Received 18 October 2006

Accepted 6 November 2006

Acta Crystallographica Section E Structure Reports Online

ISSN 1600-5368

Ilke Gurol,^a Gulay Gumus,^a Fatma Yuksel,^b Erwann Jeanneau^c* and Vefa Ahsen^d

^aTUBITAK Marmara Research Centre, Materials Institute, PO Box 21, 41470 Gebze, Kocaeli, Turkey, ^bUniversité Lyon 1, Laboratoire Multimatériaux et Interfaces - UMR 5615, 69622 Villeurbanne Cedex, France, ^cUniversité Lyon 1, Centre de Diffractométrie Henri Longchambon, 69622 Villeurbanne Cedex, France, and ^dGebze Institute of Technology, Department of Chemistry, PO Box 141, 41400 Gebz, Kocaeli, Turkey

Correspondence e-mail: erwann.jeanneau@univ-lyon1.fr

Key indicators

Single-crystal X-ray study T = 293 K Mean σ (C–C) = 0.010 Å R factor = 0.059 wR factor = 0.066 Data-to-parameter ratio = 10.3

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

Bis[N,N'-bis(octylsulfanyl)glyoximato]nickel(II)

In the title compound, $[Ni(C_{18}H_{35}N_2O_2S_2)_2]$, the coordination geometry around the Ni atom, lying on a centre of symmetry, is square planar, consisting of four N atoms from the two *vic*-dioxime ligands. The O atoms are intramolecularly hydrogen bonded, as is usually observed for *anti*-isomer metal complexes of *vic*-dioximes.

Comment

Vicinal dioximes and their complexes have undergone numerous investigations in the past decades because of their various properties (Chakravorty, 1974). The exceptional stability and unique electronic properties of *vic*-dioxime complexes can be attributed to their planar structure, which is stabilized by two hydrogen bonds (Voter *et al.*, 1951). The presence of mildly acidic hydroxyl groups and slightly basic N atoms makes *vic*-dioxime an amphoteric ligand which forms complexes with transition metal ions such as Ni^{II}, Cu^{II}, Co^{II} and Co^{III}. We present here the title complex, (I), of Ni^{II} with a *vic*-dioxime.



In complex (I), the Ni^{II} atom is coordinated by the four N atoms of the two *vic*-dioxime ligands and is in a square-planar environment (Fig. 1). The Ni atom lies on a centre of symmetry. The plane of the octyl carbon chains forms an angle of 64.05 (9)° with respect to the perfectly planar central core containing the metal atom. The mean Ni–N distance and the N1–Ni1–N2 angle (Table 1) are similar to those reported for related compounds (Gumus *et al.*, 2004).

Intramolecular $O-H\cdots O$ hydrogen bonding is observed, as expected. In the crystal structure the metal coordination planes lie parallel to the *bc* plane. The distance between two of these planes is approximately 16 Å and the octyl chains occupy the available space between the planes in a tail-to-tail fashion (Fig. 2).

Experimental

© 2006 International Union of Crystallography All rights reserved The oxime ligand was obtained in absolute ethanol at room temperature by reaction of dichloroglyoxime (Ponzio & Baldroco,

metal-organic papers

1930) with octane-1-thiol in the presence of metallic sodium, under argon (Kesimli et al., 2005). Complex (I) was synthesized by reaction of the ligand (0.25 g, 0.72 mmol) with NiCl₂·6H₂O (0.092 g, 0.092 g)0.36 mmol) and potassium hydroxide (0.04 g, 0.70 mmol) in ethanol (20 ml) at 323 K. A red precipitate was obtained upon cooling of the mixture to room temperature. Finally, compound (I) was recrystallized from hot ethanol.

Z = 4

 $D_x = 1.232 \text{ Mg m}^{-3}$

0.43 \times 0.06 \times 0.05 mm

14667 measured reflections

5083 independent reflections

2297 reflections with $I > 2\sigma(I)$

 $0.412T_2(x)$], where T_i are the Chebychev polynomials and $x = F/F_{\text{max}}$ (Watkin, 1994;

1.872 (4)

82.73 (17)

Mo $K\alpha$ radiation

 $\mu = 0.68 \text{ mm}^{-1}$

T = 293 K

Needle red

 $R_{\rm int} = 0.122$

 $\theta_{\rm max} = 27.9^\circ$

Prince, 1982) $(\Delta/\sigma)_{\rm max} < 0.001$ $\Delta \rho_{\rm max} = 0.71 \text{ e } \text{\AA}^{-3}$ $\Delta \rho_{\rm min} = -0.60 \text{ e } \text{\AA}^{-3}$

Crystal data

[Ni(C₁₈H₃₅N₂O₂S₂)₂] $M_r = 809.95$ Monoclinic, C2/c a = 50.248 (5) Å b = 5.322(5) Å c = 16.944 (5) Å $\beta = 105.536 (5)^{\circ}$ $V = 4366 (4) \text{ Å}^3$

Data collection

Nonius KappaCCD area-detector diffractometer φ and ω scans Absorption correction: analytical (de Meulenaer & Tompa, 1965) $T_{\min} = 0.77, T_{\max} = 0.97$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.066$
S = 1.06
2297 reflections
223 parameters
H-atom parameters constrained
$w = [1 - (F_{\rm o} - F_{\rm c})^2 / 36\sigma^2(F)]^2 /$
$[0.737T_{0}(x) + 0.674T_{1}(x) +$

Table 1

Selected	geometric	parameters	(A,	°).
	0			/

Ni1-N1 1.876 (4) Ni1-N2 $N2^i - Ni1 - N1$ 97.27 (17) N1-Ni1-N2 Symmetry code: (i) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$.

Table 2

Hydrogen-bond geometry (Å, °).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - H \cdot \cdot \cdot A$
$O2-H3\cdots O1^i$	0.83	1.62	2.455 (5)	175
Symmetry code: (i)	$-x + \frac{3}{2}, -y + \frac{1}{2},$	-z + 1.		

(1)

All H atoms were located in a difference Fourier map. They were initially refined using soft restraints on the bond lengths and angles to regularize their geometries, and then they were refined as riding, with C-H in the range 0.96–0.98 Å and O-H = 0.83 Å, and with $U_{iso}(H)$ $= 1.2U_{eq}(C,O).$

Data collection: COLLECT (Nonius, 2001); cell refinement: DENZO and SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO and SCALEPACK; program(s) used to solve structure: SIR97 (Altomare et al., 1999); program(s) used to refine structure: CRYSTALS (Betteridge et al., 2003); molecular graphics:



Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$.]



Figure 2 A view of the packing of (I).

DIAMOND (Brandenburg, 1996); software used to prepare material for publication: CRYSTALS.

The authors are grateful to Professor Dominique Luneau for his advice and fruitful discussions. FY thanks TUBITAK for the award of a BIDEB-2219 fellowship, and the Région Rhône-Alpes. Travel grants between France and Turkey were the CNRS-TUBITAK collaboration sponsored by programme.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). J. Appl. Cryst. 32, 115-119.

- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). J. Appl. Cryst. 36, 1487.
- Brandenburg, K. (1996). DIAMOND. University of Bonn, Germany.
- Chakravorty, A. (1974). Coord. Chem. Rev. 13, 1-46.
- Gumus, G., Ahsen, V., Lebrun, C., Luneau, D. & Pécaut, J. (2004). New J. Chem. 28, 177–179.
- Kesimli, B., Topacli, A., Topacli, C., Gumus, G., Gurol, I. & Ahsen, V. (2005). Colloids Surf. A Pysicochem. Eng. Asp., 256, 137–143.
- Meulenaer, J. de & Tompa, H. (1965). Acta Cryst. 19, 1014-1018.
- Nonius (2001). COLLECT. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Ponzio, G. & Baldroco, F. (1930). Gazz. Chim. Ital. 60, 415-429.
- Prince, E. (1982). Mathematical Techniques in Crystallography and Materials Science. New York: Springer-Verlag.
- Voter, R. C., Banks, C. V., Fassel, V. A. & Kehres, P. W. (1951). Anal. Chem. 23, 1730–1735.
- Watkin, D. (1994). Acta Cryst. A50, 411-437.