A solvent-free protocol for facile condensation of indoles with carbonyl compounds using silica chloride as a new, highly efficient, and mild catalyst

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Abstract: A simple and efficient solvent-free procedure for the preparation of bis(indolyl)methanes via electrophilic substitution reactions of indoles with aldehydes and ketones is described. The reactions took place in the presence of a catalytic amount of silica chloride at room temperature. The advantages of this method are high yields, short reaction times, low cost, and compliance with green-chemistry protocols.

Key words: silica chloride, indole, carbonyl compound, solvent-free, bis(indolyl)methane.

Résumé : On décrit une méthode très simple et efficace n'impliquant pas de solvant pour la préparation de bis(indolyl)méthanes qui s'effectue par le biais d'une réaction de substitution électrophile d'indoles avec des aldéhydes et des cétones, à la température ambiante, en présence d'une quantité catalytique de chlorure de silicium. Les avantages de cette méthode sont ses rendements élevés, ses courts temps de réaction, ses coûts peu élevés et leur appariement avec des protocoles de chimie verte.

Mots-clés : chlorure de silicium, indole, composés carbonylés, sans solvant, bis(indolyl)méthane.

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Introduction

Indole derivatives are known for their vast applications in material sciences (1), agrochemicals (2), and pharmaceuticals (3). Among these compounds, the substrates including bis(indolyl)methane moieties, such as secondary metabolites (4) and marine-sponge alkaloids (5), are important classes of bioactive metabolites. Therefore, there is a great interest in the synthesis of this class of compounds. Electrophilic substitution reaction of indole with carbonyl compounds has been used as a useful route toward the synthesis of bis(indolyl)methanes. Different reagents and catalysts have been employed to accomplish this transformation, such as acetic acid (6*a*), $InCl_3$ (6*b*), $In(OTf)_3$ (6*c*), InF_3 (6*d*), $Dy(OTf)_3$ (6*e*), $Ln(OTf)_3$ (6*f*), $FeCl_3$ (6*g*), NBS (6*h*), $PPh_3{\cdot}HClO_4$ (TPP) (6*i*), trichloro-1,3,5-triazine (6*j*), $AlPW_{12}O_{40}$ (6*k*), $ZrOCl_2{\cdot}8H_2O$ (6*l*), and Ru(III) (6*m*). The synthesis of bis(indolyl)methanes in the absence of a catalyst has been also reported (6*n*). However, some of the reported methods are associated with the following drawbacks: (*i*) the use of expensive reagents (6*c*, 6*e*, 6*f*, 6*k*), (*ii*) long reaction times (6*a*, 6*b*, 6*d*), (*iii*) low yields (6*a*), and (*iv*) the use of an additional microwave oven (6*g*). Because of the wide range of biological, industrial, and synthetic applications of bis(indolyl)methanes, their preparation has received renewed interest.

Solvent-free organic reactions have been used as a useful technique in organic synthesis. Solid-state protocols often lead to shorter reaction times, higher yields, easier workup, compliance with green-chemistry protocols, and may enhance the regio- and stereo-selectivity of the reactions (7). Solvent-free condensation of indoles with carbonyl compounds is scarce in the literature (6*l*, 8). Having those aspects in mind and also in continuation of our previous studies to explore new applications of silica chloride (9), we report a new, clean, and efficient solvent-free method for the condensation of indoles with carbonyl compounds. The method involves the use of a catalytic amount of silica chloride under extremely mild conditions (Scheme 1).

Results and discussion

First, to optimize the reaction conditions, silica chloride was used as a catalyst for the condensation of indole (2.1 mmol) with benzaldehyde (1 mmol), as a model reac-

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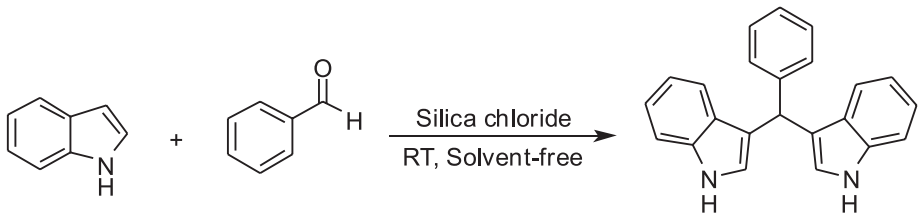
Scheme 1. Condensation of indole with benzaldehyde.



tion under solvent-free conditions (Scheme 1). Interestingly, the reaction proceeded efficiently at room temperature, and the desired bis(indolyl)methane was isolated in 93% yield after 10 min. The reaction was also examined in the presence of silica gel without using silica chloride. In these conditions, the reaction was unsuccessful, and 55% of the starting materials remained intact plus an undesired product, which has not been identified. Therefore, silica chloride was used as a catalyst for all reactions in the absence of solvent.

To compare the efficiency of the solvent-free vs. solution conditions, a model reaction was examined in several solvents, including CH_2Cl_2, $CHCl_3$, MeCN, THF, benzene, DMF, and DMSO. Thus, a mixture of indole (2.1 mmol), benzaldehyde (1 mmol), and silica chloride (0.5 g) in the appropriate solvent (10 mL) was stirred at room temperature for 4 h. Moderate yields of the product were obtained in these conditions; therefore, the solvent-free method is more efficient.

To investigate the capacity and efficiency of our catalyst, the condensation of indoles with a variety of aromatic and aliphatic aldehydes and ketones was carried out using this catalyst (Table 1). As Table 1 indicates, the reactions proceeded efficiently, and the desired products were obtained in good to excellent yields and in short reaction times.

Notably, the electronic properties of the aromatic ring of carbonyl compounds affected the results of the reaction. When an electron-withdrawing substituent, such as NO_2, was present on the aromatic ring, reaction times were decreased; however, the yields did not change significantly (Table 1, entries 2, 3, and 14). Electron-donating substituents (e.g., Me or OMe) had negligible effect on reaction times, but caused the reaction yields to decrease (Table 1, entries 6 and 7). Aliphatic aldehydes as well as ketones needed longer reaction times in comparison with aromatic aldehydes (Table 1, entries 10, 11, 12, and 15).

In conclusion, we have introduced a highly efficient catalyst for the condensation of indoles with aldehydes and ketones in the absence of solvent under mild reaction conditions. The promising points for the presented methodology are high conversion, ease of handling and low cost of the catalyst, cleaner reaction profile, and short reaction times, which make it a useful and attractive process for the rapid synthesis of substituted bis(indolyl)methanes.

Experimental

All chemicals were purchased from Merck or Fluka Chemical Companies. Silica chloride was prepared by the reaction of silica gel (item 7731 for TLC from Merck, Darmstadt, FRG) with thionyl chloride according to the literature procedure (10). ^{1}H NMR (250 MHz) and ^{13}C NMR (62.5 MHz) were run on a Bruker AVANCE DPX-250 FTNMR spectrometer. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes and are uncorrected.

General procedure for condensation of indole with benzaldehyde

To a mixture of indole (0.25 g, 2.1 mmol) and benzaldehyde (0.11 g, 1 mmol) was added silica chloride (0.5 g) and grinded at room temperature for 10 min. Afterward, the reaction mixture was suspended in acetone (20 mL) and filtered. The solvent was evaporated, and the crude product was purified by plate chromatography or recrystallization.

*3-((1*H*-Indol-3-yl)(phenyl)methyl)-1*H*-indole (1)*

Recrystallization from chloroform – petroleum ether (1:1) gave pink solid; yield: 0.301 g (93%); mp 140–142 °C (lit. 142–144 °C) (9). IR (KBr, cm^{-1}): 3402, 3051, 1618, 1600. ^{1}H NMR ($CDCl_3$, ppm) δ: 5.86 (s, 1H, ArCH), 6.66 (s, 2H), 7.11 (t, 2H, *J* = 6.9 Hz), 7.14–7.22 (m, 3H), 7.28–7.31 (m, 2H), 7.35–7.42 (m, 6H), 7.93 (br, 2H, NH). ^{13}C NMR ($CDCl_3$) δ: 31.6, 110.9, 111.9, 118.4, 119.5, 121.2, 124.0, 126.3, 127.1, 128.5, 128.6, 137.0, 145.2.

*3-((1*H*-Indol-3-yl)(4-nitrophenyl)methyl)-1*H*-indole (2)*

Recrystallization from EtOAc – petroleum ether (1:1) gave yellow needles; yield: 0.352 g (96%); mp 217–219 °C (lit. 220–222 °C) (6*c*). IR (KBr, cm^{-1}): 3423, 3051, 1593, 1510, 1458, 1337. ^{1}H NMR ($CDCl_3$, ppm) δ: 6.01 (s, 1H, ArCH), 6.72 (s, 2H), 7.02–7.08 (m, 4H), 7.36 (d, 2H, *J* = 8.1 Hz), 7.41 (d, 2H, *J* = 8.1 Hz), 7.49 (d, 2H, *J* = 8.6 Hz), 8.05 (br, 2H, NH), 8.17 (d, 2H, *J* = 8.6 Hz).

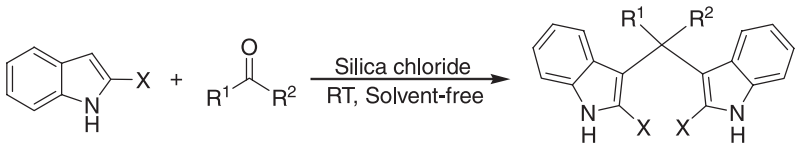
*3-((1*H*-Indol-3-yl)(3-nitrophenyl)methyl)-1*H*-indole (3)*

Recrystallization from EtOAc – petroleum ether (1:1) gave yellow solid; yield: 0.350 g (95%); mp 220–222 °C (lit. 221–223 °C) (11). IR (KBr, cm^{-1}): 3403, 3045, 2917, 1520, 1453, 1350. ^{1}H NMR ($CDCl_3$, ppm) δ: 6.23 (s, 1H, ArCH), 7.01 (t, 2H, *J* = 7.3 Hz), 7.06 (s, 2H), 7.22 (t, 2H, *J* = 7.0 Hz), 7.45 (d, 2H, *J* = 7.5 Hz), 7.53 (d, 2H, *J* = 8.0 Hz), 7.75 (m, 1H), 8.02 (d, 1H, *J* = 7.6 Hz), 8.26 (d, 1H, *J* = 7.7 Hz), 8.33 (s, 1H), 8.09 (s, 2H, NH).

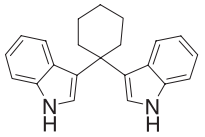
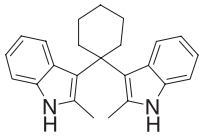
*3-((4-Chlorophenyl)(1*H*-indol-3-yl)methyl)-1*H*-indole (4)*

Recrystallization from chloroform – petroleum ether (1:1) gave pink solid; yield: 0.328 g (92%); mp 78–80 °C (lit. 76–78 °C) (12). IR (KBr, cm^{-1}): 3412, 3056, 1483. ^{1}H NMR ($CDCl_3$, ppm) δ: 5.86 (s, 1H, ArCH), 6.65 (s, 2H), 6.85–7.96 (m, 12H), 8.00 (br, 2H, NH).

Table 1. Condensation of indoles with aldehycdes and ketones using silica chloride.



1–15

Entry	Product[a]	Time (min)	Yield[b] (%)
1	X = H, R^1 = C_6H_5, R^2 = H (**1**)	10	93
2	X = H, R^1 = *p*-$NO_2C_6H_4$, R^2 = H (**2**)	5	96
3	X = H, R^1 = *m*-$NO_2C_6H_4$, R^2 = H (**3**)	7	95
4	X = H, R^1 = *p*-ClC_6H_4, R^2 = H (**4**)	10	92
5	X = H, R^1 = *o*-ClC_6H_4, R^2 = H (**5**)	20	81
6	X = H, R^1 = *p*-MeC_6H_4, R^2 = H (**6**)	10	87
7	X = H, R^1 = *p*-$OMeC_6H_4$, R^2 = H (**7**)	12	84
8	X = H, R^1 = 4-Pyridyl, R^2 = H (**8**)	8	92
9	X = H, R^1 = 2-Furyl, R^2 = H (**9**)	15	90
10	X = H, R^1 = *n*-Butyl, R^2 = H (**10**)	20	85
11	X = H, R^1 = C_6H_5, R^2 = Me (**11**)	20	70
12	 (**12**)	20	75
13	X = Me, R^1 = C_6H_5, R^2 = H (**13**)	15	91
14	X = Me, R^1 = *p*-$NO_2C_6H_5$, R^2 = H (**14**)	10	92
15	 (**15**)	25	73

[a]All compounds were identified by comparison of their melting points with the authentic samples and (or) IR as well as ^{1}H NMR data.
[b]Isolated yield.

3-((2-Chlorophenyl)(1H-indol-3-yl)methyl)-1H-indole (5)

Recrystallization from chloroform – petroleum ether (1:1) gave pink solid; yield: 0.288 g (81%); mp 74–76 °C (lit. 78–80 °C) (12). IR (KBr, cm^{-1}): 3412, 3053, 1617, 1455. ^{1}H NMR ($CDCl_3$, ppm) δ: 6.32 (s, 1H, ArCH), 6.67 (s, 2H), 7.02 (t, 2H, *J* = 7.8 Hz), 7.10–7.22 (m, 6H), 7.38–7.43 (m, 4H), 7.98 (br, 2H, NH).

3-((1H-Indol-3-yl)(p-tolyl)methyl)-1H-indole (6)

Recrystallization from chloroform – petroleum ether (1:2) gave pink solid; yield: 0.293 g (87%); mp 95–97 °C (lit. 94–96 °C) (12). IR (KBr, cm^{-1}): 3411, 3040, 2932, 1603, 1512. ^{1}H NMR ($CDCl_3$, ppm) δ: 2.33 (s, 3H, $ArCH_3$), 5.87 (s, 1H, ArCH), 6.69 (s, 2H), 7.04 (t, 2H, *J* = 7.1 Hz), 7.12 (d, 2H, *J* = 7.1 Hz), 7.23–7.28 (m, 6H), 7.41 (d, 2H, *J* = 7.2 Hz), 7.94 (br, 2H, NH).

3-((1H-Indol-3-yl)(4-methoxyphenyl)methyl)-1H-indole (7)

Recrystallization from chloroform – petroleum ether (1:1) gave brown solid; yield: 0.295 g (84%); mp 186–188 °C (lit. 187–189 °C) (13). IR (KBr, cm^{-1}): 3410, 2930, 1610, 1508. ^{1}H NMR ($CDCl_3$, ppm) δ: 3.76 (s, 3H, OCH_3), 5.85, (s, 1H, ArCH), 6.68 (s, 2H), 6.91 (d, 2H, *J* = 8.2 Hz), 7.02 (t, 2H, *J* = 7.3 Hz), 7.16 (t, 2H, *J* = 7.3 Hz), 7.20 (m, 2H), 7.35–7.41 (m, 4H), 7.95 (br, 2H, NH).

3-((1H-Indol-3-yl)(pyridin-4-yl)methyl)-1H-indole (8)

Purification by plate chromatography, eluted with EtOAc – petroleum ether (1:1), gave brown solid; yield: 0.299 g

(92%); mp 161–163 °C (lit. 163–165 °C) (6*j*). ^{1}H NMR ($CDCl_3$, ppm) δ: 5.96 (s, 1H, ArCH), 6.67 (s, 2H), 7.08–7.15 (m, 4H), 7.36 (d, 2H, *J* = 8.1 Hz), 7.43 (d, 2H, *J* = 8.1 Hz), 7.37 (d, 2H, *J* = 7.9 Hz), 7.99 (br, 2H, NH), 8.34 (d, 2H, *J* = 7.9 Hz).

3-(Furan-2-yl(1*H*-indol-3-yl)methyl)-1*H*-indole (9)

Recrystallization from EtOAc – petroleum ether (1:1) gave brown solid; yield: 0.282 g (90%); mp > 300 °C (lit. >300 °C) (12). IR (KBr, cm^{-1}): 3410, 1712, 1450. ^{1}H NMR ($CDCl_3$, ppm) δ: 5.97 (s, 1H, ArCH), 6.90 (s, 2H), 7.08–7.43 (m, 11H), 8.00 (br, 2H, NH).

3-(1-(1*H*-Indol-3-yl)pentyl)-1*H*-indole (10)

Purification by plate chromatography, eluted with EtOAc – petroleum ether (1:2), gave colorless solid; yield: 0.256 g (85%); mp 80–82 °C (6*l*). IR (KBr, cm^{-1}): 3448, 3070, 2962, 1510. ^{1}H NMR ($CDCl_3$, ppm) δ: 0.91 (t, 3H, *J* = 6.8 Hz), 1.29 (m, 2H), 1.61 (m, 2H), 2.30 (m, 2H), 4.75 (t, 1H, *J* = 6.7 Hz), 6.89 (s, 2H), 7.05–7.13 (m, 4H), 7.28 (d, 2H, *J* = 7.9 Hz), 7.52 (d, 2H, *J* = 7.9 Hz), 7.91 (br, 2H, NH).

3-(1-(1*H*-Indol-3-yl)-1-phenylethyl)-1*H*-indole (11)

Purification by plate chromatography, eluted with EtOAc – petroleum ether (1:1), gave colorless solid; yield: 0.236 g (70%); mp 164–166 °C (lit. 165–167 °C) (11). IR (KBr, cm^{-1}): 3400, 3065, 2973, 1595. ^{1}H NMR ($CDCl_3$, ppm) δ: 2.41 (s, 3H, CH_3), 6.65 (s, 2H), 6.95 (t, 2H, *J* = 7.5 Hz), 7.16 (t, 2H, *J* = 7.5 Hz), 7.27–7.44 (m, 9H), 7.92 (br, 2H, NH).

3-(1-(1*H*-Indol-3-yl)cyclohexyl)-1*H*-indole (12)

Purification by plate chromatography, eluted with chloroform – petroleum ether (1:1), gave colorless solid; yield: 0.247 g (75%); mp 163–165 °C (11). IR (KBr, cm^{-1}): 3442, 3055, 2972, 1512. ^{1}H NMR ($CDCl_3$, ppm) δ: 1.61 (m, 2H), 1.69 (m, 4H), 2.54–2.57 (m, 4H), 6.91 (s, 2H), 7.05–7.12 (m, 4H), 7.34 (d, 2H, *J* = 7.8 Hz), 7.58 (d, 2H, *J* = 7.5 Hz), 7.94 (br, 2H, NH).

2-Methyl-3-((2-methyl-1*H*-indol-3-yl)(phenyl)methyl)-1*H*-indole (13)

Purification by plate chromatography, eluted with chloroform – petroleum ether (1/2), gave pink solid; yield: 0.319 g (91%); mp 245–247 °C (6*l*). IR (KBr, cm^{-1}): 3405, 3064, 1595, 1512. ^{1}H NMR ($CDCl_3$, ppm) δ: 2.05 (s, 6H), 5.88 (s, 1H, ArCH), 6.78–7.01 (m, 6H), 7.14–7.22 (m, 5H), 7.28–7.31 (m, 2H), 8.02 (br, 2H, NH).

2-Methyl-3-((2-methyl-1*H*-indol-3-yl)(4-nitrophenyl)methyl)-1*H*-indole (14)

Recrystallization from EtOAc – petroleum ether (1:1) gave yellow solid; yield: 0.364 g (92%); mp 241–243 °C (6*l*); IR (KBr, cm^{-1}): 3398, 3051, 1590, 1515. ^{1}H NMR ($CDCl_3$, ppm) δ: 2.13 (s, 6H), 5.96 (s, 1H), 6.71–6.96 (m, 6H), 7.21 (d, 2H, *J* = 8.0 Hz), 7.47 (d, 2H, *J* = 8.5 Hz), 8.03 (d, 2H, *J* = 8.5 Hz), 8.04 (br, 2H, NH).

2-Methyl-3-(1-(2-methyl-1*H*-indol-3-yl)cyclohexyl)-1*H*-indole (15)

Purification by plate chromatography, eluted with chloroform – petroleum ether (1:1) gave colorless solid; yield: 0.262 g (73%); mp 81–84 (6*l*). IR (KBr, cm^{-1}): 3450, 3043, 2981, 1515. ^{1}H NMR ($CDCl_3$, ppm) δ: 1.55 (d, 2H, *J* = 4.1 Hz), 1.73 (d, 4H, *J* = 4.0 Hz), 2.11 (s, 6H), 2.52–2.56 (m, 4H), 6.71–6.96 (m, 6H), 7.22 (d, 2H, *J* = 7.9 Hz), 7.98 (br, 2H, NH).

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