An Efficient One-Pot Synthesis of Dispiropyrrolidine Derivatives Through 1,3-Dipolar Cycloaddition Reactions Under Ultrasound Irradiation

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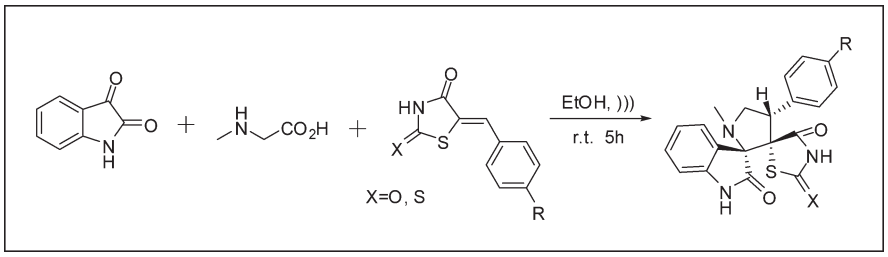

A series of dispiropyrrolidine derivatives were synthesized via the three-component 1,3-dipolar cycloaddition reaction of isatin, sarcosine and 5-arylidene-1,3-thiazolidine-2,4-dione or 5-arylidene-4-thioxo-1,3-thiazolidine-2-one in ethanol under ultrasound irradiation. This protocol has the advantages of mild reaction conditions, higher yields, and shorter reaction time.

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INTRODUCTION

Multicomponent reactions (MCRs) are a valuable approach to synthesize novel compounds [1]. The MCRs strategy offers significant advantages over conventional liner-type synthesis, by which three or more simple and flexible molecules are brought together and build up structural complexity and diversity rapidly and effectively [2]. Multicomponent 1,3-dipolar cycloaddition reactions play a key role in the synthesis of five-membered heterocyclic compounds [3]. 1,3-Dipolar cycloaddition of ylidic species, such as azomethine ylides with dipolarophiles, is a powerful method for the construction of biologically active five-membered heterocyclic compounds [4] especially pyrrolidine derivatives [5].

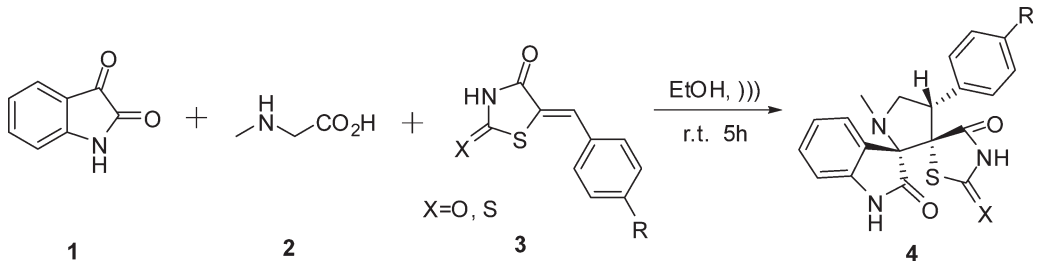
Indole and indoline fragments are important moieties of a large number of a variety of natural products and medicinal agents [6], and some of indolines, spiro-annulated with heterocycles in the 3-position, have shown high biological activity [7]. The spirooxindole system is the core structure of many pharmacological agents and natural alkaloids [8]. Pyrrolidines are important heterocycles which have glucosidase inhibitory activity, potent antiviral, antibacterial, antidiabetic, and anticancer activities [9]. Narayanan et al. recently has reported the synthesis of a series of novel dispiropyrrolidines by the 1,3-dipolar cycloaddition reaction with 5-arylidene-1,3-thiazolidine-2,4-dione and 5-arylidene-4-thioxo-1,3-thiazolidine-2-one [10]. However, this method suffers from the drawbacks of long reaction time, moderate yield, and use of toxic organic solvent. Thus, there is a certain need for the development of an alternate route for the production of dispiropyrrolidine derivatives, which surpasses those limitations.

Ultrasound has increasingly been used in organic synthesis in recent years and a large number of organic reactions can be carried out in higher yield, shorter reaction time, and milder conditions under ultrasonic irradiation [11]. As a part of our interest in the synthesis of 1,3-dipolar cycloaddition involves azomethine ylides, and in a continuation of using green chemistry tools to improve heterocyclic synthesis [12], we wish to report a rapid and efficient one-pot synthesis of dispiropyrrolidine derivatives under ultrasonic irradiation (Scheme 1).

RESULTS AND DISCUSSION

Choosing an appropriate solvent is of crucial importance for the successful organic synthesis. To search for the optimum solvent, the reaction of isatin **1a**, sarcosine **2**, and (Z)-5-(4-methylbenzylidene)-2-thioxothiazolidin-4-one **3g** was examined in different solvent for the synthesis of **4g** at different conditions (Scheme 2). The results are summarized in Table 1.

Scheme 1



First, to verify the effect of ultrasound irradiation, we have performed the reaction in methanol under ultrasonic irradiation. It can been seen from the Table 1 that the reaction time is only 6 h, and the yield of the reaction is 70%, while under the reported method the result of it was not satisfactory (Table 1, entries 1 and 2). It showed that ultrasound irradiation improved the result. The reason may be the phenomenon of cavitations produced by ultrasound [13]. Cavitation (mechanical) effect which was the physical process, creates, enlarges, and implodes gaseous and vapor-phase cavities in an irradiated liquid. Cavitation induces very high local temperatures and pressure inside the bubbles (cavities), leading to a turbulent flow in the liquid and enhanced mass transfer, thus producing a variety of high energy species in solution [14]. It has been observed that a favorable acceleration in reaction rate occurs when compared to classical conditions (i.e., under reflux).

Then, we found that the solvents also played an important role in this 1,3-dipolar cycloaddition reaction. Further studies established that absolute ethanol was the best choice among the solvents (ethanol, methanol, acetonitrile, dioxane, THF, chloroform, and water) screened. The reaction did not furnish the desired product in water and poor yields in acetonitrile, dioxane, THF, and chloroform (Table 1, entries 4–8). So ethanol was chosen as the solvent for all further reactions. Under these optimized reaction conditions, a series of dispiropyrrolidine derivatives **4** were synthesized. The results are summarized in Table 2.

As shown in Table 2, this protocol could be applied to the aromatic rings with either electron-withdrawing groups (such as halide and nitro groups) or electron-donating groups (such as alkyl group). The structures of the products were established on the spectroscopic data (IR, ^{1}H NMR, and HRMS). Compared with the reported method [10], the dramatic improvement observed is with regard to reaction time and yield. In reported methods which needed toxic organic solvent (methanol), the reaction time was about 15–25 h, and the yield was only 54–65%; whereas in the present procedure, the reactions only needed 5 h to get a good result (71–85%) rose by about 20%, and did not require toxic organic solvent.

The structures of final products **4** were established by IR, ^{1}H NMR, and HRMS spectroscopy. The structure of compound **4k** was confirmed by X-ray analysis. The X-ray crystal structure of **4k** is represented in Figure 1.

Though the detailed mechanism of this reaction has not been clarified yet, the formation of **4** can be explained by the possible mechanism presented in Scheme 3. The reaction proceeds through the generation of azomethine ylide (dipole **5**) via the condensation of isatin **1** with sarcosine **2** and decarboxylation. The dipolarophiles **3** react with azomethine ylides (dipole **5**) in ethanol to give the desired products dispiropyrrolidine derivatives **4**.

In summary, we developed an efficient three-component reaction of isatin, sarcosine and 5-arylidene-1,3-thiazolidine-2,4-dione or 5-arylidene-4-thioxo-1,3-thiazolidine-2-one for the synthesis of dispiropyrrolidine derivatives in ethanol under ultrasound irradiation. Compared with the previous method, this new protocol has the advantages of mild reaction conditions, higher yields, and shorter reaction time.

Scheme 2

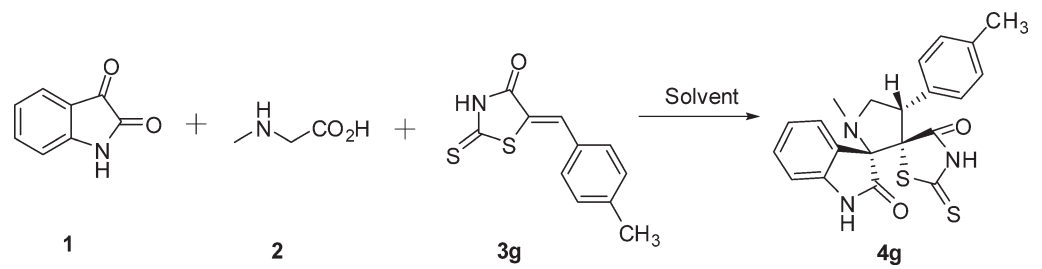


Table 1

Optimization for the synthesis of **4g**.

Entry	Solvent	Method	Time (h)	Isolated yield (%)
1	Methanol	U.S.	6	70
2	Methanol	Reflux	21	61
3	Ethanol	U.S.	5	82
4	Acetonitrile	U.S.	12	30
5	Dioxane	U.S.	12	43
6	THF	U.S.	6	51
7	Chloroform	U.S.	6	67
8	Water	U.S.	24	None

Table 2

The Synthesis of dispiropyrrolidine derivatives **4** under ultrasound irradiation.

Entry	X	R	Product	Isolated yield (%)
1	O	CH_3	**4a**	75
2	O	H	**4b**	72
3	O	NO_2	**4c**	83
4	O	F	**4d**	71
5	O	Cl	**4e**	75
6	O	Br	**4f**	78
7	S	CH_3	**4g**	82
8	S	H	**4h**	80
9	S	NO_2	**4i**	85
10	S	F	**4j**	81
11	S	Br	**4k**	84

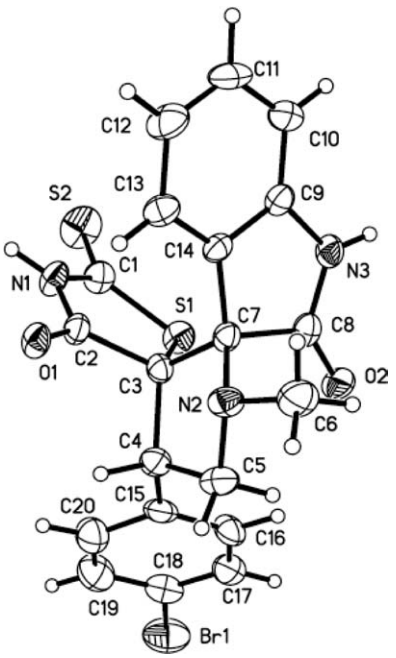


Figure 1. The X-ray structure of **4k**.

EXPERIMENTAL

Commercial solvents and reagents were used as received. Melting points are uncorrected. Ultrasonication was performed in a KQ-250E medical ultrasound cleaner with a frequency of 40 KHz and an output power of 600 W. IR spectra was recorded on Varian F-1000 spectrometer in KBr with absorptions in cm^{-1}. ^1H NMR was determined on Varian Inova-300/400 MHz spectrometer in DMSO-d_6 solution. J values are in Hz. Chemical shifts are expressed in ppm downfield from internal standard TMS. HRMS data were obtained using Bruker micrOTOF-Q instrument. The preparation of 5-arylidene-1,3-thiazolidine-2,4-dione and 5-arylidene-4-thioxo- 1,3-thiazolidine-2-one were according to the literature procedure [15].

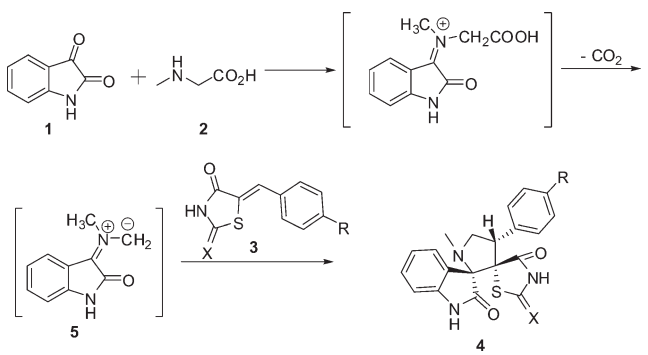
General procedure for the synthesis of dispiropyrrolidine derivatives 4. A 100-mL flask was charged with the isatin **1** (1 mmol), sarcosine **2** (1 mmol), and 5-arylidene-1,3- thiazolidine-2,4-dione or 5-arylidene-4-thioxo-1,3-thiazolidine-2-one **3** (1 mmol) in ethanol. The mixture was sonicated in the water bath of an ultrasonic cleaner under an air conditions for 5 h. After completion of reaction (monitored by TLC), the reaction mixture was filtered, and the precipitate washed with water (10 mL) and recrystallized from ethanol to afford the pure product **4**.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazoline-2″,4″-dione-4-(4-methylphenyl)pyrrolidine (4a). This compound was obtained as light yellow powder with mp 160–162°C (Lit. [10] 157°C); IR (KBr): 3222, 3059, 2945, 2866, 1703, 1618, 1468, 1387, 1159, 817, 753 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ: 2.09 (s, 3H, CH_3), 2.31 (s, 3H, CH_3), 3.44 (t, J = 7.6 Hz, 1H, CH—H), 3.88 (t, J = 9.2 Hz, 1H, CH—H), 4.43 (t, J = 8.8 Hz, 1H, CH), 6.88 (d, J = 7.6 Hz, 1H, ArH), 7.04 (t, J = 6.4 Hz, 1H, ArH), 7.19 (d, J = 7.2 Hz, 2H, ArH), 7.23 (d, J = 7.6 Hz, 1H, ArH), 7.31 (d, J = 7.2 Hz, 3H, ArH), 10.76 (s, 1H, NH), 11.97 (s, 1H, NH).

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazolidine-2″,4″-dione-4-phenyl-pyrrolidine (4b). This compound was obtained as yellow powder with mp 155–156°C (Lit. [10] 152°C); IR (KBr): 3201, 3065, 2950, 2877, 1700, 1616, 1465, 1396, 1161, 890, 751 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.06 (s, 3H, CH_3), 3.44 (t, J = 8.1 Hz, 1H, CH—H), 3.86 (t, J = 9.6 Hz, 1H, CH—H), 4.40–4.46 (m, 1H, CH), 6.84 (d, J = 7.5 Hz, 1H, ArH), 7.01 (t, J = 7.2 Hz, 1H, ArH), 7.19 (d, J = 7.5 Hz, 1H, ArH), 7.27–7.41 (m, 6H, ArH), 10.76 (s, 1H, NH), 10.99 (s, 1H, NH).

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazolidine-2″,4″-dione-4-(4-nitrophenyl)-pyrrolidine (4c). This compound was obtained as yellow powder with mp 158–160°C; IR (KBr):

Scheme 3



3218, 3063, 2947, 2864, 1759, 1689, 1604, 1519, 1468, 1345, 1160, 855, 753 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.06 (s, 3H, CH_3), 3.52 (t, J = 8.4 Hz, 1H, CH—H), 3.86 (t, J = 9.3 Hz, 1H, CH—H), 4.59 (t, J = 9.0 Hz, 1H, CH), 6.86 (d, J = 7.8 Hz, 1H, ArH), 7.02 (t, J = 7.5 Hz, 1H, ArH), 7.19 (d, J = 7.5 Hz, 1H, ArH), 7.31 (t, J = 7.8 Hz, 1H, ArH), 7.73 (d, J = 8.7 Hz, 2H, ArH), 8.21 (d, J = 8.7 Hz, 2H, ArH), 10.85 (s, 1H, NH), 12.24 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}N_4O_5S$: [M+H] 425.0914, found 425.0914.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazolidine-2″,4″-dione-4-(4-fluorophenyl)-pyrrolidine (4d). This compound was obtained as light yellow powder with mp 154–156°C (Lit. [10] 150°C); IR (KBr): 3229, 3066, 2955, 2882, 1754, 1690, 1612, 1510, 1467, 1306, 1155, 839, 737 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.06 (s, 3H, CH_3), 3.46 (t, J = 8.4 Hz, 1H, CH—H), 3.80 (t, J = 9.3 Hz, 1H, CH—H), 4.44 (t, J = 8.1 Hz, 1H, CH), 6.85 (d, J = 7.8 Hz, 1H, ArH), 7.01 (t, J = 7.5 Hz, 1H, ArH), 7.15–7.21 (m, 3H, ArH), 7.30 (t, J = 7.5 Hz, 1H, ArH), 7.44–7.49 (m, 2H, ArH), 10.78 (s, 1H, NH), 12.02 (s, 1H, NH).

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazolidine-2″,4″-dione-4-(4-chlorophenyl)-pyrrolidine (4e). This compound was obtained as light yellow powder with mp 160–162°C; IR (KBr): 3193, 3086, 2955, 2875, 1755, 1704, 1614, 1467, 1318, 1159, 826, 760 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.05 (s, 3H, CH_3), 3.46 (t, J = 8.1 Hz, 1H, CH—H), 3.80 (t, J = 9.3 Hz, 1H, CH—H), 4.43 (t, J = 8.7 Hz, 1H, CH), 6.85 (d, J = 7.8 Hz, 1H, ArH), 7.01 (t, J = 7.8 Hz, 1H, ArH), 7.18 (d, J = 7.2 Hz, 1H, ArH), 7.30 (t, J = 7.8 Hz, 1H, ArH), 7.39–7.47 (m, 4H, ArH), 10.78 (s, 1H, NH), 12.03 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}ClN_3O_3S$: [M+H] 414.0674, found 414.0673.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]thiazolidine-2″,4″-dione-4-(4-bromophenyl)-pyrrolidine (4f). This compound was obtained as light yellow powder with mp 160–161°C; IR (KBr): 3195, 3077, 2954, 2876, 1756, 1703, 1615, 1468, 1317, 1159, 823, 762 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.05 (s, 3H, CH_3), 3.46 (t, J = 8.1 Hz, 1H, CH—H), 3.80 (t, J = 9.3 Hz, 1H, CH—H), 4.42 (t, J = 8.7 Hz, 1H, CH), 6.85 (d, J = 7.8 Hz, 1H, ArH), 7.01 (t, J = 7.5 Hz, 1H, ArH), 7.18 (d, J = 7.5 Hz, 1H, ArH), 7.30 (t, J = 7.8 Hz, 1H, ArH), 7.38 (d, J = 8.4 Hz, 2H, ArH), 7.55 (d, J = 8.4 Hz, 2H, ArH), 10.78 (s, 1H, NH), 12.03 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}BrN_3O_3S$: [M+H] 458.0169, found 458.0163.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]-4″-thioxo-thiazolidine-2″-one-4-(4-methylphenyl)-pyrrolidine (4g). This compound was obtained as light yellow powder with mp 145–146°C; IR (KBr): 3199, 2948, 2849, 1711, 1617, 1513, 1428, 1323, 1296, 1198, 816, 750 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.04 (s, 3H, CH_3), 2.27 (s, 3H, CH_3), 3.42 (t, J = 8.1 Hz, 1H, CH—H), 3.82 (t, J = 9.3 Hz, 1H, CH—H), 4.36 (t, J = 8.7 Hz, 1H, CH), 6.84 (d, J = 7.8 Hz, 1H, ArH), 7.02 (t, J = 7.5 Hz, 1H, ArH), 7.15–7.21 (m, 3H, ArH), 7.30 (t, J = 7.5 Hz, 3H, ArH), 10.83 (s, 1H, NH), 13.10 (s, 1H, NH).

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]-4″-thioxo-thiazolidine-2″-one-4-phenyl-pyrrolidine (4h). This compound was obtained as red powder with mp 161–163°C; IR (KBr): 3370, 3218, 2924, 2866, 1724, 1615, 1464, 1294, 1207, 894, 757 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.05 (s, 3H, CH_3), 3.46 (t, J = 8.1 Hz, 1H, CH—H), 3.85 (t, J = 9.3 Hz, 1H, CH—H), 4.38–4.43 (m, 1H, CH), 6.84 (d, J = 7.8 Hz, 1H, ArH), 7.03 (t, J = 7.2 Hz, 1H, ArH), 7.21 (d, J = 7.5 Hz, 1H, ArH), 7.27–7.42 (m, 6H, ArH), 10.82 (s, 1H, NH), 13.11 (s, 1H, NH). HRMS Calculated for $C_{20}H_{18}N_3O_2S_2$: [M+H] 396.0835, found 396.0835.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]-4″-thioxo-thiazolidine-2″-one-4-(4-nitrophenyl)-pyrrolidine (4i). This compound was obtained as yellow powder with mp 158–159°C; IR (KBr): 3274, 2948, 2860, 1715, 1607, 1520, 1464, 1346, 1212, 855, 754 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.06 (s, 3H, CH_3), 3.53 (t, J = 8.4 Hz, 1H, CH—H), 3.85 (t, J = 9.3 Hz, 1H, CH—H), 4.58 (t, J = 8.7 Hz, 1H, CH), 6.86 (d, J = 7.5 Hz, 1H, ArH), 7.04 (t, J = 7.5 Hz, 1H, ArH), 7.21 (d, J = 7.5 Hz, 1H, ArH), 7.32 (t, J = 7.5 Hz, 1H, ArH), 7.74 (d, J = 8.7 Hz, 2H, ArH), 8.23 (d, J = 8.4 Hz, 2H, ArH), 10.90 (s, 1H, NH), 13.24 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}N_4O_4S_2$: [M+H] 441.0686, found 441.0685.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]-4″-thioxo-thiazolidine-2″-one-4-(4-fluorophenyl)-pyrrolidine (4j). This compound was obtained as light yellow powder with mp 142–143°C; IR (KBr): 3372, 3219, 3044, 2950, 2858, 1719, 1612, 1511, 1426, 1322, 1208, 835, 757 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.04 (s, 3H, CH_3), 3.46 (t, J = 8.4 Hz, 1H, CH—H), 3.78 (t, J = 6.3 Hz, 1H, CH—H), 4.39–4.44 (m, 1H, CH), 6.84 (d, J = 7.8 Hz, 1H, ArH), 7.02 (t, J = 7.5 Hz, 1H, ArH), 7.16–7.22 (m, 3H, ArH), 7.30 (t, J = 7.2 Hz, 1H, ArH), 7.45–7.49 (m, 2H, ArH), 10.84 (s, 1H, NH), 13.13 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}FN_3O_2S_2$: [M+H] 414.0741, found 414.0743.

1-N-Methyl-spiro[2.3′]oxindole-spiro[3.5″]-4″-thioxo-thiazolidine-2″-one-4-(4-bromophenyl)-pyrrolidine (4k). This compound was obtained as light yellow powder with mp 159–160°C; IR (KBr): 3199, 2954, 2870, 1711, 1619, 1429, 1327, 1208, 1076, 1007, 821, 754 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ: 2.04 (s, 3H, CH_3), 3.47 (t, J = 8.4 Hz, 1H, CH—H), 3.78 (t, J = 9.3 Hz, 1H, CH—H), 4.39 (t, J = 8.1 Hz, 1H, CH), 6.85 (d, J = 7.8 Hz, 1H, ArH), 7.03 (t, J = 7.5 Hz, 1H, ArH), 7.20 (d, J = 7.5 Hz, 1H, ArH), 7.30 (t, J = 7.2 Hz, 1H, ArH), 7.39 (d, J = 8.4 Hz, 2H, ArH), 7.56 (d, J = 8.4 Hz, 2H, ArH), 10.85 (s, 1H, NH), 13.16 (s, 1H, NH). HRMS Calculated for $C_{20}H_{17}BrN_3O_2S_2$: [M+H] 473.9940, found 473.9945.

Crystallographic data for the structure of compound **4k** has been deposited at the Cambridge Crystallographic Data Center, and the deposit number is CCDC-784779. Copy of available material can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336 033; e-mail: deposit@ccdc.cam.ac.uk).

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