Crystal structure of (2*E*,6*E*)-2-benzylidene-6-(3,4-dimethoxybenzylidene) cyclohexanone, $C_{22}H_{22}O_3$

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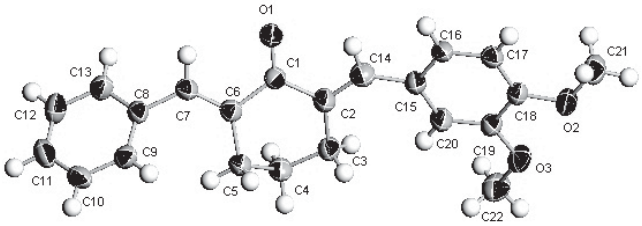

Abstract

$C_{22}H_{22}O_3$, triclinic, $P\bar{1}$ (no. 2), a = 8.641(1) Å, b = 9.808(1) Å, c = 12.420(1) Å, α = 89.263(2)°, β = 70.596(2)°, γ = 64.224(2)°, V = 883.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.0646, $wR_{ref}(F^2)$ = 0.1796, T = 293 K.

Table 1. Data collection and handling.

Crystal:	green prisms, size 0.115×0.186×0.265 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.82 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5178, 3276
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2732
$N(param)_{refined}$:	228
Programs:	SHELX [5]

Source of material

The title compound was prepared as previously described by a base catalyzed Claisen-Schmidt condensation of 2-benzylidene-cyclohexanone and 3,4-dimethoxybenzaldehyde [1, 2]. Single crystals were obtained by re-crystallization from a methanol/chloroform solution (1:3, *v/v*) at 293 K.

Experimental details

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

Discussion

Colorectal cancer is one of the most fatal malignancies worldwide and develops spontaneously or as a long-term complication of chronic bowel inflammation such as in Crohn's disease or ulcerative colitis [3]. Many naturally occurring chalcones and flavones contain an aryl ring conjugated with an alpha, beta unsaturated keto group (Ar–CH=CH–CO). Such molecules display various important therapeutic actions [2] and, therefore, these compounds have received considerable interest. Chalcones and its derivatives have also shown antitumor activity in Mice [4]. As a part of our research for novel antitumor agents, we designed and synthesized a series of chalcones analogues, and the biological activity evaluation indicated that some of these compounds are potent antitumor agents. The reported geometries, molecular properties along with the electrostatic potential surfaces and contours have also been used to understand the properties of the molecules. The central ring of the cyclohexanone ring assumes a sofa conformation with two benzylidene rings connected through *E,E* oriented groups. The carbonyl group is planar and has bond angles of (O1–C1–C2 = 120.6(2)°, O1–C1–C6 = 120.7(2)°, C2–C1–C6 = 118.44(19)°. The dihedral angle between the aromatic rings is 67.95(13)°. The torsion angle O1–C1–C2–C3 is -166.8(3)°, while the torsion angles O2–C18–C19–C20 and O3–C19–C20–C15 have magnitudes 178.6(2)° and -179.2(3)°, respectively.

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

Atom	Site	x	y	z	U_{iso}
H(3A)	2*i*	0.3431	0.2385	0.8396	0.056
H(3B)	2*i*	0.5290	0.1239	0.8517	0.056
H(4A)	2*i*	0.4712	0.3882	0.8598	0.058
H(4B)	2*i*	0.5336	0.3056	0.9579	0.058
H(5A)	2*i*	0.2781	0.5508	1.0350	0.055
H(5B)	2*i*	0.1597	0.4992	0.9891	0.055
H(7)	2*i*	0.1476	0.4022	1.2910	0.050
H(9)	2*i*	0.0041	0.7381	1.1724	0.055
H(10)	2*i*	–0.0229	0.9694	1.2352	0.063
H(11)	2*i*	0.1003	0.9864	1.3700	0.071
H(12)	2*i*	0.2365	0.7736	1.4500	0.072
H(13)	2*i*	0.2584	0.5425	1.3907	0.060
H(14)	2*i*	0.1935	0.0251	1.0532	0.060
H(16)	2*i*	0.1462	–0.1544	0.9829	0.053
H(17)	2*i*	0.1780	–0.3189	0.8394	0.050
H(20)	2*i*	0.4898	–0.0274	0.7537	0.059
H(21A)	2*i*	0.3263	–0.5254	0.7012	0.094
H(21B)	2*i*	0.3363	–0.5355	0.5730	0.094
H(21C)	2*i*	0.1651	–0.4041	0.6677	0.094
H(22A)	2*i*	0.6109	–0.0333	0.5598	0.101
H(22B)	2*i*	0.7635	–0.1747	0.4655	0.101
H(22C)	2*i*	0.7583	–0.1713	0.5929	0.101

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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)	2*i*	0.1775(2)	0.1810(2)	1.1943(1)	0.071(1)	0.051(1)	0.0418(9)	–0.0376(9)	–0.0109(8)	0.0075(7)
O(2)	2*i*	0.3862(2)	–0.3720(2)	0.6187(1)	0.058(1)	0.059(1)	0.0398(9)	–0.0341(9)	–0.0111(8)	–0.0035(7)
O(3)	2*i*	0.5672(3)	–0.2143(2)	0.5724(1)	0.065(1)	0.069(1)	0.0369(9)	–0.043(1)	0.0007(8)	–0.0039(8)
C(1)	2*i*	0.2323(3)	0.2373(2)	1.1111(2)	0.042(1)	0.043(1)	0.039(1)	–0.024(1)	–0.0145(9)	0.0074(9)
C(2)	2*i*	0.3047(4)	0.1558(3)	0.9924(2)	0.064(2)	0.048(1)	0.042(1)	–0.031(1)	–0.011(1)	0.005(1)
C(3)	2*i*	0.4087(3)	0.2090(3)	0.8927(2)	0.052(1)	0.049(1)	0.039(1)	–0.028(1)	–0.008(1)	0.003(1)
C(4)	2*i*	0.4351(3)	0.3427(3)	0.9275(2)	0.052(1)	0.051(1)	0.043(1)	–0.032(1)	–0.009(1)	0.008(1)
C(5)	2*i*	0.2599(3)	0.4638(3)	1.0181(2)	0.055(1)	0.042(1)	0.041(1)	–0.026(1)	–0.015(1)	0.0081(9)
C(6)	2*i*	0.2120(3)	0.3958(2)	1.1262(2)	0.040(1)	0.041(1)	0.041(1)	–0.022(1)	–0.0134(9)	0.0063(9)
C(7)	2*i*	0.1656(3)	0.4610(3)	1.2332(2)	0.044(1)	0.040(1)	0.040(1)	–0.021(1)	–0.0126(9)	0.0075(9)
C(8)	2*i*	0.1392(3)	0.6127(2)	1.2719(2)	0.040(1)	0.038(1)	0.037(1)	–0.0176(9)	–0.0081(9)	0.0020(9)
C(9)	2*i*	0.0523(3)	0.7441(3)	1.2279(2)	0.046(1)	0.045(1)	0.039(1)	–0.017(1)	–0.013(1)	0.005(1)
C(10)	2*i*	0.0364(4)	0.8827(3)	1.2652(2)	0.056(2)	0.037(1)	0.049(1)	–0.017(1)	–0.008(1)	0.007(1)
C(11)	2*i*	0.1078(4)	0.8933(3)	1.3467(2)	0.066(2)	0.045(1)	0.060(2)	–0.030(1)	–0.009(1)	–0.004(1)
C(12)	2*i*	0.1902(4)	0.7662(3)	1.3938(2)	0.067(2)	0.059(2)	0.054(2)	–0.027(1)	–0.024(1)	–0.006(1)
C(13)	2*i*	0.2044(3)	0.6276(3)	1.3575(2)	0.054(1)	0.045(1)	0.045(1)	–0.016(1)	–0.019(1)	0.004(1)
C(14)	2*i*	0.2638(3)	0.0391(3)	0.9830(2)	0.050(1)	0.052(1)	0.047(1)	–0.025(1)	–0.013(1)	0.007(1)
C(15)	2*i*	0.3077(4)	–0.0691(3)	0.8851(2)	0.061(2)	0.043(1)	0.040(1)	–0.029(1)	–0.012(1)	0.0038(9)
C(16)	2*i*	0.2210(3)	–0.1605(2)	0.9075(2)	0.053(1)	0.038(1)	0.034(1)	–0.021(1)	–0.008(1)	0.0050(9)
C(17)	2*i*	0.2416(3)	–0.2607(2)	0.8217(2)	0.043(1)	0.040(1)	0.040(1)	–0.021(1)	–0.012(1)	0.0073(9)
C(18)	2*i*	0.3557(3)	–0.2758(2)	0.7095(2)	0.039(1)	0.039(1)	0.037(1)	–0.0148(9)	–0.0139(9)	0.0024(9)
C(19)	2*i*	0.4530(3)	–0.1885(3)	0.6848(2)	0.042(1)	0.043(1)	0.034(1)	–0.019(1)	–0.0084(9)	0.0029(9)
C(20)	2*i*	0.4273(4)	–0.0865(3)	0.7708(2)	0.060(2)	0.048(1)	0.042(1)	–0.033(1)	–0.011(1)	0.005(1)
C(21)	2*i*	0.2961(4)	–0.4670(3)	0.6421(2)	0.076(2)	0.072(2)	0.051(2)	–0.050(2)	–0.015(1)	–0.003(1)
C(22)	2*i*	0.6845(4)	–0.1426(4)	0.5455(2)	0.070(2)	0.085(2)	0.049(2)	–0.051(2)	–0.002(1)	0.007(1)

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