Crystal structure of 1-(2-furyl)-3-(*p*-methoxyphenyl)-1,3-propanedione, $C_{14}H_{12}O_4$, *the enol form*

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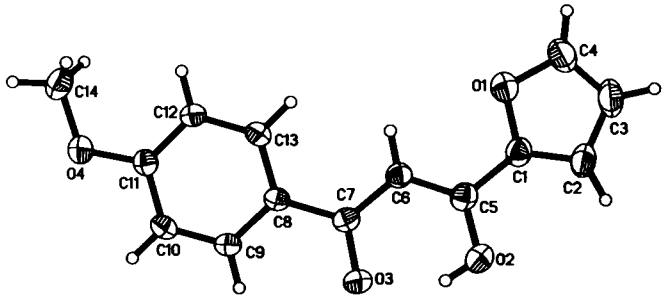
Received March 27, 2004, accepted and available on-line July 15, 2004; CCDC no. 1267/1290



Abstract

$C_{14}H_{12}O_4$, orthorhombic, *Pbca* (no. 61), $a = 10.871(3)$ Å, $b = 7.564(2)$ Å, $c = 29.083(9)$ Å, $V = 2391.4$ Å^3, $Z = 8$, $R_{gt}(F) = 0.055$, $wR_{ref}(F^2) = 0.164$, $T = 293$ K.

Source of material

At room temperature, 45 g *p*-methoxyphenylmethyl ketone was added to 150 ml toluene solution of 0.2 mol sodium ethanolate and 45g ethyl furancarboxylate within approximately 0.75 hours, under steady stirring until no heat of reaction released, then refluxed at 110 °C for 2 hours, cooled down and mixed with 100 ml ice water. The row product was partially dissolved as well as in toluene and water. Both layers were separated and washed with 200 ml diethyl ether, followed by addition of 20 ml ice acetic acid under stirring. The product was filtered off and recrystallized from tetrahydrofuran/acetone (*v*/*v* 2:1). Perfect single crystals were obtained by repeated recrystallization (yield 56 g).

Discussion

The title compound crystallizes isomorphously with 1-phenyl-3-(*p*-methoxyphenyl)-1,3-propanedione [1] and dibenzoylmethane [2]. Two C=O bond lengths are 1.286(3) Å and 1.298(3) Å, with an average of 1.292(3) Å. The bond angles of C1–C5–O2 and C8–C7–O3 are 115.3(2)° and 117.7(2)°, respectively. The average bond length and bond angle in the phenyl group are 1.383(3) Å and 120(2)°. These are in agreement with those in [1,2]. The dihedral angles range from 1.2(2)° to 10.3(2)°. The average bond lengths C—O and C—C in furyl are 1.359(3) Å and 1.356(4) Å. The average angles C–O–C, C–C–C and C–C–O in furyl are 106.5(2)°, 106.8(3)°, and 109.9(2)°, respectively. The bond length between O4 from CH_3O-group and C11 in phenyl ring is 1.358(3) Å, and the C11–O4–C14 angle is 118.1(2)°. A strong intramolecular hydrogen bond with *d*(O2–H2A···O3) = 2.495(2) Å and ∠O2–H2A···O3 = 147.2° exists in the molecule. Furthermore, the hydrogen atom in phenyl group form hydrogen bonds with the C=O double bond from neighboring molecule. The hydrogen bond of C12–H12···O3′ is 3.486(3) Å, the angle C12–H12···O3′ is 166.1°. An infinite one-dimensional chain is formed through the intermolecular hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.20 × 0.24 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12907, 2472
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1678
$N(param)_{refined}$:	166
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	8*c*	0.7152	0.0079	0.3605	0.107
H(2)	8*c*	0.5281	0.0507	0.2566	0.096
H(3)	8*c*	0.3235	0.1686	0.2302	0.110
H(4)	8*c*	0.2262	0.3046	0.2958	0.093
H(6)	8*c*	0.4843	0.2116	0.4083	0.056
H(9)	8*c*	0.7763	0.0094	0.5033	0.056
H(10)	8*c*	0.7561	0.0677	0.5802	0.060
H(12)	8*c*	0.4527	0.3528	0.5547	0.053
H(13)	8*c*	0.4735	0.2912	0.4775	0.052
H(14A)	8*c*	0.5023	0.4632	0.6258	0.107
H(14B)	8*c*	0.5103	0.3581	0.6721	0.107
H(14C)	8*c*	0.4244	0.2907	0.6325	0.107

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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(1)	8c	0.3764(2)	0.2333(3)	0.33345(6)	0.063(1)	0.090(1)	0.054(1)	0.0065(9)	−0.0045(8)	−0.0012(9)
O(2)	8c	0.6703(2)	0.0322(3)	0.33876(6)	0.061(1)	0.099(1)	0.053(1)	0.009(1)	0.0066(8)	−0.007(1)
O(3)	8c	0.7417(2)	0.0256(2)	0.42027(6)	0.0441(9)	0.080(1)	0.060(1)	0.0103(8)	0.0031(7)	−0.0090(9)
O(4)	8c	0.5996(2)	0.2454(2)	0.62132(5)	0.061(1)	0.077(1)	0.045(1)	0.0070(8)	−0.0029(7)	−0.0043(8)
C(1)	8c	0.4793(2)	0.1471(3)	0.31916(8)	0.055(1)	0.065(2)	0.046(1)	−0.006(1)	0.005(1)	0.004(1)
C(2)	8c	0.4702(3)	0.1093(5)	0.27448(9)	0.075(2)	0.124(3)	0.042(2)	0.003(2)	0.004(1)	−0.000(2)
C(3)	8c	0.3557(3)	0.1755(5)	0.2598(1)	0.080(2)	0.149(3)	0.046(2)	−0.007(2)	−0.006(2)	0.016(2)
C(4)	8c	0.3027(3)	0.2496(4)	0.2960(1)	0.066(2)	0.105(2)	0.062(2)	−0.001(2)	−0.009(1)	0.019(2)
C(5)	8c	0.5719(2)	0.1105(3)	0.35370(8)	0.049(1)	0.055(1)	0.051(1)	−0.009(1)	0.006(1)	0.002(1)
C(6)	8c	0.5552(2)	0.1523(3)	0.39933(7)	0.044(1)	0.049(1)	0.047(1)	−0.001(1)	0.0018(9)	0.000(1)
C(7)	8c	0.6432(2)	0.1072(3)	0.43248(7)	0.038(1)	0.040(1)	0.051(1)	−0.0068(9)	0.0053(9)	−0.0005(9)
C(8)	8c	0.6280(2)	0.1458(2)	0.48170(7)	0.033(1)	0.037(1)	0.047(1)	−0.0050(8)	0.0005(8)	0.0024(9)
C(9)	8c	0.7112(2)	0.0791(3)	0.51347(8)	0.039(1)	0.047(1)	0.054(1)	0.0049(9)	0.0008(9)	−0.000(1)
C(10)	8c	0.6994(2)	0.1139(3)	0.55950(8)	0.043(1)	0.054(1)	0.052(1)	0.006(1)	−0.009(1)	0.006(1)
C(11)	8c	0.6033(2)	0.2177(3)	0.57528(7)	0.044(1)	0.045(1)	0.045(1)	−0.0078(9)	−0.0016(9)	0.0006(9)
C(12)	8c	0.5180(2)	0.2841(3)	0.54440(7)	0.039(1)	0.046(1)	0.049(1)	0.0037(9)	0.0030(9)	−0.001(1)
C(13)	8c	0.5311(2)	0.2470(3)	0.49817(8)	0.038(1)	0.046(1)	0.047(1)	0.0000(9)	−0.0041(9)	0.0043(9)
C(14)	8c	0.5012(3)	0.3477(4)	0.63938(9)	0.084(2)	0.081(2)	0.049(2)	0.009(2)	0.008(1)	−0.010(1)

Acknowledgments. We are grateful to the Natural Science Foundation of Beijing (grant no. 2022007), the Science and Technology of Beijing Foundation for people studied abroad and the Project-Sponsoring by SRF for ROCS and SEM.

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