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Crystal structure of (*E*)-2,3-dihydroxybenzaldehyde *O*-(2-(((*E*)-1-(2,5-dihydroxyphenyl)ethylidene)amino)oxy)ethyl) oxime monohydrate, $C_{17}H_{20}N_2O_7$

DOI 10.1515/ncrs-2016-0388

Received March 21, 2017; accepted June 5, 2017; available online June 24, 2017

Abstract

$C_{17}H_{20}N_2O_7$, monoclinic, $P2_1/c$ (no. 14), $a = 4.6318(11)$ Å, $b = 13.472(3)$ Å, $c = 27.120(7)$ Å, $\beta = 91.081(5)^\circ$, $V = 1692.1(7)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0637$, $wR_{ref}(F^2) = 0.1297$, $T = 298(2)$ K.

CCDC no.: 1536887

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

An ethanol solution (4 mL) of 1,2-bis(aminoxy)ethane (43.2 mg, 0.469 mmol) was slowly added to the ethanol solution (4 mL) of 2,5-dihydroxyacetophenone (357.5 mg, 0.961 mmol). The mixture was stirred for 24 h at 333 K. After cooling to room temperature, the precipitate was filtered and washed with ethanol and ethanol/hexane (1:4) (12 mL),

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	$0.25 \times 0.22 \times 0.19$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.1 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω
$2\theta_{\text{max}}$, completeness:	50° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3225, 2980, 0.042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1975
$N(\text{param})_{\text{refined}}$:	238
Programs:	Bruker [1], SHELX [2]

respectively. The product was purified by recrystallization from ethanol and dried in vacuo yielding yellow powder (Yield: 61.5%, m.p. 528–530 K). Elemental analysis-Found: C, 59.66%; H, 6.12%; N, 26.49%. calc. for $C_{17}H_{20}N_2O_7$: C, 59.85%; H, 6.31%; N, 25.93%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Oxime compounds have been widely studied for many decades [3–7]. Much attentions have recently been paid to the synthesis and crystal structure of Salamo-type bisoxime compounds and their derivatives [8–11]. These compounds are an important class of organic compounds because of their applications in many fields of organic, biological, analytical chemistry and material science [12–17].

The title compound is only built up by the $C_{17}H_{20}N_2O_7$ molecules, in which all bond lengths are within the normal ranges (*cf.* the figure). There are two intramolecular hydrogen bonds ($O4-H4 \cdots N2$, $O5-H5 \cdots N1$) and intermolecular hydrogen bonds involving the water molecule [18–20]. Thus the title compound forms a chain structure along the a axis.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
C1	0.0015(9)	0.3125(3)	0.52082(16)	0.0438(11)
C2	0.1650(10)	0.3512(4)	0.55968(17)	0.0550(13)
H2	0.1411	0.4174	0.5686	0.066*
C3	0.3621(11)	0.2935(5)	0.58533(19)	0.0625(14)
H3	0.4722	0.3211	0.6109	0.075*
C4	0.3972(11)	0.1938(5)	0.5732(2)	0.0633(15)
H4A	0.5292	0.1547	0.5906	0.076*
C5	0.2370(11)	0.1543(3)	0.53568(19)	0.0542(13)
C6	0.0434(9)	0.2134(3)	0.50816(16)	0.0448(11)
C7	−0.2008(10)	0.3744(3)	0.49433(17)	0.0448(11)
H7	−0.2304	0.4390	0.5052	0.054*
C8	−0.0881(14)	0.6020(4)	0.2973(2)	0.0722(16)
H8A	0.0850	0.6237	0.3141	0.108*
H8B	−0.2449	0.6015	0.3199	0.108*
H8C	−0.1324	0.6465	0.2705	0.108*
C9	−0.6700(10)	0.3778(4)	0.39357(18)	0.0525(12)
H9A	−0.8132	0.4258	0.3825	0.063*
H9B	−0.7715	0.3179	0.4028	0.063*
C10	−0.4757(10)	0.3543(3)	0.35149(17)	0.0486(11)
H10A	−0.3239	0.3093	0.3623	0.058*
H10B	−0.5852	0.3227	0.3250	0.058*
C11	−0.0445(10)	0.5001(3)	0.27755(16)	0.0434(11)
C12	0.1555(9)	0.4824(3)	0.23658(16)	0.0409(10)
C13	0.2991(11)	0.5629(4)	0.21523(18)	0.0520(12)
H13	0.2625	0.6269	0.2263	0.062*
C14	0.4920(11)	0.5488(4)	0.17836(19)	0.0553(13)
C15	0.5494(12)	0.4561(4)	0.16075(19)	0.0589(13)
H15	0.6817	0.4474	0.1358	0.071*
C16	0.4099(11)	0.3761(4)	0.18033(18)	0.0527(12)
H16	0.4469	0.3130	0.1680	0.063*
C17	0.2135(10)	0.3871(3)	0.21829(16)	0.0432(11)
N1	−0.3411(8)	0.3438(3)	0.45641(14)	0.0448(9)
N2	−0.1705(8)	0.4234(3)	0.29504(13)	0.0454(9)
O1	−0.5229(7)	0.4164(2)	0.43602(12)	0.0514(8)
O2	−0.3529(7)	0.4456(2)	0.33456(11)	0.0507(8)
O3	0.6191(10)	0.6326(3)	0.15868(18)	0.0942(15)
H3A	0.7542	0.6183	0.1321	0.141*
O4	0.0903(8)	0.3040(2)	0.23602(13)	0.0593(9)
H4	−0.0102	0.3182	0.2596	0.089*
O5	−0.0990(8)	0.1681(3)	0.47050(13)	0.0651(10)
H5	−0.1850	0.2098	0.4538	0.098*
O6	0.2515(9)	0.0566(3)	0.52375(16)	0.0801(12)
H6	0.4473	0.0382	0.5329	0.120*
O1W	0.4427(11)	0.0850(5)	0.41109(18)	0.130(2)
H1WB	0.3073	0.1106	0.3906	0.195*
H1WA	0.3997	0.0982	0.4398	0.195*

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