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Bis(acetone 4-phenylthiosemicarbazonato-κ^2N^1,S)zinc(II)

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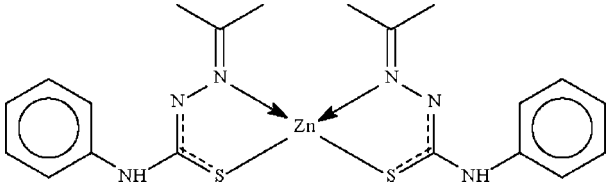
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.005 Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 18.4.

The Zn^{II} atom in the title compound, $[Zn(C_{10}H_{12}N_3S)_2]$, is N,S-chelated by the deprotonated Schiff base in a tetrahedral environment. The metal atom lies on a twofold rotation axis that relates one anion to the other. The amino H atom forms an intermolecular N—H$\cdots\pi$ interaction to an aromatic ring.

Related literature

For the two modifications of acetone 4-phenylthiosemicarbazone, see: Jian *et al.* (2005); Venkatraman *et al.* (2005).



Experimental

Crystal data

$[Zn(C_{10}H_{12}N_3S)_2]$
M_r = 477.94
Monoclinic, $C2/c$
a = 23.5203 (5) Å
b = 7.2938 (2) Å
c = 15.1134 (5) Å
β = 122.761 (2)°
V = 2180.3 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.34 mm^{-1}
T = 153 K
0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.690, T_{max} = 0.936
6983 measured reflections
2488 independent reflections
1896 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.10
2488 reflections
135 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$\cdots Cg^{i}$	0.86	2.86	3.671 (3)	157

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$. *Cg* is the centroid of the aromatic ring.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5011).

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supporting information

Acta Cryst. (2009). E**65**, m969 [doi:10.1107/S1600536809028244]

Bis(acetone 4-phenylthiosemicarbazonato-κ^2N^1,S)zinc(II)

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S1. Experimental

Zinc acetate monohydrate (0.22 g, 1 mmol) and 2,4-dihydroxybenzaldehyde 4-phenylthiosemicarbazone (0.29 g, 1 mmol) were heated in ethanol (50 ml) to form [1-(4-hydroxy-2-oxidobenzylidene)-4-phenylthiosemicarbazonato]zinc. The product was recrystallized from acetone, cleaved part of the anion.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95–0.98 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C). The amino H-atom was similarly treated (N–H 0.88 Å).

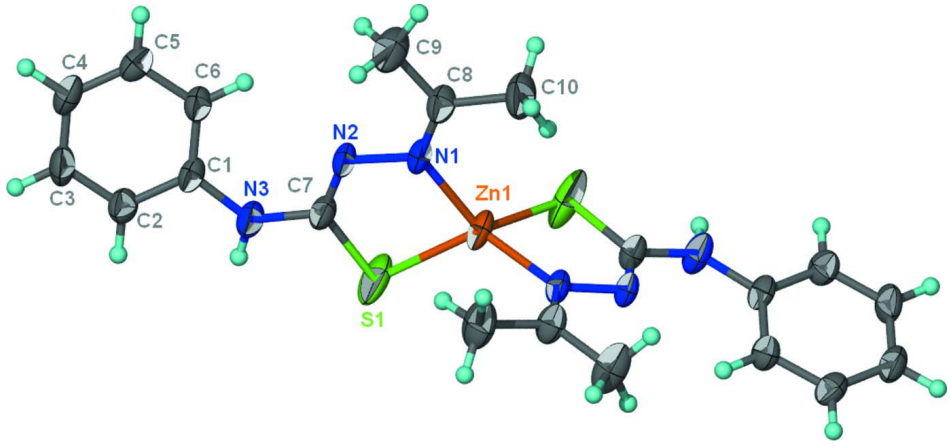

Figure 1

Anisotropic displacement ellipsoid (Barbour, 2001) plot of $Zn(C_{10}H_{12}N_3S)_2$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radii.

Bis(acetone 4-phenylthiosemicarbazonato-κ^2N^1,S)zinc(II)

Crystal data

$[Zn(C_{10}H_{12}N_3S)_2]$
M_r = 477.94
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 23.5203 (5) Å
b = 7.2938 (2) Å
c = 15.1134 (5) Å
β = 122.761 (2)°
V = 2180.3 (1) Å^3
Z = 4
F(000) = 992
D_x = 1.456 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2390 reflections
θ = 3.8–28.0°
μ = 1.34 mm^{-1}
T = 153 K
Wedge, brown
0.30 × 0.20 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.690, T_{max} = 0.936
6983 measured reflections
2488 independent reflections
1896 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 2.1°
h = −30→30
k = −9→9
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.10
2488 reflections
135 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 4.48P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.5000	0.30141 (7)	0.2500	0.03064 (17)
S1	0.55393 (4)	0.14771 (13)	0.18550 (8)	0.0501 (3)
N1	0.58756 (10)	0.4440 (3)	0.34189 (19)	0.0245 (5)
N2	0.64335 (10)	0.3888 (3)	0.33788 (19)	0.0253 (5)
N3	0.68276 (11)	0.1943 (4)	0.2634 (2)	0.0336 (6)
H3	0.6712	0.1130	0.2154	0.040*
C1	0.75216 (12)	0.2404 (4)	0.3216 (2)	0.0255 (6)
C2	0.78857 (13)	0.1872 (4)	0.2775 (2)	0.0296 (6)
H2	0.7662	0.1268	0.2113	0.035*
C3	0.85719 (14)	0.2225 (4)	0.3300 (3)	0.0348 (7)
H3A	0.8816	0.1866	0.2993	0.042*
C4	0.89046 (13)	0.3090 (4)	0.4263 (3)	0.0332 (7)
H4	0.9376	0.3330	0.4620	0.040*
C5	0.85427 (13)	0.3606 (4)	0.4703 (2)	0.0289 (6)
H5	0.8770	0.4194	0.5370	0.035*
C6	0.78518 (13)	0.3275 (4)	0.4181 (2)	0.0283 (6)
H6	0.7608	0.3646	0.4486	0.034*
C7	0.63093 (13)	0.2588 (4)	0.2715 (2)	0.0275 (6)
C8	0.59964 (13)	0.5716 (4)	0.4081 (2)	0.0302 (7)
C9	0.66790 (15)	0.6551 (5)	0.4773 (3)	0.0456 (9)
H9A	0.7007	0.5590	0.5196	0.068*
H9B	0.6663	0.7453	0.5241	0.068*
H9C	0.6815	0.7159	0.4338	0.068*
C10	0.54313 (16)	0.6384 (5)	0.4182 (3)	0.0451 (9)
H10A	0.5039	0.5575	0.3784	0.068*

H10B	0.5307	0.7633	0.3903	0.068*
H10C	0.5578	0.6380	0.4925	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0144 (2)	0.0296 (3)	0.0436 (3)	0.000	0.0129 (2)	0.000
S1	0.0171 (3)	0.0539 (6)	0.0665 (7)	−0.0064 (3)	0.0143 (4)	−0.0345 (5)
N1	0.0156 (9)	0.0257 (12)	0.0320 (14)	0.0020 (9)	0.0128 (10)	0.0034 (11)
N2	0.0148 (10)	0.0287 (12)	0.0321 (14)	0.0015 (9)	0.0124 (10)	−0.0009 (11)
N3	0.0196 (11)	0.0373 (14)	0.0408 (16)	−0.0017 (10)	0.0143 (11)	−0.0158 (13)
C1	0.0168 (11)	0.0235 (14)	0.0328 (16)	0.0030 (10)	0.0113 (12)	0.0006 (12)
C2	0.0245 (13)	0.0303 (15)	0.0332 (17)	0.0041 (12)	0.0152 (13)	−0.0016 (14)
C3	0.0241 (14)	0.0429 (18)	0.0432 (19)	0.0065 (13)	0.0221 (14)	0.0041 (16)
C4	0.0189 (12)	0.0373 (17)	0.0398 (18)	0.0023 (12)	0.0136 (13)	0.0086 (15)
C5	0.0216 (13)	0.0264 (14)	0.0310 (17)	0.0007 (11)	0.0092 (12)	0.0040 (13)
C6	0.0219 (13)	0.0307 (16)	0.0328 (17)	0.0034 (11)	0.0152 (13)	0.0004 (13)
C7	0.0171 (12)	0.0299 (15)	0.0328 (17)	−0.0001 (11)	0.0116 (12)	−0.0030 (13)
C8	0.0228 (13)	0.0258 (15)	0.0408 (18)	0.0015 (11)	0.0165 (13)	−0.0037 (14)
C9	0.0297 (15)	0.0361 (19)	0.063 (2)	−0.0023 (13)	0.0197 (17)	−0.0213 (17)
C10	0.0321 (16)	0.046 (2)	0.062 (2)	0.0046 (14)	0.0284 (17)	−0.0102 (18)

Geometric parameters (Å, º)

Zn1—N1	2.039 (2)	C3—C4	1.376 (5)
Zn1—N1^{i}	2.039 (2)	C3—H3A	0.9500
Zn1—S1	2.2702 (8)	C4—C5	1.386 (4)
Zn1—S1^{i}	2.2702 (8)	C4—H4	0.9500
S1—C7	1.754 (3)	C5—C6	1.390 (4)
N1—C8	1.281 (4)	C5—H5	0.9500
N1—N2	1.405 (3)	C6—H6	0.9500
N2—C7	1.294 (4)	C8—C9	1.492 (4)
N3—C7	1.372 (3)	C8—C10	1.499 (4)
N3—C1	1.413 (3)	C9—H9A	0.9800
N3—H3	0.8600	C9—H9B	0.9800
C1—C6	1.381 (4)	C9—H9C	0.9800
C1—C2	1.395 (4)	C10—H10A	0.9800
C2—C3	1.384 (4)	C10—H10B	0.9800
C2—H2	0.9500	C10—H10C	0.9800
N1—Zn1—N1^{i}	118.66 (13)	C5—C4—H4	120.5
N1—Zn1—S1	87.20 (6)	C4—C5—C6	120.9 (3)
N1^{i}—Zn1—S1	123.54 (7)	C4—C5—H5	119.6
N1—Zn1—S1^{i}	123.54 (7)	C6—C5—H5	119.6
N1^{i}—Zn1—S1^{i}	87.20 (6)	C1—C6—C5	119.7 (3)
S1—Zn1—S1^{i}	120.82 (6)	C1—C6—H6	120.1
C7—S1—Zn1	92.67 (9)	C5—C6—H6	120.1
C8—N1—N2	115.1 (2)	N2—C7—N3	118.8 (2)

C8—N1—Zn1	128.17 (17)	N2—C7—S1	128.47 (19)
N2—N1—Zn1	116.57 (17)	N3—C7—S1	112.8 (2)
C7—N2—N1	114.8 (2)	N1—C8—C9	123.0 (2)
C7—N3—C1	130.2 (3)	N1—C8—C10	118.8 (3)
C7—N3—H3	114.9	C9—C8—C10	118.2 (3)
C1—N3—H3	114.9	C8—C9—H9A	109.5
C6—C1—C2	119.5 (2)	C8—C9—H9B	109.5
C6—C1—N3	124.5 (2)	H9A—C9—H9B	109.5
C2—C1—N3	116.0 (3)	C8—C9—H9C	109.5
C3—C2—C1	120.1 (3)	H9A—C9—H9C	109.5
C3—C2—H2	120.0	H9B—C9—H9C	109.5
C1—C2—H2	120.0	C8—C10—H10A	109.5
C4—C3—C2	120.8 (3)	C8—C10—H10B	109.5
C4—C3—H3A	119.6	H10A—C10—H10B	109.5
C2—C3—H3A	119.6	C8—C10—H10C	109.5
C3—C4—C5	119.1 (2)	H10A—C10—H10C	109.5
C3—C4—H4	120.5	H10B—C10—H10C	109.5

N1—Zn1—S1—C7	4.02 (12)	C2—C3—C4—C5	0.0 (5)
N1^{i}—Zn1—S1—C7	126.96 (13)	C3—C4—C5—C6	−0.6 (4)
S1^{i}—Zn1—S1—C7	−123.93 (11)	C2—C1—C6—C5	−0.3 (4)
N1^{i}—Zn1—N1—C8	52.6 (2)	N3—C1—C6—C5	177.8 (3)
S1—Zn1—N1—C8	179.7 (3)	C4—C5—C6—C1	0.7 (4)
S1^{i}—Zn1—N1—C8	−54.6 (3)	N1—N2—C7—N3	−179.0 (2)
N1^{i}—Zn1—N1—N2	−132.0 (2)	N1—N2—C7—S1	1.3 (4)
S1—Zn1—N1—N2	−4.85 (18)	C1—N3—C7—N2	3.8 (5)
S1^{i}—Zn1—N1—N2	120.81 (17)	C1—N3—C7—S1	−176.5 (3)
C8—N1—N2—C7	179.4 (3)	Zn1—S1—C7—N2	−4.3 (3)
Zn1—N1—N2—C7	3.4 (3)	Zn1—S1—C7—N3	176.0 (2)
C7—N3—C1—C6	18.0 (5)	N2—N1—C8—C9	−0.7 (4)
C7—N3—C1—C2	−163.9 (3)	Zn1—N1—C8—C9	174.8 (2)
C6—C1—C2—C3	−0.3 (4)	N2—N1—C8—C10	−179.7 (3)
N3—C1—C2—C3	−178.5 (3)	Zn1—N1—C8—C10	−4.1 (4)
C1—C2—C3—C4	0.4 (5)		

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···*Cg*^{ii}	0.86	2.86	3.671 (3)	157

Symmetry code: (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.