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Crystal structure of (*E*)-1-{4-[(4-fluoro-2-hydroxybenzylidene)amino]phenyl}ethanone *O*-methyl oxime, C₁₆H₁₅FN₂O₂

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Abstract

C₁₆H₁₅FN₂O₂, monoclinic, *P*₂₁/*c* (no. 14), *a* = 6.0573(3) Å, *b* = 13.8341(6) Å, *c* = 33.0876(15) Å, β = 92.837(2)°, *Z* = 8, *V* = 2769.3(2) Å³, *R*_{gt}(*F*) = 0.0581, *wR*_{ref}(*F*²) = 0.1605, *T* = 173(2) K.

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The asymmetric unit of the title structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.19 × 0.17 × 0.14 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	19662, 4869, 0.063
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3089
<i>N</i> (<i>param</i>) _{refined} :	385
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Source of material

All chemicals were used without further purification. 1-(4-Aminophenyl)ethan-1-one *O*-methyl oxime was prepared by a method reported earlier [4, 5]. 4-Fluoro-2-hydroxybenzaldehyde (140.2 mg, 1 mmol) dissolved in EtOH (5 mL) was added to 1-(4-aminophenyl)ethanone *O*-methyl oxime (164.5 mg, 1 mmol) dissolved in EtOH (5 mL). After being stirred for 2 h at room temperature, the filtrate was allowed to stand for 21 days in a quiet environment. Several block clear yellow crystals were obtained (yield 71.4%, m.p. 374–382 K). **Anal. calcd. for C₁₆H₁₅FN₂O₂:** C, 67.12%; H, 5.28%; N, 9.78%; Found: C, 67.84%; H, 5.62%; N, 9.31%.

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}
F1	−0.0087(3)	0.12484(14)	0.50935(5)	0.0667(6)
F2	−0.4769(3)	−0.13346(13)	0.43786(5)	0.0600(5)
O1	−0.2008(3)	0.17686(15)	0.37224(5)	0.0473(5)
H1	−0.146962	0.172605	0.349388	0.071*
O2	0.1567(3)	0.15842(13)	0.08796(5)	0.0407(5)
O3	−0.6599(3)	−0.15860(16)	0.29901(5)	0.0502(6)
H3	−0.600291	−0.155096	0.276662	0.075*
O4	−0.2765(3)	−0.12463(18)	0.01773(5)	0.0592(6)
N1	0.0895(3)	0.13760(14)	0.32026(6)	0.0323(5)
N2	0.1054(4)	0.16241(15)	0.12901(6)	0.0352(5)
N3	−0.3579(3)	−0.12255(14)	0.24933(6)	0.0323(5)
N4	−0.3284(4)	−0.13147(19)	0.05872(6)	0.0473(6)
C1	−0.0477(5)	0.15035(18)	0.40104(8)	0.0366(6)
C2	−0.1042(5)	0.15280(19)	0.44138(8)	0.0417(7)
H2	−0.245997	0.174142	0.448583	0.050*
C3	0.0502(5)	0.1237(2)	0.47011(8)	0.0455(7)
C4	0.2601(5)	0.0938(2)	0.46232(8)	0.0477(8)
H4	0.362685	0.074359	0.483420	0.057*
C5	0.3146(5)	0.0933(2)	0.42240(8)	0.0415(7)
H5	0.459215	0.074089	0.415948	0.050*
C6	0.1640(4)	0.12031(18)	0.39108(7)	0.0332(6)
C7	0.2281(4)	0.11814(18)	0.34963(7)	0.0342(6)
H7	0.375936	0.101974	0.344029	0.041*
C8	0.1481(4)	0.13528(17)	0.27930(7)	0.0310(6)
C9	0.3459(5)	0.1001(2)	0.26563(8)	0.0404(7)
H9	0.454564	0.074736	0.284417	0.048*
C10	0.3856(4)	0.10185(19)	0.22475(8)	0.0387(7)
H10	0.522422	0.078087	0.216015	0.046*
C11	0.2305(4)	0.13740(17)	0.19622(7)	0.0296(6)
C12	0.0329(4)	0.17298(19)	0.21016(7)	0.0349(6)
H12	−0.076051	0.198458	0.191448	0.042*
C13	−0.0060(4)	0.17155(18)	0.25087(7)	0.0330(6)
H13	−0.142079	0.196061	0.259666	0.040*
C14	0.2703(4)	0.13629(17)	0.15238(7)	0.0312(6)
C15	0.4872(5)	0.1034(2)	0.13741(8)	0.0423(7)
H15A	0.515089	0.036525	0.145935	0.063*
H15B	0.605642	0.145117	0.148694	0.063*
H15C	0.482681	0.107172	0.107803	0.063*
C16	−0.0404(5)	0.1772(2)	0.06377(8)	0.0526(8)
H16A	−0.007535	0.175041	0.035078	0.079*
H16B	−0.097106	0.241395	0.070342	0.079*
H16C	−0.151713	0.128214	0.069283	0.079*
C17	−0.5103(4)	−0.13684(19)	0.32899(8)	0.0353(6)
C18	−0.5697(5)	−0.1462(2)	0.36885(8)	0.0418(7)
H18	−0.713108	−0.167975	0.374882	0.050*
C19	−0.4177(5)	−0.1235(2)	0.39901(8)	0.0411(7)
C20	−0.2077(5)	−0.09074(19)	0.39253(8)	0.0432(7)
H20	−0.107172	−0.074325	0.414402	0.052*
C21	−0.1499(5)	−0.08285(19)	0.35293(8)	0.0381(7)
H21	−0.005342	−0.061242	0.347588	0.046*
C22	−0.2958(4)	−0.10540(17)	0.32041(7)	0.0310(6)
C23	−0.2254(4)	−0.10107(17)	0.27955(7)	0.0332(6)
H23	−0.078258	−0.081972	0.274815	0.040*
C24	−0.2936(4)	−0.12098(17)	0.20884(7)	0.0302(6)
C25	−0.0934(4)	−0.08463(19)	0.19611(8)	0.0373(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H25	0.013033	−0.059566	0.215398	0.045*
C26	−0.0498(4)	−0.08507(18)	0.15538(7)	0.0359(6)
H26	0.087835	−0.060800	0.147215	0.043*
C27	−0.2021(4)	−0.12009(17)	0.12618(7)	0.0319(6)
C28	−0.4003(4)	−0.15880(18)	0.13945(7)	0.0332(6)
H28	−0.505579	−0.185420	0.120327	0.040*
C29	−0.4433(4)	−0.15852(18)	0.17973(7)	0.0330(6)
H29	−0.578928	−0.184660	0.187973	0.040*
C30	−0.1600(4)	−0.11467(18)	0.08251(7)	0.0334(6)
C31	0.0639(5)	−0.0896(2)	0.06844(8)	0.0448(7)
H31A	0.061275	−0.091949	0.038821	0.067*
H31B	0.172709	−0.135981	0.079646	0.067*
H31C	0.104235	−0.024292	0.077648	0.067*
C32	−0.4733(6)	−0.1422(3)	−0.00665(9)	0.0754(12)
H32A	−0.582915	−0.092238	−0.001380	0.113*
H32B	−0.532949	−0.205816	−0.000027	0.113*
H32C	−0.439331	−0.140666	−0.035294	0.113*

Experimental details

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

Comment

More and more attention is paid to the synthesis of mononuclear or multinuclear transition metal complexes with Schiff base ligands. Since the Schiff base ligand has excellent coordination ability with transition metal, it forms a stable transition metal complex [5–9]. So, we predict that the title compound is an excellent Schiff base ligand [10].

The current work reports the synthesis of title compound, which is built up by two C₁₆H₁₅FN₂O₂ molecules in the asymmetric unit. Compared with the structure already reported, all bond lengths of the title compound are in normal ranges [10–13]. There is one strong intramolecular hydrogen bond in each of the two crystallographically independent molecules (see the figure): O1–H1···N1 (*d*(N1···H1) = 1.831 Å, *d*(O1–H1) = 0.84 Å and *d*(O1···N1) = 2.578 Å) and O3–H3···N3 (*d*(N3···H3) = 1.819 Å, *d*(O3–H3) = 0.84 Å and *d*(O3···N3) = 2.568 Å).

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.

4. Kaczmarek, M. T.; Kubicki, M.: The trimorphic structure of *N,N'*-bis(5-methylsalicylidene)-4-methyl-1,3-phenylenediamine. *Acta Crystallogr.* **B70** (2014) 792–800.
5. Dong, W. K.; Gong, S. S.; Tong, J. F.; Sun, Y. X.; Wu, J. C.: Syntheses and structure of two copper(II) complexes with salicyl mono-oxime ligands. *Chin. J. Inorg. Chem.* **26** (2010) 1868–1874.
6. Li, Q. L.; Li, P. P.; Ma, J. X.; Zhao, J. X.; Zhao, L.: Crystal structure of bis{2-[(*E*)-[(4-[(*E*)-1-(methoxyimino)ethyl)phenyl]imino)methyl]phenolato- $\kappa^2 N, O$ }zinc(II), $C_{32}H_{30}N_4O_4Zn$. *Z. Kristallogr. NCS* **233** (2018) 637–639.
7. Sun, Y. X.; Zhao, Y. Y.; Li, C. Y.; Yu, B.; Guo, J. Q.; Li, J.: Supramolecular cobalt(II) and copper(II) complexes with Schiff base ligand: syntheses, characterizations and crystal structures. *Chin. J. Inorg. Chem.* **32** (2016) 913–920.
8. Sun, Y. X.; Li, C. Y.; Yang, C. J.; Zhao, Y. Y.; Guo, J. Q.; Yu, B.: Two Cu(II) complexes with Schiff base ligands: synthesis, crystal structure, spectroscopic properties, and substituent effect. *Chin. J. Inorg. Chem.* **32** (2016) 327–335.
9. Ma, J. X.; Li, Q. L.; Li, P. P.; Zhao, J. X.; Zhao, L.: Synthesis and crystal structure of bis{[(*E*)-[(4-[(*E*)-1-(methoxyimino)ethyl)phenyl]imino)methyl]-2-naphtholato- $\kappa^2 N, O$ }nickel(II), $C_{40}H_{34}N_4NiO_4$. *Z. Kristallogr. NCS* **233** (2018) 789–791.
10. Zhao, L.; Dong, X. T.; Cheng, Q.; Zhao, J. X.; Wang, L.: Synthesis, crystal structure and spectral properties of a 2D supramolecular copper(II) complex with 1-(4-[(*E*)-3-ethoxyl-2-hydroxybenzylidene]amino)phenyl ethanone oxime. *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **43** (2013) 1241–1246.
11. Sun, Y. X.; Lu, R. E.; Li, X. R.; Zhao, Y. Y.; Li, C. Y.: A Schiff base ligand containing oxime group and its Cu(II) complex: syntheses and supramolecular structures. *Chin. J. Inorg. Chem.* **31** (2015) 1055–1062.
12. Ma, J. X.; Li, Q. L.; Li, P. P.; Zhao, J. X.; Zhao, L.: Synthesis and crystal structure of (*E*)-1-(4-[(*E*)-5-chloro-2-hydroxybenzylidene]amino)phenyl)ethan-1-one *O*-methyl oxime, $C_{16}H_{15}ClN_2O_2$. *Z. Kristallogr. NCS* **233** (2018) 463–464.
13. Zhao, L.; Wang, F.; An, Q. Q.; Zhao, J. X.: Crystal structure of (*E*)-1-(4-[(*E*)-3,5-dichloro-2-hydroxybenzylidene]amino)phenyl)ethan-1-one *O*-ethyl oxime, $C_{17}H_{16}Cl_2N_2O_2$. *Z. Kristallogr. NCS* **231** (2016) 1045–1046.