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1-(2,3,4-Trihydroxybenzylidene)-4-ethylthiosemicarbazide

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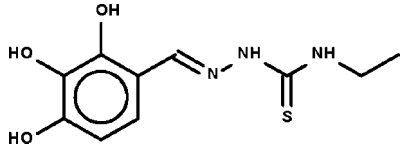
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.082; data-to-parameter ratio = 15.3.

In the title molecule, $C_{10}H_{13}N_3O_3S$, the thiosemicarbazide ═N—NH—C(═S)—NH– fragment is twisted with respect to the aromatic ring [dihedral angle = 20.5 (1)°]. A weak N—H···S hydrogen bond [3.480 (1) Å] links two molecules about a center of inversion to generate a ring. The hydroxy groups are engaged in intermolecular hydrogen bonding; the O—H···O and O—H···S hydrogen bonds generate a layer motif.

Related literature

For the crystal structures of 3,4-dihydroxybenzaldehyde 4-ethylthiosemicarbazone and 2,4-dihydroxybenzaldehyde 4-ethylthiosemicarbazone, see: Kayed *et al.* (2008); Tan *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{13}N_3O_3S$
M_r = 255.29
Monoclinic, $P2_1/c$
a = 7.5668 (5) Å
b = 14.6754 (10) Å
c = 10.8700 (7) Å
β = 104.711 (1)°
V = 1167.50 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.921, T_{max} = 0.973
10870 measured reflections
2660 independent reflections
2364 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.082
S = 1.04
2660 reflections
174 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O2	0.83 (1)	2.26 (2)	2.717 (1)	115 (2)
O1—H1*O*···S1^{i}	0.83 (1)	2.55 (1)	3.291 (1)	150 (2)
O2—H2*O*···O3	0.84 (1)	2.31 (2)	2.745 (1)	112 (2)
O2—H2*O*···O1^{ii}	0.84 (1)	2.07 (1)	2.832 (1)	151 (2)
O3—H3*O*···S1^{iii}	0.84 (1)	2.36 (1)	3.189 (1)	170 (2)
N2—H2*N*···S1^{iv}	0.87	2.62	3.480 (1)	171

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

Data collection: *APEX2* software (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2241).

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supplementary materials

Acta Cryst. (2010). E**66**, o1151 [**doi:10.1107/S1600536810014078**]

1-(2,3,4-Trihydroxybenzylidene)-4-ethylthiosemicarbazide

H. B. Shawish, M. J. Maah and S. W. Ng

Experimental

2,3,4-Trihydroxybenzaldehyde (1.54 g, 10 mmol) and 4-ethylthiosemicarbazide (1.19 g, 1 mmol) were heated in ethanol (20 ml) for 2 hours; acetic acid (0.5 ml) was also added. A brown solid separated from the cool solution; this was recrystallized from methanol.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 or 1.5U(C_{Me}).

The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints of N–H 0.86±0.01 and O–H 0.84±0.01 Å; their temperature factors were freely refined.

Figures

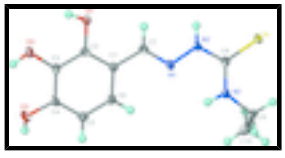

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_{13}N_3O_3S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

1-(2,3,4-Trihydroxybenzylidene)-4-ethylthiosemicarbazide

Crystal data

$C_{10}H_{13}N_3O_3S$	$F(000)$ = 536
M_r = 255.29	D_x = 1.452 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5730 reflections
a = 7.5668 (5) Å	θ = 2.4–28.3°
b = 14.6754 (10) Å	μ = 0.28 mm^{-1}
c = 10.8700 (7) Å	T = 100 K
β = 104.711 (1)°	Prism, colorless
V = 1167.50 (13) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART APEX	2660 independent reflections

diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.921, T_{max} = 0.973

10870 measured reflections

2364 reflections with $I > 2\sigma(I)$

R_{int} = 0.026

θ_{max} = 27.5°, θ_{min} = 2.4°

$h = -9 \rightarrow 9$

$k = -19 \rightarrow 19$

$l = -14 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.029

$wR(F^2)$ = 0.082

S = 1.04

2660 reflections

174 parameters

5 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.4981P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.34 e Å^{-3}

$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.70929 (4)	0.59538 (2)	0.58991 (3)	0.01292 (10)
O1	0.50792 (13)	0.28115 (6)	0.03429 (8)	0.0153 (2)
O2	0.54351 (14)	0.26317 (6)	−0.20701 (9)	0.0176 (2)
O3	0.73079 (14)	0.39064 (6)	−0.30899 (9)	0.0167 (2)
N1	0.73526 (15)	0.49383 (7)	0.26113 (10)	0.0133 (2)
N2	0.69204 (15)	0.50533 (7)	0.37631 (10)	0.0132 (2)
N3	0.91494 (16)	0.61386 (8)	0.42338 (11)	0.0150 (2)
C1	0.66760 (17)	0.42305 (8)	0.05845 (12)	0.0114 (2)
C2	0.59670 (17)	0.34728 (8)	−0.01603 (12)	0.0114 (2)
C3	0.61719 (17)	0.33861 (8)	−0.13940 (12)	0.0122 (2)
C4	0.71179 (18)	0.40505 (8)	−0.18813 (12)	0.0123 (2)
C5	0.78291 (17)	0.48082 (9)	−0.11516 (12)	0.0134 (3)
H5	0.8474	0.5261	−0.1486	0.016*
C6	0.75918 (17)	0.48975 (8)	0.00612 (12)	0.0127 (2)
H6	0.8059	0.5421	0.0550	0.015*
C7	0.64158 (17)	0.43394 (8)	0.18633 (12)	0.0124 (2)
H7	0.5559	0.3969	0.2138	0.015*
C8	0.77826 (17)	0.57177 (8)	0.45530 (12)	0.0118 (2)
C9	1.01416 (19)	0.69298 (9)	0.48851 (13)	0.0168 (3)
H9A	1.1456	0.6869	0.4917	0.020*
H9B	1.0020	0.6951	0.5770	0.020*

C10	0.9416 (2)	0.78045 (10)	0.42107 (17)	0.0283 (4)
H10A	1.0080	0.8323	0.4678	0.042*
H10B	0.8112	0.7863	0.4171	0.042*
H10C	0.9584	0.7795	0.3346	0.042*
H1O	0.472 (3)	0.2409 (11)	−0.0195 (16)	0.039 (6)*
H2O	0.554 (3)	0.2673 (15)	−0.2817 (11)	0.045 (6)*
H3O	0.731 (3)	0.4420 (9)	−0.343 (2)	0.047 (6)*
H2N	0.5992 (17)	0.4785 (11)	0.3933 (15)	0.021 (4)*
H3N	0.936 (2)	0.5975 (11)	0.3531 (11)	0.023 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01775 (18)	0.01251 (16)	0.00958 (16)	−0.00113 (11)	0.00547 (12)	−0.00111 (10)
O1	0.0228 (5)	0.0130 (4)	0.0113 (4)	−0.0065 (4)	0.0064 (4)	−0.0022 (3)
O2	0.0281 (5)	0.0147 (5)	0.0114 (5)	−0.0064 (4)	0.0078 (4)	−0.0041 (4)
O3	0.0259 (5)	0.0153 (5)	0.0110 (5)	0.0000 (4)	0.0089 (4)	0.0007 (3)
N1	0.0157 (5)	0.0150 (5)	0.0104 (5)	−0.0001 (4)	0.0056 (4)	−0.0018 (4)
N2	0.0155 (5)	0.0149 (5)	0.0109 (5)	−0.0040 (4)	0.0063 (4)	−0.0028 (4)
N3	0.0185 (6)	0.0158 (5)	0.0126 (5)	−0.0046 (4)	0.0075 (4)	−0.0047 (4)
C1	0.0116 (6)	0.0122 (5)	0.0103 (6)	0.0016 (4)	0.0024 (4)	0.0003 (4)
C2	0.0118 (6)	0.0105 (5)	0.0124 (6)	0.0007 (4)	0.0038 (5)	0.0015 (4)
C3	0.0144 (6)	0.0109 (6)	0.0109 (6)	0.0011 (5)	0.0023 (5)	−0.0012 (4)
C4	0.0137 (6)	0.0144 (6)	0.0096 (6)	0.0034 (5)	0.0042 (5)	0.0009 (4)
C5	0.0132 (6)	0.0129 (6)	0.0144 (6)	−0.0004 (5)	0.0040 (5)	0.0027 (5)
C6	0.0127 (6)	0.0118 (6)	0.0126 (6)	−0.0003 (5)	0.0016 (5)	−0.0010 (5)
C7	0.0138 (6)	0.0111 (6)	0.0128 (6)	0.0005 (5)	0.0042 (5)	0.0002 (4)
C8	0.0140 (6)	0.0104 (5)	0.0110 (6)	0.0015 (5)	0.0028 (5)	0.0011 (4)
C9	0.0182 (7)	0.0164 (6)	0.0167 (6)	−0.0067 (5)	0.0060 (5)	−0.0043 (5)
C10	0.0201 (8)	0.0167 (7)	0.0444 (10)	−0.0032 (6)	0.0012 (7)	0.0005 (6)

Geometric parameters (Å, °)

S1—C8	1.7092 (13)	C1—C6	1.4007 (17)
O1—C2	1.3707 (15)	C1—C7	1.4613 (17)
O1—H1O	0.827 (9)	C2—C3	1.3944 (17)
O2—C3	1.3669 (15)	C3—C4	1.3914 (17)
O2—H2O	0.838 (9)	C4—C5	1.3923 (17)
O3—C4	1.3738 (15)	C5—C6	1.3811 (17)
O3—H3O	0.841 (9)	C5—H5	0.9500
N1—C7	1.2819 (16)	C6—H6	0.9500
N1—N2	1.3824 (14)	C7—H7	0.9500
N2—C8	1.3523 (16)	C9—C10	1.511 (2)
N2—H2N	0.866 (9)	C9—H9A	0.9900
N3—C8	1.3244 (17)	C9—H9B	0.9900
N3—C9	1.4639 (16)	C10—H10A	0.9800
N3—H3N	0.853 (9)	C10—H10B	0.9800
C1—C2	1.3996 (17)	C10—H10C	0.9800

C2—O1—H1O	109.3 (15)	C6—C5—H5	120.2
C3—O2—H2O	109.6 (15)	C4—C5—H5	120.2
C4—O3—H3O	107.5 (15)	C5—C6—C1	121.26 (11)
C7—N1—N2	115.99 (11)	C5—C6—H6	119.4
C8—N2—N1	118.43 (11)	C1—C6—H6	119.4
C8—N2—H2N	118.7 (11)	N1—C7—C1	119.47 (11)
N1—N2—H2N	122.0 (11)	N1—C7—H7	120.3
C8—N3—C9	125.59 (11)	C1—C7—H7	120.3
C8—N3—H3N	116.0 (12)	N3—C8—N2	116.93 (11)
C9—N3—H3N	117.8 (12)	N3—C8—S1	123.94 (10)
C2—C1—C6	118.47 (11)	N2—C8—S1	119.13 (10)
C2—C1—C7	120.84 (11)	N3—C9—C10	111.14 (11)
C6—C1—C7	120.66 (11)	N3—C9—H9A	109.4
O1—C2—C3	120.30 (11)	C10—C9—H9A	109.4
O1—C2—C1	119.11 (11)	N3—C9—H9B	109.4
C3—C2—C1	120.59 (11)	C10—C9—H9B	109.4
O2—C3—C4	122.81 (11)	H9A—C9—H9B	108.0
O2—C3—C2	117.46 (11)	C9—C10—H10A	109.5
C4—C3—C2	119.72 (11)	C9—C10—H10B	109.5
O3—C4—C3	116.43 (11)	H10A—C10—H10B	109.5
O3—C4—C5	123.27 (11)	C9—C10—H10C	109.5
C3—C4—C5	120.30 (12)	H10A—C10—H10C	109.5
C6—C5—C4	119.63 (12)	H10B—C10—H10C	109.5
C7—N1—N2—C8	−175.71 (11)	O3—C4—C5—C6	−179.39 (12)
C6—C1—C2—O1	179.53 (11)	C3—C4—C5—C6	0.03 (19)
C7—C1—C2—O1	−2.47 (18)	C4—C5—C6—C1	1.21 (19)
C6—C1—C2—C3	0.05 (18)	C2—C1—C6—C5	−1.25 (19)
C7—C1—C2—C3	178.06 (11)	C7—C1—C6—C5	−179.26 (12)
O1—C2—C3—O2	0.95 (18)	N2—N1—C7—C1	174.75 (11)
C1—C2—C3—O2	−179.58 (11)	C2—C1—C7—N1	167.00 (12)
O1—C2—C3—C4	−178.31 (11)	C6—C1—C7—N1	−15.04 (18)
C1—C2—C3—C4	1.16 (19)	C9—N3—C8—N2	173.77 (12)
O2—C3—C4—O3	−0.96 (18)	C9—N3—C8—S1	−7.36 (19)
C2—C3—C4—O3	178.26 (11)	N1—N2—C8—N3	−8.18 (17)
O2—C3—C4—C5	179.58 (12)	N1—N2—C8—S1	172.89 (9)
C2—C3—C4—C5	−1.20 (19)	C8—N3—C9—C10	−97.70 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···O2	0.83 (1)	2.26 (2)	2.717 (1)	115 (2)
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N2—H2N···S1^{iv}	0.87	2.62	3.480 (1)	171

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) *x*, *y*, *z*−1; (iv) −*x*+1, −*y*+1, −*z*+1.

Fig. 1