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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(C-C)$ = 0.002 Å
R factor = 0.027
wR factor = 0.066
Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

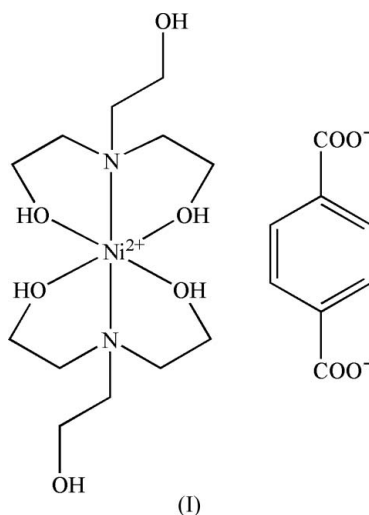
Bis(triethanolamine- κ^3N,O,O')nickel(II) benzene-1,4-dicarboxylate

The title compound, $[Ni(C_6H_{15}NO_3)_2](C_8H_4O_4)$, consists of a $[Ni(H_3tea)_2]^{2+}$ cation (H_3tea is triethanolamine) and a benzene-1,4-dicarboxylate (terephthalate) anion. The Ni^{II} atom lies on an inversion centre and exhibits a slightly distorted octahedral coordination geometry formed by two molecules of H_3tea acting as N,O,O' -tridentate ligands; the anion is also centrosymmetric. All the OH groups of H_3tea are involved in an $O-H \cdots O$ hydrogen-bonding network with terephthalate O atoms, thus linking the molecular units in chains and forming a two-dimensional infinite network.

Comment

Recently, we reported (Kirillov, Kopylovich, Kirillova, Haukka *et al.*, 2005; Kirillov, Kopylovich, Kirillova, Karabach *et al.*, 2005) a self-assembled synthesis of the one-dimensional coordination polymer $\{[Cu_2(H_2tea)_2\{\mu-C_6H_4(COO)_{2-1,4}\} \cdot 2H_2O]_n$ from copper(II) nitrate, triethanolamine (H_3tea), terephthalic acid and sodium hydroxide in an aqueous solution. This compound was found to act as a catalyst for the mild peroxidative oxidation of alkanes. This study was extended by preparing related compounds with other metals. Thus, by using nickel(II) nitrate instead of the copper salt in the above synthesis, a pale-blue crystalline material was isolated and characterized by IR spectroscopy, elemental and single-crystal X-ray diffraction analyses, which reveal the formation of the title compound $[Ni(H_3tea)_2][C_6H_4(COO)_{2-1,4}]$, (I), instead of the expected polymeric material.

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The crystal structure of (I) (Fig. 1) is composed of an $[Ni(H_3tea)_2]^{2+}$ cation, with slightly distorted centrosymmetric octahedral coordination geometry formed by two tridentate

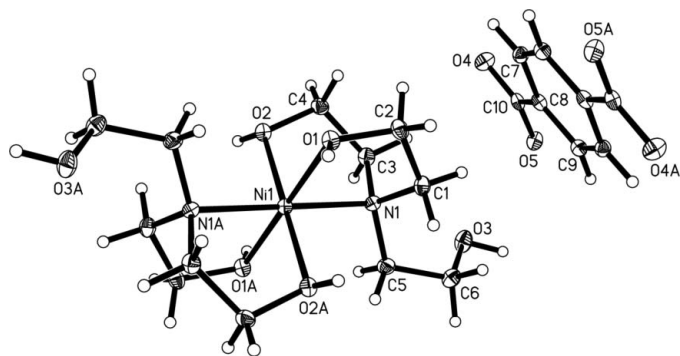


Figure 1

The molecular structure of the title compound, showing the atom-labeling scheme. The suffixes A in the cation and anion correspond to the symmetry position $(2-x, -y, 1-z)$. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

H_3tea ligands, and a centrosymmetric benzene-1,4-dicarboxylate dianion. Most of the bond parameters within the $[Ni(H_3tea)_2]^{2+}$ moiety (Table 1) agree with those of the related nickel complexes with different anions such as nitrate (Nielsen *et al.*, 1972), chloride (Icbudak *et al.*, 1995), acetate (Krabbes *et al.*, 2000), squarate (Yesilel *et al.*, 2004), saccharinate (Topcu *et al.*, 2001) and dithiodisalicylate (Ramalingam *et al.*, 1987; Marsh & Spek, 2001). All the OH groups of H_3tea are linked by $O-H \cdots O$ hydrogen bonds (Table 2) to O atoms of the terephthalate anions, leading to double chains and thus forming a two-dimensional polymeric network (Fig. 2).

Experimental

To an aqueous solution (10 ml) of $Ni(NO_3)_2 \cdot 6H_2O$ (290 mg, 1 mmol) in HNO_3 (1 mmol) (the acid was added to avoid a spontaneous hydrolysis of the metal salt) were added dropwise triethanolamine (0.27 ml, 2 mmol) and then an aqueous solution (2 ml) of NaOH (80 mg, 2 mmol) with continuous stirring at room temperature. Terephthalic acid (166 mg, 1 mmol) was dissolved in an aqueous solution (2 ml) of NaOH (80 mg, 2 mmol) and added to the resulting green cloudy solution. The reaction mixture was stirred for 10 h and then filtered off. The filtrate was left to evaporate in a beaker at ambient temperature. Pale-blue X-ray-quality crystals were formed in 3 d and were collected and dried in air (yield 76%, based on nickel nitrate). They are insoluble in common solvents. Analysis calculated for $C_{20}H_{34}N_2NiO_{10}$: C 46.09, H 6.58, N 5.37%; found: C 46.01, H 6.97, N 5.41%. FT-IR, selected bands, cm^{-1} : 3426 [$s, \nu(OH)$], 2990 [$m, \nu_{as}(CH)$], 2902 [$m, \nu_s(CH)$], 1531 [$s, \nu_{as}(COO)$], 1379 [$s, \nu_s(COO)$], 1058 [$s, \nu(C-O)$].

Crystal data

$[Ni(C_6H_{15}NO_3)_2](C_8H_4O_4)$	$Z = 1$
$M_r = 521.20$	$D_x = 1.591 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.8910$ (2) Å	Cell parameters from 7109 reflections
$b = 8.6355$ (2) Å	$\theta = 4.2\text{--}27.5^\circ$
$c = 9.2002$ (3) Å	$\mu = 0.95 \text{ mm}^{-1}$
$\alpha = 89.420$ (1)°	$T = 100$ (2) K
$\beta = 72.741$ (1)°	Pale blue, block
$\gamma = 66.316$ (1)°	$0.26 \times 0.22 \times 0.06 \text{ mm}$
$V = 543.99$ (3) Å ³	

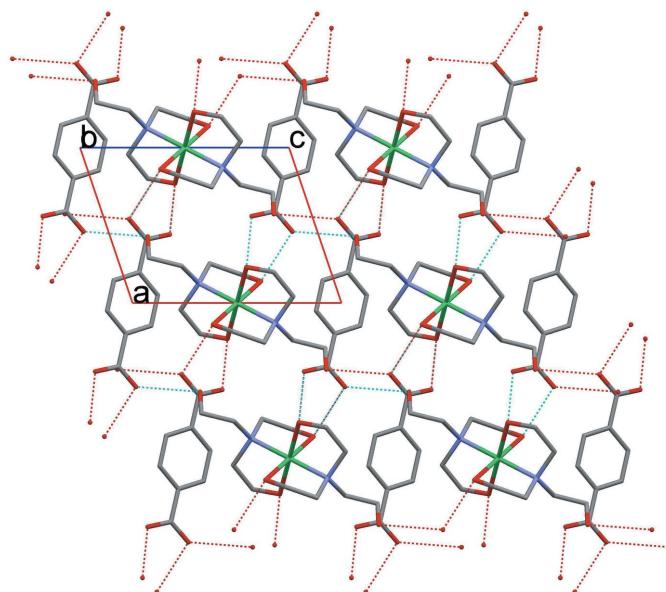


Figure 2

Fragment of the crystal packing of compound (I), viewed down the b axis, illustrating the hydrogen-bonding (dashed lines) pattern.

Data collection

Nonius KappaCCD diffractometer	2463 independent reflections
φ scans and ω scans with κ offsets	2294 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.033$
(<i>XPRED</i> in <i>SHELXTL</i> ;	$\theta_{max} = 27.5^\circ$
Sheldrick, 2005)	$h = -10 \rightarrow 10$
$T_{min} = 0.790, T_{max} = 0.945$	$k = -11 \rightarrow 11$
7109 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0156P)^2 + 0.3221P]$
$R[F^2 > 2\sigma(F^2)] = 0.027$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.066$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.46 \text{ e \AA}^{-3}$
2463 reflections	$\Delta\rho_{min} = -0.43 \text{ e \AA}^{-3}$
152 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Ni1—O1	2.0696 (10)	Ni1—N1	2.1008 (12)
Ni1—O2	2.0839 (10)		
O1 ⁱ —Ni1—O1	180.0	O1—Ni1—N1	83.35 (4)
O1—Ni1—O2	90.19 (4)	O2—Ni1—N1	83.92 (4)
O2—Ni1—O2 ⁱ	180.0	N1 ⁱ —Ni1—N1	179.999 (1)

Symmetry codes: (i) $-x + 2, -y, -z + 1$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1O \cdots O4 ⁱⁱ	0.92	1.66	2.5744 (14)	175
O3—H3O \cdots O5 ⁱⁱⁱ	0.95	1.95	2.8491 (15)	158
O2—H2O \cdots O5 ^{iv}	0.91	1.72	2.6272 (14)	172

Symmetry codes: (ii) $x + 1, y - 1, z$; (iii) $-x + 1, -y + 1, -z$; (iv) $-x + 1, -y + 1, -z + 1$.

Hydroxy atoms H10–H30 were located in a difference Fourier map and then constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent atom})$. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2005); software used to prepare material for publication: *SHELXL97*.

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