Synthesis of 3,4-Disubstituted 2*H*-1-Benzopyrans Through C-C Bond Formation via Electrophilic Cyclization

Shilpa A. Worlikar; Tanay Kesharwani; Tuanli Yao and Richard C. Larock*

Department of Chemistry, Iowa State University, Ames, Iowa 50011

Supporting Information

General. The ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75.5 MHz or 400 and 100 MHz respectively. Thin layer chromatography was performed using 60 mesh silica gel plates, and visualization was effected with short wavelength UV light (254 nm). All melting points are uncorrected. All high resolution mass spectra were recorded using EI at 70 eV. All reagents were used directly as obtained commercially unless otherwise noted.

Preparation of starting materials.

General procedure for the palladium/copper-catalyzed reaction of phenyl propargyl ether with aryl halides. To a solution of 2.5 mmol of the aryl halide in Et_3N (15 ml) was added $PdCl_2(PPh_3)_2$ (2 mol %), which was then stirred for 5 min. CuI (1.5 mol %) was then added and the flask was sealed and flushed with Ar. The reaction was stirred for 20 min. A solution of 3.0 mmol of phenyl propargyl ether in 2 mL of Et_3N was then added dropwise and the reaction mixture was allowed to stir at room temperature for the desired time. After the reaction was over, the resulting solution was diluted with H_2O (10 ml) and extracted with diethyl ether (3 x 15 mL). The combined ether fractions were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product. The crude product was purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent.

Phenyl 3-*p*-tolylprop-2-yn-1-yl ether (4). This compound was obtained as a white solid: mp 71-72 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.30 (s, 3H), 4.86 (s, 2H), 6.94-7.08 (m, 5H), 7.18-7.33 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.7, 56.9, 83.5, 87.5, 115.2,

119.5, 121.6, 129.3, 129.7, 132.0, 139.0, 158.1; IR (neat, cm^{-1}) 3032, 2914, 1598, 1490, 1214, 1029; HRMS m/z 222.10477 (calcd $C_{16}H_{14}O$, 222.10447).

3-(4-Methoxyphenyl)prop-2-yn-1-yl phenyl ether (5). This compound was obtained as a light brown solid: mp 61-62 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 3.77 (s, 3H), 4.88 (s, 2H), 6.81 (d, J = 8.8 Hz, 2H), 6.95-7.03 (m, 3H), 7.27-7.38 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.4, 56.8, 82.7, 87.2, 114.0, 114.5, 115.1, 121.5, 129.6, 133.5, 158.0, 160.0; IR (neat, cm^{-1}) 3042, 2919, 1598, 1506, 1239, 1024; HRMS m/z 238.09979 (calcd $C_{16}H_{14}O_2$, 238.09938).

3-(2-Methoxyphenyl)prop-2-yn-1-yl phenyl ether (6). This compound was obtained as a yellow oil: ^1H NMR (300 MHz, $CDCl_3$) δ 3.77 (s, 3H), 4.90 (s, 2H), 6.77-6.86 (m, 2H), 6.95 (t, J = 7.3 Hz, 1H), 7.02 (d, J = 7.8 Hz, 2H), 7.20-7.30 (m, 3H), 7.36 (dd, J = 7.6, 1.6 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.7, 56.9, 83.6, 88.1, 110.6, 111.4, 115.1, 120.4, 121.3, 129.4, 130.2, 133.8, 157.9, 160.2; IR (neat, cm^{-1}) 3057, 3032, 2934, 2243, 1603, 1270, 1024; HRMS m/z 238.09968 (calcd $C_{16}H_{14}O_2$, 238.09938).

Substituted propargyl ether **7** was prepared according to a literature procedure.[1]

3,5-Di-*tert*-butylphenyl propargyl ether (8). To a solution of 2.06 g of 3,5-di-*tert*-butylphenol (10.0 mmol) in dry acetone (50 ml) was added propargyl bromide (11.0 mmol) and anhydrous K_2CO_3 (11.0 mmol). The resulting mixture was refluxed for 24 h. The reaction mixture was diluted with H_2O (20 ml) and extracted with diethyl ether (3 x 20 ml). The combined ether layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (7:1 hexane/EtOAc) to afford 1.68 g of the indicated compound **7** (69% yield) as a yellow oil: ^1H NMR (300 MHz, $CDCl_3$) δ 1.30 (s, 18H), 2.42 (t, J = 2.5 Hz, 1H), 4.63 (d, J = 2.4

Hz, 2H), 6.83 (d, J = 1.6 Hz, 2H), 7.05 (d, J = 1.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.5, 35.0, 55.7, 75.4, 79.1, 109.4, 115.7, 152.2, 157.3; IR (neat, cm^{-1}) 3298, 2939, 1588, 1424, 1285, 1050; HRMS m/z 244.18311 (calcd $C_{17}H_{24}O$, 244.18272).

General procedure for the palladium/copper-catalyzed reaction of terminal alkynes with iodobenzene. To a solution of 4.5 mmol of iodobenzene in Et_3N (15 ml), was added $PdCl_2(PPh_3)_2$ (2 mol %), and CuI (1.5 mol %), and the mixture was stirred for 30 min under Ar. A solution of 3.0 mmol of the terminal alkyne in 2 mL of Et_3N was then added dropwise and the reaction mixture was allowed to stir at room temperature for the desired time. After the reaction was over, the resulting solution was diluted with H_2O (10 ml) and extracted with diethyl ether (3 x 15 mL). The combined ether fractions were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product. The crude product was purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent.

4-(3-Phenylprop-2-yn-1-yloxy)benzaldehyde (9). This compound was obtained as a brown solid: mp 86-87 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.00 (s, 2H), 7.14 (d, J = 8.8 Hz, 2H), 7.25-7.32 (m, 3H), 7.41-7.44 (m, 2H), 7.87 (d, J = 8.8 Hz, 2H), 9.90 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 57.0, 82.9, 88.1, 115.4, 122.0, 128.5, 129.1, 130.6, 132.0, 132.1, 162.8, 191.0; IR (neat, cm^{-1}) 3078, 2827, 1690, 1598, 1250, 1009; HRMS m/z 236.08409 (calcd $C_{16}H_{12}O_2$, 236.08373).

3,5-Di-*tert*-butylphenyl 3-phenylprop-2-yn-1-y1 ether (10). This compound was obtained as a light yellow solid: mp 54-55 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.32 (s, 18H), 4.91 (s, 2H), 6.91 (d, J = 1.6 Hz, 2H), 7.06 (t, J = 1.5 Hz, 1H), 7.26-7.31 (m, 3H), 7.41-7.45 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.7, 35.2, 56.8, 84.5, 87.2, 109.6,

115.8, 122.6, 128.5, 128.8, 132.0, 152.4, 157.6; IR (neat, cm^{-1}) 3081, 2963, 1591, 1362, 1297, 1051; HRMS m/z 320.21446 (calcd $C_{23}H_{28}O$, 320.21402).

General procedure for the triphenylphosphine/diethyl azodicarboxylate-promoted formation of the substituted phenyl propargylic ethers. To a solution of 1.31 g of PPh_3 (5.0 mmol) in dry benzene (15 ml) was added the substituted propargylic alcohol (5.0 mmol) and the substituted phenol (5.0 mmol) under an inert atmosphere with stirring. Diethyl azodicarboxylate (0.87 g, 5.0 mmol) was then added slowly and the reaction mixture was stirred at r.t. for 18 to 36 h. After the reaction was complete, the solvent was evaporated under reduced pressure and the crude product was purified by flash chromatography on silica gel using hexanes/ethyl acetate as the eluent.

3-*tert*-Butylphenyl 3-phenylprop-2-yn-1-y1 ether (11). This compound was obtained as a yellow oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.30 (s, 9H), 4.86 (s, 2H), 6.83 (dd, J = 8.0, 2.3 Hz, 1H), 6.96-7.02 (m, 1H), 7.08 (t, J = 1.9 Hz, 1H), 7.19-7.25 (m, 4H), 7.39-7.42 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.4, 34.8, 56.6, 84.3, 87.2, 111.2, 113.1, 118.6, 122.4, 128.4, 128.7, 129.1, 131.9, 153.0, 157.7; IR (neat, cm^{-1}) 3067, 2955, 2868, 1588, 1485, 1270, 1029; HRMS m/z 264.15187 (calcd $C_{19}H_{20}O$, 264.15142).

3-(Cyclohex-1-enyl)prop-2-yn-1-yl phenyl ether (12). This compound was obtained as a dark brown oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.53-1.62 (m, 4H), 2.04-2.10 (m, 4H), 4.77 (s, 2H), 6.10-6.13 (m, 1H), 6.93-6.98 (m, 2H), 7.24-7.30 (m, 2H), 7.40-7.70 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 22.3, 25.7, 29.0, 56.7, 81.3, 89.1, 115.0, 120.0, 121.3, 129.5, 136.1, 157.9; IR (neat, cm^{-1}) 3032, 2919, 2217, 1593, 1485, 1219; HRMS m/z 212.12047 (calcd $C_{15}H_{16}O$, 212.12012).

1-Naphthyl 3-phenylprop-2-yn-1-yl ether (13). This compound was obtained as a light brown solid: mp 50-51 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.08 (s, 2H), 6.99 (d, J = 7.4 Hz, 1H), 7.26-7.49 (m, 9H), 7.77-7.80 (m, 1H), 8.30-8.33 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 57.2, 84.2, 87.5, 105.9, 121.2, 122.4, 122.6, 125.6, 125.9, 126.0, 126.7, 127.7, 128.5, 128.9, 132.0, 134.8, 153.8; IR (neat, cm^{-1}) 3057, 2914, 1577, 1398, 1229, 1091; HRMS m/z 258.10497 (calcd $C_{19}H_{14}O$, 258.10447).

General procedure for iodocyclization. To a solution of 0.25 mmol of the ether and 3 mL of CH_3NO_2, 2.0 equiv of $NaHCO_3$ and 3.0 equiv of I_2 dissolved in 2 mL of CH_3NO_2 was added gradually. The reaction mixture was allowed to stir at room temperature for the desired time. Alternatively, to a solution of 0.25 mmol of the ether and 3 mL of CH_3NO_2 at -25 to -30 ^{o}C, 1.5 equiv of ICl dissolved in 2 mL of CH_3NO_2 was added gradually. The reaction mixture was allowed to stir at -25 to -30 ^{o}C for the desired time. The excess I_2 or ICl was removed by washing with satd aq $Na_2S_2O_3$. The mixture was then extracted by diethyl ether (3 x 10 mL). The combined ether layers were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using hexanes/ethyl acetate as the eluent.

3-Iodo-4-phenyl-2*H*-benzopyran (2). This compound was obtained as a pale yellow solid: mp 99-100 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.06 (s, 2H), 6.61 (dd, J = 7.7, 1.6 Hz, 1H), 6.76 (dt, J = 7.7, 1.1 Hz, 1H), 6.85 (dd, J = 8.0, 1.0 Hz, 1H), 7.14 (dd, J = 7.9, 1.6 Hz, 1H), 7.18-7.22 (m, 2H), 7.39-7.46 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.1, 91.2, 116.1, 121.7, 124.2, 126.5, 128.3, 128.7, 129.5, 129.7, 140.0, 142.0, 153.3; IR (neat, cm^{-1}) 3062, 2904, 1475, 1219, 1029, 994; HRMS m/z 333.98600 (calcd $C_{15}H_{11}IO$, 333.98547).

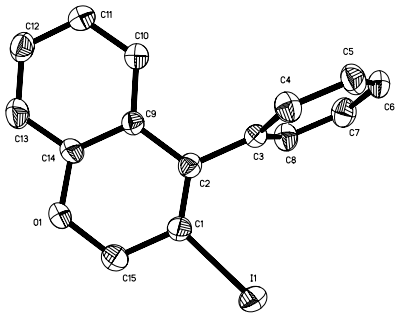

Figure 1. X-ray structure of compound **2**

3-Iodo-4-(4-methylphenyl)-2*H*-benzopyran (24). This compound was obtained as a pale brown solid: mp 68-69 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.23 (s, 3H), 4.87 (s, 2H), 6.46 (dd, J = 7.7, 1.6 Hz, 1H), 6.58 (t, J = 7.5 Hz, 1H), 6.66 (d, J = 8.0 Hz, 1H), 6.89-6.98 (m, 3H), 7.07 (d, J = 8.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.6, 75.2, 91.1, 116.1, 121.7, 124.4, 126.6, 129.4, 129.5, 129.7, 137.1, 138.1, 142.0, 153.5; IR (neat, cm^{-1}) 3032, 2914, 2842, 1475, 1219, 999; HRMS m/z 348.00051 (calcd $C_{16}H_{13}IO$, 348.00112).

3-Iodo-4-(4-methoxyphenyl)-2*H*-benzopyran (25). This compound was obtained as a pale brown solid: mp 110-111 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.85 (s, 3H), 5.05 (s, 2H), 6.66 (dd, J = 7.7, 1.2 Hz, 1H), 6.77 (t, J = 7.4 Hz, 1H), 6.84 (d, J = 8.0 Hz, 1H), 6.96 (d, J = 8.6 Hz, 2H), 7.11-7.17 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.5, 75.2, 91.4, 114.1, 116.1, 121.7, 124.6, 126.6, 129.7, 130.8, 132.3, 141.7, 153.5, 159.5; IR (neat, cm^{-1}) 2955, 2918, 2833, 1505, 1244, 1171, 1037; HRMS m/z 363.99640 (calcd $C_{16}H_{13}IO_2$, 363.99603).

3-Iodo-4-(4-nitrophenyl)-2*H*-benzopyran (26). This compound was obtained as a yellow solid: mp 140-142 ^{o}C (decomp.); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.07 (s, 2H), 6.50 (dd, J = 7.7, 1.4 Hz, 1H), 6.79 (dt, J = 7.8, 0.9 Hz, 1H), 6.88 (dd, J = 8.2, 0.9 Hz, 1H),

7.19 (dt, J = 8.0, 1.4 Hz, 1H), 7.41 (dd, J = 6.9, 1.9 Hz, 2H), 8.32 (dd, J = 6.9, 1.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.0, 91.8, 116.5, 122.0, 123.4, 124.1, 125.9, 130.4, 130.8, 140.4, 146.6, 147.8, 153.2; IR (neat, cm^{-1}) 3093, 2837, 1926, 1593, 1516, 1337, 1219; HRMS m/z 378.97119 (calcd $C_{15}H_{10}INO_3$, 378.97055).

3-Iodo-4-(2-methoxyphenyl)-2*H*-benzopyran (27). This compound was obtained as a light brown solid: mp 111-113 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.75 (s, 3H), 5.01-5.13 (m, 2H), 6.57 (dd, J = 7.8, 1.5 Hz, 1H), 6.74 (dt, J = 7.6, 1.1 Hz, 1H), 6.83 (dd, J = 7.1, 1.0 Hz, 1H), 6.96-7.14 (m, 4H), 7.37-7.42 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.9, 74.9, 92.7, 111.6, 116.0, 121.0, 121.7, 123.8, 126.1, 129.0, 129.5, 130.0, 131.0, 139.5, 153.2, 156.9; IR (neat, cm^{-1}) 2955, 2918, 1505, 1244, 1171, 1037; HRMS m/z 363.99644 (calcd $C_{16}H_{13}IO_2$, 363.99603).

3-Iodo-6-methyl-4-phenyl-2*H*-benzopyran (28). This compound was obtained as a brown oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.10 (s, 3H), 5.01 (s, 2H), 6.41 (s, 1H), 6.75 (d, J = 8.2, Hz, 1H), 6.94 (d, J = 8.2, Hz, 1H), 7.17-7.20 (m, 2H), 7.40-7.46 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 20.8, 75.1, 91.4, 115.8, 124.1, 126.8, 128.2, 128.6, 129.5, 130.2, 131.0, 140.1, 142.1, 151.1; IR (neat, cm^{-1}) 3052, 2914, 1741, 1485, 1229, 999; HRMS m/z 348.00155 (calcd $C_{16}H_{13}IO$, 348.00112).

6-*tert*-Butyl-3-iodo-4-phenyl-2*H*-benzopyran (29). This compound was obtained as a pale yellow solid: mp 84-86 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.11 (s, 9H), 5.03 (s, 2H), 6.63 (d, J = 2.4 Hz, 1H), 6.79 (d, J = 8.4 Hz, 1H), 7.16 (d, J = 2.4 Hz, 1H), 7.18-7.22 (m, 2H), 7.37-7.48 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.4, 34.3, 75.2, 90.9, 115.4, 123.5, 123.8, 126.6, 128.3, 128.6, 129.5, 140.1, 142.4, 144.5, 151.1; IR (neat, cm^{-1}) 3052, 2950, 1485, 1357, 1229, 1004; HRMS m/z 390.04856 (calcd $C_{19}H_{19}IO$, 390.04807).

3-Iodo-6-methoxy-4-phenyl-2*H*-benzopyran (30). This compound was obtained as a pale brown solid: mp 81-82 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.59 (s, 3H), 4.99 (s, 2H), 6.19 (d, J = 2.9 Hz, 1H), 6.69 (dd, J = 8.7, 2.9 Hz, 1H), 6.79 (d, J = 8.7 Hz, 1H), 7.18-7.23 (m, 2H), 7.38-7.46 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.8, 75.3, 92.4, 112.5, 114.4, 116.6, 125.1, 128.4, 128.8, 129.5, 140.0, 142.1, 147.4, 154.3; IR (neat, cm^{-1}) 2991, 2924, 2822, 1572, 1480, 1301, 1198; HRMS m/z 363.99663 (calcd $C_{16}H_{13}IO_2$, 363.99603).

6-Chloro-3-iodo-4-phenyl-2*H*-benzopyran (31). This compound was obtained as a brown solid: mp 89-90 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.07 (s, 2H), 6.59 (d, J = 2.6 Hz, 1H), 6.79 (d, J = 8.6 Hz, 1H), 7.09 (dd, J = 8.6, 2.5 Hz, 1H), 7.19 (dd, J = 7.8, 1.9 Hz 2H), 7.43-7.48 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.3, 93.0, 117.4, 125.3, 126.1, 126.7, 128.6, 129.0, 129.4, 129.5, 139.4, 141.2, 151.9; IR (neat, cm^{-1}) 2919, 2848, 1477, 1403, 1093, 638; HRMS m/z 367.94723 (calcd $C_{15}H_{10}ClIO$, 367.94649).

3-Iodo-4-phenyl-2*H*-benzopyran-6-carbaldehyde (32) and 3-(iodophenyl-methylene)-2,3-dihydrobenzofuran-5-carbaldehyde (33). These compounds were obtained as a light brown solid as a 2:1 mixture: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.07 (s, 2H), 5.19 (s, 1H), 6.95 (d, J = 8.3 Hz, 1H), 7.11 (d, J = 8.5 Hz, 3H), 7.19-7.23 (m, 3H), 7.31-7.40 (m, 4H), 7.47-7.49 (m, 2H), 7.70 (dd, J = 8.3, 1.9 Hz, 1H), 7.90 (d, J = 8.7 Hz, 2H), 9.68 (s, 1H), 9.92 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.5, 80.1, 92.7, 98.49, 99.4, 115.9, 116.9, 123.8, 128.1, 128.5, 128.7, 128.8, 129.0, 129.1, 129.3, 130.7, 130.9, 131.8, 132.2, 139.2, 141.0, 147.2, 158.6, 162.7, 190.7, 190.9; IR (neat, cm^{-1}) 3057, 2827, 1690, 1598, 1234, 1157; HRMS m/z 361.98090 (calcd $C_{16}H_{11}IO_2$, 361.98038).

5,7-Di-*tert*-butyl-3-iodo-4-phenyl-2*H*-benzopyran (34). This compound was obtained as a yellow solid: mp 142-143 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.89 (s, 9H), 1.31 (s, 9H), 4.81 (s, 2H), 6.89 (d, J = 1.9 Hz, 1H), 7.12 (d, J = 2.1 Hz, 2H), 7.24-7.27 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.3, 32.1, 35.2, 36.8, 7.8, 85.0, 109.5, 119.9, 122.7, 127.6, 128.0, 131.1, 143.7, 143.9, 149.8, 152.5, 157.2; IR (neat, cm^{-1}) 2955, 2893, 1593, 1444, 1403, 1004; HRMS m/z 446.11117 (calcd $C_{23}H_{27}IO$, 446.11067).

7-*tert*-Butyl-3-iodo-4-phenyl-2*H*-benzopyran (35). This compound was obtained as a light brown solid: mp 97-98 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.30 (s, 9H), 5.09 (s, 2H), 6.59 (d, J = 4.3 Hz, 1H), 6.83 (dd, J = 8.1, 1.8 Hz, 1H), 6.93 (d, J = 1.7 Hz, 1H), 7.21-7.25 (m, 2H), 7.40-7.44 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.3, 35.0, 75.3, 90.1, 113.3, 118.7, 121.8, 126.1, 128.3, 128.7, 129.5, 140.2, 142.0, 153.1, 153.8; IR (neat, cm^{-1}) 2965, 2904, 2356, 1603, 1485, 1004; HRMS m/z 390.04856 (calcd $C_{19}H_{19}IO$, 390.04807).

7-Chloro-3-iodo-4-phenyl-2*H*-benzopyran (36). This compound was obtained as a light brown solid: mp 90-91 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.06 (s, 2H), 6.52 (d, J = 8.3 Hz, 1H), 6.72 (dd, J = 8.4, 2.1 Hz, 1H), 6.85 (d, J = 2.1 Hz, 1H), 7.16-7.19 (m, 2H), 7.41-7.48 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.3, 91.2, 116.5, 121.9, 122.7, 127.4, 128.5, 128.8, 129.4, 134.7, 139.6, 141.3, 154.0; IR (neat, cm^{-1}) 2837, 1588, 1475, 1413, 1219, 999; HRMS m/z 367.94720 (calcd $C_{15}H_{10}ClIO$, 367.94649).

5-Chloro-3-iodo-4-phenyl-2*H*-benzopyran (37). This compound was obtained as a light brown solid: mp 73-74 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.94 (s, 2H), 6.88-6.91 (m, 2H), 7.10 (t, J = 8.0 Hz, 1H), 7.17-7.20 (m, 2H), 7.34-7.38 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 76.0, 92.8, 115.2, 123.3, 125.0, 128.0, 128.1, 129.7, 129.9, 131.7, 140.4,

141.3, 156.6; IR (neat, cm^{-1}) 3052, 2937, 1588, 1444, 1239, 999; HRMS m/z 367.94725 (calcd $C_{15}H_{10}ClIO$, 367.94649).

3-Iodo-7-methoxy-4-phenyl-2*H*-benzopyran (38) and 3-iodo-5-methoxy-4-phenyl-2*H*-benzopyran (39). These compounds were obtained as a light brown solid as a 2:3 mixture: ^1H NMR (300 MHz, $CDCl_3$) δ 3.22 (s, 3H), 3.76 (s, 2H), 4.93 (s, 2H), 5.04 (s, 1H), 6.31 (dd, J = 8.6, 2.5 Hz, 1H), 6.37-6.47 (m, 2H), 6.50-6.62 (m, 2H), 7.12-7.21 (m, 4H), 7.28-7.47 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.6, 55.8, 75.3, 75.8, 87.0, 89.0, 101.7, 106.1, 107.4, 109.2, 115.1, 117.8, 127.0, 127.6, 127.7, 128.2, 128.5, 128.6, 129.5, 130.2, 140.2, 140.5, 141.8, 143.4, 154.7, 156.0, 156.1, 161.0; IR (neat, cm^{-1}) 3016, 2934, 2827, 1608, 1465, 1270; HRMS m/z 363.99664 (calcd $C_{16}H_{13}IO_2$, 363.99603).

4-(1-Cyclohexenyl)-3-iodo-2*H*-benzopyran (40). This compound was obtained as a light brown oil: ^1H NMR (300 MHz, $CDCl_3$) δ 1.65-1.76 (m, 4H), 2.05 (m, 2H), 2.18-2.20 (m, 2H), 4.93 (s, 2H), 5.60-5.62 (m, 1H), 6.79 (dd, J = 8.0, 0.9 Hz, 1H), 6.86 (dt, J = 7.6, 1.1 Hz, 1H), 7.06 (dd, J = 7.7, 1.5 Hz, 1H), 7.13 (dt, J = 7.7, 1.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 22.2, 22.8, 25.3, 27.6, 74.7, 89.2, 116.1, 121.7, 122.8, 125.6, 129.3, 129.5, 137.5, 143.5, 153.6; IR (neat, cm^{-1}) 3027, 2914, 1598, 1475, 1209, 1034; HRMS m/z 338.01726 (calcd $C_{15}H_{15}IO$, 338.01677).

4-Hydroxymethyl-3-iodo-2*H*-benzopyran (42). This compound was obtained as a yellow solid: mp 62-63 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 2.06 (s, 1H), 4.64 (s, 2H), 4.86 (s, 2H), 6.82 (d, J = 8.0 Hz, 1H), 6.92-6.98 (m, 1H), 7.18 (dt, J = 8.0, 1.3 Hz, 1H), 7.41 (dd, J = 7.7, 1.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 66.0, 75.0, 93.8, 116.4, 121.8, 122.1, 124.3, 129.9, 137.3, 153.7; IR (neat, cm^{-1}) 3334, 2934, 1480, 1444, 1219, 1009; HRMS m/z 287.96514 (calcd $C_{10}H_9IO_2$, 287.96473).

3-Iodo-4-phenyl-2*H*-benzo[*h*]chromene (43). This compound was obtained as a light brown solid: mp 104-105 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.25 (s, 2H), 6.77 (d, J = 8.5 Hz, 1H), 7.21-7.25 (m, 3H), 7.42-7.50 (m, 5H), 7.68-7.71 (m, 1H), 8.17-8.20 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 75.6, 88.1, 118.8, 120.8, 122.2, 124.0, 124.3, 126.0, 127.1, 127.7, 128.3, 128.7, 129.6, 134.3, 140.2, 142.7, 149.4; IR (neat, cm^{-1}) 3055, 2923, 2847, 1562, 1400, 1341; HRMS m/z 384.00158 (calcd $C_{19}H_{13}IO$, 384.00112).

General procedure for the PhSeBr cyclizations. To a solution of 0.25 mmol of the substituted phenyl propargylic ether and CH_2Cl_2 (3 mL), 0.375 mmol of PhSeBr dissolved in 2 mL of CH_2Cl_2 was added dropwise. The mixture was allowed to stir at room temperature for the desired time. The reaction mixture was washed with 20 mL of water and extracted with diethyl ether (3 x 10 mL). The combined ether layers were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was further purified by flash chromatography on silica gel using hexanes/ethyl acetate as the eluent.

4-Phenyl-3-phenylselenyl-2*H*-benzopyran (23). This compound was obtained as a brown oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.84 (s, 2H), 6.84-6.87 (m, 2H), 6.94 (t, J = 7.4 Hz, 1H), 7.12-7.25 (m, 5H), 7.27-7.34 (m, 4H), 7.40-7.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 29.9, 71.1, 115.3, 121.4, 124.0, 128.2, 128.4, 128.8, 129.1, 129.2, 129.2, 129.3, 129.6, 134.6, 141.0, 158.4; IR (neat, cm^{-1}) 3052, 2919, 1582, 1480, 1224, 1024; HRMS m/z 364.03707 (calcd $C_{21}H_{16}OSe$, 364.03664).

4-Methyl-3-phenylselenyl-2*H*-benzopyran (41). This compound was obtained as a brown solid: mp 54-55 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.66 (s, 3H), 4.80 (s, 2H), 6.83 (d, J = 7.9 Hz, 2H), 6.92 (t, J = 7.3 Hz, 1H), 7.20-7.26 (m, 4H), 7.42-7.44 (m, 2H); ^{13}C

NMR (75 MHz, $CDCl_3$) δ 30.1, 72.1, 115.2, 121.3, 125.4, 127.6, 128.4, 129.5 (2C), 129.7, 130.2, 132.5, 158.5; IR (neat, cm^{-1}) 3057, 2914, 2847, 1593, 1485, 1229; HRMS m/z 302.02154 (calcd $C_{16}H_{14}OSe$, 302.02099).

3-(4-Fluoro-3-methylphenylethynyl)-4-phenyl-2*H*-benzopyran (44). To a solution of 0.17 g of 3-iodo-4-phenyl-2*H*-benzopyran **(2)** (0.5 mmol) in Et_3N (5 ml), was added $PdCl_2(PPh_3)_2$ (2 mol %) and CuI (1.5 mol %), and the mixture was stirred for 30 min under Ar. 0.6 Mmol of 5-ethynyl-2-fluorotoluene dissolved in 1 mL of Et_3N was then added dropwise and the reaction mixture was allowed to stir at room temperature for 24 h. The reaction was monitored by TLC and an additional 0.4 mmol of the 5-ethynyl-2-fluorotoluene dissolved in 1 mL of Et_3N was added slowly under an inert atmosphere and the reaction mixture was further allowed to stir at room temperature for another 24 h. After the reaction was over, the resulting solution was diluted with H_2O (5 ml) and extracted with diethyl ether (3 x 10 mL). The combined ether fractions were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using hexanes/ethyl acetate as the eluent to obtain the desired compound **44** in an 87% yield as a pale yellow solid: mp 79-80 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 2.18 (d, J = 1.6 Hz, 3H), 4.88 (s, 2H), 6.81-6.93 (m, 4H), 6.97-7.05 (m, 2H), 7.10-7.21 (m, 1H), 7.39-7.45 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.6 (d, J = 3.4 Hz), 68.0, 86.7 (d, J = 1.9 Hz), 95.1 (d, J = 0.8 Hz), 112.4, 115.3 (d, J = 9.4 Hz), 116.4, 118.9 (d, J = 3.8 Hz), 121.8, 124.3, 125.3 (d, J = 18.2 Hz), 126.8, 128.2 (d, J = 10.2 Hz), 129.8, 130.2, 130.8 (d, J = 8.4 Hz), 134.7 (d, J = 5.7 Hz), 136.6, 140.8, 154.6, 160.1, 162.6; IR (neat, cm^{-1}) 3047, 2919, 2192, 1480, 1224, 1106; HRMS m/z 340.12694 (calcd $C_{24}H_{17}FO$, 340.12634).

1,4-Dihydro-2,5-dioxacyclopenta[*a*]naphthalen-3-one (45). To a solution of 0.14 g of 4-hydroxymethyl-3-iodo-2*H*-benzopyran **(42)** (0.5 mmol) in DMF (5 ml) was added $PdCl_2(PPh_3)_2$ (5 mol %) and K_2CO_3 (2 equiv), and the mixture was stirred for 6 h under an atmosphere of CO at 60 ^oC. The reaction was monitored by TLC and, after completion of the reaction, the resulting solution was cooled to room temperature, diluted with ether (15 ml), and washed with brine (15 ml). The aqueous layer was extracted with diethyl ether (3 x 15 mL). The combined ether fractions were dried over anhydrous Na_2SO_4 and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using hexanes/ethyl acetate as the eluent to obtain the desired compound **45** in an 72% yield as a brown solid: mp 151-152 ^oC; ^1H NMR (400 MHz, $CDCl_3$) 5.13-5.15 (m, 4H), 6.92-6.99 (m, 2H), 7.07 (dd, J = 7.5, 1.5 Hz, 1H), 7.34 (dt, J = 8.2, 0.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 63.3, 68.8, 116.4, 117.2, 118.3, 122.0, 124.2, 133.8, 154.1, 154.8, 170.7; IR (neat, cm^{-1}) 2361, 1744, 1666, 1449, 1336, 1181, 1052; HRMS m/z 188.04776 (calcd $C_{11}H_8O_3$, 188.04743).

Reference

1. Pal, M.; Parasuraman, K.; Yeleswarapu, K. R. *Org. Lett*. **2003**, *5*, 349.

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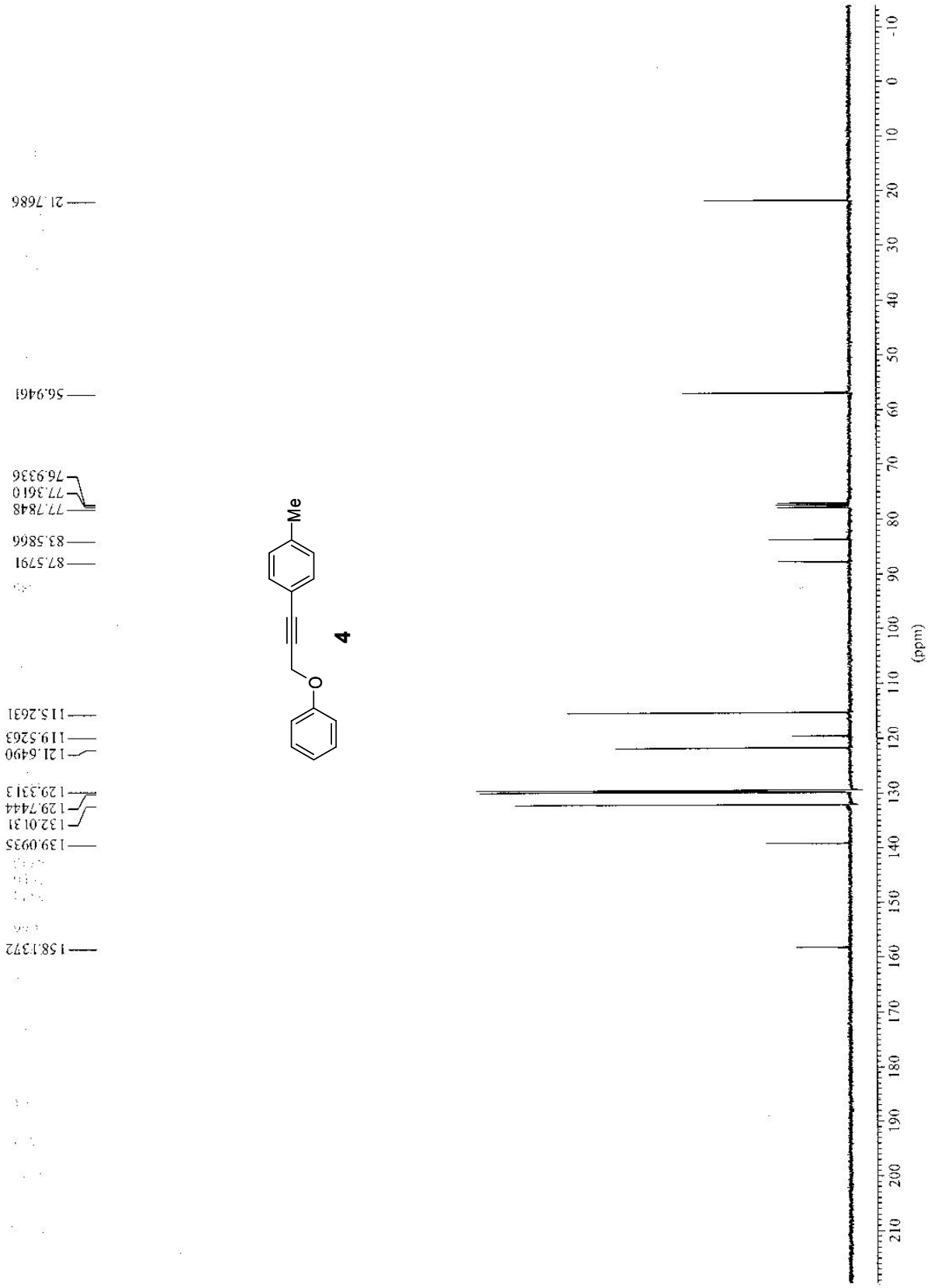
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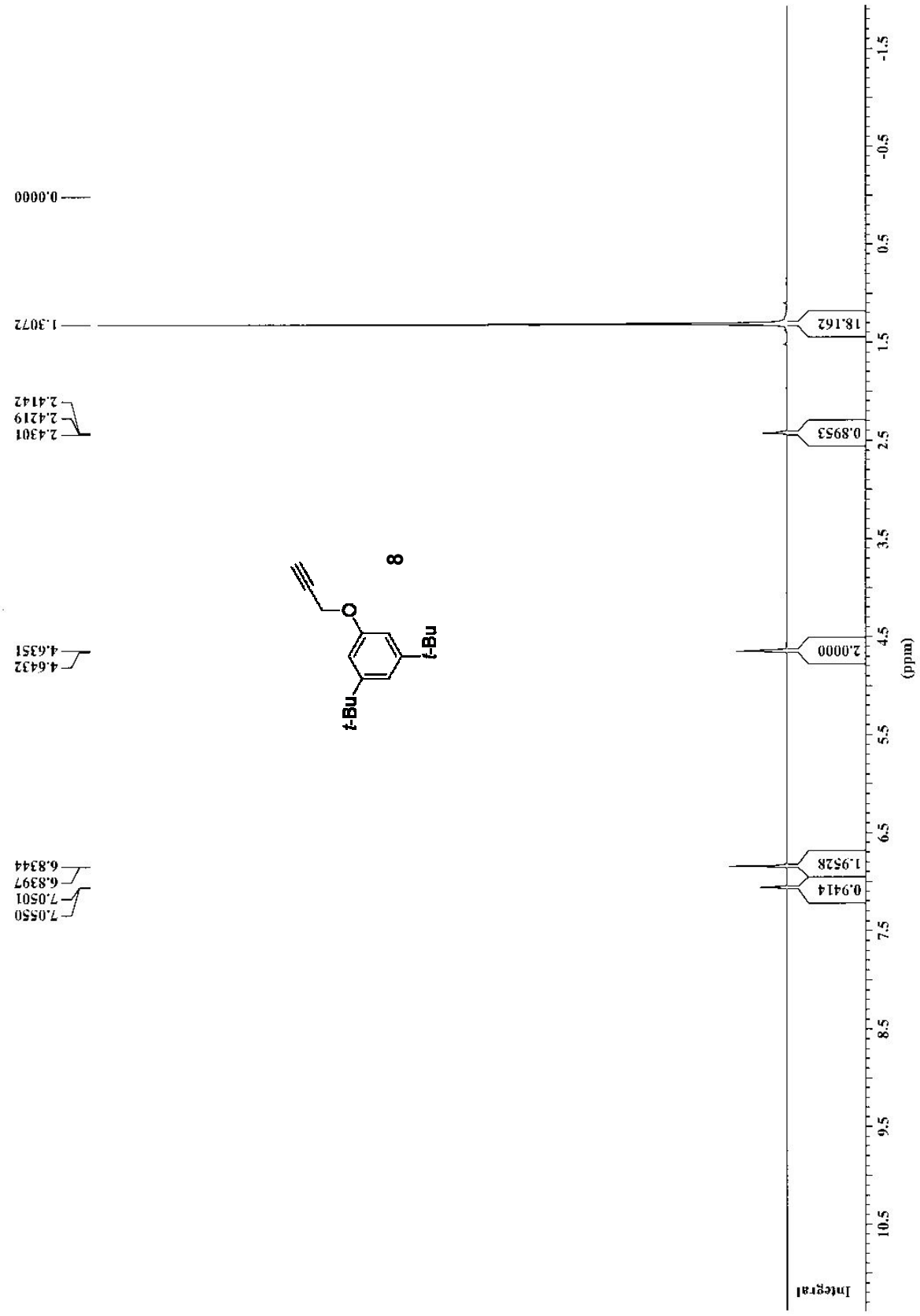
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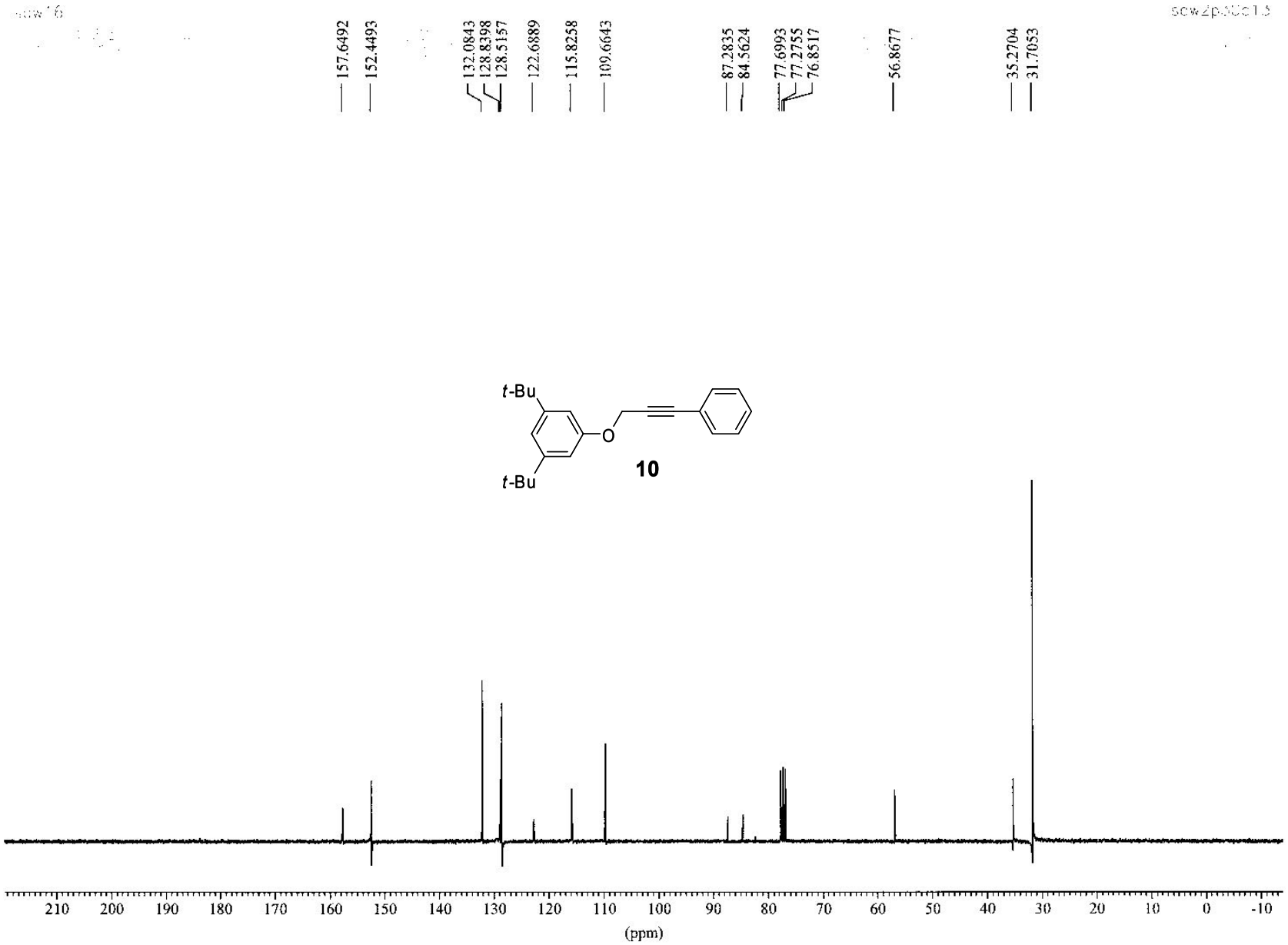
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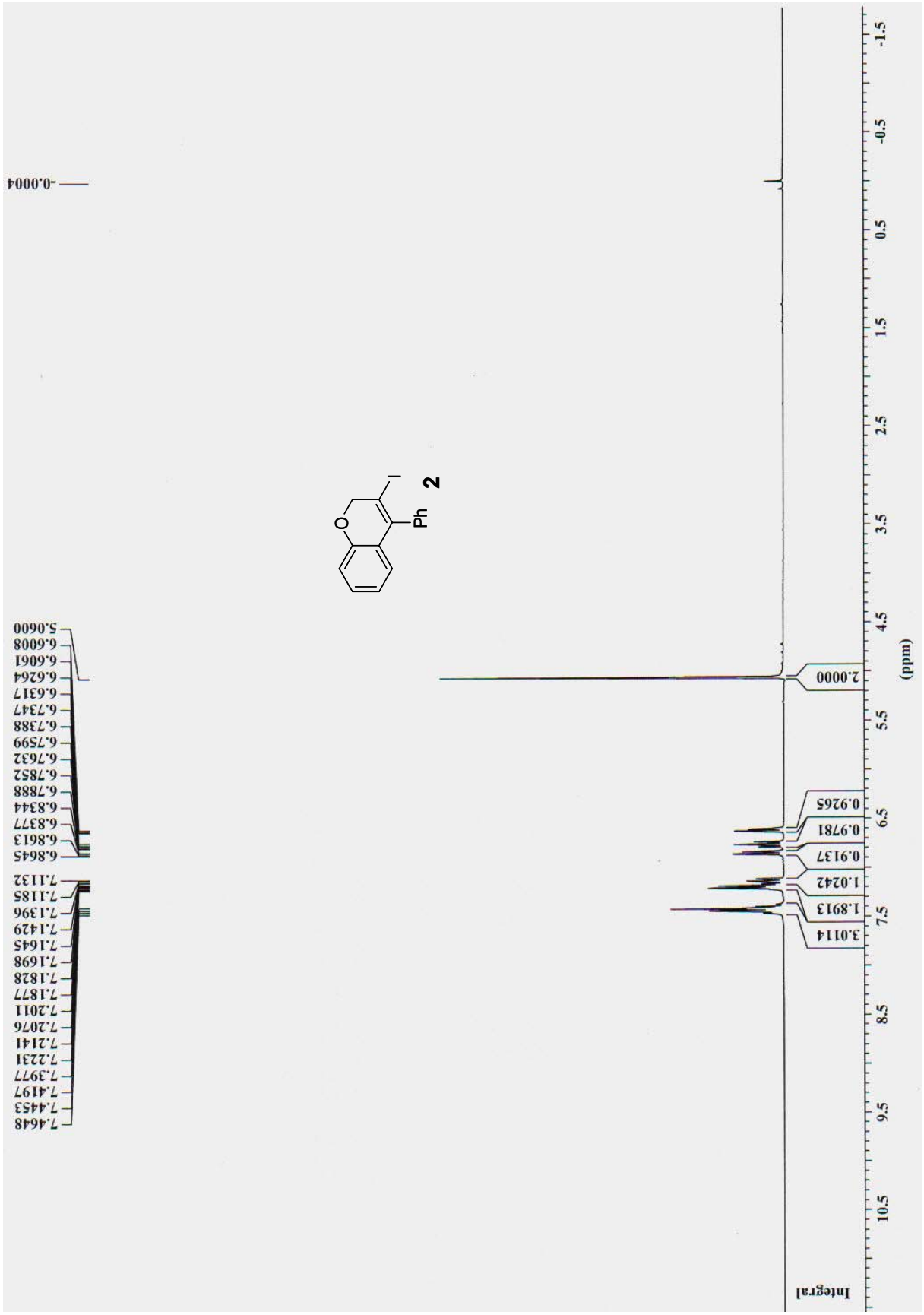
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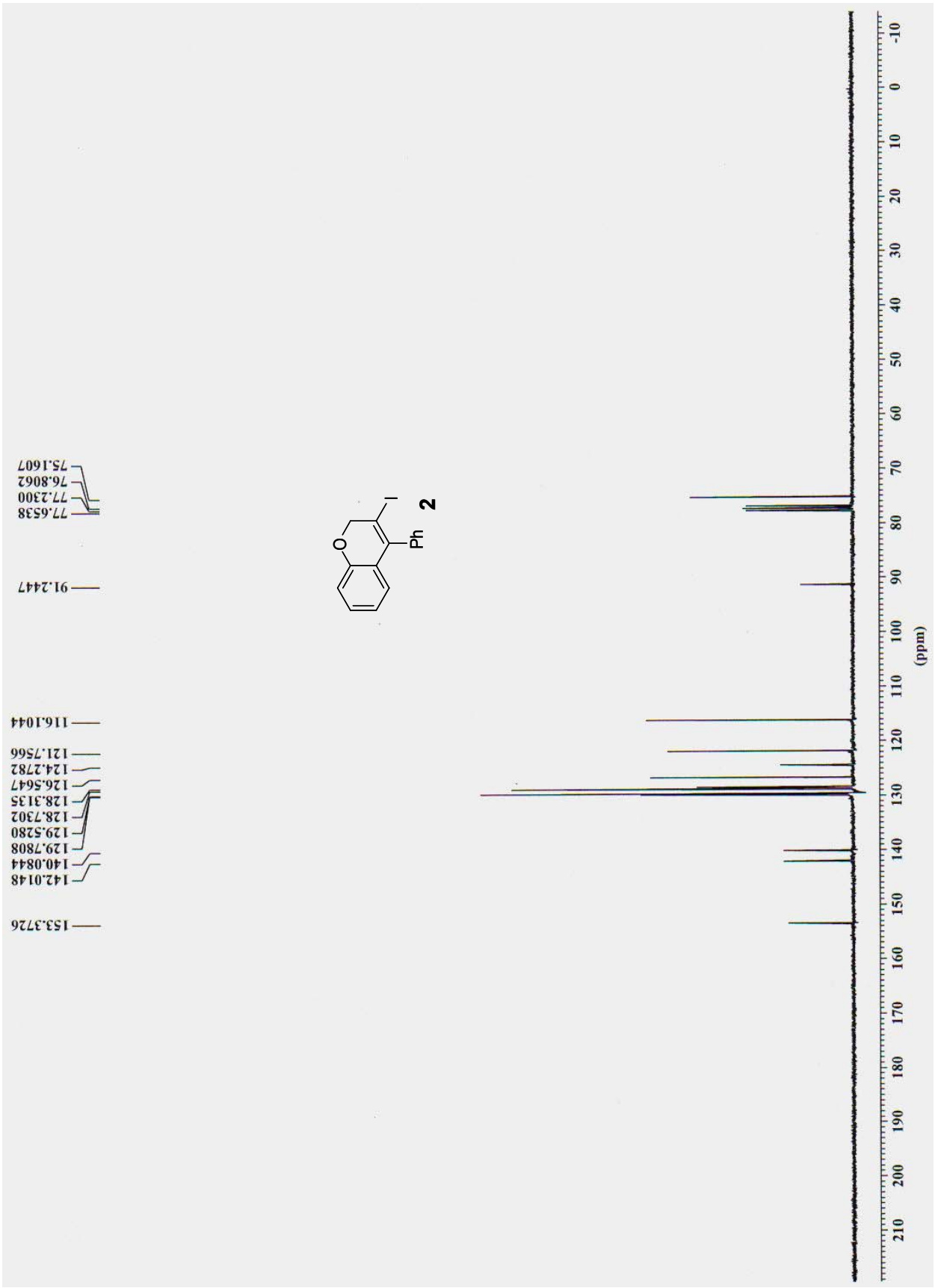
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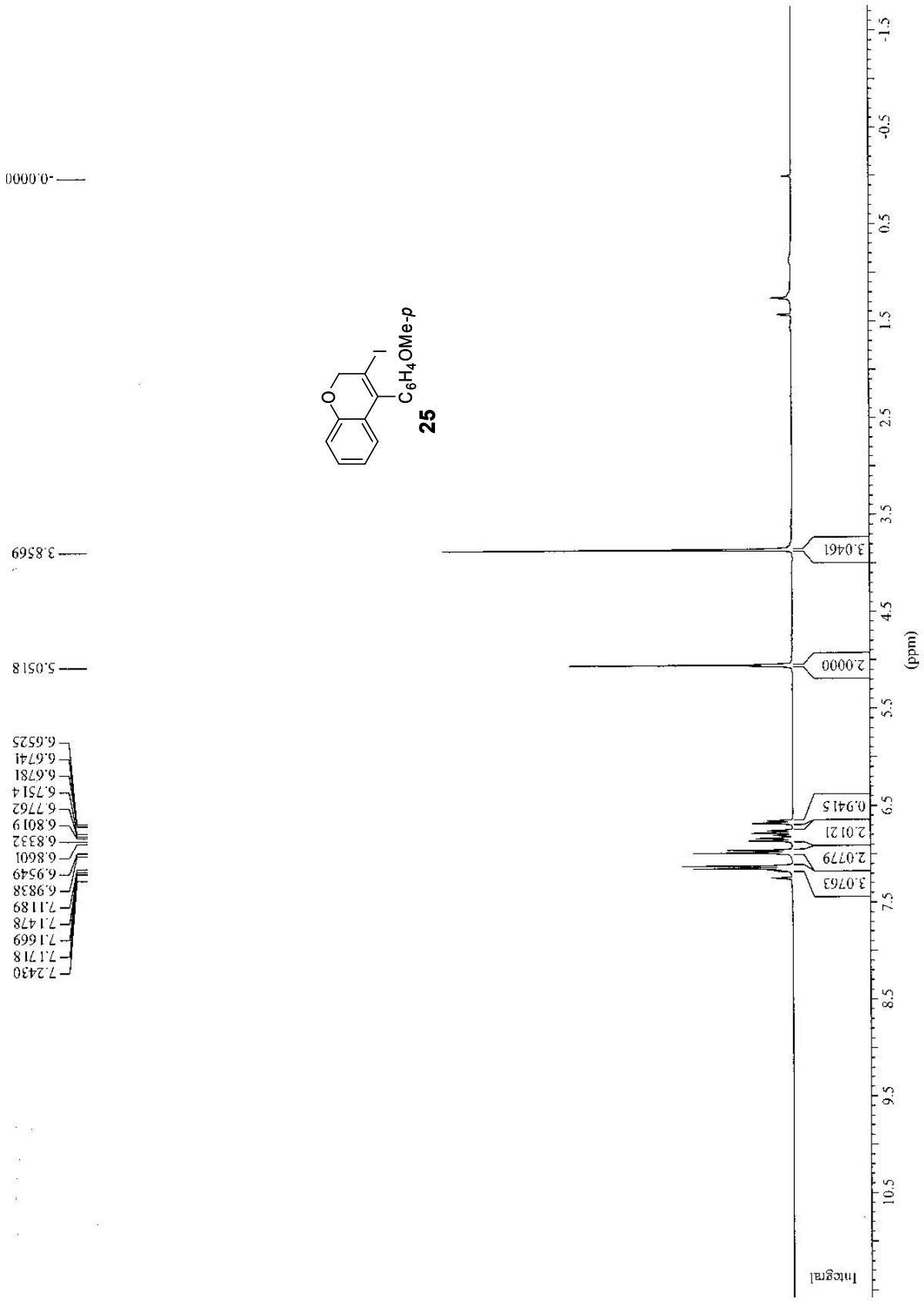
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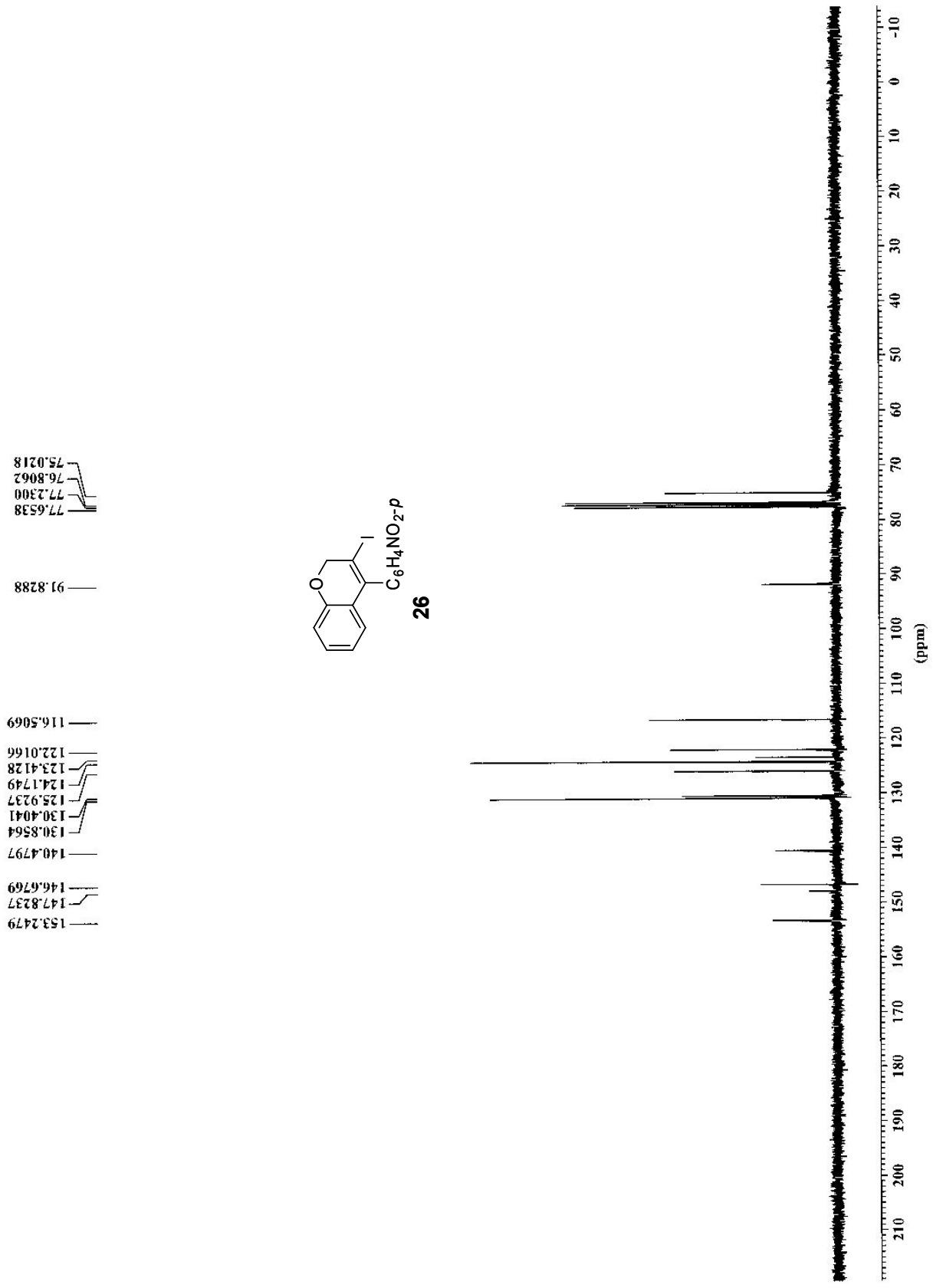
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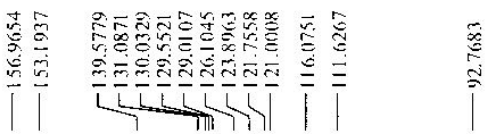
147.8237
146.6769
140.4797
130.8564
130.4041
125.9237
124.1749
123.4128
122.0166
116.5069
91.8288
77.6538
77.2300
76.8062
75.0218
O
I
C6H4NO2-p
26
(ppm)

-0.0000
3.7511
5.0148
5.0636
5.0832
5.1316
6.5548
6.5597
6.5805
6.5857
6.7180
6.7217
6.7433
6.7465
6.7685
6.7717
6.8161
6.8194
6.8430
6.8462
6.9695
6.9972
7.0049
7.0306
7.0326
7.0566
7.0599
7.0676
7.0855
7.0892
7.0941
7.1156
7.1193
7.1405
7.1458
7.2320
7.3716
7.3794
7.3952
7.3997
7.4017
7.4066
7.4225
7.4298
O
I
C6H4OMe-o
27
Integral
3.0000
2.0835
0.9256
1.9550
4.1466
0.9962
(ppm)
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5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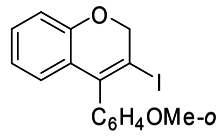
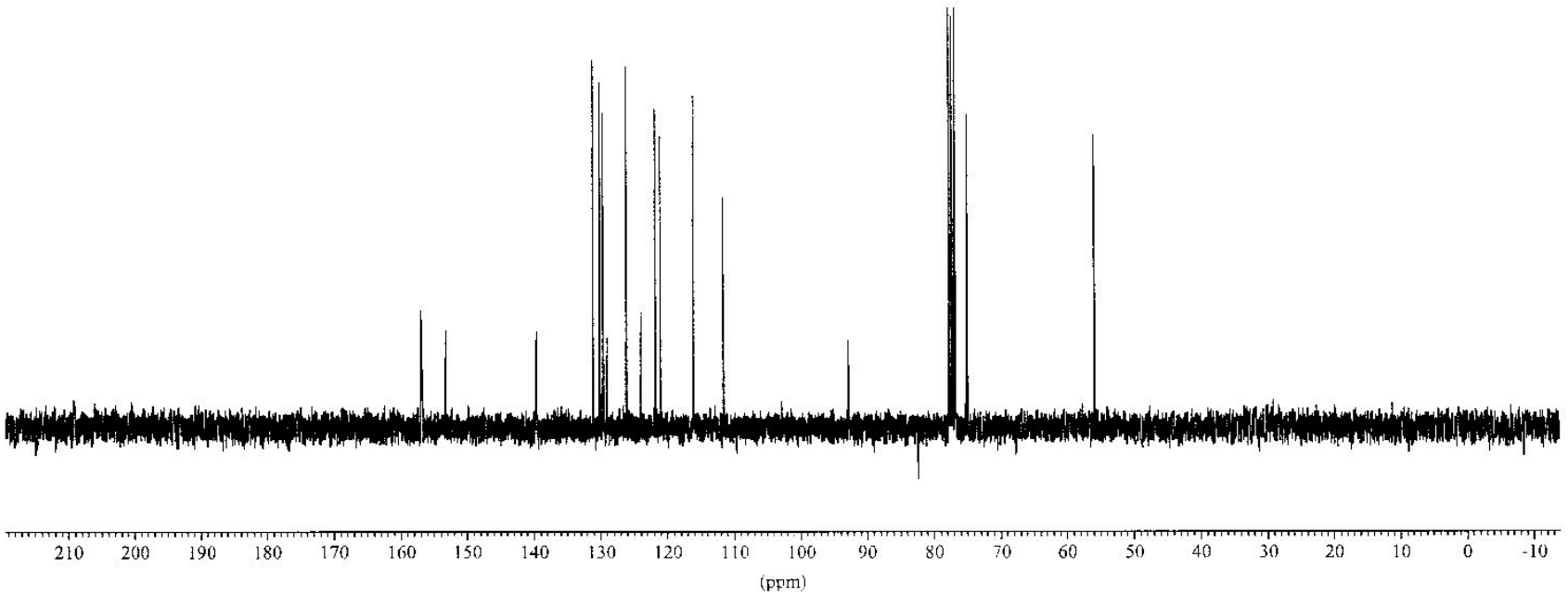
139.5779
131.0871
130.0329
129.5521
129.0107
126.1045
123.8963
121.7558
121.0008
116.0751
111.6267
92.7683
77.7171
77.2933
76.8695
74.9854
55.9631


O
I
C6H4OMe-o

27


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
(ppm)

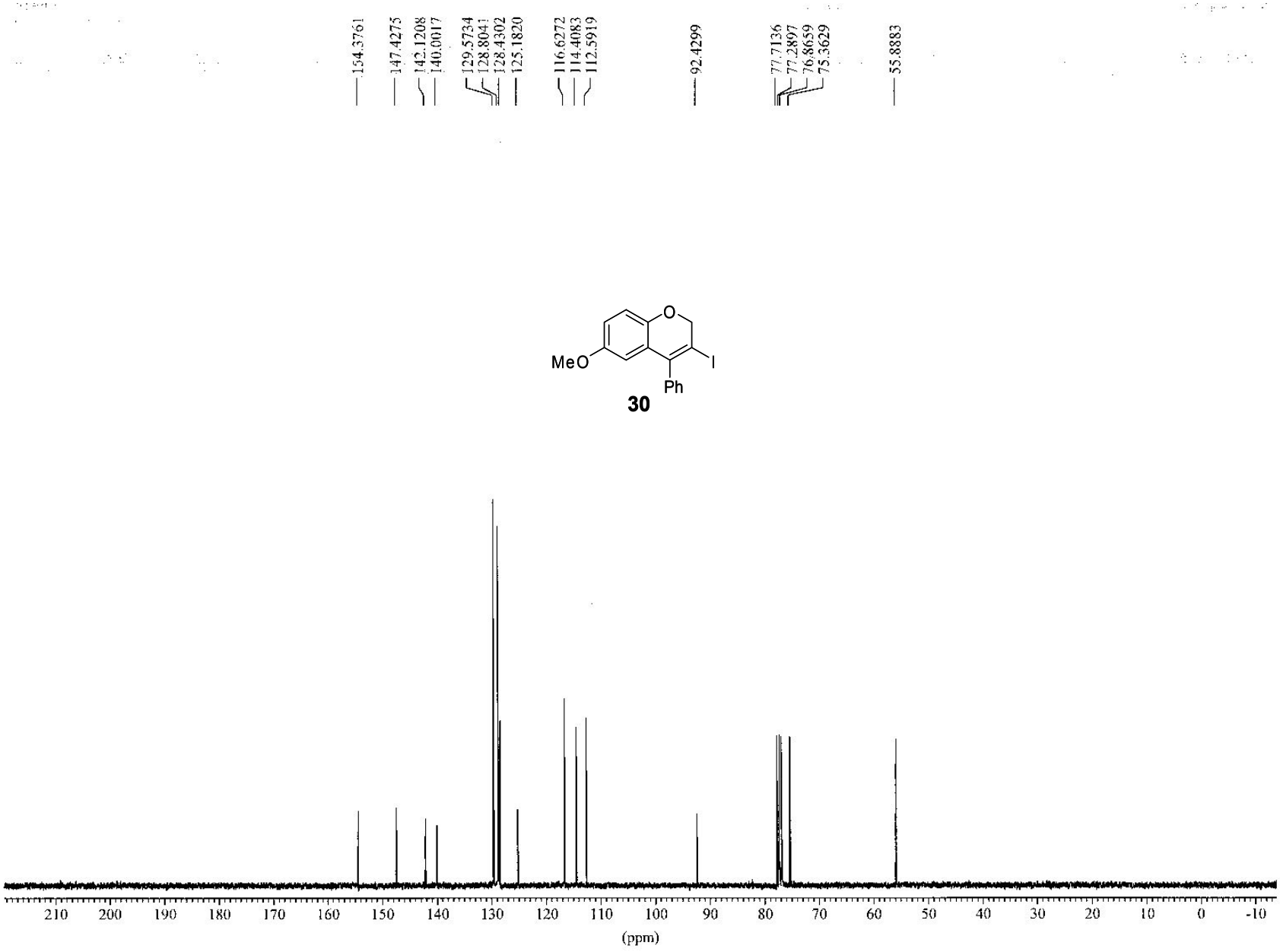
7.4656
7.4461
7.4245
7.4050
7.4005
7.2052
7.1840
7.1795
6.9561
6.9288
6.7652
6.7380
6.4136
5.0103
2.1049
0.0000
Me
O
I
Ph
28
Integral
0.9432
0.9514
1.0264
2.0270
3.2426
2.0000
2.9584
(ppm)

20.8327
75.1607
76.8062
77.2300
77.6538
91.4264
115.8480
124.1001
126.8390
128.2422
128.6981
129.5208
130.2331
131.0380
140.1948
142.1109
151.1929
I
O
Ph
Me
28
0
20
40
60
80
100
120
140
160
180
200
(ppm)

29
t-Bu
Ph
O
Integral
9.0273
2.0000
0.8893
0.9070
0.5345
2.4199
3.0297
(ppm)
1.1168
0.0000
5.0339

31.4746
34.3453
75.2533
76.8062
77.2300
77.6538
90.9278
115.4776
123.5873
123.8152
126.6289
128.3028
128.6233
129.5885
140.1094
142.4101
144.5186
151.1680
O
I
Ph
t-Bu
29
(ppm)

Integral
3.1443
2.0327
0.9739
0.9819
0.9280
2.0000
3.0906
(ppm)
7.4603
7.4396
7.4148
7.4127
7.3924
7.3895
7.2398
7.2158
7.2092
7.1893
7.1848
6.8124
6.7831
6.7119
6.7021
6.6826
6.6728
6.1991
6.1894
4.9981
3.5912
0.0000
MeO
Ph
30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147.4275
142.1208
140.0017
129.5734
128.8041
128.4302
125.1820
116.6272
114.4083
112.5919
92.4299
77.7136
77.2897
76.8659
75.3629
55.8883
MeO
O
I
Ph
30
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
(ppm)

7.4824
7.4580
7.4389
7.2598
7.2098
7.2032
7.1837
7.1788
7.1162
7.1080
7.0877
7.0791
6.8052
6.7767
6.5956
6.5871
5.0723
0.0091
Integral
2.0000
0.9085
0.9886
0.9956
2.0191
3.1369
(ppm)
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5

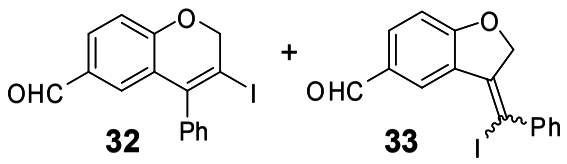
O
Ph
I
Cl

31

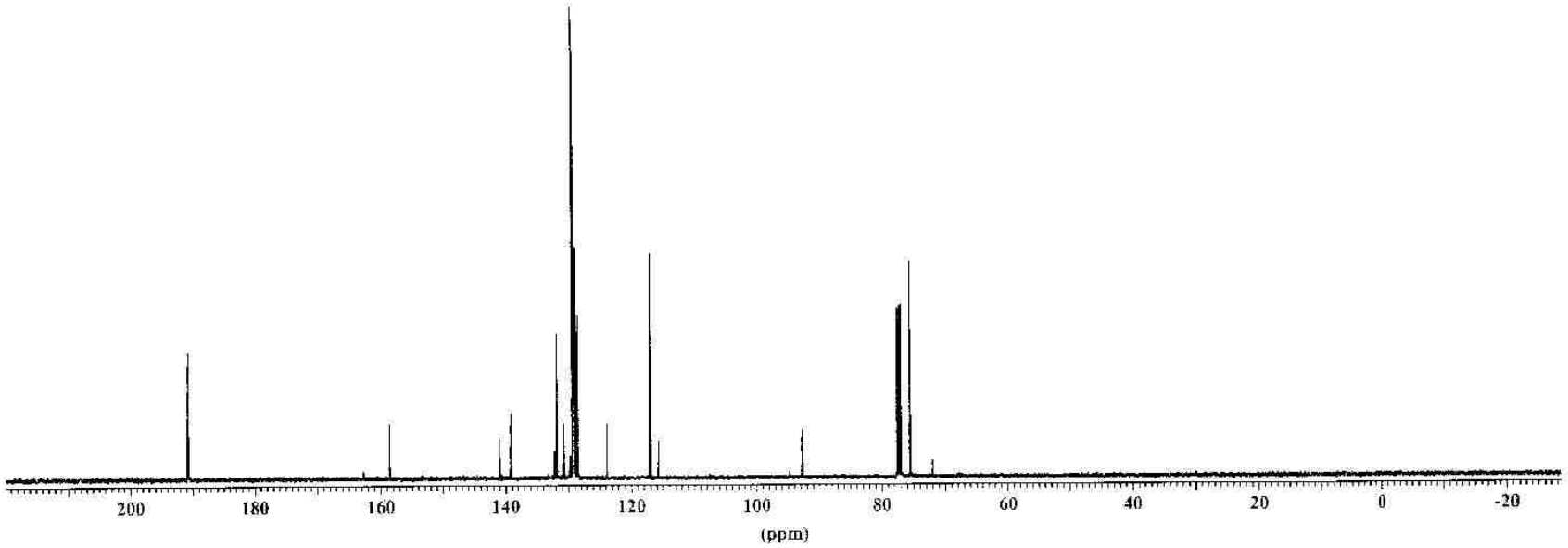
75.3344
76.8410
77.2648
77.6886
93.0033
117.4997
125.3850
126.1828
126.7313
128.6902
129.0143
129.4452
129.4666
139.4247
141.2233
151.9578
O
I
Ph
Cl
31
(ppm)

0.0031
5.1230
5.1917
6.9394
6.9603
7.1088
7.1330
7.1381
7.1934
7.1971
7.2076
7.2127
7.2170
7.2593
7.3902
7.3946
7.4335
7.4513
7.4531
7.4687
7.4733
7.4914
7.5133
7.6907
7.6955
7.7115
7.7163
7.8785
7.9003
9.6842
9.9224
OHC
Ph
I
32
+
OHC
Ph
I
33
Integral
2.1773
21.998
10.030
12.458
20.422
5.5799
33.971
11.697
1.9743
10.593
1.0000
(ppm)

190.8813
190.6197
162.7139
158.5097
140.9572
140.6236
139.1527
132.1886
131.7526
130.7935
130.7063
129.6713
129.5538
129.2922
129.0799
129.0079
128.7880
128.6023
128.4317
123.7952
116.8690
115.6256
94.6574
92.7391
77.5484
77.2300
76.9153
75.4823
71.9150



32 **33**



Integral
4.0721
1.9296
1.1700
2.0000
9.2903
9.0314
(ppm)
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
7.2752
7.2654
7.2585
7.2471
7.1283
7.1213
7.0619
6.9020
6.8955
4.8186
1.3174
0.8978
0.0000
t-Bu
t-Bu
Ph
34

31.3955
32.1469
35.1850
36.7877
76.8445
77.2684
77.3788
77.6922
85.0432
109.5610
119.9145
122.7424
127.6431
128.0135
131.1049
143.6737
143.9550
149.7853
152.5028
157.2147
t-Bu
t-Bu
Ph
34
(ppm)

Integral
3.4334
2.3076
1.0730
1.0960
1.0418
2.0000
9.4479
(ppm)
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
5.0914
1.3012
0.0380
7.5020
7.4926
7.4877
7.4727
7.4474
7.4259
7.4059
7.4002
7.2594
7.2464
7.2403
7.2203
7.2163
6.9355
6.9298
6.8439
6.8378
6.8166
6.8105
6.6050
6.5777
O
I
Ph
t-Bu
35

31.3705
35.0746
75.3380
76.8837
77.3075
77.7314
90.1292
113.3398
118.7855
121.8270
126.1971
128.3055
128.7472
129.5841
140.2367
142.0318
153.1866
153.8205
t-Bu
O
Ph
I
35
(ppm)

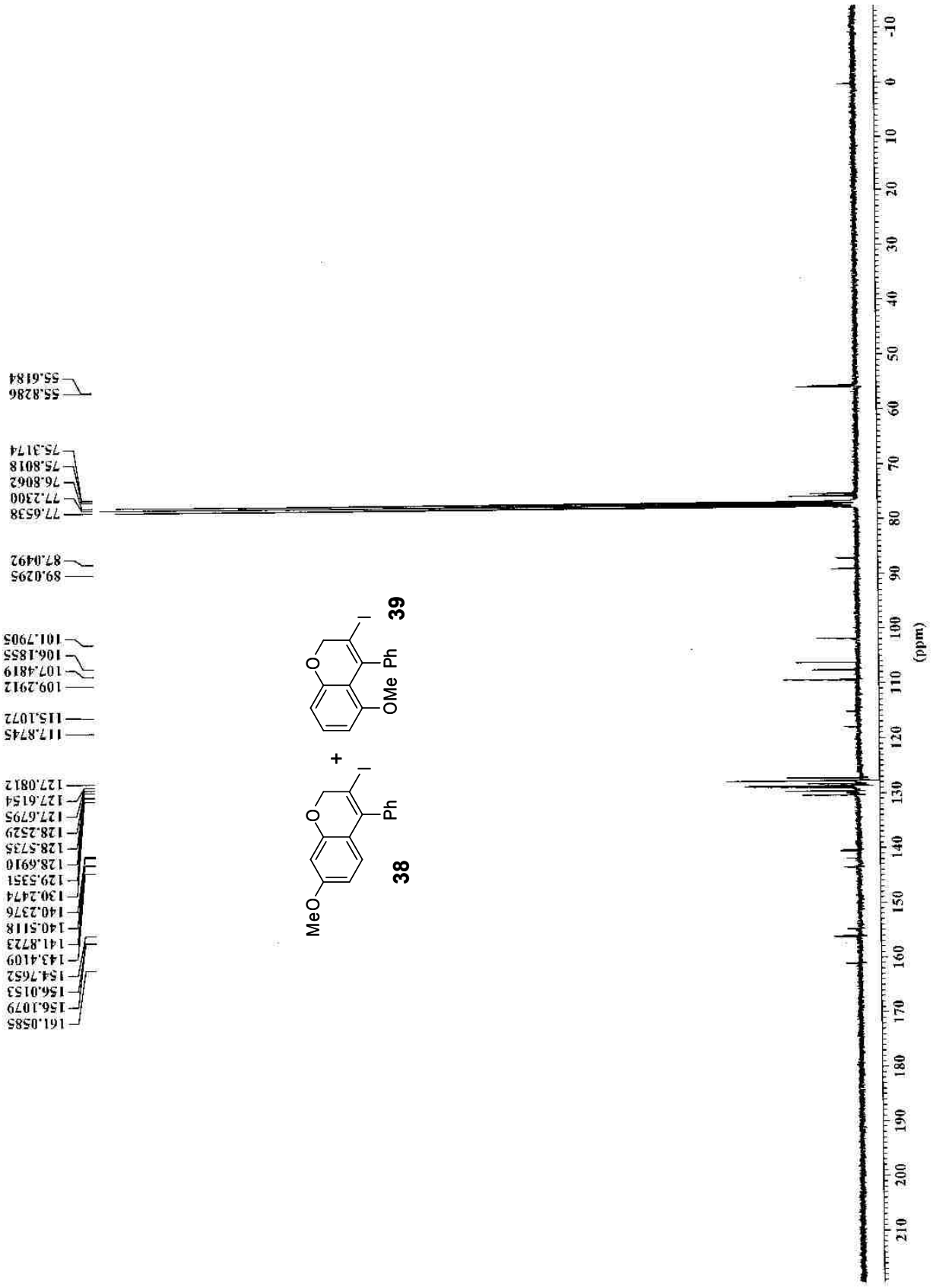
0.0000
5.0685
6.5133
6.5410
6.7091
6.7160
6.7367
6.7437
6.8519
6.8588
7.1633
7.1681
7.1877
7.1942
7.2459
7.4115
7.4306
7.4367
7.4437
7.4555
7.4689
7.4746
7.4848
Cl
O
Ph
I
36
Integral
2.0000
0.9622
0.9672
0.9216
1.9345
3.0712
(ppm)

77.6538
77.2300
76.8097
75.3424
91.2091
116.5532
121.9169
122.7467
127.4195
128.5200
128.8691
129.4318
134.7670
139.6285
141.3060
154.0066
O
I
Ph
Cl
36
(ppm)

7.3806
7.3667
7.3615
7.3501
7.3440
7.2361
7.2019
7.1881
7.1767
7.1702
7.1274
7.1006
7.0737
6.9183
6.9126
6.8946
6.8910
6.8865
4.9481
-0.0000
Cl
Ph
O
37
Integral
3.0787
1.9066
1.0255
1.9696
2.0000
(ppm)

156.6884
141.3096
140.4192
131.7433
129.9055
129.7666
128.1532
128.0107
125.0119
123.3878
115.2639
92.8368
77.6538
77.2300
76.8062
76.0761
O
I
Ph
Cl
37
(ppm)

0.0000
3.2265
3.7617
4.9318
5.0466
6.2923
6.3009
6.3212
6.3298
6.3762
6.3839
6.3989
6.4136
6.4299
6.4384
6.4645
6.4730
6.5052
6.5219
6.5503
6.5825
6.5923
6.5951
6.6110
6.6191
6.6224
7.1213
7.1319
7.1368
7.1486
7.1572
7.1633
7.1763
7.1808
7.1856
7.2060
7.2121
7.2565
7.2886
7.3041
7.3098
7.3163
7.3411
7.3566
7.3619
7.3700
7.3989
7.4140
7.4225
7.4372
MeO
Ph
38
+
OMe Ph
39
Integral
3.0000
2.1821
2.1274
1.4294
0.6560
1.6409
1.6583
4.4945
6.1588
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
(ppm)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75.3174
75.8018
76.8062
77.2300
77.6538
87.0492
89.0295
101.7905
106.1855
107.4819
109.2912
115.1072
117.8745
127.0812
127.6154
127.6795
128.2529
128.5735
128.6910
129.5351
130.2474
140.2376
140.5118
141.8723
143.4109
154.7652
156.0153
156.1079
161.0585
MeO
Ph
I
38
OMe Ph
I
39
+
(ppm)

Integral
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
(ppm)
1.0474
1.0289
1.0448
0.9487
0.9607
2.0000
4.9302
5.6017
5.6082
5.6143
5.6200
5.6265
6.7770
6.7799
6.8039
6.8067
6.8393
6.8430
6.8649
6.8678
6.8894
6.8930
7.0481
7.0530
7.0733
7.0786
7.1026
7.1079
7.1287
7.1335
7.1535
7.1592
7.2320
2.0046
2.1957
2.1851
2.0508
1.7663
1.7557
1.7411
1.7268
1.7097
1.6910
1.6764
1.6556
0.0000
4.4758
1.9771
2.2907
O
I
40

22.2146
22.8592
25.3452
27.6531
74.7227
76.8062
77.2300
77.6538
89.1577
116.1329
121.7638
122.8572
125.6245
129.2965
129.5280
137.5415
143.5035
153.5970
40
(ppm)

7.4278
7.4241
7.4022
7.3985
7.2495
7.2080
7.2035
7.1812
7.1563
7.1523
6.9801
6.9777
6.9545
6.9296
6.8360
6.8092
4.8691
4.6473
2.0666
-0.0000
42
0.9827
1.0120
1.0675
0.9350
2.1423
2.0000
1.0685
Integral
(ppm)

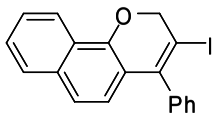
153.7537
137.3456
129.9838
124.3032
122.1698
121.8635
116.4321
93.8447
77.6538
77.2300
76.8062
75.0040
66.0360
I
CH2OH
O
42
(ppm)

Ph
O
43
Integral
0.9419
1.0810
5.2026
3.1212
0.9347
2.0000
(ppm)
0.0201
5.2713
8.2288
8.2178
8.1967
7.7352
7.7274
7.7205
7.7144
7.7038
7.5211
7.5101
7.4938
7.4869
7.4820
7.4649
7.4446
7.2773
7.2704
7.2651
7.2606
7.2517
7.2468
7.2366
6.8121
6.7837

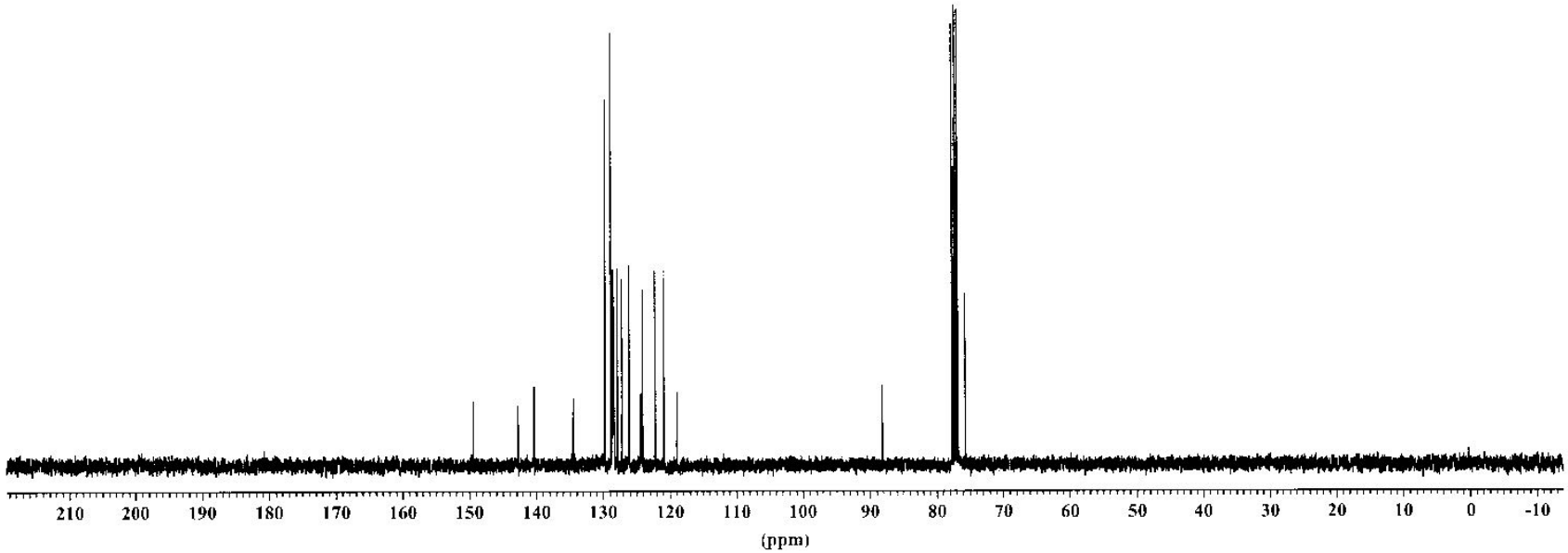
149.4015
142.7022
140.2874
134.3788
129.6348
128.7729
128.3633
127.7935
127.1382
126.0483
124.3708
124.0574
122.2125
120.8876
118.8540

88.1676

77.6538
77.2300
76.8062
75.6629



43



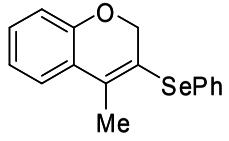
SePh
Ph
O
23
Integral
2.0000
1.9449
1.0137
4.9727
4.4209
1.9702
(ppm)
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
7.4400
7.4351
7.4152
7.4087
7.3448
7.3387
7.2780
7.2707
7.2528
7.2491
7.2308
7.2251
7.2166
7.2056
7.1962
7.1901
7.1808
7.1559
7.1417
7.1360
7.1266
6.9708
6.9459
6.9215
6.8767
6.8739
6.8478
4.8435
-0.0004

158.4150
141.0844
134.6558
129.6019
129.3918
129.2814
129.2315
129.1282
128.8504
128.4729
128.2806
124.0601
121.4353
115.3699
77.7171
77.2933
76.8730
71.1567
29.9673
SePh
Ph
O
23
(ppm)

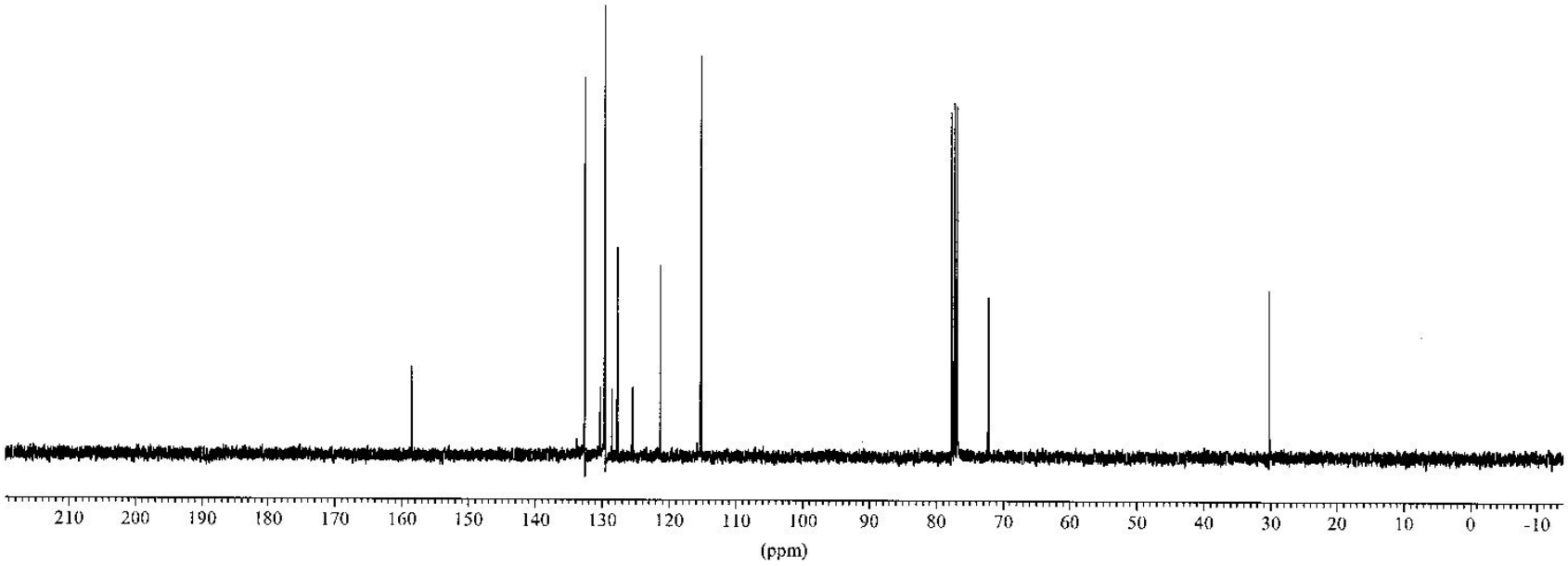
Integral
2.9346
2.0000
1.9385
1.0140
5.0590
1.9700
(ppm)
-0.0004
2.6625
4.8052
7.4547
7.4510
7.4428
7.4388
7.4315
7.4233
7.2609
7.2544
7.2369
7.2328
7.2190
7.2084
6.9516
6.9272
6.9028
6.8487
6.8222
SePh
Me
O

41

158.5076
132.5972
130.2359
129.7266
129.5948
129.5165
128.4943
127.6502
125.4242
121.3177
115.2382
77.7064
77.2826
76.8588
72.1860
30.1881



41



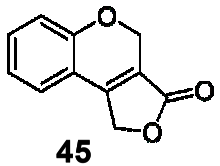
0.0000
2.1855
2.1802
7.4526
7.4473
7.4380
7.4197
7.4144
7.4083
7.3920
7.2243
7.2105
7.1803
7.1746
7.1531
7.1299
7.1242
7.1038
7.0542
7.0290
7.0115
7.0013
6.9940
6.9838
6.9777
6.9309
6.9240
6.9174
6.9032
6.8975
6.8910
6.8601
6.8377
6.8295
6.8149
6.8116
4.8817
Me
F
O
44
Integral
3.0447
2.0000
4.2214
2.1040
1.3206
4.9639
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
-1.5
(ppm)

14.5756
14.5415
68.0064
76.9153
77.2300
77.5484
86.7038
86.7227
95.1161
95.1237
112.3842
115.2465
115.4777
116.3534
118.9427
118.9806
121.7557
124.3449
125.2320
125.4140
126.8470
128.1587
128.2611
129.8154
130.2134
130.7593
130.8428
134.6414
134.6982
136.6127
140.8473
154.6011
160.0981
162.5699
Me
F
O
44
-20
0
20
40
60
80
100
120
140
160
180
200
(ppm)

7.3615
7.3576
7.3409
7.3389
7.3224
7.3183
7.2654
7.0764
7.0725
7.0574
7.0537
6.9874
6.9851
6.9686
6.9663
6.9473
6.9261
6.9247
5.1543
5.1499
5.1394
5.1348
-0.0000
4.1170
1.0000
0.9948
1.9126
Integral
(ppm)
45

s35bc

170.6713
154.8324
154.1121
133.8490
124.2198
122.0438
118.2906
117.2216
116.4368
77.5484
77.2300
76.9116
68.8291
63.2601



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