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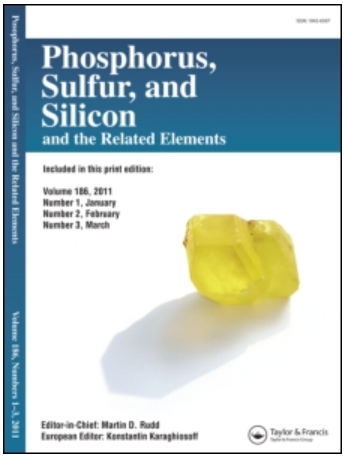

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p-TsOH Catalysed a Facile One-Pot Synthesis of Some New Substituted [1,2,4]Triazolo[3,4-*b*][1,3,4] thiadiazepines under Microwave Irradiation in Solvent-Free Conditions

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p-TsOH Catalysed a Facile One-Pot Synthesis of Some New Substituted [1,2,4]Triazolo[3,4-*b*][1,3,4] thiadiazepines under Microwave Irradiation in Solvent-Free Conditions

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A series of triazolothiadiazepines were synthesized by the reaction of 4-substituted-5-aryl-1,2,4-triazoles and chalcones using p-*TsOH as catalyst under solvent-free microwave irradiation conditions. Structure of all the prepared compounds were elucidated by elemental, IR,* ^{1}*H NMR and mass spectral data.*

Keywords Chalcones; microwave irradiation; *p*-TsOH; solvent free; thiadiazepines; triazoles

INTRODUCTION

Sulfur containing quinoline derivatives have received considerable attention due to their wide range of pharmacological activities.[1–3] Various 1,2,4-triazole derivatives are found to be associated with diverse pharmacological activities.[4–12] On the other hand, 4-substituted-5-aryl-1,2,4-triazoles are versatile synthons for constructing various biologically active heterocylcles. Further, triazolo thiadiazepine derivatives constitute an important class of organic compounds with diverse biological activities including antipharasitic, analgesic, antibacterial, anti-inflammatory and anticancer activities.[13–16] Synthetic organic

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reactions performed under non-traditional conditions are gaining popularity, primarily to circumvent growing environmental concerns.[17] Microwave–assisted heating has been shown to be an invaluable technology in synthesis since it often dramatically reduces reaction times, typically from days or hours to minutes or even seconds. And, it can also provide pure products in quantitative yields. Solvent-free reaction techniques were successfully coupled with microwave because they avoid the using of low boiling points and high vapor pressure solvents, which may sometimes lead to explosions. Additionally, it can also avoid the use of poisonous and expensive solvent, and as such can be environmentally benign, and make manipulations much easier. The use of microwave for the synthesis of organic compounds under solvent-free conditions proved to be an efficient safe and environmentally benign techniques that with shorter reaction time, high yields, and easier manipulation.[18,19] In addition, the limitations of the microwave-assisted reactions in solvents, namely the development of high pressure and need for specialized sealed vessels, are circumvented via the solid catalyst strategy, which enables organic reactions to occur rapidly at atmospheric pressures[20] and upscale the reactions on a preparative scale.[21]

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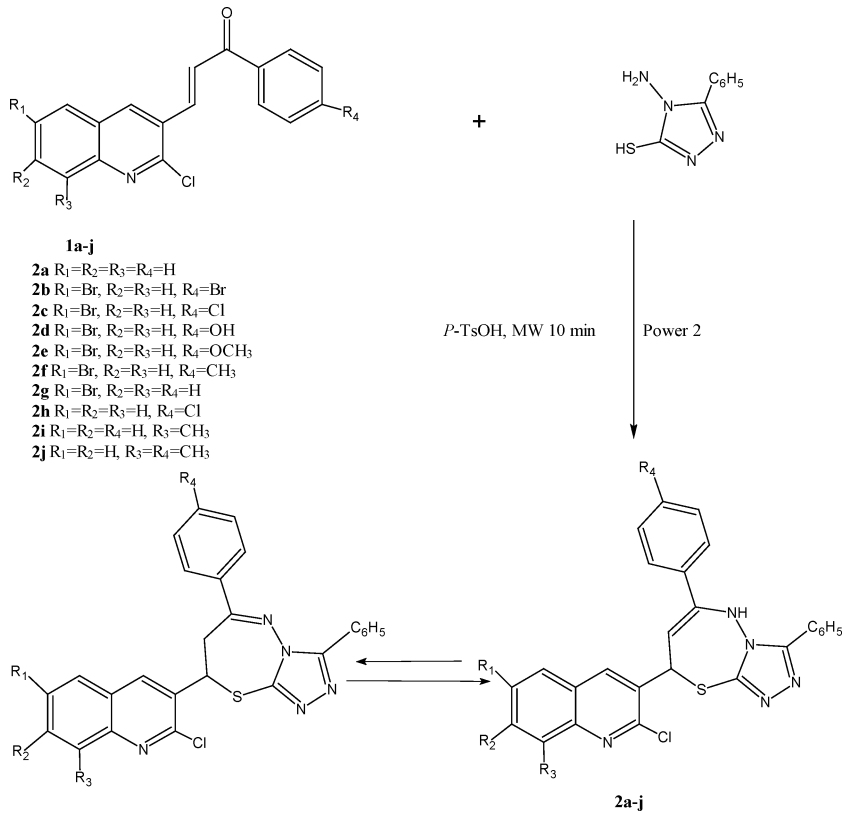
In light of the above findings and in continuation of our work on synthesis of biologically active heterocyclic compounds by the use of microwave,[22–26] we wish to report the synthesis of some new triazolo thiadiazepine derivatives **2a–j** by the condensation of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol and substituted 3-(2—chloroquinolin-3-yl)-1-phenylprop-2-en-1-ones **1a–j** and their structural elucidation by using elemental and spectral data.

RESULTS AND DISCUSSION

The starting compounds **1a–j** were prepared according to the literature method.[27] The final compounds **2a–j** were synthesised by the reaction between the substituted 3-(2—chloroquinolin-3-yl)-1-phenylprop-2-en-1-ones **1a–j** and 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol in the presence of *p*-TsOH catalyst under microwave irradiation. In presence of *p*-TsOH catalyst, the reaction takes place more efficiently, milder conditions, with greater selectively and it gives better yields within 10 min. Moreover, the work up and purification procedures are simple as the catalyst is water soluble.

The first step of a reaction catalyzed by *p*-TsOH is initiated by protonation of the oxygen atom of the chalcones, which generates cation, and it makes unsaturated group more susceptible to nucleophiles (creates more partial positive charge on the carbon atom of the unsaturated



SCHEME 1

group). So, the first step of the reaction proceeds through the conjugate addition of the highly nucleophilic -SH group to the α-β unsaturated carbon followed by subsequent condensation of -NH_2 group of triazole ring with carbonyl group of the chalcone as represented in Scheme 1 and the obtained yields are given in Table I.

In IR Spectra, of **2a** the appearance of new absorption band in the region 1610-1620 cm^{-1} indicating the presence of C = N group and also a sharp absorption band observed in the region ~760– 800 cm^{-1} corresponding to C-S-C linkage of the seven membered ring. There absence of absorption band in **2a** at 1660, 3250, 3400, and 2340 cm^{-1}, due to unsaturated carbonyl, NH_2 and SH group, respectively. The ^1H NMR spectra of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol shows signals at 13.2 ppm and δ 5.4 ppm due to –SH and —NH_2 groups, which were absent in the spectra of **2a**; this confirms that these two functional groups

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TABLE I Comparison of Obtained Yields in Microwave Method and Conventional Method

Compd.	MW method yield in g (% yield)	Conventional method yield in g (% yield)
2a	1.5537 (83)	1.254 (67)
2b	2.125 (85)	1.550 (62)
2c	1.9754 (85)	1.510 (65)
2d	1.9108 (85)	1.506 (67)
2e	2.0079 (87)	1.454 (63)
2f	1.8816 (84)	1.344 (60)
2g	1.8782 (86)	1.332 (61)
2h	1.8072 (90)	1.224 (62)
2i	1.7159 (89)	1.214 (63)
2j	1.7062 (86)	1.230 (62)

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involved in condensation. It also showed signals in the aromatic region corresponding to 16 protons, out of which 14 were attributed for aromatic protons, out of the remaining two one to —NH (δ 8.15) and another to —CH = (δ 7.65) group of the thiazepine ring. In addition, the —CH-S proton in the seven-membered ring was found to resonate at $\delta = 3.42$ ppm. The structure of **2a** was further confirmed by mass spectral studies. It gives the molecular ion peak at m/z 468 (M^+) consistent with the molecular formula $C_{26}H_{17}BrClN_5S$.

CONCLUSION

In conclusion, a simple efficient and environmentally benign method has been developed for the synthesis of novel [1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazepines under solvent-free conditions in presence of *p*—TsOH as catalyst.

EXPERIMENTAL

The purity of compounds were checked by thin layer chromatography (TLC) on silica gel using pet ether: ethyl acetate solvent system. Melting points are determined in open capillaries and are uncorrected. The FT-IR spectra were recorded on NICOLETAVATAR 360-FT-IR instrument by using KBr pellets. The ^{1}H NMR were recorded on a BRUCKER AMX-400 spectrometer operating at 400 MHz. Mass spectra were recorded on AGILENT LC-MSD-TRAP-XCT mass spectrometer. Elemental analyses were done on Vario EL. CHNOS elemental analyzer.

General Microwave Procedure for the Synthesis of Substituted- 1,2,4-Triazolo[3,4-*b*] Thiadiazepine 2a

To a mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol [6](0.768 g, 0.004 mol) and 3-(2—chloroquinolin-3-yl)-1-phenylprop-2-en-1-one **1a** (1.205 g, 0.0041 mol), catalytic amount of *p*-TsOH (50 mg) added then the mixture were grinded thoroughly, and the contents were subjected to microwave irradiation at an interval of 1 min at 160 W for about 10 min; progress of the reaction was monitored by TLC. After the completion of the reaction, the obtained product was poured into water stirred well solid obtained was recrystallized from acetonitrile—gave 83% yield. The same procedure was used for the synthesis of **2b–j.**

General Conventional Method for the Synthesis of Substituted- 1,2,4-Triazolo[3,4-*b*] Thiadiazepine 2a

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To a mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol [6](0.768 g, 0.004 mol) and 3-(2-chloroquinolin-3-yl)-1-phenylprop-2-en-1-one **1a** (1.201 g, 0.0041 mol), catalytic amount of *p*-TsOH (50 mg) was added all these were taken in 100 ml round bottom flask containing 30 ml anhydrous acetonitrile, kept for reflux for about 12 h after the completion of the reaction confirmed by TLC, reaction mixture was concentrated then poured into ice cold water. The obtained greenish-yellow colour solid was filtered washed with water then recrystallised from aqueous DMF, or from acetonitrile. The same procedure was used for the synthesis of **2b-j.**

The physicochemical data for the synthesized compounds are given below.

Physical and Spectral Data of the Products

8-(2-chloroquinolin-3-yl)-6-3-diphenyl-5,8-dihydro[1,2,4]triazolo [3,4-b][1,3,4]thiadiazepine (2a). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol) and 3-(2-chloroquinolin-3-yl)-1-phenylprop-2-en-1-one **1a** (1.201 g, 0.0041 mol) and *p*-TsOH (50 mg) to microwave irradiation as described in the general procedure, **2a** was obtained as an orange solid. The isolated compound **2a** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 250–252°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.15 (s, 1H, —NH), 6.90–7.92 (m, 14H, Ar—H), 3.45 (d, 1H, CH-S), 7.60 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1610 (C = N), 1445(C-N); MS (m/z): 547 [M^+] Anal. Calcd. for $C_{26}H_{17}BrClN_5S$ C, 57.03; H, 3.10; N, 12.79. Found: C, 57.06; H, 3.13; N, 12.81.

8-(6-Bromo-2-chloroquinolin-3-yl)-6-(4-bromophenyl)-3-phenyl-5,8-dihydro[1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2b). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl- 4H-1,2,4-triazole-3-thiol[6] (0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-(4-bromophenyl)prop-2-en-1-one **1b** (1.85 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 11 min As described in the general procedure, **2b** was obtained as an orange solid. The isolated compound **2b** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 263–265°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.16 (s, 1H, —NH), 6.90–7.94 (m, 13H, Ar—H), 3.47 (d, 1H, CH-S), 7.60 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N), 1440(C-N); MS (m/z): 625 [M^+] Anal. Calcd. for $C_{26}H_{16}Br_2ClN_5S$: C, 49.92; H, 2.56; N, 11.20. Found: C, 49.94; H, 2.58; N, 11.24.

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8-(6-bromo-2-chloroquinolin-3-yl)-6-(4-chlorophenyl)3-phenyl-5,8-dihydro[1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2c). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-(4-chlorophenyl)prop-2-en-1-one **1c** (1.669 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 10 min, **2c** was obtained as an orange solid. The isolated compound **2c** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 255–257°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.13 (s, 1H, —NH), 7.05–7.97 (m, 13H, Ar—H), 3.52 (d, 1H, CH-S), 7.65 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N), 1445(C-N); MS (m/z): 258 [M^+] Anal. Calcd. for $C_{26}H_{16}BrCl_2N_5S$: C, 53.70; H, 2.75; N, 12.05. Found: C, 53.72; H, 2.78; N, 12.07

4-[8-(6-Bromo-2-chloroquinolin-3-yl) 3-phenyl-5,8-dihydro[1,2,4] triazolo[3,4-b][1,3,4]thiadiazepin-6-yl]phenol (2d). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-(4-hydroxyphenyl)prop-2-en-1-one **1d** (1.591 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 10 min, **2d** was obtained as an orange solid. The isolated compound **2d** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 233–235°C; ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.16 (s, 1H, —NH), 6.90–7.92 (m, 14H, Ar—H), 3.50 (d, 1H, CH-S), 7.63 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1620 (C = N), 1450(C-N); MS (m/z): 563 [M^+] Anal. Calcd. for $C_{26}H_{17}BrClN_5OS$: C, 55.51; H, 3.02; N, 12.45. Found: C, 55.53; H, 3.06; N, 12.48.

8-(6-bromo-2-chloroquinolin-3-yl)-6-(4-methoxyphenyl)-3-phenyl-5,8-dihydro[1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2e). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6] (0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-(4-methoxyphenyl)prop-2-en-1-one **1e** (1.648 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 11 min, **2e** was obtained as an orange solid. The isolated compound **2e** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 252–254°C.^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.16 (s, 1H, —NH), 6.93–7.98 (m, 13H, Ar—H), 3.52 (d, 1H, CH-S), 7.65 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N), 1450(C-N); MS (m/z): 577 [M$^+$] Anal. Calcd. for $C_{27}H_{19}BrClN_5OS$: C, 56.15; H, 3.29; N, 12.13. Found: C, 56.17; H, 3.31; N, 12.16.

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8-(6-bromo-2-chloroquinolin-3-yl)-6-(4-methylphenyl)-3-phenyl-5, 8-dihydro[1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2f). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-(4-methylphenyl)prop-2-en-1-one **1f** (1.582 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 10 min, **2f** was obtained as an orange solid. The isolated compound **2f** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 231–233°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.13 (s, 1H, —NH), 6.95–8.10 (m, 13H, Ar—H), 3.54 (d, 1H, CH-S), 7.65 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1620 (C = N), 1445(C-N); MS (m/z): 560 [M$^+$] Anal. Calcd. for $C_{27}H_{19}BrClN_5S$: C, 57.85; H, 3.39; N, 12.50. Found: C, 57.83; H, 3.41; N, 12.52

8-(6-bromo-2-chloroquinolin-3-yl)-6-phenyl-5,8-dihydro[1,2,4]-triazolo[3,4-b][1,3,4]thiadiazepine (2g). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 3-(6-bromo-2-chloroquinolin-3-yl)-1-phenylprop-2-en-1-one **1g** (1.525 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 10 min, **2g** was obtained as an orange solid. The isolated compound **2g** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 243–245°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.15 (s, 1H, —NH), 6.90–7.92 (m, 15, Ar—H), 3.45 (d, 1H, CH-S), 7.60 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1620 (C = N), 1440(C-N); MS (m/z): 467 [M$^+$] Anal. Calcd. for $C_{26}H_{18}ClN_5S$: C, 66.80; H, 3.85; N, 14.99. Found: C, 66.82; H, 3.87 N, 15.03.

6-(4-chlorophenyl)-8-(2-chloroquinolin-3-yl)-3-phenyl-5,8-dihydro [1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2h). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 1-(4-chlorophenyl)-3-(2-chloroquinolin-3-yl)prop-2-en-1-one **1h** (1.345 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 11 min, **2h** was obtained as an orange solid. The isolated compound **2h** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 238–240°C.^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.16 (s, 1H, —NH), 6.90–7.92 (m, 14, Ar—H), 3.47 (d, 1H, CH-S), 7.60 (d, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N), 1450(C-N); MS (m/z): 502 [M$^+$] Anal. Calcd. for $C_{26}H_{17}Cl_2N_5S$: C, 62.15; H, 3.38; N, 13.94. Found: C, 62.17; H, 3.39; N, 13.97.

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8-(2-chloro-8-methylquinolin-3-yl)-3-6-diphenyl-5,8-dihydro[1,2,4] triazolo[3,4-b][1,3,4]thiadiazepine (2i). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6](0.768 g, 0.004 mol), 3-(2-chloro-8-methylquinolin-3-yl)-1-phenylprop-2-en-1-one **1i** (1.259 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 10 min, **2i** was obtained as an orange solid. The isolated compound **2i** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 233–235°C; (Conventional). ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.13 (s, 1H, —NH), 6.90–7.92 (m, 14, Ar—H), 3.48 (d, 1H, CH-S), 7.65 (s, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N), 1450(C-N); MS (m/z): 482 [M$^+$] Anal. Calcd. for $C_{27}H_{20}ClN_5S$: C, 67.21; H, 4.15; N, 14.52. Found: C, 67.24; H, 4.17; N, 14.54.

8-(2-chloro-8-methylquinolin-3-yl)-6-(4-methylphenyl)-3-phenyl-5, 8-dihydro[1,2,4]triazolo[3,4-b][1,3,4]thiadiazepine (2j). These compounds were prepared by subjecting the mixture of 4-amino-5-phenyl-4H-1,2,4-triazole-3-thiol[6] (0.768 g, 0.004 mol) and 3-(2-chloro-8-methylquinolin-3-yl)-1-(4-methylphenyl)prop-2-en-1-one **1j** (1.316 g, 0.0041 mol), and *p*-TsOH (50 mg) to microwave irradiation for about 11 min, **2j** was obtained as an orange solid. The isolated compound **2j** was washed and recrystallized from acetonitrile or from aqueous DMF Solid, m.p. 257–259°C. ^{1}H NMR (400 MHz, DMSO-d6) δ (ppm): 8.15 (s, 1H, —NH), 6.90–7.92 (m, 10H, Ar—H), 3.46 (d, 1H, CH-S), 7.63 (s, 1H, —CH=); IR (KBr) ν (cm^{-1}): 1615 (C = N, 1440 (C-N); MS (m/z): 496 [M$^+$] Anal. Calcd. for $C_{28}H_{22}ClN_5S$: C, 67.74; H, 4.35; N, 14.11. Found: C, 67.76; H, 4.33; N, 14.13.

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