Mengdan Wang*, Weiwei Lu, Junling Wang, Mengyang Li, Yuanyuan Feng and Jun-Ying Ma*

The crystal structure of 1-(3-oxo-1-phenyl-3-(*p*-tolyl) propylidene)-1,3-dihydro-2*H*-inden-2-one, $C_{25}H_{20}O_2$

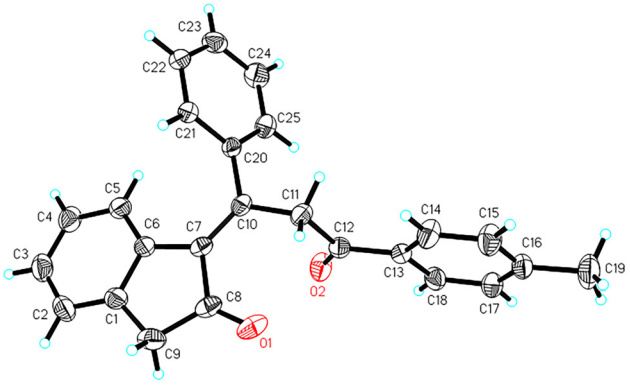

Table 1: Data collection and handling.

Crystal:	Colourless prismatic
Size:	0.20 × 0.16 × 0.13 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 94 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4808, 3160, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2395
$N(param)_{refined}$:	245
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

https://doi.org/10.1515/ncrs-2023-0418
Received September 21, 2023; accepted October 20, 2023;
published online November 8, 2023

Abstract

$C_{25}H_{20}O_2$, triclininc, $P\bar{1}$ (no. 2), a = 9.821(2) Å, b = 10.141(2) Å, c = 10.679(2) Å, α = 61.688(4)°, β = 86.002(4)°, γ = 84.220(4)°, V = 931.3(3) $Å^3$, Z = 2, $R_{gt}(F)$ = 0.0522, $wR_{ref}(F^2)$ = 0.1437, T = 293 K.

CCDC no.: 2295930

The molecular 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.

***Corresponding authors: Mengdan Wang**, School of Chemistry and Chemical Engineering, Henan University of Science and Technology, Luoyang, Henan 471003, P.R. China, E-mail: mdwang@haust.edu.cn. https://orcid.org/0000-0002-9840-5924; and **Jun-Ying Ma**, School of Chemistry and Chemical Engineering, Henan University of Science and Technology, Provincial and Ministerial Co-construction of Collaborative Innovation Center for Non-ferrous Metal New Materials and Advanced Processing Technology, Luoyang, Henan 471003, P.R. China, E-mail: majy379@163.com

Weiwei Lu, Junling Wang, Mengyang Li and Yuanyuan Feng, School of Chemistry and Chemical Engineering, Henan University of Science and Technology, Luoyang, Henan 471003, P.R. China. https://orcid.org/0000-0002-9075-4089 (J. Wang)

1 Source of material

In a Schlenk tube 3-phenyl-1-(*p*-tolyl)prop-2-yn-1-one (66.1 mg, 0.30 mmol), 1,3-dihydro-2*H*-inden-2-one (79.3 mg, 0.60 mmol), Cs_2CO_3 (195.5 mg, 0.60 mmol) and toluene (3.0 mL) were stirred at 100 °C under N_2 for 3 h. The reaction mixture was then quenched by water, and the water layers were extracted with ethyl acetate (10 mL × 3). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. Purification by chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) afforded desired compound (yellow solid, 91.8 mg, 87 %). Single crystals were obtained by crystallization of the title compound from a mixture of dichloromethane (10 mL) and petroleum ether (2 mL). Melting point: 177–178 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.39 (s, 3H), 3.52 (s, 2H), 4.80 (s, 2H), 6.38–6.35 (m, 1H), 6.92–6.87 (m, 1H), 7.46–7.13 (m, 9H), 7.91–7.88 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 206.0, 196.3, 145.3, 144.0, 142.5, 139.5, 137.7, 135.0, 133.7, 129.5, 129.4, 128.5, 128.5, 128.4, 127.1, 126.9, 125.1, 124.4, 46.0, 42.3, 21.3.

2 Experimental details

Using Olex2 [2], the structure was solved with the SHELXT [3] structure solution program and refined with the SHELXL [4] refinement package.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	1.16664 (16)	0.4745 (2)	0.16075 (18)	0.0716 (5)
O2	1.09645 (17)	0.13049 (17)	0.37400 (15)	0.0644 (5)
C1	0.9374 (2)	0.6383 (2)	0.3278 (2)	0.0505 (5)
C2	0.8906 (3)	0.7222 (2)	0.3955 (3)	0.0652 (7)
H2	0.9443	0.7926	0.3956	0.078*
C3	0.7640 (3)	0.7006 (3)	0.4624 (3)	0.0696 (7)
H3	0.7316	0.7568	0.5077	0.083*
C4	0.6852 (3)	0.5963 (3)	0.4626 (2)	0.0656 (7)
H4	0.5998	0.5824	0.5085	0.079*
C5	0.7306 (2)	0.5116 (2)	0.3959 (2)	0.0533 (6)
H5	0.6764	0.4405	0.3979	0.064*
C6	0.8572 (2)	0.5328 (2)	0.3257 (2)	0.0436 (5)
C7	0.93268 (19)	0.4553 (2)	0.25143 (19)	0.0427 (5)
C8	1.0715 (2)	0.5146 (2)	0.2154 (2)	0.0520 (6)
C9	1.0723 (2)	0.6375 (2)	0.2562 (2)	0.0616 (6)
H9A	1.1462	0.6168	0.3203	0.074*
H9B	1.0833	0.7336	0.1726	0.074*
C10	0.89310 (19)	0.3540 (2)	0.21590 (19)	0.0415 (5)
C11	0.9896 (2)	0.2779 (2)	0.1501 (2)	0.0479 (5)
H11A	1.0419	0.3535	0.0741	0.057*
H11B	0.9360	0.2349	0.1076	0.057*
C12	1.0890 (2)	0.1548 (2)	0.2521 (2)	0.0446 (5)
C13	1.17693 (19)	0.0649 (2)	0.19648 (19)	0.0418 (5)
C14	1.1787 (2)	0.0933 (3)	0.0564 (2)	0.0610 (6)
H14	1.1235	0.1736	−0.0084	0.073*
C15	1.2603 (3)	0.0055 (3)	0.0109 (3)	0.0690 (7)
H15	1.2584	0.0264	−0.0837	0.083*
C16	1.3451 (2)	−0.1134 (2)	0.1033 (3)	0.0579 (6)
C17	1.3437 (2)	−0.1408 (2)	0.2425 (3)	0.0581 (6)
H17	1.4001	−0.2201	0.3069	0.070*
C18	1.2617 (2)	−0.0547 (2)	0.2888 (2)	0.0516 (5)
H18	1.2628	−0.0769	0.3838	0.062*
C19	1.4312 (3)	−0.2121 (3)	0.0548 (3)	0.0881 (9)
H19A	1.3746	−0.2793	0.0466	0.132*
H19B	1.5009	−0.2692	0.1228	0.132*
H19C	1.4733	−0.1510	−0.0360	0.132*
C20	0.75196 (19)	0.3076 (2)	0.23806 (18)	0.0390 (5)
C21	0.6419 (2)	0.4113 (2)	0.1751 (2)	0.0459 (5)
H21	0.6573	0.5120	0.1181	0.055*
C22	0.5110 (2)	0.3679 (2)	0.1955 (2)	0.0532 (6)
H22	0.4384	0.4392	0.1534	0.064*
C23	0.4868 (2)	0.2191 (3)	0.2783 (2)	0.0602 (6)
H23	0.3979	0.1895	0.2937	0.072*
C24	0.5950 (3)	0.1146 (3)	0.3378 (3)	0.0632 (6)
H24	0.5792	0.0136	0.3922	0.076*
C25	0.7258 (2)	0.1575 (2)	0.3180 (2)	0.0523 (5)
H25	0.7980	0.0853	0.3587	0.063*

3 Comment

α,β-Unsaturated carbonyls that contain an electron-deficient carbon–carbon double bond are not only versatile synthetic intermediates but also a structural motif in biologically active molecules [5–8]. In this paper we report the synthesis and crystal structure of a novel α,β-unsaturated carbonyl compound. In the crystal structure, there are two carbonyl groups and a carbon–carbon double bond. The two carbonyl groups are linked by carbon–carbon double bond. The carbon–carbon double bond length of C(7)–C(10) is 1.350(3) Å and this is typical for carbon–carbon double bond [9]. The bond lengths of the two carbonyl groups are almost the same (1.205 Å and 1.211 Å), which is similar to that reported [10].

The complete set of X-ray diffraction data for the title compound was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 2295930).

Research funding: This work was supported by the Doctor Start-up Funds of Henan University of Science and Technology (no. 13480078).
Competing interests: The authors declare no conflicts of interest regarding this article.

References

1. BRUKER. *SAINT, APEX2 and SADABS*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. 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.* 2009, *42*, 339–341.
3. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
4. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
5. Muzart J. One-pot syntheses of α,β-unsaturated carbonyl compounds through palladium-mediated dehydrogenation of ketones, aldehydes, esters, lactones and amides. *Eur. J. Org Chem*. 2010, *2010*, 3779–3790.
6. Hayashi T., Yamasaki K. Rhodium-catalyzed asymmetric 1,4-addition and its related asymmetric reactions. *Chem. Rev.* 2003, *103*, 2829–2844.
7. Fang Z., Ning Y., Mi P., Liao P., Bi X. Catalytic C–H α-trifluoromethylation of α,β-unsaturated carbonyl compounds. *Org. Lett.* 2014, *16*, 1522–1525.
8. Xiao Q., Li J., Wang J. 1,4-Diazabicyclo[2.2.2]octane-promoted aminotrifluoromethylthiolation of α,β-unsaturated carbonyl compounds: *N*-trifluoromethylthio-4-nitrophthalimide acts as both the nitrogen and SCF_3 sources. *Org. Lett.* 2015, *17*, 6090–6093.
9. Ghorab M., Alsaid M., Al-Dosari M., Ghabbour H. Crystal structure of the co-crystalline adduct 4-((4,4-dimethyl-2,6-dioxocyclohexylidene) methylamino)-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulfonamide – acetic acid (1/1), $C_{21}H_{24}N_4O_4S{\cdot}C_2H_4O_2$. *Z. Kristallogr. N. Cryst. Struct.* 2017, *232*, 417–419.
10. Chen Q.-W., Shen D.-L. Crystal structure of 2-[(2-oxo-thiazolidine-3-carbonyl)sulfamoyl]-methy-benzoic acid methyl ester, $C_{13}H_{14}N_2O_6S_2$. *Z. Kristallogr. N. Cryst. Struct.* 2017, *232*, 67–68.