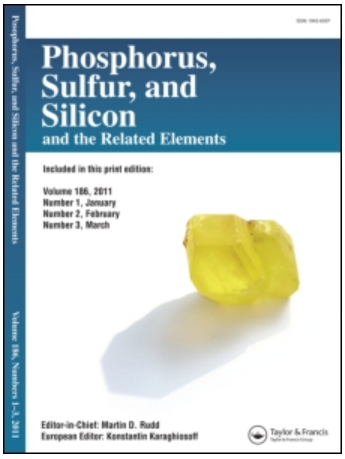

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A Diastereoselective Synthesis of Novel Multifunctional Thianes

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The reaction of 2,2′-thiobis(1,3-diarylprop-2-en-1-ones) with malononitrile in the presence of sodium ethoxide under both thermal and solvent-free microwave conditions afforded a series of hitherto unknown 2e,6e-diaroyl-3e,5e-diaryl-4,4-dicyanothianes diastereoselectively in good yields via a double Michael addition. The structures of all the compounds have been elucidated by IR, ^{1}H NMR, ^{13}C NMR, and elemental analysis.

Keywords Malononitrile; Michael addition; microwave irradiation; thianes; 2,2′-thiobis(1,3-diarylprop-2-en-1-ones)

INTRODUCTION

In continuation of our work on the synthesis of novel heterocyclic compounds[1] employing 2,2′-thio/2,2′-sulfonyl-bis(1,3-diarylprop-2-en-1-ones), we herein report a green, as well as conventional thermal approach, to the synthesis of multisubstituted thianes. The synthesis of thianes assumes importance since naturally occurring six-membered sulfur heterocycles are scant.[2] Moreover the thiane ring plays a key role in the biological activities of drugs such as cephalosporins[3] and dithiathromboxane.[4] They also display several pharmacological properties such as anti-inflammatory,[5] antihypertensive,[6] antiulcer,[7] antimicrobial,[8] antibacterial,[9] and analgesic[10] activities in addition to their use in industry as bleaching[11] and antiwear[12] agents and stabilizing agents in color photography.[13] Moreover, the title compounds possess

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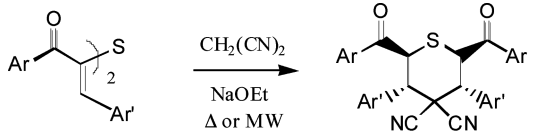
gem-dicyano groups, which provide scope for the synthesis of spiro heterocycles.[14] It is also pertinent to note that though the syntheses of a large number of a variety of heterocycles by microwave-assisted synthesis have been reported, there is no report on the synthesis of thianes using microwave irradiation.

RESULTS AND DISCUSSION

In the present investigation, 2,6-diaroyl-3,5-diaryl-4,4-dicyanothianes were obtained in good yields by the reaction of the 2,2′-thiobis(1,3-diarylprop-2-en-1-ones) **1** in DMF with malononitrile in the presence of sodium ethoxide at room temperature for 1 h (Scheme 1).

Alternatively the same reaction was carried out under solvent-free microwave irradiation in order to effect a green synthesis and to see whether there is any product/stereoselectivity, as it is not uncommon to get different products from reactions under solvent-free microwave



1 **2**

1 & 2	Ar	Ar′
a	C_6H_5	C_6H_5
b	C_6H_5	*p*-Cl-C_6H_4
c	C_6H_5	*o*-MeO-C_6H_4
d	C_6H_5	*p*-O_2N-C_6H_4
e	*p*-Cl-C_6H_4	C_6H_5
f	*p*-Cl-C_6H_4	*p*-F-C_6H_4
g	*p*-Cl-C_6H_4	*p*-Cl-C_6H_4
h	*p*-Cl-C_6H_4	*p*-Me-C_6H_4
i	*p*-Me-C_6H_4	C_6H_5
j	*p*-Me-C_6H_4	*p*-Cl-C_6H_4

SCHEME 1 Synthesis of 2e, 6e-diaroyl-3e, 5e-diaryl-4,4-dicyanothianes.

TABLE I Yields and Reaction Time for Conventional Heating and Microwave Reactions

		Microwave irradiation		Conventional at ambient temp.	
No.	Compd.	Yield (%)	Time (min)	Yield (%)	Time (min)
1	**2a**	86	5	82	60
2	**2b**	79	5	76	60
3	**2c**	66	5	65	60
4	**2d**	66	5	83	60
5	**2e**	83	5	79	60
6	**2f**	91	5	85	60
7	**2g**	93	5	84	60
8	**2h**	92	5	82	60
9	**2i**	81	5	79	60
10	**2j**	82	5	73	60

irradiation and in solution under conventional heating.[15–19] In addition, microwave reactions often proceed more rapidly than conventional thermal reactions with diminished decomposition of the products and enhanced yields.

In a typical reaction, 2,2′-thiobis(1,3-diarylprop-2-en-1-ones) **1**, malononitrile, and solid sodium ethoxide were mixed thoroughly in a borosilicate boiling tube, kept in a silica bath, which in turn was placed in a microwave oven (750 W and frequency of 2450 MHz) and irradiated at power level 4 of a total scale of 6. Completion of the reaction (TLC) requires 5 min of microwave irradiation in one stretch without intermittent cooling. The reaction mixture, after workup, afforded good yields of the same thianes obtained under thermal conditions described above (Scheme 1).

The results obtained in the present study regarding thermal versus microwave reactions in the synthesis of the title compounds are shown in Table I. It is clear that the microwave-assisted synthesis afforded better yields in all the cases, except one giving **2d**, and the reaction time is also many-fold shorter than the conventional method. It also has the obvious advantages of the absence of solvent and easy workup.

The thianes, **2a**–**2j**, obtained were characterized by elemental analysis, IR, ^{1}H NMR, and ^{13}C NMR spectroscopic data. The elucidation of the structure of **2** is discussed by taking **2h** as an example. The ^{1}H NMR spectrum of **2h** displayed two 2H doublets at 4.24 ($J = 10.8$ Hz)

and 5.62 ppm (J = 10.8 Hz) assignable to H-3,5 and H-2,6 of the thiane ring, respectively, on the basis of substituent effects and analogy with the chemical shifts of similar compounds.[1g] The thiane **2h** also has signals for the aromatic protons between 7.11 and 7.85 ppm and tolyl methyl protons as a singlet at 2.27 ppm. The coupling constant of the doublets (J = 10.8 Hz) of H-3,5 and H-2,6 indicates the diaxial relationship between the vicinal hydrogens, which points to the equatorial orientations of the aryl groups at C-3,5 and aroyl groups at C-2,6. Thus the compound is characterized as 2e,6e-diaroyl-3e,5e-diaryl-4,4-dicyanothianes. In its ^{13}C NMR spectrum, **2h** showed two signals at 112.9 and 113.8 for the diastereotopic cyanide groups besides the other expected signals. The other thianes also showed similar spectroscopic features as **2h**.

The formation of thianes displays a double Michael addition of the carbanion, generated from malononitrile, in distinct steps to the thiobis compound **1**.

CONCLUSION

The present work describes the synthesis of multisubstituted thianes by conventional solution chemistry and solvent-free, microwave-assisted synthesis. Though both the methods afforded the same products, the yields are higher with great diminution in reaction time under the solvent-free, microwave irradiation procedure. Further utility of the multifunctional thianes, as synthons in the synthesis of spiro heterocycles, currently is being explored in our group.

EXPERIMENTAL

The melting points are uncorrected. A domestic microwave oven (IFB, model electron of 750 W capacity and microwave frequency of 2450 MHz) was employed for microwave irradiation. IR spectra were recorded on a Shimadzu instrument with KBr pellet. NMR spectra were recorded at 20°C on a Bruker AMX 300 instrument operating at 300 MHz for ^{1}H and 75 MHz for ^{13}C. Solutions (in $CDCl_3$) were approximately 0.05 M, and chemical shifts were referenced internally to TMS in all cases and expressed in δ scale (ppm). Elemental analyses were performed on a Perkin-Elmer 2400 Series II Elemental CHNS Analyzer. The Michael acceptor **1** was obtained by the method in the literature.[20]

General Procedure for the Synthesis of 2e,6e-Diaroyl-3e,5e-diaryl-4,4-dicyanothianes: Synthesis of 2e,6e-Dibenzoyl-4,4-dicyano-3e,5e-diphenylthiane (2a)

Method 1: By the Solvent-Free Microwave Irradiation Method

Malononitrile (0.5 g, 5 mmol) was added dropwise with stirring into solid sodium ethoxide prepared by dissolving sodium metal (0.12 g, 5 mmol) in absolute ethyl alcohol (4 mL) and evaporating the solvent. To this mixture, 2,2′-thiobis(1,3-diphenylprop-2-en-1-one) (2.2 g., 5 mmol) was added and thoroughly mixed in a mortar. The mixture was then transferred into a borosilicate boiling tube, immersed in a silica bath, and kept in a domestic microwave oven at power level 4 (400 W) for 5 min. The completion of the reaction was followed by TLC. The reaction mixture was first washed with water, then with ethyl alcohol, and crystallized from an ethyl alcohol:chloroform mixture (3:2, 50 mL) to give 2e,6e-dibenzoyl-4,4-dicyano-3e,5e-diphenylthiane as a colorless solid, mp 282–284 °C; IR (KBr) ν 1678, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.30 (d, 2H, $J = 10.5$ Hz), 5.72 (d, 2H, $J = 10.5$ Hz), 7.28–7.62 (m,14H), 7.73 (dd, 2H, $J = 3.3, 5.7$ Hz), 7.91 (d, 4H, $J = 7.8$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 46.3, 48.8, 52.7, 113.0, 113.4, 128.6, 128.8, 128.9, 129.1, 130.9, 134.3, 135.0, 135.2, 192.2. Anal. Calcd for $C_{33}H_{24}N_2O_2S$: C, 77.32; H, 4.72; N, 5.46. Obsd: C, 77.48; H, 4.74; N, 5.43%.

Method 2: By the Conventional Thermal Method

Malononitrile (0.5 g, 5 mmol) was added dropwise with stirring into a solution of sodium ethoxide prepared by dissolving sodium metal (0.12 g, 5 mmol) in absolute ethyl alcohol (4 mL). To this mixture, a solution of 2,2′-thiobis(1,3-diphenylprop-2-en-1-one) (2.2 g, 5 mmol) in dimethylformamide (40 mL) was added with stirring and kept at room temperature for 1 h. The reaction mixture was then poured into water, and the separated solid was filtered and crystallized from an ethyl alcohol:chloroform mixture (3:2, 50 mL) to give 2e,6e-dibenzoyl-4,4-dicyano-3e,5e-diphenylthiane **2a**.

2e,6e-Dibenzoyl-3e,5e-di(*p*-chlorophenyl)-4,4-dicyanothiane (2b)

Obtained as a colorless solid, mp 262–264°C; IR (KBr) ν 1673, 2254 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.31 (d, 2H, $J = 10.5$ Hz), 5.67 (d, 2H, $J = 10.5$ Hz), 7.29–7.73 (m, 14H), 7.92 (d, 4H, $J = 7.5$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 46.2, 48.5, 51.9, 112.6, 113.4, 128.6, 129.1, 129.4, 130.0, 133.6, 134.6, 135.6, 191.8. Anal. Calcd for $C_{33}H_{22}Cl_2N_2O_2S$: C, 68.16; H, 3.81; N, 4.82. Obsd: C, 67.98; H, 3.79; N, 4.84%.

3e,5e-Di(*o*-anisyl)-2e,6e-dibenzoyl-4,4-dicyanothiane (2c)

Obtained as a colorless solid, mp 244–246°C; IR (KBr) ν 1673, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 3.91 (s, 6H), 5.12 (broad signal, 2H), 5.72 (broad signal, 2H), 6.78–7.59 (m, 14H), 7.93 (d, 4H, $J = 7.5$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 47.0, 48.0, 55.9, 111.6, 114.1, 120.7, 128.6, 128.8, 130.2, 134.1, 135.2, 192.6. Anal. Calcd for $C_{35}H_{28}N_2O_4S$: C, 73.41; H, 4.93; N, 4.89. Obsd: C, 73.58; H, 4.94; N, 4.91%.

2e,6e-Dibenzoyl-4,4-dicyano-3e,5e-di(*p*-nitrophenyl)thiane (2d)

Obtained as a colorless solid, mp 260–262°C; IR (KBr) ν 1345, 1527, 1675, 2254 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.48 (d, 2H, $J = 10.5$ Hz), 5.74 (d, 2H, $J = 10.5$ Hz), 7.49–7.73 (m,10H), 7.94 (d, 4H, $J = 7.5$ Hz), 8.23 (d, 4H, $J = 8.7$ Hz); Anal. Calcd for $C_{33}H_{22}N_4O_6S$: C, 65.77; H, 3.68; N, 9.30. Obsd: C, 65.95; H, 3.69; N, 9.35%.

2e,6e-Di(*p*-chlorobenzoyl)-4,4-dicyano-3e,5e-diphenylthiane (2e)

Obtained as a colorless solid, mp 236–238°C; IR (KBr) ν 1682, 2246 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.29 (d, 2H, $J = 10.5$ Hz), 5.64 (d, 2H, $J = 10.5$ Hz), 7.31–7.55 (m, 14H), 7.84 (d, 4H, $J = 8.7$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 46.3, 48.6, 52.4, 112.7, 113.7, 128.7, 129.2, 129.4, 129.6, 130.0, 133.2, 135.0, 141.1, 191.0. Anal. Calcd for $C_{33}H_{22}Cl_2N_2O_2S$: C, 68.16; H, 3.81; N, 4.82. Obsd: C, 68.28; H, 3.79; N, 4.79%.

2e,6e-Di(*p*-chlorobenzoyl)-4,4-dicyano-3e,5e-di (*p*-fluorophenyl)thiane (2f)

Obtained as a colorless solid, mp 288–290°C; IR (KBr) ν 1680, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.29 (d, 2H, $J = 10.5$ Hz), 5.56 (d, 2H, $J = 10.5$ Hz), 7.00–7.50 (m, 12H), 7.86 (d, 4H, $J = 8.7$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 46.2, 48.6, 51.6, 112.5, 113.5, 116.3, 116.6, 129.5, 130.0, 130.5, 130.6, 130.8, 132.9, 141.4, 190.7. Anal. Calcd for $C_{33}H_{20}Cl_2F_2N_2O_2S$: C, 64.19; H, 3.26; N, 4.54. Obsd: C, 64.01; H, 3.24; N, 4.56%.

2e,6e-Di(*p*-chlorobenzoyl)-3e,5e-di(*p*-chlorophenyl) -4,4-dicyanothiane (2g)

Obtained as a colorless solid, mp 238–240°C; IR (KBr) ν 1680, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 4.26 (d, 2H, $J = 10.2$ Hz), 5.56 (d, 2H, $J = 10.2$ Hz), 7.31–7.47 (m, 12H), 7.85 (d, 4H, $J = 8.1$ Hz). Anal. Calcd for $C_{33}H_{20}Cl_4N_2O_2S$: C, 60.94; H, 3.10; N, 4.31. Obsd: C, 60.85; H, 3.12; N, 4.29%.

2e,6e-Di(*p*-chlorobenzoyl)-4,4-dicyano-3e,5e-di (*p*-methylphenyl)thiane (2h)

Obtained as a colorless solid, mp 240–242°C; IR (KBr) ν 1688, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 2.27 (s, 6H), 4.24 (d, 2H, $J = 10.8$ Hz), 5.62 (d, 2H, $J = 10.8$ Hz), 7.11 (d, 4H, $J = 7.8$ Hz); 7.37 (d, 4H, $J = 7.8$ Hz), 7.42 (d, 4H, $J = 8.4$ Hz), 7.85 (d, 4H, $J = 8.4$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 21.1, 46.2, 49.0, 52.1, 112.9, 113.8, 128.5, 129.3, 129.9, 130.0, 132.1, 133.2, 139.5, 141.0, 191.1. Anal. Calcd for $C_{35}H_{26}Cl_2N_2O_2S$: C, 68.96; H, 4.30; N, 4.60. Obsd: C, 69.14; H, 4.28; N, 4.63%.

4,4-Dicyano-2e,6e-di(*p*-methylbenzoyl)-3e,5e-diphenylthiane (2i)

Obtained as a colorless solid, mp 266–268°C; IR (KBr) ν 1688, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 2.39 (s, 6H), 4.30 (d, 2H, $J = 10.5$ Hz), 5.73 (d, 2H, $J = 10.5$ Hz), 7.22–7.54 (m, 14H), 7.83 (d, 4H, $J = 7.5$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 21.7, 46.1, 48.9, 52.7, 112.9, 113.7, 128.7(6), 128.8(1), 129.0, 129.3, 129.6, 132.5, 135.3, 145.5, 191.6. Anal. Calcd for $C_{35}H_{28}N_2O_2S$: C, 77.75; H, 5.22; N, 5.18. Obsd: C, 77.60; H, 5.19; N, 5.21%.

4,4-Dicyano-2e,6e-di(*p*-methylbenzoyl)-3e,5e-di (*p*-chlorophenyl)thiane (2j)

Obtained as a colorless solid, mp 248–250°C; IR (KBr) ν 1688, 2250 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H 2.41 (s, 6H), 4.27 (d, 2H, $J = 10.8$ Hz), 5.64 (d, 2H, $J = 10.8$ Hz), 7.25–7.44 (m, 12H), 7.82 (d, 4H, $J = 8.0$ Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ_C 21.8, 46.0, 48.6, 52.0, 112.3, 113.4, 128.8, 129.4, 129.8, 130.0, 132.2, 133.7, 135.5, 145.9, 191.2. Anal. Calcd for $C_{35}H_{26}Cl_2N_2O_2S$: C, 68.96; H, 4.30; N, 4.60. Obsd: C, 68.80; H, 4.32; N, 4.58%.

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