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Note

Synthesis and studies of some novel *s*-triazine based aminopyrimidines, isoxazoles and 1,5-benzothiazepines

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An elegant synthesis of the titled compounds **7a-e, 8a-e** and **9a-e** have been presented starting from chalcones **6a-e** based on *s*-triazine nucleus. These chalcones **6a-e** on cyclisation with guanidine nitrate and hydroxylamine hydrochloride in the presence of alkali give aminopyrimidines **7a-e** and isoxazoles **8a-e** respectively. Further these chalcones **6a-e** on cyclisation with 2-aminothiophenol in the presence of few drops of glacial acetic acid give 1,5-benzothiazepines **9a-e**. All the products obtained from these reactions are characterized by elemental analysis, IR, ^{1}H NMR and mass spectral data.

Keywords: Aminopyrimidines, isoxazoles, 1,5-benzothiazepines, glacial acetic acid, chalcones, *s*-triazine nucleus

The quest for a more reliable and suitable drug is always fascinating and challenging. A number of drugs containing simple heterocyclic or a combination of different heterocyclic moieties have been in use these days. The *s*-triazine based chalcones and their derivatives have their own importance in heterocyclic chemistry due to their good biological activities[1]. Chalcones have been studied extensively because of their wide range of biological activity. They are found to be effective as anaesthetic[2], antibacterial[3], antiviral[4], cardiovascular[5] and anticancer[6] agents. The diverse properties of chalcones have prompted us to synthesize them in order to study their biological and pharmacological activities. Aminopyrimidines play a vital role in many biological process since their ring system is present in several vitamins, coenzymes, nucleic acid etc. Synthetic members of these groups are also important as chemotherapeutic agents. The synthesis of pyrimidines have attracted the attention of chemists because of their potential pharmacodynamic properties. Recently much interest has been focused on the synthesis of pyrimidines possessing fungicidal[7], herbicidal[8], anti-HIV[9] and antitumor[10] activity. Five-membered heterocycles like isoxazoles are found to have wide application as pharmaceutical and agrochemical agents. The synthesis of isoxazole derivatives attracted considerable attention from organic and medicinal chemists due to their considerable bioactivity. Various biological applications have been reported for isoxazoles such as antitumor[11], analgesic[12], antimicrobial[13] and chemotherapy[14]. 1,5-Benzothiazepines are gaining more attention due to their pharmacological significance. Compounds like diltiazem[15] and clentiazem are well explored as effective cardiovascular drugs and are found to contain 1,5-benzothiazepine nucleus. Some of the benzothiazepine have been claimed to exhibit antifungal, antibacterial, anticonvulsant, antispasmodic[16], neurolaptic[17] and antidepressant[18] activities.

Results and Discussion

The required precursor 2,4-bis-(phenylamino)-6-[4'-{3"-(phenyl/substituted phenyl)-2"-propenon-1"-yl}-phenylamino]-*s*-triazines **6a-e** were prepared from cyanuric chloride according to our reported procedure[19,20]. Reaction of **6e** with guanidine nitrate in the presence of alkali afforded the product **7e** in 64 % yield. The structure of the **7e** was confirmed from its spectral and analytical studies as discussed. The ^{1}H NMR spectrum of **7e** displayed two singlets at δ 3.75 and at 5.10 due to C_2'''–OCH_3 and C_2"–NH_2 protons respectively. The complex multiplet at δ 6.90-8.15 is assigned for twenty-two aromatic protons. This was supported by the electron impact mass spectrum of the compound which displayed the molecular ion peak at *m/z* 553 and elemental analysis also agreed well with the molecular formula $C_{32}H_{27}N_9O$.

The product **8e** was obtained by the treatment of **6e** with hydroxylamine hydrochloride in the presence of alkali and well characterized using its spectral and analytical data. Its IR spectra revealed the presence of –C=N group by exhibiting a strong absorption at 1583 cm^{-1}. The ^{1}H NMR spectrum of **8e** in $CDCl_3$ showed two singlets at δ 3.78 and at 6.90 due to C_2'''–OCH_3 and C_4"–H of isoxazole ring protons. The complex multiplet at δ 7.00-7.90 is assigned for twenty-one aromatic protons. This was also supported

by the mass spectrum of the compound which displayed the molecular ion peak at *m/z* 527. The CHN analysis of the compound **8e** was in good agreement with the proposed molecular formula $C_{31}H_{25}N_7O_2$.

Further, the reaction of **6e** with 2-aminothiophenol in the presence of a few drops of glacial acetic acid was carried out with an interest that the reaction would proceed as shown in **Scheme I** and at the end, this attempt yielded **9a-e**. The IR spectrum of **9e** showed strong absorption at 1573 cm^{-1} due to –C=N group. The ^1H NMR spectrum exhibited following resonance, double doublet at 3.10 for C_3"-H_a, double doublet at 3.30 for C_3"-H_b, singlet at 3.75 for C_2"'-OCH_3 and double doublet at 5.00 for C_2"-H_x proton. The aromatic cluster appeared at δ 6.90-8.10 with twenty-five aromatic protons. This was also supported by the electron impact mass spectrum of the compound which displayed the molecular ion peak at *m/z* 621. The elemental analysis was also in good agreement with the molecular formula $C_{37}H_{31}N_7OS$.

Experimental Section

All the melting points were determined in an open capillary and are uncorrected. The reactions were monitored on TLC. The IR spectra were recorded in KBr pellets on a Perkin-Elmer 237 spectrophotometer.^1H NMR spectra on a Bruker Avance DPX 300 MHz spectrometer with $CDCl_3$ as a solvent, using TMS as internal reference. Elemental analysis were carried out on a Carlo Erba 1108 model analyzer. Mass spectra were recorded on a Hewlett Packard Mass spectrometer.

Preparation of 2-phenylamino-4,6-dichloro-*s*-triazine[21] **3**. Aniline **2** (0.01 mole) was added slowly to cyanuric chloride **1** (0.01 mole) in acetone (30 mL) with constant stirring for 4 hr at 0 to 5°C. Then sodium carbonate (0.005 mole) dissolved in water (10 mL) was added dropwise to neutralize HCl evolved during the reaction. Finally the contents were poured into crushed ice. The solid separated out was filtered, washed with water, dried and recrystallized from alcohol to give **3**; m.p. 196°C, yield 86%.

Preparation of 2,4-bis-(phenylamino)-6-chloro-*s*-triazine 4. Aniline **2** (0.01 mole) was added slowly to compound **3** (0.01 mole) in acetone (35 mL) with constant stirring for 6 hr at room temperature. Then sodium carbonate (0.005 mole) dissolved in water (10 mL) was added dropwise to neutralize HCl evolved during the reaction. Finally the contents were poured into crushed ice. The solid separated out was filtered, washed with water, dried and recrystallized from alcohol to give **4**; m.p. 179°C, yield 80%; IR (KBr): 772 (C-Cl), 1359 (C-N), 805 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 7.20 to 7.80 (m, 10 Ar-H and 2 NH).

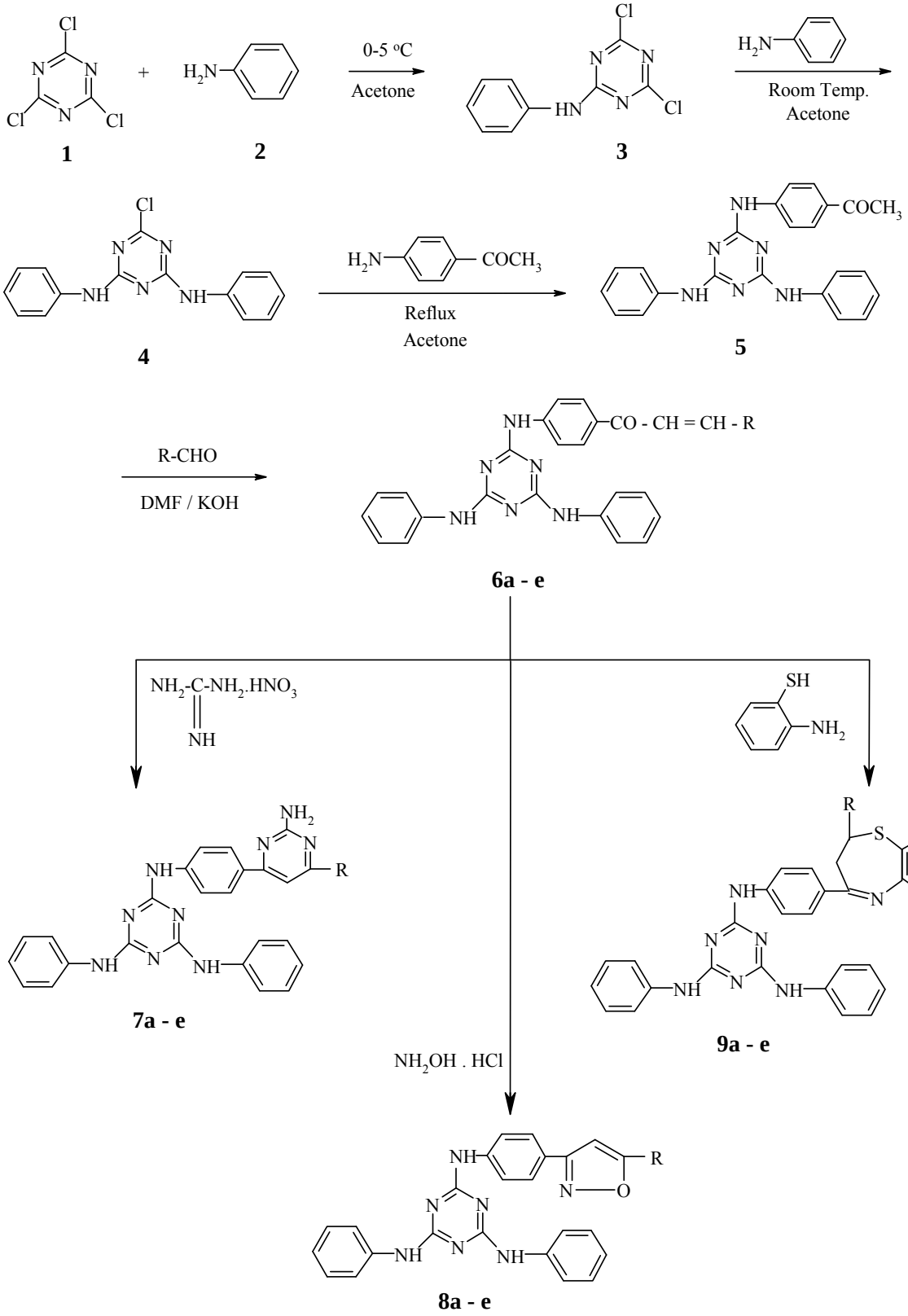
Preparation of 2,4-bis-(phenylamino)-6-(4'-acetylphenylamino)-*s*-triazine 5. 4-Aminoacetophenone (0.01 mole) and compound **4** (0.01 mole) were dissolved in acetone (40 mL). The reaction mixture was refluxed for 6 hr, cooled and poured into crushed ice. Then sodium carbonate (0.005 mole) dissolved in water (10 mL) was added to neutralize HCl evolved during the reaction. The solid separated out was filtered, washed with water, dried and recrystallized from alcohol to give **5**; m.p. 218°C, yield 75%; IR (KBr): 1662 (C=O), 1355 (C-N), 805 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 2.6 (s, 3H, -$COCH_3$), 7.0 to 7.95 (m, 14 Ar-H and 3 NH).

Preparation of 2,4-bis-(phenylamino)-6-[4'-{3''-(2'''-methoxyphenyl)-2''-propenon-1''-yl}-phenyl-amino]-*s*-triazine 6e. Compound **5** (0.01 mole) was dissolved in DMF (30 mL) and 2-methoxy-benzaldehyde (0.01 mole) was added to it. Then solution of KOH (5 mL of 40%) was added to the reaction mixture with constant stirring at room temperature. After 24 hr the reaction mixture was poured into crushed ice and neutralize with HCl. The product separated out was filtered, washed with water, dried and recrystallized from alcohol to give **6e**; m.p. 138°C, yield 68%; IR (KBr): 1647 (C=O), 1595 (-CH=CH-, str.), 1340 (C-N), 812 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.95 (s, 3H, C_2"'-OCH_3), 6.90 (d, lH, -CO-CH=), 7.15 to 7.80 (m, 18 Ar-H and 3 NH), 8.05 (d, 1H, Ar-CH=).

Similarly the remaining compounds **6a-d** were prepared by this method.

General procedure for the preparation of 2, 4-bis-(phenyl amino)-6-[4'-{2''-amino-6''-(phenyl/ substitutedphenyl)-pyrimidine-4''-yl}-phenyl amino]-*s*-triazine 7a-e. Compound **6e** (0.01 mole) was dissolved in alcohol (25 mL) and guanidine nitrate (0.01 mole) was added to it. Then solution of KOH (5 mL of 40%) was added to the reaction mixture and refluxed for 10 hr. The reaction mixture was then cooled, poured into crushed ice and product separated out was filtered, washed with water, dried and recrystallized from alcohol to give **7a-e**.

2,4-Bis-(phenylamino)-6-[4'-{2''-amino-6''-(phenyl)- pyrimidine - 4''-yl} - phenyl amino]-*s*-


0-5 °C
Acetone
Room Temp.
Acetone
Reflux
Acetone
R-CHO
DMF / KOH
NH2-C-NH2.HNO3
NH
NH2OH . HCl

Scheme I

triazine 7a: Yield 68%; m.p. 154°C; IR (KBr): 3396 (-NH_2, pyrimidine moiety), 1577 (C=N, pyrimidine moiety), 807 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 5.15 (s, 2H, C_2"-NH_2), 6.90 to 8.15 (m, 20 Ar-H and 3 NH); MS: *m/z* 523. Anal. Calcd for $C_{31}H_{25}N_9$: C, 71.13; H, 4.78; N, 24.09. Found: C, 71.16; H, 4.74; N, 24.13%.

2,4-Bis-(phenylamino)-6-[4'-{2''-amino-6''-(2'''-chlorophenyl)-pyrimidine-4''-yl} -phenylamino]-*s*-triazine 7b: Yield 67%; m.p. 165°C; IR (KBr): 3394 (-NH_2, pyrimidine moiety), 1576 (C=N, pyrimidine moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 5.12 (s, 2H, C_2"-NH_2), 6.90 to 8.15 (m, 19 Ar-H and 3 NH); MS: *m/z* 557.5. Anal. Calcd for $C_{31}H_{24}N_9Cl$: C, 66.73; H, 4.30; N, 22.60. Found: C, 66.76; H, 4.34; N, 22.63%.

2,4-Bis-(phenylamino)-6-[4'-{2''-amino-6''-(3'''-chlorophenyl)-pyrimidine-4''-yl}-phenylamino]-*s*-triazine 7c: Yield 65%; m.p. 148°C; IR (KBr): 3397 (-NH_2, pyrimidine moiety), 1573 (C=N, pyrimidine moiety), 804 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 5.10 (s, 2H, C_2"-NH_2), 6.90 to 8.15 (m, 19 Ar-H and 3 NH); MS: *m/z* 557.5. Anal. Calcd for $C_{31}H_{24}N_9Cl$: C, 66.73; H, 4.30; N, 22.60. Found: C, 66.71; H, 4.32; N, 22.57%.

2,4-Bis-(phenylamino) - 6 -[4'-{2''-amino-6''-(2'''-nitrophenyl)-pyrimidine-4''-yl} - phenyl-amino]-*s*-triazine 7d: Yield 62%; m.p. 160°C; IR (KBr): 3395 (-NH_2, pyrimidine moiety), 1575 (C=N, pyrimidine moiety), 809 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 5.20 (s, 2H, C_2"-NH_2), 6.90 to 8.15 (m, 19 Ar-H and 3 NH); MS: *m/z* 568. Anal. Calcd for $C_{31}H_{24}N_{10}O_2$: C, 65.49; H, 4.23; N, 24.65. Found: C, 65.46; H, 4.24; N, 24.63%.

2,4-Bis-(phenylamino)-6-[4'-{2''-amino-6''-(2'''-methoxyphenyl)-pyrimidine-4'' -yl}-phenylamino]-*s*-triazine 7e: Yield 64%; m.p. 140°C; IR (KBr): 3398 (-NH_2, pyrimidine moiety), 1575 (C=N, pyrimidine moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.75 (s, 3H, C_2'''-OCH_3), 5.10 (s, 2H, C_2"-NH_2), 6.90 to 8.15 (m, 19 Ar-H and 3 NH); MS: *m/z* 553. Anal. Calcd for $C_{32}H_{27}N_9O$: C, 69.44; H, 4.88; N, 22.78. Found: C, 69.46; H, 4.87; N, 22.75%.

General procedure for the preparation of of 2, 4-bis-(phenylamino)-6-[4'-{5''-(phenyl/substitute-dphenyl)-isoxazole-3''-yl}-phenylamino]-*s*-triazine 8a-e. Compound **6e** (0.01 mole) was dissolved in alcohol (25 mL) and hydroxylamine hydrochloride (0.01 mole) was added to it. Then solution of KOH (5 mL of 40%) was added to the reaction mixture and refluxed for 6 hr. The reaction mixture was then cooled, poured into crushed ice and product separated out was filtered, washed with water, dried and recrystallized from alcohol to give **8a-e**.

2,4-Bis- (phenylamino) -6- [4' - { 5''- (phenyl) - isoxazole - 3'' -yl}-phenylamino]-*s*-triazine 8a: Yield 61%; m.p. 151°C; IR (KBr): 1580 (C=N, isoxazole moiety), 807 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 6.92 (s, 1H, C_4"-CH of isoxazole moiety), 7.00 to 8.15 (m, 19 Ar-H and 3 NH); MS: *m/z* 497. Anal. Calcd for $C_{30}H_{23}N_7O$: C, 72.43; H, 4.63; N, 19.72. Found: C, 72.46; H, 4.64; N, 19.69%.

2,4-Bis-(phenylamino)-6-[4'-{ 5''-(2'''-chloro-phenyl) – isoxazole - 3'' - yl }-phenylamino]-*s*-triazine 8b: Yield 57%; m.p. 145°C; IR (KBr): 1579 (C=N, isoxazole moiety), 805 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 6.90 (s, 1H, C_4"-CH of isoxazole moiety), 7.00 to 7.90 (m, 18 Ar-H and 3 NH); MS: *m/z* 531.5. Anal. Calcd for $C_{30}H_{22}N_7OCl$: C, 67.73; H, 4.14; N, 18.44. Found: C, 67.76; H, 4.13; N, 18.41%.

2,4-Bis-(phenylamino)-6-[4'-{5''-(3'''-chloro-phenyl)-isoxazole-3''-yl}-phenylamino]-*s*-triazine 8c: Yield 59%; m.p. 139°C; IR (KBr): 1581 (C=N, isoxazole moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 6.89 (s, 1H, C_4"-CH of isoxazole moiety), 7.00 to 7.90 (m, 18 Ar-H and 3 NH); MS: *m/z* 531.5. Anal. Calcd for $C_{30}H_{22}N_7OCl$: C, 67.73; H, 4.14; N, 18.44. Found: C, 67.71; H, 4.12; N, 18.45%.

2,4-Bis-(phenylamino)-6-[4'-{5''-(2'''-nitro-phenyl)–isoxazole-3''-yl}-phenylamino]-*s*-triazine 8d: Yield 61%; m.p. 181°C; IR (KBr): 1584 (C=N, isoxazole moiety), 808 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 6.91 (s, 1H, C_4"-CH of isoxazole moiety), 7.00 to 7.90 (m, 18 Ar-H and 3 NH); MS: *m/z* 542. Anal. Calcd for $C_{30}H_{22}N_8O_3$: C, 66.42; H, 4.06; N, 20.66. Found: C, 66.44; H, 4.04; N, 20.63%.

2,4-Bis-(phenylamino)-6-[4'-{5''-(2'''-methoxy phenyl)-isoxazole-3''-yl}-phenylamino]-*s*-triazine 8e: Yield 62%; m.p. 160°C; IR (KBr): 1583 (C=N, isoxazole moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.78 (s, 3H, C_2'''-OCH_3), 6.90 (s, 1H, C_4"-CH of isoxazole moiety), 7.00 to 7.90 (m, 18 Ar-H and 3 NH); MS: *m/z* 527. Anal. Calcd for $C_{31}H_{25}N_7O_2$: C, 70.59; H, 4.74; N, 18.60. Found: C, 70.61; H, 4.73; N, 18.63%.

General procedure for the preparation of 2,4-bis-(phenylamino)-6-[4'-{2''-(phenyl/ substituted phenyl)-2'',3''-dihydro-1'',5''-benzothiazepine-4''-yl}-phenyl amino-s -triazine 9a-e. Compound

6e (0.01 mole) was dissolved in alcohol (25 mL) and 2-aminothiophenol (0.01 mole) was added to it. Then few drops of glacial acetic acid was added to the reaction mixture and refluxed for 10 hr. The reaction-mixture was then cooled, poured into crushed ice and product separated out was filtered, washed with water, dried and recrystallized from alcohol to give **9a-e**.

2,4-Bis-(phenylamino)-6-[4'-{2''-phenyl-2'',3''-dihydro-1'',5''-benzothiazepine-4''-yl}-phenyl amino]-s-triazine 9a: Yield 59%; m.p. 90°C; IR (KBr): 1570 (C=N, benzothiazepine moiety), 734 (C-S-C, benzothiazepine moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.11 (dd, 2H, C_3"-H_a of benzothiazepine moiety), 3.34 (dd, 2H, C_3"-H_b of benzothiazepine moiety), 5.10 (dd, 1H, C_2"-H_x of benzothiazepine moiety), 6.90 to 8.10 (m, 23 Ar-H and 3 NH); MS: *m/z* 591. Anal. Calcd for $C_{36}H_{29}N_7S$: C, 73.10; H, 4.91; N, 16.58. Found: C, 73.13; H, 4.94; N, 16.55%.

2,4-Bis-(phenylamino)-6-[4'-{2''-(2'''-chloro-phenyl)-2'',3''-dihydro-1'',5''-benzothiazepine-4''-yl}-phenyl amino]-s-triazine 9b: Yield 56%; m.p. 101°C; IR (KBr): 1569 (C=N, benzothiazepine moiety), 732 (C-S-C, benzothiazepine moiety), 807 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.00 (dd, 2H, C_3"-H_a of benzothiazepine moiety), 3.25 (dd, 2H, C_3"-H_b of benzothiazepine moiety), 5.10 (dd, 1H, C_2"-H_x of benzothiazepine moiety), 6.90 to 8.10 (m, 22 Ar-H and 3 NH); MS: *m/z* 625.5. Anal. Calcd for $C_{36}H_{28}N_7SCl$: C, 69.06; H, 4.48; N, 15.67. Found: C, 69.07; H, 4.46; N, 15.66%.

2,4-Bis-(phenylamino)-6-[4'-{2''-(3'''-chloro-phenyl)-2'',3''-dihydro-1'',5''-benzo thiazepine-4''-yl}-phenyl amino]-s-triazine 9c: Yield 60%; m.p. 105°C; IR (KBr): 1583 (C=N, benzothiazepine moiety), 730 (C-S-C, benzothiazepine moiety), 804 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.15 (dd, 2H, C_3"-H_a of benzothiazepine moiety), 3.36 (dd, 2H, C_3"-H_b of benzothiazepine moiety), 5.15 (dd, 1H, C_2"-H_x of benzothiazepine moiety), 6.90 to 8.10 (m, 22 Ar-H and 3 NH); MS: *m/z* 625.5. Anal. Calcd for $C_{36}H_{28}N_7SCl$: C, 69.06; H, 4.48; N, 15.67. Found: C, 69.09; H, 4.45; N, 15.69%.

2,4-Bis-(phenylamino)-6-[4'-{2''-(2'''-nitro-phenyl)-2'',3''-dihydro-1'',5''-benzothiazepine-4''-yl}-phenyl amino]-s-triazine 9d: Yield 58%; m.p. 160°C; IR (KBr): 1571 (C=N, benzothiazepine moiety), 729 (C-S-C, benzothiazepine moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.12 (dd, 2H, C_3"-H_a of benzothiazepine moiety), 3.31 (dd, 2H, C_3"-H_b of benzothiazepine moiety), 5.05 (dd, 1H, C_2"-H_x of benzothiazepine moiety), 6.90 to 8.10 (m, 22 Ar-H and 3 NH); MS: *m/z* 636. Anal. Calcd for $C_{36}H_{28}N_8SO_2$: C, 67.92; H, 4.40; N, 17.61. Found: C, 67.94; H, 4.42; N, 17.63%.

2,4-Bis-(phenylamino)-6-[4'-{2''-(2'''-methoxy-phenyl)-2'',3''-dihydro-1'',5''-benzothiazepine-4''-yl}-phenyl amino]-s-triazine 9e: Yield 63%; m.p. 104°C; IR (KBr): 1573 (C=N, benzothiazepine moiety), 731 (C-S-C, benzothiazepine moiety), 806 cm^{-1} (C-N, *s*-triazine); ^1H NMR ($CDCl_3$): δ 3.05 (dd, 2H, C_3"-H_a of benzothiazepine moiety), 3.33 (dd, 2H, C_3"-H_b of benzothiazepine moiety), 3.75 (s, 3H, C_2'''-OCH_3), 5.00 (dd, 1H, C_2"-H_x of benzothiazepine moiety), 6.90 to 8.10 (m, 22 Ar-H and 3 NH); MS: *m/z* 621. Anal. Calcd for $C_{37}H_{31}N_7OS$: C, 71.49; H, 4.99; N, 15.78. Found: C, 71.47; H, 4.98; N, 15.76%.

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