Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Hana Bashir Shawish, M. Jamil Maah* and Seik Weng Ng

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: mjamil@um.edu.my

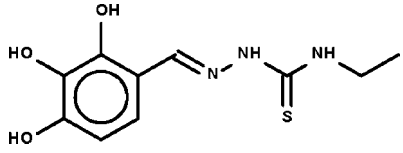
Received 8 April 2010; accepted 16 April 2010

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).

The authors thank the University of Malaya (grant No. PS354/2009) and MOHE (grant No. FRGS-FP001/2009) for supporting this study. HBS thanks the Libyan People's Bureau in Malaysia for a scholarship.

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

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2009). *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.
Tan, K. W., Ng, C. H., Maah, M. J. & Ng, S. W. (2008). *Acta Cryst.* E**64**, o2123.
Westrip, S. P. (2010). *publCIF*. In preparation.

supporting information

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

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

Hana Bashir Shawish, M. Jamil Maah and Seik Weng Ng

S1. 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.

S2. 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.

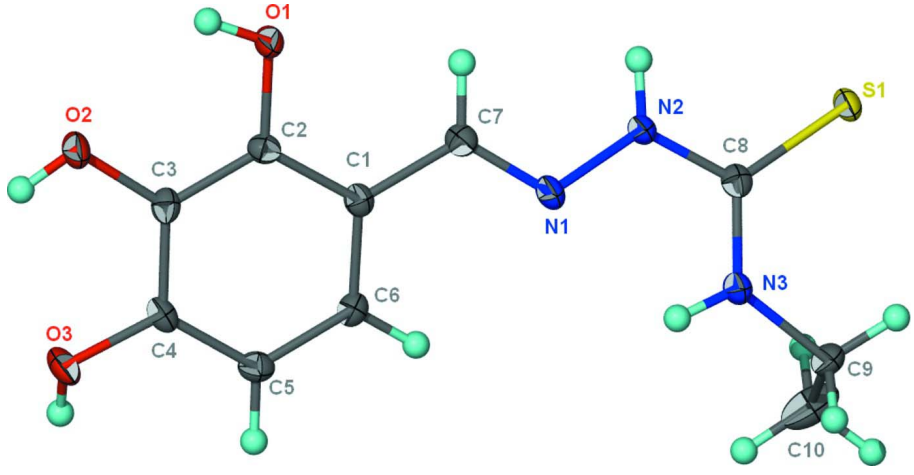


Figure 1

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

$C_{10}H_{13}N_3O_3S$
M_r = 255.29
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
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
F(000) = 536
D_x = 1.452 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5730 reflections

θ = 2.4–28.3°
μ = 0.28 mm^{-1}
T = 100 K

Prism, colorless
0.30 × 0.20 × 0.10 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} = 0.921, T_{max} = 0.973

10870 measured reflections
2660 independent reflections
2364 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −9→9
k = −19→19
l = −14→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}
$\Delta\rho_{min}$ = −0.22 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 (Å²)

	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, º)

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, º)

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)
O1—H1o···S1^{i}	0.83 (1)	2.55 (1)	3.291 (1)	150 (2)
O2—H2o···O3	0.84 (1)	2.31 (2)	2.745 (1)	112 (2)
O2—H2o···O1^{ii}	0.84 (1)	2.07 (1)	2.832 (1)	151 (2)

O3—H3o···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*−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.