

Supporting Information

DABCO Catalyzed Addition of Selenosulfonates to α,β -Unsaturated Ketones

Yong-Ling Shi and Min Shi*

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic
Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032 China,

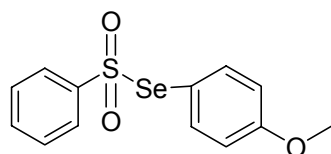
mshi@pub.sioc.ac.cn

Experimental Procedures

General Remarks. Unless otherwise stated, all reactions were carried out under argon atmosphere. All solvents were purified by distillation. Other commercially obtained reagents were used without further purification. Selenosulfonates¹, phenyl vinyl ketone² and 4-methylpent-1-en-3-one³ were prepared according to the literature. Infrared spectra were measured on a PERKIN-ELMER 983 spectrometer. ¹H-NMR spectra were recorded on a 300 MHz spectrometer in CDCl₃ using tetramethylsilane as the internal standard. Mass spectra were recorded with an HP-5989 instrument and HRMS was measured by a Finnigan MA+ mass spectrometer or an Ion Spec 4.7 Tesla FTMS mass spectrometer. Satisfactory CHN microanalyses were obtained with a Carlo-Erba 1106 analyzer. Melting points were obtained by means of a micro melting point apparatus and are uncorrected.

Typical Procedure for the Preparation of Selenosulfonates.

A suspension of PhSO₂Na·2H₂O (800 mg, 4.0 mmol) in CH₂Cl₂ (10 mL) containing 1,2-bis(4-methoxyphenyl)diselenide (372 mg, 1.0 mmol) was cooled at 0 °C and [bis(trifluoroacetoxy)iodo]benzene (475 mg, 1.1 mmol) in CH₂Cl₂ (4 mL) was added dropwise. The mixture was stirred at room temperature for 3 h. Then, the reaction mixture was washed with H₂O, dried over anhydrous MgSO₄. The solvent CH₂Cl₂ was removed under reduced pressure and the residue was purified by a flash chromatography (SiO₂, EtOAc:Petroleum ether = 1:6) to yield **3e** (479 mg, 63%) as a yellow solid.

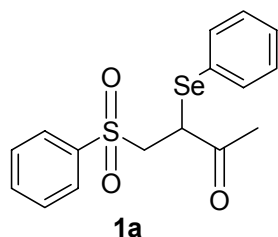


Se-4-methoxyphenyl benzeneselenosulfonate

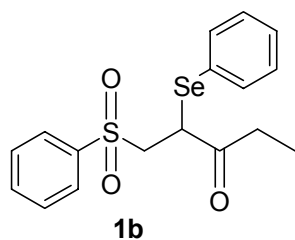
a yellow solid: mp. 62-64 °C; IR (CH₂Cl₂) ν 3060, 3012, 1582, 1567, 1491, 1302, 1253, 1183, 1129, 1072 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 3.84 (3H, s, Me), 6.84 (2H, d, *J* = 9.0 Hz, Me), 7.37-7.44 (4H, m, Ar), 7.51-7.59 (3H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 55.4, 115.1, 118.7, 127.0, 128.7, 133.4, 139.0, 145.1, 161.9. MS (EI) *m/e* 328 (M⁺, 19.3), 187 (M⁺-141, 100). Anal. Calcd. for C₁₃H₁₂O₃SSe requires C, 47.71; H, 3.70%. Found: C, 47.71; H, 3.81%.

Typical Reaction Procedure for DABCO-catalyzed Reaction of Methyl vinyl ketone with Se-phenyl benzeneselenosulfonate.

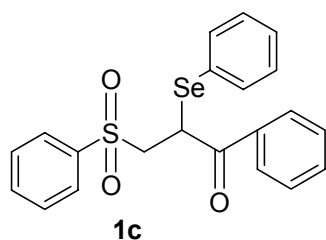
To a solution of Se-phenyl benzeneselenosulfonate (74 mg, 0.25 mmol) and DABCO (9 mg, 0.06 mmol) in THF (1.0 mL) at room temperature was added methyl vinyl ketone **2a** (42 μ L, 0.5 mmol) and the reaction mixture was further stirred at room temperature. The reaction was monitored by TLC plate. When the Se-phenyl benzeneselenosulfonate disappeared, the solvent was removed under reduced pressure and the residue was purified by a flash chromatography (SiO₂, EtOAc:Petroleum ether = 1:6) to yield **1a** (74 mg, 81 %) as a white solid.



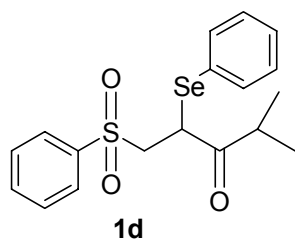
3-(phenylselanyl)-4-(phenylsulfonyl)butan-2-one (1a): a white solid: mp. 103-105 °C; IR (CHCl₃) ν 2978, 1706 (C=O), 1446, 1306, 1170, 1145, 1130 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 2.34 (3H, s, Me), 3.48 (1H, dd, *J* = 13.8, 2.7 Hz), 3.93 (1H, dd, *J* = 13.8, 10.5 Hz), 4.17 (1H, dd, *J* = 10.5, 2.7 Hz), 7.27-7.31 (2H, m, Ar), 7.36-7.44 (3H, m, Ar), 7.51-7.56 (2H, m, Ar), 7.63-7.68 (1H, m, Ar), 7.81-7.84 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 27.9, 41.7, 57.4, 125.1, 128.0, 129.3, 129.5, 129.7, 134.0, 136.1, 139.0, 199.7. MS (EI) *m/e* 368 (M⁺, 13.5), 77 (M⁺-291, 100). Anal. Calcd. for C₁₆H₁₆O₃SSe requires C, 52.32; H, 4.39%. Found: C, 52.30; H, 4.36%.



2-(phenylselanyl)-1-(phenylsulfonyl)pentan-3-one (1b): a colorless liquid: IR (CHCl₃) ν 2978, 2937, 1709 (C=O), 1447, 1307, 1144, 1085 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.05 (3H, t, J = 7.2 Hz, CH₃), 2.48 (1H, dq, J = 18.0, 7.2 Hz), 2.89 (1H, dq, J = 18.0, 7.2 Hz), 3.48 (1H, dd, J = 14.1, 2.4 Hz), 3.99 (1H, dd, J = 14.1, 10.8 Hz), 4.17 (1H, dd, J = 10.8, 2.4 Hz), 7.26-7.31 (2H, m, Ar), 7.36-7.43 (3H, m, Ar), 7.50-7.56 (2H, m, Ar), 7.62-7.67 (1H, m, Ar), 7.80-7.83 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 8.0, 33.9, 40.8, 57.5, 125.3, 128.0, 129.2, 129.4, 129.6, 133.9, 136.1, 139.0, 202.8. MS (EI) m/e 382 (M⁺, 2.0), 184 (M⁺-198, 100). HRMS Calcd. for C₁₇H₁₈O₃SSeNa⁺ requires 405.0034, Found: 405.0029.

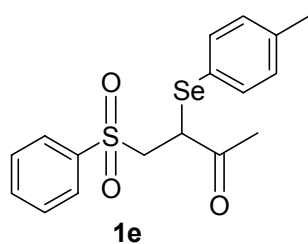


1-phenyl-2-(phenylselanyl)-3-(phenylsulfonyl)propan-1-one (1c): a colorless liquid: IR (CH₂Cl₂) ν 3059, 1675 (C=O), 1447, 1307, 1258, 1226, 1149 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 3.68 (1H, dd, J = 13.8, 2.4 Hz), 4.21 (1H, dd, J = 13.8, 10.5 Hz), 5.05 (1H, dd, J = 10.5, 2.4 Hz), 7.24-7.46 (9H, m, Ar), 7.52-7.60 (2H, m, Ar), 7.74-7.83 (4H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 36.5, 58.2, 125.5, 128.2, 128.4, 128.6, 129.2, 129.4, 129.8, 133.4, 133.8, 134.8, 136.5, 138.8, 191.7. MS (EI) m/e 430 (M⁺, 0.57), 105 (M⁺-325, 100). HRMS Calcd. for C₂₁H₁₈O₃SSeNa⁺ requires 453.0034, Found: 453.0029.

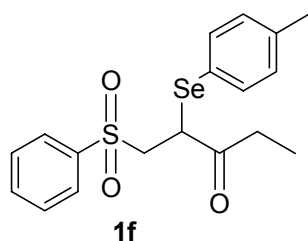


4-methyl-2-(phenylselanyl)-1-(phenylsulfonyl)pentan-3-one (1d): a colorless liquid: IR

(CH₂Cl₂) ν 2971, 2931, 1705 (C=O), 1447, 1307, 1144, 1086 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.13 (3H, d, J = 7.2 Hz, Me), 1.19 (3H, d, J = 6.6 Hz, Me), 2.98-3.12 (1H, m, CH), 3.49 (1H, dd, J = 14.1, 2.4 Hz), 3.99 (1H, dd, J = 14.1, 10.8 Hz), 4.28 (1H, dd, J = 10.8, 2.4 Hz), 7.23-7.28 (2H, m, Ar), 7.32-7.39 (3H, m, Ar), 7.47-7.52 (2H, m, Ar), 7.58-7.63 (1H, m, Ar), 7.80-7.82 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 18.4, 19.4, 38.6, 39.5, 56.9, 124.9, 127.6, 129.1, 129.3, 129.4, 133.7, 135.9, 138.9, 205.0. MS (EI) m/e 255 (M⁺-141, 2.97), 71 (M⁺-325, 100). HRMS Calcd. for C₁₈H₂₀O₃SSeNa⁺ requires 419.0191, Found: 419.0203.

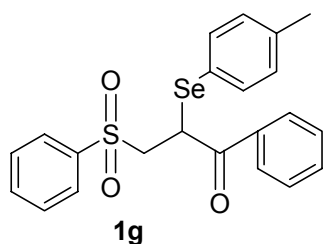


4-(phenylsulfonyl)-3-(p-tolylselanyl)butan-2-one (1e): a white solid: mp. 109-110 °C; IR (CH₂Cl₂) ν 2986, 2931, 1708 (C=O), 1446, 1306, 1144 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 2.32 (3H, s, Me), 2.33 (3H, s, Me), 3.47 (1H, dd, J = 14.1, 2.7 Hz), 3.89 (1H, dd, J = 14.1, 11.1 Hz), 4.12 (1H, dd, J = 11.1, 2.7 Hz), 7.09 (2H, d, J = 7.8 Hz, Ar), 7.29 (2H, d, J = 7.8 Hz, Ar), 7.50-7.56 (2H, m, Ar), 7.61-7.67 (1H, m, Ar), 7.81-7.85 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 21.2, 27.8, 41.6, 57.4, 121.3, 128.0, 129.3, 130.3, 133.9, 136.4, 139.0, 140.1, 199.6. MS (EI) m/e 382 (M⁺, 0.59), 43 (M⁺-339, 100). Anal. Calcd. for C₁₇H₁₈O₃SSe requires C, 53.54; H, 4.76%. Found: C, 53.36; H, 4.78%.

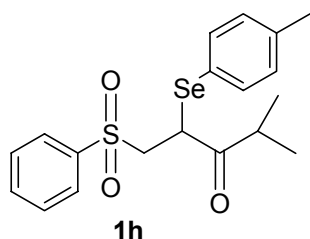


1-(phenylsulfonyl)-2-(p-tolylselanyl)pentan-3-one (1f): a colorless solid: mp. 76-78 °C; IR (CH₂Cl₂) ν 2977, 2937, 1709 (C=O), 1447, 1306, 1144 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.04 (3H, t, J = 7.2 Hz, CH₃), 2.33 (3H, s, Me), 2.46 (1H, dq, J = 18.0, 7.2 Hz), 2.90 (1H, dq, J = 18.0, 7.2 Hz), 3.47 (1H, dd, J = 13.5, 2.4 Hz), 3.94 (1H, dd, J = 13.5, 10.8 Hz), 4.10 (1H, dd, J = 10.8, 2.4 Hz), 7.08 (2H, d, J = 8.4 Hz, Ar), 7.27 (2H, d, J = 8.4 Hz, Ar), 7.49-7.54 (2H, m, Ar), 7.60-7.65 (1H, m, Ar), 7.79-7.82 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 8.0, 21.2, 33.7, 40.6, 57.4, 121.3, 127.9, 129.2, 130.2, 133.8, 136.4, 138.9, 139.9,

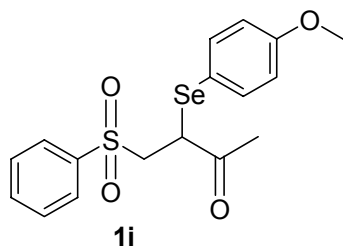
202.6. MS (EI) m/e 396 (M^+ , 24.1), 198 (M^+ -198, 100). Anal. Calcd. for $C_{18}H_{20}O_3SSe$ requires C, 54.68; H, 5.10%. Found: C, 54.51; H, 5.10%.



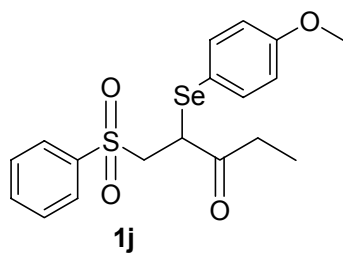
1-phenyl-3-(phenylsulfonyl)-2-(p-tolylselanyl)propan-1-one (1g): a colorless solid: mp. 100-102 °C; IR (CH_2Cl_2) ν 3059, 1675 (C=O), 1447, 1307, 1258, 1226, 1149 cm^{-1} ; 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 2.32 (3H, s, Me), 3.68 (1H, dd, $J = 14.1, 2.4$ Hz), 4.16 (1H, dd, $J = 14.1, 10.5$ Hz), 4.99 (1H, dd, $J = 10.5, 2.4$ Hz), 7.05 (2H, d, $J = 7.8$ Hz, Ar), 7.22 (2H, d, $J = 7.8$ Hz, Ar), 7.34-7.44 (4H, m, Ar), 7.49-7.57 (2H, m, Ar), 7.72-7.75 (2H, m, Ar), 7.80-7.83 (2H, m, Ar). ^{13}C NMR ($CDCl_3$, TMS, 75.44 MHz) δ 21.2, 36.2, 58.0, 121.5, 128.0, 128.3, 128.5, 129.0, 130.2, 133.2, 133.7, 134.6, 136.6, 138.6, 140.1, 191.4. MS (EI) m/e 444 (M^+ , 19.3), 105 (M^+ -339, 100). HRMS Calcd. for $C_{22}H_{20}O_3SSe^+$ requires 444.0293, Found: 444.0285.



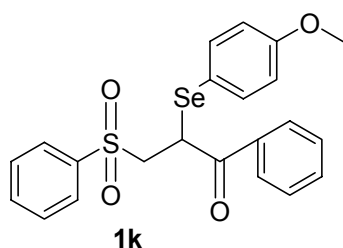
4-methyl-1-(phenylsulfonyl)-2-(p-tolylselanyl)pentan-3-one (1h): a colorless liquid: IR (CH_2Cl_2) ν 2972, 2931, 1705 (C=O), 1447, 1307, 1144, 1086 cm^{-1} ; 1H NMR ($CDCl_3$, TMS, 300 MHz) δ 1.12 (3H, d, $J = 7.2$ Hz, Me), 1.19 (3H, d, $J = 6.9$ Hz, Me), 2.32 (3H, s, Me), 2.99-3.12 (1H, m, CH), 3.47 (1H, dd, $J = 14.1, 2.4$ Hz), 3.95 (1H, dd, $J = 14.1, 10.8$ Hz), 4.28 (1H, dd, $J = 10.8, 2.4$ Hz), 7.07 (2H, d, $J = 7.8$ Hz, Ar), 7.26 (2H, d, $J = 7.8$ Hz, Ar), 7.48-7.53 (2H, m, Ar), 7.59-7.64 (1H, m, Ar), 7.80-7.83 (2H, m, Ar). ^{13}C NMR ($CDCl_3$, TMS, 75.44 MHz) δ 18.4, 19.5, 21.1, 38.6, 39.5, 57.0, 121.1, 127.7, 129.1, 130.2, 133.7, 136.3, 139.1, 139.9, 205.0. MS (EI) m/e 410 (M^+ , 0.53), 71 (M^+ -339, 100). HRMS Calcd. for $C_{19}H_{22}O_3SSeNa^+$ requires 433.0347, Found: 433.0357.



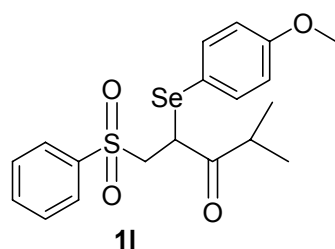
3-(4-methoxyphenylselanyl)-4-(phenylsulfonyl)butan-2-one (1i): a white solid: mp. 105-106 °C; IR (CH₂Cl₂) ν 2986, 2931, 1708 (C=O), 1446, 1306, 1144 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 2.33 (3H, s, Me), 3.45 (1H, dd, J = 13.8, 2.7 Hz), 3.79 (3H, s, Me), 3.83 (1H, dd, J = 13.8, 10.8 Hz), 4.08 (1H, dd, J = 10.8, 2.7 Hz), 6.78-6.82 (2H, m, Ar), 7.31-7.34 (2H, m, Ar), 7.51-7.56 (2H, m, Ar), 7.62-7.68 (1H, m, Ar), 7.81-7.84 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 27.9, 41.5, 55.2, 57.3, 114.7, 115.1, 128.0, 129.3, 134.0, 138.5, 139.0, 161.0, 199.5. MS (EI) m/e 398 (M⁺, 1.13), 43 (M⁺-355, 100). Anal. Calcd. for C₁₇H₁₈O₄SSe requires C, 51.39; H, 4.57%. Found: C, 51.30; H, 4.64%.



2-(4-methoxyphenylselanyl)-1-(phenylsulfonyl)pentan-3-one (1j): a colorless liquid: IR (CH₂Cl₂) ν 2976, 2938, 1708 (C=O), 1588, 1491, 1447, 1306, 1290, 1251, 1175, 1144 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.04 (3H, t, J = 7.5 Hz, CH₃), 2.45 (1H, dq, J = 18.0, 7.5 Hz), 2.91 (1H, dq, J = 18.0, 7.5 Hz), 3.46 (1H, dd, J = 13.8, 2.4 Hz), 3.78 (3H, s, Me), 3.88 (1H, dd, J = 13.8, 10.5 Hz), 4.06 (1H, dd, J = 10.5, 2.4 Hz), 6.78-6.82 (2H, m, Ar), 7.28-7.32 (2H, m, Ar), 7.49-7.54 (2H, m, Ar), 7.61-7.66 (1H, m, Ar), 7.79-7.82 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 8.0, 33.7, 40.5, 55.1, 57.2, 114.8, 114.9, 127.9, 129.2, 133.8, 138.4, 138.8, 160.8, 202.4. MS (EI) m/e 412 (M⁺, 0.68), 57 (M⁺-355, 100). HRMS Calcd. for C₁₈H₂₀O₄SSeNa⁺ requires 435.0140, Found: 435.0132.



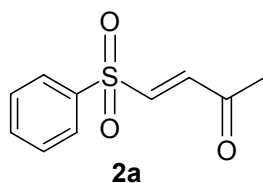
2-(4-methoxyphenylselanyl)-1-phenyl-3-(phenylsulfonyl)propan-1-one (1k): a colorless solid: mp. 133-134 °C; IR (CH₂Cl₂) ν 3062, 2937, 1674 (C=O), 1587, 1491, 1447, 1306, 1252, 1175, 1142 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 3.66 (1H, dd, *J* = 14.1, 2.4 Hz), 3.79 (3H, s, Me), 4.11 (1H, dd, *J* = 14.1, 10.5 Hz), 4.96 (1H, dd, *J* = 10.5, 2.4 Hz), 6.76-6.80 (2H, m, Ar), 7.24-7.28 (2H, m, Ar), 7.36-7.47 (4H, m, Ar), 7.51-7.59 (2H, m, Ar), 7.72-7.76 (2H, m, Ar), 7.80-7.83 (2H, m, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 36.3, 55.2, 58.0, 115.0, 115.3, 128.1, 128.4, 128.6, 129.1, 133.3, 133.8, 134.8, 138.70, 138.74, 161.1, 191.4. MS (EI) *m/e* 460 (M⁺, 17.0), 105 (M⁺-355, 100). Anal. Calcd. for C₂₂H₂₀O₄SSe requires C, 57.52; H, 4.39%. Found: C, 57.44; H, 4.48%.



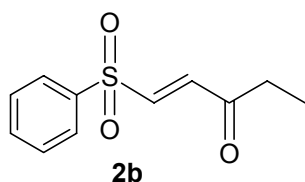
2-(4-methoxyphenylselanyl)-4-methyl-1-(phenylsulfonyl)pentan-3-one (1l): a colorless liquid: IR (CH₂Cl₂) ν 2971, 2933, 1704 (C=O), 1588, 1491, 1307, 1289, 1251, 1175, 1144 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.12 (3H, d, *J* = 6.9 Hz, Me), 1.20 (3H, d, *J* = 6.0 Hz, Me), 2.98-3.13 (1H, m, CH), 3.46 (1H, dd, *J* = 14.1, 1.8 Hz), 3.77 (3H, s, Me), 3.89 (1H, dd, *J* = 14.1, 10.2 Hz), 4.19 (1H, dd, *J* = 10.2, 1.8 Hz), 6.79 (2H, d, *J* = 7.8 Hz, Ar), 7.29 (2H, d, *J* = 7.8 Hz, Ar), 7.49-7.54 (2H, m, Ar), 7.60-7.65 (1H, m, Ar), 7.82 (2H, d, *J* = 7.8 Hz, Ar). ¹³C NMR (CDCl₃, TMS, 75.44 MHz) δ 18.4, 19.5, 38.6, 39.5, 55.1, 56.9, 114.7, 114.9, 127.7, 129.1, 133.7, 138.4, 139.1, 160.8, 204.9. MS (EI) *m/e* 426 (M⁺, 8.33), 125 (M⁺-301, 100). HRMS Calcd. for C₁₉H₂₂O₄SSe⁺ requires 426.0399, Found: 426.0406.

Typical Reaction Procedure for the Reaction of 3-(Phenylselanyl)-4-(phenylsulfonyl)butan-2-one with Hydrogen Peroxide.

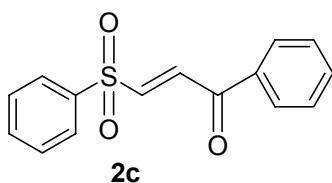
To a solution of 3-(phenylselanyl)-4-(phenylsulfonyl)butan-2-one **1a** (88 mg, 0.24 mmol) in THF (1.0 mL) at room temperature was added hydrogen peroxide (30%, 59 μ L, 0.58 mmol) and the reaction mixture was further stirred at room temperature. The reaction was monitored by TLC plate. When **1a** disappeared, the solvent was removed under reduced pressure and the residue was purified by a flash chromatography (SiO₂, EtOAc:Petroleum ether = 1:6) to yield **2a** (49 mg, 97%) as a colorless solid.



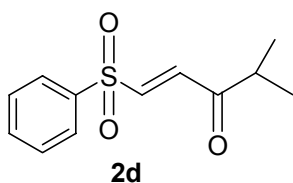
***E*-4-(phenylsulfonyl)but-3-en-2-one (2a):** a colorless solid: This is a known compound; mp. 61-63 °C; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 2.36 (3H, s, Me), 7.02 (1H, d, *J* = 15.3 Hz, CH), 7.15 (1H, d, *J* = 15.3 Hz, CH), 7.57-7.63 (2H, m, Ar), 7.68-7.71 (1H, m, Ar), 7.91-7.94 (2H, m, Ar); The mp. and ¹H NMR spectroscopic data are consistent with those reported in literature.⁴



***E*-1-(phenylsulfonyl)pent-1-en-3-one (2b):** a colorless solid: This is a known compound; mp. 86-88 °C; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 1.12 (3H, t, *J* = 7.2 Hz, CH₃), 2.68 (2H, q, *J* = 7.2 Hz, CH₂), 7.07 (1H, d, *J* = 15.6 Hz, CH), 7.18 (1H, d, *J* = 15.6 Hz, CH), 7.57-7.62 (2H, m, Ar), 7.67-7.73 (1H, m, Ar), 7.91-7.95 (2H, m, Ar); The mp. and ¹H NMR spectroscopic data are consistent with those reported in literature.⁵



***E*-1-phenyl-3-(phenylsulfonyl)prop-2-en-1-one (2c):** a colorless solid: This is a known compound; mp. 115-117 °C; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.36 (1H, d, *J* = 15.0 Hz, CH), 7.51-7.70 (6H, m, Ar), 7.92-8.01 (5H, m, Ar); The mp. and ¹H NMR spectroscopic data are consistent with those reported in literature.^{6,7}



***E*-4-methyl-1-(phenylsulfonyl)pent-1-en-3-one (2d):** (Although this compound is a known one, yet no characterization data have been reported⁸) a colorless solid: mp. 59-60 °C; IR (CH₂Cl₂) ν 3048, 2974, 1701, 1448, 1311, 1152, 1085 cm⁻¹; ¹H NMR (CDCl₃, TMS, 300 MHz)

δ 1.15 (6H, d, $J = 7.2$ Hz), 2.75-2.89 (1H, m), 7.21 (2H, s), 7.56-7.62 (2H, m, Ar), 7.67-7.72 (1H, m, Ar), 7.91-7.94 (2H, m, Ar). ^{13}C NMR (CDCl_3 , TMS, 75.44 MHz) δ 17.4, 40.7, 128.1, 129.5, 134.2, 134.3, 138.4, 140.4, 201.0. MS (EI) m/e 238 (M^+ , 0.82), 125 ($\text{M}^+ - 113$, 100). Anal. Calcd. for $\text{C}_{12}\text{H}_{14}\text{O}_3\text{S}$ requires C, 60.48; H, 5.92%. Found: C, 60.37; H, 6.05%.

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