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(*E*)-1-(2,4,6-Trihydroxybenzylidene)-4-ethylthiosemicarbazide dihydrate

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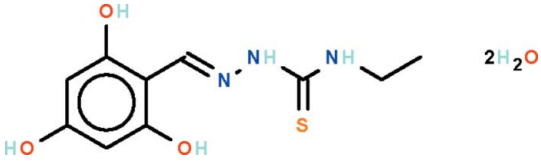
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.074; data-to-parameter ratio = 14.1.

In the title molecule, $C_{10}H_{13}N_3O_3S\cdot 2H_2O$, the thiosemicarbazide ═N—NH—C(═S)—NH– fragment [torsion angle = 0.2 (1)°] is nearly coplanar with the benzene ring [dihedral angle = 2.4 (1)°]. The benzene ring and semicarbazide moiety are located on opposite sites of the C═N bond, showing an *E* configuration. The hydroxy, imino and water H atoms are engaged in extensive hydrogen bonding, forming a three-dimensional network.

Related literature

For the crystal structure of a related compound, 1-(2,3,4-trihydroxybenzylidene)-4-ethylthiosemicarbazide, see: Shawish *et al.* (2010).



Experimental

Crystal data

$C_{10}H_{13}N_3O_3S\cdot 2H_2O$
M_r = 291.33
Monoclinic, $P2_1$
a = 4.6645 (4) Å
b = 10.4006 (9) Å
c = 13.5381 (11) Å
β = 98.674 (1)°
V = 649.27 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.27 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} = 0.923, T_{max} = 0.973
6232 measured reflections
2937 independent reflections
2826 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.03
2937 reflections
208 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1370 Friedel pairs
Flack parameter: −0.05 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···N1	0.85 (1)	1.99 (2)	2.722 (2)	144 (2)
O2—H2o···O1^{i}	0.83 (1)	2.27 (2)	2.955 (2)	140 (2)
O3—H3o···O1W	0.85 (2)	1.76 (2)	2.598 (2)	174 (2)
O1w—H11···O2w	0.84 (1)	1.94 (1)	2.785 (2)	177 (3)
O1w—H12···O3^{ii}	0.84 (1)	2.22 (1)	3.002 (2)	155 (2)
O2w—H21···S1^{iii}	0.84 (1)	2.45 (1)	3.279 (1)	169 (2)
O2w—H22···S1^{iv}	0.84 (1)	2.49 (1)	3.292 (1)	162 (2)
N2—H2n···O2w^{v}	0.86 (1)	2.13 (1)	2.965 (2)	166 (2)
N3—H3n···O2^{vi}	0.86 (1)	2.26 (2)	2.934 (2)	136 (2)

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

Data collection: *APEX2* (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).

We thank the University of Malaya (PS354/2009) and MOHE (FRGS-FP001/2009) for supporting this study. HBS also 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: XU5011).

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supporting information

Acta Cryst. (2010). E**66**, o2230 [https://doi.org/10.1107/S1600536810030783]

(*E*)-1-(2,4,6-Trihydroxybenzylidene)-4-ethylthiosemicarbazide dihydrate

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

S1. Experimental

2,4,6-Trihydroxybenzaldehyde (1.54 g, 10 mmol) and 4-ethylthiosemicarbazide (1.19 g, 1 mmol) were heated in ethanol (20 ml) for 2 h; 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 0.99 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5U(C).

The imino H 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.

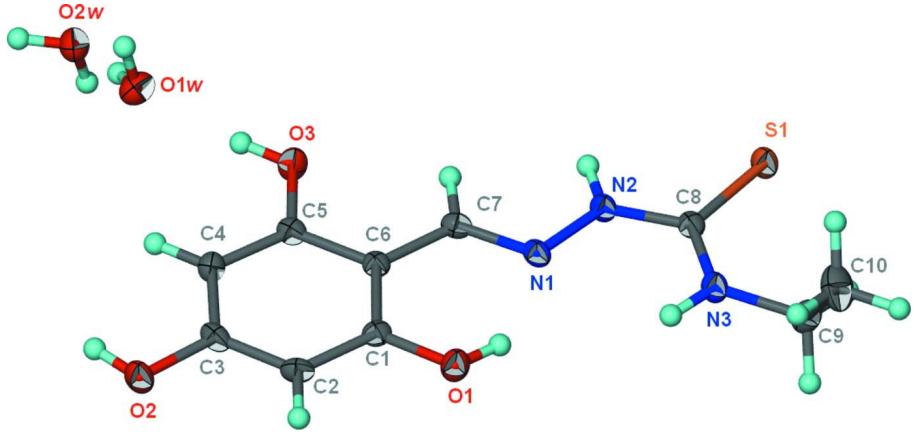

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_{13}N_3O_3S\cdot 2H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

$C_{10}H_{13}N_3O_3S\cdot 2H_2O$
M_r = 291.33
Monoclinic, $P2_1$
Hall symbol: P 2yb
a = 4.6645 (4) Å
b = 10.4006 (9) Å
c = 13.5381 (11) Å
β = 98.674 (1)°
V = 649.27 (10) Å^3
Z = 2
F(000) = 308
D_x = 1.490 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3449 reflections
θ = 2.5–28.2°
μ = 0.27 mm^{-1}

T = 100 K
Prism, yellow

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} = 0.923, T_{max} = 0.973

6232 measured reflections
2937 independent reflections
2826 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 27.5°, θ_{min} = 1.5°
h = −6→5
k = −13→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.074
S = 1.03
2937 reflections
208 parameters
12 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
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.0466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 1370 Friedel pairs
Absolute structure parameter: −0.05 (6)

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

	x	y	z	U_{iso}*/U_{eq}
S1	1.52160 (9)	1.00002 (4)	0.04613 (3)	0.01642 (10)
O1	1.0480 (3)	0.82234 (12)	0.44254 (9)	0.0175 (3)
O2	0.4114 (3)	0.51122 (13)	0.53548 (9)	0.0185 (3)
O3	0.5005 (3)	0.56687 (12)	0.18897 (9)	0.0171 (3)
O1W	0.0357 (3)	0.42702 (13)	0.15044 (10)	0.0204 (3)
O2W	0.0077 (3)	0.21977 (12)	0.01907 (10)	0.0197 (3)
N1	1.1216 (3)	0.83376 (14)	0.24708 (10)	0.0136 (3)
N2	1.2187 (3)	0.87300 (14)	0.16047 (10)	0.0139 (3)
N3	1.5378 (3)	1.01558 (15)	0.24590 (10)	0.0147 (3)
C1	0.8575 (3)	0.72575 (16)	0.41274 (13)	0.0133 (3)
C2	0.7317 (4)	0.66456 (16)	0.48651 (13)	0.0152 (3)
H2	0.7859	0.6873	0.5547	0.018*
C3	0.5256 (4)	0.56959 (17)	0.45936 (12)	0.0149 (3)
C4	0.4389 (4)	0.53585 (15)	0.36022 (12)	0.0142 (3)
H4	0.2932	0.4726	0.3427	0.017*
C5	0.5697 (4)	0.59662 (15)	0.28686 (13)	0.0136 (3)
C6	0.7848 (4)	0.69196 (15)	0.31126 (13)	0.0126 (3)
C7	0.9158 (4)	0.74927 (16)	0.23201 (13)	0.0135 (3)
H7	0.8477	0.7237	0.1652	0.016*
C8	1.4257 (3)	0.96307 (15)	0.15970 (13)	0.0131 (3)
C9	1.7695 (4)	1.11131 (17)	0.25974 (13)	0.0179 (4)

H9A	1.8548	1.1188	0.1973	0.021*
H9B	1.9242	1.0822	0.3133	0.021*
C10	1.6620 (5)	1.24156 (18)	0.28688 (17)	0.0274 (4)
H10A	1.8236	1.3028	0.2953	0.041*
H10B	1.5812	1.2350	0.3495	0.041*
H10C	1.5113	1.2715	0.2335	0.041*
H1O	1.132 (4)	0.846 (2)	0.3945 (12)	0.022 (6)*
H2O	0.280 (4)	0.462 (2)	0.5106 (17)	0.036 (7)*
H3O	0.356 (4)	0.517 (2)	0.1785 (17)	0.029 (6)*
H11	0.024 (5)	0.3659 (19)	0.1092 (18)	0.048 (8)*
H12	−0.122 (4)	0.467 (2)	0.1412 (19)	0.059 (10)*
H21	−0.127 (3)	0.1711 (18)	0.0304 (18)	0.029 (7)*
H22	0.162 (3)	0.178 (2)	0.022 (2)	0.047 (8)*
H2N	1.140 (4)	0.841 (2)	0.1046 (10)	0.020 (5)*
H3N	1.476 (4)	0.9852 (19)	0.2978 (10)	0.014 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01776 (19)	0.0203 (2)	0.01189 (19)	−0.00408 (18)	0.00442 (14)	0.00107 (17)
O1	0.0181 (6)	0.0191 (6)	0.0151 (6)	−0.0046 (5)	0.0025 (5)	−0.0017 (5)
O2	0.0223 (6)	0.0215 (6)	0.0122 (6)	−0.0044 (6)	0.0042 (5)	0.0020 (5)
O3	0.0195 (6)	0.0208 (6)	0.0113 (6)	−0.0057 (5)	0.0031 (5)	−0.0035 (5)
O1W	0.0173 (7)	0.0228 (7)	0.0204 (7)	−0.0006 (5)	0.0009 (5)	−0.0031 (5)
O2W	0.0190 (7)	0.0185 (7)	0.0215 (7)	−0.0023 (6)	0.0033 (6)	0.0019 (5)
N1	0.0141 (7)	0.0146 (6)	0.0128 (7)	0.0017 (5)	0.0042 (6)	0.0024 (5)
N2	0.0155 (7)	0.0167 (7)	0.0097 (7)	−0.0028 (5)	0.0029 (6)	0.0004 (5)
N3	0.0158 (7)	0.0176 (7)	0.0117 (6)	−0.0026 (6)	0.0052 (5)	−0.0001 (6)
C1	0.0114 (8)	0.0134 (7)	0.0150 (8)	0.0024 (6)	0.0014 (6)	−0.0002 (6)
C2	0.0165 (9)	0.0182 (8)	0.0102 (8)	0.0027 (7)	0.0001 (6)	−0.0001 (6)
C3	0.0168 (8)	0.0146 (8)	0.0142 (8)	0.0038 (7)	0.0057 (6)	0.0033 (6)
C4	0.0139 (8)	0.0136 (8)	0.0153 (8)	−0.0008 (6)	0.0033 (6)	−0.0008 (6)
C5	0.0141 (8)	0.0137 (7)	0.0133 (8)	0.0026 (6)	0.0033 (6)	0.0004 (6)
C6	0.0115 (8)	0.0129 (7)	0.0137 (8)	0.0025 (6)	0.0027 (6)	0.0011 (6)
C7	0.0150 (8)	0.0141 (8)	0.0115 (8)	0.0028 (6)	0.0020 (6)	0.0000 (6)
C8	0.0118 (8)	0.0134 (7)	0.0146 (8)	0.0019 (6)	0.0033 (6)	0.0020 (6)
C9	0.0164 (9)	0.0202 (8)	0.0171 (9)	−0.0032 (7)	0.0031 (7)	−0.0027 (7)
C10	0.0308 (11)	0.0174 (9)	0.0363 (12)	−0.0024 (8)	0.0122 (9)	0.0012 (8)

Geometric parameters (Å, º)

S1—C8	1.7085 (17)	N3—H3N	0.86 (1)
O1—C1	1.362 (2)	C1—C2	1.387 (2)
O1—H1O	0.85 (1)	C1—C6	1.409 (2)
O2—C3	1.3710 (19)	C2—C3	1.389 (2)
O2—H2O	0.83 (1)	C2—H2	0.9500
O3—C5	1.352 (2)	C3—C4	1.387 (2)
O3—H3O	0.85 (1)	C4—C5	1.393 (2)

O1W—H11	0.84 (1)	C4—H4	0.9500
O1W—H12	0.84 (1)	C5—C6	1.414 (2)
O2W—H21	0.84 (1)	C6—C7	1.442 (2)
O2W—H22	0.84 (1)	C7—H7	0.9500
N1—C7	1.294 (2)	C9—C10	1.509 (3)
N1—N2	1.3809 (19)	C9—H9A	0.9900
N2—C8	1.346 (2)	C9—H9B	0.9900
N2—H2N	0.86 (1)	C10—H10A	0.9800
N3—C8	1.323 (2)	C10—H10B	0.9800
N3—C9	1.461 (2)	C10—H10C	0.9800

C1—O1—H1O	110.3 (16)	O3—C5—C4	121.98 (15)
C3—O2—H2O	108.4 (17)	O3—C5—C6	116.47 (15)
C5—O3—H3O	111.8 (16)	C4—C5—C6	121.55 (15)
H11—O1W—H12	107.8 (15)	C1—C6—C5	117.44 (15)
H21—O2W—H22	109.8 (15)	C1—C6—C7	123.77 (15)
C7—N1—N2	113.40 (14)	C5—C6—C7	118.80 (15)
C8—N2—N1	122.67 (15)	N1—C7—C6	123.43 (15)
C8—N2—H2N	118.4 (15)	N1—C7—H7	118.3
N1—N2—H2N	118.9 (15)	C6—C7—H7	118.3
C8—N3—C9	125.54 (14)	N3—C8—N2	117.97 (15)
C8—N3—H3N	115.7 (14)	N3—C8—S1	125.35 (13)
C9—N3—H3N	118.6 (14)	N2—C8—S1	116.68 (13)
O1—C1—C2	116.95 (15)	N3—C9—C10	112.11 (15)
O1—C1—C6	121.56 (15)	N3—C9—H9A	109.2
C2—C1—C6	121.46 (15)	C10—C9—H9A	109.2
C1—C2—C3	119.14 (15)	N3—C9—H9B	109.2
C1—C2—H2	120.4	C10—C9—H9B	109.2
C3—C2—H2	120.4	H9A—C9—H9B	107.9
O2—C3—C4	121.71 (16)	C9—C10—H10A	109.5
O2—C3—C2	116.61 (15)	C9—C10—H10B	109.5
C4—C3—C2	121.67 (15)	H10A—C10—H10B	109.5
C3—C4—C5	118.68 (16)	C9—C10—H10C	109.5
C3—C4—H4	120.7	H10A—C10—H10C	109.5
C5—C4—H4	120.7	H10B—C10—H10C	109.5

C7—N1—N2—C8	178.00 (15)	O3—C5—C6—C1	179.15 (15)
O1—C1—C2—C3	176.95 (15)	C4—C5—C6—C1	−1.5 (2)
C6—C1—C2—C3	−1.2 (2)	O3—C5—C6—C7	−0.6 (2)
C1—C2—C3—O2	179.17 (15)	C4—C5—C6—C7	178.74 (15)
C1—C2—C3—C4	−1.1 (2)	N2—N1—C7—C6	178.51 (14)
O2—C3—C4—C5	−178.27 (15)	C1—C6—C7—N1	2.9 (3)
C2—C3—C4—C5	2.0 (2)	C5—C6—C7—N1	−177.42 (15)
C3—C4—C5—O3	178.64 (15)	C9—N3—C8—N2	177.96 (15)
C3—C4—C5—C6	−0.6 (2)	C9—N3—C8—S1	−2.1 (2)
O1—C1—C6—C5	−175.63 (14)	N1—N2—C8—N3	−0.2 (2)
C2—C1—C6—C5	2.5 (2)	N1—N2—C8—S1	179.78 (12)
O1—C1—C6—C7	4.1 (3)	C8—N3—C9—C10	110.57 (19)

C2—C1—C6—C7	−177.80 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N1	0.85 (1)	1.99 (2)	2.722 (2)	144 (2)
O2—H2o···O1^{i}	0.83 (1)	2.27 (2)	2.955 (2)	140 (2)
O3—H3o···O1*W*	0.85 (2)	1.76 (2)	2.598 (2)	174 (2)
O1w—H11···O2w	0.84 (1)	1.94 (1)	2.785 (2)	177 (3)
O1w—H12···O3^{ii}	0.84 (1)	2.22 (1)	3.002 (2)	155 (2)
O2w—H21···S1^{iii}	0.84 (1)	2.45 (1)	3.279 (1)	169 (2)
O2w—H22···S1^{iv}	0.84 (1)	2.49 (1)	3.292 (1)	162 (2)
N2—H2n···O2w^{v}	0.86 (1)	2.13 (1)	2.965 (2)	166 (2)
N3—H3n···O2^{vi}	0.86 (1)	2.26 (2)	2.934 (2)	136 (2)

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