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A New Method for the Preparation of α-Methylene-γ-butyrolactones Using 3-Ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone

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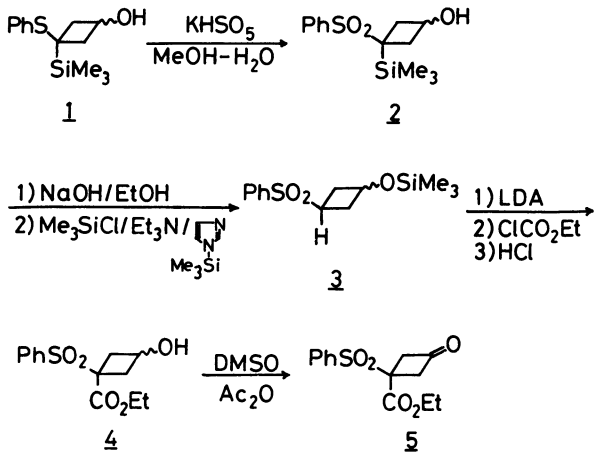
The reaction of 3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone with Grignard reagent or aryllithium gave 1-(alkyl or aryl)-3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanol which was treated with potassium hydride, reduced with $LiBH_4$ in the presence of $ZnCl_2$, and treated with *p*-TsOH or aluminum triisopropoxide successively to give α-methylene-γ-butyrolactone in good yield.

In recent years, much attention has been paid on the synthesis of the compounds possessing α-methylene-γ-butyrolactone moiety because of their wide range of biological activity and various methods have been developed for the construction of such a structural unit.[1)]

Previously, we showed that β-methylene ketones were obtained by the base-promoted ring-opening reaction of 1,3-dialkyl-3-(phenylsulfonyl)cyclobutanols.[2)] Based on the results of above study, we investigated a new synthetic route to α-methylene-γ-butyrolactone (**9**) using 3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone (**5**) as a starting material as shown in the following scheme. The cyclobutanone (**5**) was synthesized from 3-(phenylthio)-3-(trimethylsilyl)cyclobutanol (**1**) which was easily prepared by the reaction of epichlorhydrin with α,α-bis(trimethylsilyl)phenylthiomethyllithium.[3)] The cyclobutane (**1**) was oxidized with potassium peroxymonosulfate (oxone) to give the corresponding sulfone (**2**). Hydrolysis of the trimethylsilyl group and trimethylsilylation of the hydroxy group gave **3**. The trimethylsilyl ether (**3**) was treated with lithium diisopropylamide and ethyl chloroformate successively to give the hydroxy ester (**4**) which was then oxidized with dimethyl sulfoxide–acetic anhydride[4)] to afford the cyclobutanone (**5**).

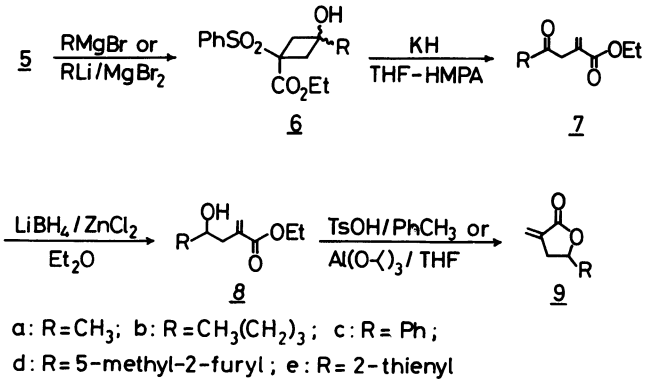


Scheme 1.

3-Ethoxycarbonyl-1-phenyl-3-(phenylsulfonyl)cyclobutanol (**6c**) was obtained in good yield by the reaction of the cyclobutanone (**5**) with phenylmagnesium bromide in THF at 0 °C. Then **6c** was treated with 4 equimolar amounts of KH in THF in the presence of HMPA to give γ-oxo-α-methylene ester (**7c**) along with a trace amount of the isomeric α,β-unsaturated ester. The selective reduction of **7c** was performed with $LiBH_4$ in ether at 0 °C in the presence of a catalytic amount of $ZnCl_2$ and a mixture of γ-hydroxy-α-methylene ester (**8c**) and α-methylene-γ-phenyl-γ-butyrolactone (**9c**) was obtained. The treatment of the mixture with *p*-TsOH in toluene at room temperature gave **9c** in good yield.

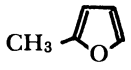
In a similar manner, several α-methylene-γ-butyrolactones (**9**) were synthesized using Grignard and aryllithium reagents and the results were summarized in Table 1. Since the treatment of **8d** and **8e** with *p*-TsOH gave complex mixtures, the lactonization of these compounds was carried out with an equimolar amount of aluminum triisopropoxide in refluxing THF.

The most coventional methods for the preparation of γ-substituted α-methylene-γ-butyrolactone (**9**) consist of the reaction of aldehyde with various carbanions which have an ester function or its equivalent. It is noted that the γ-substituent of **9** is introduced using carbanion species, which is the distinctive feature of the present method.



Scheme 2.

Table 1. Preparation of α-Methylene-γ-butyrolactones (**9**)

	R	Products (yield/%)		
		6	**7**	**9**
a	CH_3	62	67	65
b	$CH_3(CH_2)_3$	51	71	85
c	Ph	81	85	83
d		84	78	83
e		70	73	74

Experimental

Preparation of 3-(Phenylsulfonyl)-3-(trimethylsilyl)cyclobutanol (2). To a MeOH (4 ml) solution of 3-(phenylthio)-3-(trimethylsilyl)cyclobutanol (**1**)[3] (252 mg, 1 mmol) was added an aqueous solution (4 ml) of 49.5% $KHSO_5$ (922 mg, 3 mmol) under cooling with ice. After stirring for 1.5 h at room temperature, the reaction mixture was diluted with water. The organic material was extracted with CH_2Cl_2. The extract was dried over Na_2SO_4 and condensed under reduced pressure. The residue was chromatographed on silica gel (AcOEt–hexane) and 3-(phenylsulfonyl)-3-(trimethylsilyl)cyclobutanol (**2**) (265 mg) was isolated in 93% yield.

2: mp 113—115 °C (hexane–benzene); IR (KBr) 3500, 2990, 2940, 1453, 1300, 1256, 1141, 1120, 1090, 850, and 748 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.02 and 0.19 (2s, 9H), 1.98—3.11 (m, 5H), 3.34—4.25 (m, 1H), and 7.21—8.02 (m, 5H). Found: C, 54.99; H, 7.14; S, 11.24%. Calcd for $C_{13}H_{20}O_3SSi$: C, 54.89; H, 7.09; S, 11.27%.

Preparation of 1-(Phenylsulfonyl)-3-(trimethylsiloxy)cyclobutane (3). To a EtOH (200 ml) solution of the cyclobutanol (**2**) (14.22 g, 50 mmol) was added pellet NaOH (1.0 g, 25 mmol). After stirring for 1 h at room temperature, the reaction mixture was diluted with water and neutralized with 1 M hydrochloric acid (1 M=1 mol dm^{-3}). The organic material was extracted with AcOEt and the extract was dried over Na_2SO_4. The extract was condensed under reduced pressure to give crude 3-(phenylsulfonyl)cyclobutanol. A THF (80 ml) solution of the crude cyclobutanol was slowly added to a mixture of triethylamine (7.7 ml, 55 mmol), chlorotrimethylsilane (7.0 ml, 55 mmol), and *N*-(trimethylsilyl)imidazole (0.29 ml, 2 mmol) in THF (50 ml) under cooling with ice. The reaction mixture was stirred overnight at room temperature. The reaction was quenched by addition of saturated aqueous solution of $NaHCO_3$ and the organic material was extracted with AcOEt. The extract was dried (Na_2SO_4) and condensed in vacuo. The residue was purified by column chromatography on silica gel (AcOEt–hexane) to give 1-(phenylsulfonyl)-3-(trimethylsiloxy)cyclobutane (**3**) (12.84 g) in 90% yield.

3: oil; IR (neat) 3080, 2970, 1453, 1312, 1256, 1155, 1132, 1094, 993, 907, 850, 736, and 698 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.08 (s, 9H), 2.00—4.81 (m, 6H), and 7.33—7.98 (m, 5H). Found: C, 54.72; H, 7.10; S, 11.23%. Calcd for $C_{13}H_{20}O_3SSi$: C, 54.89; H, 7.09; S, 11.27%.

Preparation of 3-Ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanol (4). To a THF (45 ml) solution of lithium diisopropylamide prepared from diisopropylamine (4.912 g, 48.5 mmol) and butyllithium (46.3 mmol) was slowly added a THF (60 ml) solution of the (trimethylsiloxy)cyclobutane (**3**) (12.55 g, 44.1 mmol) under cooling with ice. After stirring for 40 min, the reaction mixture was cooled to −78 °C. A THF solution (20 ml) of ethyl chloroformate (4.6 ml, 48.6 mmol) was added to the reaction mixture and stirred for 2 h at the same temperature. Then 2 M hydrochloric acid (50 ml) was added under cooling with ice and the mixture was stirred for 15 min. The organic material was extracted with AcOEt and the extract was dried over Na_2SO_4. After evaporation of the solvent, the residue was chromatographed on silica gel (AcOEt–hexane) to give 3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanol (**4**) (10.96 g) in 87% yield.

4: viscous oil; IR (neat) 3510, 3060, 2960, 1727, 1450, 1308, 1290, 1151, 1085, 860, 730, and 695 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.11 (t, J=7 Hz, 3H), 2.33—3.40 (m, 4H), 3.40—3.82 (m, 1H), 4.03 (q, J=7 Hz, 2H), 4.25 —4.85 (m, 1H), and 7.30—8.07 (m, 5H). MS (FAB) m/z 285 (M^++H).

Preparation of 3-Ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone (5). To a dimethyl sulfoxide (31 ml) solution of the cyclobutanol (**4**) (2.92 g, 10.3 mmol) was added acetic anhydride (20.5 ml) and the mixture was stirred for 17 h at room temperature. Then it was diluted with a phosphate buffer solution (pH 7) under cooling with ice to form brownish precipitate. The precipitate was collected and dried in vacuo. The crude material was recrystallized from hexane–benzene to give pure 3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone (**5**) (1.91 g) in 68% yield.

5: mp 134—135 °C (hexane–benzene); IR (KBr) 3080, 2990, 2950, 1801, 1730, 1452, 1380, 1314, 1288, 1152, 1089, 765, 734, and 700 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.20 (t, J=7 Hz, 3H), 3.10—4.25 (m, 4H), 4.16 (q, J=7 Hz, 2H), and 7.27—8.00 (m, 5H). Found: C, 55.34; H, 4.98; S, 11.35%. Calcd for $C_{13}H_{14}O_5S$: C, 55.31; H, 5.00; S, 11.35%.

Reaction of 3-Ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone (5) with Phenylmagnesium Bromide. To a THF (0.5 ml) solution of phenylmagnesium bromide (0.55 mmol) was slowly added a THF (2 ml) solution of 3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanone (**5**) (141 mg, 0.5 mmol) under cooling with ice. After stirring for 10 min at the same temperature, the reaction was quenched by addition of saturated aqueous solution of NH_4Cl and the organic material was extracted with AcOEt. The extract was dried over Na_2SO_4 and condensed under reduced pressure. The residual crude crystal was purified by recrystallization from hexane–benzene and 3-ethoxycarbonyl-1-phenyl-3-(phenylsulfonyl)cyclobutanol (**6c**) (146 mg) was obtained in 81% yield.

6c: mp 141—142 °C (hexane–benzene); IR (KBr) 3480, 3050, 3020, 2980, 1714, 1445, 1368, 1301, 1140, 1086, 1021, 916, 870, 853, 702, and 669 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.98 and 1.10 (2t, J=7 and 6 Hz, 3H), 2.56—3.60 (m, 5H), 3.91 and 4.06 (2q, J=7 and 6 Hz, 2H), and 6.93—7.90 (m, 10H). Found: C, 63.31; H, 5.55; S, 8.88%. Calcd for $C_{19}H_{20}O_5S$: C, 63.31; H, 5.59; S, 8.90%.

By a similar manner, the cyclobutanol (**6a**) was obtained. The cyclobutanols (**6b**, **6d**, and **6e**) were prepared by the reaction of **5** with butyllithium, 5-methyl-2-furyllithium,

and 2-thienyllithium in the presence of an equimolar amount of $MgBr_2$, respectively. These compounds were isolated by TLC (AcOEt–hexane).

All the cyclobutanols (**6**) were obtained as a mixture of diastereomers and the ratio of isomers of these compounds was not determined.

3-Ethoxycarbonyl-1-methyl-3-(phenylsulfonyl)cyclobutanol (6a): viscous oil; IR (neat) 3500, 3060, 2970, 1724, 1447, 1308, 1290, 1174, 1140, 1085, 955, 725, and 690 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.10 (t, J=7 Hz, 3H), 1.29 (s, 3H), 2.42—3.28 (m, 4H) 3.28—3.80 (br s, 1H), 4.06 (q, J=7 Hz, 2H), and 7.28—7.95 (m, 5H). MS (FAB) m/z 299 (M^++H). **1-Butyl-3-ethoxycarbonyl-3-(phenylsulfonyl)cyclobutanol (6b):** viscous oil; IR (neat) 3500, 3060, 2950, 2870, 1723, 1446, 1307, 1141, 1085, 1024, 932, 850, 724, and 692 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.09 (t, J=7 Hz, 3H), 0.62—1.93 (m, 9H), 2.23—3.20 (m, 4H), 3.33—3.73 (br s, 1H), 4.03 (q, J=7 Hz, 2H), and 7.27—7.93 (m, 5H). MS (FD) m/z 341 (M^++H). **3-Ethoxycarbonyl-1-(5-methyl-2-furyl)-3-(phenylsulfonyl)cyclobutanol (6d):** mp 95—96 °C (hexane–benzene); IR (KBr) 3500, 3080, 3070, 2980, 2960, 1710, 1447, 1310, 1145, 1086, 1024, 955, 858, 802, 766, 727, and 696 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.04 (t, J=7 Hz, 3H), 2.21 (s, 3H), 3.20 (s, 4H), 3.44 (br s, 1H), 3.96 (q, J=7 Hz, 2H), 5.73—5.90 (m, 1H), 6.08 (d, J=3 Hz, 1H), and 7.30—7.98 (m, 5H). Found: C, 59.34; H, 5.52; S, 8.77%. Calcd for $C_{18}H_{20}O_6S$: C, 59.33; H, 5.53; S, 8.80%. **3-Ethoxycarbonyl-3-(phenylsulfonyl)-1-(2-thienyl)-cyclobutanol (6e):** mp 110—111 °C (hexane–benzene): IR (KBr) 3490, 3070, 2990, 1720, 1290, 1156, 1141, 1086, 729, and 703 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.00 (t, J=7 Hz, 3H), 3.27 (br s, 4H), 3.91 (q, J=7 Hz, 2H), 3.67—4.23 (m, 1H), 6.68—6.93 (m, 2H), 7.02—7.23 (m, 1H), and 7.37—7.92 (m, 5H). Found: C, 55.82; H, 4.95; S, 17.30%. Calcd for $C_{17}H_{18}O_5S_2$: C, 55.72; H, 4.95; S, 17.50%.

Preparation of Ethyl 2-Methylene-4-oxo-4-phenylbutyrate (7c). To a suspension of KH (144 mg, 3.6 mmol) in THF (4 ml)–HMPA (3 ml) was added a THF (6 ml) solution of 3-ethoxycarbonyl-1-phenyl-3-(phenylsulfonyl)cyclobutanol (**6c**) (325 mg, 0.9 mmol) at −23 °C. After being stirred for 50 min at the same temperature, the reaction was quenched by addition of a phosphate buffer solution (pH 7). The organic material was extracted with ether and the extract was dried (Na_2SO_4). After removal of the solvent, the residue was chromatographed on silica gel (AcOEt–hexane) and **7c** (168 mg) was isolated in 85% yield. **7c:** viscous oil; IR (neat) 3060, 2980, 1708, 1686, 1632, 1321, 1303, 1199, 1146, 1026, 952, 758, and 690 cm^{-1}; ^1H NMR (CCl_4) δ=1.23 (t, J=7 Hz, 3H), 3.82 (s, 2H), 4.06 (q, J=7 Hz, 2H), 5.49 (br s, 1H), 6.17 (br s, 1H), 7.13—7.53 (m, 3H), and 7.58—7.97 (m, 2H). Found: C, 71.22; H, 6.47%. Calcd for $C_{13}H_{14}O_3$: C, 71.54; H, 6.47%.

In the same procedure, α-methylene-γ-oxo esters (**7a**, **7b**, **7d**, and **7e**) were synthesized. **Ethyl 2-methylene-4-oxovalerate (7a):** viscous oil; IR (neat) 2980, 2950, 2920, 1715, 1635, 1335, 1308, 1205, 1148, 1028, and 950 cm^{-1}; ^1H NMR (CCl_4) δ=1.26 (t, J=7 Hz, 3H), 2.09 (s, 3H), 3.25 (br s, 2H), 4.13 (q, J=7 Hz, 2H), 5.51 (br s, 1H), and 6.18 (br s, 1H). MS m/z 156 (M^+). **Ethyl 2-methylene-4-oxooctanoate (7b):** viscous oil; IR (neat) 2960, 2940, 2880, 1740, 1720, 1633, 1333, 1305, 1190, 1151, 1030, and 954 cm^{-1}; ^1H NMR (CCl_4) δ=1.27 (t, J=7 Hz, 3H), 0.65—1.80 (m, 7H), 2.37 (br t, J=6 Hz, 2H), 3.21 (br s, 2H), 4.08 (q, J=7 Hz, 2H), 5.45 (br s, 1H), and 6.11 (br s, 1H). Found: C, 66.25; H, 9.22%. Calcd for $C_{11}H_{18}O_3$: C, 66.64; H, 9.15%. **Ethyl 2-Methylene-4-(5-methyl-2-furyl)-4-oxobutyrate (7d):** viscous oil; IR (neat) 3110, 2980, 1720, 1707, 1671, 1637, 1515, 1317, 1202, 1147, 1062, 1030, 956, and 804 cm^{-1}; ^1H NMR (CCl_4) δ=1.21 (t, J=7 Hz, 3H), 2.33 (s, 3H), 3.64 (s, 2H), 4.09 (q, J=7 Hz, 2H), 5.54 (br s, 1H), 6.03 (br d, J=3 Hz, 1H), 6.18 (br s, 1H), and 6.93 (d, J=3 Hz, 1H). MS (FAB) m/z 223 (M^++H). **Ethyl 2-methylene-4-oxo-4-(2-thienyl)butyrate (7e):** viscous oil; IR (neat) 3090, 2970, 1720, 1706, 1658, 1519, 1411, 1313, 1200, 1147, 1024, 951, 855, 818, and 728 cm^{-1}; ^1H NMR (CCl_4) δ=1.20 (t, J=7 Hz, 3H), 3.80 (s, 2H), 4.08 (q, J=7 Hz, 2H), 5.59 (br s, 1H), 6.21 (br s, 1H), 6.97 (dd, J=5 and 4 Hz, 1H), 7.50 (dd, J=5 and 1 Hz, 1H), and 7.62 (dd, J=4 and 1 Hz, 1H). MS (FAB) m/z 225 (M^++H).

Preparation of 4,5-Dihydro-3-methylene-5-phenyl-2(3*H*)-furanone (9c). The ethereal solution (1.8 ml) of $LiBH_4$ (1.1 mmol) was added to a suspension of $ZnCl_2$ (41 mg, 0.3 mmol) in ether (2 ml) under cooling with ice. An ether (2 ml) solution of **7c** (218 mg, 1 mmol) was slowly added to a resulting white suspension and the reaction mixture was stirred for 15 min under cooling with ice. The reaction was quenched by addition of 2 M hydrochloric acid and the organic material was extracted with ether. The extract was washed with water and dried over Na_2SO_4. After evaporation of the solvent, the residue was dissolved in toluene (4 ml) and *p*-toluenesulfonic acid (75 mg, 0.44 mmol) was added at room temperature. After being stirred for 2 h, the reaction mixture was diluted with ether (10 ml) and the organic layer was washed with water and brine, dried (Na_2SO_4), and condensed under reduced pressure. The residue was chromatographed on silica gel (AcOEt–hexane) and **9c** (138 mg) was obtained in 83% yield.

9c: mp 50—51 °C (lit,[1a] 48—49.5 °C); IR (neat) 3020, 2960, 1760, 1704, 1664, 1320, 1276, 1130, 1026, 816, 758, and 701 cm^{-1}; ^1H NMR ($CDCl_3$) δ=2.76 (ddt, J=17, 7, and 2.5 Hz, 1H), 3.39 (ddt, J=17, 8, and 2.5 Hz, 1H), 5.46 (dd, J=7 and 8 Hz, 1H), 5.63 (t, J=2.5 Hz, 1H), 6.25 (t, J=2.5 Hz, 1H), and 7.32 (s, 5H).

In a similar manner, α-methylene-γ-butyrolactones (**9a** and **9b**) were synthesized. **9d** and **9e** were prepared by the treatment of the crude reduction product (**8**) with an equimolar amount of aluminum triisopropoxide in refluxing THF (10 ml per 1 mmol of **8**) for 2 h.

4,5-Dihydro-3-methylene-5-methyl-2(3*H*)-furanone (9a): oil; IR (neat) 2980, 2930, 1760, 1665, 1440, 1400, 1340. 1280, 1261, 1116, 1042, 957, and 818 cm^{-1}; ^1H NMR (CCl_4) δ=1.42 (d, J=6 Hz, 3H), 2.47 (ddt, J=17, 6, and 3 Hz, 1H), 3.10 (ddt, J=17, 7, and 3 Hz, 1H), 4.58 (d quint, J=7 and 6 Hz, 1H), 5.55 (t, J=3 Hz, 1H), and 6.04 (t, J=3 Hz, 1H). Found: C, 63.98; H, 7.37%. Calcd for $C_6H_8O_2$: C, 64.27; H, 7.19%. **5-Butyl-4,5-dihydro-3-methylene-2(3*H*)-furanone (9b):** oil; IR (neat) 2960, 2930, 2870, 1760, 1665, 1399, 1350, 1280, 1118, 1007, 933, and 818 cm^{-1}; ^1H NMR (CCl_4) δ=0.60—1.92 (m, 9H), 2.47 (ddt, J=17, 6, and 2.5 Hz, 1H), 3.04 (ddt, J=17, 7, and 2.5 Hz, 1H), 4.01—4.63 (m, 1H), 5.48 (t, J=2.5 Hz, 1H), and 6.00 (t, J=2.5 Hz, 1H). Found: C, 69.55; H, 9.29%. Calcd for $C_9H_{14}O_2$: C, 70.10; H, 9.15%. **3,5-Dihydro-5-(5-methyl-2-furyl)-3-methylene-2(3*H*)-furanone (9d):** viscous oil; IR (neat) 3120, 3100, 2920, 1760, 1663, 1512, 1438, 1398, 1317, 1280, 1259, 1128, 1030, 966, 906, and 801 cm^{-1}; ^1H NMR (CCl_4) δ=2.17 (s, 3H), 3.01 (dt, J=7 and 2.5 Hz, 2H), 5.18 (t,

J=7 Hz, 1H), 5.45 (t, J=2.5 Hz, 1H), 5.75 (br s, 1H), 5.99 (t, J=2.5 Hz, 1H), and 6.03 (d, J=3 Hz, 1H). Found: C, 66.95; H, 5.73%. Calcd for $C_{10}H_{10}O_3$: C, 67.40; H, 5.66%. MS (FD) m/z 178 (M^+). **3,5-Dihydro-3-methylene-5-(2-thienyl)-2(3*H*)-furanone (9e):** viscous oil; IR (neat) 3100, 2970, 2930, 1759, 1664, 1435, 1277, 1251, 1125, 1020, 854, 815, and 710 cm^{-1}; ^1H NMR (CCl_4) δ=2.48—3.60 (m, 2H), 5.33—5.73 (m, 2H), 6.03 (t, J=3 Hz, 1H), and 6.67—7.28 (m, 3H). Found: C, 59.76; H, 4.60; S, 17.52%. Calcd for $C_9H_8O_2S$: C, 59.98; H, 4.47; S, 17.79%.

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