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Crystal structure of *ortho*-(2*E*,5*E*)-2,5-bis(2-methoxybenzylidene)cyclopentanone, $C_{21}H_{20}O_3$

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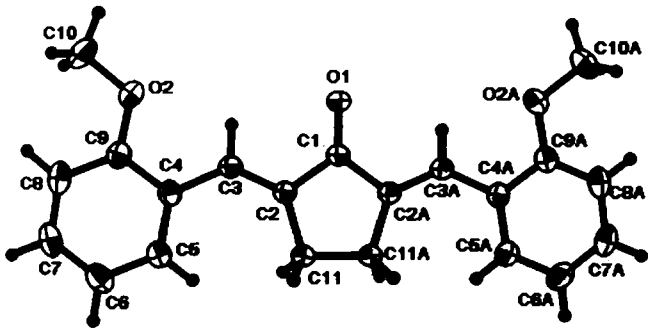

Abstract

$C_{21}H_{20}O_3$, monoclinic, *C*12/*c*1 (no. 15), a = 15.483(3) Å, b = 14.575(2) Å, c = 7.639(1) Å, β = 105.828(3)°, V = 1658.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.054, $wR_{ref}(F^2)$ = 0.155, T = 293 K.

Source of material

The title compound was prepared as previously described. This class of compounds is readily synthesized by reacting a substituted benzaldehyde with cyclopentanone [1]. Single crystals were obtained by re-crystallization from a methanol/chloroform solution (1:5, *v*/*v*) at 293 K.

Experimental details

The H atoms bound to C were positioned geometrically and allowed to ride on their parent atoms at distances of d(Csp^2—H) = 0.93 Å with U_{iso} = 1.2 U_{eq}(parent atom), d(Csp^3—H) = 0.97 Å with U_{iso} = 1.5 U_{eq}(parent atom).

Discussion

The title compound is a biologically active derivative of curcumin. During the last two decades, numerous studies have shown that curcumin has a variety of biological and pharmacological activities such as anti-carcinogen, immuno-modulation, anti-oxidant, anti-angiogenesis, anti-inflammation and chemoprevention [2-5]. However, pre-clinical and clinical studies have found that the potential beneficial effects of curcumin on various disease preventions and treatments are limited by its poor pharmacokinetic properties [6,7]. It is suggested that the presence of the methylene group and β-diketone moiety contributes to the instability of curcumin under physiological conditions [8,9]. In our previous study, a series of mono-carbonyl analogues of curcumin were designed by deleting the reactive β-diketone moiety and synthesized [10-13]. Part of bisbenzylidene cyclopentanone derivatives showed stronger bio-activities than curcumin. As part of our research in this area, the title compound was synthesized.

The $C_{21}H_{20}O_3$ molecule contains three rings. The bond lengths within phenyl rings are between 1.371(2) Å and 1.405(2) Å, which highlights the aromatic character. The molecule has crystallographic two-fold rotation symmetry and shows an *E*-configuration of the central olefinic bonds. The two independent planar benzene ring systems are essentially indentical, and the central cyclopentanone ring are almost coplanar with the two benzene rings [dihedral angle = 4.2(2)°]. The two methoxyl groups attached to atoms C9 and C9A are located on different sides of the C1=O1 double bond, as reflected by the torsion angles C5–C4–C9–O2 (178.9(1)°), C10–O2–C9–C8 (–9.3(2)°), C10–O2–C9–C4 (171.2(1)°), C7–C8–C9–O2 (–179.0(1)°) and C3–C4–C9–O2 (–1.5(2)°). The most important feature for the packing of 2,5-bis(2-methoxybenzylidene)cyclo-pentanone is stacking. This molecule demonstrates apparently a long and flat shape. There are no strong interactions between the stacked molecules.

Table 1. Data collection and handling.

Crystal:	green prismatic, size 0.342 × 0.450 × 0.492 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4444, 1628
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1141
$N(param)_{refined}$:	111
Programs:	SHELXS-97 [14], SHELXL-97 [15], SHELXTL [16]

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

Atom	Site	x	y	z	U_{iso}
H(3)	8*f*	0.8528	0.4080	1.0356	0.055
H(5)	8*f*	0.8012	0.6461	0.9611	0.071
H(6)	8*f*	0.6693	0.7057	0.7810	0.086
H(7)	8*f*	0.5546	0.6085	0.6373	0.082
H(8)	8*f*	0.5731	0.4520	0.6667	0.077
H(10A)	8*f*	0.5945	0.3158	0.8383	0.124
H(10B)	8*f*	0.6626	0.2395	0.8183	0.124
H(10C)	8*f*	0.6219	0.3064	0.6561	0.124
H(11A)	8*f*	0.9103	0.6395	1.2343	0.061
H(11B)	8*f*	0.9543	0.6405	1.0712	0.061

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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)	4*e*	0	0.3675(1)	¼	0.056(1)	0.035(1)	0.093(1)	0	–0.0055(9)	0
O(2)	8*f*	0.71505(8)	0.36286(8)	0.8536(2)	0.0543(8)	0.0572(9)	0.0822(9)	–0.0120(6)	0.0015(6)	–0.0056(6)
C(1)	4*e*	0	0.4515(1)	¼	0.043(1)	0.037(1)	0.057(1)	0	0.010(1)	0
C(2)	8*f*	0.92693(9)	0.5105(1)	1.1424(2)	0.0402(9)	0.0383(9)	0.0499(9)	0.0000(6)	0.0109(7)	0.0021(6)
C(3)	8*f*	0.85203(9)	0.4718(1)	1.0369(2)	0.0428(9)	0.0390(9)	0.0546(9)	–0.0001(6)	0.0122(7)	0.0008(7)
C(4)	8*f*	0.7695(1)	0.5128(1)	0.9239(2)	0.0393(9)	0.052(1)	0.0467(8)	0.0017(7)	0.0121(7)	0.0006(7)
C(5)	8*f*	0.7557(1)	0.6063(1)	0.9018(2)	0.049(1)	0.055(1)	0.067(1)	0.0043(8)	0.0041(8)	0.0012(8)
C(6)	8*f*	0.6764(1)	0.6425(1)	0.7946(2)	0.064(1)	0.066(1)	0.079(1)	0.0188(9)	0.008(1)	0.0075(9)
C(7)	8*f*	0.6084(1)	0.5845(2)	0.7083(2)	0.045(1)	0.091(2)	0.063(1)	0.0144(9)	0.0044(8)	0.006(1)
C(8)	8*f*	0.6194(1)	0.4910(2)	0.7261(2)	0.042(1)	0.089(2)	0.058(1)	–0.0045(9)	0.0050(8)	–0.0018(9)
C(9)	8*f*	0.6991(1)	0.4550(1)	0.8321(2)	0.046(1)	0.057(1)	0.0498(9)	–0.0031(7)	0.0129(7)	–0.0026(7)
C(10)	8*f*	0.6426(1)	0.3011(1)	0.7861(3)	0.071(1)	0.075(2)	0.094(2)	–0.027(1)	0.009(1)	–0.019(1)
C(11)	8*f*	0.9530(1)	0.6088(1)	1.1821(2)	0.047(1)	0.0376(9)	0.063(1)	0.0012(7)	0.0055(8)	0.0013(7)

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