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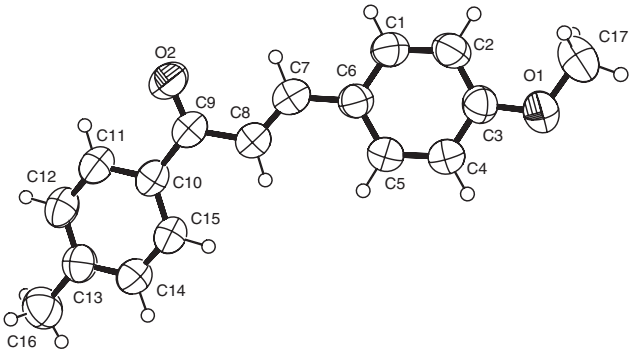
Yavuz Köysal*, Hakan Bülbül, Funda Özdemir, İlhan Özer İlhan and Necmi Dege

Crystal structure of 3-(4-methoxyphenyl)-1-(4-methylphenyl)prop-2-en-1-one, $C_{17}H_{16}O_2$



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Abstract

$C_{17}H_{16}O_2$, monoclinic, $P2_1/c$ (no. 14), $a = 11.6963(12)$ Å, $b = 11.1187(8)$ Å, $c = 11.6902(10)$ Å, $\beta = 115.545(7)°$, $V = 1371.7(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0350$, $wR_{ref}(F^2) = 0.0865$, $T = 293$ K.

CCDC no.: 1515321

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

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.36 × 0.27 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	STOE IPDS 2, ω scans
$2\theta_{max}$, completeness:	52°, 98.8%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7194, 2661, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1375
$N(param)_{refined}$:	236
Programs:	Stoe programs [1], SHELX [2, 3], ORTEP [4]

Source of material

Equimolar quanities of *p*-methylacetophenone (0.01 mol) and *p*-methoxybenzaldehyde (0.01 mol) were dissolved in ethanol (10 mL) under stirring and aq. NaOH (30%) was added dropwise. The reaction mixture was stirred at room temperature for a few hours. The reaction mixture was diluted with water & acidifed with HCl. The separated solid was filtered and recrystallised from ethanol [5].

IR (ATR) cm^{-1}: 2932 (aromatic—CH), 2837 (aliphatic—CH), 1651 (C = O), 1593–1416 (C = C); **^{1}H-NMR** (δ): 8.00–6.94 (—CH), 2.41 (—CH_3), 3.91 (—OCH_3); **^{13}C-NMR** (δ): 21.54 (—CH_3), 55.51 (—OCH_3), 188 (C = O), 163 (HC = CH), 144–120 (aromatic—CH_3), 113.81–113.75 (aliphatic—CH). **Anal.** Calcd. for $C_{17}H_{16}O_2$: C, 80.95; H, 6.34; Found: C, 80.63; H, 6.07.

Experimental details

The structure was solved by direct methods and refined by full-matrix least-square techniques. All the H atoms in the molecular structure were found and located in difference Fourier map and refined freely. H bond lengths vary between 0.952(14) Å and 0.969(15) Å for aromatic rings.

Comment

Chalcones consist of two aromatic rings which are linked by three carbon atoms with a α,β-unsaturated carbonyl system. Chalcones and their derivatives are interesting materials for their biological activities such as anti-viral, anti-malarial, anti-cancer, antioxidant, anti-inflammatory, antifungal, antileishmanial, anti-tubercular, anti-hyperglycemic properties [6–11]. They are also used in different applications such as agriculture [12], cosmetics and manufacture of pesticides [13].

The title compund, $C_{17}H_{16}O_2$, shows *E* conformation with respect to the C = C double bond (C7 = C8 = 1.318(2) Å). In the molecular structure, the methoxy substituted phenyl

***Corresponding author: Yavuz Köysal,** Yesilyurt Demir Celik Vocational School, Ondokuz Mayıs University, TR-55139, Samsun, Turkey, e-mail: yavuzk@omu.edu.tr
Hakan Bülbül and Necmi Dege: Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139, Samsun, Turkey
Funda Özdemir and İlhan Özer İlhan: Department of Chemistry, Faculty of Sciences, Erciyes University, 38039, Kayseri, Turkey

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.36180(15)	0.32004(16)	0.61559(16)	0.0787(4)
C2	0.43587(15)	0.27272(15)	0.73366(16)	0.0788(4)
C3	0.41923(13)	0.15512(14)	0.75852(14)	0.0729(4)
C4	0.32929(16)	0.08557(15)	0.66461(16)	0.0814(5)
C5	0.25615(15)	0.13408(14)	0.54835(16)	0.0749(4)
C6	0.27015(13)	0.25269(13)	0.52017(13)	0.0660(4)
C7	0.19122(15)	0.30866(15)	0.39781(15)	0.0740(4)
C8	0.10143(15)	0.25798(15)	0.29722(15)	0.0734(4)
C9	0.02624(13)	0.32677(13)	0.18103(13)	0.0704(4)
C10	−0.05096(13)	0.26101(12)	0.06163(13)	0.0662(4)
C11	−0.10413(15)	0.32522(16)	−0.05156(16)	0.0763(4)
C12	−0.17460(16)	0.26820(16)	−0.16466(16)	0.0798(5)
C13	−0.19631(13)	0.14563(15)	−0.17099(14)	0.0730(4)
C14	−0.14285(15)	0.08265(16)	−0.05865(16)	0.0779(4)
C15	−0.07184(14)	0.13783(13)	0.05549(17)	0.0746(4)
C16	−0.2733(2)	0.0836(2)	−0.2941(2)	0.0986(6)
C17	0.5723(2)	0.1671(2)	0.9754(2)	0.0991(6)
O1	0.48553(10)	0.09799(10)	0.87164(10)	0.0952(4)
O2	0.02810(11)	0.43693(9)	0.18209(9)	0.0926(4)
H1	0.3737(14)	0.4032(15)	0.5996(13)	0.093(5)*
H2	0.4969(14)	0.3226(13)	0.7989(14)	0.091(5)*
H4	0.3196(15)	0.0037(15)	0.6841(14)	0.104(5)*
H5	0.1935(13)	0.0848(12)	0.4841(13)	0.081(4)*
H7	0.2070(13)	0.3943(13)	0.3907(12)	0.085(4)*
H8	0.0821(13)	0.1756(14)	0.2962(12)	0.082(4)*
H11	−0.0863(14)	0.4102(15)	−0.0479(14)	0.097(5)*
H12	−0.2063(13)	0.3136(13)	−0.2413(14)	0.085(5)*
H14	−0.1552(13)	−0.0036(14)	−0.0611(13)	0.092(5)*
H15	−0.0377(13)	0.0903(13)	0.1305(13)	0.081(4)*
H17A	0.6045(19)	0.1107(18)	1.048(2)	0.143(8)*
H17B	0.6404(18)	0.2026(17)	0.9546(16)	0.129(7)*
H17C	0.5260(16)	0.2375(17)	0.9895(14)	0.113(6)*
H18A	−0.362(2)	0.100(2)	−0.325(2)	0.173(10)*
H18B	−0.267(2)	−0.001(2)	−0.290(2)	0.159(9)*
H18C	−0.251(2)	0.1087(19)	−0.361(2)	0.159(9)*

ring and the methyl substituted phenyl ring are inclined by 13.78(6)°. The maximum deviations within the ring systems are 0.0052(2) Å for C4 atom of the C1—C6 ring and 0.0033(2) Å for atom C12 of the C10—C15 ring. The attached methyl group is co-planar with the related aromatic ring system (C10—C15). The methoxy group is slightly twisted with the C4—C3—O1—C17 and C2—C3—O1—C17 torsion angles of −174.97(16)° and 5.0(2)°, respectively. In the molecular structure, the bond lengths and angles are consistent with the related structures [14, 15]. The crystal structure presents at least one very weak intermolecular hydrogen bond, namely C11—H11$\cdots$O2′ (with symmetry code: $-x$, $-y+1$, $-z$).

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References

1. Stoe&Cie. X-AREA (Version 1.18) and X-RED32 (Version1.04). Stoe&Cie, Darmstadt, Germany, 2002.
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Farrugia, L. J.: WinGX and ORTEP for Windows: an update. J. Appl. Cryst. **45** (2012) 849–854.
5. Jadhav, S. A.; Kulkarni, K. M.; Patil, P. B.; Dhole, V. R.; Patil, S. S.: Design, synthesis and biological evaluation of some novel pyrazoline derivatives. Der Pharma Chemica **8** (2016) 38–45.
6. Arias-Ruiz, S. N.; Romero, N.; Lobato-García, C. E.; Gómez-Rivera, A.; Mendoza, A.: Second monoclinic form of (*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one. Acta Crystallogr. **E69** (2013) o1694–o1695.
7. Dimmock, J. R.; Elias, D. W.; Beazely, M. A.; Kandepu, N. M.: Bioactivities of chalcones. Curr. Med. Chem. **6** (1999) 1125–1149.
8. Nowakowska, Z.: A review of anti-infective and anti-inflammatory chalcones. Eur. J. Med. Chem. **42** (2007) 125–137.
9. Zhang, X.-W.; Zhao, D.-H.; Quan, Y.-C.; Sun, L.-P.; Yin, X.-M.; Guan, L.-P.: Synthesis and evaluation of antiinflammatory activity of substituted chalcone derivatives. Med. Chem. Res. **19** (2010) 403–412.
10. Kouskoura, M.; Hadjipavlou-Litina, D.; Giakoumakou, M.: Synthesis and anti-inflammatory activity of chalcones and related mannich bases. Med.Chem. **4** (2008) 586–596.
11. Doan, T. N.; Tran, D. T.: Synthesis, antioxidant and antimicrobial activities of a novel series of chalcones, pyrazolic chalcones, and allylic chalcones. Pharmacol. Pharmacy **2** (2011) 282–288.
12. Pasquale, G.; Romanelli, G. P.; Autino, J. C.; García, J.; Ortiz, E. V.; Duchowicz, P. R.; Agric, J.: Quantitative structure-activity relationships of mosquito larvicidal chalcone derivatives. J. Agric. Food Chem. **60** (2012) 692–697.
13. Bhavana, P.; Sarveswari, S.; Weng Ng, S.; Tiekink, E. R. T.: Efficient ultrasound-assisted synthesis, spectroscopic, crystallographic and biological investigations of pyrazole-appended quinolinyl chalcones. J. Mol. Struct. **1081** (2015) 201–210.
14. Ahn, S.; Lee, H. J.; Lim, Y.; Koh, D.: (*E*)-1-(3,5-Dimethoxyphenyl)-3-(3-methoxyphenyl)prop-2-en-1-one. Acta Crystallogr. **E69** (2013) o666–o666.
15. Ezhilarasi, K. S.; Jonathan, D. R.; Sathya, S.; Prathebha, K.; Usha, G.: (2*E*)-1-(3,5-Dihydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one. Acta Crystallogr. **E70** (2014) o608–o609.