Ultrasonics Sonochemistry 18 (2011) 1153–1159

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultsonch

Sonochemical multi-component synthesis of spirooxindoles

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ARTICLE INFO

Article history:
Received 3 October 2010
Received in revised form 8 December 2010
Accepted 12 December 2010
Available online 21 December 2010

Keywords:
Multi-component reactions
Spirooxindole
Ultrasound
Isatin
Tetronic acid

ABSTRACT

New and efficient multi-component methods have been developed for the synthesis of spirooxindoles in the presence of a catalytic amount of *p*-TSA as an inexpensive and available catalyst in EtOH under ultrasound irradiation. The method is simple, starts from readily accessible commercial starting materials, and provides biologically interesting products in good yields and short reaction times.

1. Introduction

Ultrasonication, based on cavitation effects leading to mass transfer improvement, is an important technique that is widely used today in organic synthesis and has a profound impact on the way chemists approach organic and parallel synthesis. Reductions in reaction times, improved yields and suppression of side products, relative to traditional thermal heating, are benefits of this technology [1]. A large number of organic reactions can be carried out in higher yield, shorter reaction time and under milder conditions, by using ultrasonic irradiation [2]. Also, multi-component reactions (MCRs) are a very powerful tool for the construction of complex organic molecules by using readily available starting materials. MCRs have been frequently used by synthetic chemists as a facile means to generate molecular diversity from bifunctional substrates that react sequentially in an intramolecular fashion [3]. In this context, spirooxindoles show interesting features that make them attractive for use in MCRs under ultrasound irradiation.

The indole moiety is probably the most well-known heterocycle, a common and important feature of a variety of natural products and medicinal agents [4]. Furthermore, it has been reported that sharing of the indole 3-carbon atom in the formation of spiroindoline derivatives can highly enhance biological activity [5]. The spirooxindole system is the core structure of many pharmacological agents and natural alkaloids [6].

Tetronic acid (tetrahydrofuran-2,4-dione) is a promising convenient building block and compounds in which tetronic acid fragment is fused to a heterocyclic systems attract specific attention. Some derivatives of such polycyclic system showed anticancer [7], antirheumatic [8], antiarrythmic [9], and enzyme inhibitory activity [10]. In the literature, there are a number of reliable methods for the application of tetronic acid in the synthesis of biologically active compounds [11].

As part of our continuing efforts on the synthesis of heterocyclic compounds [12] and sonocatalysis organic transformations [13], we have recently reported the sonocatalysis synthesis of spiro[indoline-3,4′-pyrazolo[3,4-*b*]pyridine]-2,6′(1′*H*)-dione derivatives [13c]. Although several isatin-based reactions have been reported in the synthesis of spirooxindoles [14], the synthesis of spiro[benzo[*g*]-furo[3,4-*b*]quinoline-indoline]-tetraones have not been reported yet. In this paper, we report for the first time an efficient four component sonocatalysis synthesis of spiro[benzo[*g*]furo[3,4-*b*]quinoline-indoline]-tetraones.

2. Experimental

2.1. Chemicals and apparatus

The chemical used in this work were obtained from Fluka and Merck and were used without purification. Melting points were measured on an Electrothermal 9200 apparatus. IR spectra were recorded on a FT-IR 102 MB BOMEM apparatus. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. ^{1}H and ^{13}C NMR spectra were obtained on solutions in DMSO-d_6 using TMS. Ultrasound assisted reactions

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doi:10.1016/j.ultsonch.2010.12.004

were carried out using a EUROSONIC® 4D ultrasound cleaner with a frequency of 50 kHz and a nominal power of 350 W. The reactions were carried out in an open glass tube (diameter: 20 mm; thickness: 1 mm; volume: 25 mL) at 50 °C. The reaction flasks were immersed in every place of the cleaner in such a way that the surface of reactants is slightly lower than water in the cleaner, and the temperature of the water bath can be controlled by an electronic temperature control system.

2.2. Typical procedure for the preparation of 1H-spiro[benzo[g]furo[3,4-b]quinoline-11,3′-indoline]-1,2′,5,10(3H,4H)-tetraone (**5a**)

A mixture of isatin (1 mmol), tetronic acid (1 mmol), 2-hydroxy-1,4-naphthoquinone (1 mmol), ammonium acetate (1.5 mmol) and *p*-TSA (20% mol) in EtOH (5 mL) was sonicated at 50 °C for 1.5 h. After completion of the reaction, the reaction mixture was filtered and the precipitate washed with diethyl ether (2 × 5 mL) to afford the pure product **5a** as Orange powder; mp: 250–252 °C. IR (KBr) (ν_{max} cm^{-1}): 3457, 3272, 1734, 1680, 1607. MS, m/z: 384 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.46 (2H, s, CH_2), 6.84–7.88 (8H, m, H–Ar), 10.45 (1H, s, NH), 10.64 (1H, s, NH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 51.3, 61.6, 109.1, 110.1, 121.2, 122.1, 125.1, 125.6, 125.8, 126.0, 126.8, 129.5, 130.2, 131.3, 131.5, 134.4, 143.1, 165.3, 175.1, 190.0. Anal. Calcd. for $C_{22}H_{12}N_2O_5$: C, 68.75; H, 3.15; N, 7.29%. Found: C, 68.66; H, 3.22; N, 7.38%.

2.3. 5′-methyl-1H-spiro[benzo[g]furo[3,4-b]quinoline-11,3′-indoline]-1,2′,5,10(3H,4H)-tetraone (**5b**)

Brown powder; mp: 167–168 °C. IR (KBr) (ν_{max} cm^{-1}): 3193, 1740, 1679, 1639. MS, m/z (%): 398 (M^+).^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 1.99 (3H, s, CH_3), 4.58 (2H, s, CH_2), 6.86–8.00 (7H, m, H–Ar), 10.42 (1H, s, NH), 10.92 (1H, s, NH). Anal. Calcd. for $C_{23}H_{14}N_2O_5$: C, 69.34; H, 3.54; N, 7.03%. Found: C, 69.23; H, 3.42; N, 7.12% (Due to very low solubility of the product 5b, we cannot report the ^{13}C NMR data for this product).

2.4. 5′-bromo-1H-spiro[benzo[g]furo[3,4-b]quinoline-11,3′-indoline]-1,2′,5,10(3H,4H)-tetraone (**5c**)

Yellow powder; mp: 162–163 °C. IR (KBr) (ν_{max} cm^{-1}): 3449, 1715, 1620. MS, m/z (%): 463 (M^+), 461 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.55 (2H, s, CH_2), 6.76–7.98 (7H, m, H–Ar), 10.88 (1H, s, NH), 11.44 (1H, s, NH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 50.5, 66.3, 98.4, 109.2, 121.5, 125.8, 125.9, 126.0, 126.4, 126.5, 128.2, 129.8, 131.2, 133.6, 134.0, 134.9, 135.2, 143.0, 171.9, 176.9, 181.3, 184.2. Anal. Calcd. for $C_{22}H_{11}BrN_2O_5$: C, 57.04; H, 2.39; N, 6.05%. Found: C, 56.98; H, 2.45; N, 6.11%.

2.5. 5′-nitro-1H-spiro[benzo[g]furo[3,4-b]quinoline-11,3′-indoline]-1,2′,5,10(3H,4H)-tetraone (**5d**)

Yellow powder; mp: 153–155 °C. IR (KBr) (ν_{max} cm^{-1}): 3300, 1738, 1681, 1630, 1520, 1335. MS, m/z (%): 429 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.58 (2H, s, CH_2), 7.78–8.16 (7H, m, H–Ar), 10.96 (1H, s, NH), 11.16 (1H, s, NH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 50.1, 66.5, 97.1, 109.3, 120.5, 121.2, 125.9, 126.1, 126.4, 129.9, 132.3, 133.0, 132.3, 133.7, 135.2, 142.1, 149.7, 172.9, 177.0, 181.2. Anal. Calcd. for $C_{22}H_{11}N_3O_7$: C, 61.54; H, 2.58; N, 9.79%. Found: C, 61.50; H, 2.63; N, 9.85%.

2.6. 1′-benzyl-1H-spiro[benzo[g]furo[3,4-b]quinoline-11,3′-indoline]-1,2′,5,10(3H,4H)-tetraone (**5e**)

Dark yellow powder; mp: 128–130 °C. IR (KBr) (ν_{max} cm^{-1}): 3436, 2926, 1731, 1666, 1355, 1284. MS, m/z (%): 474 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.45 (2H, bs, CH_2), 4.85 (2H, s, CH_2), 6.68–7.97 (13H, m, H–Ar), 11.51 (1H, s, NH). Anal. Calcd. for $C_{29}H_{18}N_2O_5$: C, 73.41; H, 3.82; N, 5.90%. Found: C, 73.30; H, 3.90; N, 6.00% (Due to very low solubility of the product **5e**, we cannot report the ^{13}C NMR data for this product).

2.7. 1′H,2H-spiro[acenaphthylene-1,11′-benzo[g]furo[3,4-b]quinoline1′,2,5′,10′(3′H,4′H)-tetraone (**9**)

Yellow powder; mp: 163–165 °C. IR (KBr) (ν_{max} cm^{-1}): 3436, 2926, 1730, 1670, 1596, 1277. MS, m/z (%): 419 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.57 (2H, s, CH_2), 7.17–8.54 (10H, m, H–Ar), 11.46 (1H, s, NH). Anal. Calcd. for $C_{26}H_{13}NO_5$: C, 74.46; H, 3.12; N, 3.34%. Found: C, 74.40; H, 3.18; N, 3.40% (Due to very low solubility of the product **7**, we cannot report the ^{13}C NMR data for this product).

2.8. 3,3-Bis(2,5-dihydro-4-hydroxy-2-oxofuran-3-yl)indolin-2-one (**10a**)

Yellow powder; mp: 168–170 °C. IR (KBr) (ν_{max} cm^{-1}): 3263, 3012, 1732, 1664, 1618. MS, m/z (%): 329 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.56 (4H, s, 2CH_2), 6.73 (1H, d, H–Ar, J = 7.6 Hz), 6.82 (1H, t, H–Ar, J = 7.5 Hz), 7.09 (1H, t, H–Ar, J = 7.5 Hz), 7.28 (1H, d, H–Ar, J = 7.4 Hz), 10.48 (1H, s, NH), 11.88 (2H, bs, 2OH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 47.9, 66.5, 96.9, 109.5, 121.6, 125.2, 128.1, 132.2, 142.1, 172.7, 173.8, 176.7. Anal. Calcd. for $C_{16}H_{11}NO_7$: C, 58.36; H, 3.37; N, 4.25%. Found: C, 58.25; H, 3.47; N, 4.36%.

2.9. 3,3-Bis(2,5-dihydro-4-hydroxy-2-oxofuran-3-yl)-5-methylindolin-2-one (**10b**)

Yellow powder; mp: 163–165 °C. IR (KBr) (ν_{max} cm^{-1}): 3430, 3054, 1732, 1669, 1612. MS, m/z (%): 343 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 2.27 (3H, s, CH_3), 4.56 (4H, s, 2CH_2), 6.63 (1H, d, H–Ar, J = 7.7 Hz), 6.90 (1H, d, H–Ar, J = 7.6 Hz), 7.12 (1H, s, H–Ar), 10.40 (1H, s, NH), 11.79 (2H, bs, 2OH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 21.3, 48.1, 66.5, 96.9, 109.2, 125.9, 128.4, 130.2, 132.2, 139.7, 172.7, 173.9, 176.8. Anal. Calcd. for $C_{17}H_{13}NO_7$: C, 59.48; H, 3.82; N, 4.08%. Found: C, 59.41; H, 3.89; N, 4.15%.

2.10. 5-Bromo-3,3-bis(2,5-dihydro-4-hydroxy-2-oxofuran-3-yl)indolin-2-one (**10c**)

Yellow powder; mp: 181–183 °C. IR (KBr) (ν_{max} cm^{-1}): 3222, 3134, 1733, 1672, 1622. MS, m/z (%): 408 (M^+), 406 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.59 (4H, s, 2CH_2), 6.71–7.49 (3H, m, H–Ar), 10.59 (1H, s, NH), 12.01 (2H, bs, 2OH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 47.9, 66.7, 99.3, 111.5, 125.9, 128.6, 130.8, 134.7, 138.4, 172.8, 174.1, 175.8. Anal. Calcd. for $C_{16}H_{10}BrNO_7$: C, 47.08; H, 2.47; N, 3.43%. Found: C, 46.98; H, 2.56; N, 3.54%.

2.11. 3,3-Bis(2,5-dihydro-4-hydroxy-2-oxofuran-3-yl)-5-nitroindolin-2-one (**10d**)

Yellow powder; mp: 178–180 °C. IR (KBr) (ν_{max} cm^{-1}): 3255, 3067, 1733, 1667, 1617. MS, m/z (%): 374 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.61 (2H, s, CH_2), 6.96 (1H, d, H–Ar,

J = 8.3 Hz), 8.11 (2H, d, H–Ar, J = 9.3 Hz), 11.14 (1H, s, NH), 12.45 (2H, bs, 2OH). ^{13}C NMR (75.47 MHz, DMSO-d_6): δ_C 47.5, 66.9, 95.7, 109.6, 120.0, 125.8, 133.4, 142.2, 148.9, 172.8, 174.8, 176.3. Anal. Calcd. for $C_{16}H_{10}N_2O_9$: C, 51.35; H, 2.69; N, 7.49%. Found: C, 51.31; H, 2.73; N, 7.53%.

2.12. 1-Benzyl-3,3-bis(2,5-dihydro-4-hydroxy-2-oxofuran-3-yl) indolin-2-one **(10e)**

Yellow powder; mp: 168–170 °C. IR (KBr) (ν_{max} cm^{-1}): 3390, 2923, 1716, 1671, 1610. MS, m/z (%): 419 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.44 (4H, s, 2CH$_2$), 4.96 (2H, s, CH$_2$), 6.69–7.69 (9H, m, H–Ar), 8.19 (2H, bs, 2OH). Anal. Calcd. for $C_{23}H_{17}NO_7$: C, 65.87; H, 4.09; N, 3.34%. Found: C, 65.81; H, 4.26; N, 3.41% (Due to very low solubility of the product **10e**, we cannot report the ^{13}C NMR data for this product).

2.13. 3,3′-(2-oxo-1,2- dihydroacenaphthylene-1,1-diyl)bis(4-hydroxy furan-2(5H)-one) **(11)**

Yellow powder; mp: 188–190 °C. IR (KBr) (ν_{max} cm^{-1}): 3244, 1736, 1673, 1618. MS, m/z (%): 364 (M^+). ^{1}H NMR (300.13 MHz, DMSO-d_6): δ_H 4.58 (4H, s, 2CH$_2$), 7.11–7.61 (2H, m, H–Ar), 7.77 (1H, t, H–Ar, J = 7.7 Hz), 7.89 (2H, m, H–Ar), 8.21 (1H, d, H–Ar, J = 7.9 Hz), 11.81 (2H, bs, 2OH). Anal. Calcd. for $C_{20}H_{12}O_7$: C, 65.94; H, 3.32%. Found: C, 65.87; H, 3.39% (Due to very low solubility of the product **11**, we cannot report the ^{13}C NMR data for this product).

2.14. 1,3-diphenyl-7,8-dihydrospiro[furo[3,4-b]pyrazolo[4,3-e] pyridine-4,3′-indoline]-2′,5(1H)-dione **(13a)**

Yellow powder, m.p. 258–261 °C; IR (KBr) (ν_{max}, cm^{-1}): 3383, 3205, 1721; 1669; ^{1}H NMR (DMSO-d_6): δ_H 4.98 (2H, s, CH$_2$), 6.81–8.32 (14H, m, Arom), 10.94 (1H, s, NH), 11.07 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 46.9, 66.4, 96.1, 96.7, 100.3, 108.8, 120.3, 124.3, 126.3, 128.2, 128.4, 128.6, 130.1, 132.4, 135.4, 137.6, 139.5, 143.4, 149, 149.8, 160.6, 170.2, 177.4. MS (m/z) 446 (M^+); Anal. Calcd for $C_{27}H_{18}N_4O_3$: C, 72.64; H, 4.06; N, 12.55%. Found: C, 72.58; H, 4.01; N, 12.49%.

2.15. 1-(4-nitrophenyl)-3-phenyl-7,8-dihydrospiro[furo[3,4-b] pyrazolo[4,3-e]pyridine-4,3′-indoline]-2′,5(1H)-dione **(13b)**

White powder, m.p. 270–272 °C; IR (KBr) (ν_{max}, cm^{-1}): 3383, 3077, 1755, 1700; ^{1}H NMR (DMSO-d_6): δ_H 4.94 (2H, s, CH$_2$), 6.60–8.32 (13H, m, Arom), 10.47 (1H, s, NH), 10.81 (1H, s, NH). MS (m/z) 491 (M^+); Anal. Calcd for $C_{27}H_{17}N_5O_5$: C, 65.99; H, 3.49; N, 14.25%. Found: C, 65.90; H, 3.42; N, 14.17% (Due to very low solubility of the product **13b**, we cannot report the ^{13}C NMR data for this product).

2.16. 5′-bromo-1′-ethyl-1,3-diphdihydrospiro[furo[3,4-b] pyrazolo[4,3-e]pyridine-4,3′-indoline]-2′,5(1H)-dione **(13c)**

Cream powder, m.p. > 300 °C; IR (KBr) (ν_{max}, cm^{-1}): 3179, 1763, 1669; ^{1}H NMR (DMSO-d_6): δ_H 0.82 (3H, s, CH$_3$), 3.32 (2H, s, CH$_2$), 4.93 (2H, s, CH$_2$), 6.75–7.66 (13H, m, Arom), 10.79 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 12.1, 34.7, 52.1, 66.1, 99.3, 100.3, 110.7, 114.6, 124.2, 127.8, 128.2, 128.5, 130.1, 131.8, 132.8, 137.4, 137.7, 139.6, 141.9, 149.7, 158.8, 159.1, 160.5, 170, 175.6. MS (m/z) 554 (M^+), 552 (M^+); Anal. Calcd for $C_{29}H_{21}BrN_4O_3$: C, 62.94; H, 3.82; N, 10.12%. Found: C, 62.86; H, 3.88; N, 10.04%.

2.17. 5′-bromo-1-(4-bromophenyl)-1′-ethyl-3-phenyl-7,8-dihydrospiro[furo[3,4-b]pyrazolo [4,3-e]pyridine-4,3′-indoline]-2′,5(1H)-dione **(13d)**

Cream powder, m.p. > 300 °C; IR (KBr) (ν_{max}, cm^{-1}): 3185, 1768, 1670; ^{1}H NMR (DMSO-d$_6$): δ_H 0.88 (3H, bs, CH$_3$), 3.35 (2H, bs, CH$_2$), 4.94 (2H, s, CH$_2$), 6.78–7.76 (13H, m, Arom), 10.83 (1H, s, NH); ^{13}C NMR (DMSO-d$_6$): 12.4, 35.0, 52.3, 66.1, 99.6, 103.3, 111.7, 115.8, 124.3, 128.1, 128.3, 128.5, 130.0, 131.5, 132.9, 136.4, 137.0, 139.5, 142.8, 149.6, 159.6, 159.9, 160.8, 170.0, 175.8. MS (m/z) 632 (M^+); Anal. Calcd for $C_{29}H_{20}Br_2N_4O_3$: C, 55.09; H, 3.19; N, 8.86%. Found: C, 54.98; H, 3.24; N, 8.77%.

2.18. 1-(4-methoxyphenyl)-5′-nitro-3-phenyl-7,8-dihydrospiro [furo[3,4-b]pyrazolo[4,3-e]pyridine-4,3′-indoline]-2′,5(1H)-dione **(13e)**

white powder, m.p. 275–277 °C; IR (KBr) (ν_{max}, cm^{-1}): 3305, 3067, 1738, 1705; ^{1}H NMR (DMSO-d_6): δ_H 3.86 (3H, s, OCH$_3$), 4.98 (2H, s, CH$_2$), 6.65–8.47 (12H, m, Arom), 10.33 (1H, s, NH), 10.94 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 47.5, 65.9, 99.1, 102.2, 109.8, 122.3, 124, 125.1, 125.6, 128.1, 128.3, 128.8, 129.1, 132.4, 135.7, 140, 142.1, 142.8, 146.2, 151.5, 159.3, 169.9, 177.9. Anal. Calcd for $C_{28}H_{19}N_5O_6$: C, 64.49; H, 3.67; N, 13.43%. Found: C, 64.57; H, 3.60; N, 13.50%.

2.19. 5′-methyl-1,3-diphenyl-4,5-dihydrospiro[furo[3,4-e] pyrazolo[4,3-b]pyridine-8,3′-indoline]-2′,7(1H)-dione **(13f)**

White powder, m.p. 279–281 °C; IR (KBr) (ν_{max}, cm^{-1}): 3222, 3161, 1744, 1705; ^{1}H NMR (DMSO-d_6): δ_H 2.21 (3H, s, CH$_3$), 4.93 (2H, s, CH$_2$), 6.57–7.66 (13H, m, Arom), 10.21 (1H, s, NH), 10.71 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 21.0, 47.7, 65.8, 98.8, 101.1, 109.5, 124.1, 125.5, 128.0, 128.3, 128.4, 129.2, 130.2, 131.2, 133.1, 136.2, 137.8, 139.5, 139.7, 149.8, 159.2, 170.1, 178. MS (m/z) 460 (M^+); Anal. Calcd for $C_{28}H_{20}N_4O_3$: C, 73.03; H, 4.38; N, 12.17%. Found: C, 72.92; H, 4.43; N, 12.06%.

2.20. 1,3-diphenylspiro[benzo[g]pyrazolo[3,4-b]quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione **(15a)**

Red powder, m.p. 268–270 °C; IR (KBr) (ν_{max}, cm^{-1}): 3426, 3343, 1755, 1727, 1678; ^{1}H NMR (DMSO-*d6*): δ_H 6.61–8.57 (18H, m, Arom), 10.46 (1H, s, NH), 11.47 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 51.4, 105.6, 109.9, 114.8, 120.9, 122.5, 123.3, 124.0, 125.9, 126.2, 127.5, 128.0, 128.6, 128.9, 129.5, 129.7, 131.8, 132.7, 133.2, 134.3, 134.7, 138.8, 143.9, 149.2, 152.4, 153.5, 176.3, 179.3, 181.0. MS (m/z) 520 (M^+); Anal. Calcd for $C_{33}H_{20}N_4O_3$: C, 76.14; H, 3.87; N, 10.76% Found: C, 76.07; H, 3.81; N, 10.68.

2.21. 1-(4-methoxyphenyl)-3-phenylspiro[benzo[g]pyrazolo[3,4-b] quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione **(15b)**

Dark red powder, m.p. 260–262 °C; IR (KBr) (ν_{max}, cm^{-1}): 3343, 3200, 1745, 1650, 1615; ^{1}H NMR (DMSO-d_6): δ_H .3.86 (1H, s, OCH$_3$), 6.66–8.07 (17H, m, Arom), 9.90 (1H, s, NH), 10.09 (1H, s, NH); MS (m/z) 550 (M^+); Anal. Calcd for $C_{34}H_{22}N_4O_4$: C, 74.17; H, 4.03; N, 10.18% Found: C, 74.06; H, 4.08; N, 10.24 (Due to very low solubility of the product **15b**, we can not report the ^{13}C NMR data for this product).

2.22. 1-(4-nitrophenyl)-3-phenylspiro[benzo[g]pyrazolo[3,4-b] quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione **(15c)**

Red powder, m.p. > 300 °C; IR (KBr) (ν_{max}, cm^{-1}): 3210, 3061, 1715, 1676, 1644; ^{1}H NMR (DMSO-d_6): δ_H 8.57–8.86 (17H, m,

Arom), 10.05 (1H, s, NH), 10.18 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 50.6, 101.0, 101.2, 111.4, 113.5, 118.0, 123.8, 125.2, 126.4, 126.8, 127.3, 127.9, 128.0, 128.2, 128.6, 129, 130.0, 130.3, 131.2, 133.8, 135.7, 136.9, 138.5, 140.3, 141.7, 178.9, 181.0. MS (m/z) 565 (M^+); Anal. Calcd for $C_{33}H_{19}N_5O_5$: C, 70.08; H, 3.39; N, 12.38% Found: C, 70.02; H, 3.34; N, 12.30.*2.2.3.*

2.23. 5′-bromo-1′-ethyl-1,3-diphenylspiro[benzo[g]pyrazolo[3,4-b] quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione ***(15d)***

Red powder, m.p. 269–271 °C; IR (KBr) (ν_{max}, cm^{-1}): 3255, 1722, 1705, 1677; H NMR (DMSO-d_6): δ_H 0.83 (3H, bs, CH_3), 3.15 (2H, bs, CH_2), 6.63–8.05 (17H, m, Arom), 9.98 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 11.8, 34.7, 50.0, 110.4, 123.8, 126.5, 127.4, 128.0, 128.2, 128.6, 128.9, 130.0, 130.4, 131.4, 132.2, 132.6, 133.0, 133.8, 135.6, 138.5, 139.5, 142.2, 143.0, 149.7, 151.0, 152.6, 178.9, 181.0. MS (m/z) 628 (M^+), 626 (M^+); Anal. Calcd for $C_{35}H_{23}BrN_4O_3$: C, 66.99; H, 3.69; N, 8.93% Found: C, 66.90; H, 3.74; N, 9.01.

2.24. 5′-nitro-1,3-diphenylspiro[benzo[g]pyrazolo[3,4-b]quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione ***(15e)***

Dark red powder, m.p. 271–273 °C; IR (KBr) (ν_{max}, cm^{-1}): 3426, 3327, 1705, 1672, 1650; ^{1}H NMR (DMSO-d_6): δ_H 6.68–8.52 (17H, m, Arom), 10.84 (1H, s, NH), 11.16 (1H, s, NH); ^{13}C NMR (DMSO-d_6): 51.3, 83.2, 97.8, 108.9, 117.2, 118.5, 120.1, 121.3, 122.2, 124.1, 126.5, 126.8, 128.5, 129.9, 131.9, 133.2, 134.2, 135.9, 136.0, 143.5, 146.4, 150.7, 151.5, 152.4, 157.2, 160.2, 179.4, 181.7. MS (m/z) 565 (M^+); Anal. Calcd for $C_{33}H_{19}N_5O_5$: C, 70.08; H, 3.39; N, 12.38% Found: C, 70.13; H, 3.32; N, 12.31.

2.25. 5′-nitro-1-(4-nitrophenyl)-3-phenylspiro[benzo[g]pyrazolo[3,4-b]quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione ***(15f)***

Dark red powder, m.p. > 300 °C; IR (KBr) (ν_{max}, cm^{-1}): 3418, 3329, 1711, 1670, 1653; ^{1}H NMR (DMSO-d_6): δ_H 6.49–8.58 (16H, m, Arom), 10.28 (1H, s, NH), 10.59 (1H, s, NH); MS (m/z) 610 (M^+); Anal. Calcd for $C_{33}H_{18}N_6O_7$: C, 64.92; H, 2.97; N, 13.77% Found: C, 64.84; H, 2.93; N, 13.85 (Due to very low solubility of the product **15f**, we can not report the ^{13}C NMR data for this product).

2.26. 5′-methyl-1,3-diphenylspiro[benzo[g]pyrazolo[3,4-b]quinoline-4,3′-indoline]-2′,5,10(1H,11H)-trione ***(15g)***

Red powder, m.p. > 300 °C; IR (KBr) (ν_{max}, cm^{-1}): 3194, 3056, 1716, 1678; ^{1}H NMR (DMSO-d_6): 2.17 (3H, S, CH_3), 6.49–8.05 (17H, m, Arom), 10.02 (1H, s, NH), 10.07 (1H, s, NH); MS (m/z) 534 (M^+); Anal. Calcd for $C_{34}H_{22}N_4O_3$: C, 76.39; H, 4.15; N, 10.48% Found: C, 76.30; H, 4.21; N, 10.53 (Due to very low solubility of the product **15g**, we can not report the ^{13}C NMR data for this product).

3. Results and discussion

In a pilot experiment, a mixture of isatin **1a**, teronic acid **2**, 2-hydroxy-1,4-naphthoquinone **3** and ammonium acetate **4** in the presence of a catalytic amount of *p*-TSA was sonicated at 50 °C for 1.5 h. After completion of the reaction (monitored by TLC), the crude product was separated from the reaction mixture by filtration and washed with diethyl ether (2 × 5 mL) to afford product **5a** in 90% yield (Scheme 1).

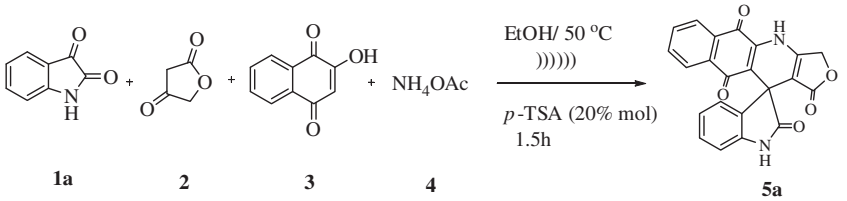
To search for the optimal reaction solvent, this reaction was examined in H_2O, CH_2Cl_2, CH_3CN and EtOH at 50 °C under ultrasonic irradiation. As shown in Table 1, the reaction using ethanol as solvent resulted in higher yields and short reaction time (Table 1, entry 3). To study the effect of amount of the catalyst, the reactions were carried out at different amount of *p*-TSA ranging from 10–30 mol%. It was found that when increasing the amount of the *p*-TSA from 10 to 20, and 30 mol%, the yields increased from 71 to 90 and 91%, respectively (Table 1, entries 2–4). It was found that 20 mol% *p*-TSA in ethanol is sufficient to push this reaction forward (Table 1, entry 3). More amounts of the additive did not improve the yields. When this reaction was carried out without *p*-TSA the yield of the expected product was very low (Table 1, Entry 1). To optimize the reaction temperature, we also performed several experiments at 25, 35, 40 and 50 °C under ultrasonic irradiation. It was found that the most suitable reaction temperature is 50 °C under ultrasonic irradiation (Entry 3). The effect of ultrasound irradiation on the reaction was also observed. As shown in Table 1, under silent condition by using stirring for 6 h, the model reaction gave **5a** in <30% yield (Table 1, Entry 11). In was found that the use of ultrasound irradiation leads to a higher yield in shorter reaction time.

Then, to delineate this approach, particularly in regard to library construction, this methodology was evaluated by using different isatins **1a**–**e**. Corresponding spiro[benzo[*g*]furo[3,4-*b*]quinoline-11,3′-indoline]-tetraones **5a**–**e** were selectively synthesized by the four-component condensation of isatins **1**, teronic acid **2**, 2-hydroxy-1,4-naphthoquinone **3** and ammonium acetate **4** in good yields under ultrasound irradiation at 50 °C in the presence of

Table 1
Optimization of conditions[a].

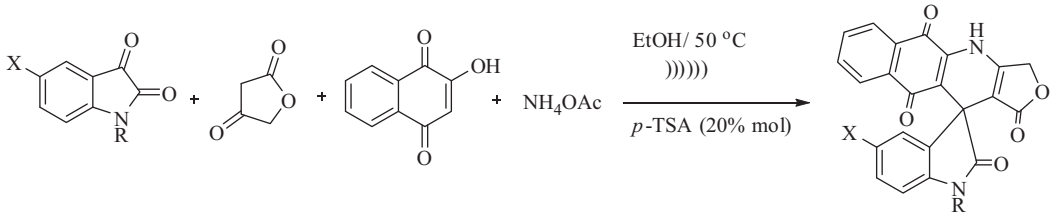
Entry	Conditions	*p*-TSA (mol%)	Time (h)	Yield (%)
1	EtOH/50 °C/ultrasound	None	5	<30
2	EtOH/50 °C/ultrasound	10	3	71
3	EtOH/50 °C/ultrasound	20	1.5	90
4	EtOH/50 °C/ultrasound	30	1.5	91
5	EtOH/40 °C/ultrasound	20	1.5	78
6	EtOH/35 °C/ultrasound	20	1.5	63
7	EtOH/25 °C/ultrasound	20	1.5	55
8	H_2O/50 °C/ultrasound	20	5	<30
9	CH_2Cl_2/50 °C/ultrasound	20	5	45
10	CH_3CN/50 °C/ultrasound	20	5	55
11	EtOH/50 °C/Silent	20	6	<30

[a] Isatin **1a** (1 mmol), teronic acid **2** (1 mmol), 2-hydroxy-1,4-naphthoquinone **3** (1 mmol), and ammonium acetate **4** (1.5 mmol).



Scheme 1.

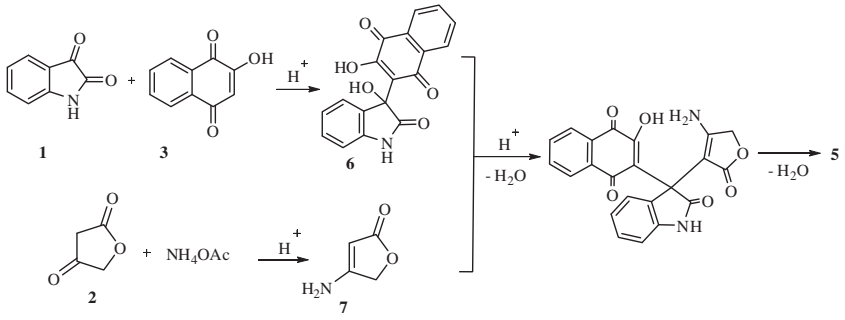
Table 2
Synthesis of spiro[benzofuroquinoline-indoline]-tetraone **5**.



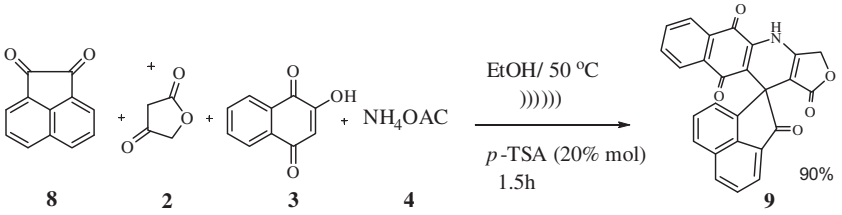
1a-e **2** **3** **4** **5a-e**

Entry	*X*	*R*	Ultrasound time (h)/yield (%)	Conventional heating yield (%)[a]
a	H	H	1.5/90	<30
b	Me	H	1.5/87	<30
c	Br	H	2/89	<30
d	NO_2	H	2/78	Trace
e	H	CH_2Ph	1.5/73	Trace

[a] Reaction time = 4 h, 50 °C in EtOH, *p*-TSA (20% mol).

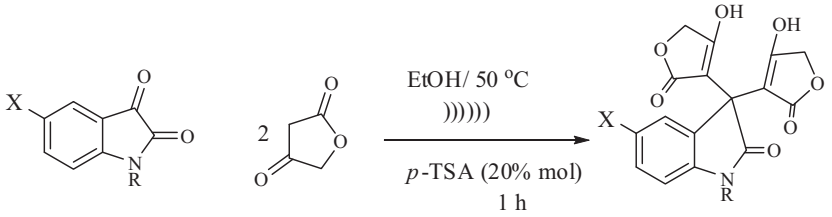


Scheme 2.



Scheme 3.

Table 3
Synthesis of oxindoles **10**.

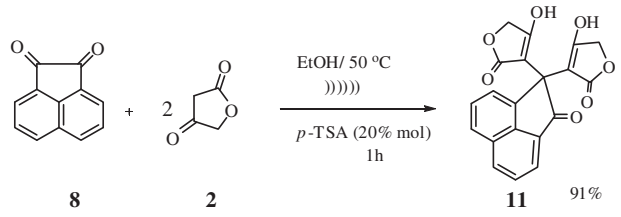


1 **2** **10a-e**

Entry	*X*	*R*	Time (h)	Yield (%)
a	H	H	1	93
b	Me	H	1.5	91
c	Br	H	2	83
d	NO_2	H	1	80
e	H	CH_2Ph	2	81

p-TSA for 1.5–2 h. The reaction can be represented as in Table 2. On the other hand, when comparing the results obtained using conventional heating with ultrasound-assisted method, we can conclude that the main advantages of ultrasound are the significant decrease of reaction times and improvement of yields (Table 2).

To the best of our knowledge, this new procedure provides the first example of an efficient method for the synthesis of spiro[benzo[*g*]furo[3,4-*b*]quinoline-11,3′-indoline]-tetraones. This method, based on four-component *p*-TSA-catalyzed reaction under ultrasonic irradiation, is the most simple and convenient and would be applicable for the synthesis of different types of spiro[benzofuroquinoline-indoline]-tetraones. Moreover, it is worth noting that two C–C and two C–N bonds were formed with concomitant creation of a spirooxindole fused benzofuroquinoline in this one-pot, four-component process.



8 2 11

Scheme 4.

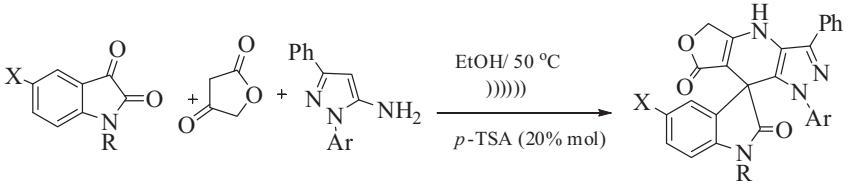
We have not established a detailed mechanism for the formation of **5**, however, a reasonable possibility is shown in Scheme 2. The formation of products **5** can be rationalized by initial formation of intermediate **6** via condensation of **1** and **3**. Subsequent addition of enamine **7** (formed in situ by reaction of **2** with ammonium acetate), to the intermediate **6** followed by cyclization afforded product **5** (Scheme 2).

As expected, when the isatin **1** was replaced by acenaphthylene-1,2-dione **8** (Scheme 3), spiro[acenaphthylene-benzo[g]furoquinoline] **9** was obtained in 90% yield under the same reaction conditions.

During our investigation on the synthesis of spirooxindole fused benzofuroquinolines, we found that in the absence of ammonium acetate and 2-hydroxy-1,4-naphthoquinone **3**, isatins **1** and tetronic acid **2** using similar conditions gave 3,3′-(2-oxoindoline-3,3-diyl)bis(4-hydroxyfuran-2(5*H*)-one) derivatives **10a–f** in 78–93% yields (Table 3). Reaction of acenaphthylene-1,2-dione **6** with tetronic acid **2** resulted in the formation of 3,3′-(2-oxo-1,2-dihydroacenaphthylene-1,1-diyl)bis(4-hydroxyfuran-2(5*H*)-one) **11** in 91% yield (Scheme 4).

To further explore the potential of this protocol for spirooxindole synthesis, we investigated the three-component reaction of

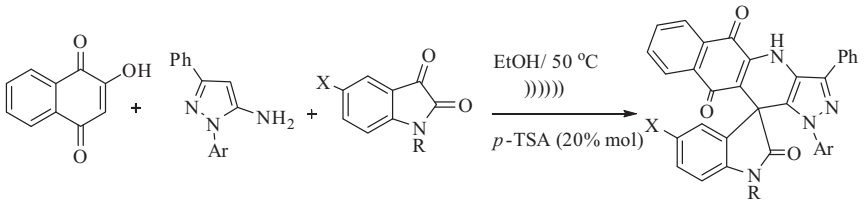
Table 4
Synthesis of spirooxindoles **13**.



1 2 12 13

Product **9**	Ar	*X*	*R*	Time (h)	Yield (%)
a	C_6H_5	H	H	3	91
b	4-NO_2–C_6H_4	H	H	2.5	92
c	C_6H_5	Br	Et	4.5	83
d	4-Br–C_6H_4	Br	Et	4	79
e	4-MeO–C_6H_4	NO_2	H	4	84
f	C_6H_5	Me	H	3.5	83

Table 5
Synthesis of spirooxindoles **15**.



14 10 1 15

Product **13**	Ar	*X*	*R*	Time (h)	Yield (%)
a	C_6H_5	H	H	2	91
b	4-MeO–C_6H_4	H	H	2.5	85
c	4-NO_2–C_6H_4	H	H	2	92
d	C_6H_5	Br	Et	2.5	83
e	C_6H_4	NO_2	H	1.5	84
f	4-NO_2–C_6H_4	NO_2	H	2	80
g	C_6H_5	Me	H	3	82

pyrazoles-5-amines **12** and isatins **1** with teronic acid **2** and obtained spiro[furo[3,4-*b*]pyrazolo[4,3-*e*]pyridine-4,3′-indoline]-diones **13** in good yields under the same reaction conditions (Table 4).

When the 2-hydroxy-1,4-naphthoquinone **14** was selected as active β-dicarbonyl compounds in the three-component reaction (Table 5), the desired spiro[benzo[*g*]pyrazolo[3,4-*b*]quinoline-11,3′-indoline]-2′,5,10(1*H*,4*H*)-triones **15** were obtained in good yields.

4. Conclusion

In conclusion, we have developed one-pot and efficient multi-component procedures for a convenient and mild synthesis of various spirooxindoles. Prominent among the advantages of these new methods are operational simplicity, good yields in short reaction times and easy work-up procedures employed.

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