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Crystal structure of 2-((*E*)-((4-((*E*)-1-(hydroxyimino)ethyl)phenyl)iminio)methyl)-5-methoxyphenolate, C₁₆H₁₆N₂O₃

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Abstract

C₁₆H₁₆N₂O₃, triclinic, $P\bar{1}$ (no. 2), $a = 8.7904(7)$ Å, $b = 12.0808(11)$ Å, $c = 14.3999(11)$ Å, $\alpha = 68.001(8)^\circ$, $\beta = 81.114(6)^\circ$, $\gamma = 83.933(7)^\circ$, $Z = 4$, $V = 1398.9(2)$ Å³, $R_{\text{gt}}(F) = 0.0658$, $wR_{\text{ref}}(F^2) = 0.1827$, $T = 290.83(10)$ K.

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The asymmetric unit of the title 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

All solvents and other reagents were of analytical grade and the title compound was prepared by modification of the reported method [1, 2]. To an ethanol solution (5 mL) of 2-hydroxy-4-methoxy benzaldehyde (456.5 mg, 3 mmol)

Table 1: Data collection and handling.

Crystal:	Clear dark yellow block
Size:	0.17 × 0.15 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ^{−1}
Diffraction, scan mode:	SuperNova, Dual, ω
θ_{max} , completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9288, 5496, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3013
$N(\text{param})_{\text{refined}}$:	385
Programs:	CrysAlis ^{PRO} [18], SHELX [19], Olex2 [20]

was added an ethanol solution (5 mL) of 1-(4-aminophenyl)-ethanone oxime (450.6 mg, 1 mmol). The solution had been stirred at 328 K for 7 h, then the mixture was filtered at room temperature. The product was dried under vacuum and a yellow solid of title compound was obtained (yield 81.3%, m.p. 392–397 K). The crystals were obtained by slow evaporation from a mixture of chloroform/ethanol (1:1) solution in a quiet environment at room temperature. Elemental analysis: Anal. calcd. for C₁₆H₁₆N₂O₃: C, 67.59%; H, 5.67%; N, 9.85%; Found: C, 67.61%; H, 5.62%; N, 9.81%.

Experimental details

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

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.6910(2)	0.46356(17)	−0.12420(14)	0.0519(6)
O2	0.4414(2)	0.84437(19)	−0.28942(15)	0.0603(6)
O3	1.2073(3)	0.0758(2)	0.51307(15)	0.0667(7)
H3	1.1986	0.0978	0.5611	0.100*
N1	0.7900(3)	0.4301(2)	0.04568(16)	0.0457(6)
H1	0.7783	0.4023	0.0004	0.055*
N2	1.1228(3)	0.1565(2)	0.43782(17)	0.0529(7)
C1	0.6387(3)	0.5701(3)	−0.1284(2)	0.0434(7)
C2	0.5637(3)	0.6489(2)	−0.2105(2)	0.0447(7)
H2	0.5504	0.6246	−0.2627	0.054*
C3	0.5107(3)	0.7607(3)	−0.2132(2)	0.0450(7)
C4	0.5247(3)	0.7990(3)	−0.1336(2)	0.0530(8)
H4	0.4870	0.8748	−0.1362	0.064*
C5	0.5930(3)	0.7245(3)	−0.0539(2)	0.0518(8)
H5	0.6001	0.7495	−0.0010	0.062*
C6	0.6543(3)	0.6096(2)	−0.0482(2)	0.0426(7)
C7	0.7293(3)	0.5368(3)	0.0349(2)	0.0457(7)
H7	0.7361	0.5663	0.0851	0.055*
C8	0.8733(3)	0.3560(3)	0.1249(2)	0.0417(7)
C9	0.8887(3)	0.2343(3)	0.1446(2)	0.0475(7)
H9	0.8437	0.2031	0.1059	0.057*
C10	0.9701(3)	0.1587(3)	0.2210(2)	0.0486(8)
H10	0.9797	0.0773	0.2325	0.058*
C11	1.0375(3)	0.2016(2)	0.28081(19)	0.0400(7)
C12	1.0234(3)	0.3246(3)	0.2589(2)	0.0470(7)
H12	1.0696	0.3562	0.2968	0.056*
C13	0.9431(3)	0.4003(3)	0.1830(2)	0.0477(8)
H13	0.9355	0.4819	0.1703	0.057*
C14	0.4177(4)	0.8153(3)	−0.3731(3)	0.0770(11)
H14A	0.3644	0.8815	−0.4194	0.115*
H14B	0.3572	0.7462	−0.3496	0.115*
H14C	0.5156	0.7987	−0.4066	0.115*
C15	1.1218(3)	0.1217(3)	0.3641(2)	0.0446(7)
C16	1.1989(4)	0.0053(3)	0.3617(2)	0.0589(9)
H16A	1.2974	−0.0061	0.3859	0.088*
H16B	1.2130	0.0072	0.2937	0.088*
H16C	1.1355	−0.0593	0.4040	0.088*
O4	1.1123(2)	0.16546(18)	0.65708(14)	0.0587(6)
O5	1.2851(2)	−0.0209(2)	0.97293(16)	0.0668(7)
O6	0.4308(3)	0.68016(19)	0.19696(16)	0.0620(6)
H6	0.4029	0.6425	0.1661	0.093*
N3	0.9266(3)	0.3468(2)	0.62452(17)	0.0444(6)
H3A	0.9827	0.2939	0.6062	0.053*
N4	0.5314(3)	0.6069(2)	0.26477(17)	0.0507(7)
C17	1.1143(3)	0.1635(2)	0.7479(2)	0.0416(7)
C18	1.1988(3)	0.0748(3)	0.8172(2)	0.0479(8)
H18	1.2568	0.0166	0.7974	0.057*
C19	1.1983(3)	0.0713(3)	0.9132(2)	0.0460(7)
C20	1.1126(3)	0.1575(3)	0.9463(2)	0.0518(8)
H20	1.1127	0.1551	1.0116	0.062*
C21	1.0290(3)	0.2450(3)	0.8799(2)	0.0489(8)
H21	0.9717	0.3022	0.9014	0.059*
C22	1.0260(3)	0.2522(2)	0.7804(2)	0.0399(7)
C23	0.9345(3)	0.3415(3)	0.7155(2)	0.0442(7)
H23	0.8784	0.3982	0.7382	0.053*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C24	0.8375(3)	0.4281(3)	0.5522(2)	0.0399(7)
C25	0.8260(3)	0.4011(3)	0.4678(2)	0.0493(8)
H25	0.8784	0.3334	0.4603	0.059*
C26	0.7374(3)	0.4744(3)	0.3955(2)	0.0491(8)
H26	0.7298	0.4548	0.3398	0.059*
C28	0.6762(3)	0.6056(3)	0.4867(2)	0.0467(7)
H28	0.6284	0.6754	0.4928	0.056*
C29	0.7640(3)	0.5313(3)	0.5604(2)	0.0481(7)
H29	0.7735	0.5512	0.6156	0.058*
C27	0.6584(3)	0.5775(2)	0.4038(2)	0.0406(7)
C30	0.5571(3)	0.6521(3)	0.3282(2)	0.0432(7)
C31	0.4851(3)	0.7692(3)	0.3310(2)	0.0574(9)
H31A	0.4615	0.8196	0.2648	0.086*
H31B	0.5557	0.8076	0.3524	0.086*
H31C	0.3920	0.7555	0.3775	0.086*
C32	1.2793(4)	−0.0378(3)	1.0766(2)	0.0721(11)
H32A	1.3206	0.0296	1.0822	0.108*
H32B	1.3392	−0.1092	1.1098	0.108*
H32C	1.1743	−0.0453	1.1078	0.108*

Comment

The structures of many compounds with oxime-type ligands have been reported, often with di, tri- or polynuclear structures [3–5]. Oximes are an important class of ligands with interesting properties due to the presence of several coordination sites [6–8]. Researchers have focused their interests on complexes, for their extensive applications [9–11]. For example: it has been used to nonlinear optical materials [12–14] and supramolecular architectures [15–17] etc.

The single crystal structure of the title compound was determined by X-ray crystallography. The asymmetric unit of the title structure contains two crystallographically independent but chemically identical molecules (*cf.* the figure). And all bond lengths are in normal ranges. In the molecules, there are strong intramolecular N–H···O hydrogen bonds (O1–H1···N1: *d*(N1···O1) = 2.598 Å, *d*(N1–H1) = 0.86 Å and *d*(H1···O1) = 1.915 Å; N3–H3A···O4: *d*(N3···O4) = 2.529 Å, *d*(N3–H3A) = 0.86 Å and *d*(H3A···O4) = 1.810 Å). In addition, there are strong intermolecular O3–H3···O4, O6–H6···O1 hydrogen bonds formed a chain structure.

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