Crystal structure of *catena*-(hexaaqua-tetrakis(isocyanurato)nickel-disodium), $Na_2Ni(H_2O)_6(C_3N_3O_3)_4$

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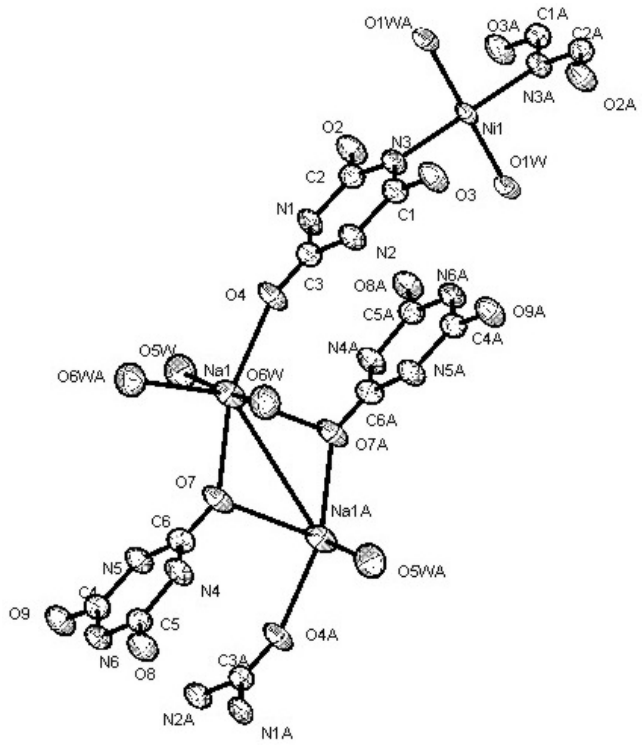

Abstract

$C_{12}H_{20}N_{12}Na_2NiO_{18}$, triclinic, $P\bar{1}$ (no. 2), a = 6.760(1) Å, b = 10.443(2) Å, c = 10.455(2) Å, α = 62.83(3)°, β = 83.72(3)°, γ = 71.82(3)°, V = 623.4 Å^3, Z = 1, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.075, T = 293 K.

Source of material

All commercially available reagents were used as supplied. A solution of cyanuric acid (0.01 mol, 1.29 g) in ethanol (10 ml) was added slowly to a solution of sodium hydroxide (0.03 mol, 1.2 g) and nickel(II) chloride (0.015 mol, 1.93 g) in ethanol. The reaction mixture was refluxed for 4 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried in vacuo (yield 86 %). Elemental analysis — found: C, 17.90 %; H, 2.54 %; N, 22.40 %; calculated for $C_{12}H_{20}N_{12}Na_2NiO_{18}$: C, 17.84 %; H, 2.50 %; N, 22.56 %. A ethanol solution of the title compound was slowly evaporated and purple block crystals were obtained after one week.

Experimental details

H atoms of water except H1WA were found in the difference Fourier map and refined freely. The other H atoms were fixed geometrically and allowed to ride on their attached atoms, with d(C—H) = 0.97 Å, d(N—H) = 0.86 Å, and U_{iso}(H) = 1.2 - 1.5 U_{eq}(parent), respectively.

Discussion

Coordination compounds of the cyanuric acid have received considerable attention in the literature. They are attractive from several points of view, in particular as the possibility of molecular self-assembly [1].

The nickel atoms in the title crystal structure are four-coordinated by a pair of water molecules with d(Ni—O) 1.934(2) Å and a pair of nitrogen atoms with d(Ni—N) 1.950(2) Å. The sodium ions are six-coordinated by three oxygen atoms of water molecules and three oxygen atoms with d(Na—O) range from 2.465(3) to 2.600(2) Å. The polymer-like chains are linked by the ligand oxygen atoms and sodium ions. All the bond lengths and angles are in the normal range comparable with similar structure reported previously [2].

Table 1. Data collection and handling.

Crystal:	purple block, size 0.18 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.62 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3471, 2305
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2143
$N(param)_{refined}$:	225
Programs:	WinGX [3], NRCVAX [4], SHELXS-97 [5], SHELXL-97 [6], SHELXTL [7]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.6140	0.1448	0.8492	0.028
H(1A)	2*i*	0.2968	0.4726	0.5194	0.028
H(5A)	2*i*	–0.1905	0.8753	0.9588	0.030
H(4A)	2*i*	0.1508	0.5537	1.2781	0.031
H(1WA)	2*i*	0.2265	–0.0997	0.6111	0.064
H(5WA)	2*i*	0.061(5)	0.728(4)	0.612(4)	0.08(1)
H(6WA)	2*i*	0.551(6)	0.239(5)	1.101(4)	0.09(1)
H(5WB)	2*i*	–0.046(6)	0.793(4)	0.692(4)	0.09(1)
H(6WB)	2*i*	0.453(6)	0.316(4)	1.176(4)	0.09(1)
H(1WB)	2*i*	0.190(5)	0.029(4)	0.653(3)	0.060(9)

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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}
Ni(1)	1*f*	½	0	½	0.0230(2)	0.0144(2)	0.0162(2)	–0.0026(1)	–0.0013(1)	–0.0100(1)
Na(1)	2*i*	0.2592(1)	0.5302(1)	0.90125(9)	0.0378(5)	0.0345(5)	0.0351(5)	–0.0062(4)	0.0016(4)	–0.0231(4)
O(9)	2*i*	–0.2843(2)	1.0646(2)	1.0435(2)	0.0394(8)	0.0223(8)	0.0273(8)	0.0008(6)	–0.0071(6)	–0.0121(6)
O(3)	2*i*	0.7041(2)	–0.0464(2)	0.7636(2)	0.0416(9)	0.0208(8)	0.0269(8)	0.0027(7)	–0.0084(6)	–0.0114(6)
O(8)	2*i*	0.1067(2)	0.6747(2)	1.4318(2)	0.0346(8)	0.0264(8)	0.0233(7)	–0.0010(6)	–0.0069(6)	–0.0124(6)
O(4)	2*i*	0.4113(2)	0.4102(2)	0.7597(2)	0.0408(9)	0.0291(8)	0.0334(8)	–0.0051(7)	–0.0007(7)	–0.0234(7)
N(3)	2*i*	0.5110(2)	0.1438(2)	0.5673(2)	0.0255(8)	0.0192(8)	0.0205(8)	–0.0043(7)	–0.0011(6)	–0.0113(7)
O(7)	2*i*	0.0174(2)	0.6121(2)	1.0437(2)	0.0405(8)	0.0323(8)	0.0327(8)	–0.0040(7)	–0.0013(7)	–0.0243(7)
N(6)	2*i*	–0.0914(2)	0.8767(2)	1.2422(2)	0.0260(8)	0.0222(9)	0.0222(8)	–0.0034(7)	–0.0011(7)	–0.0130(7)
N(2)	2*i*	0.5610(3)	0.1811(2)	0.7642(2)	0.0279(8)	0.0236(9)	0.0206(8)	–0.0043(7)	–0.0035(7)	–0.0130(7)
N(1)	2*i*	0.3676(3)	0.3793(2)	0.5648(2)	0.0289(9)	0.0169(8)	0.0235(8)	–0.0019(7)	–0.0027(7)	–0.0110(7)
O(6W)	2*i*	0.5020(3)	0.3313(2)	1.1026(2)	0.051(1)	0.033(1)	0.038(1)	–0.0123(8)	0.0005(8)	–0.0140(8)
C(3)	2*i*	0.4450(3)	0.3288(2)	0.7006(2)	0.0232(9)	0.023(1)	0.023(1)	–0.0078(8)	0.0029(8)	–0.0130(8)
O(5W)	2*i*	0.0737(3)	0.7453(2)	0.6787(2)	0.039(1)	0.039(1)	0.042(1)	–0.0111(8)	–0.0041(8)	–0.0203(8)
N(5)	2*i*	–0.1343(3)	0.8391(2)	1.0429(2)	0.0316(9)	0.0232(9)	0.0196(8)	–0.0029(7)	–0.0039(7)	–0.0120(7)
C(6)	2*i*	–0.0130(3)	0.6938(2)	1.1031(2)	0.023(1)	0.026(1)	0.025(1)	–0.0048(8)	0.0015(8)	–0.0143(9)
C(5)	2*i*	0.0297(3)	0.7324(2)	1.3085(2)	0.0207(9)	0.024(1)	0.023(1)	–0.0061(8)	0.0024(7)	–0.0135(8)
N(4)	2*i*	0.0695(3)	0.6444(2)	1.2364(2)	0.0294(9)	0.0211(9)	0.0260(9)	0.0009(7)	–0.0045(7)	–0.0135(7)
C(4)	2*i*	–0.1730(3)	0.9325(2)	1.1090(2)	0.0221(9)	0.023(1)	0.023(1)	–0.0060(8)	0.0011(8)	–0.0123(8)
C(2)	2*i*	0.3966(3)	0.2892(2)	0.4968(2)	0.0222(9)	0.024(1)	0.022(1)	–0.0073(8)	0.0019(7)	–0.0122(8)
C(1)	2*i*	0.5983(3)	0.0866(2)	0.7002(2)	0.0237(9)	0.023(1)	0.022(1)	–0.0071(8)	0.0020(8)	–0.0125(8)
O(2)	2*i*	0.3166(2)	0.3407(2)	0.3756(2)	0.0392(8)	0.0261(8)	0.0235(7)	–0.0010(6)	–0.0085(6)	–0.0133(6)
O(1W)	2*i*	0.2451(3)	–0.0215(2)	0.6002(2)	0.050(1)	0.049(1)	0.059(1)	–0.0286(9)	0.0286(9)	–0.045(1)

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