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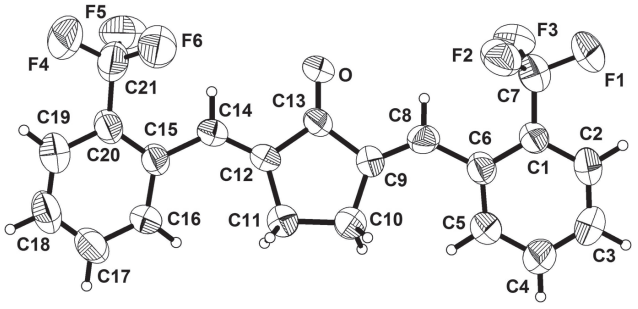
Crystal structure of 2,5-bis((*E*)-2-(trifluoromethyl) benzylidene)cyclopentan-1-one, $C_{21}H_{14}F_6O$



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Abstract

$C_{21}H_{14}F_6O$, triclinic, $P\bar{1}$ (no. 2), $a = 7.8470(16)$ Å, $b = 7.9390(16)$ Å, $c = 14.993(3)$ Å, $\alpha = 83.25(3)°$, $\beta = 89.18(3)°$, $\gamma = 73.79(3)°$, $V = 890.5(3)$ Å, $Z = 2$, $R_{gt}(F) = 0.0658$, $wR_{ref}(F^2) = 0.1261$, $T = 293$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.22 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
θ_{max}:	25.0°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3150, 3150
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1298
$N(param)_{refined}$:	253
Programs:	SHELX [13]

Source of materials

The title compound was synthesized by Aldol condensation between two molecules of 2-(trifluoromethyl)benzaldehyde and cyclopentanone, which was synthesized according our earlier published method [1, 2]. In detail: 5.0 mmol cyclopentanone was added to a solution of 10 mmol 2-(trifluoromethyl)benzaldehyde in MeOH (10 mL). The solution was stirred at room temperature for 30 min, followed by dropwise addition of $NaOCH_3/CH_3OH$ (1.0 mL, 5.0 mmol). The mixture was stirred at room temperature and monitored with TLC. When the reaction was finished, the residue was poured into saturated NH_4Cl solution and filtered. The precipitate was washed with water and cold ethanol, and dried in vacuum. The solid was further purified by silica gel chromatography (CH_2Cl_2/CH_3OH). All chemicals used for the synthesis were commercially available and were used without further purification. The yellow crystals of the title compound were obtained by slow evaporation of a CH_3OH solution at room temperature.

Experimental details

Position of the H atoms were calculated based on geometric criteria (C—H = 0.97 and 0.93 for methyl and aromatic atoms, respectively) and placed in their calculated position and refined, using a riding model with $U_{iso}(H) = 1.5\ U_{eq}$ (C) for methyl and $U_{iso}(H) = 1.2\ U_{eq}(C)$ for all others.

Discussion

Curcumin has been demonstrated to possess a wide range of pharmaceutical activities, such as anti-tumor [3, 4], anti-inflammation [5], anti-oxidation [6], anti-bacterial [7], and cardiovascular protection [8, 9]. However, its high metabolic instability and low bioavailability have dramatically limited its practical application [10]. Recently, our research group has synthesized a series of mono-carbonyl analogs of curcumin (MACs) by deleting β-diketone moiety [11, 12]. The title compound is an Curcumin analogon with one central keto group only.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O	0.3518(3)	0.1788(3)	0.12515(15)	0.0671(8)
F1	0.2333(4)	−0.1261(4)	0.51161(16)	0.1268(12)
C1	0.2977(5)	−0.2847(5)	0.3855(3)	0.0588(11)
F2	0.1382(4)	0.0185(3)	0.38446(16)	0.0995(9)
C2	0.2981(6)	−0.4343(6)	0.4439(3)	0.0852(14)
H2A	0.2831	−0.4267	0.5051	0.102*
F3	0.4119(4)	−0.0540(3)	0.41798(16)	0.0978(9)
C3	0.3207(6)	−0.5934(6)	0.4112(3)	0.0956(16)
H3A	0.3198	−0.6932	0.4503	0.115*
F4	0.0923(5)	0.7135(4)	−0.21608(18)	0.1480(14)
C4	0.3442(6)	−0.6054(6)	0.3227(3)	0.0887(15)
H4A	0.3614	−0.7137	0.3008	0.106*
F5	0.3074(4)	0.5680(4)	−0.12766(19)	0.1099(10)
C5	0.3425(6)	−0.4567(6)	0.2647(3)	0.0771(13)
H5A	0.3568	−0.4666	0.2037	0.093*
F6	0.0446(4)	0.5745(3)	−0.09335(18)	0.1072(9)
C6	0.3202(5)	−0.2930(5)	0.2941(3)	0.0612(11)
C7	0.2711(7)	−0.1148(7)	0.4241(3)	0.0746(13)
C8	0.3224(5)	−0.1373(5)	0.2300(3)	0.0609(11)
H8A	0.3457	−0.0451	0.2560	0.073*
C9	0.2966(5)	−0.1071(5)	0.1416(3)	0.0573(11)
C10	0.2523(5)	−0.2174(5)	0.0759(2)	0.0708(12)
H10A	0.1384	−0.2389	0.0893	0.085*
H10B	0.3421	−0.3302	0.0784	0.085*
C11	0.2460(5)	−0.1119(5)	−0.0179(2)	0.0659(11)
H11A	0.3397	−0.1740	−0.0549	0.079*
H11B	0.1327	−0.0962	−0.0477	0.079*
C12	0.2704(5)	0.0626(5)	−0.0040(2)	0.0596(11)
C13	0.3126(5)	0.0609(5)	0.0919(2)	0.0542(10)
C14	0.2613(5)	0.2052(5)	−0.0631(2)	0.0576(11)
H14A	0.2824	0.3005	−0.0392	0.069*
C15	0.2229(5)	0.2331(5)	−0.1600(2)	0.0591(11)
C16	0.2400(5)	0.0915(5)	−0.2090(3)	0.0710(12)
H16A	0.2774	−0.0230	−0.1798	0.085*
C17	0.2023(6)	0.1174(7)	−0.3013(3)	0.0861(14)
H17A	0.2120	0.0207	−0.3325	0.103*
C18	0.1514(6)	0.2838(7)	−0.3455(3)	0.0898(16)
H18A	0.1281	0.3011	−0.4070	0.108*
C19	0.1347(6)	0.4255(6)	−0.2989(3)	0.0817(14)
H19A	0.0989	0.5393	−0.3290	0.098*
C20	0.1705(5)	0.4016(6)	−0.2071(3)	0.0654(12)
C21	0.1536(8)	0.5632(7)	−0.1621(4)	0.0916(15)

The symmetrical unit of this complex is constructed by two 2-(trifluoromethyl)benzylidene groups and a cyclopentanone linker. The C—C bond lengths of the cyclopentanone moiety are with 1.477(4) to 1.543(4) Å quite normal. The C8—C9 and C12—C14 bond lengths of 1.326(4) and 1.340(4) Å conform to a double bond. The C6—C8 and C14—C15 bond lengths of 1.477(5) and 1.465(5) Å are between double and single bonds. The shortening of the C—C bonds showing the partial double-bond character of the C—C bonds, which are influenced by adjacent double bonds.

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