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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(C-C)$ = 0.004 Å
R factor = 0.041
wR factor = 0.115
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

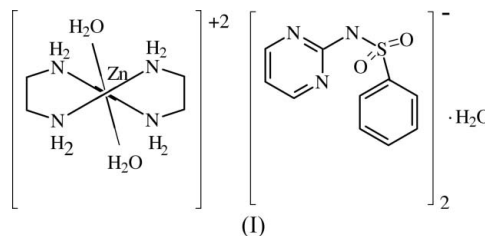
trans-Diaquabis(ethylenediamine)zinc(II) 2-(sulfanilamido)pyrimidine monohydrate

In the title compound, $[Zn(C_4H_8N_4)(H_2O)_2](C_{10}H_9N_4O_2S)_2 \cdot H_2O$, the Zn^{II} ion is octahedrally surrounded by two water O atoms [Zn—O = 2.68 (2) and 2.49 (2) Å] and the four N atoms of two ethylenediamine molecules. Intermolecular O—H...N, N—H...O and N—H...N hydrogen-bond interactions result in the formation of an intricate three-dimensional network.

Comment

Many sulfanilamide derivatives, such as 2-sulfanilamidopyrimidine (sulfadiazine), possess antibacterial activity. Thus, sulfadiazine compounds are widely used for their bacterial action (Nogrady, 1988; Silverman, 1992) and zinc salts of sulfadiazine are used to prevent bacterial infection in both humans and animals during the treatment of burns (Baezinger & Strauss, 1976; Cook & Turner, 1975). The pharmacological activity of these types of molecules is often enhanced by complexation with metal ions (Bult & Sigel, 1983; Casanova *et al.*, 1993). These metal complexes are largely insoluble, working through the slow release of the metal ions from these materials. Because the slow release of the metal ions from these drugs is strongly dependent on their binding nature, it is important to understand the coordination environment around the metal. Although interesting structural results have been described in the literature (Baenziger *et al.*, 1983; Built *et al.*, 1981; Brown *et al.*, 1987; Garcia Raso *et al.*, 1997), mixed-ligand complexes of 2-sulfanilamidopyrimidine have not been prepared and investigated sufficiently to date. Therefore, in the present paper, we report the synthesis and crystal structure of the title Zn^{II}-2-sulfanilamidopyrimidine (H-SD)-ethylenediamine (en) complex, (I).

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The title complex contains octahedral elongated *trans*-diaqua(ethylenediamine)zinc(II) cations counterbalanced with sulfadiazine anions (Fig. 1). Ethylenediamine acts, as usual, as a chelating ligand, forming five-membered rings. The Zn—N bond distances are consistent with those in a related structure (Fu *et al.*, 2005). The five-membered rings are in envelope conformations, with atoms C41 and C44 forming the flaps, as shown in Fig. 1. Sulfadiazine molecules can exist in different conformations as a result of rotation about the C—S,

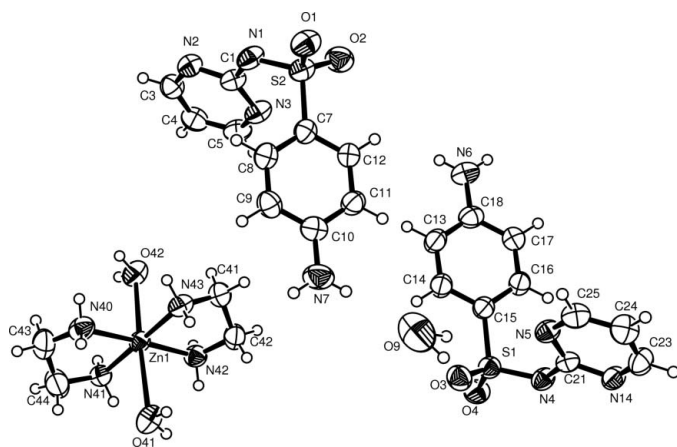


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

S—N and N—C bonds (Kokila *et al.*, 1995). The three dihedral angles describing these conformations of the sulfadiazine ligands in (I) are C15—S1—N4—C21/C7—S2—N1—C1 = 67.9 (2)/−58.9 (2)°, S1—N4—C21—N5/S2—N1—C1—N3 = −4.8 (3)/0.0 (4)° and C16—C15—S1—N4/C8—C7—S2—N1 = 25.8 (3)/−34.8 (3). Thus, both molecules have roughly the same *gauche* conformation when viewed along the S—N bond.

The H atoms of the coordinated and uncoordinated water molecules are engaged in hydrogen bonding with the sulfadiazine anions, resulting in the formation of extended chains. Moreover, there are also N—H···O hydrogen bonds, resulting in an intricate three-dimensional network (Table 1, Fig. 2).

Experimental

All reagents were purchased from commercial sources and used as supplied. An ethanol solution (20 ml) of 2-sulfanilamidopyrimidine (H-SD; 2 mmol) was added dropwise to a stirred solution of Zn^{II} acetate (1 mmol) in distilled water (30 ml). A brown precipitate began to form and the reaction mixture was stirred overnight at 313 K. The products were isolated by filtration, washed with cold distilled water and diethyl ether, and dried in air. The 2-sulfanilamidopyrimidine–ethylenediamine (en) complex was prepared as follows. A solution of en (2 mmol) in ethanol (30 ml) was added dropwise with stirring to a solution of [Zn(SD)₂] (1 mmol) dissolved in ethanol (50 ml) and stirred for 4 h at 323 K in a temperature-controlled bath. A dark-violet polycrystalline precipitate was filtered off and crystals of (I) suitable for X-ray diffraction were co-crystallized as violet crystals by slow diffusion of Et₂O into the filtrate. Analysis, calculated: C 39.14, H 5.43, N 22.83%; found: C 39.10, H 5.45, N 22.87%.

Crystal data

[Zn(C ₄ H ₈ N ₄)](C ₁₀ H ₉ N ₄ O ₂ S) ₂ ·3H ₂ O	$D_x = 1.532 \text{ Mg m}^{-3}$
$M_r = 738.17$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 28720 reflections
$a = 13.7400$ (5) Å	$\theta = 1.3\text{--}26.7^\circ$
$b = 14.6581$ (7) Å	$\mu = 0.96 \text{ mm}^{-1}$
$c = 15.9408$ (6) Å	$T = 293$ (2) K
$\beta = 94.510$ (3)°	Violet, blue
$V = 3200.6$ (2) Å ³	$0.38 \times 0.33 \times 0.22 \text{ mm}$
$Z = 4$	

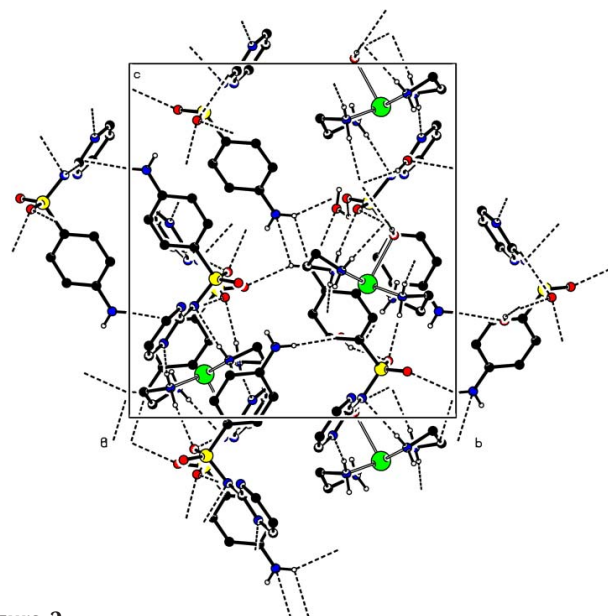


Figure 2
A packing view, showing the intricate hydrogen-bonding (dashed lines) three-dimensional network. H atoms not involved in the hydrogen bonding have been omitted for clarity.

Data collection

Stoe IPDS 2 diffractometer	4864 reflections with $I > 2\sigma(I)$
φ scans	$R_{\text{int}} = 0.062$
Absorption correction: integration	$\theta_{\text{max}} = 26.6^\circ$
<i>X-RED32</i> (Stoe & Cie, 2002)	$h = -17 \rightarrow 17$
$T_{\text{min}} = 0.749$, $T_{\text{max}} = 0.829$	$k = -18 \rightarrow 18$
32597 measured reflections	$l = -19 \rightarrow 19$
6655 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2]$
$wR(F^2) = 0.115$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{\text{max}} = 0.009$
6655 reflections	$\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{Å}^{-3}$
433 parameters	$\Delta\rho_{\text{min}} = -0.57 \text{ e } \text{Å}^{-3}$

Table 1
Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
N6—H6B···O41 ⁱ	0.86	2.31	3.165 (4)	173
N7—H7A···O9	0.86	2.28	3.114 (5)	164
N7—H7B···O2 ⁱⁱ	0.86	2.43	3.124 (4)	138
N7—H7B···N7 ⁱⁱⁱ	0.86	2.59	3.046 (5)	114
N40—H40B···N1 ^{iv}	0.90	2.37	3.204 (3)	155
N41—H41C···O4 ^v	0.90	2.24	3.128 (3)	167
N41—H41D···N14 ^{vi}	0.90	2.14	3.036 (3)	172
N42—H42C···N4 ^{vi}	0.90	2.24	3.115 (3)	165
N43—H43C···N2 ^{iv}	0.90	2.07	2.968 (3)	172
N43—H43D···O1 ⁱⁱ	0.90	2.19	3.080 (3)	172
O9—H4O···O3	0.85 (6)	2.35 (6)	2.839 (4)	116 (5)
O41—H41E···O1 ⁱⁱ	0.91 (2)	2.18 (2)	3.046 (3)	158 (4)
O41—H41F···N4 ^{vi}	0.87 (1)	2.08 (2)	2.920 (3)	163 (3)
O42—H42E···O4 ^v	0.85 (3)	2.06 (2)	2.862 (3)	158 (3)
O42—H42F···N1 ^{iv}	0.86 (1)	2.03 (1)	2.884 (3)	174 (3)
O42—H42F···O1 ^{iv}	0.86 (1)	2.59 (3)	3.142 (3)	123 (2)

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

Water H atoms were located in a difference Fourier map and refined using distance restraints of O–H = 0.85 Å and H···H = 1.39 Å, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms (C or N), with C–H and N–H distances in the range 0.85–0.93 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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