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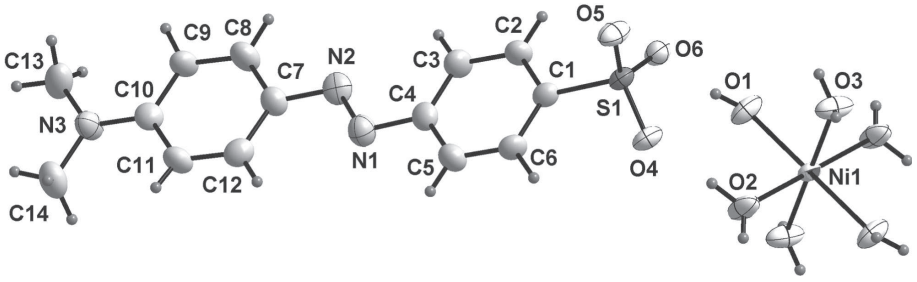
Crystal structure of hexaaquanickel(II) bis((*E*)-4-((4-(dimethylamino)phenyl)diazenyl)benzenesulfonate), $C_{28}H_{40}N_6NiO_{12}S_2$



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Abstract

$C_{28}H_{40}N_6NiO_{12}S_2$, monoclinic, $P2_1/c$ (no. 14), $a = 6.202(1)$ Å, $b = 7.126(1)$ Å, $c = 38.499(2)$ Å, $\beta = 91.258(2)°$, $V = 1701.1(4)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0698$, $wR_{ref}(F^2) = 0.1515$, $T = 300$ K.

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The cation and the anion forming the title crystal structure are shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

$NiCl_2{\cdot}6H_2O$ (0.364 g) was added to a solution of $C_{14}H_{14}N_3NaO_3S$ (0.491 g) in methanol (200 mL) and the mixture was slowly evaporated. Small red-brown crystals were obtained after approximately 1 week.

Table 1: Data collection and handling.

Crystal:	Red-brown prism
Size:	0.57 × 0.48 × 0.37 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	7.6 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	34163, 3000, 0.119
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2062
$N(param)_{refined}$:	283
Programs:	SHELX [1], Bruker programs [2], SIR92 [3], DIAMOND [4], publCIF [5]

Experimental details

All H atoms were located in a difference Fourier map and refined with $U_{iso}(H) = 1.2$ of their parent atoms with the exception of H atoms in the methyl groups and water molecules. H atoms in methyl groups and water molecules were refined using $U_{iso}(H) = 1.5U_{eq}(C)$ or $1.5U_{eq}(O)$.

Comment

Recently, owing to the various properties, for example, a low-dimensional magnetism, photovoltaic effects, and thermoelectric applications, inorganic-organic hybrid systems have attracted considerable interest [6–8]. As a part of this approach, we prepared a series of inorganic-organic layered materials [9–11].

In the layered title structure $Ni(H_2O)_6$ layers are placed between organic bilayers. The crystal structure is similar to

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	1.0000	0.5000	0.5000	0.0265(3)
S1	0.61159(17)	−0.0048(2)	0.43946(3)	0.0311(3)
O1	0.7066(6)	0.4952(7)	0.47613(11)	0.0443(10)
H1O1	0.654(12)	0.588(10)	0.4697(19)	0.066*
H2O1	0.641(12)	0.410(10)	0.4665(19)	0.066*
O2	1.1044(7)	0.2850(7)	0.46915(14)	0.0510(14)
H1O2	1.007(12)	0.209(11)	0.4599(19)	0.077*
H2O2	1.213(13)	0.261(12)	0.465(2)	0.077*
O3	0.8978(8)	0.3037(7)	0.53500(15)	0.0494(13)
H1O3	0.783(13)	0.256(11)	0.535(2)	0.074*
H2O3	0.958(13)	0.234(12)	0.538(2)	0.074*
O4	0.8461(5)	−0.0042(6)	0.44346(9)	0.0405(9)
O5	0.5172(7)	−0.1773(6)	0.45212(11)	0.0444(11)
O6	0.5158(6)	0.1616(6)	0.45484(10)	0.0413(10)
N1	0.4072(8)	0.0013(8)	0.28748(12)	0.0491(12)
N2	0.2172(8)	0.0427(5)	0.27879(12)	0.0403(12)
N3	−0.0081(7)	0.0152(8)	0.13739(11)	0.0467(12)
C1	0.5524(7)	0.0051(8)	0.39443(13)	0.0329(11)
C2	0.3503(9)	0.0698(8)	0.38326(16)	0.0389(14)
H2	0.255(10)	0.095(8)	0.4010(15)	0.047*
C3	0.2987(10)	0.0709(9)	0.34817(16)	0.0448(15)
H3	0.159(10)	0.115(9)	0.3399(16)	0.054*
C4	0.4466(9)	0.0127(9)	0.32395(14)	0.0393(12)
C5	0.6476(10)	−0.0542(8)	0.33564(16)	0.0430(15)
H5	0.738(10)	−0.084(9)	0.3192(16)	0.052*
C6	0.7007(9)	−0.0555(8)	0.37060(15)	0.0394(14)
H6	0.845(10)	−0.091(8)	0.3788(15)	0.047*
C7	0.1724(9)	0.0309(8)	0.24258(14)	0.0405(14)
C8	−0.0348(11)	0.0781(10)	0.23174(17)	0.0489(16)
H8	−0.135(10)	0.107(9)	0.2493(17)	0.059*
C9	−0.0968(10)	0.0725(9)	0.19712(15)	0.0435(15)
H9	−0.250(10)	0.101(9)	0.1916(15)	0.052*
C10	0.0481(9)	0.0158(8)	0.17194(14)	0.0386(12)
C11	0.2549(10)	−0.0420(9)	0.18356(16)	0.0462(16)
H11	0.337(10)	−0.103(9)	0.1699(16)	0.055*
C12	0.3165(10)	−0.0368(8)	0.21804(16)	0.0430(15)
H12	0.444(10)	−0.051(9)	0.2238(16)	0.052*
C13	−0.2199(12)	0.0699(12)	0.1255(2)	0.059(2)
H13A	−0.330(13)	−0.018(12)	0.133(2)	0.088*
H13B	−0.257(13)	0.180(12)	0.134(2)	0.088*
H13C	−0.244(13)	0.020(11)	0.104(2)	0.088*
C14	0.1426(14)	−0.0469(12)	0.11170(19)	0.061(2)
H14A	0.270(13)	0.032(12)	0.111(2)	0.092*
H14B	0.183(13)	−0.170(12)	0.115(2)	0.092*
H14C	0.061(13)	−0.048(11)	0.092(2)	0.092*

the previously reported Mn analogue [11]. Many crystal structures containing methyl-orange have been reported [12–15]. Through the hydrogen bonds all water ligands are linked and as a result hydrogen bonded layers are in an *ab* plane. Based on the bond lengths, each nickel(II) is almost octahedrally surrounded by six water molecules. The distances between Ni—O range from 2.02(3) Å to 2.052(5) Å. Compared to the Mn compound, the difference is very tiny. Organic chains are parallel to the *c* axis. The hydrogen bonds in the title compound are classified as medium strong [16, 17]. Similar to the Mn compound, the C—N bond lengths are shorter than the sum of the covalent radii, 1.47 Å (N1—C4 = 1.422 Å, N2—C7 = 1.418 Å, N3—C10 = 1.367 Å). Some 'sacrificial' structures can explain this shortening of the bond lengths [11, 18–20].

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