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The 1:1 adduct of 2,3,5,6-tetramethylpyrazine 1,4-dioxide and diaquabis(thiocyanato-*κN*)-zinc(II) (ATD)

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Key indicators

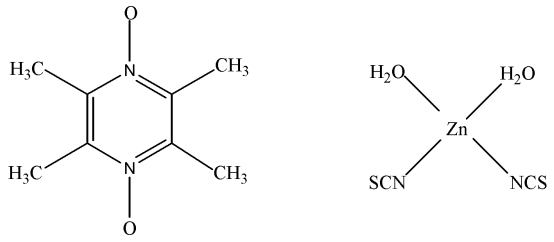
Single-crystal X-ray study
T = 298 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.096
Data-to-parameter ratio = 16.8

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

In the crystal structure of the title adduct, $C_8H_{12}N_2O_2\cdot[Zn(NCS)_2(H_2O)_2]$, the organic and inorganic molecules are connected by O—H···O hydrogen bonds, with O···O distances ranging from 2.640 (2) to 2.677 (3) Å. There are two independent 2,3,5,6-tetramethylpyrazine 1,4-dioxide molecules, both of which lie on crystallographic inversion centers; the asymmetric unit contains two organic half-molecules and one inorganic molecule.

Comment

Many multinuclear and polymeric complexes have been synthesized using thiocyanate as a bridging ligand, and some exhibit special physical properties (Shen & Xu, 2001; Shi *et al.*, 2005). In our recent experiments, we have shown that 2,3,5,6-tetramethylpyrazine 1,4-dioxide exhibits bridging properties. Our intention was to design a polymeric complex using zinc(II) ions and the bridging ligands thiocyanate and 2,3,5,6-tetramethylpyrazine 1,4-dioxide, but in our attempt only the title adduct, (I), was obtained. The crystal structure is described here.



(I)

In the title structure, the asymmetric unit contains one Zn^{II} complex and two half-molecules of 2,3,5,6-tetramethylpyrazine 1,4-dioxide, the complete molecules being generated by inversion symmetry. In the metal complex, the Zn atom is coordinated by two N atoms from two isothiocyanate anions and two O atoms from two water molecules. The Zn atom is in a distorted tetrahedral coordination environment (see Table 1). The non-H atoms of 2,3,5,6-tetrapyrazine 1,4-dioxide are essentially coplanar. Fig. 1 shows the Zn complex and two complete molecules of 2,3,5,6-tetramethylpyrazine 1,4-dioxide. Fig. 2 displays the unit cell and the arrangements of the two components. The 2,3,5,6-tetramethylpyrazine 1,4-dioxide molecules are approximately perpendicular to the *b* axis, but there are no significant *π*–*π* stacking interactions. In the crystal structure, the Zn^{II} complex molecules and the organic molecules are connected through O—H···O hydrogen bonds to form sheets in the [010] plane (see Table 2 and Fig. 2).

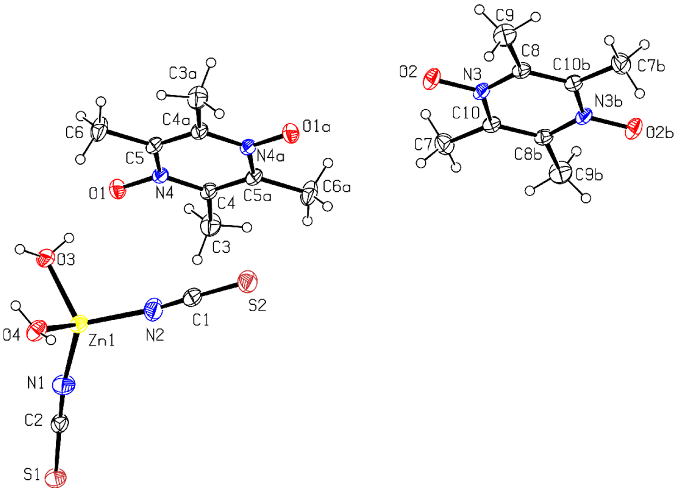


Figure 1
View of the adduct, with the atom-numbering scheme and 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii. Although the asymmetric unit contains two half molecules of 2,3,5,6-tetramethylpyrazine 1,4-dioxide both complete molecules are shown, and atoms labeled with suffixes '*a*' and '*b*' are related by the symmetry operators $(1 - x, 1 - y, -1 - z)$ and $(2 - x, 2 - y, 1 - z)$, respectively.

Experimental

To a solution (15 ml) containing $Zn(ClO_4)\cdot 6H_2O$ (0.2540 g, 0.68 mmol), NaSCN (0.1120 g, 1.38 mmol) and 2,3,5,6-tetramethylpyrazine 1,4-dioxide (0.1156 g, 0.69 mmol) were added and the solution was stirred for a few minutes. Colorless single crystals were obtained after the solution was allowed to stand at room temperature for two weeks.

Crystal data

$C_8H_{12}N_2O_2\cdot[Zn(NCS)_2(H_2O)_2]$
$M_r = 385.80$
Monoclinic, $P2_1/c$
$a = 15.563\ (4)$ Å
$b = 7.2595\ (17)$ Å
$c = 15.425\ (4)$ Å
$\beta = 113.412\ (3)^\circ$
$V = 1599.2\ (7)$ Å^3
$Z = 4$

$D_x = 1.602$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4535 reflections
$\theta = 2.4$–26.9°
$\mu = 1.82$ mm^{-1}
$T = 298\ (2)$ K
Prism, colorless
0.46 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.527$, $T_{\max} = 0.697$
9037 measured reflections

3454 independent reflections
2918 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.0^\circ$
$h = -12 \rightarrow 19$
$k = -9 \rightarrow 8$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.096$
$S = 1.05$
3454 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.12P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0302 (15)

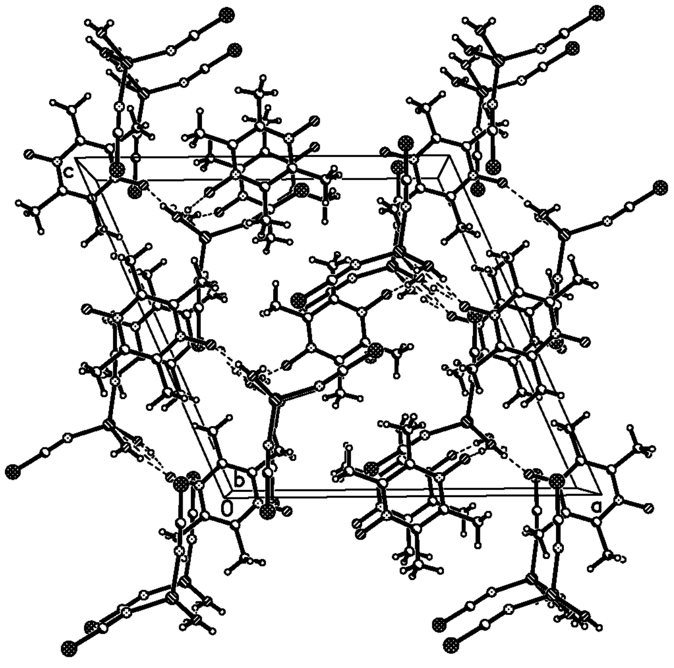


Figure 2
Packing diagram, with O—H···O hydrogen bonds are shown as dashed lines.

Table 1
Selected geometric parameters (Å, °).

Zn1—N1	1.924 (2)	Zn1—O3	1.9806 (16)
Zn1—N2	1.930 (2)	Zn1—O4	1.9820 (17)
N1—Zn1—N2	116.23 (10)	N1—Zn1—O4	108.49 (9)
N1—Zn1—O3	111.67 (9)	N2—Zn1—O4	112.67 (9)
N2—Zn1—O3	108.14 (8)	O3—Zn1—O4	98.20 (8)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O1	0.82	1.82	2.640 (2)	176
O3—H2···O2$^{\rm i}$	0.87 (3)	1.84 (3)	2.677 (3)	163 (3)
O4—H3···O1$^{\rm ii}$	0.77 (3)	1.90 (3)	2.656 (2)	165 (3)
O4—H4···O2$^{\rm i}$	0.82	1.88	2.676 (3)	162

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

H atoms bonded to C atoms were included in calculated positions, with C—H distances of 0.96 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(C). Of the four H atoms (H1, H2, H3 and H4) bonded to the coordinated water molecules (O1 and O2), H1 and H4 were included in calculated positions, with O—H = 0.82 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(O), while H2 and H3 were refined independently with isotropic displacement parameters.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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