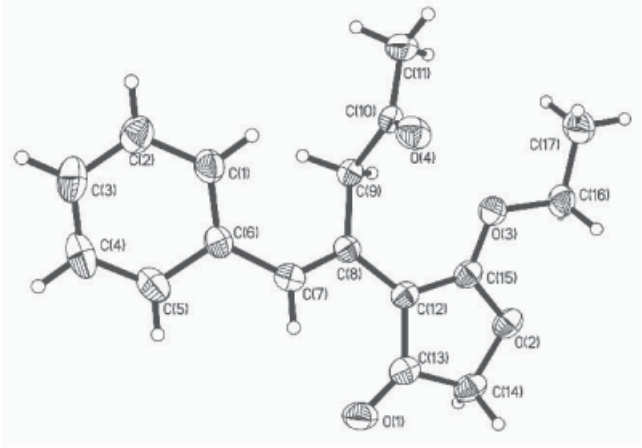
Crystal structure of (*E*)-5-ethoxy-4-(4-oxo-1-phenylpent-1-en-2-yl)furan-3-(2*H*)-one, $C_{17}H_{18}O_4$

Qiang Wang[*, I], Shuqi Ma[I] and Lin Feng[II]

[I] Henan Polytechnic University, School of Physics-Chemistry, Jiaozuo 454000, Henan Province, P. R. China
[II] Jiaozuo Entry-Exit Inspection and Quarantine Bureau, Jiaozuo 454001, Henan Province, P. R. China

Received January 13, 2013, accepted April 09, 2013, available online June 28, 2013, CCDC no. 909448



Abstract

$C_{17}H_{18}O_4$, monoclinic, $P2_1/c$ (no. 14), a = 9.437(2) Å, b = 17.895(4) Å, c = 8.661(2) Å, β = 91.58(3)°, V = 1462.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.0531, $wR_{ref}(F^2)$ = 0.1336, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.3×0.3×0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.92 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7987, 2556
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2213
$N(param)_{refined}$:	192
Programs:	SHELX [5]

Source of material

Phenylbuta-2,3-dien-1-one (1 mmol) and ethyl 4-chloroaceto-acetate (1.2 mmol) in CH_3CN (5 mL) were added anhydrous K_2CO_3 (1.0 mmol). The solution was stirred at room temperature for 1 h. The reaction was then quenched with aqueous NH_4Cl and extracted with ethyl acetate (5 mL × 3). The combined organic phases were dried, filtered and concentrated under vacuum. The residue was purified by column chromatography over silica gel using ethyl acetate/hexane (v:v = 1:10) as eluent to give pure the product. Single crystals suitable for X-ray diffraction were prepared by slow evaporation from a solution of ethyl acetate and petroleum ether (v:v = 1:10).

Experimental details

H atoms were located in a difference map and treated as riding with C–H = 0.96 Å for methyl groups and C–H = 0.93 Å for all other hydrogens with U_{iso}(H) = 1.2 U_{eq} (aromatic, methine) or U_{iso}(H) = 1.5 U_{eq} (methyl).

Discussion

Furan-3(2*H*)-ones are attractive synthetic targets due to exhibit potential biological activities and the furanone scaffold is found in a number of naturally occurring antitumoragents such as jatrophone [1], geiparvarin [2], eremantholides [3], or lychnophorolide [4]. For this reason, efficient and rapid synthesis of furan-3(2*H*)-ones is of significant importance. All bond lengths and angles of the title structure are in the expected ranges. The furan-3(2*H*)-one ring system (C8/O1–O3/C12–C15) is planar with a maximum deviation of -0.0097 (3) Å for atom C14. The dihedral angles between the benzene ring and furan-3(2*H*)-one ring is 15.142 (63)°. The crystal packing is stabilized by van der Waals forces only.

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

Atom	Site	x	y	z	U_{iso}
H(9A)	4*e*	0.9720	0.8717	–0.0206	0.052
H(9B)	4*e*	0.8499	0.9163	0.0558	0.052
H(7)	4*e*	1.2379	0.9648	0.1917	0.061
H(14A)	4*e*	0.9831	1.1200	0.4989	0.071
H(14B)	4*e*	0.9268	1.1534	0.3402	0.071
H(1)	4*e*	1.1433	0.7887	0.0323	0.069
H(16A)	4*e*	0.5717	1.0000	0.3546	0.072
H(16B)	4*e*	0.6481	0.9675	0.5040	0.072
H(11A)	4*e*	0.7551	0.7302	0.1555	0.094
H(11B)	4*e*	0.7953	0.7583	–0.0090	0.094
H(11C)	4*e*	0.6879	0.8039	0.0888	0.094
H(5)	4*e*	1.4091	0.9603	–0.0160	0.079
H(2)	4*e*	1.2672	0.7225	–0.1461	0.083
H(3)	4*e*	1.4623	0.7738	–0.2592	0.091
H(4)	4*e*	1.5373	0.8917	–0.1871	0.093
H(17A)	4*e*	0.5159	0.8805	0.2720	0.112
H(17B)	4*e*	0.4581	0.8914	0.4385	0.112
H(17C)	4*e*	0.5984	0.8464	0.4149	0.112

* Correspondence author (e-mail: wangqiang@hpu.edu.cn)

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

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	4*e*	0.8242(2)	1.06010(7)	0.4016(2)	0.069(1)	0.0468(8)	0.0577(9)	0.0109(7)	0.0026(7)	–0.0099(7)
O(3)	4*e*	0.7550(1)	0.94759(7)	0.3102(2)	0.0532(8)	0.0522(8)	0.0691(9)	0.0006(6)	0.0149(7)	–0.0113(7)
O(4)	4*e*	0.9535(2)	0.79725(8)	0.2800(2)	0.071(1)	0.0585(9)	0.0559(9)	–0.0098(7)	–0.0040(8)	0.0121(7)
C(10)	4*e*	0.8917(2)	0.8176(1)	0.1636(2)	0.043(1)	0.043(1)	0.042(1)	0.0019(8)	0.0082(8)	–0.0065(8)
C(9)	4*e*	0.9344(2)	0.8868(1)	0.0777(2)	0.046(1)	0.045(1)	0.040(1)	0.0029(8)	0.0019(8)	–0.0006(8)
O(1)	4*e*	1.1800(2)	1.08326(8)	0.2941(2)	0.075(1)	0.0542(9)	0.068(1)	–0.0174(8)	–0.0040(8)	–0.0022(8)
C(8)	4*e*	1.0423(2)	0.9363(1)	0.1586(2)	0.050(1)	0.0380(9)	0.041(1)	–0.0013(8)	0.0016(8)	0.0052(8)
C(7)	4*e*	1.1814(2)	0.9323(1)	0.1331(2)	0.050(1)	0.047(1)	0.055(1)	–0.0048(9)	–0.0009(9)	0.0010(9)
C(13)	4*e*	1.0574(2)	1.0623(1)	0.3114(2)	0.070(1)	0.041(1)	0.043(1)	–0.001(1)	–0.006(1)	0.0064(9)
C(12)	4*e*	0.9868(2)	0.9949(1)	0.2596(2)	0.053(1)	0.0381(9)	0.043(1)	0.0019(8)	–0.0009(8)	0.0020(8)
C(15)	4*e*	0.8543(2)	0.9989(1)	0.3206(2)	0.059(1)	0.040(1)	0.046(1)	0.0068(9)	–0.0012(9)	–0.0019(9)
C(14)	4*e*	0.9486(2)	1.1075(1)	0.3955(3)	0.085(2)	0.039(1)	0.053(1)	0.001(1)	–0.007(1)	–0.0032(9)
C(6)	4*e*	1.2581(2)	0.8839(1)	0.0259(2)	0.043(1)	0.055(1)	0.052(1)	0.0027(8)	0.0036(9)	0.0058(9)
C(1)	4*e*	1.2207(2)	0.8108(1)	–0.0137(3)	0.053(1)	0.053(1)	0.069(1)	0.0033(9)	0.013(1)	0.002(1)
C(16)	4*e*	0.6254(2)	0.9580(1)	0.3959(3)	0.053(1)	0.064(1)	0.064(1)	0.010(1)	0.015(1)	–0.005(1)
C(11)	4*e*	0.7718(2)	0.7736(1)	0.0935(3)	0.059(1)	0.058(1)	0.072(1)	–0.011(1)	0.002(1)	–0.012(1)
C(5)	4*e*	1.3792(2)	0.9123(1)	–0.0416(3)	0.049(1)	0.065(1)	0.083(2)	–0.005(1)	0.008(1)	0.010(1)
C(2)	4*e*	1.2960(2)	0.7707(1)	–0.1197(3)	0.066(2)	0.064(1)	0.078(2)	0.013(1)	0.011(1)	–0.006(1)
C(3)	4*e*	1.4129(3)	0.8009(2)	–0.1865(3)	0.069(2)	0.086(2)	0.073(2)	0.028(1)	0.019(1)	0.008(1)
C(4)	4*e*	1.4562(3)	0.8714(2)	–0.1453(3)	0.052(1)	0.092(2)	0.089(2)	0.012(1)	0.024(1)	0.026(2)
C(17)	4*e*	0.5421(2)	0.8879(1)	0.3788(3)	0.061(1)	0.073(2)	0.092(2)	0.001(1)	0.019(1)	–0.007(1)

Acknowledgments. This work was supported by the Natural Science Research Programme of the Education Department of Henan Province (grant No. 2011 A150015), the Basic and Cutting-Edge Technology Research Project of Henan Province (No.132300410293) and the Henan Polytechnic University Foundation for Doctoral Teachers (grant No. B2010-65). The authors thank Dr Z. Xu for assistance with the data analysis.

References

1. Kupchan, M.; Sigel, C. W.; Matz, M. J.; Gilmore, C. J.; Bryan, R. F.: Tumor inhibitors. 111. Structure and stereochemistry of jatrophone, a novel macrocyclic diterpenoid tumor inhibitor. J. Am. Chem. Soc. **98** (1976) 2295-2300.
2. Lahey, F. N.; MacLeod, J. K.: The coumarins of Geijera parviflora Lindl. Aust. J. Chem. **20** (1967) 1943-1955.
3. Le Quesne, P. W.; Levery, S. B.; Menachery, M. D.; Brennan, T. F.; Raffauf, R. F.: Antitumour plants. Part 6. Novel modified germacranolides and other constituents of Eremanthus elaeagnus Schultz-bip (compositae). J. Chem. Soc. PerkinTrans. **1** (1978) 1572-1580.
4. Le Quesne, P. W.; Menachery, M. D.; Pastore, M. P.; Kelley, C. J.; Brennan, T. F.; Onan, K. D.; Raffauf, R. F.: Raffauf, Antitumor plants. 12. Further sesquiterpenoid constituents of Lychnophora affinis Gardn. (Compositae). X-ray structure analysis of lychnophorolide A. J. Org. Chem. **47** (1982) 1519-1521.
5. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.