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Bis(adamantylamine-κN)bis(1-phenylbutane-1,3-dionato-$\kappa^2 O,O'$)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 14.6

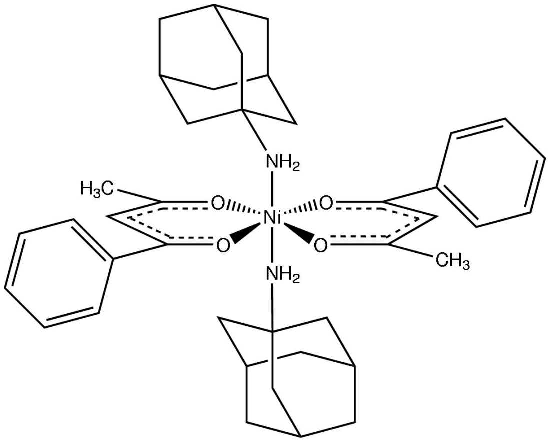
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title complex, [Ni($C_{10}H_9O_2$)$_2$($C_{10}H_{17}N$)$_2$], the Ni atom, which occupies a special position at an inversion centre, has an octahedral environment formed by four O atoms of two chelate benzoylacetonates [Ni—O 2.0409 (16) and 2.0187 (16) Å, and O—Ni—O 90.36 (6)°] and two N atoms of the adamantylamine ligands [Ni—N = 2.176 (2) Å]. The amine H atoms of the adamantylamine ligands do not participate in any hydrogen bonds, due to their steric shielding and the lack of accessible acceptors.

Comment

Metal complexes of β-diketones have been studied very intensively for years. These compounds have found their place in catalytic and extraction processes and have been used very successfully as precursors for the formation of thin films. Their significant potential as hosts in soft (and smart) supramolecular materials was recognized five years ago (Soldatov *et al.*, 1999). These products have many attributes which qualify them for use as supramolecular ion-exchange materials, flexible and smart sorbents, and as functional organic zeolite analogues (Soldatov *et al.*, 2003).

Soldatov and co-workers have reported a series of modified metal dibenzoylmethanates which were used for designing supramolecular host–guest materials (Soldatov *et al.*, 1999; Soldatov & Ripmeester, 2001*a*,*b*; Soldatov *et al.*, 2001; Soldatov *et al.*, 2002). A search of the Cambridge Structural Database (CSD, Version 5.25; Allen, 2002) yielded 1912 entries of metal complexes containing β-diketones as a ligand; only 24 entries, however, contain 1-phenyl-1,3-butanedionate (benzoylacetonate) as a diketonate ligand. Just three of these are nickel(II) benzoylacetonates, *viz*. refcodes BEBMUQ (Yoshida *et al.*, 1999), AMIQIW (Bučar & Meštrović, 2003) and ASOWUA (Meštrović *et al.*, 2004). The scarcity of information on metal benzoylacetonates prompted us to investigate the applicability of adducts of bis(benzoylacetonato)nickel(II) with some pyridine derivatives in the design of host–guest materials (Bučar, 2004). In further research, among other addends, adamantylamine was introduced as a ligand to complete the coordination of the Ni atom in the basic metal bis-chelate unit, bis(benzoylacetonato)nickel(II). Metal complexes with adamantylamine are extremely rare. Adamantylamine is normally introduced into organometallics as the adamantylamide anion and in the CSD there is only one structure that contains adamantylamine as a neutral ligand (refcode QUBNAC; Westerhausen *et al.*, 2001). In this communication, the crystal structure of bis(benzoylacetonato)bis(adamantylamine)nickel(II), the title complex, (I), is reported.



(I)

In the title complex (Fig. 1), the Ni atom, which occupies a special position at an inversion centre, has an octahedral environment formed by two chelate benzoylacetonates [Ni—O1 2.0409 (16) and Ni—O2 2.0187 (16) Å, and O1—Ni—O2 90.36 (6)°] and two adamantylamine molecules [Ni—N 2.176 (2) Å]. The O1···O2 bite distance [2.880 (2) Å] is in good agreement with those in BEBMUQ and AMIQIW and is slightly longer than that in ASOWUA. The chelate rings have the conformation of a shallow sofa, with a folding angle of 11.00 (7)° along the O1···O2 line. The C5–C10 phenyl ring mean plane forms a dihedral angle of 13.43 (9)° with the O1—C2—C3—C4—O2 plane of the chelate ring.

As the amine H atoms are shielded by bulky ligands and, probably even more importantly, there are no accessible acceptors, no intermolecular hydrogen bonds are found in the structure of (I). Molecules of the complex in the crystal structure are linked only by van der Waals interactions.

Experimental

Bis(1-phenyl-1,3-butanedionato)nickel(II) was prepared by refluxing an ethanol solution (100 ml) of $NiCl_2 \cdot 6H_2O$ (0.475 g) and 1-phenyl-1,3-butanedione (0.645 g) for 5 h. The reaction mixture was filtered. Adamantylamine (0.037 g) was added and dissolved in 15 ml of the filtrate. The mixture was then kept at 348 K for 10 min. The resulting dark-green solution was filtered and left to evaporate slowly at 253 K. Single crystals of (I) suitable for X-ray diffraction analysis precipitated after two weeks.

Crystal data

$[Ni(C_{10}H_9O_2)_2(C_{10}H_{17}N)_2]$
M_r = 683.55
Triclinic, $P\bar{1}$
a = 6.738 (3) Å
b = 12.028 (3) Å
c = 12.063 (3) Å
α = 70.02 (3)°
β = 86.88 (3)°
γ = 76.45 (3)°
V = 892.8 (5) Å^3

Z = 1
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4811 reflections
θ = 2.1–30.4°
μ = 0.59 mm^{-1}
T = 295 (1) K
Plate, green
0.6 × 0.3 × 0.1 mm

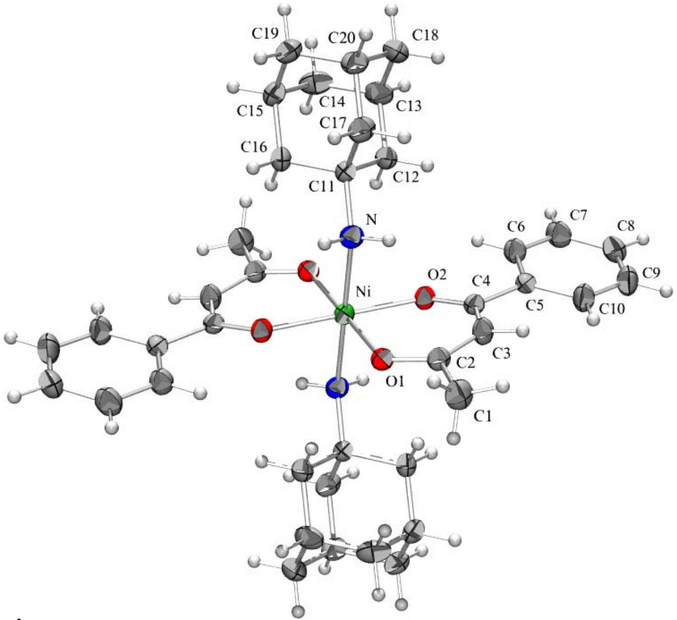


Figure 1
A view of (I), showing the atom-numbering scheme and with displacement ellipsoids drawn at the 30% probability level. Unlabelled atoms are related to labelled atoms by the symmetry code ($-x + 2$, $-y$, $-z$).

Data collection

Oxford Diffraction Xcalibur 3 CCD area-detector diffractometer
ω scans
Absorption correction: analytical (Alcock, 1970)
T_{min} = 0.810, T_{max} = 0.941
12 713 measured reflections

3129 independent reflections
2545 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.0°
$h = -8 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.07
3129 reflections
215 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—O1	2.0409 (16)	N—C11	1.484 (3)
Ni—O2	2.0187 (16)	C1—C2	1.505 (3)
Ni—N	2.176 (2)	C2—C3	1.392 (3)
O1—C2	1.263 (3)	C3—C4	1.390 (3)
O2—C4	1.269 (3)	C4—C5	1.506 (3)
O2—Ni—O1	90.36 (6)	C3—C2—C1	118.5 (2)
O2—Ni—N	91.10 (7)	C4—C3—C2	127.1 (2)
O1—Ni—N	82.72 (7)	O2—C4—C3	124.8 (2)
C2—O1—Ni	124.67 (14)	O2—C4—C5	114.84 (19)
C4—O2—Ni	125.71 (14)	C3—C4—C5	120.4 (2)
C11—N—Ni	130.22 (14)	C6—C5—C10	118.2 (2)
O1—C2—C3	125.6 (2)	C6—C5—C4	119.0 (2)
O1—C2—C1	115.8 (2)		

All H atoms were placed geometrically and included in the refinement in the riding-model approximation, with C—H distances in the range 0.93–0.98 Å and N—H distances of 0.90 Å, and with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom ($1.5U_{eq}$ in the case of the methyl H atoms).

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2003); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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