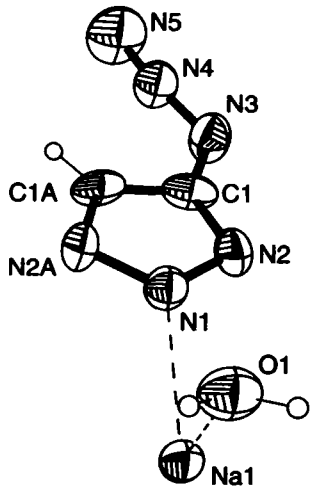
Received August 1, 2005, accepted and available on-line December 5, 2005; CCDC no. 1267/1616



Abstract

$C_2H_3N_6NaO$, monoclinic, $P12_1/m1$ (no. 11), $a = 3.6146(8)$ Å, $b = 7.146(1)$ Å, $c = 11.258(2)$ Å, $\beta = 92.02(1)°$, $V = 290.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.042$, $wR_{ref}(F^2) = 0.110$, $T = 233$ K.

Source of material

The sodium salt of 4-azido-1,2,3-triazole [1,2] was obtained as a hydrate by refluxing perfluoroallylbenzene [CA-registry no. 67899-41-6] with an excess of sodium azide in moist acetone. Crystals of this unexpected product were collected in low yield after filtration and evaporization of the solvent. This compound is certainly an energetic material, but no hazard assessment has been conducted so far.

Experimental details

Thin plate with thickness of 0.01 mm and its small X-ray scattering power led to a reduced number of observed reflections and a low N_{gt}/N_{param} ratio. Hydrogen atom at C1 (or C1A) could not be found from Fourier difference map because of the overlying with N3 from the disordered azido group. Therefore, it was calculated geometrically. The water hydrogen atom (with O1 in a special position) was found and refined with an isotropic displacement parameter and an O—H bond restraint of 0.85 Å.

Discussion

The title compound crystallizes centrosymmetrically with a positional disorder of the azido group N3-N4-N5 by a mirror plane through the atoms O1, Na1, N1 and N5. The nitrogen atoms N3 and N4 in general positions were refined with an occupancy of 0.5. In the figure the disordered azido group at C1A and the disordered hydrogen atom at C1 were omitted for clarity. The azido group is not linear with an N3–N4–N5 angle of 172.5° (maybe an indication that the nitrogen atom N5 lies not exactly on the mirror plane), and it is tilted slightly out of the ring plane. Hydrogen bridges are observed between the water molecule and the azido group with d(O1–H···N3′) = 2.009 Å. The sodium ion has a distorted octahedral coordination by four nitrogen atoms from different azolide rings and two oxygen atoms from water molecules with distances between 2.347 Å – 2.721 Å and deviations from the right angle of ±10°. By the coordination of the sodium ion a monolayer is formed and the azido groups penetrate into the adjacent layers which are stacked along the c axis.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.01 × 0.25 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.00 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	43.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1328, 392
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 342
$N(param)_{refined}$:	66
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1O)	4*f*		0.76(1)	–0.162(5)	0.782(3)	0.10(2)
H(1)	4*f*	0.5	0.4769	–0.0796	0.1971	0.048

Table 3. 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)	2*e*		1.2529(4)	–¼	0.5939(1)	0.040(1)	0.034(1)	0.038(1)	0	–0.0074(7)	0
O(1)	2*e*		0.7817(9)	–¼	0.7321(3)	0.049(2)	0.064(3)	0.039(2)	0	–0.005(2)	0
N(1)	2*e*		0.7784(9)	–¼	0.4267(3)	0.040(2)	0.035(2)	0.034(2)	0	–0.002(2)	0

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Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(2)	4*f*		0.6961(7)	–0.0979(3)	0.3630(2)	0.046(2)	0.026(1)	0.054(2)	0.004(1)	0.000(1)	0.004(1)
C(1)	4*f*		0.5604(7)	–0.1563(5)	0.2603(2)	0.034(2)	0.065(2)	0.021(2)	0.004(1)	–0.008(1)	0.009(1)
N(3)	4*f*	0.5	0.427(2)	–0.0824(8)	0.1552(4)	0.065(3)	0.039(3)	0.042(3)	0.000(3)	–0.007(3)	–0.003(3)
N(4)	4*f*	0.5	0.275(1)	–0.1802(7)	0.0767(5)	0.057(3)	0.044(4)	0.039(3)	–0.001(2)	–0.005(3)	0.002(2)
N(5)	2*e*		0.131(2)	–¼	0.0007(4)	0.097(4)	0.086(4)	0.045(3)	0	–0.025(3)	0

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