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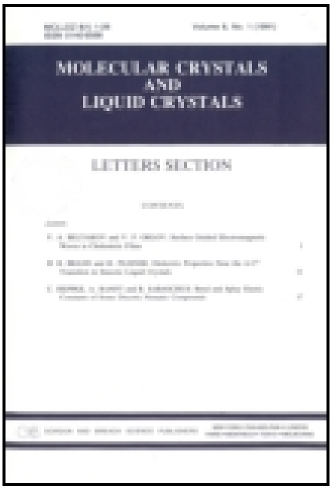

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Synthesis and Crystal Structures of (E)-1-Phenyl-3-[(2,4,6-Trimethylphenyl)]prop-2-En-1-One and (E)-1-Phenyl-3-[(4-Trifluoromethylphenyl)]prop-2-En-1-One

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Synthesis and Crystal Structures of (*E*)-1-Phenyl-3-[(2,4,6-Trimethylphenyl)]prop-2-En-1-One and (*E*)-1-Phenyl-3-[(4-Trifluoromethylphenyl)]prop-2-En-1-One

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*Two chalcone compounds, namely (*E*)-1-phenyl-3-[(2,4,6-trimethylphenyl)]prop-2-en-1-one (**1**) and (*E*)-1-phenyl-3-[(4-trifluoromethylphenyl)]prop-2-en-1-one (**2**), have been synthesized and structurally characterized by elemental analysis, ^1H NMR spectrum, and single-crystal X-ray diffraction analysis. The chalcone molecules in (**1**) and (**2**) have the common skeleton of 1,3-diaryl-2-propen-1-one and adopt an (*E*)-configuration about the $C=C$ double bonds. In addition, X-ray analysis reveals that the $\pi\cdots\pi$ stacking interactions are well observed in the crystal structure of (**1**) and (**2**).*

Keywords Chalcone; crystal structures; intermolecular interaction; synthesis

Introduction

Chalcones that possess the common skeleton of 1,3-diaryl-2-propen-1-ones have attracted much attention in recent years due to their extensive use or application in the field of the synthetical and biological chemistry. On one hand, chalcones and their derivatives are regarded as the signigficant precursors for the synthesis of different types of heterocyclic compounds such as flavones, which are an important group of bioactive organic compounds [1, 2]. On the other hand, natural and synthetic chalcones have been reported to show many biological properties including antibacterial [3], antiinflammatory [4], antifungal [5], antituberculosis [6, 7], and anti-HIV [8], *etc*. Eespecially, the unique structures of the chalcone compounds, which contain two aromatic rings separated by α,β-unsaturated ketone, are resposiber for various activites of these molecules [9]. This seems very important to better understand the relationship between molecular structure and biological activity through the study on the synthesis and structures of the chalcone

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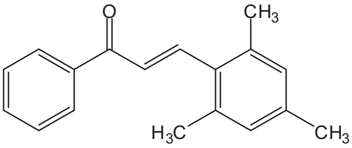

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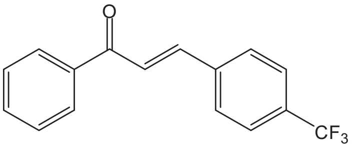


2

Figure 1. The molecular structures of the title compounds (**1**) and (**2**).

compounds. In view of the above-mentioned information, we herein report that two chalcone compounds, chemically (*E*)-1-phenyl-3-[(2,4,6-trimethylphenyl)]prop-2-en-1-one (**1**) and (*E*)-1-phenyl-3-[(4-trifluoromethylphenyl)]prop-2-en-1-one (**2**), have been synthesized and structurally characterized using the single-crystal X-ray diffraction method as well as spectroscopic techniques. Their molecular structures are as shown in Fig. 1. In this context, we now wish to present the detailed crystal structure analyses of the title compounds (**1**) and (**2**) based on X-ray crystallography.

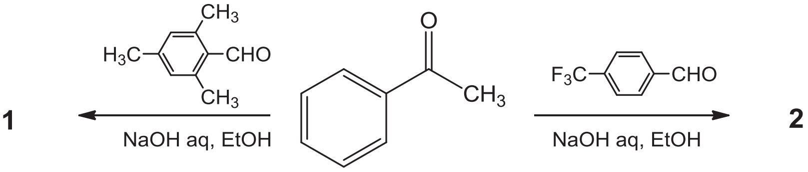
Experimental

Materials and Instruments

All the chemicals were of reagent grade and used without further purification. Melting points were determined on a YRT-3 apparatus and are uncorrected. Elemental analyses for carbon and hydrogen were performed on a Perkin-Elmer 240C analyzer. ^{1}H NMR spectra were obtained on a Bruker Avance 400 MHz spectrometer.

Synthetic Procedures

The synthetic route of the title compounds (**1**) and (**2**) is presented in Scheme 1.



Scheme 1. Synthetic route for the title compounds (**1**) and (**2**).

Synthesis of (E)-1-phenyl-3-[(2,4,6-trimethylphenyl)]prop-2-en-1-one (1)

In 25 mL round-bottomed flask, the acetophenone (0.60 g, 5.0 mmol) and sodium hydroxide (0.30 g, 7.5 mmol) were dissolved in ethanol + water (2 mL + 3 mL), and the mixture was stirred at room temperature for 5 min followed by addition of 2,4,6-trimethylbenzaldehyde (0.74 g, 5.0 mmol). The reaction mixture was then stirred at room temperature and monitored by TLC until the reaction completed. The solid was filtered, washed with cold water, recrystallized from ethanol, and dried *in vacuo* to give a white solid (0.90 g, yield of 72%). Mp: 101–103°C. Anal. Calcd. (%) for $C_{18}H_{18}O$: C, 86.36, H, 7.25. Found (%): C, 86.20,

Table 1. Crystal data and structural refinement details for (**1**) and (**2**)

Compounds	(**1**)	(**2**)
CCDC number	944793	944794
Empirical formula	$C_{18}H_{18}O$	$C_{16}H_{11}F_3O$
Formula weight	250.32	276.25
Temperature (K)	113 (2)	113 (2)
Wavelength (Å)	0.71073	1.54187
Crystal system	Monoclinic	Monoclinic
space group	P2(1)	P2(1)/c
Unit cell dimensions (Å, °)	$a = 10.882\ (8), \alpha = 90$	$a = 14.7469\ (5), \alpha = 90$
	$b = 5.661\ (4), \beta = 106.880\ (9)$	$b = 14.5697\ (4), \beta = 92.8540\ (10)$
	$c = 11.726\ (9), \gamma = 90$	$c = 5.8430\ (2), \gamma = 90$
Volume ($Å^3$), Z	691.3 (9), 2	1253.86 (7), 4
Calculated density (g cm^{-3})	1.203	1.463
Absorption coefficient μ (mm^{-1})	0.073	0.121
$F(000)$	268	568
Crystal size (mm^3)	0.20 × 0.18 × 0.12	0.20 × 0.18 × 0.08
θ range for data collection (°)	1.81–27.89	1.97–27.93
Limiting indices	$-14 < = h < = 13$, $-7 < = k < = 7$, $-15 < = l < = 15$	$-19 < = h < = 19$, $-14 < = k < = 19$, $-7 < = l < = 7$
Reflection collected	7270	12984
Independent reflection	3199 ($R_{int} = 0.0436$)	3005 ($R_{int} = 0.0491$)
Completeness to $\theta_{\max}$ (%)	99.7	99.8
Data/restraints/parameters	3199/1/175	3005/0/181
Goodness-of-fit on F^2	0.988	1.117
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0398$, $wR_2 = 0.0712$	$R_1 = 0.0582$, $wR_2 = 0.0730$
R indices (all data)	$R_1 = 0.0615$, $wR_2 = 0.0778$	$R_1 = 0.0542$, $wR_2 = 0.0777$
Largest diff.e peak and hole (e A^{-3})	0.158 and −0.197	0.221 and −0.267

H, 7.12. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 2.31 (s, 3H, CH_3), 2.40 (s, 6H, CH_3), 6.93 (s, 2H, Ph*H*), 7.16 (d, J = 16.0 Hz, 1H, = C*H*), 7.50 (t, J = 8.0 Hz, 2H, Ph*H*), 7.59 (dd, J = 5.2 Hz and 7.8 Hz, 1H, Ph*H*), 7.98 (m, 3H, = C*H* + Ph*H*) ppm.

Synthesis of (E)-1-phenyl-3-[(4-trifluoromethylphenyl)]prop-2-en-1-one (2)

The procedure was similar to that of **1** except 4-trifluoromethylbenzaldehyde (0.87 g, 5.0 mmol) was used instead of 2,4,6-trimethylbenzaldehyde (0.74 g, 5.0 mmol). Compound **2** (1.35 g, 98%) was obtained as a slightly-yellow solid. Mp: 130–132 °C. Anal. Calcd.

Table 2. Selected bond lengths (Å) and bond angles (°) for (**1**) and (**2**)

Compound (**1**)					
O(1)–C(7)	1.231(2)	C(8)–C(9)	1.340 (2)	C(13)–C(18)	1.511(2)
C(6)–C(7)	1.497(2)	C(9)–C(10)	1.480 (2)	C(15)–C(16)	1.510(2)
C(7)–C(8)	1.476(2)	C(11)–C(17)	1.517 (2)	C(10)–C(15)	1.418(2)
O(1)–C(7)–C(8)	121.50(15)	C(9)–C(8)–C(7)	120.70 (16)	C(10)–C(11)–C(17)	123.21(16)
O(1)–C(7)–C(6)	119.27(15)	C(8)–C(9)–C(10)	126.73 (17)	C(14)–C(13)–C(18)	121.61(15)
C(1)–C(6)–C(7)	121.97(15)	C(11)–C(10)–C(9)	122.80 (15)	C(12)–C(13)–C(18)	120.85(16)
C(5)–C(6)–C(7)	118.45(15)	C(15)–C(10)–C(9)	117.77 (15)	C(14)–C(15)–C(16)	119.80(15)
C(8)–C(7)–C(6)	119.23(16)	C(12)–C(11)–C(17)	117.91 (15)	C(7)–C(8)–C(9)–C(10)	–179.24(15)
Compound (**2**)					
O(1)–C(7)	1.2244(14)	C(8)–C(9)	1.3249 (16)	F(1)–C(16)	1.3361(14)
C(6)–C(7)	1.4897(16)	C(9)–C(10)	1.4715 (16)	F(2)–C(16)	1.3439(14)
C(7)–C(8)	1.4896(16)	C(13)–C(16)	1.4920 (16)	F(3)–C(16)	1.3301(14)
O(1)–C(7)–C(8)	121.12(11)	C(1)–C(6)–C(7)	118.44 (11)	F(3)–C(16)–F(2)	105.91(10)
O(1)–C(7)–C(6)	120.32(11)	C(11)–C(10)–C(9)	117.90 (11)	F(1)–C(16)–F(2)	105.12(9)
C(8)–C(7)–C(6)	118.56(11)	C(15)–C(10)–C(9)	123.42 (11)	F(3)–C(16)–C(13)	113.26(10)
C(9)–C(8)–C(7)	119.56(11)	C(12)–C(13)–C(16)	119.73 (11)	F(1)–C(16)–C(13)	113.02(10)
C(8)–C(9)–C(10)	127.71(12)	C(14)–C(13)–C(16)	119.94 (11)	F(2)–C(16)–C(13)	112.27(10)
C(5)–C(6)–C(7)	122.13(11)	F(3)–C(16)–F(1)	106.62 (10)	C(7)–C(8)–C(9)–C(10)	175.00(10)

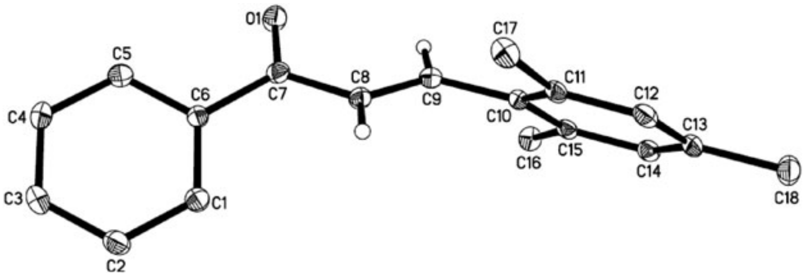


Figure 2. Crystal structure of (**1**) with 30% probability thermal ellipsoids.

(%) for $C_{16}H_{11}F_3O$: C, 69.56, H, 4.01. Found (%): C, 69.38, H, 4.21. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): 7.49 (d, $J = 15.6$ Hz, 1H, = C*H*), 7.55 (d, $J = 7.6$ Hz, 1H, Ph*H*), 7.60 (m, 2H, Ph*H*), 7.68 (d, $J = 8.0$ Hz, 2H, Ph*H*), 7.75 (d, $J = 8.0$ Hz, 2H, Ph*H*), 7.82 (d, $J = 15.6$ Hz, 1H, = C*H*), 8.03 (d, $J = 8.4$ Hz, 2H, Ph*H*) ppm.

Single-Crystal X-ray Diffraction

Single crystals of the title compounds (**1**) and (**2**) suitable for X-ray diffraction analyses were grown by slow evaporation of the CH_2Cl_2/hexane solution (v/v = 1:1) at 5 °C. Two single crystals with dimensions of 0.20 mm × 0.18 mm × 0.12 mm for (**1**) and 0.16 mm × 0.14 mm × 0.12 mm for (**2**) were mounted on a Rigaku Saturn CCD area deterctor. Data were collected at 113 (2) K by using a graphite monochromator with MoKα radiation ($\lambda = 0.71073$ Å) for (**1**) and (**2**) in the ω-ϕ scanning mode. Absorption corrections were performed by SADABS program [10]. The structures were solved by direct methods using the SHELXS-97 program [11] and refined by full-matrix least-squares techniques (SHELXL-97) [12] on F^2. Hydrogen atoms were located using the geometric method. Details of crystal data, data collection, and structure refinement for (**1**) and (**2**) are listed in Table 1. Selected bond lengths and angles for (**1**) and (**2**) are given in Table 2.

Results and Discussion

Spectral Analyses

The as-prepared chalcone compounds were characterized by elemental analysis and ^{1}H NMR spectrum. Their structures were unambiguously determined using X-ray diffraction analysis. The elemental analysis and NMR spectra have been in well agreement with the formulae proposed by the X-ray crystallography for (**1**) and (**2**), respectively.

Crystal Structure Description of the Title Compound (1)

As depicted in Fig. 2, the chalcone molecule in (**1**) consists of a free phenyl ring and a 2,4,6-trimethylsubstituted phenyl ring linked by a $-C(=O)-CH{=}CH-$ unit, in which the (*E*)-configuration of the central propenone is similar to that observed in 1-phenyl-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one [13] but different from the (*Z*)-configuration reported in 1-phenyl-3-(2,4,6-triphenyphenyl)prop-2-en-1-one [14]. In central propenone system, the C8 = C9 bond length (1.340 (2) Å) and the C7 = O1 one (1.231 (2) Å) are much shorter than the C=C bond distance (1.3317 (19) Å or 1.322 (3) Å) and the C=O one

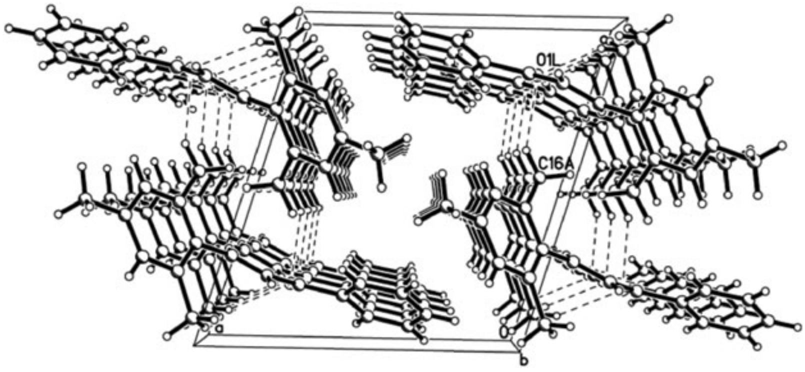


Figure 3. Crystal packing diagram of (**1**) along the *b*-axis.

(1.2244 (17) Å or 1.214 (3) Å) is observed in its analogues [13, 14], respectively. This suggests that (i) in comparasion with the methoxyl and benzene group, the introduction of the methyl group into 2,4,6-positions of the phenyl group low the electron density within the aromatic ring, which is in turn conjugated with the exocyclic propenone moiety [15]; (ii) the extended π-electron conjugated system (—C(=O)—CH=CH—) is mainly induced by the 2,4,6-trimethylsubstituted phenyl group rather than the free phenyl one [16]. Furthermore, defined the C1–C6 phenyl ring as plane-I (r.m.s. deviation 0.0076 Å), the C10–C15 phenyl ring as plane-II (r.m.s. deviation 0.0011 Å), and the central C7–C8 = C9–C10 unit as plane-III (r.m.s. deviation 0.0040 Å), the dihedral angles between them (namely $\mathrm{PL}_{\mathrm{I,II}}$, $\mathrm{PL}_{\mathrm{I,III}}$ and $\mathrm{PL}_{\mathrm{II,III}}$) are 77.0°, 34.4°, and 45.1°, respectively, indicating that the two phenyl rings are rotated in opposite directions relative to the central unit of plane-III [17]. The C7–C8 = C9–C10 torsional angle is 179.2° and the ketone O1 atom deviates from the above-defined plane-III by 0.3039 Å, showing that the C=O bond is not coplanar with the central C7–C8 = C9–C10 moiety [18].

As shown in Fig. 3, the crystal packing of (**1**) is stabilized by the C—H···O short contacts involving the H atoms of the methyl group and the O atoms of the ketone group [19]. For instance, the short nonbonded contact between the O1L atom of the ketone group and the H atom linked to the methxyl C16A atom, which is 2.453 Å, exits in the adjacent molecules. Apart from the short intermolecular contacts, there are weak $\pi \cdots \pi$ stacking interactions involving the phenyl rings of two adjacent molecules [15], in which the phenyl rings are parallel to each other with the centroid-to-centroid separation of 5.661 Å. As

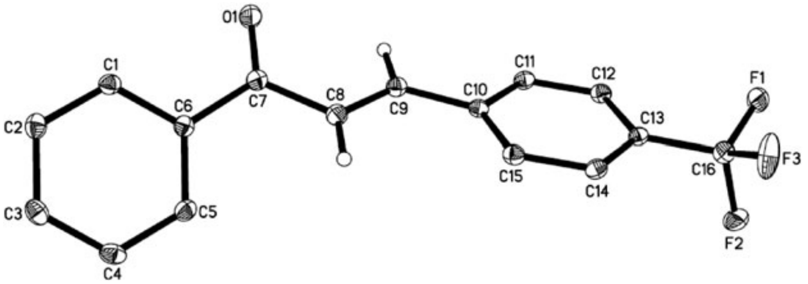


Figure 4. Crystal structure of (**2**) with 30% probability thermal ellipsoids.

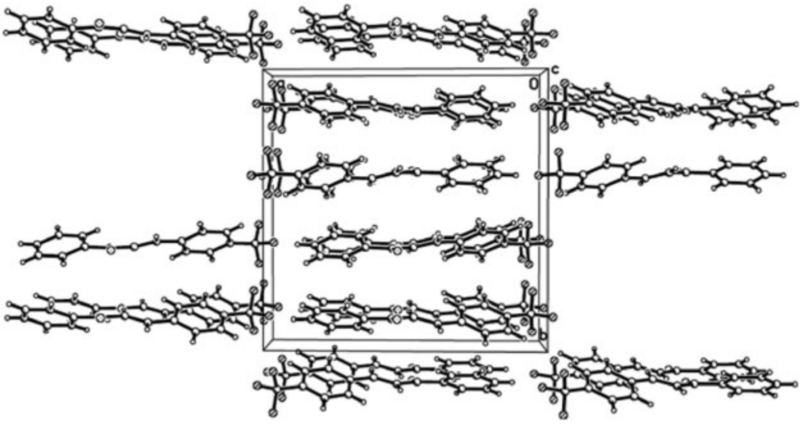


Figure 5. Crystal packing diagram of (**2**) along the *c*-axis.

a result, the two kinds of intermolecular interactions link the molecule into an infinite three-dimensional network (Fig. 3).

Crystal Structure Description of the Title Compound (2)

As depicted in Fig. 4, the chalcone molecule of (**2**) has the similar skeleton of two phenyl rings and one central propenone group as (**1**), where the (*E*)-conformation of the exocyclic olefinic bond agrees quite well with that reported in its analogues such as 1-phenyl-3-(4-fluorophenyl)prop-2-en-1-one [20], 1-phenyl-3-(4-methoxyphenyl)prop-2-en-1-one [21], and 1-phenyl-3-(4-pentyloxyphenyl)prop-2-en-1-one [22], *etc.* In central propenone system, the C8 = C9 bond length (1.3249 (16) Å) and the C7 = O1 one (1.2244 (14) Å) are within the corresponding bond distance (C=C for 1.319–1.333 Å and C=O for 1.180–1.231 Å) found in its analogues [15 , 16 , 20–22], suggesting that the central π-electron conjugated system (−C(=O)−CH=CH−) is mainly induced by the mono-substituent in 4-position of the phenyl ring [16]. Moreover, the dihedral angle between the C10–C15 plane of the 4-trifluoromethylphenyl group and the C7–C8 = C9–C10 plane of the central propenone moiety is 14.2°, which is sharply larger than the corresponding angle (45.1°) in (**1**). The dihedral angle made by the C10–C15 plane of the 4-trifluoromethylphenyl ring and the C1–C6 plane of the free phenyl ring, which is 49.7°, is much greater than that (77.0°) found in (**1**). These observations indicate that the planarity of the entire molecule for (**2**) is much better than that of (**1**), probably because of the steric repulsion of the 2,4,6-trimethylphenyl substituent in (**1**) that is a bigger group with respect to the 4-trifluoromethylphenyl group in (**2**).

As shown in Fig. 5, the $\pi \cdots \pi$ stacking interactions are observed in the crystal packing of (**2**), together with normal van der Waals' contancts. The adjacent molecules are collinear to each other and arranged in layers via the weak $\pi \cdots \pi$ stacking interactions of the parallel adjacent phenyl rings with the centroid-to-centroid separation of 5.843 Å [15].

Conclusions

In summary, two chalcone compounds [i.e., (*E*)-1-phenyl-3-[(2,4,6-trimethylphenyl)]prop-2-en-1-one (**1**) and (*E*)-1-phenyl-3-[(4-trifluoromethylphenyl)]prop-2-en-1-one (**2**)] have been synthesized and structurally characterized elemental analysis and ^{1}H NMR spectroscopy. Particularly, the molecular structures of (1) and (2) were unequivocally determined by single-crystal X-ray diffraction analysis, where the exocyclic olefinic bond reside in a (*E*)-configuration in their solid state. In addition, the weak $\pi \cdots \pi$ stacking interactions are observed in the crystal structures of (1) and (2).

Acknowledgments

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Supplementary Material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 944793 and 944794 for the title compounds (**1**) and (**2**), respectively. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving. html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk.

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