DOI: 10.1002/ejoc.201000276

An Unprecedented Route for the Synthesis of 3,3′-Biindoles by Reductive Cyclization of 3-[2-Nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles Mediated by Iron/Acetic Acid

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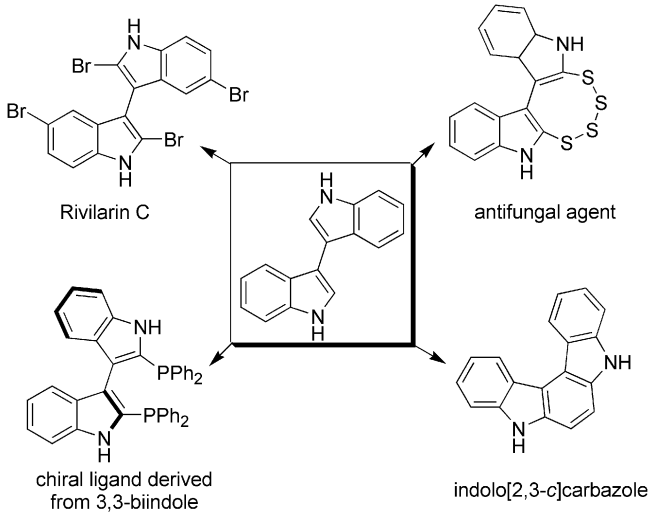
Keywords: Nitrogen heterocycles / Iron / Michael addition / Nitroalkenes / Cyclization

An unprecedented route for the synthesis of 3,3′-biindoles is developed. Both symmetrical and unsymmetrical 3,3′-biindoles could be generated by this method. Mild conditions, high yields of the products, and environmentally acceptable reagent are the merits of the procedure.

Introduction

Indoles, a major class of nitrogen heterocycles, are embedded in a number of natural and pharmaceutically important compounds and they have been the focus of numerous biological studies during the last several years.[1] Derivatives of indole are known to possess various biological properties including antibacterial, cytotoxic, antioxidative, and insecticidal activities.[2] Among the indole derivatives, biindoles are most important because of their wide range of applications in pharmaceuticals and functional materials.[3] In addition, biindoles are known to be very good diene systems for Diels–Alder reactions for the construction of indolo carbazoles, which are the core skeleton of structurally unique natural products possessing a wide range of biological activities.[4] In particular, 3,3′-biindoles are found in many natural products and several biologically active compounds.[5] Moreover, 3,3′-biindole derivatives have been used for treating a protein folding disorder such as Alzheimer's disease, dementia, Parkinson's disease, Huntington's disease, and prion-based spongiform encephalopathy.[6] Some of the synthetic utilities of the 3,3′-biindoles are shown in the Scheme 1.



Scheme 1. Synthetic utilities of 3,3′-biindole derivatives.

Owing to the importance of biindoles, numerous methods have been reported for the synthesis of biindole derivatives. However, the majority of the currently available protocols describe the synthesis of 2,3′-biindoles,[7] where few of them report on the construction of 2,2′- and other biindoles.[8] As far as the synthesis of 3,3′-biindoles is concerned, to the best of our knowledge only three multistep synthetic routes are known to reach the target. The most frequently used method involves the reaction of isatin with indole in the presence of a basic catalyst to obtain an intermediate hydroxy compound, which upon treatment with a Lewis acid generates the 3,3′-biindole.[9] The other two methods are based on palladium-catalyzed cross-coupling reactions (Scheme 2).[10] A noticeable limitation with the former method is that it requires longer times and the overall yields of the products are moderate. Whereas in the latter two cases, prefunctionalization of the indoles to their halides and the use of complex starting materials are required. The above-described facts drove us to search for a better alternative.

On the other hand, the reductive cyclization of nitro compounds is the predominantly employed method for the construction of indoles and related N-heterocompounds.[11] Leimgruber-Batcho, Bartoli, and Reissert reactions, the

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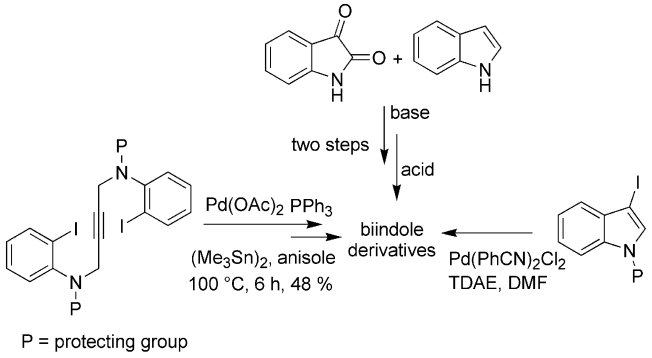
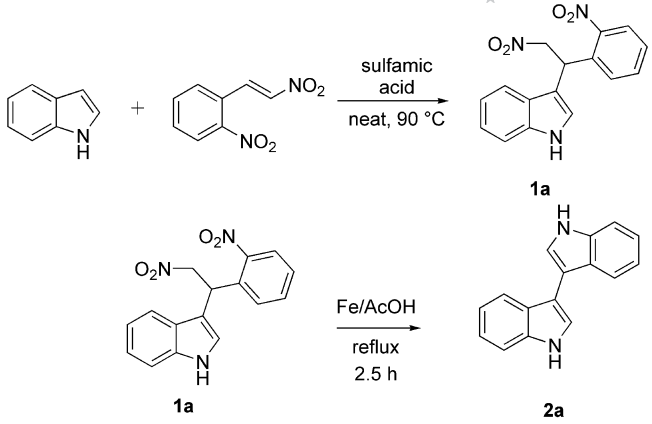
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/ejoc.201000276.



Scheme 2. Previously reported methods for the synthesis of 3,3′-biindole derivatives.



Scheme 3. Route to the synthesis of 3,3′-biindolyl derivatives.

transition-metal-catalyzed reductive N-heteroannulation of *o*-nitrostyrenes, and the Cadogan cyclization are all well-known methods in this category.[12] Recently, we have adopted Fe/acetic acid as a reductive cyclizing agent for the synthesis of N-heterocompounds,[13] as it is an inexpensive, environmental friendly, readily available reagent. A plethora of literature cited this reagent for the construction of several heterocyclic compounds including quinolines, 2*H*-pyrrolo-[3,4-*c*]quinolines, thieno[2,3-*b*][1,4]thiazin-2-(3*H*)-ones, 3,6-dimethyl-9*H*-4,5,9-triazaphenanthren-10-ones, pyrrolo[3,2-*b*]indole, chiral 3-substituted benzodiazepinone aziridines, pyrrolo[2,1-*c*][1,4]benzodiazepines derivatives, and α-methylene-γ-butyrolactams.[14] Furthermore, this reagent system has successfully been applied to the synthesis of indole derivatives.[15] Herein, we wish to introduce an unprecedented approach towards the synthesis of 3,3′-biindole derivatives through the reductive cyclization of 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles.

Results and Discussion

Our two-step synthetic strategy of 3,3′-biindoles involved the Michael addition of indole to a 1-nitro-2-(2-nitrovinyl)-benzene to obtain a precursor compound, which upon further treatment with iron/acetic acid produces the corresponding 3,3′-biindole derivative (Scheme 3). Initially, we focused our attention on the synthesis of precursor compounds such as 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles. A wide range of methods are available for the Michael addition of indoles to nitroalkenes.[16] However, very few methods have described the Michael addition of indoles to 1-nitro-2-(2-nitrovinyl)benzenes.[17] At this juncture, we found the sulfamic acid was the suitable catalyst in terms of reaction time and yield. The use of this catalytic system for the Michael addition of indoles to nitroalkenes has been described by Zuo et al.[18] However, they did not show any example with 1-nitro-2-(2-nitrovinyl)benzenes. Hence, by modifying the reaction conditions, we prepared structurally diverse 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles in good yields.

Previously, we reported that the reagent Fe/AcOH is an excellent reductive cyclizing agent for the synthesis of indolylquinoline derivatives.[13] Taking cues from these experiments, we anticipated that the precursor compounds, 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles, could be easily converted into their corresponding 3,3′-biindoles by reductive cyclization. In this regard, we first carried out the reaction of 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles with Fe/acetic acid. The best results could be obtained when **1a** (1 mmol) was treated with Fe powder (6 mmol) in acetic acid (5 mL) at reflux for 2.5 h to furnish desired 3,3′-biindole **2a** in excellent yield.

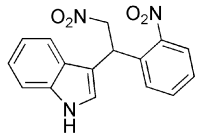
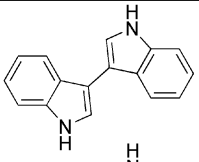
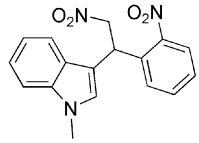
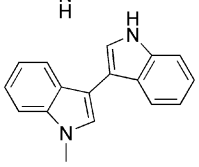
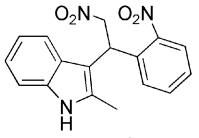
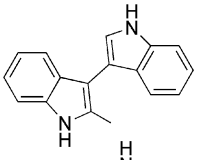
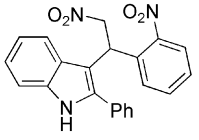
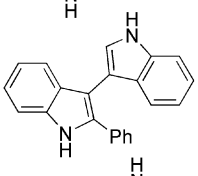
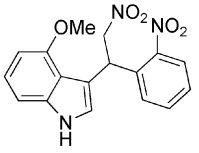
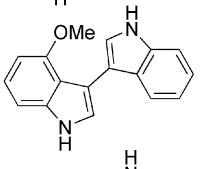
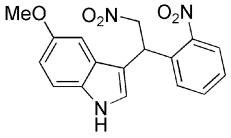
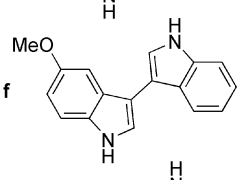
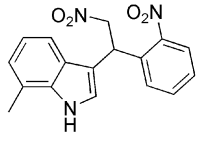
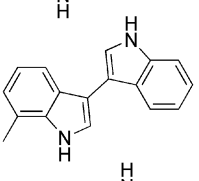
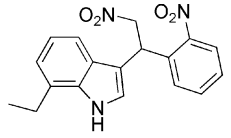
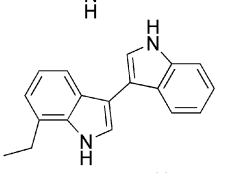
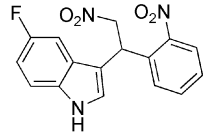
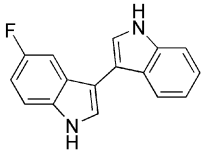
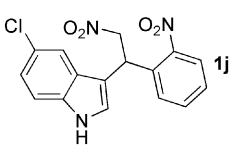
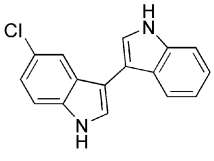
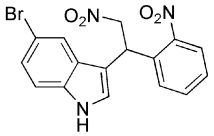
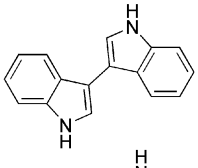
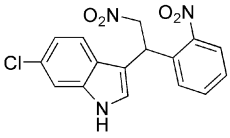
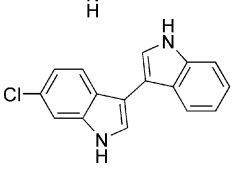
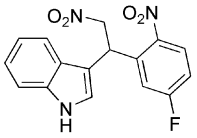
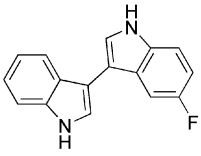
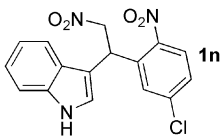
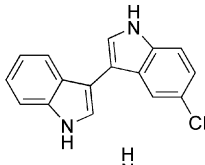
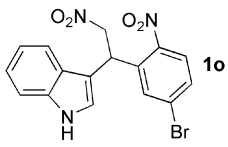
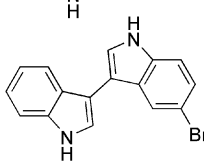
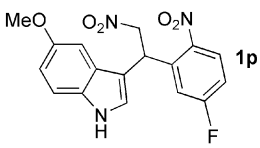
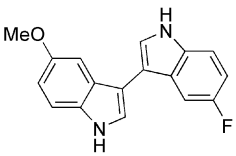
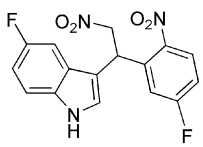
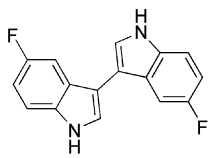
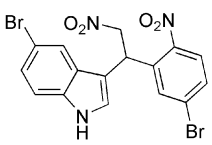
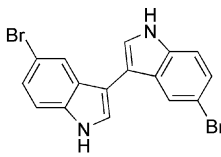
Encouraged by this result, we applied this methodology for Michael adducts **1a**–**r**, which were obtained from different substituted 1-nitro-2-(2-nitrovinyl)benzenes and various substituted indoles, to synthesize substituted 3,3′-biindoles **2a**–**o** in excellent yields. Product yields were not influenced by substituents present on either the indole or benzene components in the precursor compounds. It is interesting to note that methoxy (Table 1, Entries 5, 6, and 16), chloride and bromide (Table 1, Entries 14, 15, and 18), and fluoride (Table 1, Entries 13, 16, and 17) substituents are well tolerated under the present reaction conditions.

It is known that usually, under Fe/AcOH conditions, nitro compounds give *N*-acetylated products as side products,[19] But under our conditions, we did not find any such products, as evidenced from analysis of the ^{1}H NMR spectra of the crude product.

All the compounds were characterized by ^{1}H and ^{13}C NMR spectroscopy and HRMS. Further, the structures of the compounds were confirmed by single-crystal X-ray analysis of representative examples of symmetrical and unsymmetrical biindoles **2a** and **2d**. The crystal structures both compounds are depicted in Figure 1.

Three plausible mechanistic routes can be envisaged for the formation of 3,3′-biindole derivatives from 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indole. In the first route, the nitro group on the aromatic ring is reduced in the presence of Fe/AcOH to obtain the intermediate 2-[1-(1*H*-indol-3-yl)-2-nitroethyl]aniline, which further undergoes tautomerization followed by cyclization to form intermediate B. Upon losing

Table 1. Reductive cyclization of 3-[2-nitro-1-(2-nitrophenyl)ethyl]-1*H*-indoles using Fe/acetic acid (**1a**–**r**, **2a**–**o**).

Entry	Substrate	Product	Yield[a,b] [%]
1	 **1a**	 **2a**	79
2	 **1b**	 **2b**	83
3	 **1c**	 **2c**	82
4	 **1d**	 **2d**	81
5	 **1e**	 **2e**	83
6	 **1f**	 **2f**	83
7	 **1g**	 **2g**	80
8	 **1h**	 **2h**	83
9	 **1i**	 **2i**	83
10	 **1j**	 **2j**	84
11	 **1k**	 **2k**	83
12	 **1l**	 **2l**	84
13	 **1m**	 **2i**	82
14	 **1n**	 **2j**	80
15	 **1o**	 **2k**	83
16	 **1p**	 **2m**	84
17	 **1q**	 **2n**	83
18	 **1r**	 **2o**	82

[a] All the reactions were performed on a 1-mmol scale under reflux for 2.5 h. [b] Isolated yields.

a water molecule from B, C is formed. Intermediate C undergoes aromatization by loss of HNO to biindole. In an alternative assumption, the reduction of both nitro functionalities of the precursor compound in the presence of Fe/AcOH to form intermediate D has been considered. Protonation of D is followed by elimination of ammonia to

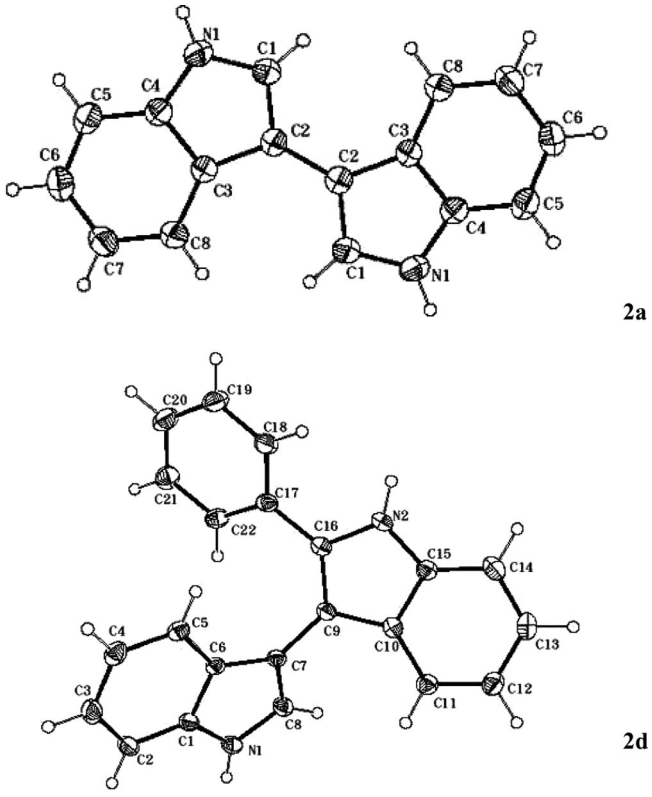


Figure 1. Crystal structures of compounds **2a** and **2d**.

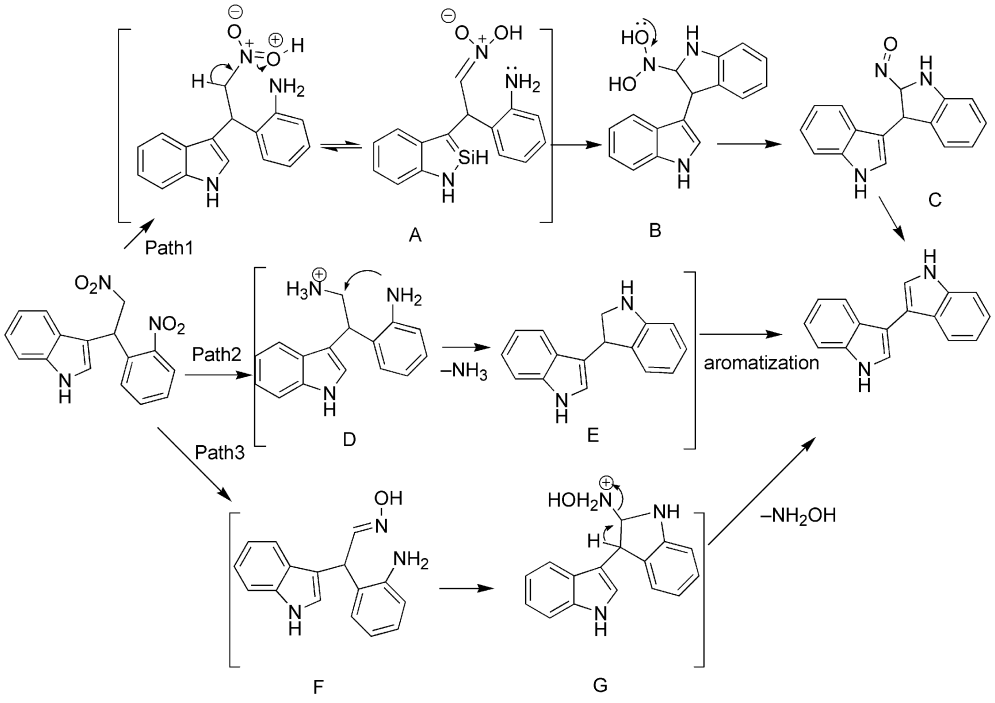
form intermediate E. Intermediate E is later aromatized to give the biindole. A third approach towards the rationalization of the conversion deals with the partial reduction of the aliphatic nitro group to the corresponding oxime F.[20] Oxime F undergoes cyclization to give intermediate G. Compound G, upon loss of a hydroxylamine molecule, results in the formation of biindole.[20] All these plausible mechanisms are depicted in Scheme 4. Although we do not have concrete evidence for any one of the proposed mechanisms, the first and the third mechanisms seem to be more reasonable than the second.

Conclusions

In conclusion, we have successfully developed an unprecedented route for the synthesis of 3,3′-biindoles. The procedure is operationally simple, convenient, and can be applicable to synthesize a wide range of 3,3′-biindoles. This methodology is useful for the synthesis of both symmetrical and unsymmetrical 3,3′-biindoles. Devoid of side products, clean reactions, and high yields of the products constitute the additional attractive features of this methodology. To the best of our knowledge, this procedure represents the simplest method for the construction of 3,3′-biindoles from simple and readily available starting materials.

Experimental Section

Representative Procedure for the Synthesis of 3,3′-Biindoles: To a stirred solution of **1a** (1 mmol) in acetic acid (5 mL) was added powdered Fe (6 mmol), and the reaction mixture was then heated at reflux for 2.5 h. The mixture was cooled to room temperature, and acetic acid was removed under reduced pressure. EtOAc (10 mL) was added; the mixture was stirred for 2 min and then filtered to remove any iron impurities. The insoluble iron residue was washed with EtOAc (10 mL). The filtrate and washings were



Scheme 4. Plausible mechanistic routes for the formation of 3,3′-biindole.

combined and dried with anhydrous $MgSO_4$. The solvent was removed under reduced pressure, and the crude product was purified by flash column chromatography (petroleum ether/ethyl acetate) to yield the expected product.

1*H*,1′*H*-3,3′-Biindole (2a): White solid, m.p. 274–276 °C. ^{1}H NMR (400 MHz, [D$_6$]DMSO): δ = 11.14 (br. s, 2 H), 7.77 (d, J = 7.8 Hz, 2 H), 7.63 (d, J = 2.0 Hz, 2 H), 7.44 (d, J = 8.0 Hz, 2 H), 7.14 (t, J = 7.2 Hz, 2 H), 7.05 (t, J = 7.1 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, [D$_6$]DMSO): δ = 136.4, 126.0, 121.8, 121.2, 119.5, 118.8, 111.5, 109.7 ppm. MS (EI): *m*/*z* (%) = 232 (100) [M]$^+$. HRMS: calcd. for $C_{16}H_{12}N_2$ [M]$^+$ 232.0995; found 232.0996.

2-Phenyl-1*H*,1′*H*-3,3′-biindole (2d): White solid, m.p. 227–229 °C. ^{1}H NMR (400 MHz, [D$_6$]DMSO) δ = 11.47 (br. s, 1 H), 11.21 (br. s, 1 H), 7.56 (s, 1 H), 7.55 (s, 1 H), 7.47–7.43 (m, 2 H), 7.40 (s, 1 H), 7.37 (d, J = 7.8 Hz, 1 H), 7.28–7.24 (m, 2 H), 7.20 (d, J = 6.9 Hz, 1 H), 7.18–7.12 (m, 1 H), 7.07 (t, J = 7.3 Hz, 1 H), 7.01–6.97 (m, 2 H), 6.82 (t, J = 7.3 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, [D$_6$]DMSO): δ = 136.4, 136.2, 133.8, 133.2, 129.5, 128.2, 127.2, 126.9, 126.7, 124.2, 121.7, 120.9, 119.6, 119.3, 119.0, 118.5, 111.6, 111.2, 108.6, 106.5 ppm. MS (EI): *m*/*z* (%) = 308 (100) [M]$^+$. HRMS: calcd. for $C_{22}H_{16}N_2$ [M]$^+$ 308.1308; found 308.1313.

CCDC-777828 (**2a**) and -777829 (**2d**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supporting Information (see footnote on the first page of this article): Experimental procedures, spectral data of **2a**–**o**, and copies of the NMR spectra of **2a**–**o**.

Acknowledgments

We would like to express our sincere gratitude to the National Science Council of the Republic of China and National Taiwan Normal University for providing financial support to pursue this work.

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Received: March 1, 2010
Published Online: June 9, 2010

