5371 measured reflections
5010 independent reflections

3 standard reflections
every 200 reflections
intensity decay: 0.372%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.167$
$S = 1.138$
5010 reflections
343 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 17.9297P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.626$ e Å^{-3}
$\Delta\rho_{\min} = -0.870$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ru1—B2	2.036 (9)	Ru1—P1	2.487 (2)
Ru1—B3	2.062 (8)	S1—C1	1.641 (8)
Ru1—B4	2.329 (8)	S2—C2	1.641 (8)
Ru1—B5	2.341 (8)	O1—C1	1.296 (9)
Ru1—B7	2.393 (9)	O1—B2	1.447 (10)
Ru1—B6	2.393 (8)	O2—C2	1.301 (9)
Ru1—S2	2.443 (2)	O2—B3	1.437 (10)
Ru1—S1	2.445 (2)		
B2—Ru1—S2	151.7 (3)	B6—Ru1—S1	114.8 (2)
B3—Ru1—S2	74.7 (2)	S2—Ru1—S1	84.87 (7)
B4—Ru1—S2	159.7 (2)	B2—Ru1—P1	109.7 (3)
B5—Ru1—S2	115.2 (2)	B3—Ru1—P1	112.7 (2)
B7—Ru1—S2	114.6 (2)	B4—Ru1—P1	93.1 (2)
B6—Ru1—S2	85.8 (2)	B5—Ru1—P1	94.6 (2)
B2—Ru1—S1	74.8 (2)	B7—Ru1—P1	155.6 (2)
B3—Ru1—S1	152.6 (2)	B6—Ru1—P1	159.4 (2)
B4—Ru1—S1	115.3 (2)	S2—Ru1—P1	86.82 (7)
B5—Ru1—S1	159.8 (2)	S1—Ru1—P1	83.63 (7)
B7—Ru1—S1	86.7 (2)		

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *PROCESS* in *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL*97.

We thank the NSFC (grant No. 29873009) for support of this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1533). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1226–1228

A three-ring *exo*-polyhedral-cyclized nickelaundecaborane cluster: [$(CH_3COS)_3NiB_{10}H_7(PPh_3)$]

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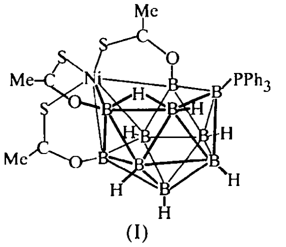
(*Received 4 January 1999; accepted 30 March 1999*)

Abstract

The title compound 3,7:7,8:7,11-tris(μ-thioacetate-*S*:*O*)-10-(triphenylphosphine-*P*)-8,9-μH-7-nickela-*nido*-undecaborane, [$Ni(C_{24}H_{31}B_{10}O_3PS_3)$], has a *nido*-type $\{NiB_{10}\}$ cage fused to three five-membered Ni—S—C—O—B rings.

Comment

The chemistry of metallaborane clusters is still attracting a great deal of interest and a large variety of novel structural motifs is continually revealed (Greenwood, 1983, 1991; Kennedy, 1986, 1997). We have synthesized and characterized a series of metallaborane compounds which contain *exo*-polyhedral cyclization of metal ligands to the cluster. Some examples of groups involved in cyclization are thiobenzoate (Dou, Hu, Sun *et al.*, 1997; Dou, Hu, Yao *et al.*, 1997; Hu *et al.*, 1997; Hu, Dou, Sun *et al.*, 1998; Hu, Dou, Yao *et al.*, 1998; Yao *et al.*, 1998), benzoate (Yao *et al.*, 1995) and acetate (Fontaine *et al.*, 1987). As a continuation of this investigation on *exo*-polyhedral cyclization clusters, we studied the reaction of [NiCl$_2$(PPh$_3$)$_2$] with [B$_{10}$H$_{10}$]$^{2-}$ and thioacetatic acid. The preparation and structure of the title compound, (I), are reported here.



(I)

The cluster structure of (I) is shown in Fig. 1. The compound has a *nido*-11-vertex {NiB$_{10}$} cage, with the Ni atom in the open NiB$_4$ face. There are three five-membered rings, *i.e.* Ni7—S1—C1—O1—B3, Ni7—S2—C2—O2—B8 and Ni7—S3—C3—O3—B11. The Ni7—S1, Ni7—S2 and Ni7—S3 distances are 2.3766 (14), 2.3405 (13) and 2.2469 (13) Å, respectively, which are consistent with the corresponding Ni—S bond lengths in the cluster [(PhCOS)$_3$NiB$_{10}$H$_7$(PPh$_3$)] (Dou, Hu, Sun *et al.*, 1997). The distances Ni7—B3 [2.082 (4) Å], Ni7—B8 [2.109 (4) Å] and Ni7—B11 [2.112 (4) Å] are significantly shorter than reported corresponding Ni—B distances [2.18 (1), 2.24 (1) and 2.22 (1) Å, respectively] in the non-cyclized nickelaborane (Guggenberger, 1972). This further demonstrates that cycloboronation can strengthen the nickel-to-boron bonding (Dou, Hu, Sun *et al.*, 1997; Dou, Hu, Yao *et al.*, 1997; Hu *et al.*, 1997). In the cluster, the PPh$_3$ ligand is bonded to the B10 atom of the cage. The bond lengths P1—B10 [1.906 (5) Å], B10—B11 [1.682 (6) Å] and B9—B10 [1.823 (6) Å] are similar to those in [(PhCOS)$_2$NiB$_{10}$H$_8$(PPh$_3$)] and [(PhCOS)$_3$NiB$_{10}$H$_7$(PPh$_3$)] (Dou, Hu, Sun *et al.*, 1997; Dou, Hu, Yao *et al.*, 1997). There is a bridging H atom between the B8 and B9 atoms.

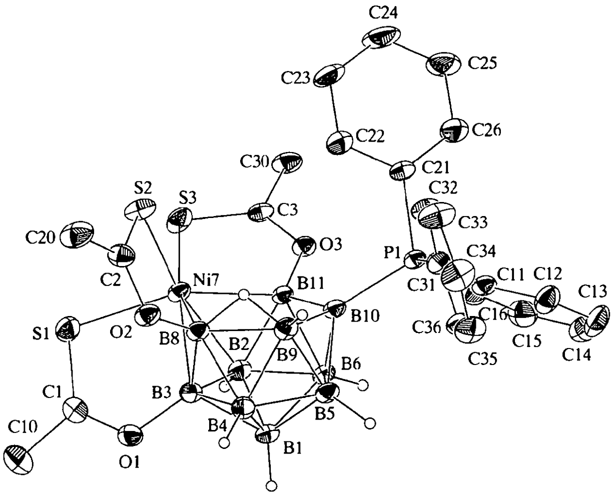


Fig. 1. The molecular structure of (I) shown with 30% probability displacement ellipsoids.

Experimental

The synthesis of (I) was carried out by reaction of [NiCl$_2$(PPh$_3$)$_2$] (0.6 mmol), [Et$_4$N]$_2$B$_{10}$H$_{10}$ (0.6 mmol) and CH$_3$COSH (1.2 mmol) in refluxing dichloromethane for 65 h under an atmosphere of dry nitrogen. The resulting solution was reduced in volume and chromatographed using dichloromethane/light petroleum (4:1) as the eluting medium to give the dark-green compound at R_f = 0.85. The product was recrystallized from an *n*-hexane/dichloromethane solution.

Crystal data

[Ni(C$_{24}$H$_{31}$B$_{10}$O$_3$PS$_3$)]
M_r = 661.45
Triclinic
$P\bar{1}$
a = 11.957 (3) Å
b = 12.966 (3) Å
c = 13.041 (3) Å
α = 114.31 (2)°
β = 109.41 (2)°
γ = 94.62 (2)°
V = 1681.3 (7) Å^3
Z = 2
D_x = 1.307 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10.0–13.7°
μ = 0.837 mm^{-1}
T = 294 (2) K
Square prism
0.42 × 0.36 × 0.18 mm
Dark green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.720, $T_{\max}$ = 0.864
6229 measured reflections
5915 independent reflections

3932 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.017
$\theta_{\max}$ = 25°
$h = 0 \rightarrow 14$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.149
S = 1.047
5915 reflections
407 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0847P)^2 + 1.5226P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.894 e Å^{-3}
$\Delta\rho_{\min}$ = −0.707 e Å^{-3}
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni7—B3	2.082 (4)	S2—C2	1.665 (5)
Ni7—B8	2.109 (4)	S3—C3	1.676 (4)
Ni7—B11	2.112 (4)	P1—B10	1.906 (5)
Ni7—B2	2.119 (5)	O1—B3	1.480 (5)
Ni7—S3	2.2469 (13)	O2—B8	1.457 (5)
Ni7—S2	2.3405 (13)	O3—B11	1.492 (5)
Ni7—S1	2.3766 (14)	B9—B10	1.823 (6)
S1—C1	1.658 (4)	B10—B11	1.682 (6)
B3—Ni7—B8	50.29 (18)	B8—Ni7—S2	75.25 (13)
B3—Ni7—B11	85.06 (17)	B11—Ni7—S2	117.41 (12)
B8—Ni7—B11	88.79 (17)	B2—Ni7—S2	159.91 (13)
B3—Ni7—B2	49.89 (18)	S3—Ni7—S2	91.97 (5)
B8—Ni7—B2	88.50 (18)	B3—Ni7—S1	82.27 (13)
B11—Ni7—B2	49.25 (17)	B8—Ni7—S1	101.17 (13)
B3—Ni7—S3	146.36 (14)	B11—Ni7—S1	152.86 (12)
B8—Ni7—S3	159.62 (13)	B2—Ni7—S1	105.30 (13)
B11—Ni7—S3	83.04 (12)	S3—Ni7—S1	94.45 (5)
B2—Ni7—S3	100.01 (13)	S2—Ni7—S1	89.63 (5)
B3—Ni7—S2	121.35 (14)		

The cage H atoms were located using Fourier methods and refined isotropically. H atoms on phenyl and CH_3 groups were included at riding positions, with C—H distances and U(H) fixed at 0.93 Å/1.2U_{eq}(C) and 0.96 Å/1.5U_{eq}(C), respectively.

Data collection: *CAD*-4 *Manual* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *Manual*. Data reduction: *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL*97.

The authors acknowledge the support of the Chinese National Science Foundation. JMD acknowledges the support of the Science Foundation of Shandong Province.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1304). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1228–1230

Bis(1-methylimidazole-N^3)bis(salicylato-O,O')copper(II)

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(*Received 23 September 1998; accepted 9 February 1999*)

Abstract

The crystal structure of the title complex, [Cu($C_7H_5O_3$)$_2$($C_4H_6N_2$)$_2$], is built of monomeric [Cu(mim)$_2$(Sal)$_2$] molecules (mim is N-methylimidazole and Sal is salicylate), which display a distorted octahedral $CuN_2O_2\cdots O_2'$ chromophore. An intramolecular hydrogen bond exists between the hydroxyl group and the carbonyl O atom of the carboxylate group [O···O 2.581 (3) Å].

Comment

The number of reports on monomeric copper(II) complexes containing carboxylate and basic ligands is quite limited. It has been found that by increasing the acidity of the alkylcarboxylate ligands or by increasing the basicity of the basic ligands, the tendency towards formation of monomeric complexes increases (Melnik, 1981; Ahmed & Abuhijleh, 1982; Uruska *et al.*, 1990). Complexes of copper(II) with carboxylate and imidazole ligands have been studied as models for copper proteins containing both functionalities in the side chain (Sigel, 1980; Bernarducci *et al.*, 1983). Some of these copper(II) complexes have been found to possess a variety of pharmacological activities (Tamura *et al.*, 1987; Bhirud & Śrivastava, 1990).

The crystal structure of the title complex, (I), consists of monomeric units in which the copper(II) ion assumes a centrosymmetric distorted octahedral geom-