

Acta Crystallographica Section C

**Crystal Structure
Communications**

ISSN 0108-2701

**[*N,N*-Bis(2-hydroxyethyl)dithiocarbamato-*S*]bis(triphenylphosphine-*P*)copper(I) tri-
phenylphosphine solvate**

Fangfang Jian *et al.*

Electronic paper

This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

© 2000 International Union of Crystallography • Printed in Great Britain – all rights reserved

[*N,N*-Bis(2-hydroxyethyl)dithiocarbamate-*S*]bis(triphenylphosphine-*P*)-phosphine solvate

Fangfang Jian,^a Fengli Bei,^a Lude Lu,^a Xujie Yang,^a Xin Wang,^a Ibrahim Abdul Razak,^b S. Shanmuga Sundara Raj^b and Hoong-Kun Fun^{b*}

^aMaterial Chemistry Laboratory, Nanjing University of Science and Technology, Nanjing 210094, People's Republic of China, and ^bX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

Received 10 May 2000

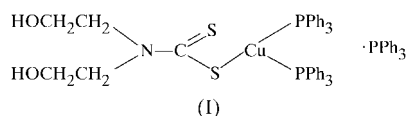
Accepted 30 May 2000

Data validation number: IUC0000154

In the title compound, [Cu(C₅H₁₀NO₂S₂)(C₁₈H₁₅P)₂] \cdot C₁₈H₁₅P, the Cu atom is in a distorted tetrahedral coordination, with two triphenylphosphine P atoms and two S atoms from an *N,N*-bis(2-hydroxyethyl)dithiocarbamate ligand occupying the vertices. The crystal structure is characterized by alternate layers of complex and triphenylphosphine molecules.

Comment

Group 12 metals form a wide range of tertiary phosphine (R₃P) complexes of the general stoichiometry L_nMX (L = R₃P, n = 1–4; Attar, Alcock, Frye *et al.*, 1991; Attar, Alcock, Bowmaker *et al.*, 1991; Alyea *et al.*, 1992). The value of n and the net electronic charge on the complex are somewhat dependent upon the coordinating ability of X. Also, the transition metal dithiolates containing MS₄ chromophores show interesting variations in their reactions with Lewis bases (Cordes & Draganjac, 1988; Venkatachalam *et al.*, 1996).



The asymmetric unit of the title structure, (I), contains one molecule of [Cu{(C₂H₄OH)₂S₂CN}(PPh₃)₂] together with a molecule of uncoordinated triphenylphosphine. The Cu1 atom is in a distorted tetrahedral environment, with the P atoms of the triphenylphosphine ligands and the S atoms of the bidentate dithiocarbamate ligand occupying the vertices. The angles around Cu1 range between 74.77 (4)° (S1–Cu1–S2) and 124.51 (4)° (P1–Cu1–P2). The small S1–Cu1–S2 bond angle is due to the restricted bite angle of the ligand. The P1–Cu1–P2 bond angle is comparable with values reported for other bis(triphenylphosphine)copper(I) compounds (Kara-

glannidis *et al.*, 1989; Long *et al.*, 1996). The Cu–P distances are comparable to those found in (PPh₃)₂Cu(C₂H₄N₂S₂)Br (Long *et al.*, 1996). The bond lengths of Cu–S1 and S2–C37 are longer than those of Cu–S2 and S1–C37, respectively. One of the S–C bond lengths (S1–C37) is close to the C=S distance of 1.69 Å, while S2–C37 shows a value intermediate between single- and double-bond distances, indicating partial double-bond character. The longer Cu–S bond length (Cu1–S1) lies adjacent to C–S bond which is closer to the C=S bond distance. The same characteristic is found in the structure of [Ni(pipdte)(PPh₃)₂]ClO₄(PPh₃) \cdot H₂O (pipdte is piperidinedithiocarbamate; Venkatachalam *et al.*, 1996), but different from the complex of [Au(S₂CPh)(PPh₃)₂] (Lanfredi *et al.*, 1992), where the shorter Au–S bond lies adjacent to the C–S bond length which is closer to the C=S bond distance. The four-membered Cu₂C is nearly planar with normal bond lengths. The bond length of C37–N1 [1.360 (4) Å] has double-bond character, but slightly longer than those observed in a related structure (Jian *et al.*, 1999). All the phenyl rings show normal bond parameters. The P–C bond distances are also normal. The C–P–C angles deviate appreciably from the ideal tetrahedral angle of 109.5°. The crystal structure is characterized by alternate layers of the complexes and the triphenylphosphine ligands which lie parallel to the *bc* plane. The structure is stabilized by C–H $\cdots\pi$ and van der Waals interactions (Table 2).

Experimental

Sodium *N,N*-bis(2-hydroxyethyl)dithiocarbamate, Na[S₂CN(C₂H₄OH)₂], was dissolved in H₂O. CuCl₂ was then added at room temperature with stirring. The brown reaction mixture was filtered and the filtrate was washed with water and dried overnight *in vacuo*. The dried brown solid was dissolved in EtOH and triphenylphosphine was added at 253–263 K with stirring and the resulting solution refluxed for 4–5 h. Upon cooling, the colourless solution obtained was filtered and left to evaporate. After a few days, colourless solids were separated out and recrystallized from EtOH/CHCl₃. Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature of the EtOH solvent.

Crystal data

[Cu(C₅H₁₀NO₂S₂)(C₁₈H₁₅P)₂] \cdot C₁₈H₁₅P
M_r = 1030.61
Triclinic, P $\bar{1}$
a = 12.2892 (4) Å
b = 13.1612 (5) Å
c = 19.3696 (7) Å
α = 95.512 (1)°
β = 103.177 (1)°
γ = 116.801 (1)°
V = 2650.47 (16) Å³

Z = 2
D_x = 1.291 Mg m⁻³
Mo Kα radiation
Cell parameters from 7234 reflections
θ = 1.11–28.34°
μ = 0.624 mm⁻¹
T = 293 (2) K
Slab, colourless
0.40 × 0.30 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (SADABS; Sheldrick, 1996)
T_{min} = 0.788, T_{max} = 0.918
18 118 measured reflections

11 791 independent reflections
5663 reflections with I > 2σ(I)
R_{int} = 0.065
θ_{max} = 27.5°
h = -15 → 14
k = -17 → 17
l = -18 → 25

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.057$
 $wR(F^2) = 0.139$
 $S = 0.86$
 11791 reflections
 624 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0271P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.44 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.76 \text{ e } \text{\AA}^{-3}$

Table 1
 Selected geometric parameters (\AA , $^\circ$).

Cu1—P2	2.2595 (9)	S1—C37	1.702 (4)
Cu1—P1	2.2849 (10)	S2—C37	1.710 (4)
Cu1—S2	2.3946 (11)	N1—C37	1.360 (5)
Cu1—S1	2.4287 (10)		
P2—Cu1—P1	124.51 (4)	P2—Cu1—S1	122.04 (4)
P2—Cu1—S2	112.35 (4)	P1—Cu1—S1	102.49 (4)
P1—Cu1—S2	109.85 (4)	S2—Cu1—S1	74.77 (4)

Table 2
 Hydrogen-bonding geometry (\AA , $^\circ$).

$Cg1$ and $Cg2$ are the centroids of the C19–C24 and C7–C12 rings, respectively.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C12—H12A \cdots Cg1	0.93	2.84	3.633	144
C44—H44A \cdots Cg2 ¹	0.93	2.97	3.821	153

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

After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms. The O1 atom in one of the hydroxyethyl groups is disordered and was refined with an occupancy ratio of of 78:22.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/622004. SSSR thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

References

Alyea, E. C., Malito, J., Attar, S. & Nelson, J. H. (1992). *Polyhedron*, **18**, 2409–2413.
 Attar, S., Alcock, N. W., Bowmaker, G. A., Frye, J. S., Bearden, W. H. & Nelson, J. H. (1991). *Inorg. Chem.* **30**, 4166–4176.
 Attar, S., Alcock, N. W., Frye, J. S., Bearden, W. H. & Nelson, J. H. (1991). *Inorg. Chem.* **30**, 4743–4753.
 Cordes, A. W. & Draganjac, M. (1988). *Acta Cryst.* **C44**, 363–364.
 Jian, F. F., Zhu, D. R., Fun, H.-K., Chinnakali, K., Razak, I. A. & You, X. Z. (1999). *Acta Cryst.* **C55**, 940–942.
 Karaglannidis, P., Aslanidis, P., Kessissoglou, D. P., Krebs, B. & Dartmann, M. (1989). *Inorg. Chim. Acta*, **156**, 47–56.
 Lanfredi, A. M. M., Ugozzoli, F., Asaro, F., Pellizer, G., Marsich, N. & Camus, A. (1992). *Inorg. Chim. Acta*, **192**, 271–282.
 Long, D. L., Zeng, D. X., Xin, X. Q., Huang, X. Y. & Kang, B. S. (1996). *Synth. React. Inorg. Met-Org. Chem.* **26**, 723–733.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
 Venkatachalam, V., Ramalingam, K., Akilan, R., Sivakumar, K., Chinnakali, K. & Fun, H.-K. (1996). *Polyhedron*, **15**, 1289–1294.