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Redetermination of bis(dimethylglyoximato-κ^2N,N')nickel(II)

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Key indicators

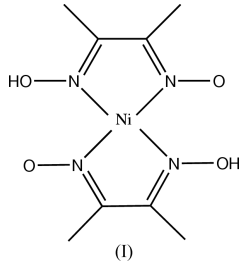
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.103
Data-to-parameter ratio = 13.3

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

The crystal structure of the title complex, $[Ni(C_4H_7N_2O_2)_2]$, was first determined by Godycki & Rundle [*Acta Cryst.* (1953), **6**, 487–495] and rerefined by Williams, Wohlauer & Rundle [*J. Am. Chem. Soc.* (1959), **81**, 755–756]. We present here a redetermination, of improved precision, in which disordered H atoms have been experimentally located.

Comment

Previous structure determinations of (I) have been reported by Godycki & Rundle (1953) and Williams *et al.* (1959). The molecular structure of (I) is shown in Fig. 1. The complex molecule is located on a mirror plane, apart from some methyl H atoms, and the central Ni atom is located on an inversion centre. Two dimethylglyoximate monoanions chelate to the Ni^{II} atom, with Ni—N distances of 1.859 (3) and 1.861 (3) Å (Table 1). A difference Fourier map clearly showed two H atoms bound to oxime atoms O1 and O2. This means that this is an average structure of disordered molecules with deprotonation of dimethylglyoxime occurring randomly at either O1 or O2. Intramolecular O—H···O hydrogen bonding is observed as expected (Fig. 1 and Table 2). The molecular packing diagram shows the overlapped arrangement of neighbouring parallel molecules. The separation of 3.2372 (7) Å between the molecular planes, *i.e.* the Ni···Ni distance, may be indicative of some bonding contribution.



(I)

Experimental

The title complex is a by-product in the reaction for preparing a μ-dibromosuccinate complex. An aqueous solution (10 ml) containing $NiCl_2{\cdot}6H_2O$ (0.24 g, 1 mmol) and dimethylglyoxime (0.12 g, 1 mmol) was mixed with another aqueous solution (20 ml) of dibromosuccinic acid (0.28 g, 1 mmol) and NaOH (0.08 g, 2 mmol). The mixture was refluxed for 4 h and then filtered. Single crystals of the title compound were obtained from the filtrate after 2 weeks, along with needle-shaped crystals. The latter were not, however, suitable for X-ray analysis.

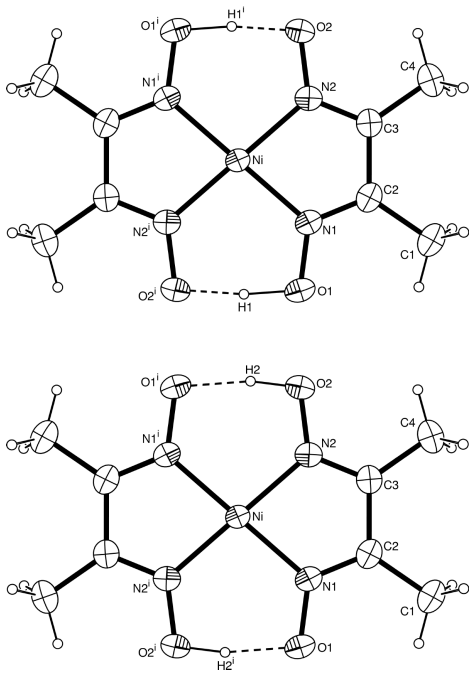


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids. Dashed lines indicates the hydrogen bonding. The two components of the disorder, involving H1 and H2, are shown in the top and bottom views, respectively. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

Crystal data

$[Ni(C_4H_7N_2O_2)_2]$	Mo $K\alpha$ radiation
M_r = 288.92	Cell parameters from 7201 reflections
Orthorhombic, *Ibam*	θ = 2.3–27.4°
a = 16.5752 (17) Å	μ = 1.75 mm^{-1}
b = 10.4233 (13) Å	T = 295 (2) K
c = 6.4744 (14) Å	Prism, brown
V = 1118.6 (3) Å^3	0.3 × 0.2 × 0.1 mm
Z = 4	
D_x = 1.716 Mg m^{-3}	

Data collection

Rigaku R-AXIS RAPID diffractometer	705 independent reflections
ω scans	632 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	R_{int} = 0.028
T_{min} = 0.590, T_{max} = 0.840	θ_{max} = 27.5°
4806 measured reflections	$h = -21 \rightarrow 21$
	$k = -13 \rightarrow 13$
	$l = -7 \rightarrow 8$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2 + 1.2037P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.103	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
705 reflections	
53 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å).

Ni—N2	1.859 (3)	N1—O1	1.350 (4)
Ni—N1	1.861 (3)	N2—C3	1.292 (4)
N1—C2	1.292 (5)	N2—O2	1.341 (4)

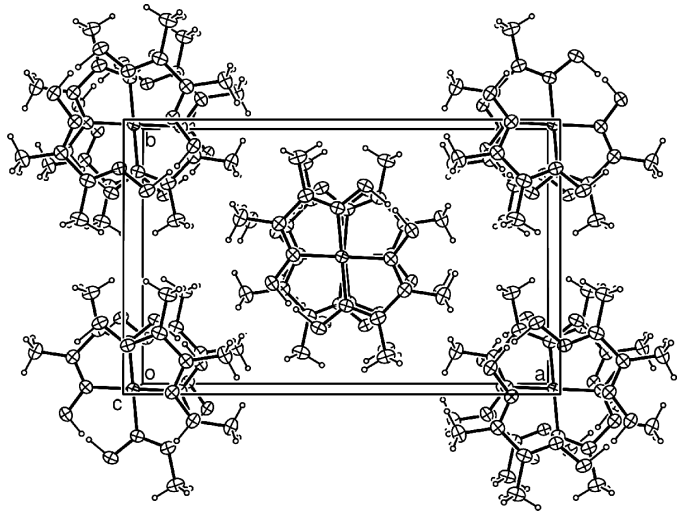


Figure 2
A molecular packing diagram, showing the overlapped arrangement of neighboring parallel molecules.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	1.11	1.35	2.457 (4)	172
O2—H2···O1^{i}	0.99	1.48	2.457 (4)	169

Symmetry code: (i) $1-x, 1-y, 1-z$.

H atoms on C atoms were placed in calculated positions, with C—H = 0.96 Å, and refined as riding, with $U_{iso}(H) = 1.5U_{eq}$ of the carrier atoms. A difference Fourier map clearly showed that two peaks on a mirror plane, with approximately identical electron density, are close to O1 and O2. They were assigned to H atoms bound to O1 and O2, with half occupancy each, and were included in structure-factor calculations with fixed positional parameters and a displacement parameter of U_{iso} = 0.08 Å^2.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR*-92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku Corporation (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Version 3.00. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.
Godycki, L. B. & Rundle, R. E. (1953). *Acta Cryst.* **6**, 487–495.
Williams, D. E., Wohlauer, G. & Rundle, R. E. (1959). *J. Am. Chem. Soc.* **81**, 755–756.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.