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by considerations of sizes and electrostatic and covalent binding forces. The thiosulfate group can act as a monodentate or bridging ligand. When the anion coordinates through sulfur, a significant lengthening of the S—S bond has been observed, in comparison with the dimensions in the ionic moiety (Baggio & Baggio, 1973). Complexes of phenanthroline (hereafter phen) with transition metals have been found to be useful for examining distinctive conformations along the DNA helix (Barton, 1989).

The determination of the structure of the title compound, (I), of empirical formula $Zn_2(phen)_3(S_2O_3)_2$.-$3H_2O$ is of interest to determine the coordination around the metal atoms and to define the role played by the thiosulfate group as a ligand.

cis-Diaquabis(1,10-phenanthroline-N,N')-zinc(II) (1,10-Phenanthroline-N,N')bis(thio-sulfato-S)zincate(II) Monohydrate

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Abstract

The structure of the title compound, $[Zn(C_{12}H_8N_2)_2$-$(H_2O)_2][Zn(S_2O_3)_2(C_{12}H_8N_2)].H_2O$, consists of cationic $[Zn(phen)_2(H_2O)_2]^{2+}$ and anionic $[Zn(phen)(S_2O_3)_2]^{2-}$ moieties. They are connected by hydrogen bonding into a three-dimensional network with the participation of a disordered solvent water molecule. The thiosulfate groups, one of which is disordered, are coordinated through sulfur. The phenanthroline groups coordinate as usual through both N atoms. One of them is planar and the remaining two present shallow-boat conformations.

Comment

To date, very few zinc thiosulfate complexes have been studied by X-ray diffraction (Andreetti, Cavalca, Domiano & Musatti, 1968; Baggio, Baggio & de Perazzo, 1974). Since the Zn^{II} ion has its $3d$ shell completed, there are no ligand-field stabilization effects and thus the stereochemistry of the compounds is determined solely

(I)

The structure consists of cationic $[Zn(phen)_2(H_2O)_2]^{2+}$ and anionic $[Zn(phen)(S_2O_3)_2]^{2-}$ moieties, in the general positions of space group $P\bar{1}$. Figs. 1 and 2 show the structure of both moieties and the crystal packing, respectively. The $[Zn(phen)_2(H_2O)_2]^{2+}$ cation displays distorted octahedral coordination, with phen acting as a bidentate ligand through both N atoms. Two water molecules complete the environment around the metal atom. The same ligand distribution was found in the cation of $[Zn(phen)_2(H_2O)_2]SO_4.6H_2O$ (Hu & Liu, 1991).

The metal coordination geometry of the $[Zn(phen)-(S_2O_3)_2]^{2-}$ anion is distorted tetrahedral, with both thiosulfate groups bonded to the Zn atom through sulfur, and phen occupying the other two coordination sites. The three independent phen molecules in the cell display distances and angles that are in good agreement with the values reported for the free base (Nishigaki, Yoshioka & Nakatsu, 1978) and also in many zinc complexes (Bencini, Midollini & Zanchini, 1989). One of the phen molecules is planar within experimental error; the other two (those labelled *a* and *b* in the diagrams) assume shallow-boat shapes deviating significantly from the expected regular conformation. This can be seen
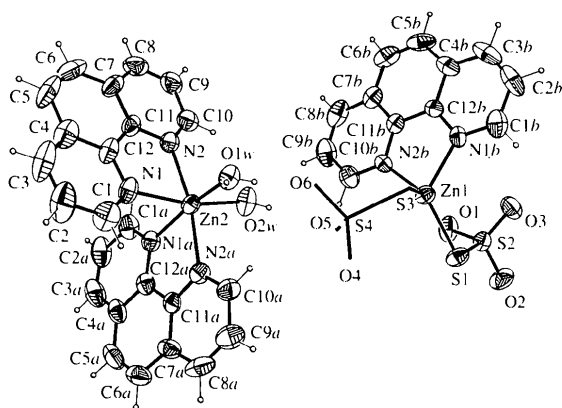
Fig. 1. View of the molecule showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level. The disordered thiosulfate group is depicted schematically.
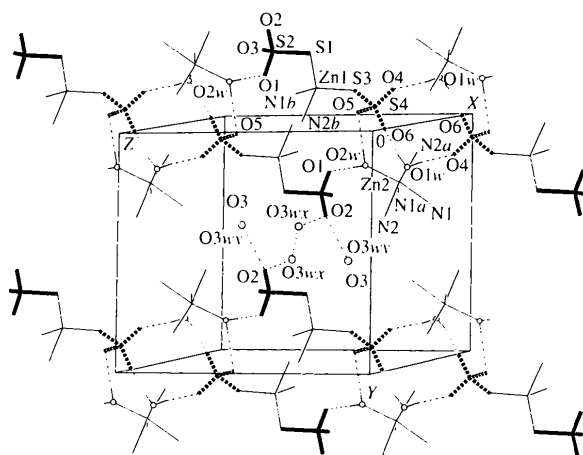

Fig. 2. Simplified packing diagram showing the proposed hydrogen-bonding interactions (water molecules are represented by small circles; phen molecules are omitted except for the metal-bound N atoms; disordered thiosulfates are represented in broken heavy lines by a single group located at the site of largest population).

from the maximum individual departures from the least-squares planes, which are found to be 0.017(11) for C9, 0.100(11) for C9a, and 0.058(11) Å for C1b. The general behaviour, however, is best described by the sums of the squared normalized deviations, $\sum(d_i/\sigma_i)^2$, which amount to 9.12, 360.82 and 169.83, respectively. Comparison with the expected χ-squared value for 95% confidence level and eleven degrees of freedom (19.70) confirms the stated results.

In the octahedral moiety, the mean Zn—N bond length [2.17(3) Å] is in good accordance with those reported in other six-coordinate Zn–phen complexes. In the sulfate salt of this same cation (Hu & Liu, 1991), bond distances which are *trans* to water molecules are significantly longer than the others. In the present structure, however, this feature is only shown in one bond, namely Zn2—N1 [2.210(7) Å]. Owing

to the change of coordination number from six to four, Zn—N distances are considerably shorter in the anion [mean Zn—N 2.07(2) Å]. Zn—O distances are normal for six-coordinate Zn complexes. The thiosulfate groups occupy two coordination sites in the complex anion, being bonded through sulfur. One of the groups is severely disordered and could only be refined with the help of an idealized rigid-body model in four different positions of rather similar occupancy. For this reason it has not been taken into account in the discussion of chemical bonding. The Zn1—S1 bond [2.252(2) Å] is shorter than the sum of the covalent radii (2.33 Å), and slightly shorter than the Zn—S bond length reported in Zn(etu)$_2$S$_2$O$_3$ (2.32 Å; Baggio, Baggio & de Perazzo, 1974). Coordination through sulfur, with a Zn1—S1—S2 angle of 102.6(1)°, reduces the C_{3v} symmetry of the anion, and this can account for the splitting of the asymmetric S—O stretching modes observed in the IR spectrum of this and other thiosulfate complexes (Costamagna & Levitus, 1966). The thiosulfate group keeps the pyramidal shape, with a slight lengthening of the S1—S2 bond, in good agreement with the results observed in other S-bonded thiosulfate complexes (Baggio & Baggio, 1973).

The crystal packing is dominated by hydrogen bonding from coordinated as well as non-coordinated water molecules. Only the H atoms belonging to the former could be located in a difference Fourier synthesis, and all of them were found to be engaged in hydrogen bonding with O atoms from the thiosulfate groups, defining chains parallel to the c axis. Although three out of the four H···O contacts involve O atoms of the disordered thiosulfate group, and accordingly could not be defined with certainty, they span values well within the range of commonly accepted hydrogen bonding distances [O1w—H1wa···O 1.63–2.01, O1w—H1wb···O 1.68–1.81, O2w—H1wa···O 1.70–2.15 Å; normalized values following Jeffrey & Lewis (1978)], which leaves almost no doubt about the character of the interactions. The fourth H···O contact is well defined: O2w—H2wb···O1 1.74 Å.

The disordered water molecule of crystallization O3wx/O3wy seems to participate in the linkage of parallel chains, as suggested by the O···O distances involved [O3wx···O2(1 − x, 1 − y, 1 − z) 2.86(2), O3wx···O3wx(1 − x, 1 − y, 1 − z) 2.46(3), O3wy···O2(−x, 1 − y, 1 − z) 2.86(1), O3wy···O3 2.90(1) Å]. An accurate description of the interactions, however, is precluded by the disordered character of the molecule. The final array showing the proposed hydrogen-bonding contacts (phen omited for clarity) can be seen in Fig. 2.

Experimental

The compound was prepared by mixing an alcoholic solution of phenanthroline with an aqueous solution of zinc acetate

and sodium thiosulfate. After several hours, well developed prismatic crystals were obtained.

Crystal data

$[Zn(C_{12}H_8N_2)_2(H_2O)_2][Zn-(S_2O_3)_2(C_{12}H_8N_2)].H_2O$
$M_r = 949.64$
Triclinic
$P\bar{1}$
$a = 10.545\,(2)$ Å
$b = 12.924\,(2)$ Å
$c = 14.856\,(2)$ Å
$\alpha = 90.60\,(1)°$
$\beta = 100.06\,(1)°$
$\gamma = 99.47\,(1)°$
$V = 1964.7\,(6)$ Å^3
$Z = 2$
$D_x = 1.605$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–12.5°$
$\mu = 1.495$ mm^{-1}
$T = 293\,(2)$ K
Prism
$0.32 \times 0.20 \times 0.18$ mm
Colourless

Data collection

Siemens $R3m$ diffractometer
$\omega/2\theta$ scans
Absorption correction:
  ψ scan (*XEMP* in *SHELXTL/PC*; Sheldrick, 1990)
$T_{min} = 0.762$, $T_{max} = 0.945$
3787 measured reflections
3663 independent reflections

2868 observed reflections
  $[I > 2\sigma(I)]$
$R_{int} = 0.0377$
$\theta_{max} = 20.0°$
$h = 0 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$
2 standard reflections
  monitored every 98
  reflections
  intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0458$
$wR(F^2) = 0.1333$
$S = 0.986$
3662 reflections
516 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 8.9981P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.072$
$\Delta\rho_{max} = 0.569$ e Å^{-3}
$\Delta\rho_{min} = -0.535$ e Å^{-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 isotropic or equivalent isotropic displacement parameters* $(Å^2)$

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}/U_{iso}
Zn1	0.23586(10)	0.83724(7)	0.31813(6)	0.0461(3)
Zn2	0.68777(9)	1.26371(7)	0.16577(6)	0.0429(3)
S1	0.3179(2)	0.6973(2)	0.3771(2)	0.0554(7)
S2	0.2544(2)	0.6843(2)	0.49982(15)	0.0483(6)
O1	0.3081(6)	0.7824(5)	0.5523(4)	0.066(2)
O2	0.3073(7)	0.5956(5)	0.5411(5)	0.079(2)
O3	0.1140(6)	0.6665(5)	0.4832(4)	0.073(2)
S3†	0.2256(10)	0.8475(11)	0.1643(5)	0.0413(7)
S4†	0.4047(9)	0.9285(6)	0.1556(7)	0.0413(7)
O4†	0.4695(13)	0.8600(12)	0.1081(11)	0.051(2)
O5†	0.4734(11)	0.9552(15)	0.2484(9)	0.051(2)
O6†	0.3844(15)	1.0214(9)	0.1049(16)	0.051(2)
S3x†	0.2527(11)	0.8300(10)	0.1649(5)	0.0413(7)
S4x†	0.4319(10)	0.9205(7)	0.1776(8)	0.0413(7)
O4x†	0.4993(15)	0.8784(14)	0.1122(13)	0.051(2)
O5x†	0.4979(13)	0.913(2)	0.2709(9)	0.051(2)
O6x†	0.4116(17)	1.0267(9)	0.158(2)	0.051(2)
S3y†	0.2268(12)	0.8626(18)	0.1615(6)	0.0413(7)
S4y†	0.4212(10)	0.9158(8)	0.1730(6)	0.0413(7)
O4y†	0.4569(17)	0.8931(16)	0.0858(10)	0.051(2)
O5y†	0.4873(15)	0.8602(18)	0.2462(13)	0.051(2)
O6y†	0.4423(18)	1.0278(8)	0.1942(15)	0.051(2)
S3z†	0.2363(15)	0.8758(19)	0.1660(7)	0.0413(7)
S4z†	0.4284(16)	0.9207(11)	0.1608(9)	0.0413(7)
O4z†	0.4899(19)	0.974(2)	0.2476(12)	0.051(2)
O5z†	0.436(2)	0.990(2)	0.0851(15)	0.051(2)
O6z†	0.481(2)	0.8262(16)	0.146(2)	0.051(2)
N1	0.7429(7)	1.3665(6)	0.0560(5)	0.055(2)
N2	0.6076(7)	1.4022(5)	0.1888(5)	0.048(2)
N1a	0.8783(7)	1.3254(6)	0.2451(5)	0.048(2)
N2a	0.8028(7)	1.1458(5)	0.1415(5)	0.048(2)
N1b	0.0424(7)	0.8484(6)	0.3207(5)	0.047(2)
N2b	0.2632(7)	0.9814(5)	0.3852(4)	0.041(2)
C1	0.8096(11)	1.3491(8)	-0.0087(7)	0.076(3)
C2	0.8385(14)	1.4236(11)	-0.0729(7)	0.103(4)
C3	0.7982(15)	1.5161(11)	-0.0672(9)	0.107(5)
C4	0.7291(11)	1.5401(9)	-0.0011(8)	0.072(3)
C5	0.6827(14)	1.6367(9)	0.0072(9)	0.093(4)
C6	0.6163(12)	1.6530(9)	0.0734(10)	0.091(4)
C7	0.5872(10)	1.5767(7)	0.1369(8)	0.065(3)
C8	0.5192(10)	1.5892(9)	0.2077(10)	0.081(3)
C9	0.4984(9)	1.5138(9)	0.2657(8)	0.073(3)
C10	0.5439(9)	1.4203(7)	0.2549(7)	0.060(3)
C11	0.6311(8)	1.4800(7)	0.1308(7)	0.052(2)
C12	0.7026(9)	1.4603(7)	0.0602(6)	0.053(2)
C1a	0.9156(10)	1.4129(8)	0.2967(6)	0.063(3)
C2a	1.0457(12)	1.4517(9)	0.3334(7)	0.080(3)
C3a	1.1407(11)	1.3990(10)	0.3160(7)	0.077(3)
C4a	1.1062(9)	1.3061(8)	0.2620(6)	0.059(3)
C5a	1.1983(10)	1.2437(11)	0.2409(8)	0.076(3)
C6a	1.1578(11)	1.1512(10)	0.1922(8)	0.076(3)
C7a	1.0247(10)	1.1132(9)	0.1585(6)	0.058(3)
C8a	0.9754(13)	1.0156(9)	0.1102(7)	0.075(3)
C9a	0.8453(13)	0.9863(8)	0.0807(7)	0.076(3)
C10a	0.7612(10)	1.0536(8)	0.0965(6)	0.059(3)
C11a	0.9324(9)	1.1739(7)	0.1740(6)	0.045(2)
C12a	0.9718(9)	1.2714(7)	0.2276(6)	0.048(2)
C1b	-0.0657(12)	0.7805(8)	0.2887(7)	0.072(3)
C2b	-0.1904(11)	0.8058(12)	0.2870(8)	0.094(4)
C3b	-0.2014(11)	0.9002(13)	0.3204(8)	0.085(4)
C4b	-0.0925(10)	0.9729(9)	0.3556(6)	0.059(3)
C5b	-0.0917(13)	1.0747(11)	0.3965(7)	0.075(3)
C6b	0.0169(14)	1.1384(9)	0.4338(7)	0.072(3)
C7b	0.1418(10)	1.1107(8)	0.4317(6)	0.054(2)
C8b	0.2595(14)	1.1744(8)	0.4676(6)	0.074(3)
C9b	0.3744(12)	1.1416(9)	0.4620(7)	0.078(3)
C10b	0.3721(10)	1.0451(8)	0.4194(6)	0.059(3)
C11b	0.1492(9)	1.0135(7)	0.3914(5)	0.040(2)
C12b	0.0308(8)	0.9433(7)	0.3547(5)	0.043(2)
O1w	0.5254(7)	1.1887(4)	0.0715(4)	0.056(2)
O2w	0.6022(7)	1.1865(5)	0.2677(4)	0.065(2)
O3w·x‡	0.4251(14)	0.4257(12)	0.4568(11)	0.035(6)
O3w·y‡	-0.1657(8)	0.6037(6)	0.4235(6)	0.056(3)

† Disordered thiosulfate atom sites; U_{iso} given. ‡ Disordered (see below).

Table 2. *Selected geometric parameters* $(Å, °)$

Zn1—N2b	2.057(7)	Zn2—N1a	2.165(7)
Zn1—N1b	2.076(7)	Zn2—N1	2.210(7)
Zn1—S1	2.252(2)	S1—S2	2.048(3)
Zn1—S3	2.275(8)	S2—O3	1.437(6)
Zn1—S3x	2.316(7)	S2—O2	1.451(6)
Zn1—S3z	2.320(10)	S2—O1	1.458(6)
Zn1—S3y	2.340(9)	S3—S4	2.03(2)
Zn2—O2w	2.077(6)	S4—O5	1.45(2)
Zn2—O1w	2.102(6)	S4—O6	1.45(2)
Zn2—N2	2.150(7)	S4—O4	1.45(2)
Zn2—N2a	2.162(7)		
N2b—Zn1—N1b	80.7(3)	O2w—Zn2—N2a	96.4(3)
N2b—Zn1—S1	124.4(2)	O1w—Zn2—N2a	92.1(3)
N1b—Zn1—S1	119.7(2)	N2—Zn2—N2a	168.9(3)
S1—Zn1—S3	113.8(4)	O2w—Zn2—N1a	99.2(3)
S1—Zn1—S3x	104.8(4)	O1w—Zn2—N1a	167.9(3)

N2b—Zn1—S3z	104.5 (6)	N2—Zn2—N1a	93.9 (3)
N1b—Zn1—S3z	97.9 (5)	N2a—Zn2—N1a	77.2 (3)
S1—Zn1—S3z	120.5 (6)	O2w—Zn2—N1	167.1 (3)
S1—Zn1—S3Y	118.0 (6)	O1w—Zn2—N1	88.5 (2)
O2w—Zn2—O1w	87.4 (3)	N2—Zn2—N1	76.7 (3)
O2w—Zn2—N2	91.6 (3)	N2a—Zn2—N1	95.9 (3)
O1w—Zn2—N2	96.0 (2)	N1a—Zn2—N1	87.1 (3)

The structure was solved through a combination of direct methods and difference Fourier synthesis. Refinement was performed on F^2 using the whole data set. H atoms from coordinated water molecules were detected in a difference Fourier synthesis and subsequently refined with a rigid-body model. Those of the solvent water molecule could not be found. H atoms in the phen groups were included at their expected positions and allowed to ride on their host atoms. Fixed isotropic displacement parameters were used for all H atoms. One of the thiosulfate groups was found to be severely disordered and was refined as four different images of an idealized rigid group with occupancies constrained to sum to 1. The solvent water molecule was also found to be split in two positions. Lowering of the space-group symmetry did not result in a simpler model.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to solve structure: *XS* in *SHELXTL/PC*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* in *SHELXL93* and *PARST* (Nardelli, 1983).

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The Five-Coordinate Cadmium Halide Bis(diethyldithiocarbamate) Complexes [PPh4][Cd(S2CNEt2)2Cl] and [PPh4][Cd(S2CNEt2)2Br]

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Abstract

The title compounds, tetraphenylphosphonium chloro-bis(diethyldithiocarbamato-S,S)cadmium(II), ($C_{24}H_{20}P$)-[CdCl($C_5H_{10}NS_2$)$_2$], (1), and tetraphenylphosphonium bromobis(diethyldithiocarbamato-S,S)cadmium(II), ($C_{24}H_{20}P$)[CdBr($C_5H_{10}NS_2$)$_2$], (2), are isomorphous. The compexes are mononuclear with coordination spheres of the types S_4Cl and S_4Br, respectively. The central Cd atom is linked to four S atoms and two bidentate dithiocarbamate ligands and to the halide atom. The resulting Cd-atom coordination geometry is halfway between trigonal bipyrimidal and square pyramidal. Principal dimensions include: Cd—S 2.573 (1)–2.682 (1) in (1) and 2.571 (3)–2.736 (2) Å in (2); Cd—Cl and Cd—Br 2.462 (1) and 2.626 (1) Å, respectively.

Comment

The characterization of mononuclear five-coordinate cadmium(II) compounds with sulfur ligation is of current interest with respect to structural and spectroscopic models for cadmium binding sites in biological molecules, particularly in a number of metallothioneins and metallo-regulatory proteins (Reddy, Zhang, Schlemper & Schrauzer, 1992, and references therein).

The reactions of the sulfur-bridged cadmium bis-(diethyldithiocarbamate) dimer with potentially monodentate anionic ligands to give the 1:1 adducts [Cd(S2CNEt2)2X]$^-$ (X = Cl, Br, I or NCS) have been reported recently (Baggio, Frigerio, Halac, Vega & Perec, 1992). The complexes were isolated as their [NEt4]$^+$ or [N^{n}Bu4]$^+$ salts and characterized by elemental analysis and by spectroscopic techniques. In the