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Crystal structure of dichlorido(pyridine-κN)(2,4,6-tri-2-pyridyl-1,3,5-triazine-κ⁴N²,N¹,N⁶)nickel(II), C₂₃H₁₇Cl₂N₇Ni

Abstract

C₂₃H₁₇Cl₂N₇Ni, monoclinic, P₂₁/c (no. 14), a = 12.5392(5) Å, b = 11.5655(6) Å, c = 15.5085(8) Å, β = 102.4634(17)°, V = 2196.07(18) Å³, Z = 4, Rgt(F) = 0.0348, wRref(F²) = 0.0792, T = 223(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

To a solution of NiCl₂·6H₂O (0.2670 g, 1.123 mmol) in EtOH (30 mL) was added 2,4,6-tri-2-pyridyl-1,3,5-triazine (tptz; 0.2814 g, 0.901 mmol) and stirred for 12 h at room temperature. The formed precipitate was separated by filtration, washed with EtOH and acetone, and dried at 50 °C, to give a pale green powder (0.3363 g). Crystals were obtained by slow evaporation from a pyridine (py) solution at 60 °C.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.94 Å and Uiso(H) = 1.2 Ueq(C) with the help of the SHELXL program (AFIX 43 option) [2]. The highest peak (0.36 e Å⁻³) and the deepest hole (−0.24 e Å⁻³) in the difference Fourier map are located 1.19 Å and 1.02 Å from the atoms Ni₁ and C₂, respectively.

Comment

This contribution is part of my continuing interest in the structural chemistry of metal complexes containing tptz ligand [5–8]. Single crystals of the title compound [NiCl₂(py)(tptz)] were unexpectedly obtained during crystallization from a pyridine solution of the reaction product.

In the title complex, the central Ni(II) ion is six-coordinated in a distorted octahedral manner by three N atoms of the tridentate tptz ligand, two Cl atoms and one N atom from the pyridine ligand. The tight N—Ni—N chelating angles (<N₁—Ni₁—N₄ = 77.23(8)° and <N₁—Ni₁—N₆ = 76.73(8)°) significantly contribute the distortion of the octahedron. The apical Cl₁—Ni₁—Cl₂, N₁—Ni₁—N₇, and N₄—Ni₁—N₆ bond angles are 172.62(2)°, 176.66(7)° and 153.93(8)°, respectively. The Ni—N₄/N₆(pyridyl) bonds (2.128(2) Å).
and 2.152(2) Å) are slightly longer than the Ni—N$_{triazine}$ bond (1.978(2) Å) and Ni—N$_{py}$ bond (2.076(2) Å). In the crystal structure, the two pyridyl rings coordinated to the Ni atom are located approximately parallel to the triazine ring, making dihedral angles of 2.0(2)$^\circ$ and 3.7(2)$^\circ$. The dihedral angle between the uncoordinated pyridyl ring and triazine ring is 11.3(1)$^\circ$. The nearly planar pyridine (N7—C23) rings are twisted with the dihedral angle of 65.88(5)$^\circ$ between the ring plane and the least-squares plane of the ptz ligand. The complex displays intramolecular C—H···Cl hydrogen bonds with d(C····Cl) = 3.227(3)–3.05(3) Å. Pairs of complex molecules are assembled by intermolecular C—H···Cl hydrogen bonds with d(C····Cl) = 3.347(3) Å. These pairs are stacked in columns along [100]. In the columns, numerous π···π interactions are present. The shortest distance between Cg1 (the centroid of ring N1—C13) and Cg2 (the centroid of ring N6—C18; symmetry code i: $-x$, $-y$, $1-z$) is 4.049(1) Å, and the dihedral angle between the ring planes is 3.7(1)$^\circ$.[6]

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**References**

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