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Preparation of 1,5-Diketones by Addition of Cyclohexanone to Chalcones under Solvent-free Phase Transfer Catalyst Condition

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Eight different chalcone-1,5-diketone derivatives (**5a-h**) were prepared by the reaction of chalcone derivatives (**3a-h**) with cyclohexanone under the solvent-free phase transfer catalyst condition with modarate to high yields. The mechanistic pathway of the reaction can be explained by the Michael-type addition of cyclohexanone to chalcone derivatives (**3a-h**).

Key Words: Chalcones, 1,5-Diketones, Solvent-free, Phase Transfer Catalyst, Michael Addition

Introduction

Chalcones, either natural or synthetic, are known to exhibit various biological activities,[1] such as antioxidant,[2] antiinflammatory,[3] antimalarial,[4] antileishmanial,[5] anticancer[6]and antitumor.[7] In addition, chalcones are very important compounds as a Michael acceptor in organic syntheses. The Michael addition reaction is one of the most fundamental C-C bond-forming reactions in the synthesis of 1,5-dicarbonyl compounds. 1,5-Diketones are extremely important synthetic intermediates in their own right and are desirable starting materials for generating many heterocyclic[8,9] and polyfunctional compounds.[10,11]

The solid-state Michael additions have performed well recently.[12,13] For example, Liu et al. have reported the addition of acetophenone to ferrocenylchalcone as Michael acceptor under the solvent-free condition[14] in which excess amount of acetophenone and NaOH as the catalyst are used. However, there are a few examples related to the addition of cyclic ketones to chalcone derivatives.[15,16] Herein this kind of reaction is described. It is shown that the 1,5-diketones (**5a-h**) containing chalcone can be prepared in considerable yield by Michael-type addition of cyclohexanone to chalcone derivatives (**3a-h**) under solvent-free phase transfer catalyst condition.

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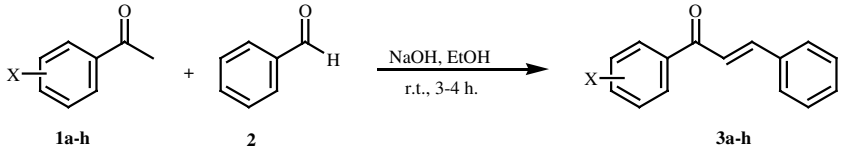
Results and Discussion

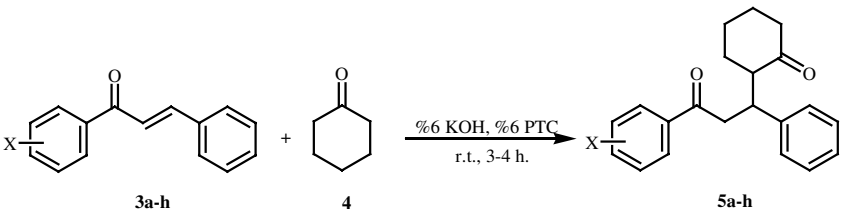
The general synthetic strategy employed to prepare the chalcone derivatives (**3a-h**) was based on Claisen-Schmidt condensation, which was reported previously.[17] As shown in Scheme 1 and Table 1, a series of 8 chalcone derivatives (**3a-h**)[18−27] were prepared by base catalyzed condensation of appropriately substituted acetophenone with benzaldehyde in yields of 69%-97% (Scheme 1). The structures of all the 8 chalcone derivatives (**3a-h**) synthesized in this research were established on the basis of IR, ^{1}H-NMR, and ^{13}C-NMR spectral data.



1a) X = H; **b**) X = *o*-Cl; **c**) *m*-Cl; **d**) X = *p*-Cl; **e**) X = *o*-Br
f) X = *o*-OCH_3; **g**) X = *m*-OCH_3; **h**) X = *p*-OCH_3

Scheme 1

The synthesized chalcones derivatives (**3a-h**) were submitted to the Michael addition reaction. A series of 8 chalcone-1,5-diketones (**5a-h**) was prepared by base catalyzed addition of cyclohexanone to chalcones (**3a-h**) under solvent-free phase transfer catalyst condition. In this reaction, chalcone **3** (1 mole), cyclohexanone **4** (2 mole), KOH (6% mole) and PTC (benzyltriethylammonium chloride) (6% mole) were used. After the purification of the crude products, the chalcone-1,5-diketones (**5a-h**) were obtained in yields of 40%-83% (Scheme 2, Table 1). The advantages of this method are neat conditions, less reaction times, usage of catalytic amount of base.



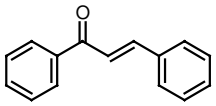
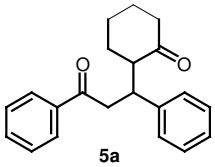
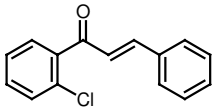
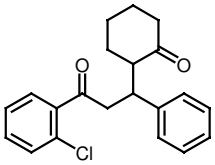
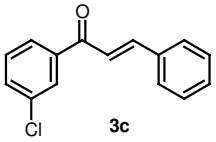
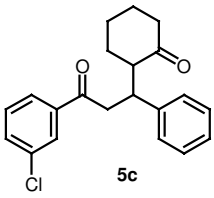
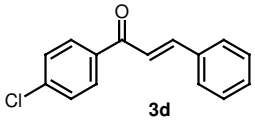
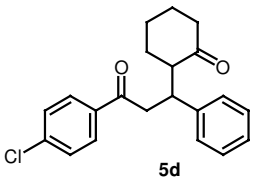
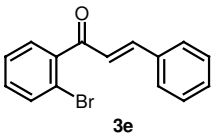
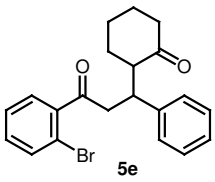
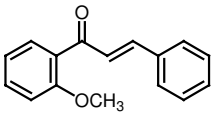
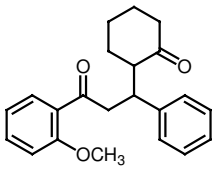
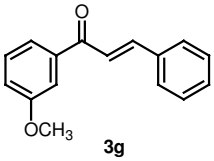
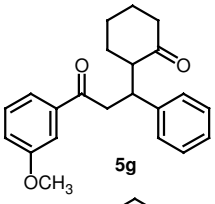
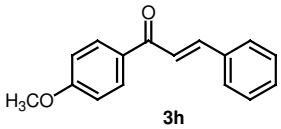
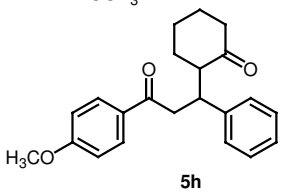
3a) X = H; **b**) X = *o*-Cl; **c**) *m*-Cl; **d**) X = *p*-Cl; **e**) X = *o*-Br
f) X = *o*-OCH_3; **g**) X = *m*-OCH_3; **h**) X = *p*-OCH_3

Scheme 2

It is observed that the Michael-type addition of cyclohexanone to chalcone derivatives (**3a-h**) can be easily carried out and gives product **5** in satisfactory yield. However, the position of substituents affects the reaction yields. While the high yields were achieved in *m*- and *p*-substituted **5a, c, d, g, h**, the low yields were observed to *o*-substituted **5b, e,** and **f** as seen in Table 1. It is assumed that the low yields could be attributed to the steric hindrance of *o*-substituents.

In this series, compounds **5a**[23], **5d**[28] and **5h**[29] are known in the literature. The structures of other 1,5-diketones (**5b, c, e, f** and **g**) were determined on the basis of spectral data (^{1}H-, ^{13}C-NMR, IR, MS, and elemental analysis). In the ^{1}H-NMR spectrum of **5a-h**, the protons of PhCO$\underline{CH_2}$ gave an AB system that is characteristic signals for these compounds. While, part A of the AB system is shown as a doublet of doublet at δ = 3.50 -3.42 (J = 15.7-16.7, 3.9-4.5 Hz) and that of part B as a doublet of doublet at δ = 3.23-3.15 (J = 15.7-16.7, 9.5-9.6 Hz). In addition, all spectral data are consistent with the titled compounds.

Table 1. Prepared 1,5-diketones.

Reagents	Products	Yield (%)[a]	mp (°C)
 **3a**	 **5a**	83	146-148[b]
 **3b**	 **5b**	65	120-124
 **3c**	 **5c**	75	107-109
 **3d**	 **5d**	72	113-116
 **3e**	 **5e**	63	120-122
 **3f**	 **5f**	40	108-111
 **3g**	 **5g**	78	89-92
 **3h**	 **5h**	78	128-130

[a]Isolated yield.

[b]Lit.[16], 148-149 °C.

Experimental

^{1}H and ^{13}C-NMR spectra were recorded with a Varian Gemini 200 MHz and an AC Bruker 400 MHz. As internal standards served TMS (δ 0.00) for ^{1}H-NMR and $CDCl_3$ (δ 77.0) for ^{13}C-NMR spectroscopy Jvalues were given in Hz. IR spectra were recorded on a Jasco FT/IR-430 spectrometer. Mass spectra were taken with a Thermofinnigan Trace GC/Trace DSQ/A1300, (E.I Quadrapole, 70 eV) equipped with a SGE-BPX5 MS capilary column (30 m x 0.25 mm i.d., 0.25 μm). Elemental analyses were obtained using a LECO CHNS 932 elemental analyzer. Melting points were measured on an Electrothermal 9100 apparatus.

General procedure for preparation of 1,5-diketones 5a-h

To a mixture of chalcone **1a** (10 mmol) and cycylohexanone **4** (20 mmol) solid KOH (0.6 mmol) and PTC (0.6 mmol) were added and stirred for 3 or 4 h at room temperature. Then, the mixture was extracted with $CHCl_3$(20 mL) and dried over Na_2SO_4. After the solvent removed in vacua, the product was precipitated in CCl_4/hexane (3:1).

2-(3-oxo-1,3-diphenylpropyl) cyclohexanone (5a): Yield: 83%; colorless solid; mp 146-148 $^\circ$C (CCl_4-hexane, 3:1) (Lit.[16] 148-149 $^\circ$C). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.93-7.89 (m, 2H), 7.55-7.41 (m, 3H), 7.37-7.13 (m, 5H), 3.78-3.68 (m, 1H), 3.50 A part of AB system (dd, 1H, J= 16.2, 4.1 Hz), 3.23 B part of AB system (dd, 1H, J= 16.2, 9.5 Hz), 2.75- 2.68 (m, 1H), 2.53-2.32 (m, 2H), 2.01-1.94 (m, 1H), 1.79-1.51 (m, 4H), 1.49-1.24 (m, 1H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.6, 200.8, 144.1, 139.1, 134.8, 130.4 (2C), 130.4 (2C), 130.3 (2C), 130.2 (2C), 128.6, 57.8, 46.2, 44.3, 43.1, 34.5, 30.5, 26.1. **IR** (KCl): 3056, 33025**,** 2939, 2918, 2854, 1708, 1683, 1596, 1446, 1340, 1216, 746, 696, 567 cm^{-1}. **MS** m/z (relative intensity): 306.5 (M^+, 1), 287.5 (3), 209.3 (7), 187.2 (98), 105.0 (100), 77.0 (79). **Anal. Calcd** for $C_{21}H_{22}O_2$(306.40): C 82.32, H 7.24; found: C 81.98, H 7.22.

2-(3-(2-chlorophenyll)-3-oxo-1-phenylpropyl) cyclohexanone (5b): Yield: 65%; colorless solid; mp 121-124 $^\circ$C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.43-7.11 (m, 9H), 3.71-3.59 (m, 1H), 3.46 (dd, 1H, J= 16.6, 4.5 Hz), 3.23 (dd, 1H, J= 16.6, 9.5 Hz), 2.72-2.60 (m, 1H), 2.55-1.22 (m, 8H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.2, 203.8, 143.7, 141.5, 132.7, 133.3, 132.2 (2C), 130.9, 130.5, 130.4 (2C), 128.7, 128.6, 57.7, 50.2, 44.2, 43.0, 34.2, 30.4, 26.0. **IR** (KCl): 3058, 3026, 2933, 2918, 2854, 1705, 1691, 1431, 1369, 1122, 1072, 983, 750, 721, 567 cm^{-1}. **MS** m/z (relative intensity): 340.7 (M^+, 2), 322.5 (6), 293.4 (15), 243.3 (100), 187.3 (81), 139.0 (61), 77.1 (3). **Anal. Calcd** for $C_{21}H_{21}ClO_2$(340.84): C 74.00, H 6.21; found: C 73.74, H 6.23.

2-(3-(3-chlorophenyll)-3-oxo-1-phenylpropyl) cyclohexanone (5c): Yield: 75%; colorless solid; mp 107-109 $^\circ$C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.87-7.79 (m, 2H), 7.50-7.14 (m, 7H), 3.72-3.62 (m, 1H), 3.50 (dd, 1H, J= 16.1, 3.9 Hz), 3.17 (dd, 1H, J= 16.1, 9.6 Hz), 2.79-2.66 (m, 1H), 2.55-2.39 (m, 2H), 2.02-1.98 (m, 1H), 1.83-1.52 (m, 4H), 1.28-1.22 (m, 1H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.5, 199.4, 143.8, 140.6, 136.7, 134.7, 131.8, 130.5 (2C), 130.3, 130.2 (2C), 128.7, 128.3, 57.7, 46.5, 44.4, 43.2, 34.6, 30.6, 26.3. **IR** (KCl): 3066, 3030, 2941, 2922, 2850, 1707, 1683, 1413, 1363, 1226, 1215, 1124, 700, 569 cm^{-1}. **MS** m/z (relative intensity): 340.7 (M^+, 1), 322.5 (3), 293.3 (10), 243.3 (62), 187.2 (100), 139.1 (40), 77.1 (3.5). **Anal. Calcd** for $C_{21}H_{21}ClO_2$(340.84): C 74.00, H 6.21; found: C 73.86, H 6.25.

2-(3-(4-chlorophenyll)-3-oxo-1-phenylpropyl) cyclohexanone (5d): Yield: 72%; colorless solid; mp 113-116 $^\circ$C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.94-7.83 (m, 2H), 7.42-7.29 (m, 2H), 7.27-7.12 (m, 5H), 3.68-3.61 (m, 1H) ,3.54 (dd, 1H, J= 15.8, 4.0 Hz,), 3.15 (dd, 1H,J= 15.8,

9.6 Hz), 2.74-2.66 (m, 1H), 2.52-2.35 (m, 2H), 1.99-1.84 (m, 1H), 1.82-1.57 (m, 4H), 1.54-1.21 (m, 1H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.7, 199.6, 143.7, 141.2, 137.4, 131.6 (2C), 130.7 (2C), 130.5 (2C), 130.3 (2C), 128.7, 57.8, 46.4, 44.5, 43.4, 34.7, 30.6, 26.3. IR (KCl): 3057, 3024, 2939, 2918, 2852, 1707, 1685, 1589, 1446, 1398, 1215, 1095, 982, 816, 696, 57 cm^{-1} **MS** m/z (relative intensity): 340.6 (M^+, 1), 322.5 (1), 293.0 (6), 243.3 (60), 187.2 (100), 139.0 (49), 77.1 (2). **Anal. Calcd** for $C_{21}H_{21}ClO_2$(340.84): C 74.00, H 6.21; found: C 73.68, H 6.26.

2-(3-(2-bromophenyl)-3-oxo-1-phenylpropyl) cyclohexanone (5e): Yield: 63%; colorless solid; mp 120-122 °C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.53-7.49 (m, 1H), 7.31-7.09 (m, 8H), 3.72-3.60 (m, 1H), 3.46 (dd, 1H, J= 16.7, 4.4 Hz), 3.21 (dd, 1H, J= 16.7, 9.5 Hz), 2.73-2.32 (m, 1H), 2.55-2.32 (m, 2H), 1.98-1.83 (m, 1H), 1.80-1.49 (m, 4H), 1.32-1.22 (m, 1H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.2, 204.5, 143.6, 143.6, 135.5, 133.3, 130.5 (2C), 130.5 (2C), 130.3, 129.2, 128.7, 120.6, 57.6, 49.9, 44.2, 42.9, 34.2, 30.4, 26.0. **IR** (KCl): 3055, 3026, 2933, 2918, 2854, 1705, 1693, 1404, 1369, 1122, 1030, 983, 750, 698, 567 cm^{-1}. **MS** m/z (relative intensity): 384.4 (M^+, 0.5), 366.5 (3), 289.3 (61), 287.2 (74), 286.3 (26), 187.1 (100), 185.1 (70), 183.1 (55), 77.0 (5.5). **Anal. Calcd** for $C_{21}H_{21}BrO_2$(385.29): C 65.46, H 5.49; found: C 65.03, H 5.93.

2-(3-(2-methoxyphenyl)-3-oxo-1-phenylpropyl) cyclohexanone (5f): Yield: 40%; colorless solid; mp 108-111 °C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.43-7.41 (m, 2H), 7.38-7.12 (m, 5H), 6.95-6.85 (m, 2H), 3.83 (s, 3H), 3.80-3.68 (m, 1H), 3.38-3.34 (m, 2H), 2.72-2.28 (m, 3H), 1.95-1.24 (m, 6H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.4, 203.23, 160.1, 144.5, 134.9, 132.1, 130.9, 130.6 (2C), 130.2 (2C), 128.3, 122.5, 113.3, 57.9, 57.5, 50.8, 43.8, 42.6, 33.8, 30.3, 25.6. **IR** (KCl): 3058, 3026, 2925, 2854, 1703, 1666, 1483, 1433, 1284, 1242, 1022, 752, 698, 567 cm^{-1}. **MS** m/z (relative intensity): 336.6 (M^+, 0.5), 318.5 (41), 239.3 (42), 187.3 (12), 135.0 (100), 77.0 (9). **Anal. Calcd** for $C_{22}H_{24}O_3$(336.42): C 78.54, H 7.19; found: C 78.15, H 7.48.

2-(3-(3-methoxyphenyl)-3-oxo-1-phenylpropyl) cyclohexanone (5g): Yield: 78%; colorless solid; mp 89-92 °C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (200 MHz, $CDCl_3$): δ = 7.55-7.43 (m, 2H), 7.42-7.01 (m, 7H), 3.81 (s, 3H), 3.78-3.66 (m, 1H), 3.50 (dd, 1H, J= 16.1, 4.0 Hz), 3.20 (dd, 1H, J= 16.1, 9.6 Hz), 2.79-2.55 (m, 1H), 2.52-2.38 (m, 2H), 1.99-1.56 (m, 5H), 1.28-1.22 (m, 1H). ^{13}**C-NMR** (50 MHz, $CDCl_3$): δ = 215.5, 200.6, 161.7, 143.9, 140.4, 131.4, 130.5 (2C), 130.4 (2C), 128.6, 122.8, 121.5, 114.5, 57.8, 57.4, 46.4, 44.4, 43.3, 34.5, 30.6, 26.2. **IR** (KCl): 3058, 3028, 2931, 2912, 2852, 1709, 1678, 1581, 1431, 1259, 1049, 987, 700, 573 cm^{-1}. **MS** m/z (relative intensity): 336.5 (M^+, 10), 318.5 (6), 239.4 (100), 187.2 (89), 150.0 (77), 135.1 (71), 77.1 (13). **Anal. Calcd** for $C_{22}H_{24}O_3$(336.42): C 78.54, H 7.19; found: C 78.20, H 7.42.

2-(3-(4-methoxyphenyl)-3-oxo-1-phenylpropyl) cyclohexanone (5h): Yield: 68%; colorless solid; mp 128-130 °C (CCl_4-hexane, 3:1). ^{1}**H-NMR** (400 MHz, $CDCl_3$): δ = 7.91-7.89 (m, as brd., 2H,J = 8.8 Hz, AA' part of AA'XX' system), 7.26-7.23 (m, 2H), 7.18-7.14 (m, 3H), 6.91-6.87 (m, as brd., 2H,J = 8.8 Hz, XX' part of AA'XX' system),3.84 (s, 3H), 3.75-3.69 (dt, J = 9.8, 4.4 Hz, 1H), 3.42 (dd, 1H, J= 15.7, 4.0 Hz), 3.16 (dd, 1H, J= 15.7, 9.5 Hz), 2.75-2.69 (dt, J = 9.8 5.1 Hz, 1H), 2.55-2.48 (m, 1H), 2.42-2.35 (, 1H), 2.00-1.93 (m, 1H), 1.80-1.72 (m, 2H), 1.68-1.61 (m, 1H), 1.59-1.50 (m, 1H), 1.31-1.26 (m, 1H). ^{13}**C-NMR** (100 MHz, $CDCl_3$): δ = 213.9, 197.5, 163.5, 142.3, 130.7 (2C), 130.4, 128.7 (2C), 128.6 (2C), 126.8, 113.8 (2C), 56.1, 55.6, 44.1, 42.5, 41.5, 32.6, 28.7, 24.2. **IR** (KCl): 3057, 3026, 2933, 2852, 1707, 1672, 1603, 1420, 1255, 1167, 984, 816, 698, 565 cm^{-1}. **MS** m/z (relative intensity): 336.4 (M^+, 4), 318.5 (2.5), 239.3 (43), 187.2 (5), 150.1 (100), 135.0 (61), 77.0 (2). **Anal. Calcd** for $C_{22}H_{24}O_3$(336.42): C 78.54, H 7.19; found: C 78.30, H 7.18.

Conclusion

In conclusion, the Michael addition of cyclohexanone to chalcone derivatives **3a-h** under solvent-free phase transfer catalyst condition is a fast, mild, cheap and simple method to prepare chalcone-1,5-diketones (**5a-h**). Products are isolated by reasonably high yields with simple extraction into organic solvent.

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