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2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

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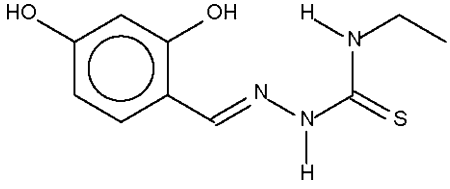
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 17.0.

The molecular conformation of the title compound, $C_{10}H_{13}N_3O_2S$, is stabilized by an intramolecular O—H···N hydrogen bond. Adjacent molecules are linked by O—H···O hydrogen bonds to furnish a zigzag chain.

Related literature

For the structure of 3,4-dihydroxybenzaldehyde 4-ethylthiosemicarbazone, see: Kayed *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{13}N_3O_2S$
M_r = 239.29
Monoclinic, $P2_1/n$
a = 4.6592 (6) Å
b = 24.067 (3) Å
c = 10.047 (1) Å
β = 99.060 (2)°
V = 1112.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 100 (2) K
0.40 × 0.12 × 0.06 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.896, T_{max} = 0.983
6303 measured reflections
2517 independent reflections
1972 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.08
2517 reflections
148 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3	0.84	1.84	2.583 (2)	147
O2—H2···O1^{i}	0.84	1.92	2.714 (2)	158

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2806).

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supporting information

Acta Cryst. (2008). E**64**, o2123 [doi:10.1107/S160053680803300X]

2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

Kong Wai Tan, Chew Hee Ng, Mohd Jamil Maah and Seik Weng Ng

S1. Experimental

4-Ethylthiosemicarbazide (1.19 g, 10 mmol) and 2,4-dihydroxybenzaldehyde (1.38 g, 10 mmol) were refluxed in ethanol (40 ml) for 6 h. Slow evaporation of the solvent yielded yellow crystals.

S2. Refinement

H-atoms were placed in calculated positions (C—H 0.95 Å, N—H 0.88 Å, O—H 0.85 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C,N) or U(H) set to 1.5U(O).

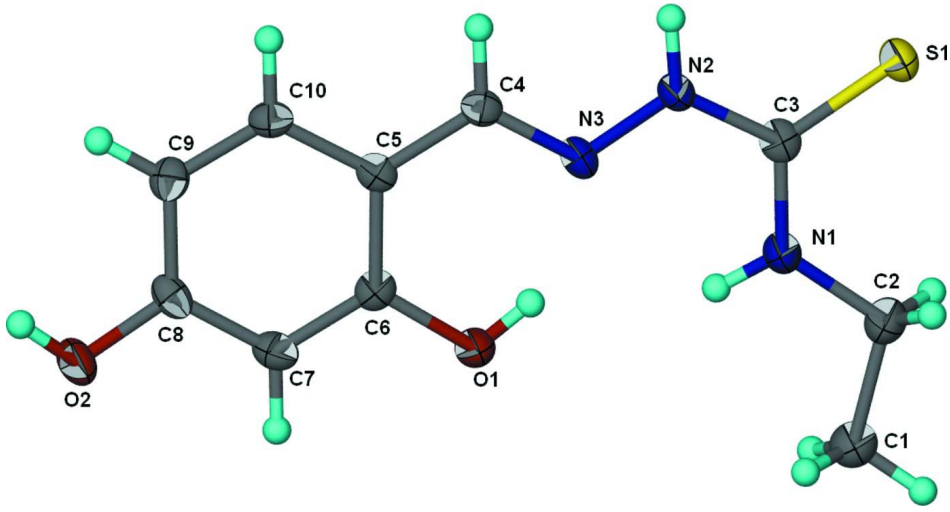

Figure 1

Displacement ellipsoid (Barbour, 2001) plot of the title compound at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

Crystal data

$C_{10}H_{13}N_3O_2S$
M_r = 239.29
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 4.6592 (6) Å
b = 24.067 (3) Å
c = 10.047 (1) Å
β = 99.060 (2)°
V = 1112.5 (2) Å^3
Z = 4
F(000) = 504
D_x = 1.429 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1634 reflections
θ = 2.6–28.1°
μ = 0.28 mm^{-1}
T = 100 K
Plate, yellow
0.40 × 0.12 × 0.06 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} = 0.896, T_{max} = 0.983
6303 measured reflections
2517 independent reflections
1972 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 27.5°, θ_{min} = 1.7°
$h = -6 \rightarrow 4$
$k = -30 \rightarrow 31$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.08
2517 reflections
148 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
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.3651P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	1.01421 (11)	0.514276 (19)	0.28484 (5)	0.02112 (15)
O1	0.1480 (3)	0.32859 (6)	0.28304 (13)	0.0240 (3)
H1	0.2707	0.3544	0.2908	0.036*
O2	−0.4004 (3)	0.21979 (5)	0.53651 (13)	0.0238 (3)
H2	−0.4152	0.2117	0.6165	0.036*
N1	0.6825 (4)	0.43087 (7)	0.17727 (16)	0.0225 (4)
H1N	0.5495	0.4056	0.1845	0.027*
N2	0.6791 (3)	0.44770 (6)	0.40159 (15)	0.0175 (3)
H2N	0.7378	0.4666	0.4760	0.021*
N3	0.4854 (3)	0.40468 (6)	0.40248 (15)	0.0169 (3)
C1	0.6265 (5)	0.39432 (9)	−0.0504 (2)	0.0270 (5)
H1A	0.6985	0.3971	−0.1367	0.040*
H1B	0.4169	0.4015	−0.0644	0.040*
H1C	0.6644	0.3569	−0.0132	0.040*
C2	0.7804 (5)	0.43673 (9)	0.0471 (2)	0.0276 (5)
H2A	0.9932	0.4309	0.0578	0.033*
H2B	0.7371	0.4747	0.0115	0.033*
C3	0.7800 (4)	0.46113 (7)	0.28620 (18)	0.0174 (4)
C4	0.3905 (4)	0.39384 (7)	0.51361 (17)	0.0162 (4)
H4	0.4568	0.4152	0.5920	0.019*
C5	0.1834 (4)	0.34936 (7)	0.51966 (18)	0.0154 (4)
C6	0.0690 (4)	0.31772 (7)	0.40586 (18)	0.0174 (4)
C7	−0.1245 (4)	0.27525 (8)	0.41357 (19)	0.0195 (4)
H7	−0.1983	0.2545	0.3352	0.023*
C8	−0.2121 (4)	0.26265 (7)	0.53563 (18)	0.0179 (4)

C9	−0.1092 (4)	0.29412 (7)	0.65039 (18)	0.0181 (4)
H9	−0.1729	0.2864	0.7338	0.022*
C10	0.0857 (4)	0.33653 (7)	0.64102 (18)	0.0171 (4)
H10	0.1560	0.3577	0.7192	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0235 (3)	0.0188 (2)	0.0214 (2)	−0.0054 (2)	0.00430 (19)	0.00073 (18)
O1	0.0316 (9)	0.0240 (7)	0.0169 (6)	−0.0099 (6)	0.0059 (6)	−0.0026 (5)
O2	0.0275 (8)	0.0207 (7)	0.0225 (7)	−0.0089 (6)	0.0019 (6)	0.0036 (5)
N1	0.0235 (9)	0.0242 (8)	0.0207 (8)	−0.0087 (7)	0.0063 (7)	−0.0028 (7)
N2	0.0181 (8)	0.0176 (8)	0.0167 (7)	−0.0047 (6)	0.0022 (6)	−0.0009 (6)
N3	0.0154 (8)	0.0139 (7)	0.0211 (8)	−0.0016 (6)	0.0019 (6)	0.0017 (6)
C1	0.0302 (12)	0.0283 (11)	0.0228 (10)	−0.0037 (9)	0.0056 (9)	−0.0022 (8)
C2	0.0301 (12)	0.0332 (11)	0.0209 (10)	−0.0089 (10)	0.0081 (9)	−0.0025 (8)
C3	0.0141 (10)	0.0168 (9)	0.0209 (9)	0.0029 (7)	0.0017 (7)	0.0019 (7)
C4	0.0151 (10)	0.0162 (8)	0.0165 (9)	0.0004 (7)	0.0004 (7)	−0.0011 (7)
C5	0.0138 (9)	0.0133 (8)	0.0185 (9)	0.0021 (7)	0.0006 (7)	0.0002 (7)
C6	0.0176 (10)	0.0179 (9)	0.0166 (9)	0.0025 (7)	0.0027 (7)	0.0007 (7)
C7	0.0208 (10)	0.0169 (9)	0.0195 (9)	−0.0012 (8)	−0.0010 (8)	−0.0014 (7)
C8	0.0154 (10)	0.0139 (8)	0.0232 (9)	−0.0007 (7)	−0.0005 (8)	0.0031 (7)
C9	0.0184 (10)	0.0190 (9)	0.0168 (9)	0.0012 (7)	0.0021 (7)	0.0033 (7)
C10	0.0180 (10)	0.0170 (9)	0.0155 (8)	0.0015 (8)	−0.0003 (7)	−0.0017 (7)

Geometric parameters (Å, º)

S1—C3	1.6826 (19)	C1—H1C	0.9800
O1—C6	1.367 (2)	C2—H2A	0.9900
O1—H1	0.8400	C2—H2B	0.9900
O2—C8	1.355 (2)	C4—C5	1.449 (2)
O2—H2	0.8400	C4—H4	0.9500
N1—C3	1.333 (2)	C5—C10	1.401 (2)
N1—C2	1.458 (2)	C5—C6	1.407 (2)
N1—H1N	0.8800	C6—C7	1.373 (3)
N2—C3	1.357 (2)	C7—C8	1.386 (3)
N2—N3	1.374 (2)	C7—H7	0.9500
N2—H2N	0.8800	C8—C9	1.400 (3)
N3—C4	1.291 (2)	C9—C10	1.379 (3)
C1—C2	1.515 (3)	C9—H9	0.9500
C1—H1A	0.9800	C10—H10	0.9500
C1—H1B	0.9800		
C6—O1—H1	109.5	N2—C3—S1	120.03 (14)
C8—O2—H2	109.5	N3—C4—C5	120.45 (16)
C3—N1—C2	124.66 (16)	N3—C4—H4	119.8
C3—N1—H1N	117.7	C5—C4—H4	119.8
C2—N1—H1N	117.7	C10—C5—C6	117.04 (17)

C3—N2—N3	120.08 (15)	C10—C5—C4	120.59 (16)
C3—N2—H2N	120.0	C6—C5—C4	122.37 (16)
N3—N2—H2N	120.0	O1—C6—C7	117.76 (17)
C4—N3—N2	118.21 (15)	O1—C6—C5	120.57 (17)
C2—C1—H1A	109.5	C7—C6—C5	121.67 (17)
C2—C1—H1B	109.5	C6—C7—C8	120.03 (17)
H1A—C1—H1B	109.5	C6—C7—H7	120.0
C2—C1—H1C	109.5	C8—C7—H7	120.0
H1A—C1—H1C	109.5	O2—C8—C7	116.98 (17)
H1B—C1—H1C	109.5	O2—C8—C9	123.01 (17)
N1—C2—C1	109.36 (17)	C7—C8—C9	120.00 (17)
N1—C2—H2A	109.8	C10—C9—C8	119.21 (17)
C1—C2—H2A	109.8	C10—C9—H9	120.4
N1—C2—H2B	109.8	C8—C9—H9	120.4
C1—C2—H2B	109.8	C9—C10—C5	122.00 (17)
H2A—C2—H2B	108.3	C9—C10—H10	119.0
N1—C3—N2	116.86 (17)	C5—C10—H10	119.0
N1—C3—S1	123.11 (14)		

C3—N2—N3—C4	−178.76 (17)	C10—C5—C6—C7	−1.5 (3)
C3—N1—C2—C1	178.52 (18)	C4—C5—C6—C7	179.20 (17)
C2—N1—C3—N2	−175.78 (18)	O1—C6—C7—C8	−179.83 (17)
C2—N1—C3—S1	4.9 (3)	C5—C6—C7—C8	0.1 (3)
N3—N2—C3—N1	0.0 (2)	C6—C7—C8—O2	−179.15 (16)
N3—N2—C3—S1	179.30 (13)	C6—C7—C8—C9	1.6 (3)
N2—N3—C4—C5	179.27 (15)	O2—C8—C9—C10	179.01 (17)
N3—C4—C5—C10	178.30 (17)	C7—C8—C9—C10	−1.8 (3)
N3—C4—C5—C6	−2.4 (3)	C8—C9—C10—C5	0.3 (3)
C10—C5—C6—O1	178.39 (16)	C6—C5—C10—C9	1.3 (3)
C4—C5—C6—O1	−0.9 (3)	C4—C5—C10—C9	−179.39 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.84	1.84	2.583 (2)	147
O2—H2···O1^{i}	0.84	1.92	2.714 (2)	158

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