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One-pot approach for the stereoselective synthesis of spirocyclopropyl oxindoles from isatins and arsonium salts

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ABSTRACT

A one-pot approach for highly stereoselective synthesis of spirocyclopropyl oxindoles **3** with good to excellent yields from the reaction of isatins **1** and arsonium salts **2** in the presence of K_2CO_3 is described. The structure of product **3** was confirmed by ^1H NMR, ^{13}C NMR, IR, MS, EA, and X-ray diffraction as well.

1. Introduction

Cyclopropane derivatives are versatile building blocks for organic synthesis because of their ability to undergo various synthetically useful transformations.[1]

Spirocyclopropyl oxindoles have been employed as building blocks for construction of more complex oxindole compounds that exhibit biological and pharmaceutical activities. Wood and co-workers reported an approach for construction of the welwitindolinone carbon skeleton with spirocyclopropyl oxindole as precursor.[2] Spirocyclopropyl oxindoles have been successfully employed as building blocks in the total synthesis of (±)-strychnofoline[3] and (−)-spirotryprostatin B[4] by Carreira and co-workers.

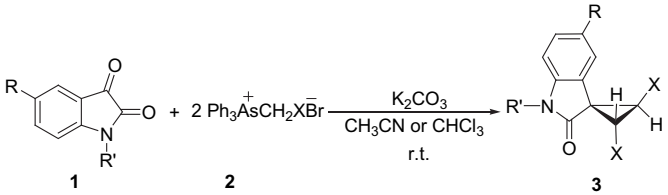
Recently, Shanmugam and co-workers reported a method for synthesis of 3-spirocyclopropane-2-indolones from the Baylis–Hillman adducts of isatin by reductive cyclization, but a mixture of cis/trans isomers was obtained from this method.[5] Thus, the development of new approach for stereoselective synthesis of spirocyclopropyl oxindoles is required.

One-pot methodology offers significant advantages such as reduction in the number of synthetic steps, energy consumption and waste production, and high efficiency.[6] Thus, considerable efforts have been made to develop new one-pot processes.

Olefins are commonly employed as starting materials in preparation of cyclopropane derivatives by Simmons–Smith reaction, transition metal-catalyzed decomposition of diazoalkanes, and Michael-initiated ring closure.[7] To our knowledge, there are no examples of the construction of spirocyclopropane-oxindole using isatin as substrate in the literature.

2. Results and discussion

In this work, we describe a new one-pot approach for stereoselective construction of spirocyclopropyl oxindoles from isatins and arsonium salts (Scheme 1).



R= H, Cl, Br, NO_2, CH_3; R'= H, Ph
X= CO_2CH_3, CN

Scheme 1. Synthesis of spirocyclopropyl oxindoles **3**.

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The experiment began with the reaction of isatin and benzoylmethylenetriphenylarsonium bromide in the presence of K_2CO_3 in DME at room temperature. A complex mixture was obtained from this reaction. We envisaged that the benzoyl group, possessing on the compounds (olefin or cyclopropane) formed from isatin and benzoylmethylenetriphenylarsonium bromide, could continue to undergo further Wittig reaction with arsonium ylide to give the complex mixture. The desired *trans*-dihydrospirocyclopropyl oxindoles were obtained when methoxycarbonyltriphenylarsonium bromide or cyanomethyltriphenylarsonium bromide was used instead of benzoylmethylenetriphenylarsonium bromide under the same reaction conditions.

To optimize the reaction condition, we then screened the bases and solvents. The results are shown in Table 1. Of particular note was that the reaction of isatin with methoxycarbonyltriphenylarsonium bromide in CH_3CN provided product **3** in nearly quantitative yield (Table 1, entry 5) and with cyanomethyltriphenylarsonium bromide in $CHCl_3$ afforded product **3** in high yield (Table 1, entry 8).

To determine the scope and limit of this reaction, a number of substituted isatins was examined. As shown in Table 2, the reaction proceeded smoothly and the desired spirocyclopropyl oxindoles **3a**–**k** were obtained in high to excellent yields. R could be H, electron-withdrawing groups or electron-donating group. Even for the *N*-phenyl substituted isatin, the reaction completed within 1 h and product **3f** was obtained in 78% yield. However when an acyclic substrate *N*-cyclohexy-2-oxo-2-phenylacetamide was used as starting material, there was no cyclopropane product observed, but a Wittig reaction product was formed. The structures of compounds **3a**–**k** were confirmed by ^1H NMR, ^{13}C NMR, MS, IR, elemental analysis, and X-ray diffraction (Fig. 1).

Table 1
Optimization of reaction condition of isatin with arsonium salts

Entry	Base	Solvent	X	R=R′	Time	Yield (%)
1	K_2CO_3	DME	CO_2CH_3	H	1 h	70
2	$NaHCO_3$	DME	CO_2CH_3	H	48 h	30
3	$KF{\cdot}2H_2O$	DME	CO_2CH_3	H	48 h	28
4	K_2CO_3	DME	CN	H	30 min	65
5	K_2CO_3	CH_3CN	CO_2CH_3	H	40 min	98
6	K_2CO_3	CH_3CN	CN	H	1 h	82
7	K_2CO_3	$CHCl_3$	CO_2CH_3	H	12 h	97
8	K_2CO_3	$CHCl_3$	CN	H	30 min	90

DME=dimethoxyethane.

Table 2
Synthesis of spirocyclopropyl oxindoles **3**

Entry	Solvent	R	R′	X	Time (min)	Yield[a] (%)
1	CH_3CN	H	H	CO_2CH_3	40	98 (**3a**)
2	CH_3CN	Cl	H	CO_2CH_3	50	97 (**3b**)
3	CH_3CN	Br	H	CO_2CH_3	40	94 (**3c**)
4	CH_3CN	NO_2	H	CO_2CH_3	60	92 (**3d**)
5	CH_3CN	CH_3	H	CO_2CH_3	60	83(**3e**)
6	CH_3CN	H	Ph	CO_2CH_3	60	78 (**3f**)
7	$CHCl_3$	H	H	CN	30	90 (**3g**)
8	$CHCl_3$	Cl	H	CN	35	75 (**3h**)
9	$CHCl_3$	Br	H	CN	30	85 (**3i**)
10	$CHCl_3$	NO_2	H	CN	35	78 (**3j**)
11	$CHCl_3$	CH_3	H	CN	40	91(**3k**)

[a] Isolated yield by silica gel chromatography.

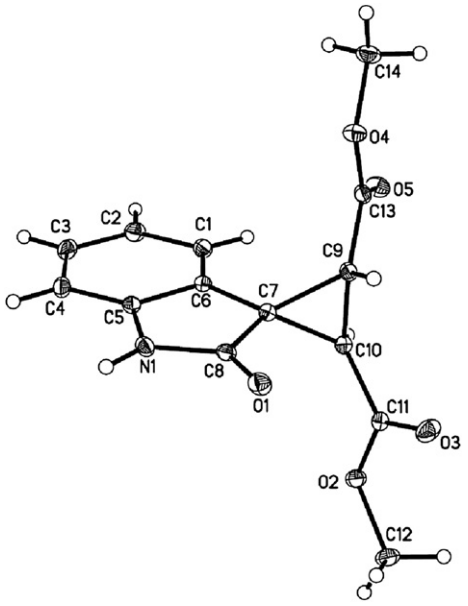
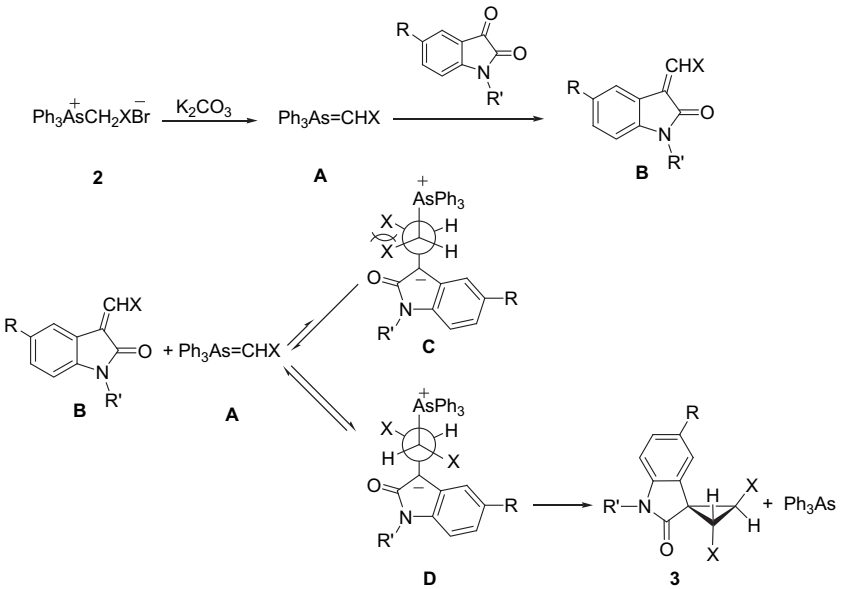


Figure 1. X-ray crystal structure of **3a**.

A plausible mechanism for this reaction is outlined in Scheme 2. First, Wittig reaction of isatin and arsonium ylide **A**, derived from arsonium salt **2** with K_2CO_3 as base, to generate olefin **B**. Then, the



Scheme 2. Mechanism for stereoselective synthesis of spirocyclopropyl oxindole **3**.

arsonium ylide **A** nucleophilically attacks the olefin **B** to provide two of intermediates **C** or **D**. Apparently the intermediate **D** should be favored over the intermediate **C**, due to the steric repulsion between two bulky groups (X) in the conformation of **C**. The selectivity may simply come from the more thermodynamically stable intermediate **D**.[8] Finally, product **3** is obtained through the intramolecular ring closure of the intermediate **D**.

3. Conclusions

In summary, we have developed a new one-pot approach for stereoselective construction of spirocyclopropyl oxindoles. This method has the advantages of reduction in the number of synthetic steps and the waste produced, mild reaction condition, simplicity in operation, high to excellent yields of products, and high stereoselectivity as well.

4. Experimental

4.1. General information

All reagents and solvents were obtained from commercial sources and used without further purification. All melting points were uncorrected. Melting points were determined on a WRS-1 digital melting point apparatus made by Shanghai Physical Optical Instrument Factory (SPOIF), China. IR spectra were measured on an AVATAR370 FT spectrometer and expressed in cm^{-1} (KBr disc). All ^1H, ^{13}C, and ^{19}F NMR spectra were recorded on a Bruker AM-500, using $CDCl_3$ as solvent. Mass spectra were recorded on an HP5989A mass spectrometer. Elemental analyses were measured on the elementar vario EL III. X-ray crystal data were collected with a Bruker Smart Apex2 CCD. Flash chromatography was performed on columns of silica gel (20–30 μ).

4.2. Typical procedure for the preparation of *trans*-2,3-dihydrospiro[cyclopropane-isatin] 3a–k

A mixture of isatin **1** (1 mmol), arsonium salt **2** (2 mmol), and K_2CO_3 (828 mg, 6 mmol) was stirred at room temperature in CH_3CN or $CHCl_3$ (5 mL). The completion of the reaction was determined by TLC. The solvent CH_3CN or $CHCl_3$ was removed under reduced pressure and the residue was run on a silica gel chromatographic column (petroleum ether–ethyl acetate, 4:1). The desired products **3** could be obtained.

4.2.1. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-1′,3′-dihydro-indol-2′-one ***3a***. White solid, mp 123–124 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.28 (d, J=7.5 Hz, 1H, CH), 3.33 (d, J=7.5 Hz, 1H, CH), 3.71 (s, 3H, CH_3), 3.74 (s, 3H, CH_3), 6.99–7.03 (m, 2H, Ph–H), 7.24–7.27 (m, 1H, Ph–H), 7.30–7.32 (m, 1H, Ph–H), 9.41 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 35.2, 35.3, 38.0, 52.6, 52.8, 110.3, 122.5, 122.9, 124.6, 128.6, 141.6, 166.3, 167.5, 173.9. IR (KBr) 3496, 1740, 1697, 1623, 1319, 1220 cm^{-1}. MS (EI) (m/z) (%): 275 (M^+, 9), 195 (100). Anal. Calcd for $C_{14}H_{13}NO_5$ (%): C, 61.09; H, 4.76; N, 5.09. Found (%): C, 60.97; H, 4.82; N, 5.22.

4.2.2. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-5′-chloro-1′,3′-dihydro-indol-2′-one ***3b***. White solid, mp 178–179 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.27 (d, J=7.5 Hz, 1H, CH), 3.30 (d, J=7.5 Hz, 1H, CH), 3.75 (s, 3H, CH_3), 3.76 (s, 3H, CH_3), 6.86 (d, J=8.5 Hz, 1H, Ph–H), 7.24 (d, J=8.5 Hz, 1H, Ph–H), 7.36 (s, 1H, Ph–H), 7.85 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 35.5, 35.6, 37.8, 52.8, 52.9, 111.2, 123.6, 126.3, 128.1, 128.6, 140.1, 165.1, 167.3, 173.5. IR (KBr) 3177, 1737, 1710, 1622, 1322, 1211 cm^{-1}. MS (EI) (m/z) (%): 311 (M^++2, 26), 310 (M^++1, 10), 309 (M^+, 86), 250 (100). Anal. Calcd for $C_{14}H_{12}ClNO_5$ (%): C, 54.29; H, 3.91; N, 4.52. Found (%): C, 54.30; H, 3.81; N, 4.52.

4.2.3. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-5′-bromo-1′,3′-dihydro-indol-2′-one ***3c***. White solid, mp 175–176 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.26 (d, J=7.5 Hz, 1H, CH), 3.31 (d, J=7.5 Hz, 1H, CH), 3.73 (s, 3H, CH_3), 3.75 (s, 3H, CH_3), 6.89 (d, J=8.5 Hz, 1H, Ph–H), 7.39 (d, J=8.5 Hz, 1H, Ph–H), 7.47 (s, 1H, Ph–H), 9.35 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 35.5, 35.6, 37.7, 52.8, 52.9, 111.7, 115.3, 126.3, 126.7, 131.5, 140.6, 165.9, 167.3, 173.4. IR (KBr) 3304, 1734, 1619, 1326, 1212 cm^{-1}. MS (EI) (m/z) (%): 356 (M^++2, 14), 355 (M^++1, 86), 353 (M^+−1, 87), 296 (100). Anal. Calcd for $C_{14}H_{12}BrNO_5$ (%): C, 47.48; H, 3.42; N, 3.95. Found (%): C, 47.44; H, 3.46; N, 4.00.

4.2.4. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-5′-nitro-1′,3′-dihydro-indol-2′-one ***3d***. Light yellow solid, mp 241–242 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.34 (d, J=8.0 Hz, 1H, CH), 3.45 (d, J=8.0 Hz, 1H, CH), 3.77 (s, 6H, CH_3), 7.06 (d, J=8.5 Hz, 1H, Ph–H), 8.26 (d, J=8.5 Hz, 1H, Ph–H), 8.32 (s, 1H, Ph–H), 8.53 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 36.0, 37.4, 53.0, 109.6, 119.6, 125.4, 125.5, 143.6, 146.7, 165.4, 166.9, 173.0. IR (KBr) 3276, 1737, 1627, 1338, 1227 cm^{-1}. MS (EI) (m/z) (%): 320 (M^+, 48), 261 (100). Anal. Calcd for $C_{14}H_{12}N_2O_7$ (%): C, 52.51; H, 3.78; N, 8.75. Found (%): C, 52.52; H, 3.83; N, 8.75.

4.2.5. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-5′-methyl-1′,3′-dihydro-indol-2′-one ***3e***. Light yellow solid, 289–290 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 2.31 (s, 3H), 3.26 (d, J=7.5 Hz, 1H, CH), 3.28 (d, J=7.5 Hz, 1H, CH), 3.72 (s, 3H), 3.74 (s, 3H), 6.82 (d, J=8.0 Hz, 1H, Ph–H), 7.05 (d, J=8.0 Hz, 1H, Ph–H), 7.13 (s, 1H, Ph–H), 8.21 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 21.3, 35.5, 37.9, 53.0, 110.0, 123.7, 124.7, 125.4, 128.9, 143.6, 166.3, 173.5. IR (KBr) 3377, 1731, 1703, 1627, 1319, 1222 cm^{-1}. MS (EI) (m/z) (%): 291 (M^++2, 2), 290 (M^++1, 15), 289 (M^+, 90), 61 (100). Anal. Calcd for $C_{15}H_{15}NO_9$ (%): C, 62.28; H, 5.23; N, 4.84. Found (%): C, 62.31; H, 5.32; N, 4.98.

4.2.6. trans-2,3-Dihydrospiro[2,3-dicarbomethoxycyclopropane]-5′-phenyl-3′-hydro-indol-2′-one ***3f***. Yellow solid, mp 61–62 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.36 (d, J=7.5 Hz, 1H, CH), 3.39 (d, J=7.5 Hz, 1H, CH), 3.71 (s, 3H, CH_3), 3.73 (s, 3H, CH_3), 6.87–7.51 (m, 9H, Ar–H). IR (KBr) 3436, 1725, 1612, 1337, 1214 cm^{-1}. MS (EI) (m/z) (%): 351 (M^+, 72), 292 (100). Anal. Calcd for $C_{20}H_{17}NO_5$ (%): C, 68.37; H, 4.88; N, 3.99. Found (%): C, 68.32; H, 4.83; N, 3.75.

4.2.7. trans-2,3-Dihydrospiro[2,3-dicyanocyclopropane]-1′,3′-dihydro-indol-2′-one ***3g***. White solid, decomp. 268 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 2.88 (s, 2H, CH), 7.05–7.07 (m, 1H, Ph–H), 7.24–7.26 (m, 1H, Ph–H), 7.44–7.47 (m, 1H, Ph–H), 7.53–7.55 (m, 1H, Ph–H), 7.77 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.2, 35.5, 111.5, 114.5, 121.4, 123.0, 123.4, 130.8, 144.2, 171.7. IR (KBr) 3364, 2253, 1721, 1625, 1472, 1221 cm^{-1}. MS (EI) (m/z) (%): 210 (M^++1, 20), 209 (M^+, 100). Anal. Calcd for $C_{12}H_7N_3O$ (%): C, 68.89; H, 3.37; N, 20.09. Found (%): C, 68.67; H, 3.32; N, 20.09.

4.2.8. trans-2,3-Dihydrospiro[2,3-dicyanocyclopropane]-5′-chloro-1′,3′-dihydro-indol-2′-one ***3h***. White solid, decomp. 290 °C. ^1H NMR (500 MHz, $CDCl_3$) δ: 3.28 (s, 2H, CH), 7.16 (d, J=8.5 Hz, 1H, Ph-H), 7.37 (s, 1H), 7.48 (d, J=8.5 Hz, 1H, Ph–H), 10.25 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.7, 35.4, 112.9, 114.4, 123.4, 123.6, 127.7, 130.8, 143.3, 171.5. IR (KBr) 3277, 2257, 1725, 1624, 1479, 1214 cm^{-1}. MS (EI) (m/z) (%): 245 (M^++2, 32), 244 (M^++1, 14), 243 (M^+, 100). Anal. Calcd for $C_{12}H_6ClN_3O$ (%): C, 59.15; H, 2.48; N, 17.25. Found (%): C, 58.92; H, 2.48; N, 16.96.

4.2.9. trans-2,3-Dihydrospiro[2,3-dicyanocyclopropane]-5′-bromo-1′,3′-dihydro-indol-2′-one ***3i***. White solid, decomp. 275 °C. ^1H NMR

(500 MHz, $CDCl_3$) δ: 2.88 (s, 2H, CH), 6.95 (d, J=8.5 Hz, 1H, Ph–H), 7.59 (d, J=8.5 Hz, 1H, Ph–H), 7.63 (s, 1H), 7.76 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 19.8, 35.3, 113.4, 114.5, 114.9, 124.0, 126.2, 133.7, 143.7, 171.4. IR (KBr) 3279, 2257, 1724, 1620, 1476, 1212 cm^{-1}. MS (EI) (m/z) (%): 290 (M^++2, 14), 289 (M^++1, 100), 288 (M^+, 18), 287 (M^+−1, 100). Anal. Calcd for $C_{12}H_6BrN_3O$ (%): C, 50.03; H, 2.10; N, 14.59. Found (%): C, 49.69; H, 2.07; N, 14.52.

4.2.10. trans-2,3-Dihydrospiro[2,3-dicyanocyclopropane]-5′-nitro-1′,3′-dihydro-indol-2′-one ***3j***. Light yellow solid, decomp. 310 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 3.40 (s, 2H, CH), 7.37 (d, J=8.5 Hz, 1H, Ph–H), 8.30 (s, 1H), 8.43 (d, J=8.5 Hz, 1H, Ph–H), 10.68 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 20.2, 35.3, 111.6, 114.3, 118.7, 123.0, 127.6, 143.8, 150.4, 172.1. IR (KBr) 3383, 2258, 1750, 1624, 1329, 1206 cm^{-1}. MS (EI) (m/z) (%): 255 (M^++1, 17), 254 (M^+, 100). Anal. Calcd for $C_{12}H_6N_4O_3$ (%): C, 56.70; H, 2.38; N, 22.04. Found (%): C, 56.81; H, 2.32; N, 21.98.

4.2.11. trans-2,3-Dihydrospiro[2,3-dicyanocyclopropane]-5′-methyl-1′,3′-dihydro-indol-2′-one ***3k***. Light yellow solid, 262–263 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 2.41 (s, 3H), 2.86 (s, 2H, CH), 6.94 (d, J=8.0 Hz, 1H, Ph-H), 7.23 (d, J=8.0 Hz, 1H, Ph–H), 7.31 (s, 1H, Ph–H), 7.88 (br s, 1H, NH). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 18.3, 21.4, 35.0, 110.9, 112.4, 124.0, 124.7, 131.0, 133.8, 145.4, 174.8. IR (KBr) 3349, 2254, 1719, 1631, 1489, 1216 cm^{-1}. MS (EI) (m/z) (%): 224 (M^++1, 15), 223 (M^+, 65), 57 (100). Anal. Calcd for $C_{13}H_9N_3O$ (%): C, 69.95; H, 4.06; N, 18.82. Found (%): C, 69.81; H, 4.02; N, 18.98.

4.3. X-ray crystal structure data of compound 3a

Intensity data were collected at 293(2) K on Bruker P4 diffractometer with graphite monochromatized and Mo Kα radiation (λ=0.71073 Å). The structure was solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically, hydrogen atoms were included but not refined. The final cycle of full matrix least-squares refinement was based on F^2, respectively. All calculations were performed using SHELXS-97 and SHELXL-97 programs.

The crystallographic date of **3a** has been deposited to the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-682951. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, GB-Cambridge CB2 1EZ; fax: +44 1223 336 033; or e-mail: deposit@ccdc.ca.ac.uk or http://www.ccdc.ac.uk, upon request.

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