Supporting Information for

**Novel and efficient Synthesis of Iminocoumarins via Copper-Catalyzed
Multicomponent Reaction**

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Table of Contents

General Considerations	1
General Procedures for the Reactions	1-2
Characterization Data for All Products	2-12
References and Notes	12-3
Copies of ^1H and ^{13}C NMR Spectra for All Products	14-58

General Considerations

Column chromatography was carried out on silica gel (300-400 mesh) with mixed solvents (hexane-ethyl acetate). THF and triethyl amine (TEA) were distilled from Sodium and CaH₂ respectively. Infrared spectra were obtained on a FTIR spectrometer. NMR spectra were recorded for ¹H NMR at 400 MHz otherwise noted, for ¹³C NMR at 100 MHz at 293 K otherwise noted. Chemical shifts are reported relative to residue peaks of CDCl₃ (7.27 ppm for ¹H and 77.27 ppm for ¹³C) and DMSO-*d*₆ (2.50 ppm for ¹H and 40.0 ppm for ¹³C). The following abbreviations are used to describe peak patterns where appropriate: b = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants are reported in Hertz (Hz). Low-resolution MS and HRMS were obtained using ESI ionization. Melting points were measured with micro melting point apparatus. Sulfonyl azides were prepared according the published methods.^{1,2}

General Procedures for the Reaction

To a solution of sulfonyl azide (1 mmol), alkynes (1 mmol), CuI (0.1 mmol) and salicylaldehyde or *o*-hydroxyacetophenone (1.1 mmol) in THF (3 mL) in Schlenk tube was added TEA (2 mmol) slowly via syringe. The reaction solution was stirred at room temperature under N₂ for 12 h. The solution was concentrated, and the residue was diluted with CH₂Cl₂ (30 mL), washed with water (10 mL), brine (10 mL) and dried over sodium sulfate. The solvent was removed in vacuum, and the residue was purified by column

chromatography on silica gel with hexane-ethyl acetate (2:1) to give corresponding iminocoumarin. All the compounds were further recrystallized from EA.

Characterization Data for All Products

Pale yellow solid; mp. 194-195 °C; ^1H NMR (CDCl_3) δ 7.94 (d, J = 8.4 Hz, 2H), 7.68 (s, 1H), 7.54 (m, 4H), 7.42 (d, J = 8.0 Hz, 1H), 7.34 (m, 6H), 2.38 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 157.6, 152.4, 143.2, 139.9, 139.4, 134.5, 132.1, 130.1, 129.7, 129.4, 129.2, 129.1, 128.43, 128.40, 127.4, 126.6, 125.9, 119.9, 116.4, 21.7 ppm; IR (KBr) ν 3065, 1635, 1628, 1558, 1305, 1182, 1088, 863 cm^{-1} ; MS (ESI) m/z 397.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{17}\text{NO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 398.0821; found, 398.0803.

Pale yellow solid; mp. 202-204 °C; ^1H NMR (CDCl_3) δ 8.03 (m, 2H), 7.67-7.44 (m, 8H), 7.37 (m, 4H) ppm; ^{13}C NMR (CDCl_3) δ 157.1, 151.3, 142.2, 138.3, 134.8, 134.1, 132.7, 131.5, 130.6, 129.6, 129.3, 128.9, 128.5, 127.2, 121.6, 118.7, 118.4 ppm; IR (KBr) ν 3066, 1617, 15560, 1545, 1320, 1149, 818 cm^{-1} ; MS (ESI) m/z 461.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{14}\text{BrNO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 461.9770; found, 461.9750.

Pale yellow solid; mp. 261-265 °C; ^1H NMR (DMSO-*d*₆) δ 8.20(s, 1H), 8.09 (d, J = 8.4 Hz, 2H), 7.76 (d, J = 8.4 Hz, 1H), 7.68 (d, J = 8.8 Hz, 2H), 7.61 (d, J = 6.8 Hz, 2H), 7.43 (m, 3H), 7.10 (dd, J_1 = 2.0 Hz, J_2 = 8.4 Hz, 1H), 7.03 (d, J = 2.0 Hz, 1H), 3.94 (s, 3H) ppm; ^{13}C NMR (DMSO-*d*₆) δ 163.5, 158.3, 153.8, 142.6, 141.1, 138.0, 135.0, 130.8, 129.8, 129.60, 129.57, 128.9, 128.6, 125.2, 115.1, 113.9, 100.1, 56.8 ppm; IR (KBr) ν 3088, 1635, 1578, 1558, 1288, 1087, 895, 767 cm^{-1} ; MS (ESI) m/z 447.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{16}\text{ClNO}_4\text{S}$ ($[\text{M}+\text{Na}]^+$), 448.0381; found, 448.0381.

Pale yellow crystal; mp. 152-154 °C; ^1H NMR (CDCl_3) δ 7.71 (s, 1H), 7.59 (m, 2H), 7.54 (m, 3H), 7.42 (m, 3H), 7.34 (m, 1H), 3.08 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 157.9, 152.4, 139.9, 134.7, 132.1, 129.9, 129.2, 129.1, 128.4, 128.3, 125.9, 119.8, 116.9, 43.2 ppm; IR (KBr) ν 3032, 2930, 1624, 1558, 1550, 1305, 1139, 954, 767 cm^{-1} ; MS (ESI) m/z 321.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{13}\text{NO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 322.0508; found, 322.0500.

White crystal; mp. 214-216 °C; ^1H NMR (CDCl_3) δ 7.66 (m, 2H), 7.62 (s, 1H), 7.59 (m, 2H), 7.45 (m, 4H), 3.09 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 157.2, 151.3, 138.2, 134.8, 134.4, 131.4, 130.5, 129.5, 129.2, 128.6, 121.5, 118.8, 118.6, 43.3 ppm; IR (KBr) ν 3050, 2930, 1621, 1576, 1560, 1304, 1130, 817 cm^{-1} ; MS (ESI) m/z 400.1 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{12}\text{BrNO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 399.9613; found, 399.9595.

Green solid; mp. 213-217 °C; ^1H NMR (CDCl_3) δ 8.40 (d, J = 4.0 Hz, 1H), 8.22 (m, 1H), 8.00 (d, J = 8.0 Hz, 2H), 7.97 (d, J = 3.6 Hz, 1H), 7.90 (d, J = 8.0 Hz, 1H), 7.70 (m, 3H), 7.54 (m, 2H), 7.42 (m, 3H), 7.30 (d, J = 8.0 Hz, 2H), 2.40 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 157.6, 152.1, 143.2, 139.7, 135.8, 134.9, 133.5, 130.9, 129.42, 129.38, 129.3, 128.8, 128.7, 128.5, 127.4, 126.9, 121.7, 116.2, 114.6, 21.7 ppm; IR (KBr) ν 3060, 1631, 1609, 1545, 1513, 1291, 1087, 870 cm^{-1} ; MS (ESI) m/z 448.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{19}\text{NO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 448.0978; found, 448.0957.

Pale yellow solid; mp. 175-178 °C; ^1H NMR (CDCl_3) δ 7.94 (d, J = 8.4 Hz, 2H), 7.63 (m, 2H), 7.58 (s, 1H), 7.52 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.4 Hz, 1H), 7.29 (m, 2H), 7.23 (d, J = 7.6 Hz, 2H), 2.68 (q, J = 7.6 Hz, 2H), 2.41 (s, 3H), 1.26 (t, J = 7.6 Hz, 3H) ppm; ^{13}C NMR

(CDCl₃) δ 157.0, 151.2, 146.0, 143.4, 139.5, 137.5, 134.6, 131.6, 131.5, 130.5, 129.5, 129.3, 128.1, 127.5, 121.7, 118.6, 118.3, 28.9, 21.8, 15.6 ppm; IR (KBr) ν 2970, 1621, 1581, 1570, 1302, 1089, 937, 813, 683 cm⁻¹; MS (ESI) m/z 504.2 ([M+Na]⁺); HRMS (ESI) calcd for C₂₄H₂₀BrNO₃S ([M+Na]⁺), 504.0239; found, 504.0216.

Green solid; mp. 201-204 °C; ¹H NMR (CDCl₃) δ 8.06 (d, J = 7.2 Hz, 2H), 7.71 (s, 1H), 7.54 (m, 7H), 7.44 (d, J = 8.4 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.07 (t, J = 8.8 Hz, 2H) ppm; ¹³C NMR (CDCl₃) δ 163.3 (d, J_{C-F} = 247.2 Hz), 157.7, 152.5, 142.3, 139.9, 132.7, 132.3, 131.2 (d, J_{C-F} = 8.3 Hz), 130.5 (d, J_{C-F} = 3.0 Hz), 129.2, 128.9, 128.4, 127.3, 126.1, 119.9, 116.6, 115.5 (d, J_{C-F} = 21.5 Hz) ppm; IR (KBr) ν 3100, 1624, 1585, 1546, 1323, 1161, 1090, 758 cm⁻¹; MS (ESI) m/z 401.8 ([M+Na]⁺); HRMS (ESI) calcd for C₂₁H₁₄FNO₃S ([M+Na]⁺), 402.0571; found, 402.0560.

Yellow solid; mp. 173-174 °C; ¹H NMR (CDCl₃) δ 7.80 (d, J = 8.4 Hz, 2H), 7.63 (s, 1H), 7.55 (d, J = 8.8 Hz, 2H), 7.49 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 8.0 Hz, 1H), 7.29 (m, 3H), 6.88 (d, J = 8.4 Hz, 2H), 3.80 (s, 3H), 2.38 (s, 3H) ppm; ¹³C NMR (CDCl₃) δ 160.4, 157.8, 152.2, 143.2, 139.5, 138.7, 131.7, 130.6, 129.7, 129.4, 128.2, 127.5, 126.8, 125.9, 120.1, 116.4,

113.9, 55.5, 21.7 ppm; IR (KBr) ν 3034, 2926, 1624, 1608, 1540, 1301, 1152, 1086, 832 cm^{-1} ; MS (ESI) m/z 427.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{19}\text{NO}_4\text{S}$ ($[\text{M}+\text{Na}]^+$), 428.0927; found, 428.0906.

Brown solid; mp. 115-119 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 8.70 (s, 1H), 8.60 (s, 1H), 8.24 (d, $J = 8.0$ Hz, 1H), 7.76 (m, 1H), 7.60 (m, 3H), 7.37 (m, 1H), 7.31 (m, 1H), 3.20 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 157.4, 152.7, 151.3, 149.7, 142.7, 136.4, 132.9, 129.2, 127.3, 126.1, 124.9, 123.9, 119.8, 116.9, 43.2 ppm; IR (KBr) ν 3067, 1628, 1570, 1286, 1122, 871, 772 cm^{-1} ; MS (ESI) m/z 322.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{12}\text{N}_2\text{O}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 323.0461; found, 323.0450.

Pale yellow crystal; mp. 187-190 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 8.02 (dd, $J_1 = 2.0$ Hz, $J_2 = 8.0$ Hz, 2H), 7.47 (m, 3H), 7.35 (m, 1H), 6.89 (m, 2H), 3.87 (s, 3H), 2.47 (t, $J = 7.8$ Hz, 2H), 1.56 (m, 2H), 1.34 (m, 2H), 0.90 (m, 3H) ppm; ^{13}C NMR (CDCl_3) δ 162.6, 159.5, 153.6, 141.3, 138.72, 138.68, 129.0, 128.8, 128.6, 127.4, 114.6, 113.5, 100.3, 56.2, 30.3, 30.0, 22.5, 14.0 ppm; IR (KBr) ν 3092, 2958, 1635, 1539, 1312, 1158, 1086, 837 cm^{-1} ; MS (ESI) m/z 427.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{ClNO}_4\text{S}$ ($[\text{M}+\text{Na}]^+$), 428.0694; found, 428.0676.

White crystal; mp. 195-198 °C; ^1H NMR (CDCl_3) δ 8.00 (d, J = 8.8 Hz, 2H), 7.61 (m, 2H), 7.51 (m, 3H), 7.32 (d, J = 8.4 Hz, 1H), 1.36 (s, 9H) ppm; ^{13}C NMR (CDCl_3) δ 156.6, 150.7, 141.2, 138.99, 138.95, 135.8, 134.4, 130.5, 129.3, 128.6, 121.3, 118.4, 118.0, 35.7, 28.9 ppm; IR (KBr) ν 3080, 2959, 1619, 1542, 1314, 1157, 1081, 833 cm^{-1} ; MS (ESI) m/z 476.1 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{BrClINO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 475.9693; found, 475.9693.

White crystal; mp. 158-161 °C; ^1H NMR (CDCl_3) δ 7.99 (d, J = 8.4 Hz, 2H), 7.84 (s, 1H), 7.52 (m, 2H), 7.42 (d, J = 8.4 Hz, 1H), 7.31 (m, 3H), 4.69 (m, 2H), 4.40 (dd, J_1 = 1.2 Hz, J_2 = 16.0 Hz, 1H), 3.85 (m, 1H), 3.53 (m, 1H), 2.41 (s, 3H), 1.89-1.56 (m, 6H) ppm; ^{13}C NMR (CDCl_3) δ 156.5, 152.2, 143.5, 139.2, 137.0, 131.7, 129.5, 128.3, 127.9, 127.7, 125.9, 119.6, 116.6, 99.3, 63.9, 62.9, 30.7, 25.5, 21.8, 19.8 ppm; IR (KBr) ν 3062, 2945, 1635, 1558, 1325, 1163, 1029, 831 cm^{-1} ; MS (ESI) m/z 436.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$ ($[\text{M}+\text{Na}]^+$), 436.1189; found, 436.1171.

Yellow solid. Mp. 139-143 °C; ^1H NMR (CDCl_3) δ 8.55 (s, 1H), 8.28 (d, J = 8.4 Hz, 1H), 7.99 (d, J = 9.2 Hz, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.71 (m, 1H), 7.59 (m, 2H), 3.17 (s, 3H), 0.42 (s, 9H) ppm; ^{13}C NMR (CDCl_3) δ 160.7, 153.2, 144.2, 133.8, 130.9, 129.6, 129.3, 128.7, 128.6, 126.7, 121.4, 117.0, 114.0, 43.2, -1.57 ppm; IR (KBr) ν 3049, 2933, 1675, 1635, 1512, 1315, 1241, 1144, 966, 816 cm^{-1} ; MS (ESI) m/z 367.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{19}\text{NO}_3\text{SSi}$ ($[\text{M}+\text{Na}]^+$), 368.0747; found, 368.0744.

Yellow solid; mp. 151-153 °C; ^1H NMR (CDCl_3) δ 8.29 (s, 1H), 7.99 (d, J = 8.8 Hz, 2H), 7.65 (m, 1H), 7.58 (d, J = 7.6 Hz, 1H), 7.46 (m, 2H), 7.30 (m, 2H), 4.33 (q, J = 7.2 Hz, 2H), 2.41 (s, 3H), 1.30 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR (CDCl_3) δ 162.8, 154.1, 153.5, 145.7, 143.3, 139.5, 134.6, 129.7, 129.4, 127.3, 126.2, 121.2, 118.2, 117.0, 62.4, 21.7, 14.2 ppm; IR (KBr) ν 3077, 1700, 1630, 1584, 1309, 1239, 1090, 833 cm^{-1} ; MS (ESI) m/z 394.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{NO}_5\text{S}$ ($[\text{M}+\text{Na}]^+$), 394.0720; found, 394.0704.

White solid; mp. 139-141 °C; ^1H NMR (CDCl_3) δ 7.77 (s, 1H), 7.68 (dd, J_1 = 1.6 Hz, J_2 = 8.4

Hz, 2H), 7.50 (m, 2H), 7.41 (m, 3H), 7.32 (d, $J = 8.4$ Hz, 1H), 7.27 (m, 1H) ppm; ^{13}C NMR (CDCl_3) δ 160.7, 163.6, 140.0, 134.8, 131.5, 129.0, 128.7, 128.6, 128.4, 128.1, 124.7, 119.8, 116.5 ppm; IR (KBr) ν 3056, 1719, 1612, 1453, 1123, 1118, 961, 751 cm^{-1} ; MS (ESI) m/z 244.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{10}\text{O}_2$ ($[\text{M}+\text{Na}]^+$), 245.0573; found, 245.0567.

Yellow crystal; mp. 236-240 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 7.82 (d, $J = 8.4$ Hz, 2H), 7.70 (dd, $J_1 = 1.6$ Hz, $J_2 = 8.0$ Hz, 1H), 7.57 (m, 1H), 7.48 (m, 1H), 7.40 (m, 4H), 7.23 (m, 4H), 2.37 (s, 3H), 2.28 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 158.4, 151.8, 147.6, 142.8, 140.0, 134.2, 132.0, 130.2, 129.2, 128.8, 128.5, 128.4, 127.2, 125.7, 125.4, 120.9, 117.1, 21.7, 17.0 ppm; IR (KBr) ν 3067, 3044, 1620, 1539, 1299, 1155, 1081, 782 cm^{-1} ; MS (ESI) m/z 412.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{19}\text{NO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 412.0978; found, 412.0964.

Brown solid; mp. 215-219 $^{\circ}\text{C}$; ^1H NMR (CDCl_3) δ 7.93 (d, $J = 7.2$ Hz, 2H), 7.69 (d, $J = 8.4$ Hz, 1H), 7.57 (m, 1H), 7.41 (m, 8H), 7.24 (d, $J = 6.8$ Hz, 2H), 2.27 (s, 3H) ppm; ^{13}C NMR (CDCl_3) δ 158.6, 151.6, 148.0, 142.8, 134.1, 132.2, 132.0, 130.2, 128.61, 128.58, 128.5, 128.4, 127.0, 125.8, 120.8, 116.9, 17.0 ppm; IR (KBr) ν 3060, 1618, 1539, 1317, 1303, 1151, 1080, 838, 783, 756 cm^{-1} ; MS (ESI) m/z 398.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for

C₂₂H₁₇NO₃S ([M+Na]⁺), 398.0821; found, 398.0803.

White crystal; mp. 233-236 °C; ¹H NMR (CDCl₃) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.58 (m, 2H), 7.41 (m, 4H), 7.25 (d, *J* = 7.6 Hz, 2H), 2.95 (s, 3H), 2.28 (s, 3H) ppm; ¹³C NMR (CDCl₃) δ 158.6, 151.7, 147.6, 134.3, 131.9, 130.2, 128.5, 128.4, 128.3, 125.7, 125.3, 120.8, 117.4, 43.1, 16.9 ppm; IR (KBr) ν 3021, 1623, 1558, 1301, 1137, 974, 850, 795 cm⁻¹; MS (ESI) *m/z* 335.8 ([M+Na]⁺); HRMS (ESI) calcd for C₁₇H₁₅NO₃S ([M+Na]⁺), 336.0665; found, 336.0650.

White solid; mp. 240-243 °C; ¹H NMR (CDCl₃) δ 7.86 (d, *J* = 8.8 Hz, 2H), 7.72 (dd, *J*₁ = 0.8 Hz, *J*₂ = 8.0 Hz, 1H), 7.59 (m, 1H), 7.48 (m, 1H), 7.38 (m, 3H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.31 (s, 3H), 1.29 (t, *J* = 7.6 Hz, 3H) ppm; ¹³C NMR (CDCl₃) δ 159.1, 151.6, 148.3, 144.6, 141.5, 138.4, 132.1, 131.2, 130.1, 128.9, 128.7, 128.6, 128.0, 125.9, 125.5, 121.0, 117.0, 28.9, 17.1, 15.6 ppm; IR (KBr) ν 3088, 2965, 1620, 1539, 1325, 1156, 1080, 830 cm⁻¹; MS (ESI) *m/z* 460.2 ([M+Na]⁺); HRMS (ESI) calcd for C₂₄H₂₀ClNO₃S ([M+Na]⁺), 460.0745; found, 460.0725.

Brown crystal; mp. 197-199 °C; ^1H NMR (CDCl_3) δ 7.97 (d, J = 8.8 Hz, 2H), 7.60 (dd, J_1 = 1.2 Hz, J_2 = 7.6 Hz, 1H), 7.45 (m, 1H), 7.30 (m, 4H), 2.65 (t, J = 7.6 Hz, 2H), 2.40 (s, 3H), 2.37 (s, 3H), 1.47 (m, 2H), 1.36 (m, 2H), 0.89 (t, J = 7.6 Hz, 3H) ppm; ^{13}C NMR (CDCl_3) δ 158.6, 151.1, 146.1, 143.0, 139.9, 131.1, 129.3, 128.0, 127.4, 125.5, 124.7, 120.9, 116.7, 30.5, 27.5, 22.9, 21.7, 15.2, 14.1 ppm; IR (KBr) ν 2950, 2870, 1620, 1558, 1312, 1154, 1082, 769 cm^{-1} ; MS (ESI) m/z 391.8 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{23}\text{NO}_3\text{S}$ ($[\text{M}+\text{Na}]^+$), 392.1291; found, 392.1307.

Yellow crystal; mp. 172-174 °C; ^1H NMR (CDCl_3) δ 8.10 (dd, J_1 = 1.2 Hz, J_2 = 7.6 Hz, 2H), 7.69 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz, 1H), 7.52 (m, 4H), 7.37 (m, 2H), 4.82 (d, J = 11.6 Hz, 1H), 4.71 (m, 1H), 4.56 (d, J = 10.8 Hz, 1H), 3.86 (m, 1H), 3.43 (m, 1H), 2.56 (s, 3H), 1.60 (m, 2H), 1.49 (m, 4H) ppm; ^{13}C NMR (CDCl_3) δ 158.7, 151.8, 151.0, 142.7, 132.4, 132.3, 128.8, 127.4, 125.8, 125.3, 123.6, 120.8, 117.0, 99.7, 62.7, 61.7, 30.7, 25.5, 19.7, 15.5 ppm; IR (KBr) ν 2935, 2884, 1621, 1539, 1449, 1317, 1163, 1086, 862, 762 cm^{-1} ; MS (ESI) m/z 436.2 ($[\text{M}+\text{Na}]^+$); HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{23}\text{NO}_5\text{S}$ ($[\text{M}+\text{Na}]^+$), 436.1189; found, 436.1169.

References and Notes

1. (a) Danheiser, R. L.; Miller, R. F.; Brisbois, R. G.; Park, S. Z. *J. Org. Chem.*; **1990**; 55, 1959-1964. (b)

Hazen, G. G.; Bollinger, F. W.; Roberts, F. E.; Russ, W. K.; Seman, J. J.; Staskiewicz, S. *Org. Synth.* **1998**, *Coll. Vol. 9*, 400; **1996**, *Vol. 73*, 144.

2. Caution: Although the mixture of sulfonyl azides is the safest of a group of diazo compounds, one should keep in mind the inherent instability, shock sensitivity, and explosive power of azides. All users should exercise appropriate caution.

Copies of ^1H and ^{13}C NMR Spectra for All Products