ISSN 1070-3632, Russian Journal of General Chemistry, 2016, Vol. 86, No. 5, pp. 1144–1150. © Pleiades Publishing, Ltd., 2016.

Efficient Synthesis of Bis-indolyloxindoles from (Phenylimino)indolin-2-ones and 1*H*-Indole Catalyzed by *p*-Toluenesulfonic Acid[1]

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Received November 19, 2015

Abstract—*p*-Toluenesulfonic acid (*p*-TSA) efficiently catalyzed the reaction of (phenylimino)indolin-2-ones with 1*H*-indole in dichloromethane at room temperature to afford 3,3-di(indolyl)indolin-2-ones in 2-3 min with high yields.

Keywords: 3,3-di(indolyl)indolin-2-ones, 1*H*-indole, (phenylimino)indolin-2-ones

DOI: 10.1134/S1070363216050273

INTRODUCTION

Oxindole is a structural block of a variety of pharmacologically and biologically active compounds [1, 2]. 3,3-Diaryloxindoles exhibit mechanism-specific antibacterial, antiprotozoal, antiproliferative, and anti-inflammatory activities [3]. These compounds have also been used as laxatives [4] and lead compounds for Ca_2C-depletion mediated inhibition of translation initiation [5]. The parent compound, trisindoline, is a natural product and has been isolated by Kabayshi and co-workers [6]. Ru(III)-catalyzed synthesis of 3,3-di-(indolyl)indolin-2-ones by the reaction of (phenyl-imino)indolin-2-ones and indoles was presented [7]. The use of mild acids is essential in developing efficient methods of synthesis of 3,3-di(indolyl)oxindoles from (phenylimino)indolin-2-ones and indoles. In some protocols sulfonic acids have been used as catalysts [8]. Some other methods have also been reported for the synthesis of this class of compounds [9].

p-Toluenesulfonic acid (*p*-TsOH) has received considerable attention as a nontoxic, inexpensive and readily available catalyst for various organic reactions that lead to the corresponding products with high selectivity and excellent yields [10-12]. Therefore, *p*-TsOH could be an effective Bronshed acid in activation of (phenylimino)indolin-2-ones and 1*H*-indole under mild conditions. There have been no reports on the use of *p*-TsOH in the synthesis of 3,3-di(indolyl)-oxindoles from (phenylimino)indolin-2-ones.

As a part of development of new synthetic methods [13] we, herein, disclose our results on *p*-TsOH catalyzed synthesis of 3,3-di(indolyl)oxindoles from (phenylimino)indolin-2-ones and 1*H*-indole.

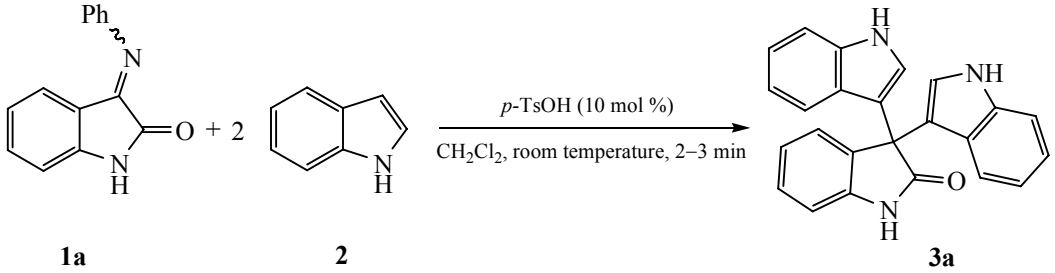
RESULTS AND DISCUSSION

Initially, we have studied the reaction of 3-(phenyl-imino)indolin-2-one (**1a**) with 1*H*-indole **2** in the presence of 10 mol % of *p*-TsOH in CH_2Cl_2 at room temperature (Scheme 1). The reaction reached its completion in 2–3 min and gave 3,3-di(1*H*-indol-3-yl)-indolin-2-one (**3a**) (85%, see the table).

Encouraged by that result, we turned our attention to various substituted 3-(phenylimino)indolin-2-ones. 5-Methyl-3-(phenylimino)indolin-2-one (**1b**) reacted smoothly with **2** to give the corresponding 3,3-di(1*H*-indol-3-yl)-5-methylindolin-2-one (**3b**). The scope of the reaction was exemplified with a disubstituted compound, 5,7-dimethyl-3-(phenylimino)indolin-2-one (**1c**), to produce the desired product **3c**. Thus, 1*H*-indole was used as a representative starting material. The reaction was performed with various substituted (phenylimino)indolin-2-ones such as 5-methoxy, 5-fluoro, 5-chloro, 5-bromo, 6-bromo, 5-nitro derivatives and gave the corresponding products with high yields (see the table). This method was further extended to *N*-methyl, *N*-benzyl, and *N*-acetyl (phenylimino)indolin-

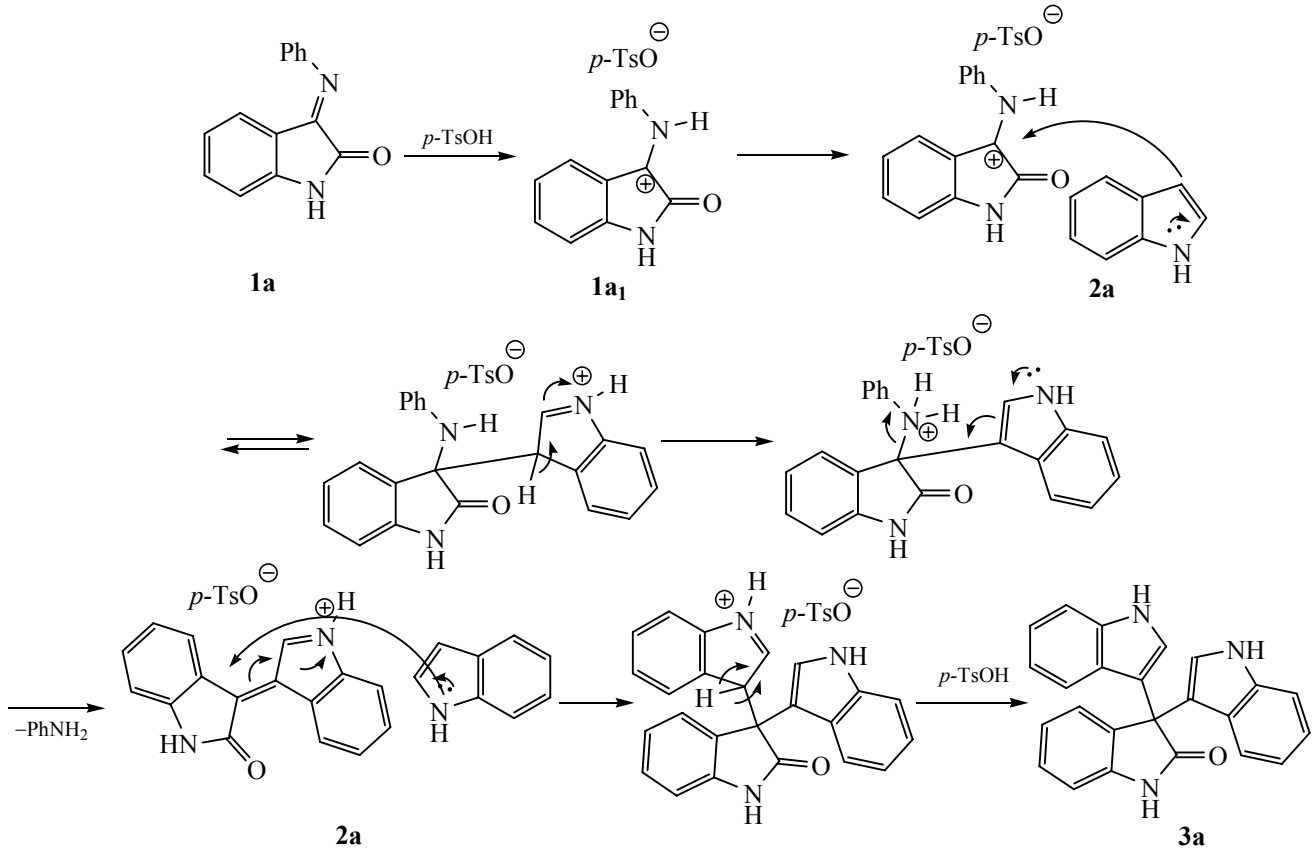
[1] The text was submitted by the authors in English.

Scheme 1. Synthesis of 3,3-di(1*H*-indol-3-yl)indolin-2-one.



1a **2** **3a**

Scheme 2. Proposed mechanism for the synthesis of **3a**.



2-ones that produced the desired products (see the table).

Without catalysis by *p*-TsOH the reaction did not proceed even at reflux temperature. Lowering the reaction temperature was detrimental to the efficiency of this procedure. The mild reaction conditions resulted in formation of no side products. All products were characterized by ^{1}H and ^{13}C NMR and mass spectrometry.

The reaction mechanism may be presented as follows (Scheme 2). Initially, *p*-TsOH activates (phenylimino)-indolin-2-ones **1a** to generate *p*-TsOH-(phenylimino)-indolin-2-one carbenium ion (**1a$_1$**), which subsequently reacts with 2 equiv. of 1*H*-indole **2** to furnish the 3,3-di-(1*H*-indol-3-yl)indolin-2-one (**3a**).

EXPERIMENTAL

All reagents were used without further purification unless specified otherwise. Solvents were dried and distilled by conventional methods. All air or moisture-sensitive reactions were conducted under the atmosphere of N_2 or Ar in flame-dried or oven-dried

p-TSA catalyzed synthesis of bis-indolyloxindoles from (phenylimino)indolin-2-ones and 1*H*-indole[a]

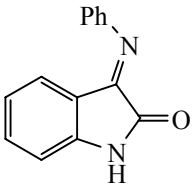
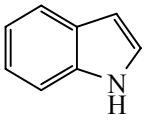
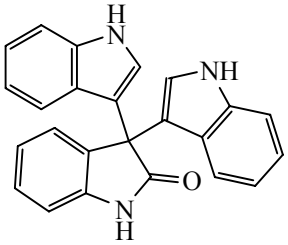
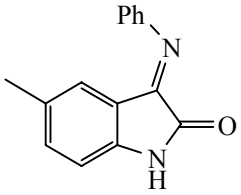
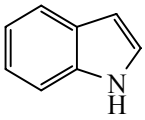
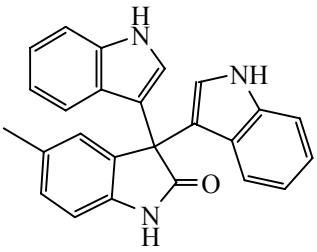
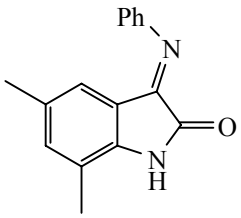
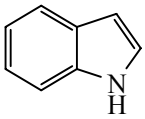
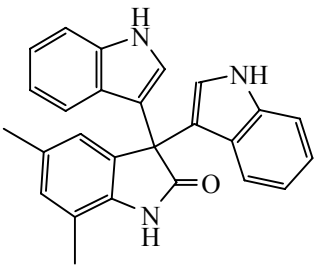
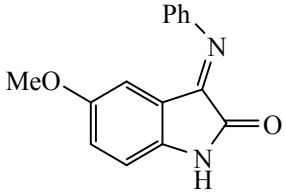
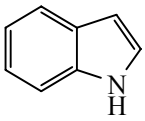
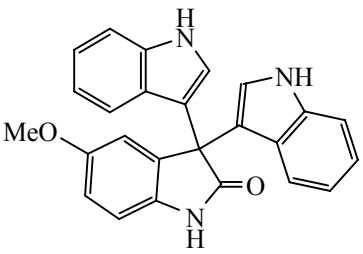
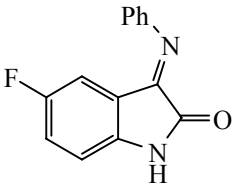
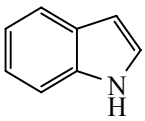
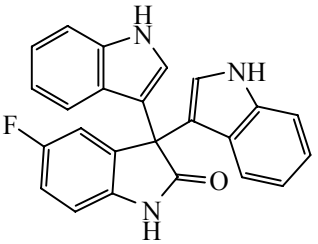
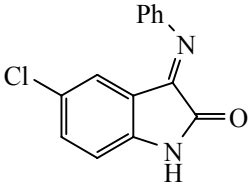
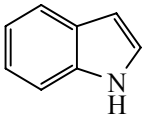
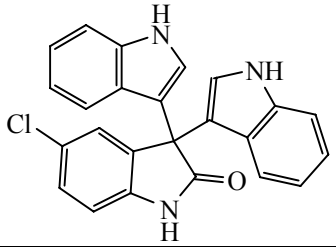
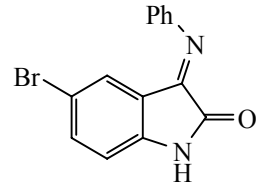
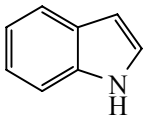
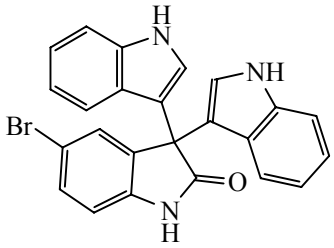
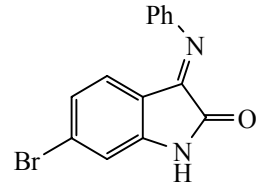
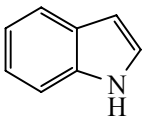
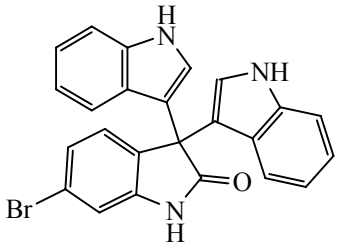
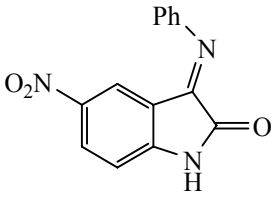
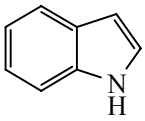
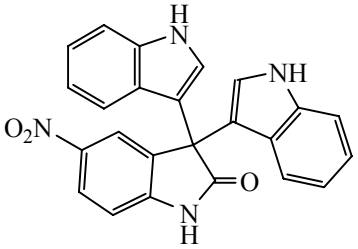
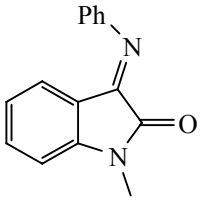
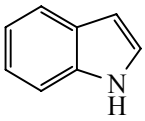
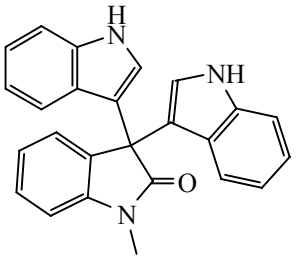
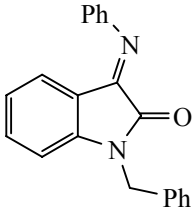
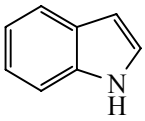
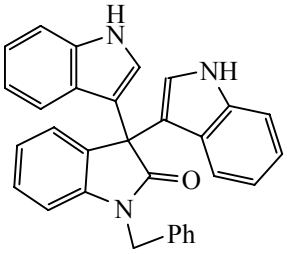
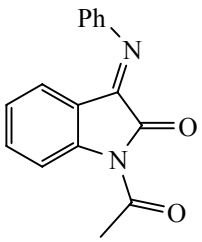
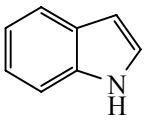
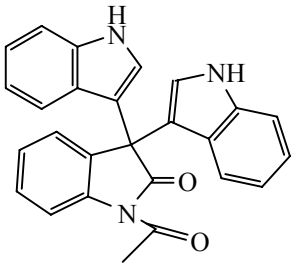
Isatin-imine **1**	Indole **2**	Product **3**	Yield,[b] %
			85
			80
			81
			82
			78
			80

Table 1. (Contd.)

Isatin-imine **1**	Indole **2**	Product **3**	Yield,[b] %
			75
			72
			70
			78
			80
			72

[a] The reaction time was 2–3 min. All products were characterized by ^{1}H and ^{13}C NMR, IR, and mass spectroscopy. [b] Yield of isolated product.

glassware with magnetic stirring. IR spectra (KBr discs) were recorded on a Bruker Vector 22 FT-IR spectrophotometer. ^{1}H and ^{13}C NMR spectra were measured in DMSO-d_6 (500 and 125 MHz) with TMS as the internal standard on a Bruker AV 300 or 500 MHz NMR spectrometers. Mass spectra were measured on a Bruker Micro-TOF-Q mass spectrometer using electrospray ionization (ESI) technique. Column chromatography was carried out using silica gel (60–120 or 100–200 mesh) packed in glass columns. Distilled ethyl acetate and petroleum ether were used as eluents.

General synthesis of 3,3-di(1*H*-indol-3-yl)indolin-2-ones (3a–3l). To the mixture of (phenylimino)indolin-2-ones (**1a**) (1 mmol) and 1*H*-indole **2** (2 mmol) in anhydrous CH_2Cl_2 (10 mL) was added *p*-TsOH (10 mol %) in a 50 mL flask and stirred at room temperature for 2–3 min. Upon completion of the process the reaction mixture was diluted, washed with water (3 × 15 mL) and extracted with ethyl acetate. The combined organic layers were concentrated to afford the crude mixture which was purified by hexane/AcOEt flash chromatography to give the pure product **3a** (0.30 g, 80%).

3,3-Di(1*H*-indol-3-yl)indolin-2-one (3a). White solid, mp 317–319°C. IR spectrum, ν, cm^{-1}: 3429, 3326, 3040, p1709, 1613, 1471, 1106, 930, 736. ^{1}H NMR spectrum, δ, ppm: 10.82 s (2H, NH), 10.50 s (1H, NH), 7.43 d (2H, *J* = 8.1 Hz), 7.33–7.28 m (4H), 7.01 m (3H, *J* = 8.0 Hz), 6.96 t (1H, *J* = 7.5 Hz), 6.71 s (2H), 6.80 t (2H, *J* = 7.5 Hz). ^{13}C NMR spectrum, δ, ppm: 178.7, 141.3, 136.9, 134.6, 127.8, 125.7, 124.9, 124.2, 121.4, 120.9, 120.7, 118.2, 114.3, 111.6, 109.5, 52.5. MS (ESI, MeOH): *m/z* 364 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-methylindolin-2-one (3b). White solid, mp 306–307°C. IR spectrum, ν, cm^{-1}: 3375 (NH), 3325 (NH), 3041, 1704, 1489, 1100, 809, 736. ^{1}H NMR spectrum, δ, ppm: 10.93 s (2H, NH), 10.50 s (1H, NH), 7.35 d (2H, *J* = 8.1 Hz), 7.24 d (2H, *J* = 7.9 Hz), 7.05–7.00 (4H, m), 6.90–6.88 m (1H), 6.85 s (2H), 6.83–6.80 m (2H), 2.19 m (3H, –CH_3). ^{13}C NMR spectrum, δ, ppm: 178.5, 138.6, 136.8, 134.5, 130.0, 128.2, 125.8, 125.5, 124.5, 121.0, 120.8, 118.0, 114.5, 111.2, 109.1, 52.7, 20.1 (–CH_3). MS (ESI, MeOH): *m/z* 378 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5,7-dimethylindolin-2-one (3c). White solid, mp 296–297°C. IR spectrum, ν, cm^{-1}: 3457 (NH), 3337 (NH), 1680, 1456, 1101, 850, 742. ^{1}H NMR spectrum, δ, ppm: 10.92 s (2H, NH), 10.51 s (1H, NH), 7.35 d (2H, *J* = 8.2 Hz), 7.24 d (2H, *J* = 8.0 Hz), 7.35–7.00 m (2H), 6.85–6.81 m (6H), 2.30 m (3H, –CH_3), 2.16 m (3H, –CH_3). ^{13}C NMR spectrum, δ, ppm: 179.3, 137.3, 137.1, 134.2, 130.1, 129.3, 125.5, 124.1, 122.5, 121.1, 120.7, 118.5, 118.2, 114.7, 111.6, 52.9, 20.8, 18.6. MS (ESI, MeOH): *m/z* 392 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-methoxylindolin-2-one (3d). White solid, mp 288–290°C. IR spectrum, ν, cm^{-1}: 3364, 3048, 1689, 1487, 1148, 1015, 858, 742. ^{1}H NMR spectrum, δ, ppm: 10.95 s (2H, NH), 10.42 s (1H, NH), 7.35 d (2H, *J* = 8.0 Hz), 7.22 d (2H, *J* = 8.1 Hz), 7.02–7.02 m (2H), 6.92–6.90 m (1H), 6.88 s (2H), 6.84–6.81 m (4H), 3.61 m (3H, OCH_3). ^{13}C NMR spectrum, δ, ppm: 178.4, 154.5, 136.7, 135.9, 134.6, 125.3, 124.4, 120.9, 120.6, 118.1, 114.2, 112.1, 112.1, 115.7, 109.9, 55.4, 53.2 (OCH_3). MS (ESI, MeOH): *m/z* 394 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-fluoroindolin-2-one (3e). White solid, mp 239–240°C. IR spectrum, ν, cm^{-1}: 3436, 3316, 1696, 1481, 1104, 861, 734. ^{1}H NMR spectrum, δ, ppm: 10.95 s (2H, NH), 10.62 s (1H, NH), 7.34 d (2H, *J* = 8.1 Hz), 7.22 d (2H, *J* = 8.0 Hz), 7.05–7. 21 m (5H), 6.91 s (2H), 6.84 t (2H, *J* = 7.5 Hz). ^{13}C NMR spectrum, δ, ppm: 178.7, 157.9, 137.6, 136.8, 136.4, 125.6, 124.5, 121.2, 120.4, 118.2, 114.1, 113.4, 112.2, 111.6, 110.4, 53.0. MS (ESI, MeOH): *m/z* 382 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-chloroindolin-2-one (3f). White solid, mp 290–291°C. IR spectrum, ν, cm^{-1}: 3440, 3362, 3037, 1699, 1476, 1105, 817, 736. ^{1}H NMR spectrum, δ, ppm: 10.35 s (1H, NH), 10.25 s (2H, NH), 7.39 d (2H, *J* = 8.1 Hz), 7.31 d (1H, *J* = 8.3Hz), 7.19–7.21 m (3H), 7.05–7.01 m (3H), 6.89 s (2H), 6.84 t (2H, *J* = 7.5 Hz). ^{13}C NMR spectrum, δ, ppm: 178.3, 140.3, 136.9, 136.6, 127.8, 125.5, 125.4, 124.6, 124.4, 121.0, 120.5, 118.4, 113.5, 111.7, 111.1, 52.8. MS (ESI, MeOH): *m/z* 398 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-bromoindolin-2-one (3g). White solid, mp 304–306°C. IR spectrum, ν, cm^{-1}: 3355 (NH), 3123 (NH), 3075, 1693, 1615, 1475, 745. ^{1}H NMR spectrum, δ, ppm: 10.14 s (1H, NH), 10.03 s (2H, NH), 7.47 s (3H), 7.32 d (1H, *J* = 2 Hz), 7.26 d (2H, *J* = 8 Hz), 7.05 t (2H, *J* = 8 Hz), 6.99 d (1H, *J* = 8 Hz), 6.91–6.87 m (2H), 6.86–6.82 m (2H). ^{13}C NMR spectrum, δ, ppm: 178.2, 140.7, 136.9, 130.6, 127.2, 125.3, 124.32, 121.0, 120.7, 120.4, 118.3, 113.5, 113.1, 111.4, 111.5, 52.8. MS (ESI, MeOH): *m/z* 444 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-6-bromoindolin-2-one (3h). White solid, mp 309–311°C. IR spectrum, ν, cm^{-1}: 3405 (NH), 3110 (NH), 3030, 1710, 1608, 1452, 1115,

744. ^{1}H NMR spectrum, δ, ppm: 11.00 s (2H, NH); 10.74 s (1H, NH), 7.36 d (2H, *J* = 8.0 Hz), 7.20 d (2H, *J* = 8.0 Hz), 7.10–7.16 m (3H), 7.02 t (2H, *J* = 7.6 Hz), 6.84 d (2H, *J* = 2.4 Hz), 6.81 t (2H, *J* = 7.6 Hz). ^{13}C NMR spectrum, δ, ppm: 178.4, 143.1, 136.7, 133.9, 126.8, 125.5, 124.4, 124.1, 121.0, 120.8, 120.2, 118.6, 113.7, 112.5, 111.8, 52.2. MS (ESI, MeOH): *m/z* 444 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-5-nitroindolin-2-one (3i). Yellow solid, mp 281–282°C. IR spectrum, ν, cm^{-1}: 3366, 3112, 1710, 1697, 1626, 1470, 1335, 735 cm^{-1}. ^{1}H NMR spectrum, δ, ppm: 11.33 s (1H, NH), 11.09 s (2H, NH), 8.25 d (1H, *J* = 8.7 Hz), 7.90 (1H, s), 7.39 d (2H, *J* = 8.1 Hz), 7.20–7.22 m (3H), 7.04 t (2H, *J* = 7.8 Hz), 6.96 s (2H), 6.83 t (2H, *J* = 7.3 Hz). ^{13}C NMR spectrum, δ, ppm: 179.0, 147.5, 142.5, 137.9, 136.1, 126.2, 125.6, 125.4, 122.1, 121.2, 119.4, 119.4, 113.6, 112.7, 110.2, 52.4. MS (ESI, MeOH): *m/z* 409 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-1-methylindolin-2-one (3j). White solid, mp 277–279°C. IR spectrum, ν, cm^{-1}: 3360 (NH), 3118 (NH), 3047, 2950, 1695, 1670, 1611, 1472, 734. ^{1}H NMR spectrum, δ, ppm: 10.17 s (2H, NH), 7.56 d (2H, *J* = 8 Hz), 7.35–7.31 m (2H), 7.24 d (2H, *J* = 8 Hz), 7.12–7.15 m (1H), 7.05–7.00 m (3H), 6.91 t (2H, *J* = 7.6 Hz), 6.83 t (2H, *J* = 7.4 Hz), 3.32 m (3Hs, *N*–CH_3). ^{13}C NMR spectrum, δ, ppm:. 176.95, 142.69, 136.95, 133.69, 127.88, 125.62, 124.55, 124.29, 122.07, 120.91, 120.71, 118.26, 114.05, 111.55, 108.46, 52.17, 26.17 (NCH_3). MS (ESI, MeOH): *m/z* 378 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-1-benzylindolin-2-one (3k). White solid, mp 198–200°C. IR spectrum, ν, cm^{-1}: 3410, 3120, 1702, 1472, 734. ^{1}H NMR spectrum, δ, ppm: 9.81 s (2H), 7.35–7.37 m (4H), 7.24–7.33 m (5H), 7.10–7.13 m (3H), 6.98–7.03 m (3H), 6.87 s (2H), 6.74 t (2H, *J* = 7.2 Hz), 5.01 s (2H, CH_2). ^{13}C NMR spectrum, δ, ppm: 177.93, 142.57, 137.78, 137.41, 134.63, 129.44, 128.75, 128.47, 128.37, 126.44, 125.65, 125.26, 125.10, 123.12, 121.94, 121.66, 119.18, 119.18, 119.18, 114.83, 112.54, 110.21, 53.12, 43.84. MS (ESI, MeOH): *m/z* 454 $[M + H]^+$.

3,3-Di(1*H*-indol-3-yl)-*N*-acetylindolin-2-one (3l). White solid, mp 310–312°C. IR spectrum, ν, cm^{-1}: 3420 (NH), 3323 (NH), 3052, 1747, 1720, 1460, 1371, 1349, 1299, 1267, 1245, 1170, 764, 750, 748. ^{1}H NMR spectrum, δ, ppm: 11.05 s (2H, NH), 7.38–6.86 m (14H), 2.06 m (3H, CH_3). ^{13}C NMR spectrum, δ, ppm: 176.95, 142.69, 136.95, 133.69, 127.88, 125.62, 124.55, 124.29, 122.07, 120.91, 120.71, 118.26, 114.05, 111.55, 108.46, 52.17, 23.05. MS (ESI, MeOH): *m/z* 406 $[M + H]^+$.

CONCLUSIONS

Here is presented a simple and efficient method of synthesis of 3,3-di(1*H*-indol-3-yl)indolin-2-ones from (phenylimino)indolin-2-ones with *p*-TSA catalysis. This method offers such advantages as low cost, short reaction time, high yield, and mild reaction conditions.

ACKNOWLEDGMENTS

The authors thankful to DST-SERB and UGC New Delhi for financial assistance.

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