

**Crystal data**[Zn(C<sub>7</sub>H<sub>13</sub>O<sub>2</sub>)<sub>2</sub>] $M_r = 323.72$ 

Orthorhombic

 $Pbc2_1$  $a = 4.7651 (6) \text{ \AA}$  $b = 9.3404 (15) \text{ \AA}$  $c = 37.066 (6) \text{ \AA}$  $V = 1649.7 (4) \text{ \AA}^3$  $Z = 4$  $D_x = 1.303 \text{ Mg m}^{-3}$  $D_m$  not measured**Data collection**Enraf–Nonius CAD-4  
diffractometer $\omega/2\theta$  scans

Absorption correction:

 $\psi$  scan fitted by spherical  
harmonic functions

(SORTAV; Blessing, 1995)

 $T_{\min} = 0.76$ ,  $T_{\max} = 0.99$ 

4714 measured reflections

1582 independent reflections

**Refinement**Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.055$  $wR(F^2) = 0.159$  $S = 0.908$ 

1582 reflections

173 parameters

H atoms not refined

 $w = 1/[\sigma^2(F_o^2)]$  $(\Delta/\sigma)_{\max} < 0.001$ Cu  $K\alpha$  radiation $\lambda = 1.5418 \text{ \AA}$ Cell parameters from 25  
reflections $\theta = 20\text{--}40^\circ$  $\mu = 2.129 \text{ mm}^{-1}$  $T = 293 (2) \text{ K}$ 

Plate

 $0.175 \times 0.135 \times 0.004 \text{ mm}$ 

Colorless

847 reflections with

 $I > 2\sigma(I)$  $R_{\text{int}} = 0.063$  $\theta_{\max} = 69.81^\circ$  $h = 0 \rightarrow 5$  $k = 0 \rightarrow 11$  $l = 0 \rightarrow 44$ 

3 standard reflections

frequency: 180 min

intensity decay: 3.8%

 $\Delta\rho_{\max} = 0.502 \text{ e \AA}^{-3}$  $\Delta\rho_{\min} = -0.536 \text{ e \AA}^{-3}$ 

Extinction correction:

SHELXL97

Extinction coefficient:

0.0026 (4)

Scattering factors from

International Tables for  
Crystallography (Vol. C)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1286). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 2065–2068**[Co(phen)(thiourea)(H<sub>2</sub>O)Cl<sub>2</sub>]-thiourea  
(phen is 1,10-phenanthroline)**LEOPOLDO SUESCUN, RAÚL A. MARIEZCURRENA AND  
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**Abstract**

The structure of aquadichloro(1,10-phenanthroline)-(thiourea-*S*)cobalt(II)-thiourea (1/1), [CoCl<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)-(CH<sub>4</sub>N<sub>2</sub>S)(H<sub>2</sub>O)]·CH<sub>4</sub>N<sub>2</sub>S, is formed by the neutral cobalt(II) complex and a thiourea molecule. High-spin Co<sup>II</sup> is present in the structure as can be deduced from the Co–N bond distances. The packing of the structure is directed by hydrogen bonds which stabilize the presence of both thiourea molecules.

**Comment**

The change in the electronic configuration of Co<sup>II</sup> cations from high- to low-spin states has been studied

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

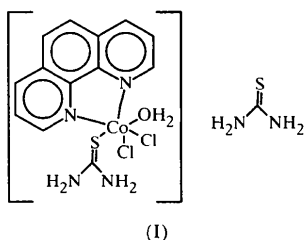
Zn1—O12	1.920 (11)	O12—C11 <sup>i</sup>	1.282 (16)
Zn1—O11	1.946 (8)	O21—C21	1.257 (12)
Zn1—O21	1.975 (11)	O22—C21 <sup>ii</sup>	1.245 (14)
Zn1—O22	1.976 (6)	C11—O12 <sup>iii</sup>	1.282 (16)
O11—C11	1.274 (14)	C21—O22 <sup>iv</sup>	1.245 (14)
O12—Zn1—O11	107.0 (4)	C21—O21—Zn1	113.8 (9)
O12—Zn1—O21	112.7 (3)	C21 <sup>iii</sup> —O22—Zn1	128.0 (7)
O11—Zn1—O21	117.2 (4)	O11—C11—O12 <sup>iii</sup>	120.1 (12)
O12—Zn1—O22	114.6 (4)	O11—C11—C12	120.5 (12)
O11—Zn1—O22	100.1 (3)	O12 <sup>iii</sup> —C11—C12	119.1 (12)
O21—Zn1—O22	104.8 (3)	O22 <sup>iv</sup> —C21—O21	120.5 (11)
C11—O11—Zn1	133.0 (8)	O22 <sup>iv</sup> —C21—C22	120.4 (9)
C11 <sup>i</sup> —O12—Zn1	118.4 (9)	O21—C21—C22	119.1 (11)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *DREAR97* (Blessing, 1987). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ATOMS* (Dowty, 1995). Software used to prepare material for publication: *WINWORD* (Version 5.0).

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extensively (Adams *et al.*, 1993; Faus *et al.*, 1993, 1994). The synthesis and structural characterization of new Co<sup>II</sup> compounds with multiple ligands could add important information to this field. The title compound, (I), can be included in this category.



The octahedral complex [Co(phen)(tu)(H<sub>2</sub>O)Cl<sub>2</sub>] is neutral, with one 1,10-phenanthroline (phen) molecule coordinated in a bidentate manner, one thiourea molecule coordinated through the S atom, two Cl ions placed *trans* with respect to the phen N atoms and a water molecule (Fig. 1). The Co—N bond distances are 2.136(2) and 2.162(2) Å, similar to the Co—N distances in high-spin Co complexes, such as [Co(dmvi)<sub>2</sub>phen]·2CHCl<sub>3</sub> (dmvi is dimethylviolate; Faus *et al.*, 1994). The low-spin complexes reported previously contain shorter Co—N distances of about 1.97(4) Å (Faus *et al.*, 1993). The coordination around the Co atom is a distorted octahedron, expected because of the presence of the rigid phen ligand. Because of the fixed bite of this bidentate molecule, the N11—Co—N12 angle is expected to be near 79° for Co complexes and this leads to a distortion in the entire coordination sphere of the central atom. Table 1 gives selected bond distances and angles about the Co1 atom. The equatorial plane of the octahedron formed by both phen N atoms and both Cl atoms is slightly distorted from planarity, with maximum deviations of 0.072 Å for Cl41 and 0.14 Å for N12. The apical positions are occupied by atoms S21 and O51. Atom Co1 is positioned 0.072 Å from this plane towards atom S21. The S21—Co1—O51 angle is 174.39(6)°, lower than that expected for regular octahedral geometry. This is also a consequence of the rigid coordination of the phen molecule. The phen molecule is planar with a maximum deviation of 0.070(2) Å for N11. The dihedral angle between the phen and equatorial planes is 11.91(8)° with the phen plane tilted towards the water molecule and Co1 shifted by 0.228(2) Å from the mean plane of the phen ligand. This shift is not frequent in complexes containing more than one phen molecule in the coordination sphere (Hazell *et al.*, 1997; Faus *et al.*, 1993). Bond distances and angles of the phen residue have expected values. Both thiourea molecules found in the structure have similar geometric parameters (Table 1). This suggests that the electronic distribution in the thiourea molecule is not strongly affected by coordination. Previous work (Spofford *et al.*, 1970; Baggio *et al.*, 1975) showed that the

thiourea molecule can coordinate through the π cloud, leading to increased C—S and decreased C—N bond lengths. The Co—S distance is 2.5503(9) Å, similar to that observed in other multiple-ligand Co<sup>II</sup> complexes (O'Connor & Amma, 1969) with no π coordination. Both Co—Cl bond distances have expected values, as observed in Co<sup>II</sup> complexes containing phen (Hazell *et al.*, 1997) or thiourea (O'Connor *et al.*, 1969) together with chloride ligands. The packing is mainly directed by hydrogen bonds between both the thiourea and the water molecules. There is an intramolecular hydrogen bond connecting the Cl41 atom with the coordinated thiourea molecule. This has been observed in other thiourea and chloride complexes (O'Connor *et al.*, 1969; Suescun *et al.*, 1999). Table 2 shows the hydrogen-bonding geometry. There are two H atoms belonging to the free thiourea molecule that are not involved in hydrogen bonding. One of them (H62A) is located at distances of 2.91(3), 2.98(3) and 3.06(3) Å from the C131<sup>i</sup>, C141<sup>i</sup> and S21<sup>i</sup> atoms [symmetry code: (i)  $-x, 1-y, 1-z$ ], respectively. These contacts are not to be considered hydrogen bonds because these distances are very close to the sum of the van der Waals radii of the involved atoms (2.95 for Cl···H and 3.0 for S···H; Bondi, 1964). The other H atom (H62B) interacts with the π cloud of the C111<sup>ii</sup> → N12<sup>ii</sup> ring of phen [symmetry code: (ii)  $1-x, 1-y, 1-z$ ], with a distance of 3.38 Å from H62B to the ring centroid and a perpendicular distance of 2.71(4) Å. Fig. 2 shows the unit cell and the hydrogen bonds for the title compound. There is a stacking interaction between equivalent phen ligands ( $1-x, 2-y, 1-z$ ), with a mean perpendicular distance between planes of 3.41 Å. The stacking is along the *c* axis, as can also be seen in Fig. 2.

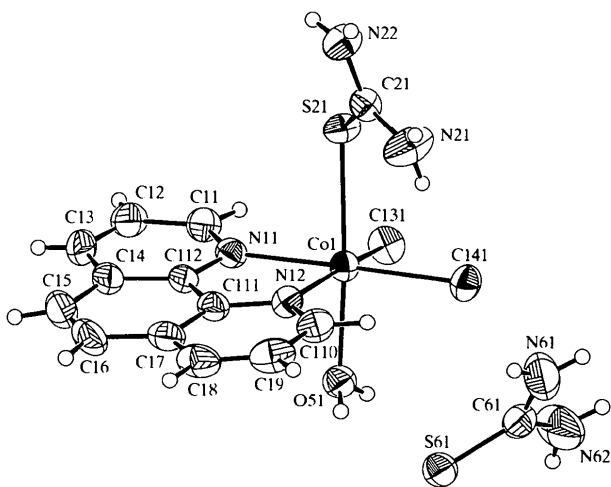


Fig. 1. ZORTEP (Zsolnai, 1995) drawing showing the title compound with that atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been excluded for clarity.

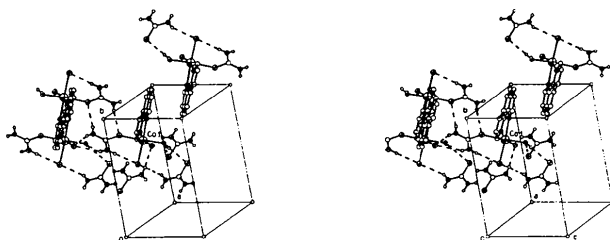


Fig. 2. Stereoview of the crystal packing and unit cell of the title compound. Hydrogen bonds are marked as dashed lines. Some atoms have been excluded for clarity.

## Experimental

The title compound was obtained as a by-product of the synthesis of the 1,10-phenanthroline and thiourea complexes of cobalt. Over 50 ml of a hot acid (HCl 0.5 *M*) water solution of  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  (1 *M*), 25 ml of a water solution of thiourea (1 *M*) and 25 ml of a water solution of 1,10-phenanthroline (1 *M*) were added. The final solution was kept at 353 K for 10 min with continuous stirring and was then left undisturbed to favour slow evaporation. After five weeks of slow evaporation, irregular violet single crystals of the title compound were obtained.

### Crystal data

$[\text{CoCl}_2(\text{C}_{12}\text{H}_8\text{N}_2)(\text{CH}_4\text{N}_2\text{S}) \cdot (\text{H}_2\text{O})] \cdot \text{CH}_4\text{N}_2\text{S}$

$M_r = 480.29$

Triclinic

$P\bar{1}$

$a = 9.719$  (3) Å

$b = 11.682$  (3) Å

$c = 8.7504$  (13) Å

$\alpha = 92.932$  (14)°

$\beta = 90.434$  (17)°

$\gamma = 85.99$  (2)°

$V = 989.8$  (4) Å<sup>3</sup>

$Z = 2$

$D_x = 1.612$  Mg m<sup>-3</sup>

$D_m$  not measured

### Data collection

Rigaku AFC-7S diffractometer

$\theta/2\theta$  scans

Absorption correction:

$\psi$  scan (Molecular Structure Corporation, 1993)

$T_{\min} = 0.685$ ,  $T_{\max} = 0.822$

4814 measured reflections

4544 independent reflections

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.122$

$S = 1.024$

4544 reflections

309 parameters

Mo  $K\alpha$  radiation

$\lambda = 0.71070$  Å

Cell parameters from 6 reflections

$\theta = 18.62$ – $19.67^\circ$

$\mu = 1.364$  mm<sup>-1</sup>

$T = 293$  (2) K

Irregular

$0.30 \times 0.15 \times 0.15$  mm

Violet

3379 reflections with

$I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 27.5^\circ$

$h = 0 \rightarrow 12$

$k = -15 \rightarrow 15$

$l = -11 \rightarrow 11$

3 standard reflections

every 150 reflections

intensity decay: none

$w = 1/[\sigma^2(F_o^2) + (0.0848P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.87$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.51$  e Å<sup>-3</sup>

Extinction correction: none

H atoms treated by a mixture of independent and constrained refinement

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Co1—N12	2.136 (2)	S21—C21	1.709 (3)
Co1—O51	2.149 (2)	C21—N21	1.321 (4)
Co1—N11	2.162 (2)	C21—N22	1.323 (4)
Co1—Cl41	2.4187 (9)	S61—C61	1.708 (3)
Co1—Cl31	2.4498 (9)	C61—N62	1.310 (5)
Co1—S21	2.5503 (9)	C61—N61	1.321 (5)
N12—Co1—O51	82.63 (8)	O51—Co1—S21	174.39 (6)
N12—Co1—N11	77.33 (9)	N11—Co1—S21	84.84 (6)
O51—Co1—N11	90.57 (8)	Cl41—Co1—S21	94.81 (3)
N12—Co1—Cl41	95.83 (7)	Cl31—Co1—S21	89.57 (3)
O51—Co1—Cl41	90.15 (6)	N21—C21—N22	118.1 (3)
N11—Co1—Cl41	172.97 (6)	N21—C21—S21	121.5 (2)
N12—Co1—Cl31	165.15 (6)	N22—C21—S21	120.4 (2)
O51—Co1—Cl31	87.37 (6)	N62—C61—N61	117.5 (4)
N11—Co1—Cl31	91.87 (7)	N62—C61—S61	120.2 (3)
Cl41—Co1—Cl31	95.15 (3)	N61—C61—S61	122.3 (3)
N12—Co1—S21	99.44 (7)		

Table 2. Hydrogen-bonding geometry (Å, °)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N21—H21A $\cdots$ Cl41	0.80 (3)	2.46 (3)	3.252 (3)	171 (3)
N21—H21B $\cdots$ Cl31 <sup>i</sup>	0.85 (3)	2.74 (3)	3.497 (3)	149 (3)
N22—H22A $\cdots$ S21 <sup>ii</sup>	0.82 (3)	2.61 (2)	3.388 (3)	157 (3)
N22—H22B $\cdots$ Cl31 <sup>i</sup>	0.83 (3)	2.45 (3)	3.245 (3)	161 (3)
O51—H51A $\cdots$ S61	0.86 (3)	2.39 (3)	3.206 (2)	158 (3)
O51—H51B $\cdots$ S61 <sup>iii</sup>	0.85 (3)	2.44 (3)	3.264 (2)	162 (3)
N61—H61A $\cdots$ Cl31 <sup>iv</sup>	0.84 (4)	2.44 (4)	3.271 (4)	171 (4)
N61—H61B $\cdots$ Cl41	0.83 (4)	2.71 (3)	3.439 (4)	149 (3)

Symmetry codes: (i)  $x, y, z - 1$ ; (ii)  $-x, 2 - y, -z$ ; (iii)  $1 - x, 1 - y, 1 - z$ ; (iv)  $-x, 1 - y, 1 - z$ .

All the NH<sub>2</sub> residues from both thiourea molecules were restrained to be planar with equal N—H and H $\cdots$ H distances [0.82 (2) and 1.47 (2) Å, respectively]. Both O—H bond distances of the water molecule were restrained to be equal [0.85 (2) Å]. All other H atoms were freely refined; the range of C—H distances for the freely refined atoms is 0.81 (4)–1.00 (3) Å and for the restrained N—H distances, the range is 0.81 (2)–0.86 (2) Å.

Data collection: *MSCI/AF C Diffractometer Control Software* (Molecular Structure Corporation, 1993). Cell refinement: *MSCI/AF C Diffractometer Control Software*. Data reduction: *MSCI/AF C Diffractometer Control Software*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *PLATON98* (Spek, 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1094). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 2068–2070

## Bis[( $\mu$ -*O,O'*-salicylato)(2,2'-bipyridyl)-copper(II)] acetylsalicylic acid dihydrate

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### Abstract

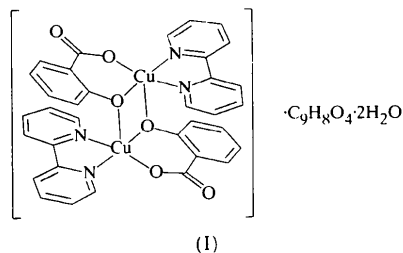
The asymmetric unit of the title compound, bis( $\mu$ -2-hydroxybenzoato-*O*<sup>1</sup>, *O*<sup>2</sup>:*O*<sup>2</sup>)bis[(2,2'-bipyridyl-*N,N'*)copper(II)]-acetylsalicylic acid-water (1/1/2), [Cu<sub>2</sub>(C<sub>7</sub>H<sub>4</sub>O<sub>3</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>].C<sub>9</sub>H<sub>8</sub>O<sub>4</sub>.2H<sub>2</sub>O, contains a binuclear copper complex, one acetylsalicylic acid molecule and two water molecules. Both crystallographically independent Cu<sup>II</sup> atoms of the mixed binuclear unit show analogous distorted elongated square-pyramidal coordination (of the type 4+1). Each Cu<sup>II</sup> atom is surrounded by one 2,2'-bipyridyl chelate and two salicylate

anions. The crystal packing is characterized by several intermolecular hydrogen bonds.

### Comment

Copper(II) complexes with non-steroidal anti-inflammatory drugs (NSAIDs) have been studied extensively since Sorenson (1976) demonstrated that they are more active than their parent drugs and exhibit an anti-ulcer activity. Sorenson also showed that 3,5-diisopropylsalicylic acid which is an inactive agent becomes a potent anti-inflammatory compound when it chelates to copper(II). Moreover, binary complexes of Cu<sup>II</sup> with 3,5-disubstituted salicylates and ternary complexes containing phenanthrolines have been prepared and characterized by Randford *et al.* (1993). These complexes have been tested for antiviral and cytotoxic activities; ternary complexes were ten times as cytotoxic as their binary analogues.

The crystal and molecular structure of the title compound, (I), has been determined in connection with investigations of binary complexes formed by Cu<sup>II</sup> salts with some NSAIDs as indomethacin (Guessous *et al.*, 1998) and niflumic acid (Greenaway *et al.*, 1999) in our department. Following the work of Randford, we decided to synthesize and characterize ternary complexes of Cu<sup>II</sup> with acetylsalicylic acid and heteroaromatic nitrogen bases (1,10-phenanthroline or bipyridine). Their biological activities will be tested and described elsewhere. The title compound consists of a binuclear copper complex, one acetylsalicylic acid and two water molecules. The two Cu atoms are crystallographically independent and present a similar environment. Each Cu<sup>II</sup> atom is surrounded by one 2,2'-bipyridyl chelate *via N,N'* atoms, and two salicylate anions (obtained by deacetylation of acetylsalicylate during the synthesis). Each salicylate anion is bidentate *via* the carboxylate and hydroxyl groups [average bond length 1.895 (2) Å]; moreover, this hydroxyl group is bonded to the second Cu atom [average length 2.428 (3) Å] and so the salicylate anion acts as a bridging ligand between the Cu atoms [average angle Cu—O—Cu' 94.34 (9)°].



The Cu—N bond lengths are all equivalent [average 1.994 (2) Å]. So the coordination polyhedron around each Cu atom is a distorted square pyramid. The Cu and Cu' atoms are, respectively, 0.082 (1) Å below and