

| | | | | |
|-------|-------------|------------|------------|--------|
| O(8) | -0.1308 (2) | 0.4851 (2) | 0.2140 (3) | 0.0293 |
| O(9) | 0.2876 (2) | 0.1573 (3) | 0.0099 (3) | 0.0360 |
| O(10) | 0.2309 (3) | 0.1449 (4) | 0.4774 (3) | 0.0416 |

Table 2. Selected geometric parameters (Å, °)

| | | | |
|--|------------|--|-----------|
| Fe(1)—N(1) | 2.047 (2) | C(6)—N(1) | 1.333 (4) |
| Fe(1)—N(2) | 2.053 (2) | C(7)—O(3) | 1.225 (4) |
| Fe(1)—O(1) | 2.012 (2) | C(7)—O(4) | 1.277 (4) |
| Fe(1)—O(4) | 2.030 (2) | C(8)—C(9) | 1.510 (4) |
| Fe(1)—O(5) | 2.034 (2) | C(8)—O(5) | 1.290 (4) |
| Fe(1)—O(7) | 2.013 (2) | C(8)—O(6) | 1.214 (4) |
| C(1)—C(2) | 1.515 (4) | C(9)—C(10) | 1.384 (4) |
| C(1)—O(1) | 1.285 (4) | C(9)—N(2) | 1.327 (4) |
| C(1)—O(2) | 1.225 (4) | C(10)—C(11) | 1.389 (5) |
| C(2)—C(3) | 1.392 (4) | C(11)—C(12) | 1.390 (5) |
| C(2)—N(1) | 1.318 (4) | C(12)—C(13) | 1.389 (4) |
| C(3)—C(4) | 1.386 (4) | C(13)—C(14) | 1.516 (4) |
| C(4)—C(5) | 1.394 (5) | C(13)—N(2) | 1.327 (4) |
| C(5)—C(6) | 1.387 (4) | C(14)—O(7) | 1.281 (4) |
| C(6)—C(7) | 1.514 (4) | C(14)—O(8) | 1.218 (4) |
| Na(1)···O(2 ⁱ) | 2.409 (3) | Na(1)···O(8 ⁱⁱⁱ) | 2.450 (3) |
| Na(1)···O(5 ⁱⁱ) | 2.683 (3) | Na(1)···O(9 ⁱⁱ) | 2.331 (3) |
| Na(1)···O(6 ⁱⁱ) | 2.500 (3) | Na(1)···O(10) | 2.363 (3) |
| N(1)—Fe(1)—N(2) | 168.26 (9) | C(6)—C(7)—O(4) | 113.5 (2) |
| N(1)—Fe(1)—O(1) | 76.20 (9) | O(3)—C(7)—O(4) | 126.2 (3) |
| N(2)—Fe(1)—O(1) | 109.04 (9) | C(9)—C(8)—O(5) | 113.7 (2) |
| N(1)—Fe(1)—O(4) | 75.82 (9) | C(9)—C(8)—O(6) | 122.0 (3) |
| N(2)—Fe(1)—O(4) | 99.57 (9) | O(5)—C(8)—O(6) | 124.3 (3) |
| O(1)—Fe(1)—O(4) | 151.39 (9) | C(8)—C(9)—C(10) | 128.3 (3) |
| N(1)—Fe(1)—O(5) | 93.74 (9) | C(8)—C(9)—N(2) | 111.1 (2) |
| N(2)—Fe(1)—O(5) | 75.82 (9) | C(10)—C(9)—N(2) | 120.5 (3) |
| O(1)—Fe(1)—O(5) | 91.83 (9) | C(9)—C(10)—C(11) | 118.1 (3) |
| O(4)—Fe(1)—O(5) | 95.8 (1) | C(10)—C(11)—C(12) | 120.5 (3) |
| N(1)—Fe(1)—O(7) | 114.22 (9) | C(11)—C(12)—C(13) | 117.9 (3) |
| N(2)—Fe(1)—O(7) | 76.20 (9) | C(12)—C(13)—C(14) | 128.4 (3) |
| O(1)—Fe(1)—O(7) | 96.13 (9) | C(12)—C(13)—N(2) | 120.5 (3) |
| O(4)—Fe(1)—O(7) | 89.9 (1) | C(14)—C(13)—N(2) | 111.1 (2) |
| O(5)—Fe(1)—O(7) | 152.00 (8) | C(13)—C(14)—O(7) | 113.6 (2) |
| C(2)—C(1)—O(1) | 113.3 (2) | C(13)—C(14)—O(8) | 120.5 (3) |
| C(2)—C(1)—O(2) | 120.6 (2) | O(7)—C(14)—O(8) | 125.9 (3) |
| O(1)—C(1)—O(2) | 126.1 (3) | Fe(1)—N(1)—C(2) | 118.3 (2) |
| C(1)—C(2)—C(3) | 127.7 (2) | Fe(1)—N(1)—C(6) | 119.0 (2) |
| C(1)—C(2)—N(1) | 111.4 (2) | C(2)—N(1)—C(6) | 122.6 (3) |
| C(3)—C(2)—N(1) | 120.9 (3) | Fe(1)—N(2)—C(9) | 119.1 (2) |
| C(2)—C(3)—C(4) | 117.7 (3) | Fe(1)—N(2)—C(13) | 118.2 (2) |
| C(3)—C(4)—C(5) | 120.6 (3) | C(9)—N(2)—C(13) | 122.5 (2) |
| C(4)—C(5)—C(6) | 118.0 (3) | Fe(1)—O(1)—C(1) | 119.5 (2) |
| C(5)—C(6)—C(7) | 128.7 (3) | Fe(1)—O(4)—C(7) | 120.6 (2) |
| C(5)—C(6)—N(1) | 120.2 (3) | Fe(1)—O(5)—C(8) | 119.6 (2) |
| C(7)—C(6)—N(1) | 111.1 (2) | Fe(1)—O(7)—C(14) | 119.8 (2) |
| C(6)—C(7)—O(3) | 120.4 (3) | | |
| O(2 ⁱ)···Na(1)···O(5 ⁱⁱ) | 84.14 (8) | O(6 ⁱⁱ)···Na(1)···O(9 ⁱⁱ) | 86.5 (1) |
| O(2 ⁱ)···Na(1)···O(6 ⁱⁱ) | 130.9 (1) | O(8 ⁱⁱⁱ)···Na(1)···O(9 ⁱⁱ) | 90.3 (1) |
| O(5 ⁱⁱ)···Na(1)···O(6 ⁱⁱ) | 50.43 (7) | O(2 ⁱ)···Na(1)···O(10) | 91.4 (1) |
| O(2 ⁱ)···Na(1)···O(8 ⁱⁱⁱ) | 91.20 (9) | O(5 ⁱⁱ)···Na(1)···O(10) | 105.7 (1) |
| O(5 ⁱⁱ)···Na(1)···O(8 ⁱⁱⁱ) | 168.3 (1) | O(6 ⁱⁱ)···Na(1)···O(10) | 85.3 (1) |
| O(6 ⁱⁱ)···Na(1)···O(8 ⁱⁱⁱ) | 136.99 (9) | O(8 ⁱⁱⁱ)···Na(1)···O(10) | 85.0 (1) |
| O(2 ⁱ)···Na(1)···O(9 ⁱⁱ) | 105.9 (1) | O(9 ⁱⁱ)···Na(1)···O(10) | 162.2 (1) |
| O(5 ⁱⁱ)···Na(1)···O(9 ⁱⁱ) | 80.7 (1) | | |

Symmetry codes: (i) $x, -y, \frac{1}{2} + z$; (ii) $x, y, 1 + z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

The structure was solved by the Patterson method and successive Fourier maps. H atoms were found from difference Fourier syntheses and their positions were subsequently refined. Anomalous dispersion was taken into account. The absolute configuration was confirmed; the Flack enantiopole parameter χ was refined [$\chi = 0.06(2)$] using the expression $F_c = [(1-\chi)F(h)^2 + \chi F(-h)^2]^{1/2}$. All calculations were performed using *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985) on a VAX 6310 and a PC computer.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Data reduction: *CRYSTALS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine

structure: *CRYSTALS*. Molecular graphics: *CRYSTALS*. Software used to prepare material for publication: *CRYSTALS*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1094). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 840–843

{*exo*-2,7,11- μ_3 -[(μ -H)₂Cu(PPh₃)]-2-(PPh₃)-*closo*-2,1-CuTeB₁₀H₈}.CH₂Cl₂

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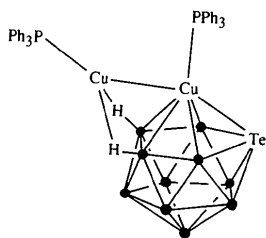
Abstract

The title compound, 2-triphenylphosphine-2,7,11- μ_3 -[dihydro(triphenylphosphine)copper-2Cu,7H,11H]-octahydro-2-cupra-1-tellura-*closo*-dodecaborane (C₃₆H₄₀B₁₀Cu₂P₂Te.CH₂Cl₂), has a triangulated icosahedral

closo-CuTeB₁₀ cage, somewhat distorted from regular, with the Cu and Te atoms adjacent. A triphenylphosphine ligand is attached to the Cu cage atom and a [Cu*(PPh₃)] unit is bonded to one CuB₂ triangular face of the CuTeB₁₀ cage *via* one Cu—Cu* bond and two B—H—Cu* interactions. Whereas the Cu cage atom is essentially symmetrically bonded in the CuB₂ face, the *exo*-cage Cu* atom is unsymmetrically bonded.

Comment

The overall cluster structure of the title compound (1), Fig. 1, may be viewed as a triangulated icosahedron, somewhat distorted from regular, fused to a tetrahedron of Cu₂B₂ atoms with a common CuB₂ face. Alternatively, if Wade's (1976) electron-counting rules are rigidly applied, the *exo*-cluster unit [Cu₃(PPh₃)] behaves as a single-electron 'ligand' *L* to the cage and together, 'Cu₂(PPh₃)*L*', Te and ten B—H units generate a 12 vertex *closo*-cluster. An analogous cupradicarbaborane {*exo*-3,4,8-[(μ-H)₂Cu(PPh₃)]-3-PPh₃-*closo*-3,1,2-Cu₂B₉H₉}, (2), has been reported (Do, Kang, Knobler & Hawthorne, 1987).



The Cu₃—H—B interactions are notable features. The *exo*-Cu₃...B₇ and *exo*-C₃...B₁₁ distances [2.207 (4) and 2.161 (5) Å, respectively] are slightly longer than the cage Cu₂—B₇ and Cu₂—B₁₁ distances [2.132 (5) and 2.138 (5) Å, respectively]. The similarity of these bonding distances is rather unexpected since the bonding interactions of the two Cu atoms must be quite different in each case. Moreover, the bonding of the Cu₂ atom to the B atoms of the TeB₄ face is almost symmetrical, but the *exo*-Cu₃ atom is asymmetrically bonded to B₇ and B₁₁. Asymmetric Cu—B bonding was also reported for the *exo*-cage Cu atom in (2) (Do *et al.*, 1987), but in this compound one Cu—H—B bond was adjacent to a C atom and the other adjacent to a B atom.

The Cu₂—B₃ and Cu₂—B₆ linkages which flank the Te atom [2.302 (5) and 2.320 (5) Å, respectively] are significantly longer than the Cu₂—B₇ or Cu—B₁₁ distances. Indeed, the former are longer than any previously reported Cu—B distances in *closo* Cu-containing metallaheteroboranes; in (2), the Cu—B distances are between 2.140 (5) and 2.216 (5) Å (Do *et al.*, 1987) and in the related compound [3-(PPh₃)-4-(SMe₂)-*closo*-3,1,2-Cu₂B₉H₁₀] the Cu—B distances

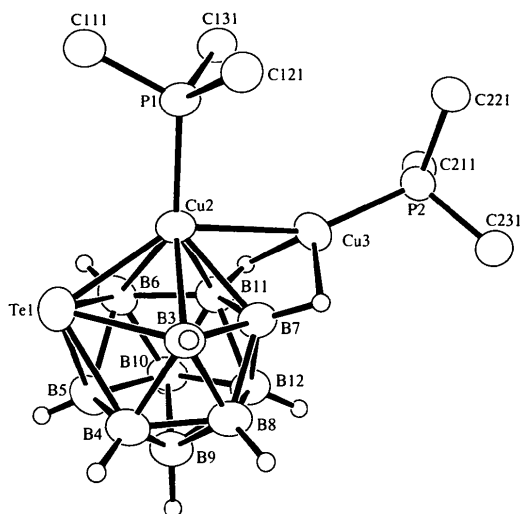


Fig. 1. Molecular structure of {*exo*-2,7,11-μ₃-[(μ-H)₂Cu(PPh₃)]-2-(PPh₃)-*closo*-2,1-CuTeB₁₀H₈}. Displacement ellipsoids are shown at the 50% probability level. For clarity, all phenyl ring atoms other than the ψ-C atoms have been omitted and H atoms are drawn as small circles of arbitrary radii.

are between 2.130 (4) and 2.212 (4) Å (Hamilton & Welch, 1991).

The Cu—Cu distance [2.6915 (8) Å] falls within the very wide range of 2.35–3.6 Å which has been reported by Do *et al.* (1987) and it is significantly longer than the inter-copper distance in (2). The Cu—Te distance of 2.499 (3) Å is notably short, shorter even than the previously reported Cu^I—Te distances which lie within the range 2.535 (1)–3.574 (4) Å (Rice, 1978; Chada & Drake, 1985). This suggests a strong bond between the Cu and Te atoms, possibly of the two-electron two-centre variety.

Experimental

The reaction between equimolar amounts of the caesium salt of the [nido-7-TeB₁₀H₁₁][−] anion and [Cu(NO₃)(PPh₃)₂] in ethanol at room temperature afforded the title compound in 28% yield after preparative chromatography on silica [eluant: CH₂Cl₂/cyclohexane (7:2)].

Crystal data

[Cu₂Te(H)₂(B₁₀H₈)-
(C₁₈H₁₅P)₂].CH₂Cl₂

M_r = 982.38

Triclinic

P $\bar{1}$

a = 11.6125 (9) Å

b = 12.6271 (12) Å

c = 16.4500 (14) Å

α = 86.415 (7)°

β = 87.307 (7)°

γ = 63.307 (7)°

V = 2150.3 (3) Å³

Z = 2

D_x = 1.517 Mg m^{−3}

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 25

reflections

θ = 15.0–17.5°

μ = 1.880 mm^{−1}

T = 293 (2) K

Prism

0.70 × 0.30 × 0.20 mm

Pale yellow

Data collection

Nicolet P3/F four-circle diffractometer
 $\theta_{\max} = 25.05^\circ$
 $h = -13 \rightarrow 13$
 $\omega/2\theta$ scans
 $k = -14 \rightarrow 15$
Absorption correction:
 $l = 0 \rightarrow 19$
 ψ scans
3 standard reflections
 $T_{\min} = 0.482$, $T_{\max} = 0.638$
monitored every 100 reflections
7254 measured reflections
intensity decay: none
7254 independent reflections
6365 observed reflections
 $[I > 2\sigma(I)]$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.0482$
 $wR(F^2) = 0.1267$
 $S = 1.066$
7251 reflections
527 parameters
 $w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 2.9792P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.008$
 $\Delta\rho_{\max} = 1.249 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.341 \text{ e } \text{\AA}^{-3}$
Extinction correction: none
Atomic scattering factors
from *International Tables for Crystallography* (1992), Vol. C, Tables 4.2.6.8 and 6.1.1.4

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

| | x | y | z | U_{eq} |
|------|--------------|-------------|-------------|-----------------|
| Te1 | 1.10250 (3) | 0.26730 (3) | 0.25502 (2) | 0.05607 (13) |
| Cu2 | 0.93278 (5) | 0.47563 (4) | 0.27027 (3) | 0.03992 (14) |
| Cu3 | 0.67801 (5) | 0.61695 (5) | 0.25821 (4) | 0.0516 (2) |
| P1 | 0.98418 (10) | 0.61502 (9) | 0.30370 (6) | 0.0373 (2) |
| C111 | 1.1439 (4) | 0.5671 (4) | 0.3455 (2) | 0.0420 (9) |
| C112 | 1.2506 (5) | 0.4992 (5) | 0.2972 (3) | 0.0609 (12) |
| C113 | 1.3743 (5) | 0.4645 (5) | 0.3244 (4) | 0.077 (2) |
| C114 | 1.3925 (5) | 0.4931 (5) | 0.4008 (4) | 0.0696 (15) |
| C115 | 1.2887 (5) | 0.5581 (5) | 0.4489 (3) | 0.0672 (14) |
| C116 | 1.1644 (5) | 0.5947 (4) | 0.4219 (3) | 0.0516 (11) |
| C121 | 0.9865 (4) | 0.7128 (4) | 0.2184 (2) | 0.0399 (9) |
| C122 | 0.9468 (4) | 0.7015 (4) | 0.1431 (2) | 0.0467 (10) |
| C123 | 0.9495 (5) | 0.7735 (5) | 0.0767 (3) | 0.0575 (12) |
| C124 | 0.9902 (5) | 0.8580 (5) | 0.0859 (3) | 0.0638 (14) |
| C125 | 1.0305 (6) | 0.8707 (5) | 0.1607 (3) | 0.0658 (14) |
| C126 | 1.0304 (5) | 0.7982 (4) | 0.2270 (3) | 0.0559 (12) |
| C131 | 0.8752 (4) | 0.7077 (4) | 0.3814 (2) | 0.0401 (9) |
| C132 | 0.8465 (5) | 0.6486 (4) | 0.4481 (3) | 0.0539 (11) |
| C133 | 0.7679 (6) | 0.7120 (6) | 0.5108 (3) | 0.0699 (15) |
| C134 | 0.7147 (6) | 0.8319 (6) | 0.5071 (3) | 0.083 (2) |
| C135 | 0.7387 (7) | 0.8918 (5) | 0.4415 (4) | 0.081 (2) |
| C136 | 0.8205 (5) | 0.8297 (4) | 0.3784 (3) | 0.0600 (12) |
| P2 | 0.50903 (10) | 0.79065 (9) | 0.25715 (7) | 0.0407 (2) |
| C211 | 0.3994 (4) | 0.8122 (3) | 0.3443 (2) | 0.0412 (9) |
| C212 | 0.4515 (5) | 0.7700 (4) | 0.4205 (3) | 0.0562 (12) |
| C213 | 0.3718 (6) | 0.7898 (5) | 0.4885 (3) | 0.0661 (14) |
| C214 | 0.2406 (5) | 0.8494 (5) | 0.4813 (3) | 0.0636 (13) |
| C215 | 0.1881 (5) | 0.8902 (5) | 0.4061 (3) | 0.0645 (14) |
| C216 | 0.2673 (4) | 0.8722 (4) | 0.3374 (3) | 0.0531 (11) |
| C221 | 0.5365 (4) | 0.9210 (4) | 0.2518 (2) | 0.0416 (9) |
| C222 | 0.6324 (5) | 0.9234 (4) | 0.1985 (3) | 0.0598 (12) |
| C223 | 0.6569 (5) | 1.0205 (5) | 0.1921 (4) | 0.0682 (14) |
| C224 | 0.5871 (5) | 1.1166 (5) | 0.2382 (4) | 0.0670 (14) |
| C225 | 0.4910 (6) | 1.1164 (5) | 0.2900 (4) | 0.074 (2) |
| C226 | 0.4661 (5) | 1.0193 (4) | 0.2969 (3) | 0.0568 (12) |
| C231 | 0.4172 (4) | 0.8070 (4) | 0.1666 (2) | 0.0438 (9) |
| C232 | 0.4067 (5) | 0.7095 (4) | 0.1419 (3) | 0.0622 (13) |
| C233 | 0.3439 (6) | 0.7160 (6) | 0.0703 (4) | 0.079 (2) |
| C234 | 0.2937 (6) | 0.8185 (6) | 0.0235 (3) | 0.073 (2) |
| C235 | 0.3013 (6) | 0.9169 (5) | 0.0484 (3) | 0.0708 (15) |
| C236 | 0.3631 (5) | 0.9111 (4) | 0.1199 (3) | 0.0589 (12) |

| | | | | |
|------|-------------|-------------|------------|-------------|
| B3 | 0.9670 (5) | 0.3842 (4) | 0.1486 (3) | 0.0409 (10) |
| B4 | 0.9913 (5) | 0.2254 (5) | 0.1582 (3) | 0.0478 (11) |
| B5 | 0.9593 (6) | 0.1847 (4) | 0.2714 (3) | 0.0503 (12) |
| B6 | 0.9157 (5) | 0.3163 (4) | 0.3378 (3) | 0.0448 (11) |
| B7 | 0.8065 (4) | 0.4684 (4) | 0.1831 (3) | 0.0361 (9) |
| B8 | 0.8459 (5) | 0.3410 (4) | 0.1269 (3) | 0.0423 (10) |
| B9 | 0.8476 (5) | 0.2244 (4) | 0.1940 (3) | 0.0465 (11) |
| B10 | 0.8003 (5) | 0.2811 (4) | 0.2932 (3) | 0.0460 (11) |
| B11 | 0.7775 (5) | 0.4293 (4) | 0.2905 (3) | 0.0399 (10) |
| B12 | 0.7302 (5) | 0.3749 (4) | 0.2070 (3) | 0.0419 (10) |
| C11S | 0.3905 (6) | 0.3927 (4) | 0.0978 (2) | 0.218 (2) |
| C12S | 0.3357 (4) | 0.2128 (4) | 0.0393 (3) | 0.202 (2) |
| C1S | 0.3189 (15) | 0.3615 (11) | 0.0278 (7) | 0.192 (3) |

Table 2. Selected geometric parameters (\AA , $^\circ$)

| | | | |
|------------|-------------|-------------|-------------|
| Te1—B4 | 2.323 (5) | Cu2—B3 | 2.302 (5) |
| Te1—B5 | 2.326 (6) | Cu2—B6 | 2.320 (5) |
| Te1—B6 | 2.360 (5) | Cu2—Cu3 | 2.6915 (8) |
| Te1—B3 | 2.360 (5) | Cu3—B11 | 2.161 (5) |
| Te1—Cu2 | 2.4991 (6) | Cu3—P2 | 2.1893 (12) |
| Cu2—B7 | 2.132 (5) | Cu3—B7 | 2.207 (4) |
| Cu2—B11 | 2.138 (5) | Cu3—H7 | 1.89 (4) |
| Cu2—P1 | 2.2028 (11) | Cu3—H11 | 1.83 (6) |
| B11—Cu2—P1 | 138.47 (13) | P1—Cu2—Cu3 | 96.17 (4) |
| P1—Cu2—B6 | 135.84 (14) | Te1—Cu2—Cu3 | 143.09 (2) |
| P1—Cu2—Te1 | 120.73 (4) | P2—Cu3—Cu2 | 152.49 (4) |

Absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Table 4.2.4.2). Geometrical restraints were applied to phenyl rings so that they were flat with overall C_{2v} symmetry. While phenyl H atoms were constrained in calculated positions, the borane cage atoms were located on a difference synthesis and were freely refined.

Rigid bond restraints were applied to the CH_2Cl_2 solvate molecule which displayed considerable thermal motion.

Data collection: Nicolet P3/F software (Nicolet, 1984). Cell refinement: Nicolet P3/F software. Data reduction: *SYNTLP* (McDonald, 1984). Program(s) used to solve structure: *SHELX90* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Ternary Ni^{II} Complexes: Bisglycinato(1,10-phenanthroline)nickel(II) Trihydrate and Bisglycinato(2,2'-bipyridine)nickel(II) Trihydrate

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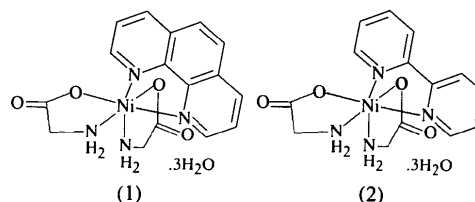
Abstract

The two title compounds, $[\text{Ni}(\text{C}_2\text{H}_4\text{NO}_2)_2(\text{C}_{12}\text{H}_8\text{N}_2)] \cdot 3\text{H}_2\text{O}$ and $[\text{Ni}(\text{C}_2\text{H}_4\text{NO}_2)_2(\text{C}_{10}\text{H}_8\text{N}_2)] \cdot 3\text{H}_2\text{O}$, are isostructural. Their structures consist of discrete complex molecules, together with water molecules of crystallization. All ligands are bidentate and the coordination polyhedron is a distorted octahedron. Distinct segregation of hydrophilic and hydrophobic regions appears along the *c* axis. In the hydrophilic region a three-dimensional network of hydrogen bonds is formed involving principally the water molecules. Aromatic interactions exist in the hydrophobic region.

Comment

It is widely known that crystallization kinetics and consequently crystal morphology may be influenced by the presence of small amounts of foreign substances (Addadi, Berkovitch-Yellin, Weissbuch, Van Mil, Shimon, Lahav & Leiserowitz, 1985). In a previous study (Skoulika, Michaelides & Aubry, 1991), we reported the crystallization of the complex $[\text{Ni}(\beta\text{-ala})_2] \cdot 2\text{H}_2\text{O}$ ($\beta\text{-ala}$ is $\beta\text{-alanine}$) in the presence of glycine. By stereochemical considerations we showed that it is probably the ternary complex $[\text{Ni}(\beta\text{-ala})(\text{gly})] \cdot 2\text{H}_2\text{O}$ (gly is glycine) that is selectively adsorbed onto a particular crystal face and inhibits deposition onto that face. As part of a systematic investigation of similar systems, we undertook a study of the crystallization of $[\text{Ni}(\text{gly})_2] \cdot 2\text{H}_2\text{O}$ in the presence of the aromatic amines

1,10-phenanthroline and 2,2'-bipyridine. In order to elucidate the geometry of the host-guest system, we determined the crystal structures of the two title ternary complexes $[\text{Ni}(\text{gly})_2(o\text{-phen})] \cdot 3\text{H}_2\text{O}$ (1) (*o*-phen is 1,10-phenanthroline) and $[\text{Ni}(\text{gly})_2(2,2'\text{-bpy})] \cdot 3\text{H}_2\text{O}$ (2) (2,2'-bpy is 2,2'-bipyridine).



Compounds (1) and (2) are isostructural. Their crystal structures consist of discrete complex molecules, which are very similar in shape and geometry, and water molecules of crystallization (Figs. 1 and 2). The coordination polyhedron around the Ni^{II} atom is represented by a distorted octahedron. The equatorial positions [O(1), N(3), N(2) and O(3)] are occupied by the N atom and one O atom of one glycine ligand, the O atom of a second glycine ligand and one N atom belonging to the N–N ligand, while the axial positions [N(1) and N(4)] are occupied by the N atom of the second glycine and the remaining N atom of the N–N ligand. The geometry of the glycine ligands is as expected (Freeman & Guss, 1968). They have ‘bite’ angles of 80.7° for compound (1) and 80.6 and 80.7° for (2). The *o*-phenanthroline moiety is not exactly planar; the dihedral angles between the mean plane through the central six-membered ring and those through the adjacent aromatic rings are 3.7 and 1.8°. It is interesting to note that recently the structure of another isomer, $[\text{Ni}(\text{gly})_2(\text{bpy})] \cdot 5.5\text{H}_2\text{O}$ (space group *Fddd*), has been published (Žák, Głowiak, Kameníček & Březina, 1990). In this case, compared with the title structures, one glycine O atom of the equatorial plane is replaced by a glycine N atom, while the remaining O atom occupies an axial position.

The pyridine rings of the bipyridine ligand are planar but twisted with respect to each other by an angle of 9.4° due to chelation strain. The *o*-phen and 2,2'-bpy ligands have bite angles of 78.6 and 77.9°, respectively. Their dimensions compare well with those observed in other metal complexes (Antolini, Marcotrigiano, Menabue, Pellacani, Saladini & Sola, 1985; Poletti, Stojaković, Prelesnik & Manojlović-Muir, 1990). The bond distances between Ni^{II} and the ligands are within the ranges of literature values (Poletti, Stojaković, Prelesnik & Manojlović-Muir, 1990; Freeman & Guss, 1978). The equatorial coordination plane, from which the Ni atom deviates towards the axial N(1) atom by 0.033 Å for compound (1) and 0.038 Å for (2), shows a very slight tetrahedral distortion.

The molecules are ordered in the crystal lattice so that distinct segregation of hydrophobic and hydrophilic