Pergamon

Tetrahedron 58 (2002) 4551–4554

TETRAHEDRON

Studies on sulfoxide rearrangement: synthesis of dimedone-annelated heterocycles

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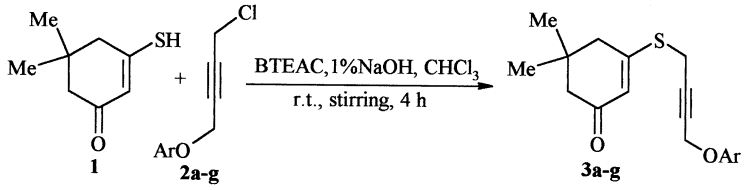
Received 1 January 2002; revised 7 March 2002; accepted 4 April 2002

Abstract—A number of 3-(4′-aryloxybut-2′-ynyl)thio-5,5-dimethyl cyclohex-2-enones are synthesized in 80–90% yield by phase transfer catalyzed reaction of 5,5-dimethyl-3-merca-ptocyclohex-2-enone with a number of 1-aryloxy-4-chlorobut-2-ynes. The sulfides are oxidized with 1 equiv. of *m*-chloroperoxybenzoic acid and the resulting sulfoxides are then stirred at r.t. for 6–8 h to give 2-aryloxymethyl-3-(*m*-chlorobenzoyloxy)-6,6-dimethyl-5,6,7-trihydro benzo(*b*)thiophene-4-ones in 70–80% yield. © 2002 Published by Elsevier Science Ltd.

Aliphatic Claisen rearrangement[1] has become a powerful tool for carbon–carbon bond formation.[2] Its importance is partly due to the subsequent development of a series new variants[3] of the [3,3] sigmatropic rearrangement and partly due to its versatile application in the synthesis of various heterocycles using oxygen,[4] nitrogen,[5] and sulfur[6] Claisen rearrangements. Our recent work on the synthesis of various heterocycles by the application of [3,3] sigmatropic rearrangement[7] motivated us to study the synthesis of dimedone-annelated heterocycles.[8] In continuation, we undertook a study on the synthesis of dimedone-annelated thiophene by the application of sulfoxide rearrangement, a mild and simple methodology we had reported earlier.[9]

The starting materials (**3a**–**g**) for this study were prepared in 80–90% yield by the phase transfer catalyzed alkylation of 5,5-dimethyl-3-mercaptocyclohex-2-enone[10] (**1**) with a number of 1-aryloxy-4-chlorobut-2-ynes (**2a**–**g**) in chloroform and 1% aqueous NaOH solution in the presence of benzyltriethylammonium chloride at room temperature for 4 h (Scheme 1).

Substrate **3a** was treated with 1 equiv. of 3-chloroperoxybenzoic acid in chloroform at 0–5°C for 10 min. TLC showed a highly polar single spot indicating the formation of sulfoxide (**4a**). This product exhibited tendency to further reorganization. The sulfoxide could not be isolated in the pure form. It rearranged even during workup. Therefore, no attempt was made to characterize the sulfoxide. The reaction mixture was stirred for 6–8 h at room temperature to give a single product. Its mass spectrum indicated the incorporation of *m*-chloroperoxy unit as appendage. From



3.a. Ar = -C_6H_5 (90%)
b. Ar = 2-ClC_6H_4 (87%)
c. Ar = 4-ClC_6H_4 (85%)
d. Ar = 4-$MeOC_6H_4$ (80%)
e. Ar = 4-MeC_6H_4 (88%)
f. Ar = 2,4-$Cl_2C_6H_3$ (85%)
g. Ar = 2-MeC_6H_4 (86%)

Scheme 1.

Keywords: [2,3] sigmatropic rearrangement; sulfoxide; *m*-chloroperoxybenzoic acid; 2-aryloxymethyl-3-(*m*-chlorobenzoyloxy)-6,6-dimethyl-5,6,7-trihydro benzo(*b*)thiophene-4-ones; regioselective heterocyclization.

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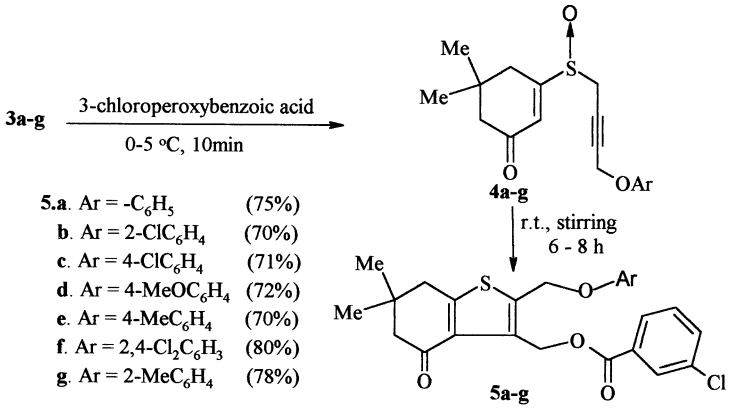
0040–4020/02/$ - see front matter © 2002 Published by Elsevier Science Ltd.
PII: S0040-4020(02)00396-4



Scheme 2.

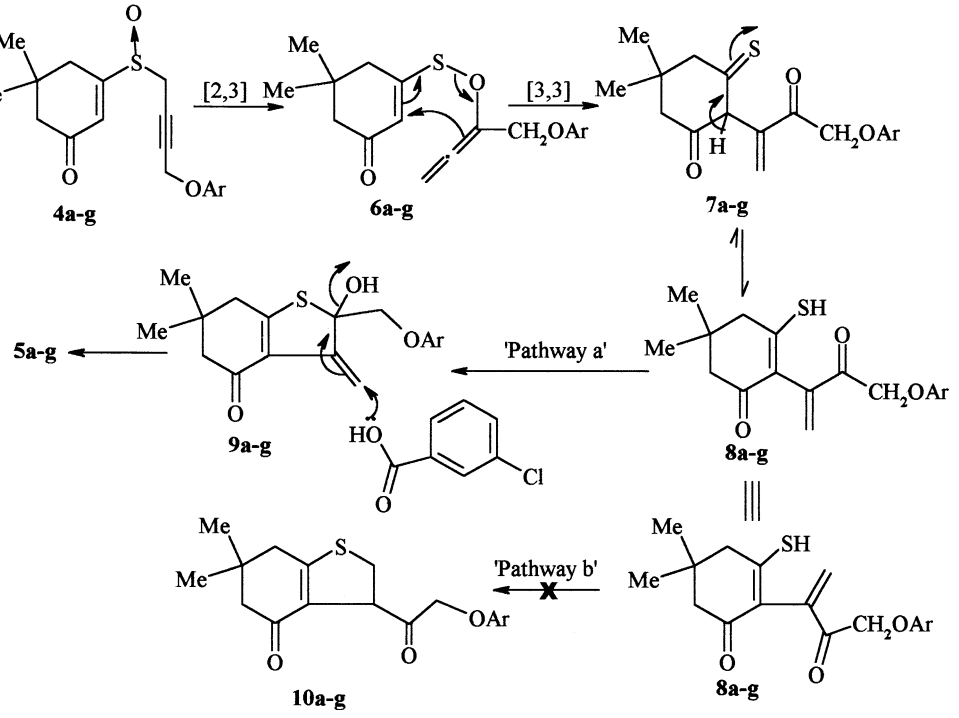
its elemental analysis and spectroscopic data, the product was characterized as 3-(*m*-chlorobenzoyloxymethyl)-2-phenoxymethyl-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-one **5a**. Encouraged by this result, other substrates **3b**–**g** were similarly treated to give products **5b**–**g** in 70–80% yields (Scheme 2). The formation of products **5a**–**g** from the substrates **3a**–**g** may be easily explained by the facile oxidation of **3a**–**g** with 1 equiv. of 3-chloroperoxybenzoic acid to the corresponding sulfoxides **4a**–**g**. A [2,3] sigmatropic rearrangement of the vinyl propargyl sulfoxides (**4a**–**g**) may give unstable allenesulfenates (**6a**–**g**) which may then undergo a [3,3] sigmatropic rearrangement giving intermediates (**7a**–**g**). In intermediates (**7a**–**g**), the carbonyl function and –SH group are suitably juxtaposed to give thioketols (**8a**–**g**). An S_N2/displacement in **9a**–**g** may afford the final products **5a**–**g** (pathway a). Alternatively, intermediate **8a**–**g** could have provided **10a**–**g** by an internal Michael addition (pathway b). However, no such product (**10a**–**g**) was obtained (Scheme 3).

Sulfoxide rearrangements reported earlier usually require refluxing[9a] in CCl_4. In the present instance, room temperature stirring brings about the sulfoxide rearrangement. Incorporation of *m*-chlorobenzoyloxy unit as an appendage was earlier observed only in the case of amine–oxide rearrangement.

The reaction described here is found to be general. The oxidation step is chemoselective as the other sensitive functionalities remain unaffected by *m*-chloroperoxybenzoic acid. This as a mild and simple method for the synthesis of the hydroaromatic system 6,6-trihydro benzo(*b*)thiophene-4-ones.

1. Experimental

Melting points were measured on a sulfuric acid bath and are uncorrected. UV absorption spectra were recorded in



Scheme 3.

EtOH on a Hitachi 200-20 Spectrophotometer. IR spectra were run on KBr disks on a Perkin–Elmer 1330 apparatus. ^{1}H NMR, ^{13}C NMR and DEPT Spectra were determined for solutions in $CDCl_3$ with TMS as internal standard on a Bruker 300 (300 MHz) instrument. Elemental analyses and recording of mass spectra were carried out by RSIC (CDRI) Lucknow on a [JEOL D-300 (El)] instrument. Silica gel (60–120 mesh), Spectrochem, India, was used for chromatographic separation. Petroleum ether refers to the fraction boiling between 60 and 80°C.

1.1. General procedure for the synthesis of 3a–g

To a mixture of 1-aryloxy-4-chlorobut-2-yne **2a–g** (1 mmol) and 3-mercapto-5,5-dimethyl cyclohex-2-enone (1 mmol) **2a–g** in chloroform (100 mL) was added to a solution of benzyltriethylammonium chloride (BTEAC, 0.5 g, 1.8 mmol) in 1% aqueous NaOH (100 mL) and the mixture was magnetically stirred at room temperature for 4 h. The reaction mixture was then diluted with water (50 mL). Chloroform layer was taken out and washed with 2N HCl (20 mL), brine (20 mL), water (20 mL) and dried (Na_2SO_4). Evaporation of chloroform left a gummy residue which was subjected to column chromatography. Elution of the column with benzene afforded compounds **3a–g**.

1.1.1. 3-(4′-Phenoxybut)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3a). Yield 90%; viscous liquid; $\lambda_{max:}$ 218, 285 nm; ν_{max}: 1140, 1590, 1660, 2980 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 3H, *Me*), 1.07 (s, 3H, *Me*), 2.25 (s, 2H, CH_2CO), 2.42 (s, 2H, =CCH_2), 3.57–3.59 (t, J=1.9 Hz, 2H, SCH_2), 4.77–4.78 (t, J=1.9 Hz, 2H, OCH_2), 5.93 (s, 1H, =C*H*), 6.92–7.30 (m, 5H, Ph); MS *m*/*z* 300 (M$^+$); Anal. calcd for $C_{18}H_{20}O_2S$: C, 71.97; H, 6.71; found C, 72.12; H, 6.49%.

1.1.2. 3-(4′-(2′-Chlorophenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3b). Yield 87%; viscous liquid; λ_{max}: 218, 282 nm; ν_{max}: 1130, 1580, 1650, 2970 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 3H, *Me*), 1.07 (s, 3H, *Me*), 2.26 (s, 2H, CH_2CO), 2.29 (s, 2H, =CCH_2), 3.57–3.58 (t, J=1.9 Hz, 2H, SCH_2), 4.75–4.76 (t, J=1.9 Hz, 2H, OCH_2), 5.92 (s, 1H, =C*H*), 6.93–7.37 (m, 4H, Ph); MS *m*/*z* 334, 336 (M$^+$); Anal. calcd for $C_{18}H_{19}ClO_2S$: C, 64.59; H, 5.72; found C, 64.50; H, 5.52%.

1.1.3. 3-(4′-(4′-Chlorophenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3c). Yield 85%; viscous liquid; λ_{max}: 227, 282 nm; ν_{max}: 1140, 1580, 1650, 2970 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 3H, *Me*), 1.09 (s, 3H, *Me*), 2.25 (s, 2H, CH_2CO), 2.28 (s, 2H, =CCH_2), 3.57–3.58 (t, J=1.9 Hz, 2H, SCH_2), 4.65–4.67 (t, J=1.9 Hz, 2H, OCH_2), 5.92 (s, 1H, =C*H*), 6.84–6.89 (d, J=12 Hz, 2H, Ph), 7.22–7.25 (d, J=12 Hz, 2H, Ph); MS *m*/*z* 334, 336 (M$^+$); Anal. calcd for $C_{18}H_{19}ClO_2S$: C, 64.59; H, 5.72; found C, 64.43; H, 5.61%.

1.1.4. 3-(4′-(4′-Methoxyphenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3d). Yield 80%; viscous liquid; λ_{max}: 225, 285 nm; ν_{max}: 1140, 1580, 1640, 2970 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 3H, *Me*), 1.08 (s, 3H, *Me*), 2.25 (s, 2H, CH_2CO), 2.38 (s, 2H, =CCH_2), 3.58 (brs, 2H, SCH_2), 3.77 (s, 3H, –O*Me*), 4.62 (brs, 2H, OCH_2), 5.94 (s, 1H, =C*H*), 6.81–6.89 (m, 4H, Ph); MS *m*/*z* 330 (M$^+$); Anal. calcd for $C_{19}H_{22}O_3S$: C, 69.06; H, 6.71; found C, 68.94; H, 6.53%.

1.1.5. 3-(4′-(4′-Methylphenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3e). Yield 88%; viscous liquid; λ_{max}: 221, 285 nm; ν_{max}: 1130, 1580, 1650, 2970 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.24 (s, 3H, Ph*Me*), 2.28 (s, 4H, CH_2CO, =CCH_2), 3.58–3.59 (t, J=1.9 Hz, 2H, SCH_2), 4.64–4.65 (t, J=1.9 Hz, 2H, OCH_2), 5.94 (s, 1H, =C*H*), 6.81–6.84 (d, J=12 Hz, 2H, Ph), 7.06–7.09 (d, J=12 Hz, 2H, Ph); MS *m*/*z* 314 (M$^+$); Anal. calcd for $C_{19}H_{22}O_2S$: C, 72.57; H, 7.05; found C, 72.39; H, 6.87%.

1.1.6. 3-(4′-(2′,4′-Dichlorophenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3f). Yield 85%; viscous liquid; λ_{max}: 227, 282 nm; ν_{max}: 1140, 1580, 1650, 2980 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 3H, *Me*), 1.07 (s, 3H, *Me*), 2.25 (s, 2H, CH_2CO), 2.33 (s, 2H, =CCH_2), 3.58–3.59 (t, J=1.9 Hz, 2H, SCH_2), 4.68–4.69 (t, J=1.9 Hz, 2H, OCH_2), 5.94 (s, 1H, =C*H*), 6.92–7.31 (m, 3H, Ph); MS *m*/*z* 368, 370, 372 (M$^+$); Anal. calcd for $C_{18}H_{18}Cl_2O_2S$: C, 58.54; H, 4.91; found C, 58.52; H, 4.71%.

1.1.7. 3-(4′-(2′-Methylphenoxy)-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (3g). Yield 86%; viscous liquid; λ_{max}: 220, 282 nm; ν_{max}: 1130, 1570, 1640, 2980 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.22 (s, 3H, Ph*Me*), 2.25 (s, 2H, CH_2CO), 2.33 (s, 2H, =CCH_2), 3.58 (brs, 2H, SCH_2), 4.70 (brs, 2H, OCH_2), 5.94 (s, 1H, =C*H*), 6.87–7.17 (m, 4H, Ph); MS *m*/*z* 314 (M$^+$); Anal. calcd for $C_{19}H_{22}O_2S$: C, 72.57; H, 7.05; found C, 72.49; H, 6.89%.

1.2. General procedure for the oxidation and subsequent rearrangement of 3-(4′-aryloxybut-2′-ynylthio)-5,5-dimethylcyclohex-2-enone (5a–g)

A solution of *m*-CPBA [5 mmol, 1.91 g (45%)] in chloroform (100 mL) was slowly added to a well stirred solution of the sulfide (**3a–g**) (5 mmol) in chloroform (50 mL) at 0–5°C over a period of 10 min. The reaction mixture was stirred at room temperature for an additional 6–8 h before being washed with 10% aqueous Na_2CO_3 (3×25 mL) and dried (Na_2SO_4). Removal of the solvent gave a crude mass which was purified by column chromatography over silica gel. Elution of the column by 70% benzene–petroleum ether gave compounds (**5a–g**).

1.2.1. 2-Phenoxymethyl-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones (5a). Yield 85%; viscous liquid; λ_{max}: 219, 275 nm; ν_{max}: 1120, 1590, 1610, 1680, 2920 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.30 (s, 2H, CH_2CO), 2.52 (s, 2H, =CCH_2), 3.99 (brs, 2H, CH_2OAr), 4.70 (brs, 2H, CH_2–OCO), 6.70–7.33 (m, 9H, Ph); MS *m*/*z* 454, 456 (M$^+$); Anal. calcd for $C_{25}H_{23}ClO_4S$: C, 66.00; H, 5.09; found C, 66.21; H, 5.12%.

1.2.2. 2-(2′-Chlorophenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones (5b). Yield 70%; viscous liquid; λ_{max}: 218, 273 nm; ν_{max}: 1120, 1580, 1620, 1690, 2920 cm^{-1}; ^{1}H NMR ($CDCl_3$,

300 MHz): δ 1.05 (s, 6H, 2*Me*), 2.30 (s, 2H, CH_2CO), 2.52 (s, 2H, =CCH_2), 4.00 (brs, 2H, CH_2OAr), 4.75 (brs, 2H, CH_2–OC=O), 6.67–7.39 (m, 8H, Ph); MS *m*/*z* 488, 490, 492 (M^+); Anal. calcd for $C_{25}H_{22}Cl_2O_4S$: C, 61.36; H, 4.53; found C, 61.23; H, 4.39%.

1.2.3. 2-(4′-Chlorophenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones (5c). Yield 71%; viscous liquid; λ_{max}: 226, 274 nm; ν_{max}: 1130, 1590, 1610, 1690, 2930 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.06 (s, 6H, 2*Me*), 2.31 (s, 2H, CH_2CO), 2.51 (s, 2H, =CCH_2), 4.98–3.99 (t, *J*=2 Hz, 2H, CH_2OAr), 4.67–4.68 (t, *J*=2 Hz, 2H, CH_2–OC=O), 6.81–6.86 (m, 4H, Ph), 7.23–7.27 (m, 4H, Ph); MS *m*/*z* 488, 490, 492 (M^+); Anal. calcd for $C_{25}H_{22}Cl_2O_4S$: C, 61.36; H, 4.53; found C, 61.31; H, 4.31%.

1.2.4. 2-(4′-Methoxyphenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones (5d). Yield 72%; viscous liquid; λ_{max}: 219, 273 nm; ν_{max}: 1130, 1580, 1620, 1690, 2960 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.06 (s, 6H, 2*Me*), 2.26 (s, 2H, CH_2CO), 2.52 (s, 2H, =CCH_2), 3.77 (s, 3H, –OMe), 3.99–4.00 (t, *J*=2 Hz, 2H, CH_2OAr), 4.63–4.65 (t, *J*=2 Hz, 2H, CH_2–OC=O), 6.69–7.25 (m, 8H, Ph); MS *m*/*z* 484, 486 (M^+); Anal. calcd for $C_{26}H_{25}ClO_5S$: C, 64.38; H, 5.18; found C, 64.31; H, 4.98%.

1.2.5. 2-(4′-Methylphenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-one (5e). Yield 70%; viscous liquid; λ_{max}: 222, 275 nm; ν_{max}: 1120, 1560, 1610, 1690, 2960 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ_H 1.05 (s, 6H, 2*Me*), 2.24 (s, 2H, CH_2CO), 2.29 (s, 3H, Ph*Me*), 2.52 (s, 2H, =CCH_2), 3.99 (brs, 2H, CH_2OAr), 4.70 (brs, 2H, CH_2–OC=O), 6.69–7.98 (m, 8H, Ph); ^{13}C NMR ($CDCl_3$, 300 MHz): δ_c 20.85, 28.14, 28.21, 34.705, 38.66, 45.65, 51.32, 56.12, 74.13, 84.53, 99.08, 99.65, 114.94, 115.26, 128.69, 130.27, 130.46, 130.53, 130.50, 131.50, 131.59, 133.23, 134.16, 154.52, 170.50 (ester carbonyl) 197.96 (keto carbonyl); MS *m*/*z* 468, 470 (M^+); Anal. calcd for $C_{26}H_{25}ClO_4S$: C, 66.58; H, 5.37; found C, 66.49; H, 5.22%.

1.2.6. 2-(2′,4′-Dichlorophenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones (5f). Yield 80%; mp 110°C; λ_{max}: 221, 285 nm; ν_{max}: 1130, 1570, 1600, 1690, 2940 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.07 (s, 6H, 2*Me*), 2.31 (s, 2H, CH_2CO), 2.57 (s, 2H, =CCH_2), 3.99 (brs, 2H, CH_2OAr), 4.77 (brs, 2H, CH_2–OCO), 6.68–7.38 (m, 7H, Ph); MS *m*/*z* 522, 524, 526 (M^+); Anal. calcd for $C_{25}H_{21}Cl_3O_4S$: C, 57.31; H, 4.04; found C, 57.52; H, 3.89%.

1.2.7. 2-(2′-Methylphenoxy)-3-(3′-chlorobenzoyloxymethyl)-6,6-dimethyl-5,6,7-trihydrobenzo(*b*)thiophene-4-ones(5g). Yield 78%; viscous liquid; λ_{max}: 222, 287 nm; ν_{max}: 1120, 1570, 1610, 1680, 2950 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 1.06 (s, 6H, 2*Me*), 2.28 (s, 2H, CH_2CO), 2.29 (s, 3H, Ph*Me*), 2.52 (s, 2H, =CCH_2), 3.92 (brs, 2H, CH_2OAr), 4.65 (brs, 2H, CH_2–OC=O), 6.69–7.58 (m, 8H, Ph); MS *m*/*z* 468, 470 (M^+); Anal. calcd for $C_{26}H_{25}ClO_4S$: C, 66.58; H, 5.37; found C, 66.43; H, 5.23%.

Acknowledgements

We are thankful to the CSIR (New Delhi) for financial assistance. One of us (S. K. S.) is grateful to the CSIR (New Delhi) for a fellowship.

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