Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

Kong Wai Tan,[a] Chew Hee Ng,[b] Mohd Jamil Maah[a]* and Seik Weng Ng[a]

[a]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and [b]Faculty of Engineering and Science, Universiti Tunku Abdul Rahman, 53300 Kuala Lumpur, Malaysia
Correspondence e-mail: mjamil@um.edu.my

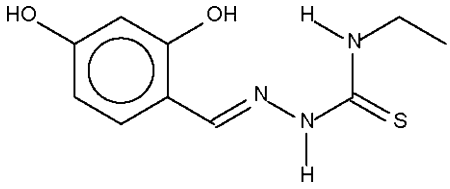
Received 6 October 2008; accepted 12 October 2008

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).

We thank the University of Malaya (grant No. PJP F316/2008C) for supporting this study. KWT thanks the Ministry of Higher Education for an SLAI scholarship in this research.

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

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kayed, S. F., Farina, Y., Baba, I. & Simpson, J. (2008). *Acta Cryst.* E**64**, o824–o825.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2008). *publCIF*. In preparation.

supplementary materials

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

2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

K. W. Tan, C. H. Ng, M. J. Maah and S. W. Ng

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.

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).

Figures

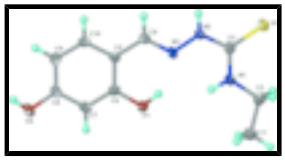


Fig. 1. Displacement ellipsoid (Barbour, 2001) plot of the title compound at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

2,4-Dihydroxybenzaldehyde 4-ethylthiosemicarbazone

Crystal data

$C_{10}H_{13}N_3O_2S$	F_{000} = 504
M_r = 239.29	D_x = 1.429 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1634 reflections
a = 4.6592 (6) Å	θ = 2.6–28.1º
b = 24.067 (3) Å	μ = 0.28 mm^{-1}
c = 10.047 (1) Å	T = 100 (2) K
β = 99.060 (2)º	Plate, yellow
V = 1112.5 (2) Å^3	0.40 × 0.12 × 0.06 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	2517 independent reflections
Radiation source: fine-focus sealed tube	1972 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 100(2) K	θ_{max} = 27.5º

ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −6→4
T_{min} = 0.896, T_{max} = 0.983	k = −30→31
6303 measured reflections	l = −13→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.109	$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.3651P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
2517 reflections	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
148 parameters	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	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 (Å^2)

	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, °)

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, °)

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

Fig. 1