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

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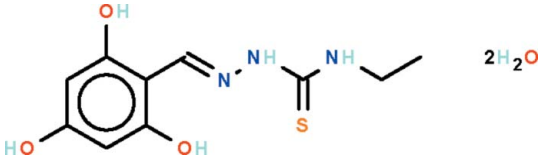
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.074; data-to-parameter ratio = 14.1.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5011).

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supplementary materials

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

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

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

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.

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 1.5*U*(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.

Figures

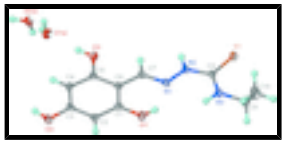

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{10}H_{13}N_3O_3S\cdot 2H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{10}H_{13}N_3O_3S\cdot 2H_2O$	$F(000) = 308$
$M_r = 291.33$	$D_x = 1.490$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 3449 reflections
$a = 4.6645$ (4) Å	$\theta = 2.5$–$28.2°$
$b = 10.4006$ (9) Å	$\mu = 0.27$ mm^{-1}
$c = 13.5381$ (11) Å	$T = 100$ K
$\beta = 98.674$ (1)°	Prism, yellow
$V = 649.27$ (10) Å^3	0.30 × 0.20 × 0.10 mm
$Z = 2$	

Data collection

Bruker SMART APEX diffractometer	2937 independent reflections

Radiation source: fine-focus sealed tube	2826 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.025$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 1.5°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -6 \rightarrow 5$
$T_{min} = 0.923$, $T_{max} = 0.973$	$k = -13 \rightarrow 13$
6232 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.074$	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.0466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2937 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
12 restraints	Absolute structure: Flack (1983), 1370 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.05 (6)

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

	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 (Å^2)

	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, °)

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, °)

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)
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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.

Fig. 1