A Novel Synthesis of Sulfone Systems as Antimicrobial Agents

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Z. Naturforsch. **55b,** 127–132 (2000); received August 3, 1999

Sulfone Systems, Antimicrobial Agents

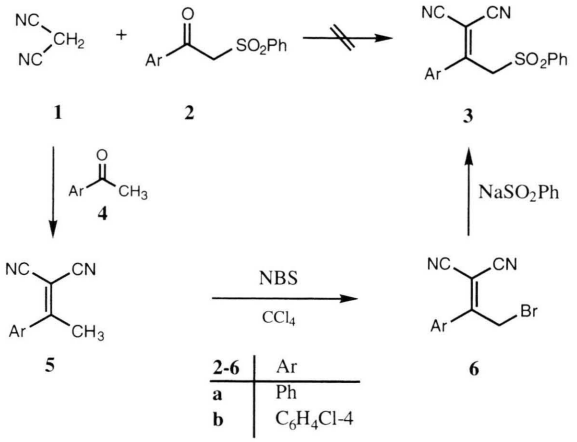
The applicability and synthetic potency of novel reagent, 2-aryl-1,1-dicyano-3-(phenylsulfonyl)propenes **3** to develop an expeditious convenient synthetic route of unique polyfunctionally substituted carbocyclic and heterocyclic sulfone systems is reported. Chemical and spectroscopic evidence for the structures of the newly synthesized compounds are described. Some of the obtained compounds were tested for their antimicrobial activity.

Introduction

In view of the diverse biological and physiological activities of sulfones [1–7], and in connection with our previous efforts directed towards the facile synthesis of heterocyclic ring systems [8–12], we designed a specific simple program aimed at the development of convenient synthetic approach for the construction of several new hitherto unreported carbocyclic and heterocyclic sulfone systems of expected potential bioresponses utilizing the novel reagent **3** as a unique key precursor. The newly synthesized derivatives possess latent functional substituents and appear to be promising for further chemical transformations as well as biological activity evaluations.

Investigations, Results and Discussion

Our attempts to prepare the key precursor, 2-aryl-1,1-dicyano-3- phenylsulfonylpropenes **3,** *via* condensation of equimolar amounts of malononitrile **1** with the appropriate phenylsulfonylacetophenones **2** utilizing a variety of acidic or alkaline conditions were unsuccessful. In our hands, the precursor **3** could be prepared stepwise. Thus, condensation of **1** with the appropriate acetophenones **4** afforded the corresponding condensation products **5** [5,13], which could be brominated with *N*-bromosuccinimide in carbon tetrachloride solution in the presence of a catalytic amount of benzoyl peroxide to afford the corresponding α-bromo derivatives **6**. The latter reacted with equimolar amounts of sodium benzenesulfinate in ethanol solution to furnish the target novel key reagent **3** in acceptable yields (Scheme 1). The elemental and spectroscopic data of **3** are entirely compatible with the assigned structure.



Scheme 1

Compound **3** proved to be highly reactive towards various reagents and underwent numerous chemical transformations, resulting in the construction of a wide range of carbocyclic and heterocyclic aromatic sulfone systems. Thus, when equimolar amounts of each of **3a,b** and elemental sulfur in dioxane were heated under reflux, 2-amino-4-aryl-5-phenylsulfonylthiophene-3-carbonitriles **7** could be isolated in good yields (Scheme 2).

The methylene group in **3** proved to be highly reactive towards electrophilic reagents. Thus, compound **3a** reacted with an equimolar amount of the

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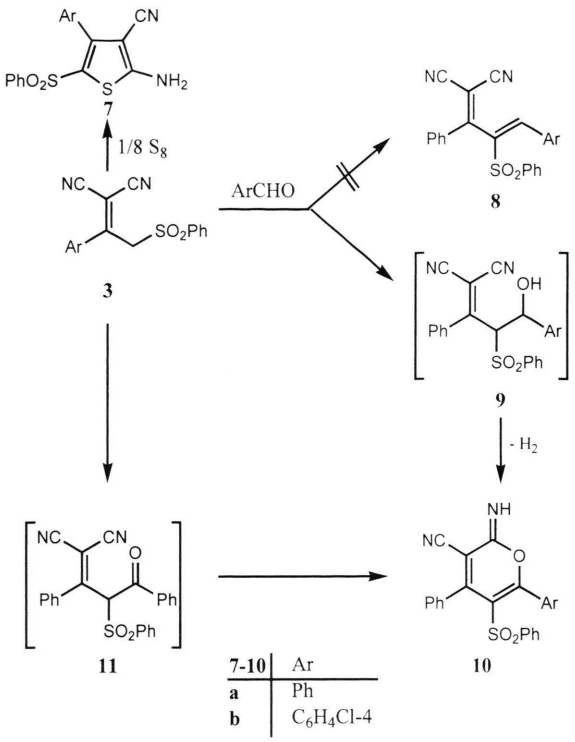
appropriate aromatic aldehyde in dioxane containing potassium *t*-butoxide to afford the corresponding pyran derivatives **10** instead of the expected arylidene derivatives **8**. The reaction apparently involves formation of the hydroxy intermediate **9**, intramolecular heterocyclization *via* an anticipated Michael-type addition of the OH group to the CN function and spontaneous autoxidation *via* loss of hydrogen molecule under the experimental reaction conditions to give the final isolable products **10**. Similar autoxidations have been reported previously [5,14–16]. Both elemental analyses and spectral data are in agreement with the proposed structure **10**. Thus the IR spectrum of **10a** as a representative example, displayed an NH absorption peak near 3436 cm^{-1} and only one CN absorption band at 2220 cm^{-1}. Its ^1H NMR spectrum (DMSO-d_6) showed beside the aromatic protons signal at δ = 6.92–7.80 ppm, a singlet at δ = 12.4 ppm corresponding to a NH function.

Alternatively, compound **10a** could be prepared *via* an independent route involving the reaction of **3a** with equimolar amount of benzoyl chloride in pyridine, under reflux, to afford a single product which was found to be identical in all aspects (m.p., mixed m.p. and IR data) with **10a**, apparently *via* intermediacy of the benzoyl derivative **11**.

The reaction of each of **3a,b** with equimolar amounts of phenacyl bromide in dry dioxane in the presence of potassium *t*-butoxide, under reflux, afforded the corresponding 5-(phenylsulfonyl)cyclopentadiene-2-carbonitrile derivatives **12a,b**, respectively (Scheme 3). Assignment of structure **12** for the reaction products was based on analytical and spectral data. Thus, as an example, the IR spectrum of **12a** showed absorption bands at ν 3400, 3300 (NH_2), 2220 (CN) and 1695 (CO) cm^{-1}. Its ^1H NMR spectrum (DMSO-d_6) showed a singlet at δ = 3.92 for NH_2 protons, singlet at δ = 4.42 for methine proton in addition to a multiplet at δ = 6.71–7.80 for the aromatic protons.

Compound **3a** reacted with two-fold molar equivalent of phenacyl bromide under the same experimental conditions to yield the 5-phenacyl derivatives of **12a** cyclopentadiene **13**.

Compounds **12a,b** reacted with equimolar amounts of phenyldiazonium chloride in ethanolic sodium acetate solution (pH≃8) at 0–5 °C to yield



Scheme 2

the corresponding coupling products 5-(phenylazo)cyclopentadiene derivatives **14a,b**.

As an extension of the synthetic route the behavior of **12** in cycloaddition reactions was investigated, and it was found to be a highly reactive candidate in [4+2] cycloaddition reactions with electronpoor π-deficient systems. Thus compounds **12a,b** add equimolar amounts of dimethyl acetylenedicarboxylate in dry acetonitrile to furnish the corresponding norbornadiene **15**. (Scheme 3). Both elemental analysis and spectroscopic data of **15** are consistent with the assigned structure. Thus, the mass spectrum of **15a** revealed a molecular ion peak at *m/z* 568 corresponding to the molecular formula $C_{31}H_{24}N_2O_7S$. Its IR spectrum showed absorption peaks at ν 3400, 3290, (NH_2), 2219 (CN) and 1680, 1675, 1672 (3 CO) cm^{-1}. Similarly, compounds **12a,b** add to equimolar amounts of tetracyanoethylene in dry acetonitrile to give the corresponding [4+2] cycloadducts **16a,b** respectively.

In summary, the results presented in this article demonstrate a general simple applicable method-

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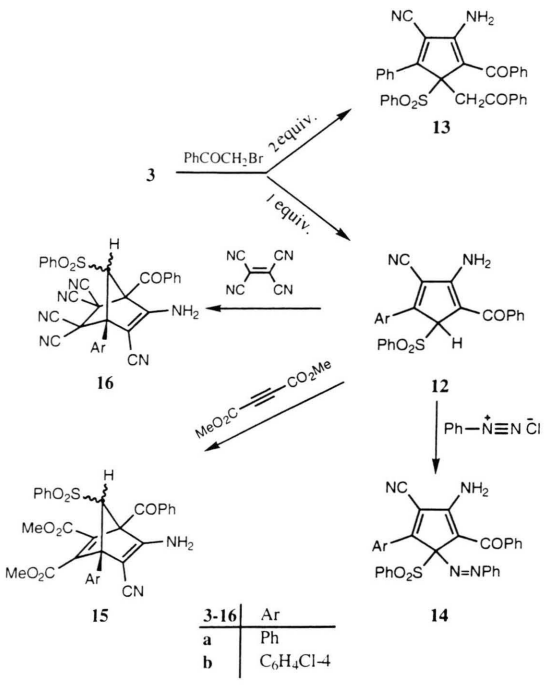

Scheme 3

ology for the construction of a wide variety of polyfunctionally substituted carbocyclic and heterocyclic sulfone systems obtainable only with difficulty otherwise. The importance of such derivatives is due to their expected wide spectrum of potential bioresponses. Work along the expansion of such synthetic approach is now in progress.

Antimicrobial Activity

Table I shows the antimicrobial activity of some of the above mentioned compounds against a variety of bacteria and yeast. Also, in the same table there is a comparison between the activity of these compounds and some known antimicrobial agents. The microbial activity of the compounds considered were tested on the Gram negative bacteria *Salmonella typhimurium* CAIM 1350, *Pseudomonas spp.* CAIM 13, *Shigalla, Brodiatella*; the Gram positive bacteria *Bacillus subtilis* CAIM 1007, *Staphylococcus aureus* CAIM 1352, *Micrococcus lutea*, and the yeast *Candida albicans* CAIM 22, and *Saccharomyces cerevisiae* CAIM 14.

All strains were kindly supplied from Microbiological Research Center, Cairo-Mircen, Egypt (CAIM). Bacterial test organisms were inoculated on nutrient agar slants for 24 h at 37 °C. Yeast organisms were inoculated on molt extract agar slants and incubated at 28 °C for 48 h.

Experimental

Melting points are uncorrected. IR spectra were recorded (KBr) on a Pye Unicam SP-1000 spectrophotometer. ^{1}H NMR spectra were obtained on Varian Gemini 200 MHz spectrometer using TMS as internal reference. Chemical shifts are expressed as δ (ppm). Mass spectra were recorded on a GCMS-QP 1000 EX mass spectrometer operating at 70 eV. The elemental analyses were performed by the Microanalytical Unit, Cairo University.

Table I. Antimicrobial activity of compounds considered against bacteria and yeast (diameter of inhibition zones in mm).

Compound	*Salm.*	*Pseudo.*	*Staph.*	*Bacil.*	*Cand.*	*Sac.*
3b	16	–	12	7	–	9
7	29	30	20	18	–	–
10a	12	10	7	8	–	–
10b	15	11	8	12	–	–
12b	20	20	18	18	–	–
15a	16	19	12	8	–	–
16a	11	13	15	11	2	17
Tetracycline	25	30	25	27	–	–
Gentamycine	25	40	24	26	–	–
Chloramphenicol	–	–	–	10	–	–
Ampicillin sodium	23	12	12	15	–	–
Ampicillin anhydrous	20	10	15	15	–	–
Amoxacillin trihydrate	25	15	10	15	–	–

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2-Aryl-1,1-dicyano-3-(phenylsulfonyl)propenes (**3a,b**)

General procedure: To a solution of **5a,b** (0.1 mol) [5] in carbon tetrachloride (150 ml), N-bromosuccinimide (0.15 mol) and benzoyl peroxide (0.5 g) were added. The reaction mixture was boiled under reflux for 2 h. The solvent was then evaporated "in vacuo". The residue obtained (highly lachrymatory) was dissolved in ethanol (150 ml) and sodium benzenesulfinate (0.1 mol) was added. The reaction mixture was boiled under reflux for 2 h, left aside to cool at room temperature and then poured onto cold water. The solid product, which formed, was collected by filtration, washed three times with water, dried and crystallized from the appropriate solvent.

1,1-Dicyano-2-phenyl-3-(phenylsulfonyl)propene (**3a**)

Yield: 80%; m.p. 125 °C (ethanol). IR: ν = 3010 cm^{-1} (CH_2), 2220, 2218 cm^{-1} (2 CN), 1630 cm^{-1} (C=C). – ^1H NMR ($CDCl_3$): δ = 4.25 (s, 2H, CH_2), 6.81–7.20 (m, 5H, aromatic-H), 7.42–7.80 (m, 5H, SO_2Ph). – MS (70 eV): *m/z* (%): 308 (18) [M^+].

$C_{17}H_{12}N_2O_2S$ (308.36)
Calcd C 66.22 H 3.92 N 9.09 S 10.39%,
Found C 66.4 H 3.0 N 9.2 S 10.2%.

2-(p-Chlorophenyl)-1,1-dicyano-3-(phenylsulfonyl)propene (**3b**)

Yield: 80%; m.p. 131 °C (ethanol). – IR: ν = 3006 cm^{-1} (CH_2), 2222, 2219 cm^{-1} (2 CN), 1630 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 4.13 (s, 2H, CH_2), 6.63–7.10 (m, 4H, aromatic-H), 7.51–7.90 (m, 5H, SO_2Ph).

$C_{17}H_{11}ClN_2O_2S$ (342.80)
Calcd C 59.56 H 3.23 Cl 10.34 N 8.17 S 9.35%,
Found C 59.4 H 3.2 Cl 10.3 N 8.0 S 9.3%.

2-Amino-4-aryl-5-(phenylsulfonyl)thiophene-3-carbonitriles (**7a,b**)

General procedure: To a solution of **3a,b** (0.005 mol) in dioxane (20 ml), elemental sulfur (0.005 mol) was added. The reaction mixture was boiled under reflux for 30 min, left to cool at room temperature and then poured into cold water whereby the solid product that separated was collected by filtration and crystallized from the appropriate solvent.

2-Amino-4-phenyl-5-(phenylsulfonyl)thiophene-3-carbonitrile (**7a**)

Yield: 55%; m.p. 295 °C (dioxane). – IR: ν = 3400, 3320 cm^{-1} (NH_2), 2222 cm^{-1} (CN), 1640 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 6.25 (s,2H, NH_2), 6.71–6.95 (m, 5H, aromatic-H), 7.40–7.80 (m, 5H, SO_2Ph). – MS (70 eV): *m/z* (%): 340 (22) [M^+].

$C_{17}H_{12}N_2O_2S_2$ (340.42)
Calcd C 59.98 H 3.55 N 8.23 S 18.84%,
Found C 60.2 H 3.5 N 8.4 S 18.7%.

2-Amino-4-(p-chlorophenyl)-5-(phenylsulfonyl)-thiophene-3-carbo-nitrile (**7b**)

Yield: 61%; m.p. 221 °C (dioxane). – IR: ν = 3410, 3318 cm^{-1} (NH_2), 2220 cm^{-1} (CN), 1636 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 6.55 (s, 2H, NH_2), 6.95–7.23 (m, 4H, aromatic-H), 7.53–7.96 (m, 5H, SO_2Ph).

$C_{17}H_{11}ClN_2O_2S_2$ (374.87)
Calcd C 54.46 H 2.95 Cl 9.45 N 7.47 S 17.11%,
Found C 54.3 H 2.9 Cl 9.3 N 7.4 S 17.0%.

6-Aryl-2-imino-4-phenyl-5-(phenylsulfonyl)-pyran-3-carbonitriles (**10a,b**)

Method a: To a solution of **3a** (0.005 mol) and potassium *t*-butoxide (0.005 mol) in dry dioxane (15 ml), the appropriate aromatic aldehyde (0.005 mol) was added. The reaction mixture was boiled under reflux for 2 h, poured onto ice/water and neu-tralized with dilute hydrochloric acid (pH ≃ 7). The solid product so formed was collected by filtration, dried and crystallized from the appropriate solvent.

4,6-Diphenyl-2-imino-5-(phenylsulfonyl)pyran-3-carbonitrile (**10a**)

Yield: 45%; m.p. 185 °C (dioxane). – IR: ν = 3436 cm^{-1} (NH), 2220 cm^{-1} (CN), 1640 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 6.92–7.80 (m, 15 H, aromatic protons), 12.41 (s, 1H, NH).

$C_{24}H_{16}N_2O_3S$ (412.46)
Calcd C 69.88 H 3.91 N 6.79 S 7.77%,
Found C 70.1 H 3.7 N 6.7 S 7.8%.

6-(p-Chlorophenyl)-2-imino-4-phenyl-5-(phenylsulfonyl)pyran-3-carbonitrile (**10b**)

Yield: 52%; m.p. 214 °C (dioxane). – IR: ν = 3445 cm^{-1} (NH), 2222 cm^{-1} (CN), 1636 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 6.78–7.91 (m, 14H, aromatic-H),12.71 (s, lH, NH).

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$C_{24}H_{15}ClN_2O_3S$ (446.91)
Calcd C 64.50 H 3.38 Cl 7.93 N 6.26 S 7.17%,
Found C 64.3 H 3.3 Cl 7.8 N 6.2 S 7.0%.

Method b: To a solution of **3a** (0.005 mol) in dry dioxane (15 ml), benzoyl chloride (0.005 mol) was added. The reaction mixture was boiled under reflux for 2 h, stirred overnight at room temperature and then poured onto cold water. The solid product which formed was filtered off, crystallized from dioxane (yield 75%) and it was found to be identical in all aspects (m.p., mixed m.p. and IR data) with an authentic sample of **10a** prepared according to method (a).

3-Amino-1-aryl-4-benzoyl-5-(phenylsulfonyl)-cyclopentadiene-2-carbonitriles (**12a,b**)

General procedure: To a solution of **3a,b** (0.01 mol) and potassium *t*-butoxide (0.01 mol) in dry dioxane (25 ml), phenacyl bromide (0.01 mol) was added. The reaction mixture was refluxed for 5 h. The solid product, which formed on dilution with water and neutralization with dilute HCl was collected by filtration, dried and crystallized from the appropriate solvent.

3-Amino-4-benzoyl-1-phenyl-5-(phenylsulfonyl)-cyclopentadiene-2-carbonitrile (**12a**)

Yield: 60%; m.p. 130 °C (ethanol). – IR: ν = 3400, 3330 cm^{-1} (NH_2), 2220 (CN), 1695 (CO). – ^1H NMR (DMSO-d_6): δ = 3.92 (s, 2H, NH_2), 4.42 (s, 1H, CH), 6.71–7.80 (m, l5H, aromatic-H).

$C_{25}H_{18}N_2O_3S$ (426.87)
Clacd C 70.34 H 4.25 N 6.56 S 7.60%,
Found C 70.6 H 4.2 N 6.6 S 7.2%.

3-Amino-4-benzoyl-1-(p-chlorophenyl)-5-(phenylsulfonyl)cyclopentadiene-2-carbonitrile (**12b**)

Yield: 54%; m.p. 142 °C (dioxane). – IR: ν = 3415, 3326 cm^{-1} (NH_2), 2222 cm^{-1} (CN), 1690 cm^{-1} (CO). – ^1H NMR (DMSO-d_6): δ = 4.12 (s, 2H, NH_2), 4.75 (s, lH, CH), 6.91–7.92 (m, 14H, aromatic-H).

$C_{25}H_{17}ClN_2O_3S$ (461.32)
Calcd C 65.08 H 3.71 Cl 7.68 N 6.07 S 7.03%,
Found C 65.0 H 3.6 Cl 7.4 N 5.9 S 6.8%.

3-Amino-4-benzoyl-5-phenacyl-1-phenyl-5-(phenylsulfonyl)cyclopentadiene-2-carbonitrile (**13**)

The same experimental procedure described above for the synthesis of **12** has been followed except for using phenacyl bromide (0.02 mol). The solid product was collected by filtration, washed several times with boiling ethanol. Yield: 46%; m.p. 260 °C (DMF). – IR: ν = 3400, 3340 cm^{-1} (NH_2), 2220 cm^{-1} (CN), 1695 cm^{-1} (CO). – ^1H NMR: insoluble in common NMR solvents. – MS (70 eV): *m/z* (%) 544 (26) [M^+].

$C_{33}H_{24}N_2O_4S$ (544.63)
Calcd C 72.77 H 4.44 N 5.14 S 5.88%,
Found C 72.9 H 4.2 N 5.3 S 5.9%.

3-Amino-1-aryl-4-benzoyl-5-phenylazo-5-(phenylsulfonyl)cyclopentadiene-2-carbonitriles (**14a,b**)

General procedure: To a stirred solution of **12a,b** (0.005 mol) in ethanol (50 ml) containing sodium acetate (4 g), benzene diazonium chloride (0.005 mol)[prepared by adding sodium nitrite (0.005 mol) to aniline (0.005 mol) in concentrated hydrochloric acid (2 ml) at 0–5 °C while stirring] was added dropwise while cooling at 0–5 °C and stirring. The reaction mixture was then left at room temperature for 1h and the solid product which formed in each case was collected by filtration and crystallized from the appropriate solvent.

3-Amino-4-benzoyl-1-phenyl-5-phenylazo-5-(phenylsulfonyl)cyclo-pentadiene-2-carbonitrile (**14a**)

Yield: 54%; m.p. 218 °C (ethanol). – IR: ν = 3400, 3330 cm^{-1} (NH_2), 2220 cm^{-1} (CN), 1695 cm^{-1} (CO). – ^1H NMR ($CDCl_3$): δ = 3.91 (s, 2H, NH_2), 6.75–7.80 (m, 20H, aromatic-H).

$C_{31}H_{22}N_4O_3S$ (530.60)
Calcd C 70.17 H 4.18 N 10.55 S 6.04%,
Found C 70.3 H 3.9 N 10.6 S 5.8%.

3-Amino-4-benzoyl-1-(p-chlorophenyl)-5-phenylazo-5-(phenylsulfonyl)cyclopentadiene-2-carbonitrile (**14b**)

Yield: 58%; m.p. 226 °C (ethanol). – IR: ν = 3410, 3337 cm^{-1} (NH_2), 2218 cm^{-1} (CN), 1690 (CO). – ^1H NMR (DMSO-d_6): δ = 3.51 (s, 2H, NH_2), 6.93–7.95 (m, l9H, aromatic-H).

$C_{31}H_{21}ClN_4O_3S$ (565.05)
Calcd C 65.89 H 3.74 Cl 6.27 N 9.91 S 5.67%,
Found C 65.6 H 3.7 Cl 6.2 N 9.8 S 5.6%.

Cycloaddition Reactions of 12a,b with Electron Poor Olefins

Synthesis of **15a,b** *and* **16a,b**

General procedure: To a solution of **12a,b** (0.005 mol) in dry acetonitrile (l0 ml), either dimethyl

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acetylenedicarboxylate or tetracyanoethylene (0.005 mol) was added. The reaction mixture was boiled under reflux for 15 min, then left aside at room temperature overnight. The solid product obtained in each case was filtered off and crystallized from the appropriate solvent.

Compound **15a**: Yield: 71%; m.p. 156 °C (acetonitrile). – IR: ν = 3400, 3290 cm^{-1} (NH_2), 2219 cm^{-1} (CN), 1680–1672 cm^{-1} (CO), 1620 cm^{-1} (C=C). – ^1H NMR ($CDCl_3$): δ = 3.70 (s, 3H, CH_3), 3.78 (s, 3H, CH_3), 4.31 (s, 1H, CH), 6.95–7.80 (m, 15H, aromatic-H), 8.10 (br s, 2H, NH_2). – MS (70 eV): *m/z* (%) 568 (12) [M^+].

$C_{31}H_{24}N_2O_7S$ (568.60)
Calcd C 65.48 H 4.25 N 4.93 S 5.64%,
Found C 65.2 H 4.0 N 5.1 S 5.6%.

Compound **15b**: Yield: 68%; m.p. 164 °C (ethanol). – IR: ν = 3406, 3295 cm^{-1} (NH_2), 2220 cm^{-1} (CN), 1670–1660 cm^{-1} (CO), 1625 cm^{-1} (C=C). – ^1H NMR (DMSO-d_6): δ = 3.62 (s, 3H, CH_3), 3.81 (s, 3H, CH_3), 4.20 (s, 1H, CH), 6.90–7.73 (m, 14H, aromatic-H), 8.53 (br s, 2H, NH_2).

$C_{31}H_{23}ClN_2O_7S$ (603.05)
Calcd C 61.74 H 3.84 Cl 5.88 N 4.64 S 5.32%,
Found C 61.5 H 3.8 Cl 5.7 N 4.6 S 5.1%.

Compound **16a**: Yield: 66%; m.p. 215 °C (acetonitrile). – IR: ν = 3400, 3260 (NH_2), 2225–2218 cm^{-1} (CN), 1670 cm^{-1} (CO). – ^1H NMR (DMSO-d_6): δ = 4.39 (s, 1H, CH), 6.90–7.80 (m, 15H, aromatic-H), 8.91 (br s, 2H, NH_2).

$C_{31}H_{18}N_6O_3S$ (554.58)
Calcd C 67.14 H 3.27 N 15.15 S 5.78%,
Found C 66.9 H 3.3 N 15.2 S 5.9%.

Compound **16b**: Yield: 69%; m.p. 221 °C (DMF). – IR: ν = 3410, 3262 (NH_2), 2226–2215 (CN), 1675(CO). – ^1H NMR (DMSO-d_6): δ = 4.28 (s, 1H, CH), 6.83–7.65 (m, 14H, aromatic-H), 8.57 (br s, 2H, NH_2).

$C_{31}H_{17}ClN_6O_3S$ (587.03)
Calcd C 63.21 H 2.91 Cl 6.02 N 14.26 S 5.44%,
Found C 63.0 H 2.9 Cl 5.8 N 14.10 S 5.2%.

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