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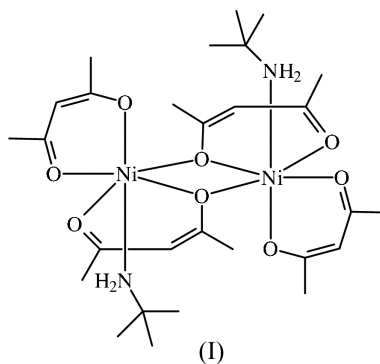
Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
 R factor = 0.062
 wR factor = 0.193
Data-to-parameter ratio = 19.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Di- μ -acetylacetonato-bis[diacetylacetonato-
(*tert*-butylamine)nickel(II)]

In the centrosymmetric dinuclear title compound, $[\text{Ni}_2(\text{C}_5\text{H}_7\text{O}_2)_4(\text{C}_4\text{H}_{11}\text{N})_2]$, the six-coordinate Ni^{II} atom displays pseudo-octahedral coordination, with chelation by two acetylacetonate groups. The other coordination sites are occupied by the N atom of the amine and the O atom of one of the two acetylacetonate groups.

Comment

The title compound, (I), was synthesized in the course of our studies of the catalytic activity of Ni complexes in olefin polymerization (Luo *et al.*, 2002). The compound exists as a centrosymmetric dinuclear compound in which both acetylacetonate groups chelate to the Ni atom in each half $[\text{Ni}-\text{O}$ 2.076 (3) and 2.014 (4) \AA , and 2.028 (3) and 2.001 (3) \AA], but one of them also interacts with the other Ni atom $[\text{Ni}-\text{O}$ 2.156 (3) $\text{\AA}]$. The octahedral geometry is completed by the N atom of the amine. Probably because of the steric bulk of the *tert*-butyl group, the $\text{Ni}-\text{N}-\text{C}$ angle is larger than expected $[135.4(4)^\circ]$.



Experimental

tert-Butylammonium acetylacetonate was the unexpected product obtained by reacting butylamine and acetylacetone, in a 1:1 molar stoichiometry, in an attempt to synthesize a Schiff base. Bis(tetraethylammonium) tetrabromonickelate was synthesized using the literature procedure of Gill & Nyholm (1959). The two reagents were then reacted according to the published procedure (Ernst *et al.*, 1967; Everett & Holm, 1965, 1966, 1968). Potassium metal (0.29 g, 0.7 mmol) was placed in *tert*-butanol (25 ml). After it had dissolved completely, the solution was warmed to 323 K and the ammonium salt (1.99 g, 1.1 mmol) was added to give a yellow–orange solution. The nickel reagent (3.06 g, 5 mmol) was added to the cooled solution to afford an immediate precipitate. The product was purified by recrystallization from *n*-pentane–toluene (3:1) in about 40% yield. CHN analysis: calculated for $\text{C}_{28}\text{H}_{50}\text{N}_2\text{Ni}_2\text{O}_8$: C 50.95, H 7.64, N 4.22%; found: C 50.88, H 7.59, N 4.14%.

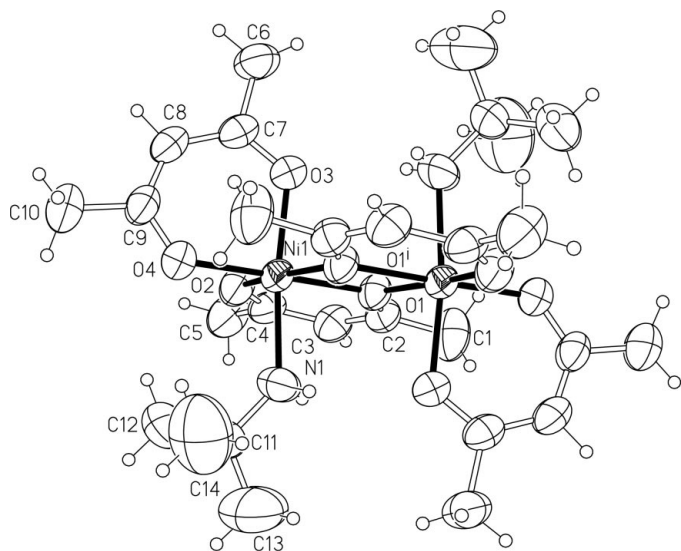


Figure 1
A view of the molecule of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Crystal data

$[\text{Ni}_2(\text{C}_5\text{H}_7\text{O}_2)_4(\text{C}_4\text{H}_{11}\text{N})_2]$
 $M_r = 660.12$
 Triclinic, $P\bar{1}$
 $a = 9.073$ (1) Å
 $b = 9.141$ (1) Å
 $c = 11.513$ (1) Å
 $\alpha = 110.555$ (2)°
 $\beta = 99.252$ (2)°
 $\gamma = 100.637$ (2)°
 $V = 851.9$ (2) Å³

$Z = 1$
 $D_x = 1.287$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 972 reflections
 $\theta = 3.4\text{--}26.7^\circ$
 $\mu = 1.15$ mm⁻¹
 $T = 298$ (2) K
 Block, green
 $0.41 \times 0.31 \times 0.15$ mm

Data collection

Bruker SMART area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.650$, $T_{\max} = 0.847$
 5189 measured reflections

3608 independent reflections
 2817 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\text{max}} = 27.2^\circ$
 $h = -11 \rightarrow 11$
 $k = -11 \rightarrow 10$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.193$
 $S = 1.08$
 3608 reflections
 185 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1088P)^2 + 0.8863P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.78$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------------------|-----------|-------------------------|-----------|
| Ni1—O1 | 2.076 (3) | Ni1—O3 | 2.028 (3) |
| Ni1—O1 ⁱ | 2.156 (3) | Ni1—O4 | 2.001 (3) |
| Ni1—O2 | 2.014 (4) | Ni1—N1 | 2.100 (4) |
| O1—Ni1—O1 ⁱ | 79.7 (1) | O1 ⁱ —Ni1—N1 | 87.7 (2) |
| O1—Ni1—O2 | 91.0 (1) | O2—Ni1—O3 | 90.2 (2) |
| O1—Ni1—O3 | 90.0 (1) | O2—Ni1—O4 | 88.1 (1) |
| O1—Ni1—O4 | 178.9 (1) | O2—Ni1—N1 | 92.3 (2) |
| O1—Ni1—N1 | 83.5 (2) | O3—Ni1—O4 | 90.7 (1) |
| O1 ⁱ —Ni1—O2 | 170.7 (1) | O3—Ni1—N1 | 173.1 (2) |
| O1 ⁱ —Ni1—O3 | 88.8 (1) | O4—Ni1—N1 | 95.9 (2) |
| O1 ⁱ —Ni1—O4 | 101.2 (1) | | |

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

The C—C and C—N distances in the *tert*-butylamine unit were restrained to be approximately equal, as were the C···C distances. Additionally, the displacement parameters of the three methyl C atoms were restrained to be approximately isotropic. The final difference map had a peak larger than 1 e \AA^{-3} in the vicinity of the *tert*-butyl group, for which disorder is likely but has not been resolved. H atoms were positioned geometrically and treated as riding on their parent C and N atoms ($\text{C—H}_{\text{aromatic}} = 0.93$, $\text{C—H}_{\text{aliphatic}} = 0.96$ and $\text{N—H} = 0.90$ Å). The displacement parameters of all H atoms were set to 1.5 times those of the equivalent isotropic displacement parameters of their parent atoms.

Data collection: SMART (Bruker, 1999); cell refinement: SMART; data reduction: SAINT (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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