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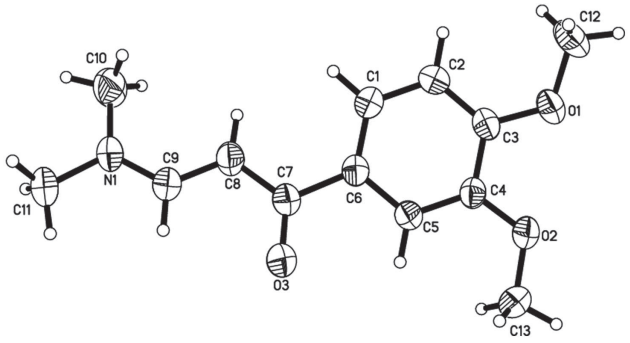
Crystal structure of (*E*)-1-(3,4-dimethoxyphenyl)-3-(dimethylamino)prop-2-en-1-one, $C_{13}H_{17}NO_3$



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Abstract

$C_{13}H_{17}NO_3$, monoclinic, $P2_1/c$, $a = 7.7486(4)$ Å, $b = 8.5148(3)$ Å, $c = 19.9822(9)$ Å, $\beta = 100.341(2)°$, $V = 1296.97(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0620$, $wR_{ref}(F^2) = 0.1814$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow, block, size 0.368×0.451×0.578 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 venture, φ and ω scans
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18633, 2952
$N(param)_{refined}$:	158
Programs:	SHELX [11], Bruker programs [12]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*e*	0.5533	0.0784	0.6997	0.063
H(2A)	4*e*	0.6537	0.0458	0.8158	0.066
H(5A)	4*e*	0.7660	0.5123	0.7096	0.055
H(8A)	4*e*	0.4080	0.1519	0.6006	0.067
H(9A)	4*e*	0.4915	0.3267	0.4915	0.073
H(10A)	4*e*	0.1492	0.0871	0.5231	0.126
H(10B)	4*e*	0.2818	−0.0431	0.5044	0.126
H(10C)	4*e*	0.1189	0.0126	0.4484	0.126
H(11A)	4*e*	0.3736	0.2797	0.3845	0.117
H(11B)	4*e*	0.1717	0.2295	0.3755	0.117
H(11C)	4*e*	0.3182	0.1002	0.3698	0.117
H(12A)	4*e*	0.8755	0.1188	0.9822	0.139
H(12B)	4*e*	0.9140	0.0373	0.9142	0.139
H(12C)	4*e*	0.7162	0.0584	0.9258	0.139
H(13A)	4*e*	0.9710	0.7333	0.8475	0.105
H(13B)	4*e*	0.8010	0.7091	0.7899	0.105
H(13C)	4*e*	0.9872	0.6437	0.7785	0.105

Source of material

The most important and straight forward method for the synthesis of the title compound involves the direct condensation of β-dicarbonyl compounds with amines at reflux in an aromatic solvent with azeotropic removal of water [8, 9].

To a solution of 3,4-dimethoxyacetophenone (1.80 g, 0.01 mol) in dimethylformamide-dimethylacetal (1.91 g, 0.015 mol) in dry xylene (20 mL) was refluxed for 24 h. The obtained solid was collected by filteration and recrystallized from dioxane to give the title compound.

Yield, 66%; m.p. 123.7 °C. IR (KBr, cm^{-1}): 3080 (CH arom.), 2997, 2931, 2839 (CH aliph.), 1635 (C = O). ^{1}H-NMR

Table 3: Atomic coordinates and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*	0.8252(2)	0.2547(2)	0.90088(7)	0.098(1)	0.068(1)	0.0404(8)	−0.0044(8)	−0.0068(7)	0.0090(7)
O(2)	4*e*	0.8774(2)	0.5170(2)	0.84231(7)	0.079(1)	0.0573(9)	0.0449(8)	−0.0114(7)	−0.0071(7)	−0.0030(6)
O(3)	4*e*	0.6798(4)	0.4273(3)	0.59200(8)	0.192(2)	0.108(2)	0.0440(9)	−0.083(2)	−0.003(1)	0.0047(9)
N(1)	4*e*	0.3138(2)	0.1676(2)	0.46542(8)	0.069(1)	0.068(1)	0.0407(9)	−0.0067(9)	−0.0034(8)	−0.0020(8)
C(1)	4*e*	0.6169(3)	0.1607(2)	0.7252(1)	0.056(1)	0.052(1)	0.048(1)	−0.0064(9)	0.0034(9)	−0.0043(9)
C(2)	4*e*	0.6753(3)	0.1417(2)	0.7944(1)	0.063(1)	0.050(1)	0.050(1)	−0.0051(9)	0.0071(9)	0.0075(9)
C(3)	4*e*	0.7645(3)	0.2610(2)	0.83254(9)	0.054(1)	0.056(1)	0.0391(9)	0.0032(9)	0.0033(8)	0.0044(8)
C(4)	4*e*	0.7947(2)	0.4030(2)	0.80069(9)	0.043(1)	0.048(1)	0.0416(9)	0.0020(8)	0.0000(7)	−0.0027(8)
C(5)	4*e*	0.7409(2)	0.4181(2)	0.73148(9)	0.051(1)	0.045(1)	0.042(1)	0.0003(8)	0.0042(8)	0.0023(8)
C(6)	4*e*	0.6499(2)	0.2976(2)	0.69275(9)	0.047(1)	0.050(1)	0.0400(9)	0.0007(8)	0.0035(8)	−0.0021(8)
C(7)	4*e*	0.6009(3)	0.3230(3)	0.6174(1)	0.086(2)	0.057(1)	0.040(1)	−0.011(1)	0.004(1)	−0.0011(9)
C(8)	4*e*	0.4683(3)	0.2278(3)	0.5787(1)	0.060(1)	0.061(1)	0.043(1)	−0.000(1)	0.0005(9)	−0.0015(9)
C(9)	4*e*	0.4286(3)	0.2466(3)	0.5099(1)	0.075(1)	0.059(1)	0.046(1)	−0.008(1)	0.003(1)	−0.0040(9)
C(10)	4*e*	0.2075(4)	0.0465(4)	0.4870(1)	0.071(2)	0.108(2)	0.065(2)	−0.028(2)	−0.007(1)	0.009(1)
C(11)	4*e*	0.2926(4)	0.1966(3)	0.3930(1)	0.102(2)	0.085(2)	0.041(1)	−0.012(2)	0.000(1)	−0.006(1)
C(12)	4*e*	0.8334(5)	0.1057(3)	0.9333(1)	0.135(3)	0.081(2)	0.055(1)	0.003(2)	−0.003(2)	0.026(1)
C(13)	4*e*	0.9118(3)	0.6620(3)	0.8122(1)	0.081(2)	0.062(1)	0.064(1)	−0.022(1)	0.003(1)	−0.003(1)

(DMSO-d_2): 3.1 [s, 6H, $2NCH_3$], 3.8 [s, 6H, $2OCH_3$], 5.8, 6.9 [2d, 2H, CH = CH, J = 7.8 Hz], 7.4–7.6 [m, 3H, Ar—H]. ^{13}C-NMR (DMSO-d_6): 44.8(2), 56.0(2), 91.1, 110.8, 111.1, 121.2, 133.4, 148.7, 151.7, 154.1, 185.3. MS m/z (%): 235 (M^+) (12.73), 136 (100) Anal. Calcd. For $C_{13}H_{17}NO_3$ (235): C, 66.36; H, 7.28; N, 5.95. Found: C, 66.07; H, 7.54; N, 6.23.

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT and SHELXS-97 [10, 11]. All hydrogen atoms were idealized and refined using a riding model with the help of of the SHELX-2008 program (AFIX43 or AFIX 137 option) [10].

Discussion

Enaminones or enamine of β-dicarbonyl compounds and their chemistry has been reviewed [1, 2]. These enaminones have demonstrated a potential as multipurpose synthetic intermediates in organic synthesis [3, 4], in the pharmaceutical development [4–7] and in heterocyclic synthesis [4].

The aim of this work was to design a novel *E*-1-(3,4-dimethxyphenyl)-3-(dimethylamino)prop-2-en-1-one having a biologically active 3,4-dimethoxyphenyl moiety.

The crystal structure of the title compound contains one molecule in the asymmetric unit. In the title molecule C8—C9 bond length is 1.363(3) Å and this is typical C—C double bond. In the crystal structure the molecules are stabilized by two intermolecular non-classical hydrogen bonds, of which O1 and O3 work as hydrogen bond acceptors and C10 and C11 work as hydrogen bond donors. The distance of the interactions between C10—H10C···O2 is 2.58(2) Å and C11—H11A···O3 is 2.59 Å and the angles are 168° and 122°, respectively.

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References

1. Greenhill, J. V.: Enaminones. Chem. Soc. Rev. **6** (1977) 277–294.
2. Elassar, A. Z. A.; El-Khair, A. A.: Recent developments in the chemistry of enaminones. Tetrahedron **59** (2003) 8463–8480.
3. Huang, Y.; Hartmann, R. W.: The improved preparation of 7,8-dihydroquinoline-5(6*H*)-one and 6,7-dihydro-5*H*-1-pyrindin-5-one. Synth. Commun. **28** (1998) 1197–1200.
4. Baraldi, P. G.; Simoni, D.; Manfredini, S.: An improved reparation of enaminones from 1,3-diketones and ammonium acetate or amine acetates. Synthesis **11** (1983) 902–903.
5. Cartaya-Marin, C. P.; Henderson, D. G.; Soeder, R. W.: Synthesis of enaminones using trimethylsilyl trifluoromethane sulfonate as an activator. Synth. Commun. **27** (1997) 4275–4283.
6. Edafiogho, I. O.; Alexander, M. S.; Moore, J. A.; Farrar, V. A.; Scott, K. R.: Anticonvulsant enaminones: With emphasis on methyl 4-[(p-chlorophenyl)amino]-6-methyl-2-oxocyclohex-3-en-1-oate (ADD 196022). Curr. Med. Chem. **1** (1994) 159–175.
7. Eddington, N. D.; Cox, D. S.; Roberts, R. R.; Stables, J. P.; Powell, C. B.; Scott, K. R.: Enaminones versatile the rapeutic pharmacophores. Further advances. Curr. Med. Chem. **7** (2000) 417–436.
8. Govindh, B.; Diwakar, B. S. D.; Murthy, Y. L. N.: A brief review on synthesis and applications of β-enamino carbonyl compounds. Org. Commun. **5** (2012) 105–119.
9. Martin, D. F.; Janusonis, G. A.; Martin, B. B.: Stabilities of bivalent metal complexes of some β-ketoimines. J. Am. Chem. Soc. **83** (1961) 73–75.
10. Baraldi, P. G.; Simoni, D.; Manfredini, S.: An improved preparation of enaminones from 1,3-diketones and ammonium acetate or amine acetates. Synthesis **11** (1983) 902–903.
11. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
12. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA, 2009.