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Michael Adducts-Synthons for Pyrazolidinediones, Isoxazolidinediones, Pyrimidinetriones, and Thioxopyrimidinediones

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ABSTRACT: *4-Substituted pyrazolidinediones, isoxazolidinediones, and 5-substituted pyrimidinetriones, thioxopyrimidinediones were obtained by exploiting gem-diester functionality in mono- and di-substituted dimethyl malonates.* © 2004 Wiley Periodicals, Inc. Heteroatom Chem 15:477–481, 2004; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20053

INTRODUCTION

The heterocyclic compounds particularly nitrogen-containing heterocycles are synthetically challenging ones as models for a number of physiologically active natural products. In fact pyrimidinetriones, pyrazolidinediones, and isoxazolidinediones have attracted the attention of synthetic organic chemists because of their chemotherapeutic properties [1]. In recent past, we have been actively engaged in the synthesis of nitrogen-containing heterocycles by exploiting the *gem*-diester/cyanoester/dicyano functionalities [2]. In continuation of our ongoing program to develop some more interesting heterocycles, a new, simple, multifunctional reactive intermediates, dimethyl-2,2-bis-(2′-benzenesulfonylethyl)-malonate, dimethyl-2-(2′-arylsulfonyl-1′-arylethyl)-malonate, and dimethyl-2-[2′-(arylmethanesulfonyl)-1′-arylethyl]-malonate have been synthesized [3]. The latter has been utilized to get a variety of heterocycles, pyrimidine, pyrazole, and isoxazole derivatives.

RESULTS AND DISCUSSION

When vinyl sulfone was treated with dimethyl malonate instead of expected mono-addition product, dimethyl-2-(2′-benzenesulfonylethyl)-malonate, dimethyl-2,2-bis-(2′-benzenesulfonylethyl)-malonate (**1**) was also obtained. However, repetition of this reaction with 2 moles of vinyl sulfone and one mole of dimethyl malonate exclusively **1** was formed. On the other hand, the reaction of aryl styryl sulfone/benzyl styryl sulfone with dimethyl malonate gave dimethyl-2-(2′-arylsulfonyl-1′-arylethyl)-malonate (**7**)/dimethyl-2-[2′-(arylmethanesulfonyl)-1′-arylethyl]-malonate (**8**) [3]. The **1, 7**, and **8** have been used as synthons to get the desired products. The cyclocondensation of **1** with hydrazine, hydroxylamine, urea, *N,N*-dimethyl urea, and thiourea in the presence of sodium methoxide resulted in the formation of 4,4-bis-(2′-benzenesulfonylethyl)-pyrazolidine-3,5-dione (**2**), 4,4-bis-(2′-benzenesulfonylethyl)-isoxazolidine-3,5-dione (**3**), 5,5-bis-(2′-benzenesulfonylethyl)-pyrimidine-2,4,6-trione (**4**), 5,5-bis-(2′-benzenesulfonylethyl)-1,3-dimethylpyrimidine-2,4,6-trione (**5**), and 5,5-bis-(2′-benzenesulfonylethyl)-2-thioxodihydropyrimidine-4,6-dione (**6**) (Scheme 1 and Table 1). The absence of a band

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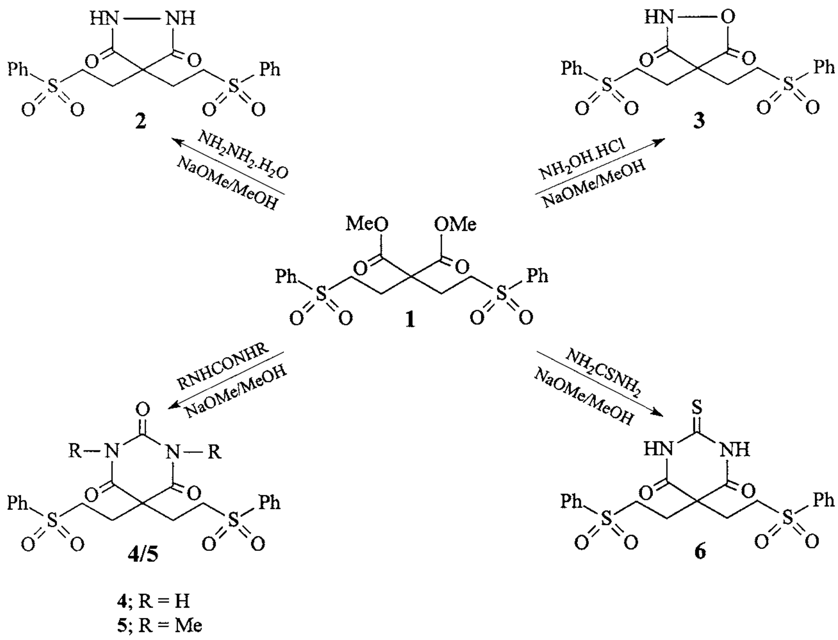


SCHEME 1

at 1760 cm^{-1} in the IR spectra of these compounds (**2–6**) due to carbonyl absorption of carbomethoxy group and the presence of absorption bands at 1100–1150, 1310–1350 (SO_2), and 1655–1725 cm^{-1} (CONH) indicated their formation. In addition to these, all the compounds except **5** exhibited an absorption band at 3300–3450 cm^{-1} (NH). The ^{1}H NMR spectra of **2–6** displayed two triplets at 2.30–2.35 and 3.40–3.44 ppm due to methylene protons. The downfield absorption was assigned to the one adjacent to sulfonyl group. Apart from these, compounds **2, 3, 4**, and **6** showed a broad singlet around 9.04–10.13 ppm for NH, which disappeared on deuteration. On the other hand, **5** showed a singlet at 2.83 ppm for *N*-methyl group (Table 2).

Adopting similar methodology, the reaction of dimethyl-2-(2′-arylsulfonyl-1′-arylethyl)-malonate (**7**)/dimethyl-2-[2′-(arylmethanesulfonyl)-1′-arylethyl]-malonate (**8**) with hydrazine, hydroxylamine, urea, *N,N*-dimethyl urea, and thiourea furnished 4-(2′-arylsulfonyl-1′-arylethyl)-pyrazolidine-3,5-dione (**9**)/4-[2′-(arylmethanesulfonyl)-1′-arylethyl]-pyrazolidine-3,5-dione (**10**), 4-(2′-arylsulfonyl-1′-arylethyl)-isoxazolidine-3,5-dione (**11**)/4-[2′-(arylmethanesulfonyl)-1′-arylethyl]-isoxazolidine-3,5-dione (**12**), 5-(2′-arylsulfonyl-1′-arylethyl)-pyrimidine-2,4,6-trione (**13**)/5-[2′-(arylmethanesulfonyl)-1′-arylethyl]-pyrimidine-2,4,6-trione (**14**), 5-(2′-arylsulfonyl-1′-arylethyl)-1,3-dimethylpyrimidine-2,4,6- trione (**15**)/5-[2′-(arylmethanesulfonyl)-1′-arylethyl]-1,3-dimethylpyrimidine-2,4,6-trione (**16**), and 5-(2′-arylsulfonyl-1′-arylethyl)-2-thioxodihydropyrimidine-4,6-dione (**17**)/5-(2′-arylmethanesulfonyl-1′-arylethyl)-2-thioxodihydropyrimidine-4,6-dione (**18**) (Scheme 2 and Table 1). The IR spectra of **9–18** exhibited absorption bands due to sulfonyl and amidic carbonyl functional groups at 1315–1340, 1130–1145, and 1650–1728 cm^{-1}. In addition to these compounds **9–14, 17** and **18** showed bands for NH group at 3325–3455 cm^{-1}. In the ^{1}H NMR spectra, a doublet was observed in the region 3.65–3.80 ppm for C_4—H in **9–12** and at 3.74–3.98 ppm for C_5—H in **13–18**. The compounds **9–18** showed a multiplet in the region 3.18–3.65 ppm for $C_{1'}$—H and a double doublet at 3.68–3.98 ppm for $C_{2'}$—H. Apart from these **10, 12, 14, 16**, and **18** showed a sharp singlet at 4.67–4.78 ppm for benzylic protons. Besides, the compounds **9–14**, and **17, 18** exhibited a broad singlet at 9.02–10.21 ppm for NH protons that disappeared on deuteration. The compounds **15** and **16** displayed a singlet at 2.80–2.85 ppm, which was accounted for N-Me group (Table 2).

In conclusion, interesting mono- and disubstituted pyrazolidinediones, isoxazolidinediones, pyrimidinetriones, and thioxopyrimidinediones were prepared from simple substrates by exploiting *gem*-diester functionality.

EXPERIMENTAL

Melting points were determined in open capillaries on a Mel-Temp apparatus and are uncorrected. The purity of the compounds was checked by TLC (silica gel H, BDH, ethyl acetate/hexane, 3:1). The IR

TABLE 1 Physical Data of Compounds **2–6** and **9–18**

						Found (calcd.) (%)		
Product	*Ar*	*Ar′*	*Melting Point (°C)*	*Yield (%)*	*Molecular Formula (Mol.Wt.)*	*C*	*H*	*N*
2	–	–	165–167	74	$C_{19}H_{20}N_2O_6S_2$ (436.51)	52.07 (52.28)	4.69 (4.62)	6.33 (6.42)
3	–	–	170–172	76	$C_{19}H_{19}NO_7S_2$ (437.50)	52.29 (52.16)	4.27 (4.38)	3.26 (3.20)
4	–	–	190–192	72	$C_{20}H_{20}N_2O_7S_2$ (464.52)	51.59 (51.71)	4.42 (4.34)	5.87 (6.03)
5	–	–	194–196	71	$C_{22}H_{24}N_2O_7S_2$ (492.57)	53.51 (53.64)	4.99 (4.91)	5.76 (5.68)
6	–	–	199–201	70	$C_{20}H_{20}N_2O_6S_3$ (480.59)	50.06 (49.98)	4.11 (4.19)	5.90 (5.83)
9a	C_6H_5	C_6H_5	205–207	79	$C_{17}H_{16}N_2O_4S$ (344.40)	59.39 (59.23)	4.74 (4.68)	8.23 (8.13)
9b	C_6H_5	4-Cl.C_6H_4	208–210	82	$C_{17}H_{15}ClN_2O_4S$ (378.84)	53.73 (53.90)	3.87 (3.99)	7.44 (7.39)
10a	4-Cl.C_6H_4	C_6H_5	187–189	77	$C_{18}H_{17}ClN_2O_4S$ (392.86)	54.86 (55.03)	4.28 (4.36)	7.22 (7.13)
10b	4-Cl.C_6H_4	4-Cl.C_6H_4	192–194	79	$C_{18}H_{16}Cl_2N_2O_4S$ (427.32)	50.65 (50.59)	3.65 (3.77)	6.61 (6.55)
11a	C_6H_5	C_6H_5	195–197	80	$C_{17}H_{15}NO_5S$ (345.39)	59.27 (59.12)	4.29 (4.38)	4.13 (4.05)
11b	C_6H_5	4-Cl.C_6H_4	198–200	78	$C_{17}H_{14}ClNO_5S$ (379.83)	53.71 (53.76)	3.80 (3.71)	3.55 (3.69)
12a	4-Cl.C_6H_4	C_6H_5	180–182	80	$C_{18}H_{16}ClNO_5S$ (393.86)	54.95 (54.89)	4.00 (4.09)	3.43 (3.55)
12b	4-Cl.C_6H_4	4-Cl.C_6H_4	185–187	82	$C_{18}H_{15}Cl_2NO_5S$ (428.31)	50.33 (50.48)	3.66 (3.53)	3.33 (3.27)
13a	C_6H_5	C_6H_5	210–212	75	$C_{18}H_{16}N_2O_5S$ (372.41)	57.97 (58.05)	4.26 (4.33)	7.65 (7.52)
13b	C_6H_5	4-Cl.C_6H_4	215–217	77	$C_{18}H_{15}ClN_2O_5S$ (406.85)	53.28 (53.14)	3.66 (3.72)	6.77 (6.88)
14a	4-Cl.C_6H_4	C_6H_5	190–192	78	$C_{19}H_{17}ClN_2O_5S$ (420.87)	54.34 (54.22)	4.11 (4.07)	6.73 (6.66)
14b	4-Cl.C_6H_4	4-Cl.C_6H_4	196–198	81	$C_{19}H_{16}Cl_2N_2O_5S$ (455.33)	49.98 (50.12)	3.45 (3.54)	6.01 (6.15)
15a	C_6H_5	C_6H_5	213–215	76	$C_{20}H_{20}N_2O_5S$ (400.46)	59.76 (59.99)	5.12 (5.03)	6.84 (6.99)
15b	C_6H_5	4-Cl.C_6H_4	208–210	78	$C_{20}H_{19}ClN_2O_5S$ (434.90)	55.41 (55.23)	4.51 (4.40)	6.36 (6.44)
16a	4-Cl.C_6H_4	C_6H_5	201–203	69	$C_{21}H_{21}ClN_2O_5S$ (448.93)	56.26 (56.18)	4.80 (4.71)	6.33 (6.24)
16b	4-Cl.C_6H_4	4-Cl.C_6H_4	208–210	72	$C_{21}H_{20}Cl_2N_2O_5S$ (483.38)	52.04 (52.18)	4.25 (4.17)	5.66 (5.79)
17a	C_6H_5	C_6H_5	218–220	73	$C_{18}H_{16}N_2O_4S_2$ (388.47)	55.48 (55.65)	4.22 (4.15)	7.14 (7.21)
17b	C_6H_5	4-Cl.C_6H_4	222–224	74	$C_{18}H_{15}ClN_2O_4S_2$ (422.91)	51.02 (51.12)	3.66 (3.57)	6.74 (6.62)
18a	4-Cl.C_6H_4	C_6H_5	207–209	73	$C_{19}H_{17}ClN_2O_4S_2$ (436.93)	52.45 (52.23)	3.87 (3.92)	6.49 (6.41)
18b	4-Cl.C_6H_4	4-Cl.C_6H_4	212–214	74	$C_{19}H_{16}Cl_2N_2O_4S_2$ (471.39)	48.63 (48.41)	3.33 (3.42)	5.88 (5.94)

spectra were recorded on a Perkin-Elmer grating infrared spectrophotometer, model 337 in KBr pellets. The ^{1}H NMR spectra were recorded in $CDCl_3$/DMSO-d_6 on a Varian EM-360 spectrometer (300 MHz) with TMS as an internal standard. The elemental analyses were performed at Punjab University, Chandigarh, India. The compounds dimethyl-2,2-bis-(2′-benzenesulfonylethyl)-malonate (**1**) and dimethyl-2-(2′-arylsulfonyl-1′-arylethyl)-malonate/dimethyl-2-[2′-(arylmethanesulfonyl)-1′-arylethyl]-malonate (**7/8**) were prepared according to the literature procedure [3].

TABLE 2 ^{1}H NMR Spectral Data for Compounds **2–6** and **9–18**

Product	*δ ^{1}H (ppm)*
2	2.33 (t, 4H, $C_{1'}$—H), 3.41 (t, 4H, $C_{2'}$—H), 7.24–7.95 (m, 10H, Ar-H), 9.11 (bs, 2H, NH)
3	2.30 (t, 4H, $C_{1'}$—H), 3.42 (t, 4H, $C_{2'}$—H), 7.28–7.93 (m, 10H, Ar-H), 10.13 (bs, 1H, NH)
4	2.31 (t, 4H, $C_{1'}$—H), 3.40 (t, 4H, $C_{2'}$—H), 7.30–7.96 (m, 10H, Ar-H), 9.04 (bs, 2H, NH)
5	2.30 (t, 4H, $C_{1'}$—H), 2.83 (s, 6H, N—CH_3), 3.19 (t, 4H, $C_{2'}$—H), 7.23–7.94 (m, 10H, Ar-H)
6	2.35 (t, 4H, $C_{1'}$—H), 3.44 (t, 4H, $C_{2'}$—H), 7.21–7.92 (m, 10H, Ar-H), 9.06 (bs, 2H, NH)
9a	3.21 (m, 1H, $C_{1'}$—H), 3.72 (dd, 2H, $C_{2'}$—H),3.76 (d, 1H, C_4—H),7.07–7.94 (m, 10H, Ar-H), 9.02 (bs, 2H, NH)
9b	3.23 (m, 1H, $C_{1'}$—H), 3.73 (dd, 2H, $C_{2'}$—H), 3.78 (d, 1H, C_4-H), 7.06–7.92 (m, 9H, Ar-H), 9.04 (bs, 2H, NH)
10a	3.22 (m, 1H, $C_{1'}$—H), 3.75 (d, 1H, C_4—H), 3.80 (dd, 2H, $C_{2'}$—H), 4.67 (s, 2H, Ar—CH_2—SO_2—), 7.01–7.19 (m, 9H, Ar-H), 9.44 (bs, 2H, NH)
10b	3.24 (m, 1H, $C_{1'}$—H), 3.76 (d, 1H, C_4—H), 3.84 (dd, 2H, $C_{2'}$—H), 4.70 (s, 2H, Ar—CH_2—SO_2—), 6.98–7.20 (m, 8H, Ar-H), 9.48 (bs, 2H, NH)
11a	3.63 (m, 1H, $C_{1'}$—H), 3.65 (d, 1H, C_4—H), 3.71 (dd, 2H, $C_{2'}$—H), 7.09–7.94 (m, 10H, Ar-H), 10.17 (bs, 1H, NH)
11b	3.65 (m, 1H, $C_{1'}$—H), 3.67 (d, 1H, C_4—H), 3.72 (dd, 2H, $C_{2'}$—H), 7.06–7.97 (m, 9H, Ar-H), 10.21 (bs, 1H, NH)
12a	3.55 (m, 1H, $C_{1'}$—H), 3.78 (d, 1H, C_4—H), 3.83 (dd, 2H, C2'—H), 4.72 (s, 2H, Ar—CH_2—SO_2—), 7.00–7.18 (m, 9H, Ar-H), 10.19 (bs, 1H, NH)
12b	3.58 (m, 1H, $C_{1'}$—H), 3.80 (d, 1H, C_4—H), 3.86 (dd, 2H, $C_{2'}$—H), 4.70 (s, 2H, Ar—CH_2—SO_2—), 7.02–7.20 (m, 8H, Ar-H), 10.14 (bs, 1H, NH)
13a	3.22 (m, 1H, $C_{1'}$—H), 3.75 (dd, 2H, $C_{2'}$—H), 3.78 (d, 1H, C_5—H), 7.08–7.93 (m, 10H, Ar-H), 9.20 (bs, 2H, NH)
13b	3.25 (m, 1H, $C_{1'}$—H), 3.70 (dd, 2H, $C_{2'}$—H), 3.76 (d, 1H, C_5—H), 7.08–7.94 (m, 9H, Ar-H), 9.24 (bs, 2H, NH)
14a	3.19 (m, 1H, $C_{1'}$—H), 3.74 (d, 1H, C_5—H), 3.82 (dd, 2H, $C_{2'}$—H), 4.74 (s, 2H, Ar—CH_2—SO_2—), 6.96–7.14 (m, 9H, Ar-H), 9.95 (bs, 2H, NH)
14b	3.21 (m, 1H, $C_{1'}$—H), 3.77 (d, 1H, C_5—H), 3.88 (dd, 2H, $C_{2'}$—H), 4.71 (s, 2H, Ar—CH_2—SO_2—), 7.01–7.18 (m, 8H, Ar-H), 9.92 (bs, 2H, NH)
15a	2.80 (s, 6H, N—CH_3), 3.20 (m, 1H, $C_{1'}$—H), 3.85 (dd, 2H, $C_{2'}$—H), 3.96 (d, 1H, C_5-H), 7.02–7.89 (m, 10H, Ar-H)
15b	2.85 (s, 6H, N—CH_3), 3.24 (m, 1H, $C_{1'}$—H), 3.90 (dd, 2H, $C_{2'}$—H), 3.98 (d, 1H, C_5-H), 7.10–7.95 (m, 9H, Ar-H)
16a	2.82 (s, 6H, N—CH_3), 3.18 (m, 1H, $C_{1'}$—H), 3.68 (dd, 2H, $C_{2'}$—H), 3.75 (d, 1H, C_5—H), 4.72 (s, 2H, Ar—CH_2—SO_2—), 7.12–7.20 (m, 9H, Ar-H)
16b	2.84 (s, 6H, N—CH_3), 3.20 (m, 1H, $C_{1'}$—H), 3.72 (dd, 2H, $C_{2'}$—H), 3.76 (d, 1H, C_5—H), 4.74 (s, 2H, Ar—CH_2—SO_2—), 7.10–7.21 (m, 8H, Ar-H)
17a	3.28 (m, 1H, $C_{1'}$—H), 3.87 (dd, 2H, $C_{2'}$—H), 3.92 (d, 1H, C_5—H), 7.12–7.98 (m, 10H, Ar-H), 9.32 (bs, 2H, NH)
17b	3.25 (m, 1H, $C_{1'}$—H), 3.82 (dd, 2H, $C_{2'}$—H), 3.93 (d, 1H, C_5—H), 7.05–7.92 (m, 9H, Ar-H), 9.30 (bs, 2H, NH)
18a	3.22 (m, 1H, $C_{1'}$—H), 3.78 (d, 1H, C_5—H), 3.98 (dd, 2H, $C_{2'}$—H), 4.75 (s, 2H, Ar—CH_2—SO_2—), 7.00–7.18 (m, 9H, Ar-H), 9.20 (bs, 2H, NH)
18b	3.25 (m, 1H, $C_{1'}$—H), 3.80 (d, 1H, C_5—H), 3.90 (dd, 2H, $C_{2'}$—H), 4.78 (s, 2H, Ar—CH_2—SO_2—), 6.96–7.16 (m, 8H, Ar-H), 9.22 (bs, 2H, NH)

*4,4-Bis-(2′-benzenesulfonylethyl)-pyrazolidine-3,5-dione (**2**)/4-(2′-arylsulfonyl-1′-arylethyl)-pyrazolidine-3,5-dione (**9**)/4-(2′-arylmethanesulfonyl-1′arylethyl)-pyrazolidine-3,5-dione (**10**)*

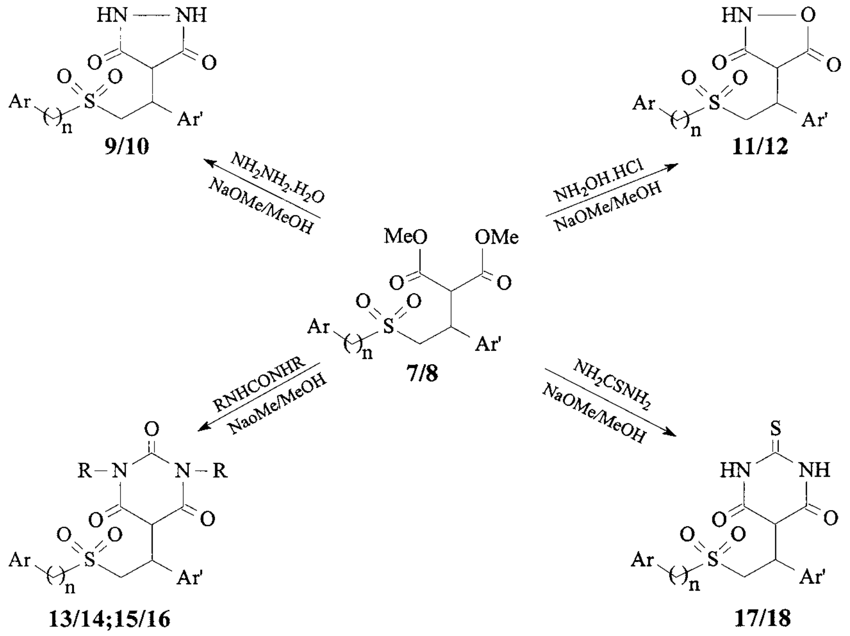
A mixture of **1/7/8** (10 mmol), hydrazine hydrate (15 mmol), MeOH (20 mL), and 10% NaOMe (5 mL) was refluxed for 5–6 h. The solution was cooled and poured onto crushed ice containing hydrochloric acid. The solid obtained was filtered, dried, and recrystallized from MeOH.

*4,4-Bis-(2′-benzenesulfonylethyl)-isoxazolidine-3,5-dione (**3**)/4-(2′-arylsulfonyl-1′-aryl-ethyl)-isoxazolidine-3,5-dione (**11**)/4-(2′-arylmethanesulfonyl-1′ arylethyl)-isoxazolidine-3,5-dione (**12**)*

To a solution of **1/7/8** (10 mmol) in MeOH (20 mL), hydroxylamine hydrochloride (10 mmol) and 10% NaOMe (5 mL) were added and refluxed for 6–9 h. The reaction mixture was cooled and poured onto crushed ice containing acetic acid. The solid separated was filtered, dried, and recrystallized from MeOH.

*5,5-Bis-(2′-benzenesulfonylethyl)-pyrimidine-2,4,6-trione (**4**)/5,5-bis-(2′-benzenesulfonylethyl)-1,3-dimethylpyrimidine-2,4,6-trione (**5**)/5-(2′-arylsulfonyl-1′-arylethyl)-pyrimidine-2,4,6-trione (**13**)/5-(2′-arylmethanesulfonyl-1′ arylethyl)-pyrimidine-2,4,6-trione (**14**)/5-(2′-arylsulfonyl-1′-arylethyl)-1,3-dimethylpyrimidine-2,4,6-trione (**15**)/5-(2′-arylmethanesulfonyl-1′ arylethyl)-1,3-dimethylpyrimidine-2,4,6-trione (**16**)*

The compound **1/7/8** (10 mmol) was dissolved in MeOH (10 mL). To this urea/*N*,*N*-dimethyl urea (10 mmol) in MeOH (10 mL) was added and refluxed



n = 0 (**7,9,11,13,15,17**); n = 1 (**8,10,12,14,16,18**)

R=H (**13,14**); R=Me (**15,16**)

Ar=Ar'=Ph(**7a,9a,11a,13a,15a,17a**); Ar=Ph, Ar'=4-Cl.Ph (**7b,9b,11b,13b,15b,17b**)

Ar=4-Cl.Ph, Ar'=Ph(**8a,10a,12a,14a,16a,18a**); Ar=Ar'=4-Cl.Ph (**8b,10b,12b,14b,16b,18b**)

SCHEME 2

for 8–12 h. The contents were cooled and poured onto crushed ice containing hydrochloric acid. The separated solid was filtered and recrystallized from MeOH.

*5,5-Bis-(2′-benzenesulfonylethyl)-2-thioxodihydro-pyrimidine-4,6-dione (**6**)/5-(2′-arylsulfonyl-1′-arylethyl)-2-thioxodihydropyrimidine-4,6-dione (**17**)/5-(2′-arylmethanesulfonyl-1′ arylethyl)-2-thioxodihydropyrimidine-4,6-dione (**18**)*

To a solution of **1/7/8** (10 mmol) in MeOH (20 mL), thiourea (10 mmol), and NaOMe (5 mL) were added and refluxed for 7–12 h. The reaction mixture was cooled and poured onto crushed ice containing hydrochloric acid. The solid separated was filtered and purified by recrystallization from MeOH.

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