

Hypoiodous acid initiated rearrangement of tertiary propargylic alcohols to α -iodoenones

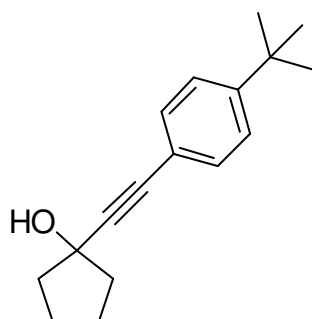
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General. ^1H NMR spectra were recorded at either 400 or 500 MHz. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl_3 : 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), and coupling constants (Hz). ^{13}C NMR were recorded with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard (CDCl_3 : 77.4 ppm). Mass spectrometry (m/z) was performed in ESI mode, with only molecular ions being reported. Infrared (IR) spectra ν_{max} are reported in cm^{-1} . Bands are characterized as broad (br), strong (s), medium (m) and weak (w). All purchased reagents were used as received without further purification. THF was dried with 3\AA molecular sieves then distilled from sodium benzophenone ketyl. Petroleum ether refers to the fraction boiling at 40-60 °C. **2o**¹ is a known compound and its analytical data matched that in the literature.

Representative procedure for the preparation of the starting materials: Synthesis of 1-((4-tert-butylphenyl)ethynyl)cyclopentanol, 2d



¹ T. Ishikawa, T. Mizuta, K. Hagiwara, T. Aikawa, T. Kudo and S. Saito, *J. Org. Chem.*, 2003, **68**, 3702.

4-*tert*-Butylphenylacetylene (0.88 mL, 4.9 mmol) was dissolved in THF (20 mL) at -78 °C under a N₂ atmosphere. *n*-BuLi (2.2 M, 2.2 mL, 4.9 mmol) was added dropwise. After 0.5 hours, the resulting mixture was added *via* cannula to a solution of cyclopentanone (0.43 mL, 4.9 mmol) in THF (20 mL) and stirred at -78 °C for a further 2 hours. The reaction was quenched by addition of water, extracted with EtOAc, dried (MgSO₄), filtered and concentrated. The residue was purified by flash chromatography (silica gel; 20:1 petroleum ether/EtOAc) to provide a white solid (1.06 g, 89%).

Mp: 65-67 °C

IR (neat): 997 (s), 2958 (m), 3267 (br) cm⁻¹.

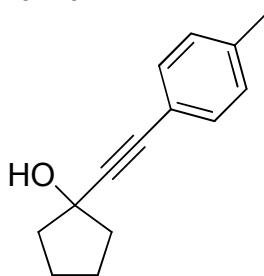
¹H NMR (500 MHz, CDCl₃): δ 1.30 (9H, s), 1.72-2.11 (9H, m), 7.32 (2H, d, *J* = 9.5 Hz), 7.36 (2H, d, *J* = 9.5 Hz).

¹³C NMR (126 MHz, CDCl₃): δ 22.9 (2C), 30.6 (3C), 34.1, 42.0 (2C), 74.4, 82.6, 91.6, 119.2, 124.6 (2C), 130.7 (2C), 150.8.

MS: *m/z* (M+23) 265.2

HRMS: *m/z* calc'd for C₁₇H₂₂NaO 265.1563, found 265.1560

Data for 1-(*p*-tolylethynyl)cyclopentanol **2e**



Mp: 55-57 °C

IR (neat): 990 (s), 2963 (m), 3283 (br) cm⁻¹.

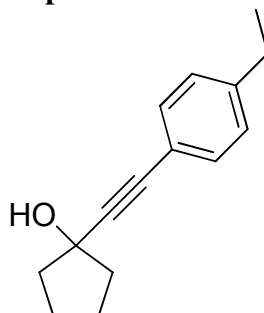
¹H NMR (500 MHz, CDCl₃): δ 1.72-1.92 (5H, m), 1.96-2.11 (4H, m), 2.34 (3H, s), 7.10 (2H, d, *J* = 7.9 Hz), 7.31 (2H, d, *J* = 8.1 Hz).

¹³C NMR (126 MHz, CDCl₃): δ 21.8, 23.9 (2C), 43.0 (2C), 75.4, 83.6, 92.5, 120.1, 129.4 (2C), 131.9 (2C), 138.6.

MS: m/z (M+23) 223.1

HRMS: m/z calc'd for $C_{14}H_{16}NaO$ 223.1093, found 223.1089

Data for 1-((4-ethylphenyl)ethynyl)cyclopentanol 2f



Mp: 50-52 °C

IR (neat): 991 (s), 3966 (m), 3276 (br) cm^{-1} .

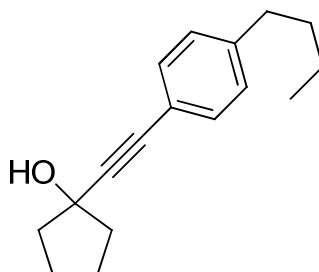
1H NMR (500 MHz, $CDCl_3$): δ 1.22 (3H, t, $J = 7.6$ Hz), 1.72-1.94 (5H, m), 1.96-2.12 (4H, m), 2.63 (2H, q, $J = 7.6$ Hz), 7.13 (2H, d, $J = 8.2$ Hz), 7.34 (2H, d, $J = 8.2$ Hz).

^{13}C NMR (126 MHz, $CDCl_3$): δ 15.7, 23.9 (2C), 29.2, 43.0 (2C), 75.4, 83.7, 92.5, 120.4, 128.2 (2C), 132.0 (2C), 145.0.

MS: m/z (M+23) 237.1

HRMS: m/z calc'd for $C_{15}H_{18}NaO$ 237.1250, found 237.1255

Data for 1-((4-butylphenyl)ethynyl)cyclopentanol 2g



Oil

IR (neat): 992 (s), 2929 (m), 2956 (m), 3341 (br) cm^{-1} .

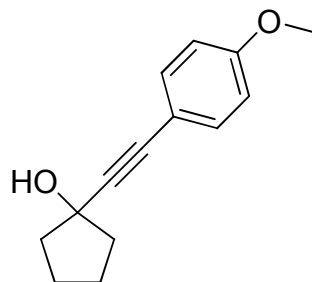
^1H NMR (500 MHz, CDCl_3): δ 0.92 (3H, t, $J = 7.4$ Hz), 1.34 (2H, hexet, $J = 7.6$ Hz), 1.53-1.62 (2H, m), 1.72-1.92 (4H, m), 1.97-2.11 (5H, m), 2.59 (2H, t, $J = 7.8$ Hz), 7.11 (2H, d, $J = 8.2$ Hz), 7.33 (2H, d, $J = 8.2$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 14.3, 22.7, 23.9 (2C), 33.8, 35.9, 43.0 (2C), 75.4, 83.7, 92.6, 120.3, 128.7 (2C), 131.9 (2C), 143.7.

MS: m/z ($M+23$) 265.2

HRMS: m/z calc'd for $\text{C}_{17}\text{H}_{22}\text{NaO}$ 265.1563, found 265.1550

Data for 1-((4-methoxyphenyl)ethynyl)cyclopentanol 2h



Oil

IR (neat): 988 (s), 1170 (s), 1247 (s), 1509 (s), 1608 (m), 2954 (w), 3297 (br) cm^{-1} .

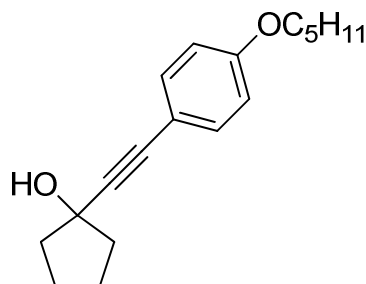
^1H NMR (500 MHz, CDCl_3): δ 1.71-1.91 (4H, m), 1.96-2.10 (5H, m), 3.80 (3H, s), 6.82 (2H, d, $J = 8.8$ Hz), 7.35 (2H, d, $J = 8.8$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 23.9 (2C), 42.9 (2C), 55.6, 75.3, 83.3, 91.8, 114.2 (2C), 115.4, 133.4 (2C), 159.8.

MS: m/z ($M+23$) 239.1

HRMS: m/z calc'd for $\text{C}_{14}\text{H}_{16}\text{NaO}_2$ 239.1043, found 239.1046

Data for 1-((4-(pentyloxy)phenyl)ethynyl)cyclopentanol 2i



Oil

IR (neat): 1244 (s), 1508 (s), 1606 (m), 2955 (m), 3380 (br) cm^{-1} .

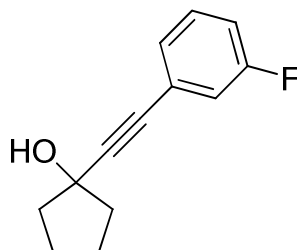
^1H NMR (500 MHz, CDCl_3): δ 0.93 (3H, t, $J = 7.2$ Hz), 1.32-1.47 (4H, m), 1.71-1.91 (6H, m), 1.96-2.10 (5H, m), 3.93 (2H, t, $J = 6.6$ Hz), 6.81 (2H, d, $J = 8.9$ Hz), 7.33 (2H, d, $J = 8.9$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 14.4, 22.8, 23.9 (2C), 28.5, 29.3, 42.9 (2C), 68.4, 75.3, 82.4, 91.7, 114.8 (2C), 115.1, 133.4 (2C), 159.5.

MS: m/z ($M+23$) 295.2

HRMS: m/z calc'd for $\text{C}_{18}\text{H}_{24}\text{NaO}_2$ 295.1669, found 295.1662

Data for 1-((3-fluorophenyl)ethynyl)cyclopentanol 2j



Oil

IR (neat): 992 (s), 1579 (s) 1608 (m), 2965 (w), 3332 (br) cm^{-1} .

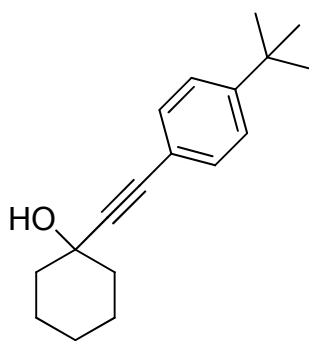
^1H NMR (500 MHz, CDCl_3): δ 1.75-1.96 (4H, m), 1.99-2.13 (5H, m), 7.03 (1H, tdd, $J = 8.5, 2.6, 1.2$ Hz), 7.14 (1H, ddd, $J = 9.6, 2.6, 1.4$ Hz), 7.22 (1H, dt, $J = 7.7, 1.3$ Hz), 7.25-7.31 (1H, m).

^{13}C NMR (126 MHz, CDCl_3): δ 23.9 (2C), 42.9 (2C), 75.2, 82.3 (d, $J = 3.0$ Hz), 94.2, 115.9 (d, $J = 20$ Hz), 118.8 (d, $J = 23$ Hz), 125.1 (d, $J = 9.6$ Hz), 127.9 (d, $J = 2.6$ Hz), 130.2 (d, $J = 9.6$ Hz), 162.6 (d, $J = 247$ Hz).

MS: m/z ($M+23$) 227.1

HRMS: m/z calc'd for $\text{C}_{13}\text{H}_{13}\text{FNaO}$ 227.0843, found 227.0840

Data for 1-((4-*tert*-butylphenyl)ethynyl)cyclohexanol 2k



Mp: 120-122 °C

IR (neat): 962 (s)m 1069 (m), 2930 (m), 3248 (br) cm^{-1} .

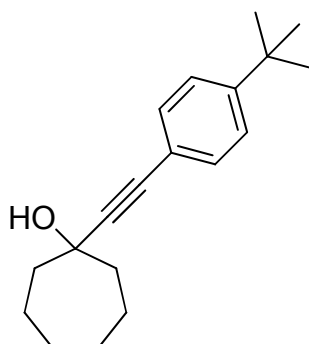
^1H NMR (500 MHz, CDCl_3): δ 1.30 (9H, s), 1.51-1.78 (8H, m), 1.95-2.08 (3H, m), 7.32 (2H, d, $J = 8.7$ Hz), 7.37 (2H, d, $J = 8.6$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 23.8 (2C), 25.6, 31.5 (3C), 35.1, 40.5 (2C), 69.5, 84.9, 92.5, 120.2, 125.6 (2C), 131.8 (2C), 151.9.

MS: m/z ($M+23$) 279.2

HRMS: m/z calc'd for $\text{C}_{18}\text{H}_{24}\text{NaO}$ 279.1719, found 279.1720

Data for 1-((4-*tert*-butylphenyl)ethynyl)cycloheptanol 2m



Mp: 89-90 °C

IR (neat): 1028 (s), 2923 (m), 3243 (br) cm^{-1} .

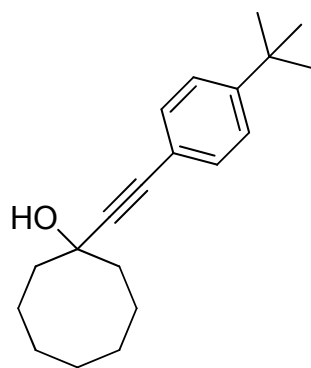
^1H NMR (500 MHz, CDCl_3): δ 1.35 (9H, s), 1.55-1.82 (8H, m), 1.94-2.04 (2H, m), 2.14-2.23 (2H, m), 2.92 (1H, br), 7.35 (2H, *d*, $J = 8.4$ Hz), 7.44 (2H, *d*, $J = 8.4$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 22.6 (2C), 28.1 (2C), 31.4 (3C), 34.9, 43.4 (2C), 72.4, 83.9, 93.6, 120.3, 125.4 (2C), 131.6 (2C), 151.4.

MS: m/z ($M+23$) 293.2

HRMS: m/z calc'd for $\text{C}_{19}\text{H}_{26}\text{NaO}$ 293.1876, found 293.1876

Data for 1-((4-*tert*-butylphenyl)ethynyl)cyclooctanol 2n



Mp: 115-116 °C

IR (neat): 980.1 (s), 2916 (m), 3264 (br) cm^{-1} .

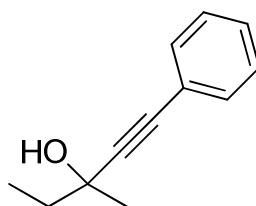
^1H NMR (500 MHz, CDCl_3): δ 1.34 (9H, s), 1.49-1.60 (3H, m), 1.62-1.81 (7H, m), 2.00-2.14 (4H, m), 2.45 (1H, br), 7.34 (2H, *d*, $J = 8.5$ Hz), 7.41 (2H, *d*, $J = 8.4$ Hz).

^{13}C NMR (126 MHz, CDCl_3): δ 22.5 (2C), 24.8, 28.3 (2C), 31.4 (3C), 34.9, 38.7 (2C), 72.0, 83.6, 93.5, 120.3, 125.5 (2C), 131.7 (2C), 151.5.

MS: m/z ($M+23$) 307.2

HRMS: m/z calc'd for $\text{C}_{20}\text{H}_{28}\text{NaO}$ 307.2032, found 307.2036

Data for 3-methyl-1-phenylpent-1-yn-3-ol 2p



Colourless oil

IR (neat): 1124 (m), 1153 (m), 2971 (m), 3362 (br) cm^{-1} .

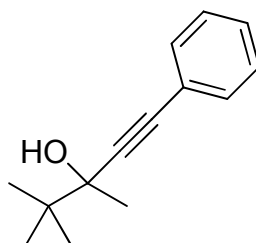
^1H NMR (400 MHz, CDCl_3): δ 1.11 (3H, t, $J = 7.4$ Hz), 1.57 (3H, s), 1.79 (2H, qd, $J = 7.3, 2.9$ Hz), 2.13 (1H, br), 7.27-7.33 (3H, m), 7.38-7.46 (2H, m).

^{13}C NMR (100 MHz, CDCl_3): δ 9.5, 29.7, 37.0, 69.5, 83.7, 93.0, 123.1, 128.6 (3C), 132.0 (2C).

MS: m/z (M+23) 197.1

HRMS: m/z calc'd for $\text{C}_{12}\text{H}_{14}\text{NaO}$ 197.0937, found 197.0941

Data for 3,4,4-trimethyl-1-phenylpent-1-yn-3-ol 2q



Colourless oil

IR (neat): 901 (m), 1070 (m), 2968 (m), 3453 (br) cm^{-1} .

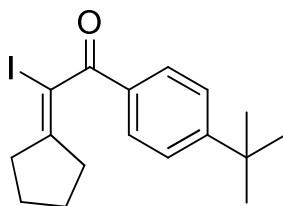
^1H NMR (400 MHz, CDCl_3): δ 1.12 (9H, s), 1.54 (3H, s), 1.99 (1H, s), 7.27-7.34 (3H, m), 7.38-7.47 (2H, m).

^{13}C NMR (100 MHz, CDCl_3): δ 25.1, 25.6 (3C), 38.9, 74.7, 84.2, 93.2, 123.3, 128.5, 128.6 (2C), 134.0 (2C).

MS: m/z (M+23) 225.1

HRMS: m/z calc'd for $\text{C}_{14}\text{H}_{18}\text{NaO}$ 225.1250, found 225.1253

General procedure for the oxidative rearrangement to α -iodoenones 4: Synthesis of 1-(4-*tert*-butylphenyl)-2-cyclopentylidene-2-iodoethanone **4d.**



1-(4-*tert*-Butylphenyl)ethynyl)cyclopentanol **2d** (0.21 mmol), *m*-CPBA (109 mg, 0.63 mmol), sodium iodide (31 mg, 0.21 mmol) and trichloroacetic acid (51 mg, 0.31 mmol) were dissolved in acetonitrile (1 mL) at room temperature under a nitrogen atmosphere and stirred overnight. The reaction mixture was quenched with saturated aqueous sodium thiosulfate solution and extracted with CH₂Cl₂. The organic layer was washed with saturated aqueous sodium bicarbonate solution, dried over MgSO₄, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel; 9:1 petroleum ether/ethyl acetate) to afford 1-(4-*tert*-butylphenyl)-2-cyclopentylidene-2-iodoethanone **4d** as a pale yellow oil (58 mg, 75%).

IR (neat): 1108 (m), 1187 (m), 1255 (s), 1602 (s), 1656 (s), 2959 (w) cm⁻¹.

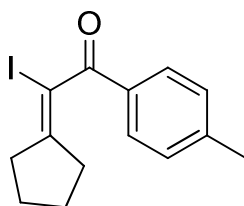
¹H NMR (400 MHz, CDCl₃): δ 1.35 (9H, s), 1.72-1.86 (4H, m), 2.26 (2H, t, J = 6.6 Hz), 2.53 (2H, t, J = 7.3 Hz), 7.47 (2H, d, J = 8.6 Hz), 7.86 (2H, d, J = 8.6 Hz).

¹³C NMR (100 MHz, CDCl₃): δ 25.8, 29.0, 31.4 (3C), 34.3, 35.6, 41.1, 85.8, 126.1 (2C), 130.3 (2C), 132.1, 157.8, 158.2, 193.1.

MS: m/z (M+23) 391.1

HRMS: m/z calc'd for C₁₇H₂₁INaO 391.0529, found 391.0542.

Data for 2-cyclopentylidene-2-iodo-1-*p*-tolylethanone **4e**



Oil

IR (neat): 1175 (s), 1243 (s), 1601 (s), 1652 (s), 2952 (w) cm⁻¹.

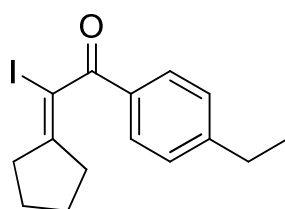
^1H NMR (400 MHz, CDCl_3): δ 1.71-1.86 (4H, m), 2.21-2.28 (2H, m), 2.42 (3H, s), 2.49-2.56 (2H, m), 7.26 (2H, d, $J = 7.9$ Hz), 7.82 (2H, d, $J = 8.2$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 22.3, 25.9, 29.1, 34.5, 41.2, 86.1, 129.8 (2C), 130.5 (2C), 132.5, 144.8, 158.5, 193.1.

MS: m/z ($M+23$) 349.0

HRMS: m/z calc'd for $\text{C}_{14}\text{H}_{15}\text{INaO}$ 349.0060, found 349.0065.

Data for 2-cyclopentylidene-1-(4-ethylphenyl)-2-iodoethanone 4f



Oil

IR (neat): 1060 (m), 1177 (m), 1253 (s), 1602 (s), 1655 (s), 2869 (w), 2961 (w) cm^{-1} .

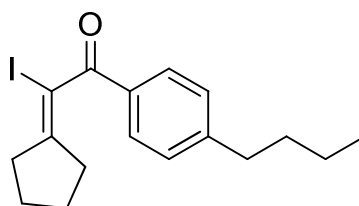
^1H NMR (400 MHz, CDCl_3): δ 1.26 (3H, t, $J = 7.6$ Hz), 1.77-1.87 (4H, m), 2.25 (2H, t, $J = 6.3$ Hz), 2.53 (2H, t, $J = 7.1$ Hz), 2.67-2.77 (2H, m), 7.28 (2H, d, $J = 8.1$ Hz), 7.85 (2H, d, $J = 8.2$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 15.5, 25.8, 29.0, 29.4, 34.4, 41.1, 85.9, 128.6 (2C), 130.5 (2C), 132.4, 151.0, 158.4, 193.2.

MS: m/z ($M+23$) 363.0

HRMS: m/z calc'd for $\text{C}_{15}\text{H}_{17}\text{INaO}$ 363.0216, found 363.0220.

Data for 1-(4-butylphenyl)-2-cyclopentylidene-2-iodoethanone 4g



Oil

IR (neat): 1177 (m), 1248 (m), 1603 (s), 1656 (s), 2954 (m) cm^{-1} .

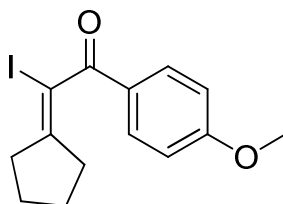
^1H NMR (400 MHz, CDCl_3): δ 0.93 (3H, t, $J = 7.3$ Hz), 1.31-1.42 (2H, m), 1.56-1.70 (2H, m), 1.72-1.85 (4H, m), 2.25 (2H, t, $J = 6.1$ Hz), 2.52 (2H, t, $J = 7.2$ Hz), 2.67 (2H, t, $J = 7.7$ Hz), 7.26 (2H, d, $J = 8.3$ Hz), 7.83 (2H, d, $J = 8.3$ Hz)..

^{13}C NMR (100 MHz, CDCl_3): δ 14.3, 22.8, 25.8, 29.1, 33.6, 34.4, 36.2, 41.1, 85.9, 129.2 (2C), 130.5 (2C), 132.4, 149.8, 158.4, 193.2.

MS: m/z ($M+23$) 391.0

HRMS: m/z calc'd for $\text{C}_{17}\text{H}_{21}\text{INaO}$ 391.0529, found 391.0535

Data for 2-cyclopentylidene-2-iodo-1-(4-methoxyphenyl)ethanone 4h



Oil

IR (neat): 1022 (m), 1158 (s), 1247 (s), 1454 (m), 1592 (s), 1650 (m), 2956 (w) cm^{-1} .

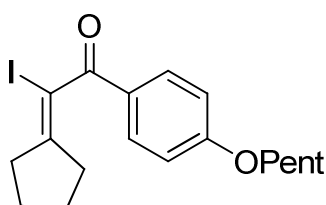
^1H NMR (400 MHz, CDCl_3): δ 1.72-1.86 (4H, m), 2.25 (2H, t, $J = 6.1$ Hz), 2.51 (2H, t, $J = 7.2$ Hz), 3.87 (3H, s), 6.94 (2H, d, $J = 8.9$ Hz), 7.91 (2H, d, $J = 9.0$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 25.8, 29.0, 34.2, 40.9, 55.9, 85.7, 114.4 (2C), 127.4, 132.8 (2C), 157.6, 164.3, 192.2.

MS: m/z ($M+23$) 365.0

HRMS: m/z calc'd for $\text{C}_{14}\text{H}_{15}\text{INaO}_2$ 365.0009, found 365.0009.

Data for 2-cyclopentylidene-2-iodo-1-(4-(pentyloxy)phenyl)ethanone 4i



Oil

IR (neat): 1158 (s), 1246 (s), 1595 (s), 1650 (m), 2954 (w) cm^{-1} .

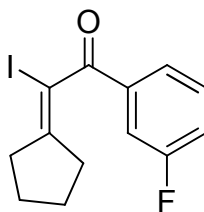
^1H NMR (400 MHz, CDCl_3): δ 0.93 (3H, t, $J = 7.0$ Hz), 1.32-1.50 (4H, m), 1.72-1.85 (6H, m), 2.25 (2H, t, $J = 7.0$ Hz), 2.51 (2H, t, $J = 7.2$ Hz), 4.02 (2H, t, $J = 6.5$ Hz), 6.92 (2H, d, $J = 8.9$ Hz), 7.89 (2H, d, $J = 8.9$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 14.4, 22.8, 25.9, 28.5, 29.0, 29.1, 34.2, 40.9, 68.7, 85.7, 114.8 (2C), 127.1, 132.8 (2C), 157.4, 164.0, 192.2.

MS: m/z ($M+23$) 421.1

HRMS: m/z calc'd for $\text{C}_{18}\text{H}_{23}\text{INaO}_2$ 421.0635, found 421.0650.

Data for 2-cyclopentylidene-1-(3-fluorophenyl)-2-iodoethanone **4j**



Oil

IR (neat): 1257 (s), 1440 (m), 1587 (m), 1663 (m), 2959 (w) cm^{-1} .

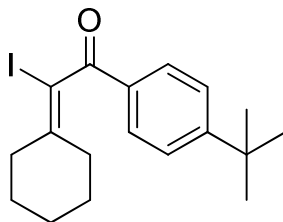
^1H NMR (400 MHz, CDCl_3): δ 1.73-1.88 (4H, m), 2.26 (2H, t, $J = 6.0$ Hz), 2.53 (2H, t, $J = 7.4$ Hz), 7.23-7.29 (1H, m), 7.40-7.47 (1H, m), 7.55-7.60 (1H, m), 7.66-7.70 (1H, m).

^{13}C NMR (100 MHz, CDCl_3): δ 25.7, 29.0, 34.6, 41.4, 85.2, 116.7 (d, $J = 22$ Hz), 120.8 (d, $J = 21$ Hz), 125.9 (d, $J = 3.0$ Hz), 130.7 (d, $J = 7.8$ Hz), 137.3 (d, $J = 6.2$ Hz), 160.4, 163.1 (d, $J = 248$ Hz), 192.1.

MS: m/z ($M+23$) 353.0

HRMS: m/z calc'd for $\text{C}_{13}\text{H}_{12}\text{FINaO}$ 352.9809, found 352.9812.

Data for 1-(4-*tert*-butylphenyl)-2-cyclohexylidene-2-iodoethanone 4k



Oil

IR (neat): 1184 (s), 1245 (s), 1264 (s), 1602 (s), 1659 (s), 2929 (w) cm^{-1} .

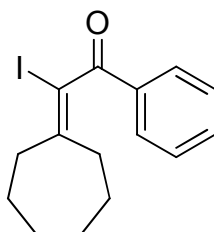
^1H NMR (400 MHz, CDCl_3): δ 1.34 (9H, s), 1.40-1.48 (2H, m), 1.52-1.60 (2H, m), 1.66-1.75 (2H, m), 2.18-2.25 (2H, m), 2.49-2.58 (2H, m), 7.48 (2H, d, $J = 8.6$ Hz), 7.91 (2H, d, $J = 8.6$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 26.2, 27.8, 28.0, 31.4 (3C), 33.6, 35.6, 39.2, 88.3, 126.2 (2C), 130.4 (2C), 131.6, 149.4, 158.0, 193.3.

MS: m/z ($M+23$) 405.1

HRMS: m/z calc'd for $\text{C}_{18}\text{H}_{23}\text{INaO}$ 405.0686, found 405.0689.

Data for 2-cycloheptylidene-2-iodo-1-phenylethanone 4l



Oil

IR (neat): 1172 (m), 1235 (s), 1447 (m), 1659 (s), 2921 (w) cm^{-1} .

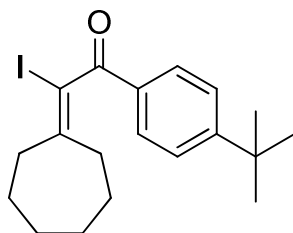
^1H NMR (400 MHz, CDCl_3): δ 1.46-1.56 (4H, m), 1.57-1.65 (2H, m), 1.70-1.79 (2H, m), 2.31-2.39 (2H, m), 2.56-2.64 (2H, m), 7.47 (2H, t, $J = 7.7$ Hz), 7.55-7.61 (1H, m), 7.95-7.99 (2H, m).

^{13}C NMR (100 MHz, CDCl_3): δ 26.8, 27.9, 29.0, 29.9, 33.9, 40.8, 91.5, 129.1 (2C), 130.4 (2C), 134.0, 134.3, 151.6, 193.5.

MS: m/z ($M+23$) 363.0

HRMS: m/z calc'd for $\text{C}_{15}\text{H}_{17}\text{INaO}$ 363.0216, found 363.0217

Data for 1-(4-*tert*-butylphenyl)-2-cycloheptylidene-2-iodoethanone 4m



Oil

IR (neat): 1108 (m), 1183 (s), 1252 (s), 1602 (s), 1658 (s), 2924 (w) cm^{-1} .

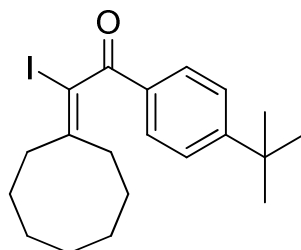
^1H NMR (400 MHz, CDCl_3): δ 1.35 (9H, s), 1.48-1.55 (4H, m), 1.58-1.65 (2H, m), 1.70-1.77 (2H, m), 2.33-2.38 (2H, m), 2.57-2.62 (2H, m), 7.48 (2H, d, $J = 8.6$ Hz), 7.91 (2H, d, $J = 8.6$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 26.8, 28.0, 29.0, 30.0, 31.4 (3C), 33.8, 35.6, 40.8, 91.7, 126.1 (2C), 130.4 (2C), 131.5, 151.0, 157.9, 193.2.

MS: m/z ($M+23$) 419.1

HRMS: m/z calc'd for $\text{C}_{19}\text{H}_{25}\text{INaO}$ 419.0842, found 419.0851.

Data for 1-(4-*tert*-butylphenyl)-2-cyclooctylidene-2-iodoethanone 4n



Oil

IR (neat): 1105 (m), 1183 (m), 1253 (s), 1602 (s), 1658 (s), 2923 (w) cm^{-1} .

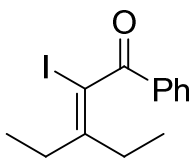
^1H NMR (400 MHz, CDCl_3): δ 1.35 (9H, s), 1.41-1.50 (2H, m), 1.52-1.68 (6H, m), 1.81-1.88 (2H, m), 2.25-2.31 (2H, m), 2.53-2.58 (2H, m), 7.48 (2H, d, $J = 8.6$ Hz), 7.90 (2H, d, $J = 8.5$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 25.6, 26.1, 26.5, 26.8, 27.6, 31.5 (3C), 34.1, 35.6, 38.6, 91.1, 126.1 (2C), 130.4 (2C), 131.6, 151.6, 157.9, 193.3.

MS: m/z ($M+23$) 433.1

HRMS: m/z calc'd for $\text{C}_{20}\text{H}_{27}\text{INaO}$ 433.0999, found 433.1005.

Data for 3-ethyl-2-iodo-1-phenylpent-2-en-1-one 4o



Oil

IR (neat): 1233 (s), 1448 (m), 1662 (s), 2968 (w) cm^{-1} .

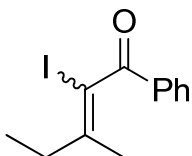
^1H NMR (400 MHz, CDCl_3): δ 0.97 (3H, t, $J = 7.7$ Hz), 1.16 (3H, t, $J = 7.5$ Hz), 2.20 (2H, q, $J = 7.5$ Hz), 2.48 (2H, q, $J = 7.5$ Hz), 7.47 (2H, t, $J = 7.7$ Hz), 7.55-7.59 (1H, m), 7.94-7.97 (2H, m).

^{13}C NMR (100 MHz, CDCl_3): δ 12.3, 13.5, 27.0, 32.6, 90.8, 129.1 (2C), 130.4 (2C), 134.1, 134.4, 153.2, 193.3.

MS: m/z ($M+23$) 337.0

HRMS: m/z calc'd for $\text{C}_{13}\text{H}_{15}\text{INaO}$ 337.0060, found 337.0060.

Data for (*E*)- and (*Z*)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



Formed as a 1:1 mixture of alkene geometrical isomers which can be partially separated.

Oil

IR (neat): 1238 (s), 1448 (m), 1661 (s), 2970 (w) cm^{-1} .

NMR data for (*E*)-isomer (determined by NOESY experiments):

^1H NMR (400 MHz, CDCl_3): δ 0.98 (3H, t, $J = 7.5$ Hz), 2.12 (3H, s), 2.19 (2H, q, $J = 7.5$ Hz), 7.47 (2H, t, $J = 7.7$ Hz), 7.58 (1H, t, $J = 7.7$ Hz), 7.97 (2H, d, $J = 7.7$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 13.1, 26.4, 29.5, 91.3, 129.1 (2C), 130.4 (2C), 134.1, 134.2, 148.6, 193.5.

NMR data for (*Z*)-isomer:

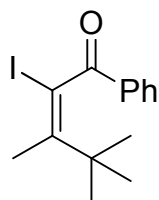
^1H NMR (400 MHz, CDCl_3): δ 1.14 (3H, t, $J = 7.6$ Hz), 1.81 (3H, s), 2.47 (2H, q, $J = 7.6$ Hz), 7.47 (2H, t, $J = 7.6$ Hz), 7.58 (1H, t, $J = 7.4$ Hz), 7.94 (2H, d, $J = 7.4$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 11.8, 19.6, 35.8, 89.8, 129.1 (2C), 130.3 (2C), 134.1 (2C), 147.9, 193.6.

MS: m/z ($M+23$) 323.0

HRMS: m/z calc'd for $\text{C}_{12}\text{H}_{13}\text{INaO}$ 322.9904, found 322.9903.

Data for (*E*)-2-iodo-3,4,4-trimethyl-1-phenylpent-2-en-1-one 4q



Alkene geometry determined by NOESY experiments.

Oil

IR (neat): 1231 (s), 1451 (m), 1661 (s), 2969 (w) cm^{-1} .

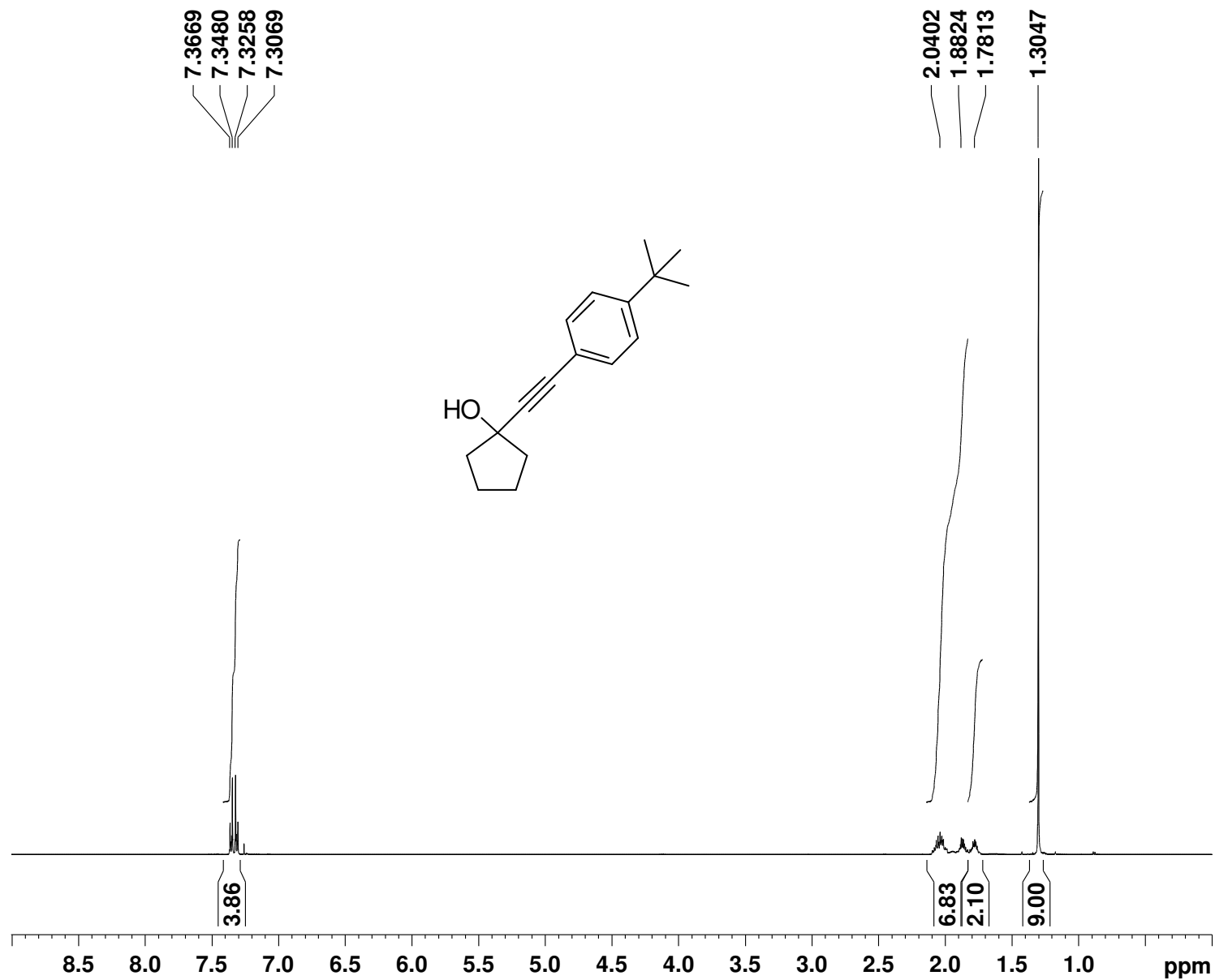
^1H NMR (400 MHz, CDCl_3): δ 1.10 (9H, s), 2.25 (3H, s), 7.47 (2H, t, $J = 7.4$ Hz), 7.55 (1H, t, $J = 7.2$ Hz), 7.99 (2H, d, $J = 7.5$ Hz).

^{13}C NMR (100 MHz, CDCl_3): δ 28.8, 30.5 (3C), 40.6, 96.0, 129.0 (2C), 130.4 (2C), 133.7, 134.2, 151.2, 193.2.

MS: m/z ($M+23$) 351.0

HRMS: m/z calc'd for $\text{C}_{14}\text{H}_{17}\text{INaO}$ 351.0216, found 351.0219.

¹H NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclopentanol, 2d



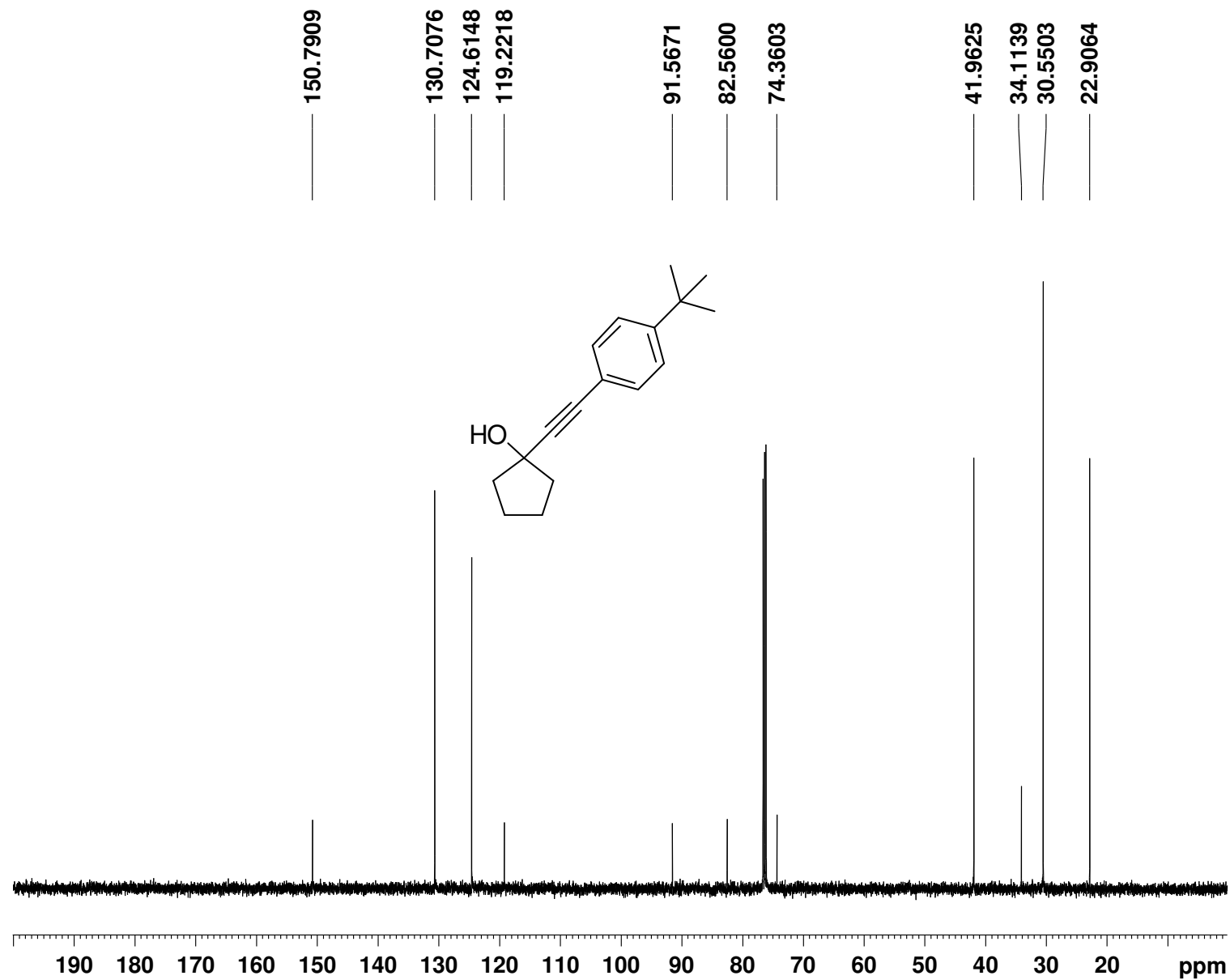
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EXPNO 10
PROCNO 1

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PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 144
DW 48.400 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TDO 1

==== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclopentanol, 2d



Current Data Parameters
NAME ARM-1162
EXPNO 11
PROCNO 1

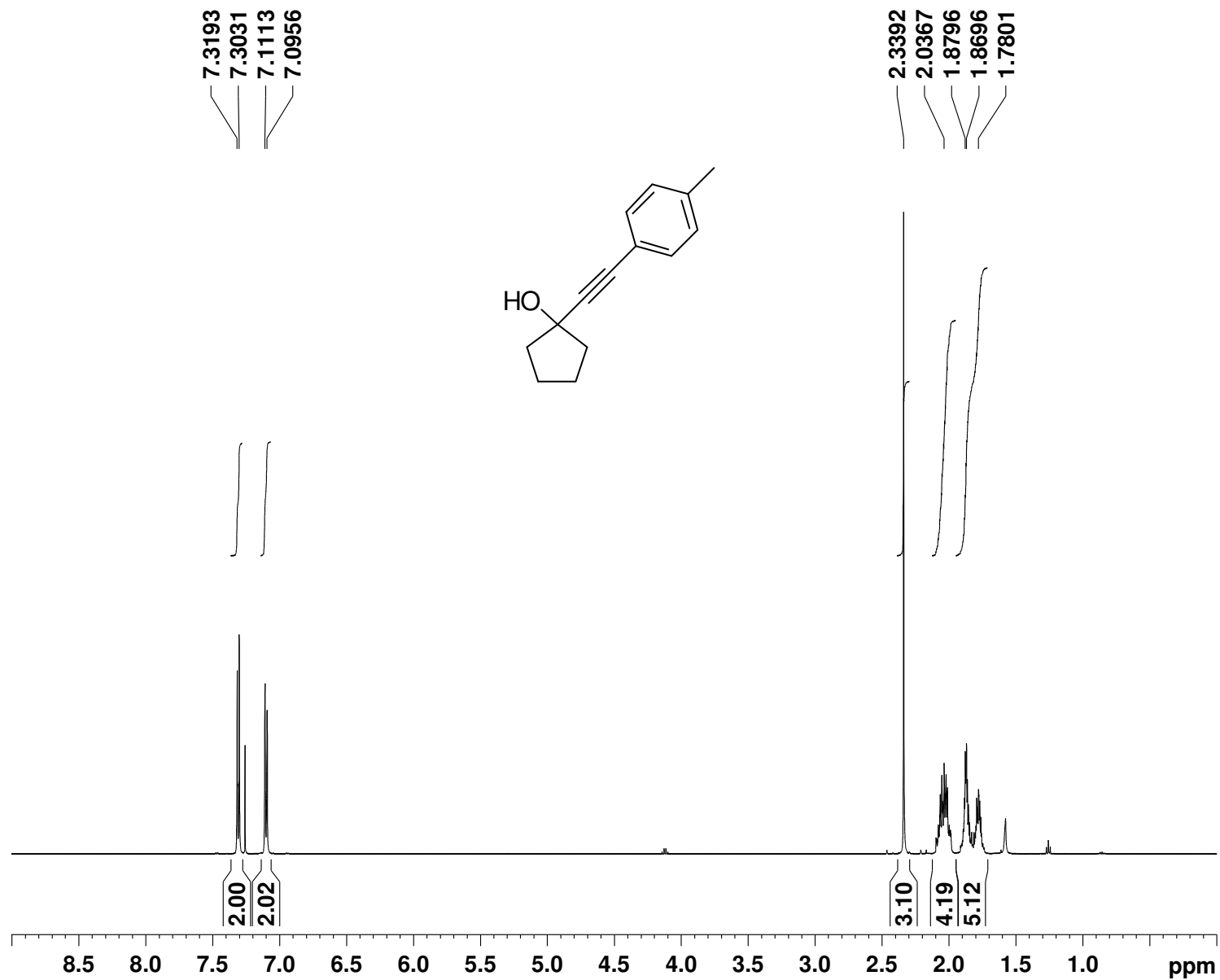
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Time 13.24
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PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 81
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 13000
DW 16.650 usec
DE 6.00 usec
TE 297.0 K
D1 2.0000000 sec
D11 0.03000000 sec
TDO 8

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7578687 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-(*p*-tolylethynyl)cyclopentanol 2e



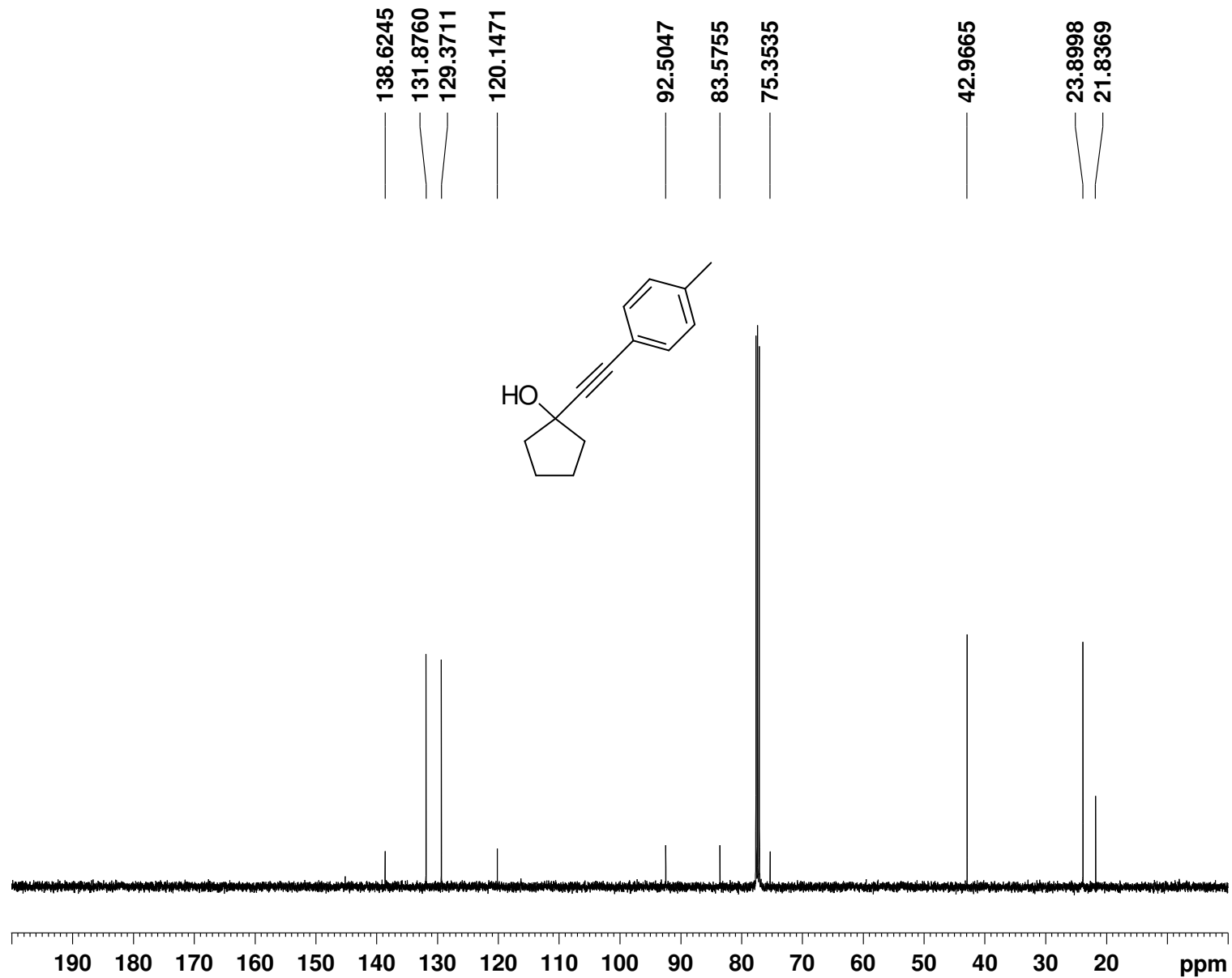
Current Data Parameters
NAME ARM-1192
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 13.42
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PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 322
DW 48.400 usec
DE 6.00 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-(*p*-tolylethynyl)cyclopentanol 2e



```
Current Data Parameters
NAME      ARM-1192
EXPNO    11
PROCNO   1

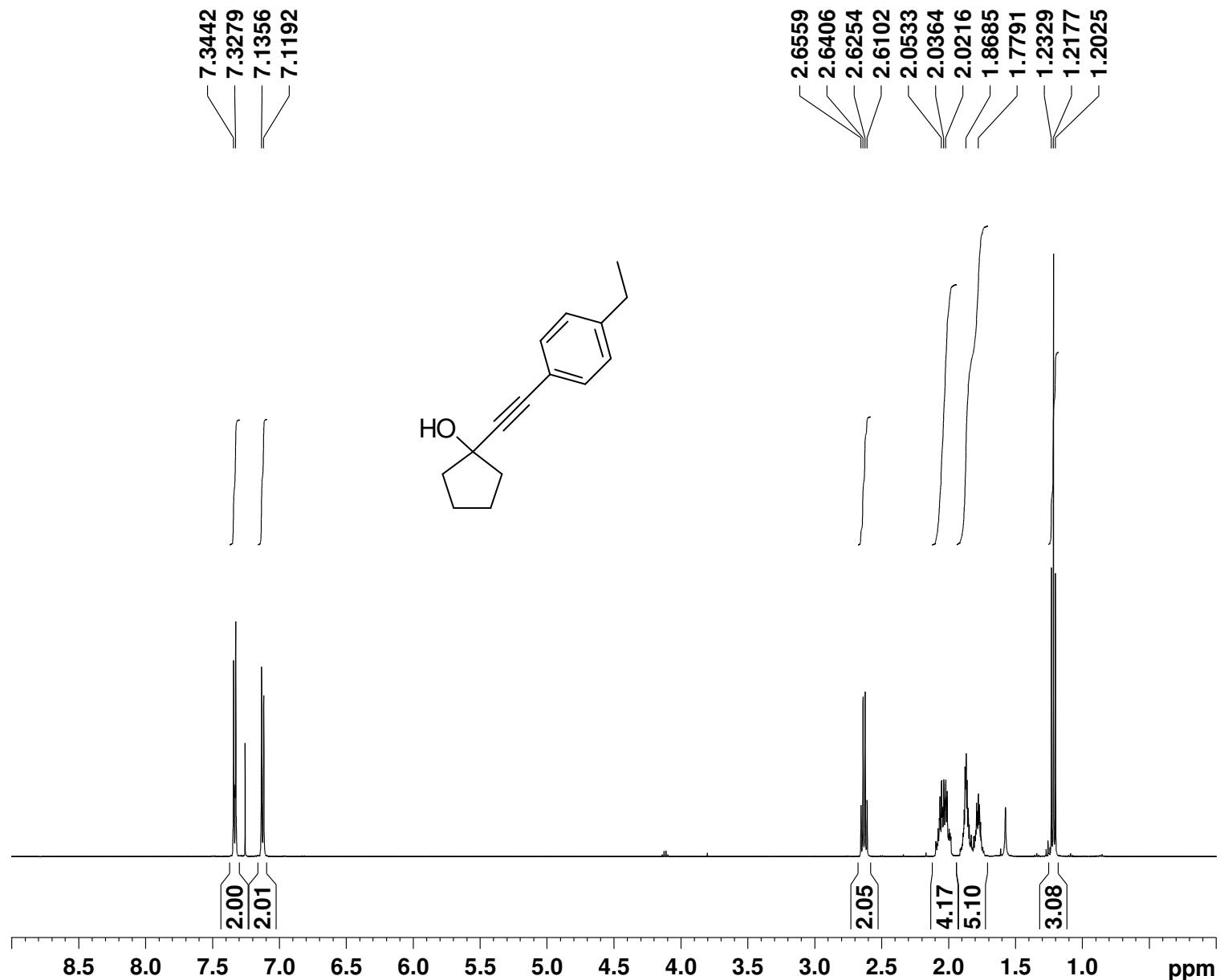
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PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       4
SWH      30030.029 Hz
FIDRES   0.458222 Hz
AQ       1.0912244 sec
RG       14600
DW       16.650 usec
DE       6.00 usec
TE       297.1 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      8

===== CHANNEL f1 =====
NUC1     13C
P1       9.00 usec
PL1      0.00 dB
SFO1     125.7703637 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     18.00 dB
PL13     18.00 dB
PL2W     23.36054420 W
PL12W    0.37023968 W
PL13W    0.37023968 W
SFO2     500.1320005 MHz

F2 - Processing parameters
SI       32768
SF       125.7577435 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
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¹H NMR spectrum for 1-((4-ethylphenyl)ethynyl)cyclopentanol 2f



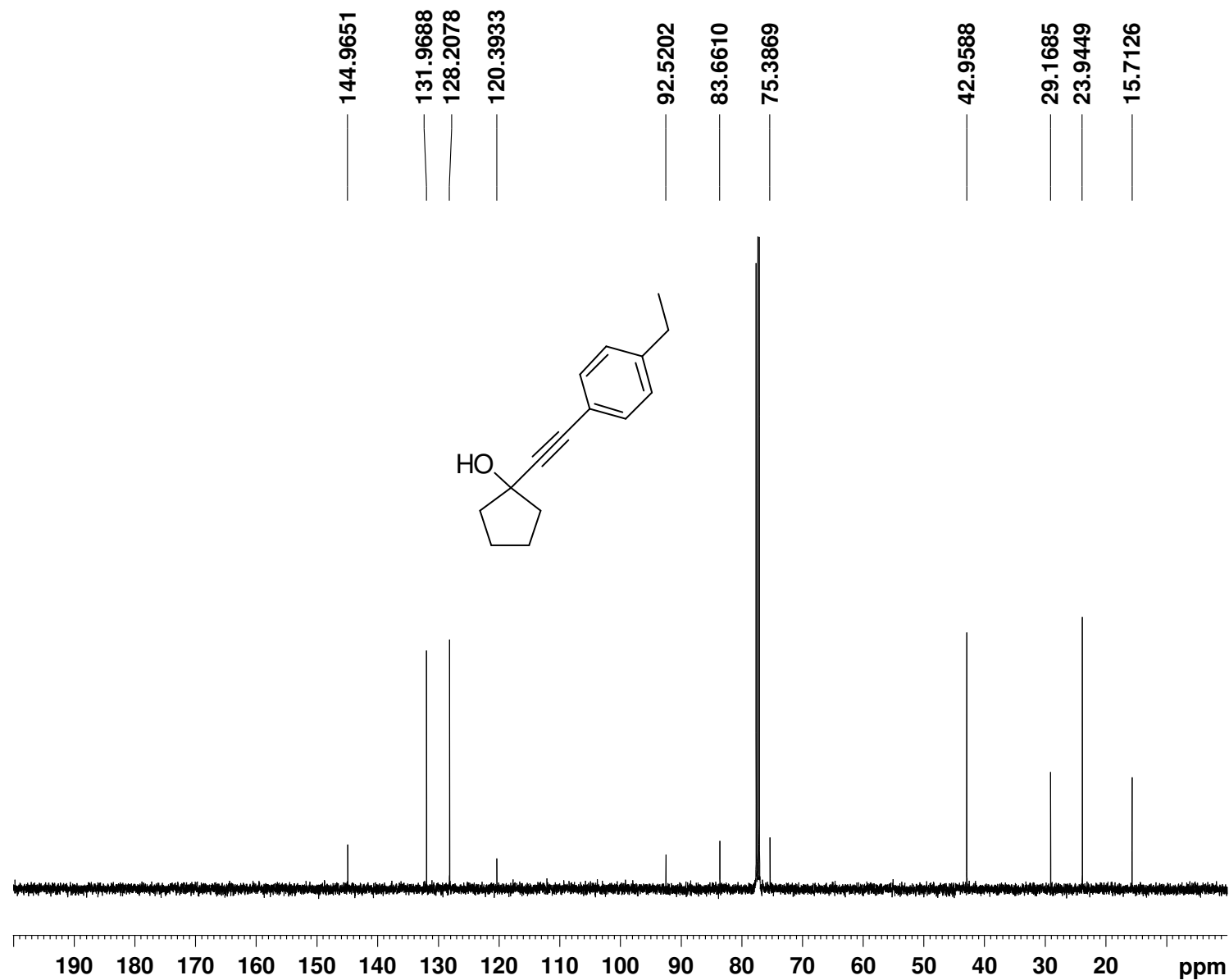
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EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
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INSTRUM   av500sgu
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       10330.578 Hz
FIDRES    0.157632 Hz
AQ        3.1719923 sec
RG        322
DW        48.400 usec
DE        6.00 usec
TE        296.2 K
D1        1.00000000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      1H
P1        9.70 usec
PL1       0.10 dB
PL1W     22.82879257 W
SFO1     500.1330885 MHz

F2 - Processing parameters
SI        32768
SF        500.1300144 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
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¹³C NMR spectrum for 1-((4-ethylphenyl)ethynyl)cyclopentanol 2f



```
Current Data Parameters
NAME      ARM-1193
EXPNO     11
PROCNO    1

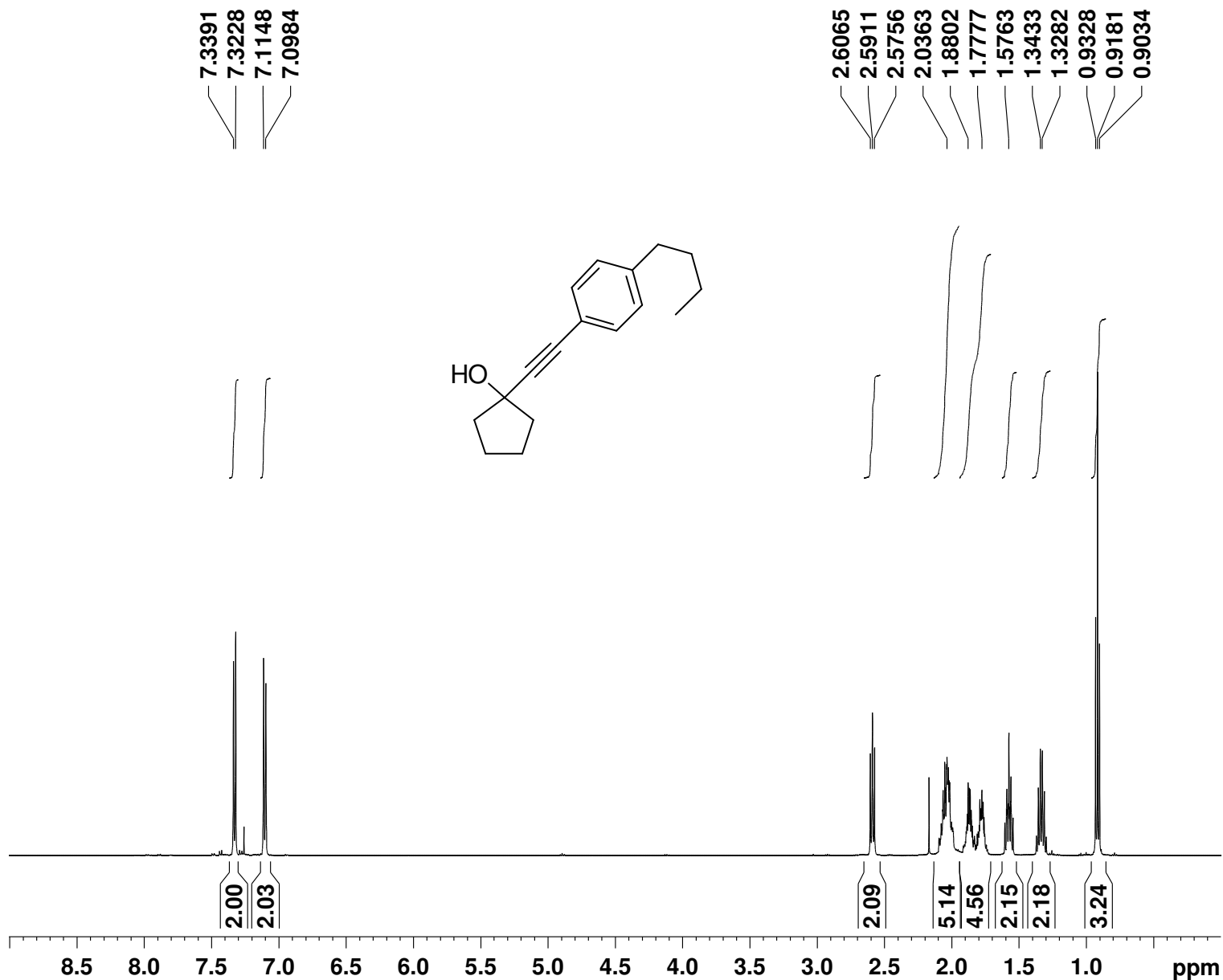
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Time      14.17
INSTRUM   av500sgu
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        256
DS        4
SWH       30030.029 Hz
FIDRES    0.458222 Hz
AQ        1.0912244 sec
RG        14600
DW        16.650 usec
DE        6.00 usec
TE        297.3 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       8

===== CHANNEL f1 =====
NUC1      13C
P1        9.00 usec
PL1       0.00 dB
SFO1     125.7703637 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12     18.00 dB
PL13     18.00 dB
PL2W     23.36054420 W
PL12W    0.37023968 W
PL13W    0.37023968 W
SFO2     500.1320005 MHz

F2 - Processing parameters
SI        32768
SF        125.7577420 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
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¹H NMR spectrum for 1-((4-butylphenyl)ethynyl)cyclopentanol 2g



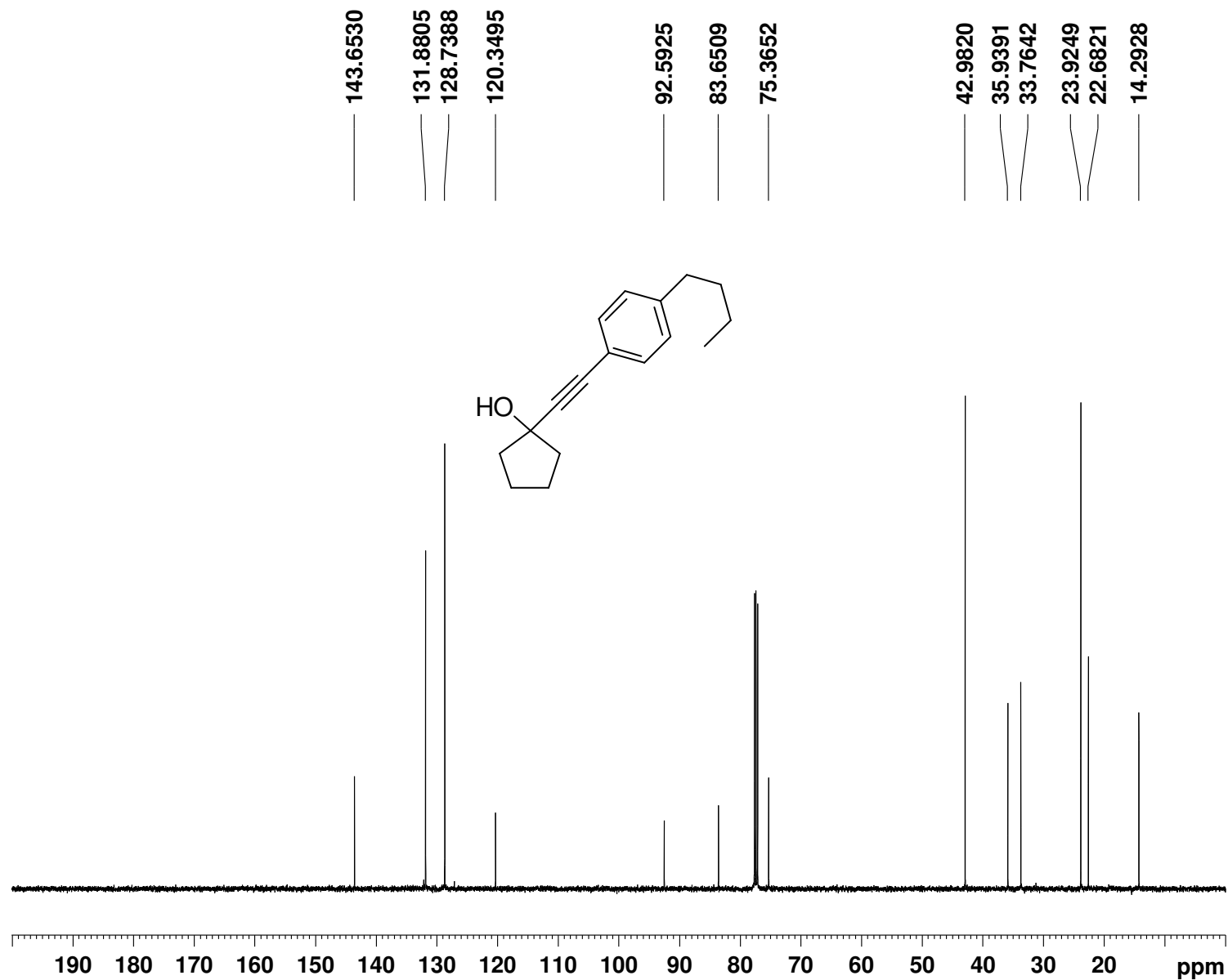
Current Data Parameters
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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-butylphenyl)ethynyl)cyclopentanol 2g



Current Data Parameters
NAME ARM-1179
EXPNO 11
PROCNO 1

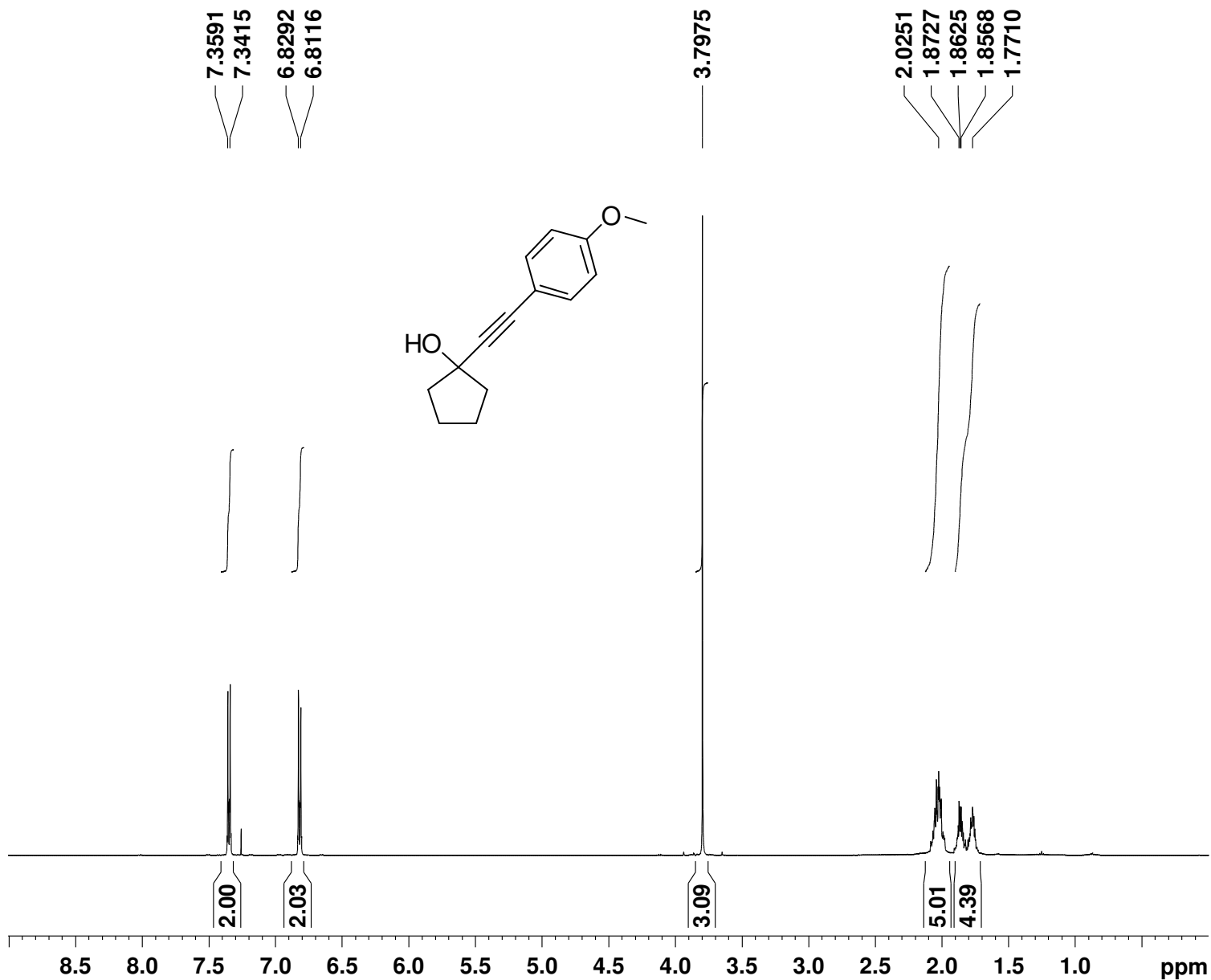
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PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 14600
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577447 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-((4-methoxyphenyl)ethynyl)cyclopentanol 2h



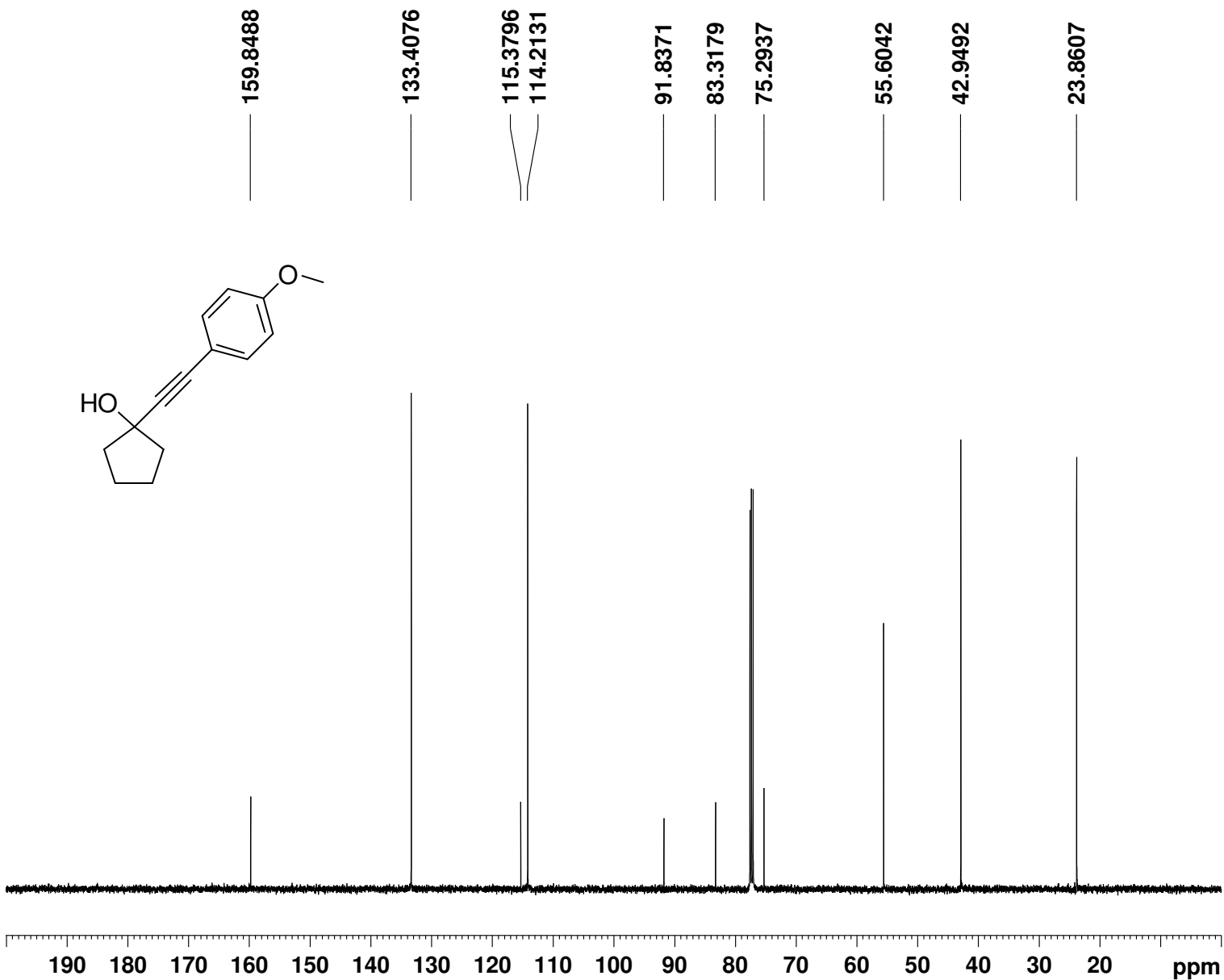
Current Data Parameters
NAME ARM-1188
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120119
Time 12.58
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 144
DW 48.400 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-methoxyphenyl)ethynyl)cyclopentanol 2h



Current Data Parameters
NAME ARM-1188
EXPNO 11
PROCNO 1

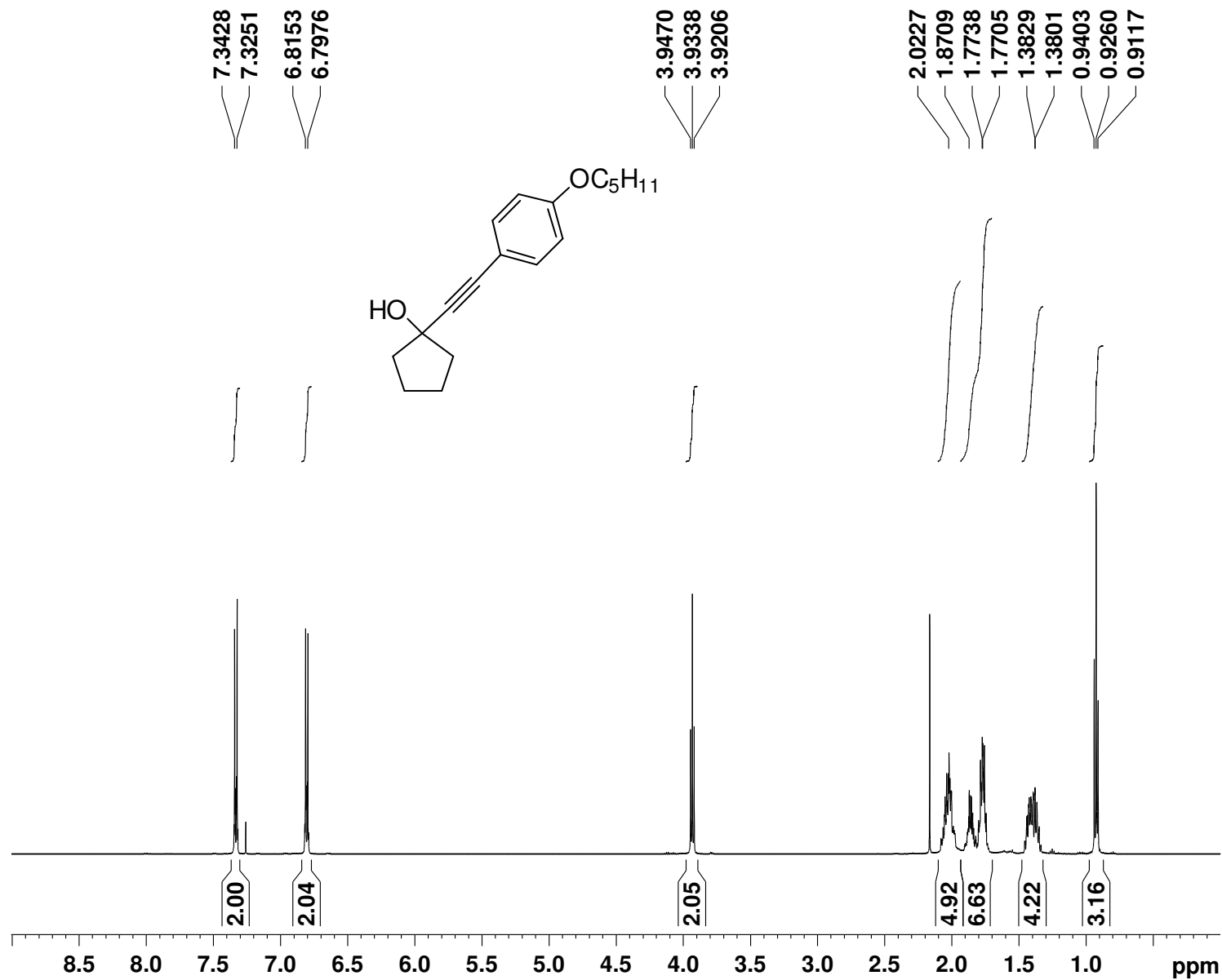
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Time 13.00
INSTRUM av500sq
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 14600
DW 16.650 usec
DE 6.00 usec
TE 297.5 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577482 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-((4-(pentyloxy)phenyl)ethynyl)cyclopentanol 2i



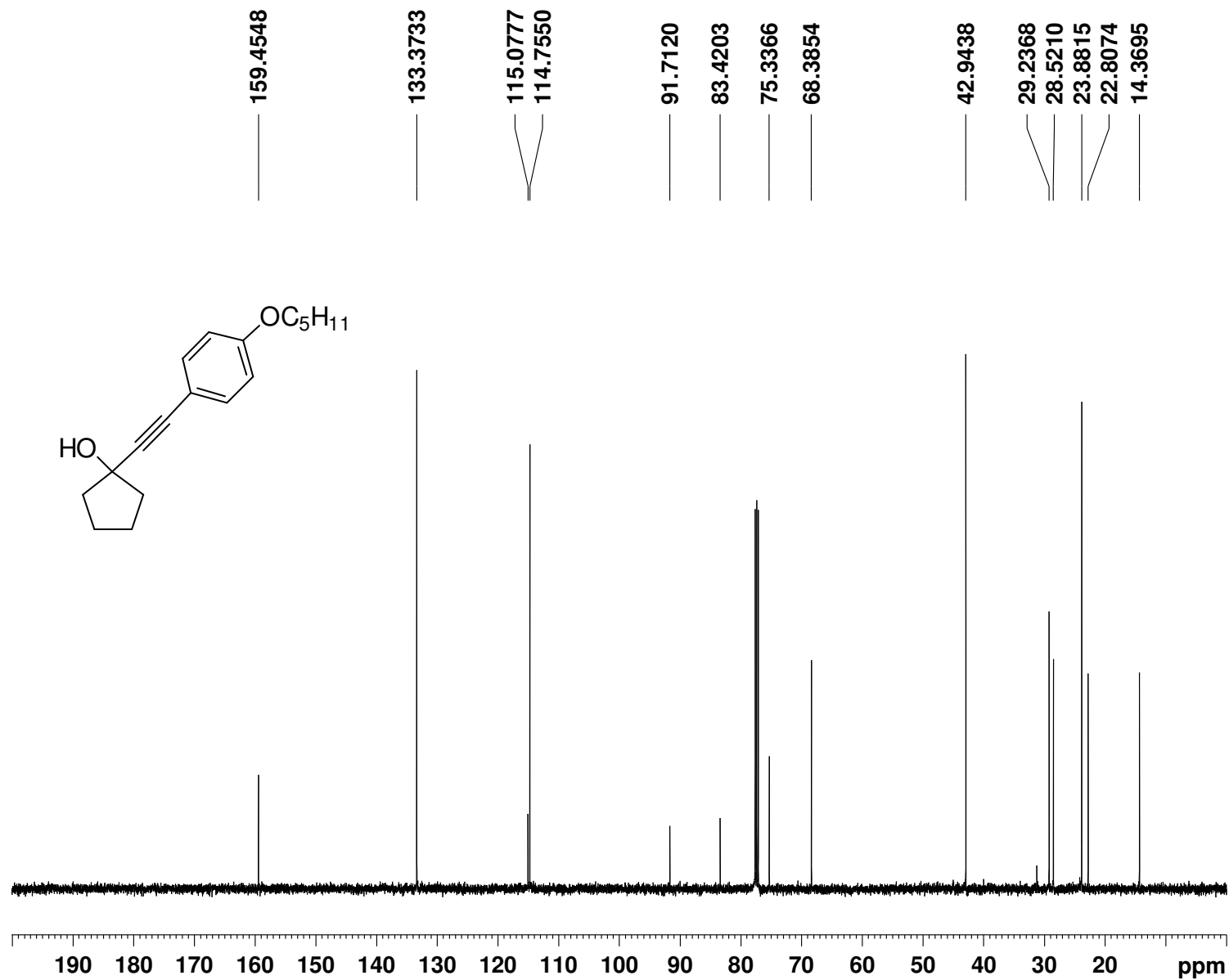
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EXPNO 10
PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 114
DW 48.400 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300133 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-(pentyloxy)phenyl)ethynyl)cyclopentanol 2i



Current Data Parameters
NAME ARM-1189
EXPNO 11
PROCNO 1

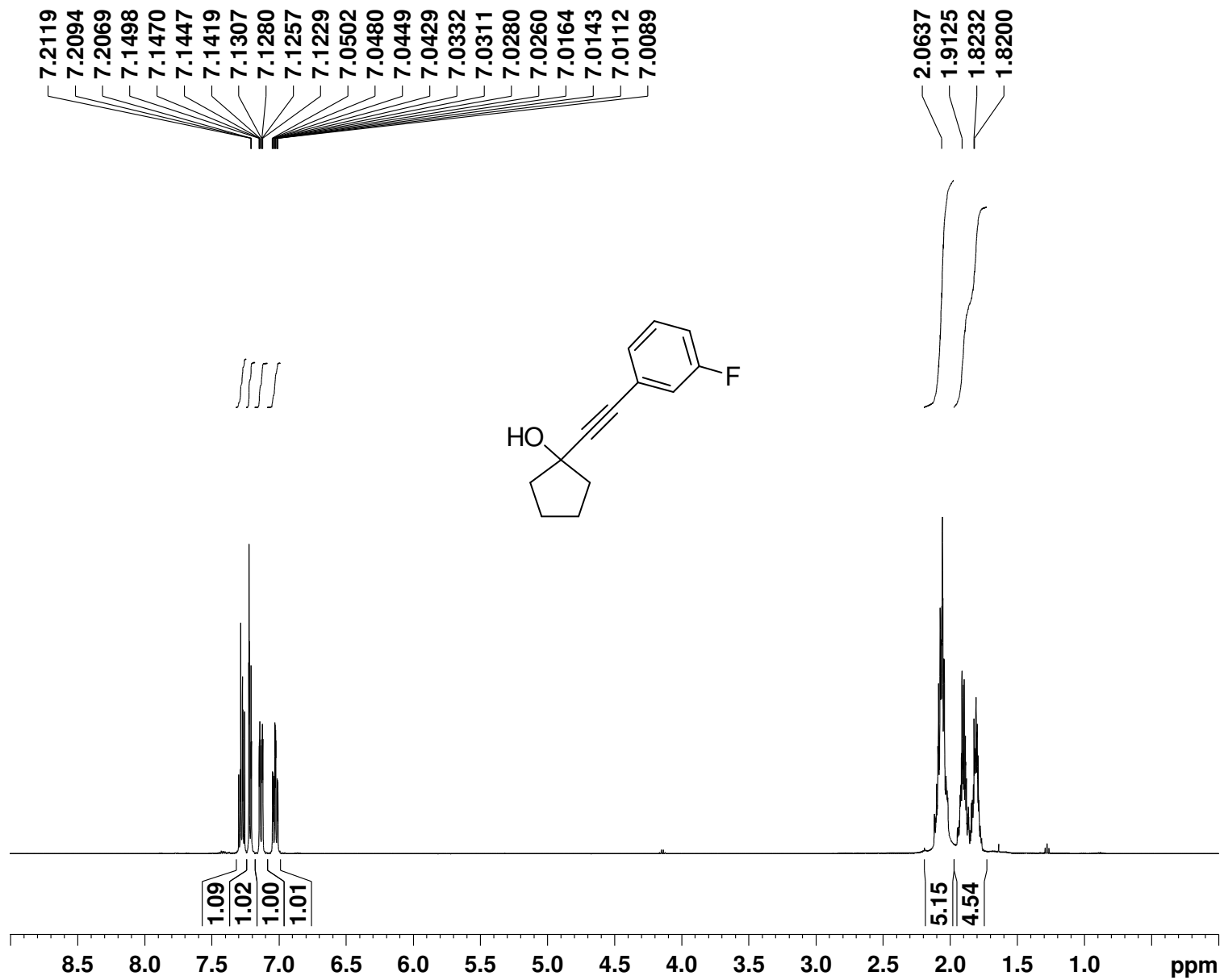
F2 - Acquisition Parameters
Date_ 20120119
Time 13.43
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 14600
DW 16.650 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577453 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-((3-fluorophenyl)ethynyl)cyclopentanol 2j



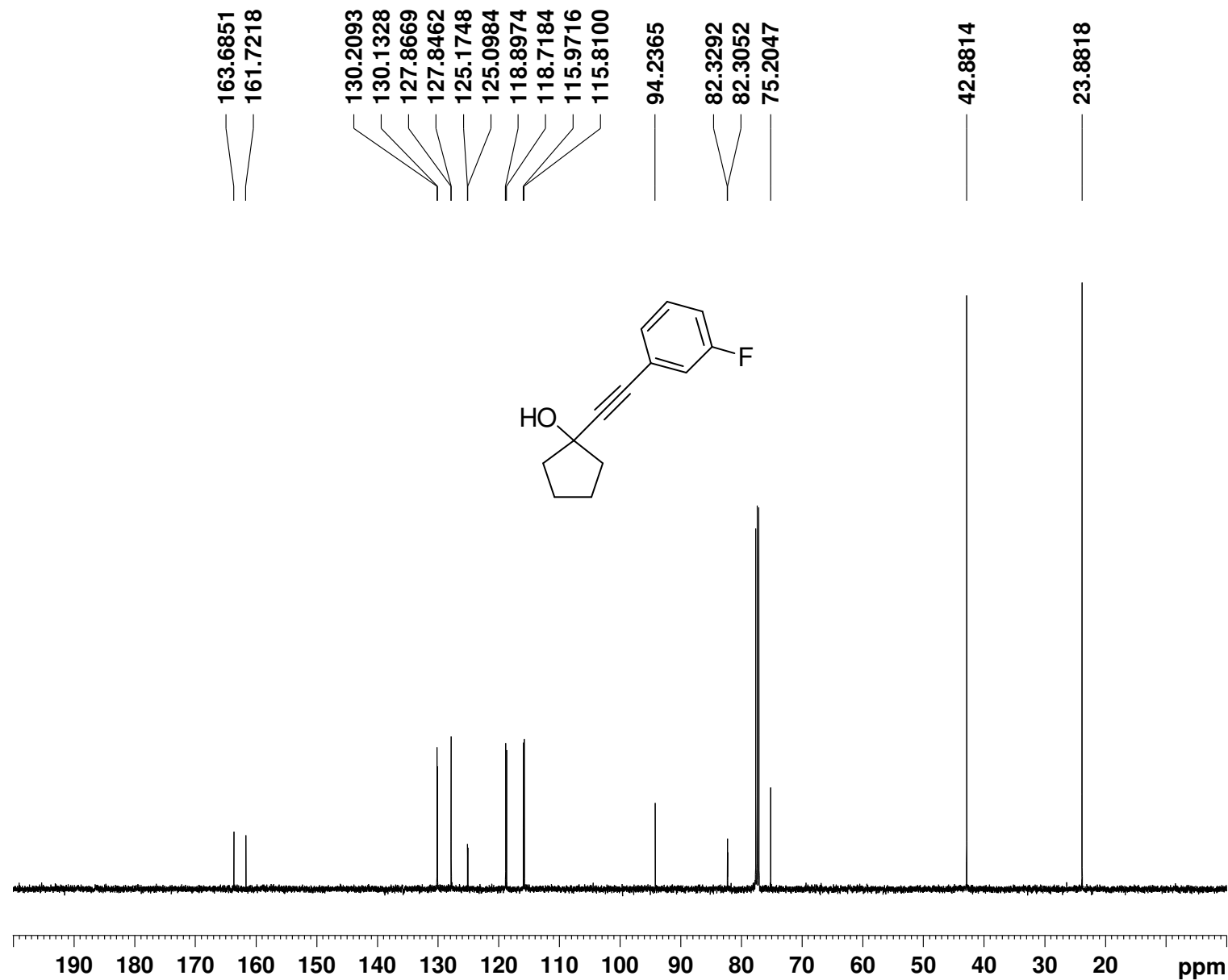
Current Data Parameters
NAME ARM-1204
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120119
Time 9.22
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 144
DW 48.400 usec
DE 6.00 usec
TE 296.7 K
D1 1.0000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((3-fluorophenyl)ethynyl)cyclopentanol 2j



Current Data Parameters
NAME ARM-1204
EXPNO 11
PROCNO 1

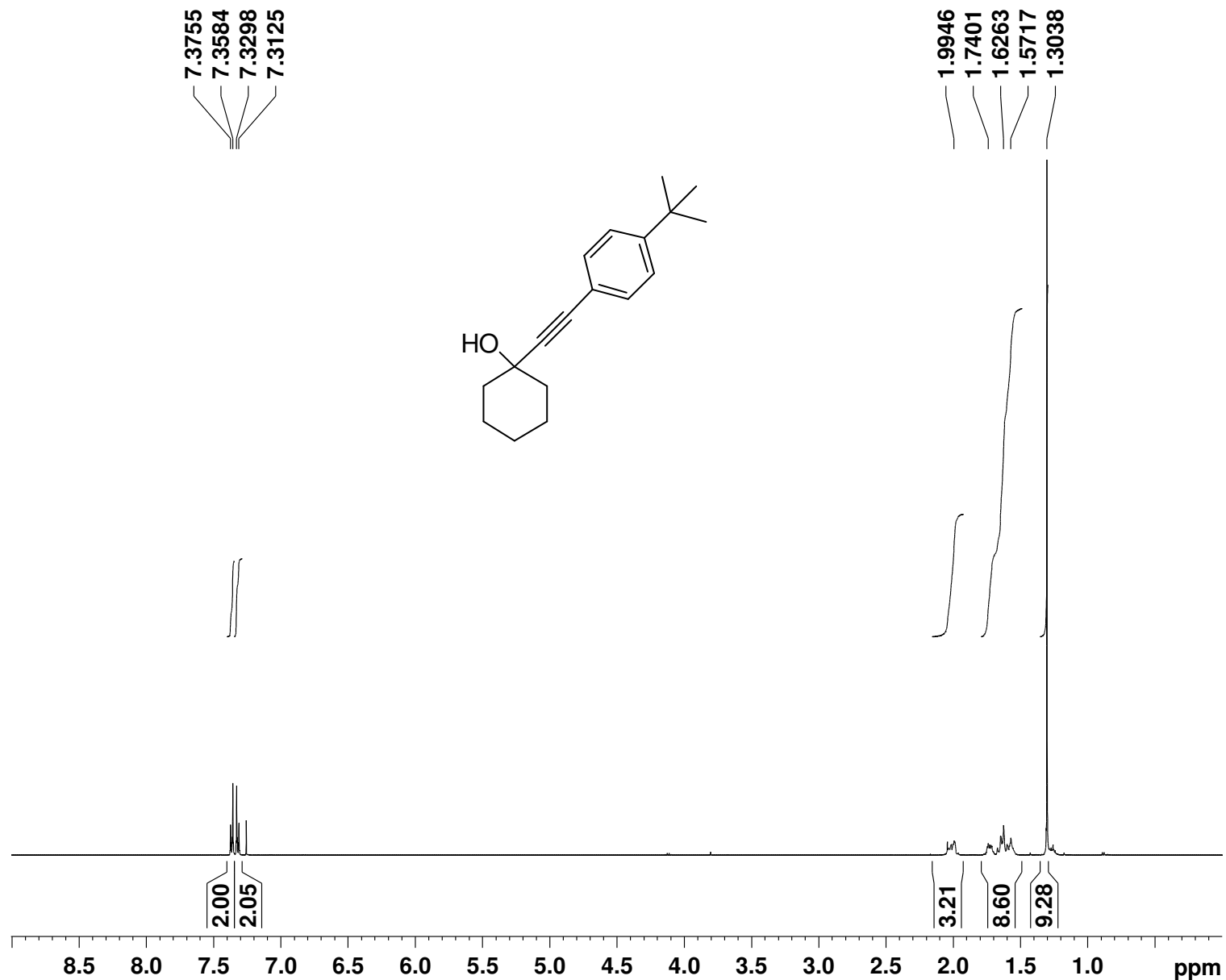
F2 - Acquisition Parameters
Date_ 20120119
Time 9.24
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 14600
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SF01 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SF02 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577434 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclohexanol 2k



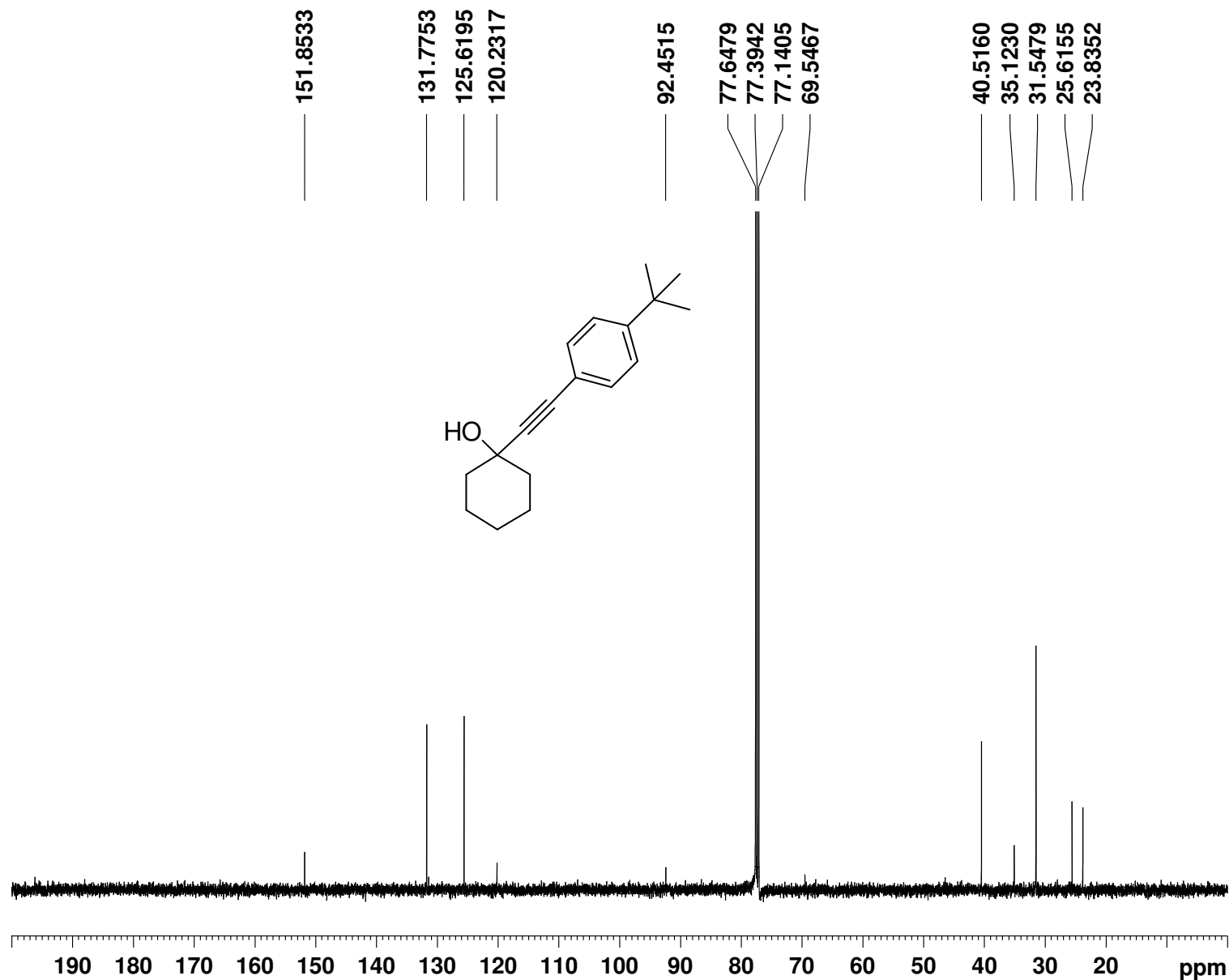
Current Data Parameters
NAME ARM-1178
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120119
Time 14.09
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 406
DW 48.400 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclohexanol 2k



```
Current Data Parameters
NAME      ARM-1178
EXPNO    11
PROCNO   1

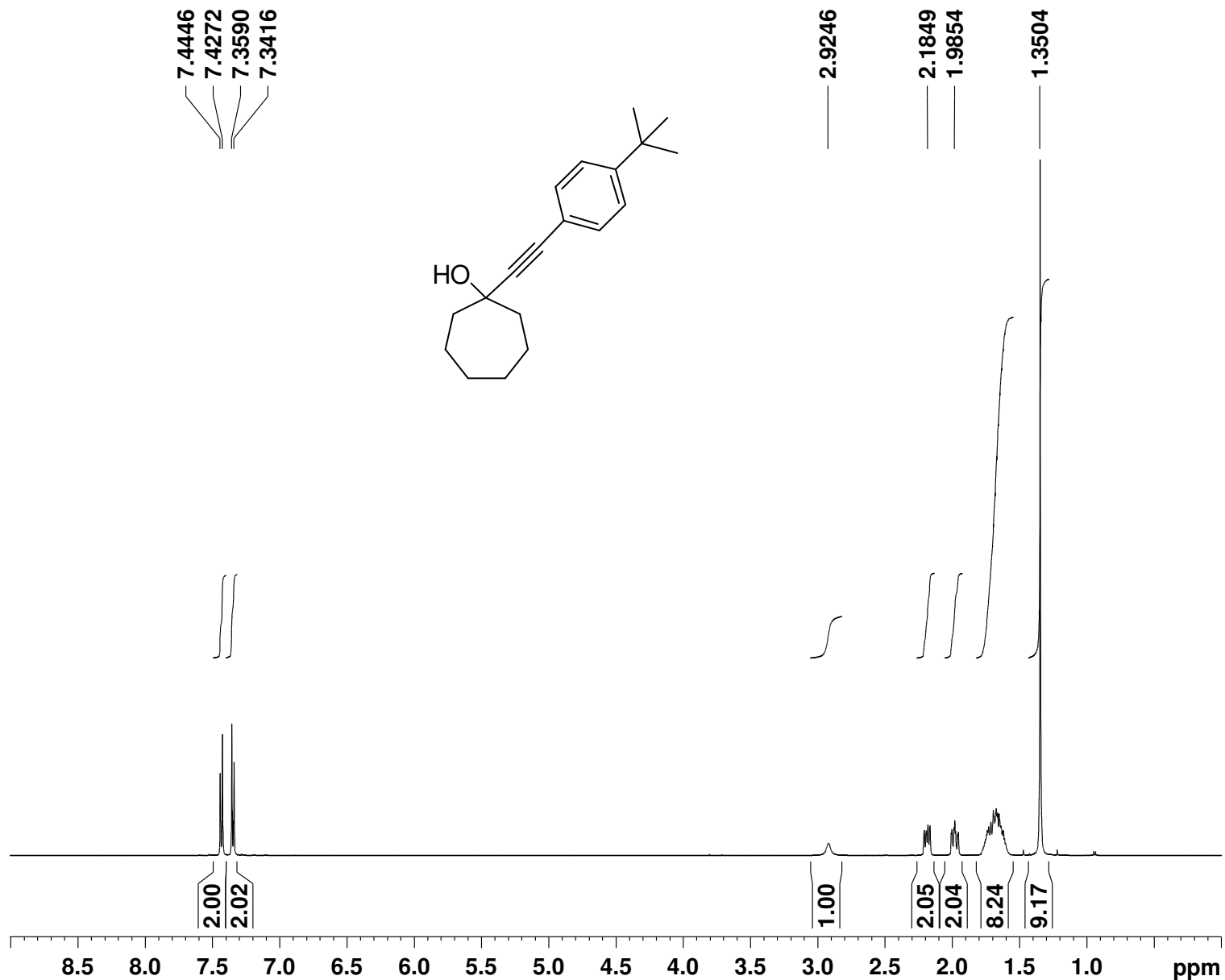
F2 - Acquisition Parameters
Date_    20120119
Time     14.11
INSTRUM  av500sgu
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       4
SWH      30030.029 Hz
FIDRES   0.458222 Hz
AQ       1.0912244 sec
RG       14600
DW       16.650 usec
DE       6.00 usec
TE       297.6 K
D1       2.0000000 sec
D11      0.0300000 sec
TDO      8

===== CHANNEL f1 =====
NUC1     13C
P1       9.00 usec
PL1      0.00 dB
SFO1    125.7703637 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2      0.00 dB
PL12     18.00 dB
PL13     18.00 dB
PL2W     23.36054420 W
PL12W    0.37023968 W
PL13W    0.37023968 W
SFO2    500.1320005 MHz

F2 - Processing parameters
SI       32768
SF       125.7577426 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```


¹H NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cycloheptanol 2m



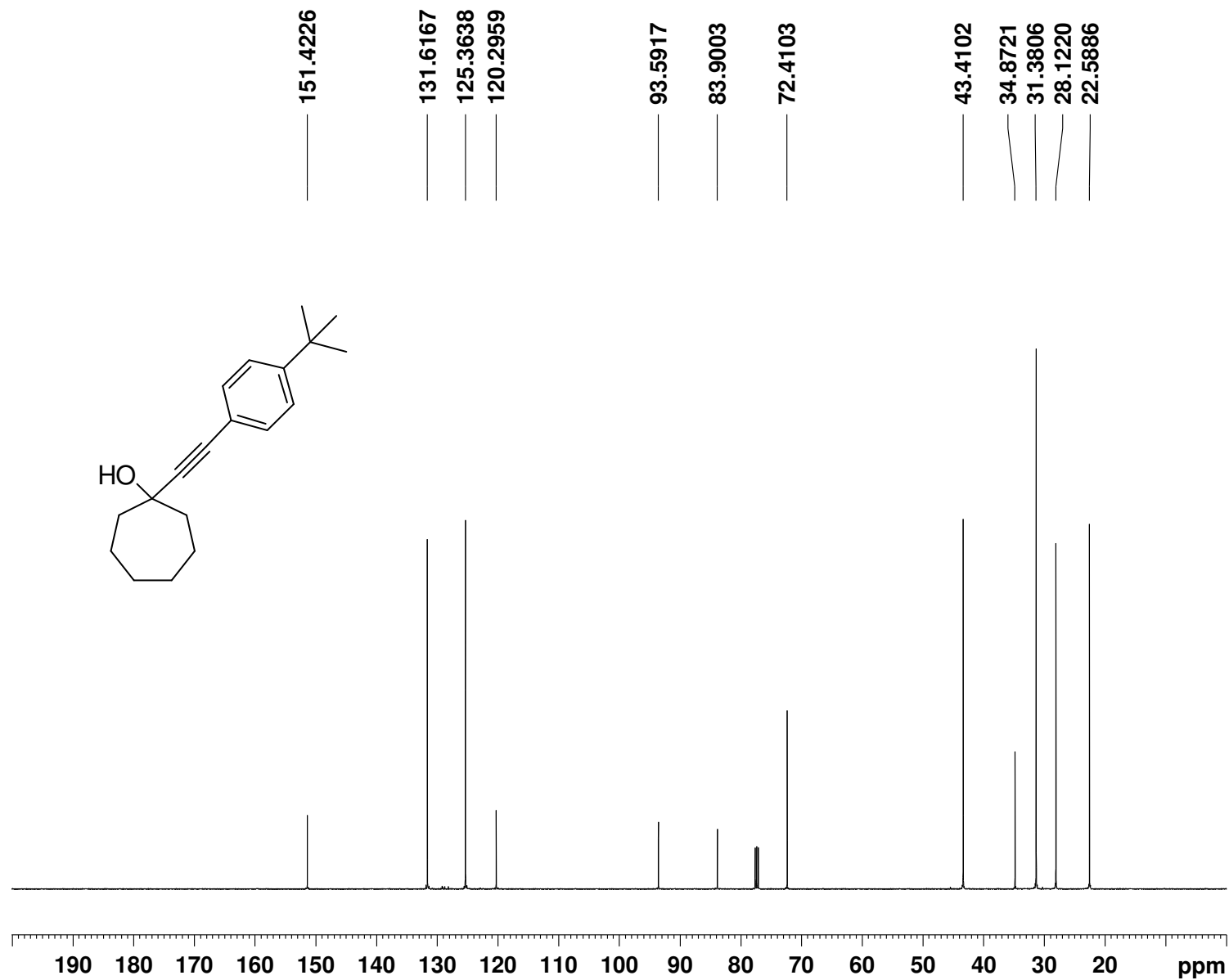
Current Data Parameters
NAME ARM-1205_3
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120130
Time 11.23
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 12.7
DW 48.400 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SF01 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cycloheptanol 2m



Current Data Parameters
NAME ARM-1205_3
EXPNO 11
PROCNO 1

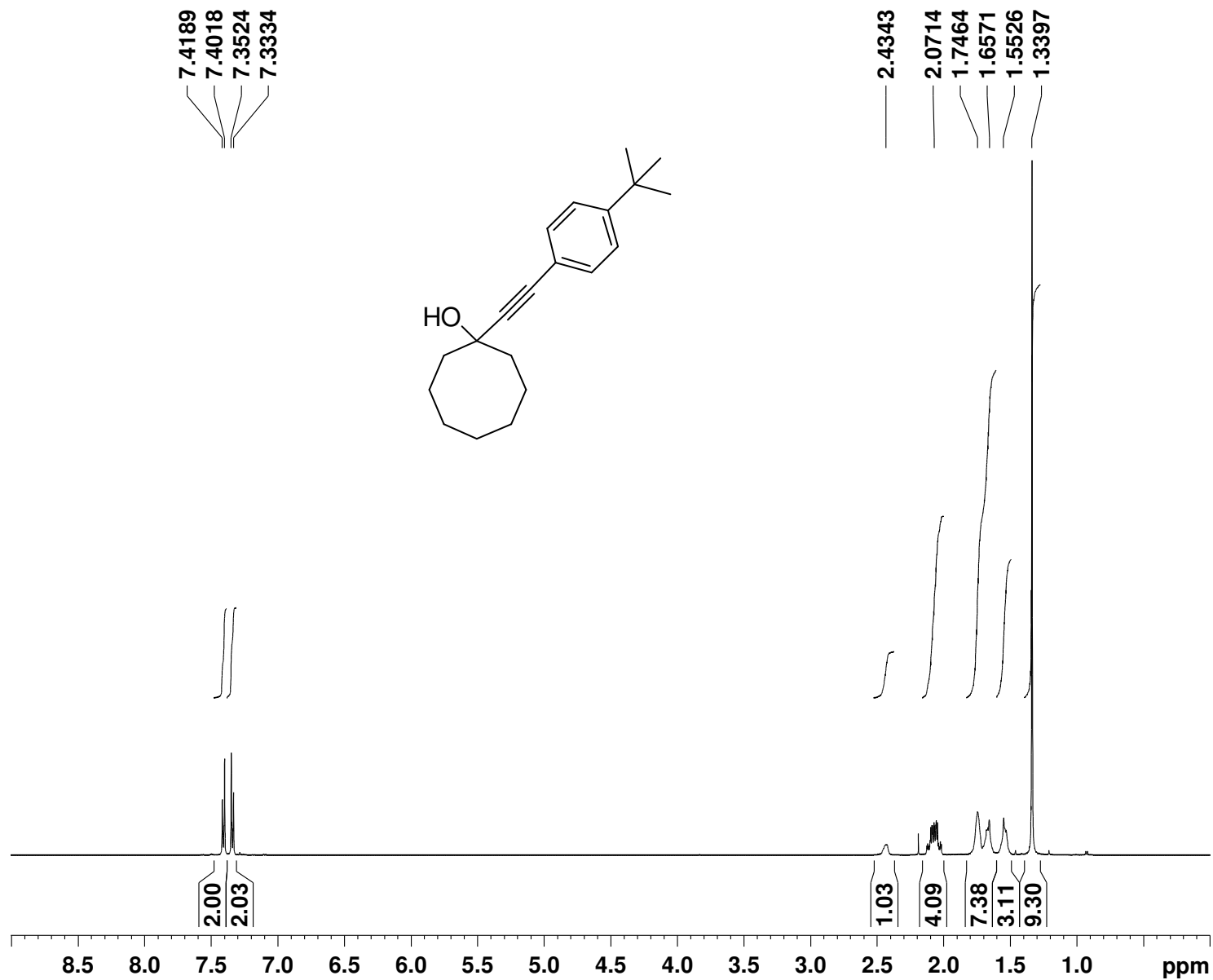
F2 - Acquisition Parameters
Date_ 20120130
Time 11.26
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 11500
DW 16.650 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclooctanol 2n



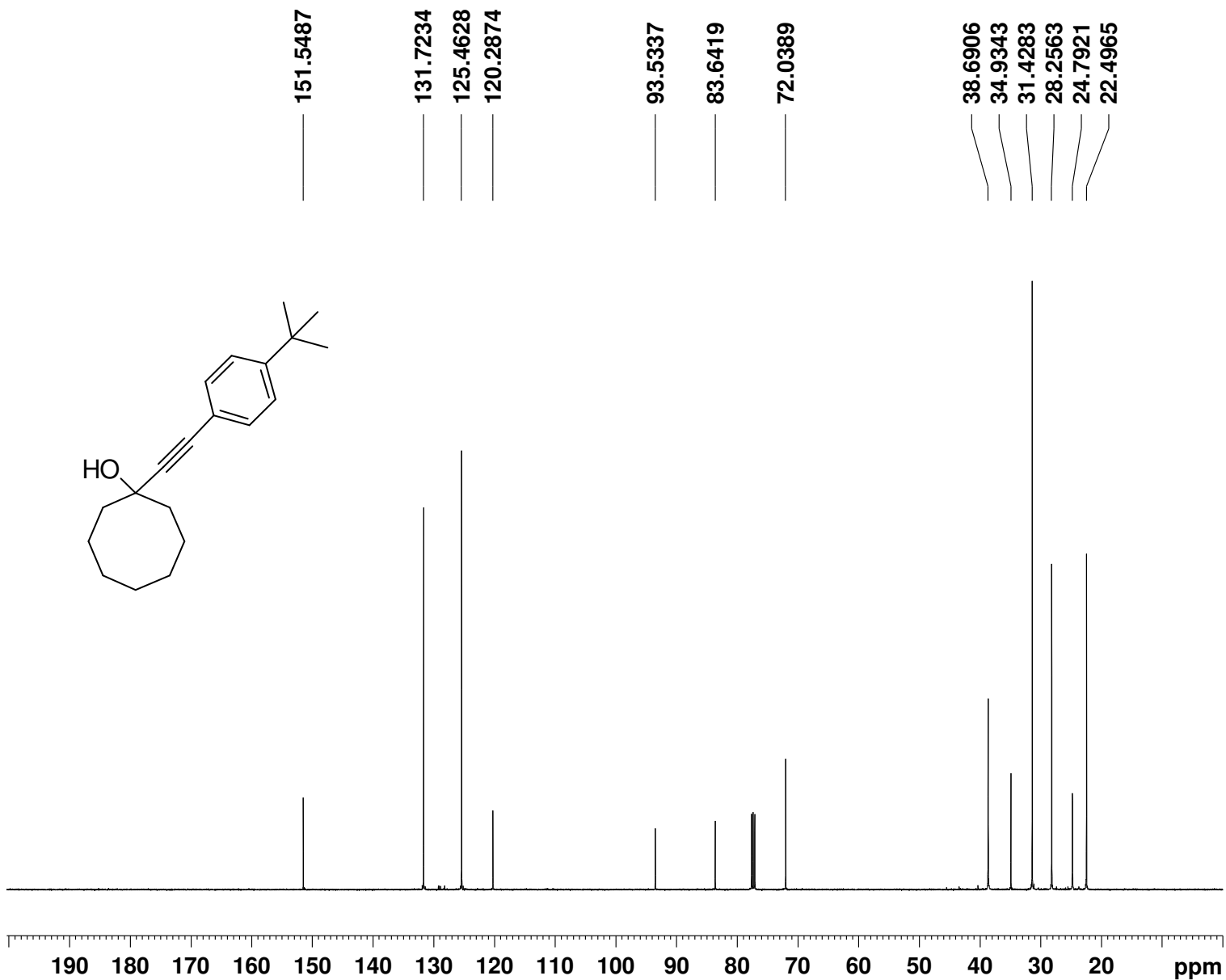
Current Data Parameters
NAME ARM-1206_3
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120130
Time 11.01
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.2
DW 48.400 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
PL1W 22.82879257 W
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-((4-*tert*-butylphenyl)ethynyl)cyclooctanol 2n



Current Data Parameters
NAME ARM-1206_3
EXPNO 11
PROCNO 1

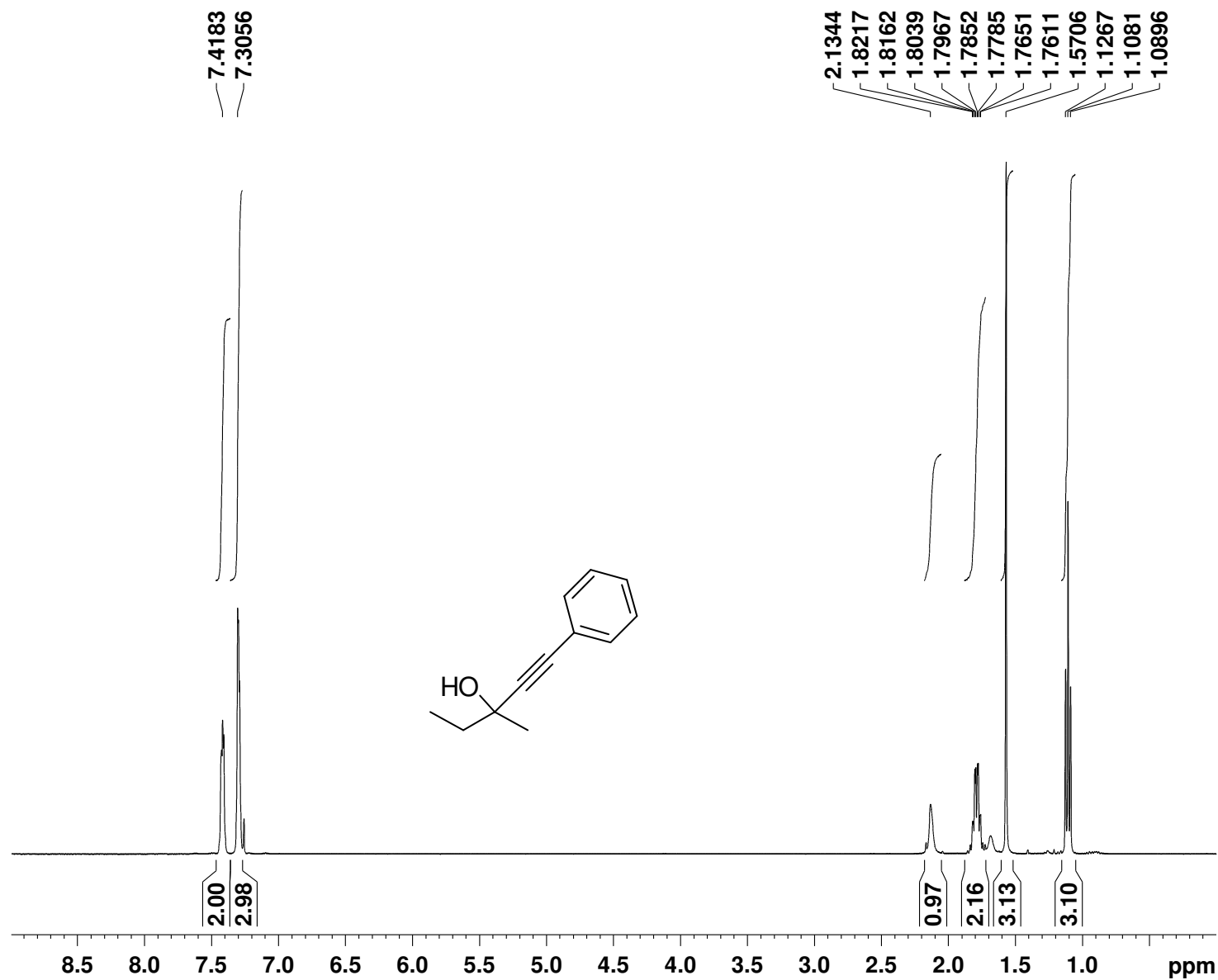
F2 - Acquisition Parameters
Date_ 20120130
Time 11.05
INSTRUM av500sgu
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 13000
DW 16.650 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 8

=====
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

=====
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
PL2W 23.36054420 W
PL12W 0.37023968 W
PL13W 0.37023968 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577611 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 3-methyl-1-phenylpent-1-yn-3-ol 2p



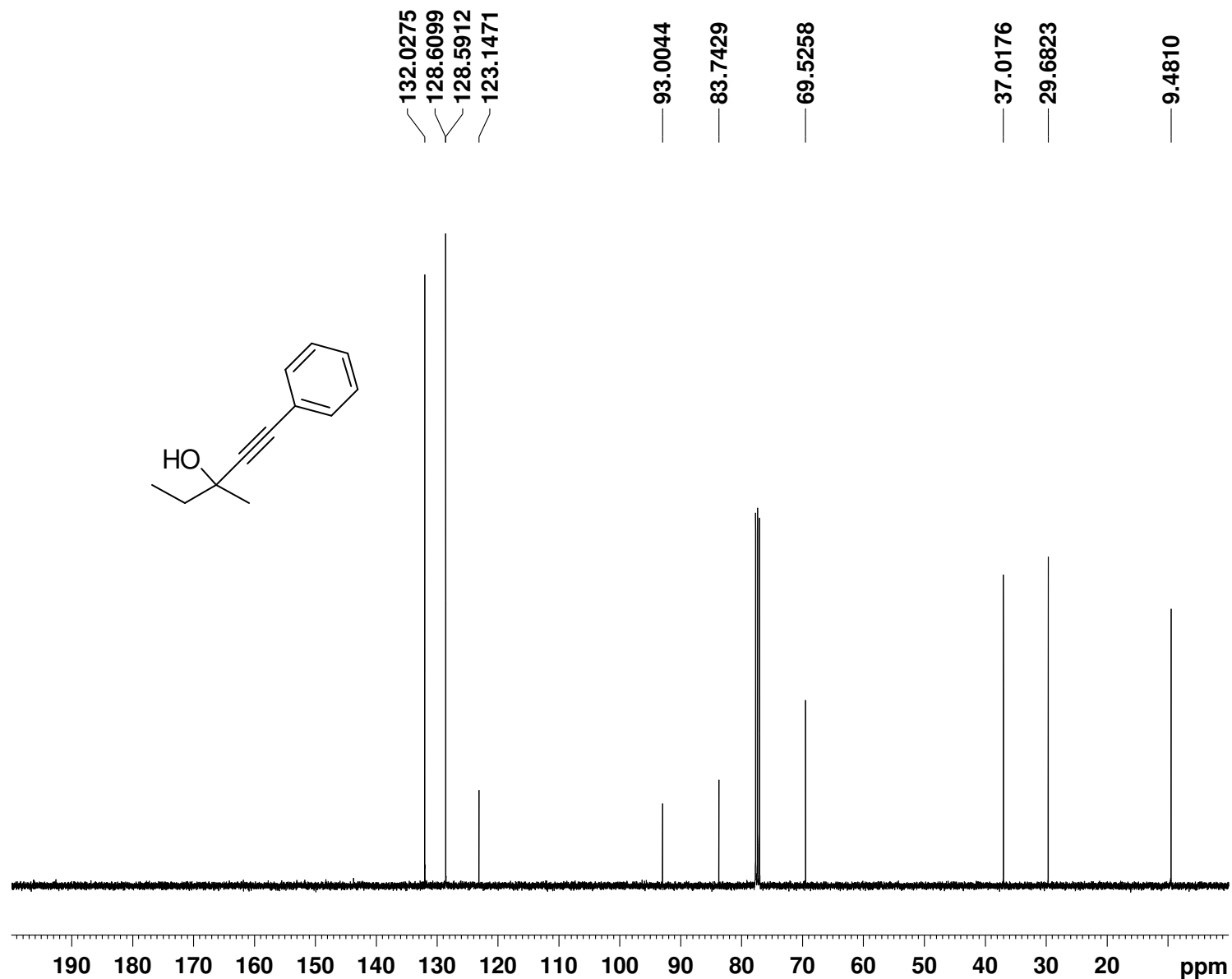
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Current Data Parameters
NAME          WM-502
EXPNO         20
PROCNO        1

F2 - Acquisition Parameters
Date_         20120814
Time          12.38
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG            59.2
DW            60.800 usec
DE            10.69 usec
TE            294.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SF01          400.1324710 MHz
NUC1          1H
P1            8.00 usec
PLW1         24.00000000 W

F2 - Processing parameters
SI            131072
SF            400.1300103 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.50
```

¹³C NMR spectrum for 3-methyl-1-phenylpent-1-yn-3-ol 2p



```
Current Data Parameters
NAME          WM-502
EXPNO         21
PROCNO        1

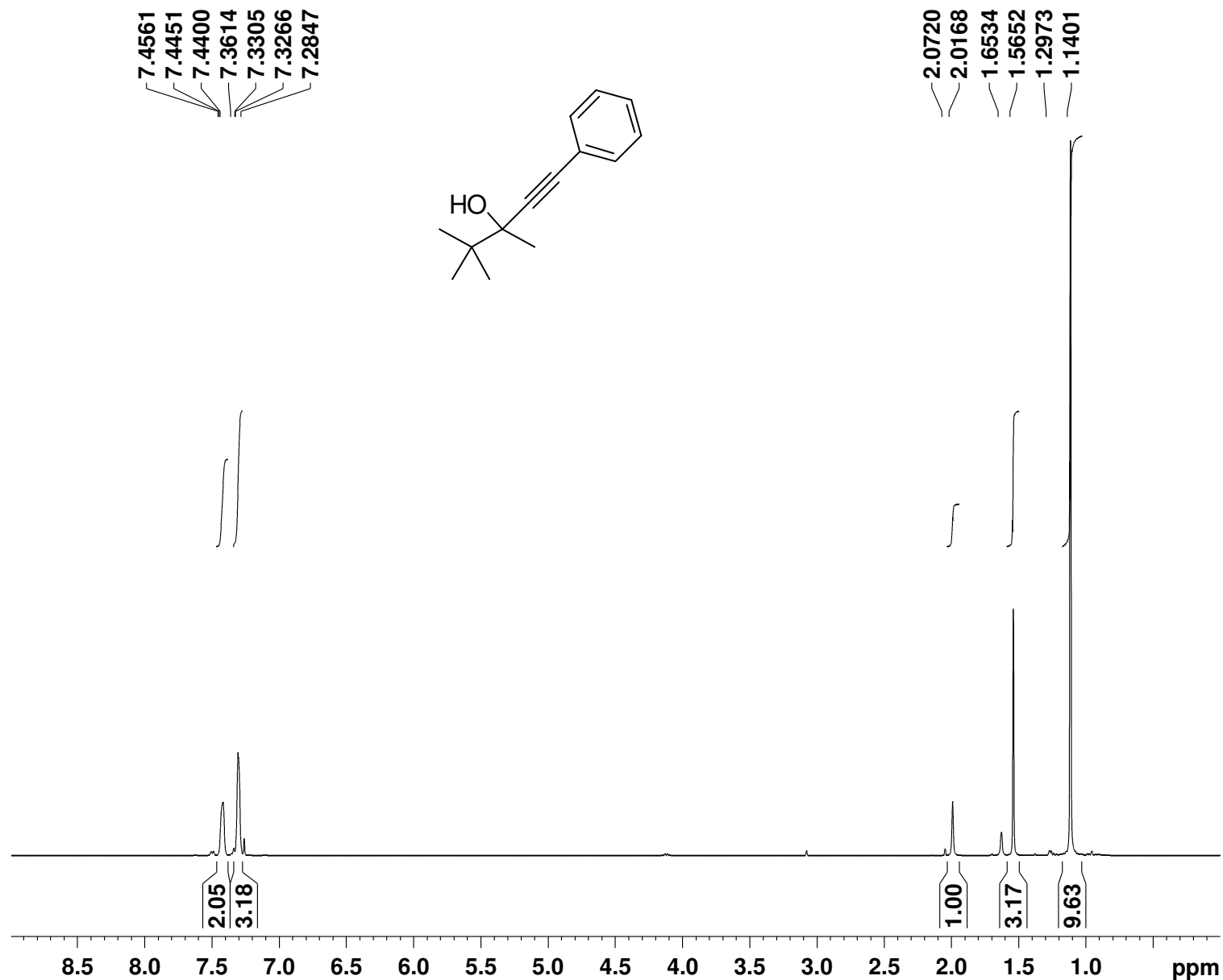
F2 - Acquisition Parameters
Date_         20120814
Time          12.54
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG            181.72
DW            20.800 usec
DE            8.18 usec
TE            294.5 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
SFO1          100.6228293 MHz
NUC1          13C
P1            9.00 usec
PLW1          77.00000000 W

===== CHANNEL f2 =====
SFO2          400.1316005 MHz
NUC2          1H
CPDPRG[2]    waltz16
PCPD2        90.00 usec
PLW2          24.00000000 W
PLW12         0.17567000 W
PLW13         0.14229999 W

F2 - Processing parameters
SI            65536
SF            100.6127342 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.40
```

¹H NMR spectrum for 3,4,4-trimethyl-1-phenylpent-1-yn-3-ol 2q



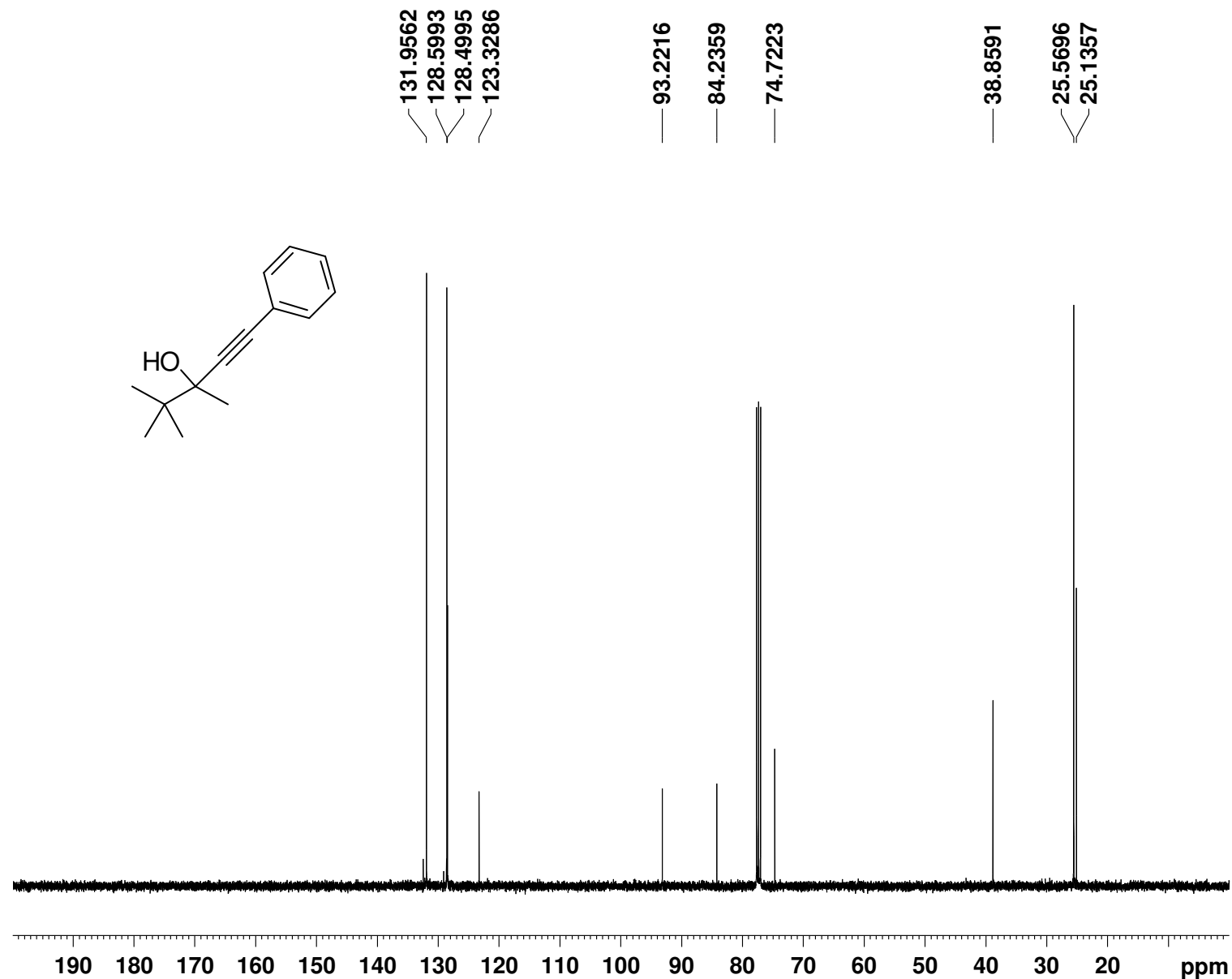
Current Data Parameters
NAME WM-505
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120830
Time 15.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 65.91
DW 60.800 usec
DE 10.69 usec
TE 294.1 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 8.00 usec
PLW1 24.00000000 W

F2 - Processing parameters
SI 131072
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.50

¹³C NMR spectrum for 3,4,4-trimethyl-1-phenylpent-1-yn-3-ol 2q



```
Current Data Parameters
NAME          WM-505
EXPNO         20
PROCNO        1

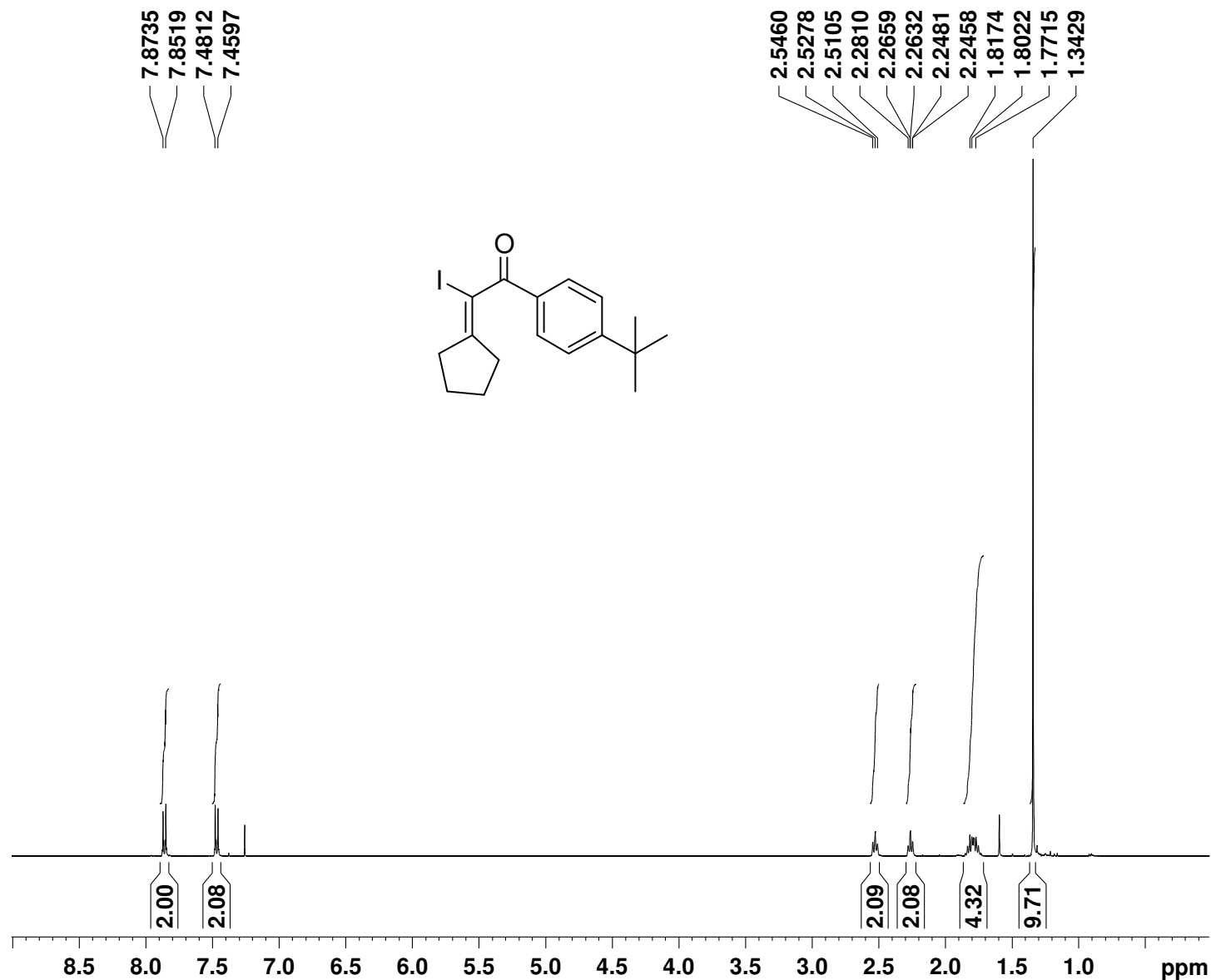
F2 - Acquisition Parameters
Date_         20120830
Time          16.52
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG            181.72
DW            20.800 usec
DE            8.18 usec
TE            294.5 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
SF01          100.6228293 MHz
NUC1           13C
P1             9.00 usec
PLW1          77.0000000 W

===== CHANNEL f2 =====
SF02          400.1316005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2         90.00 usec
PLW2          24.0000000 W
PLW12         0.1756700 W
PLW13         0.14229999 W

F2 - Processing parameters
SI            65536
SF            100.6127350 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.40
```


¹H NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclopentylidene-2-iodoethanone 4d



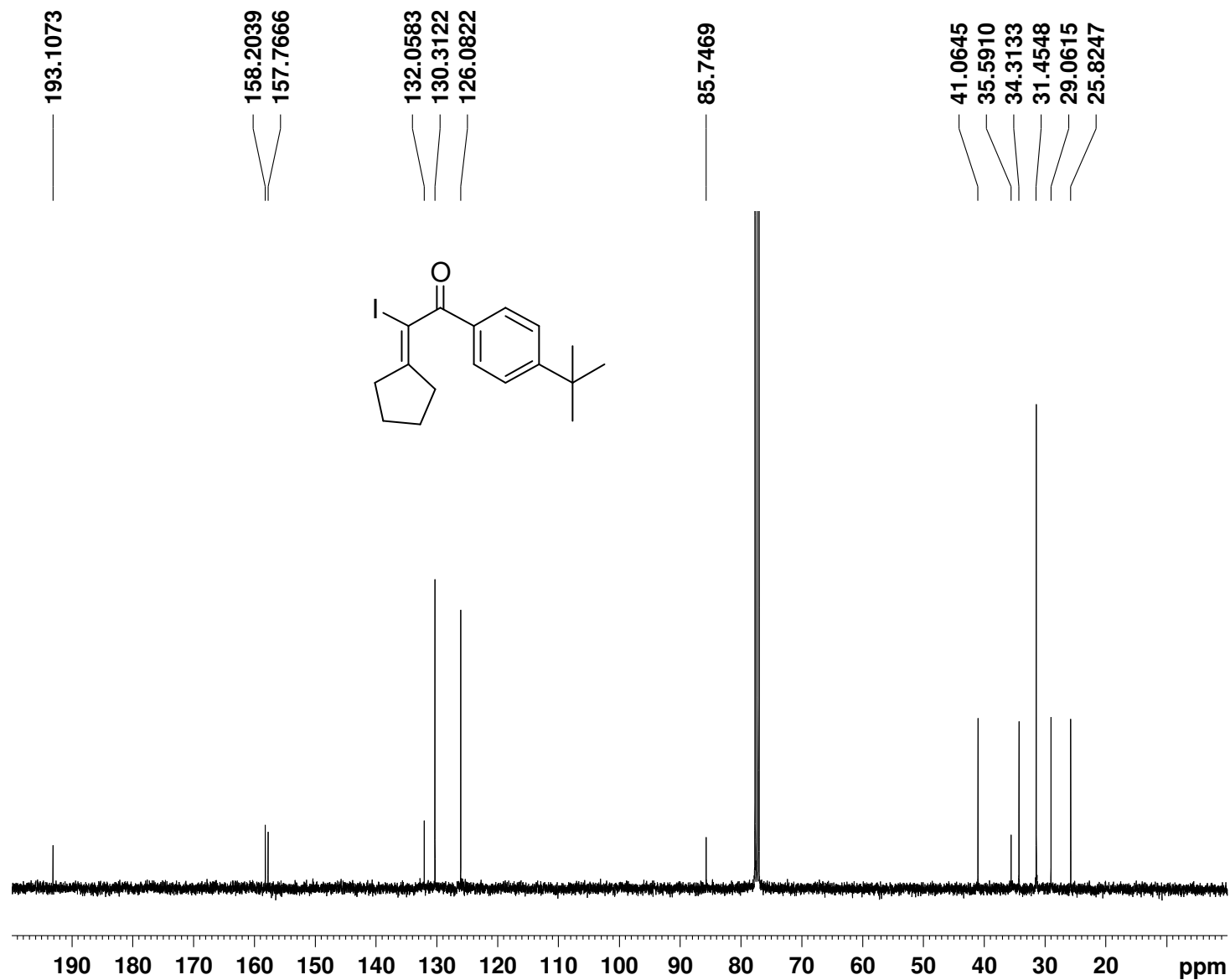
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Current Data Parameters
NAME      ARM-1235
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20110922
Time     13.57
INSTRUM  dpx400
PROBHD   5 mm QNP 1H/1
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ       3.9584243 sec
RG       228.1
DW       60.400 usec
DE       6.00 usec
TE       292.1 K
D1       1.00000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     1H
P1       8.40 usec
PL1     -3.00 dB
SFO1    400.1324710 MHz

F2 - Processing parameters
SI       32768
SF       400.1300096 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
```

¹³C NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclopentylidene-2-iodoethanone 4d



```
Current Data Parameters
NAME      ARM-1235
EXPNO     8
PROCNO    1

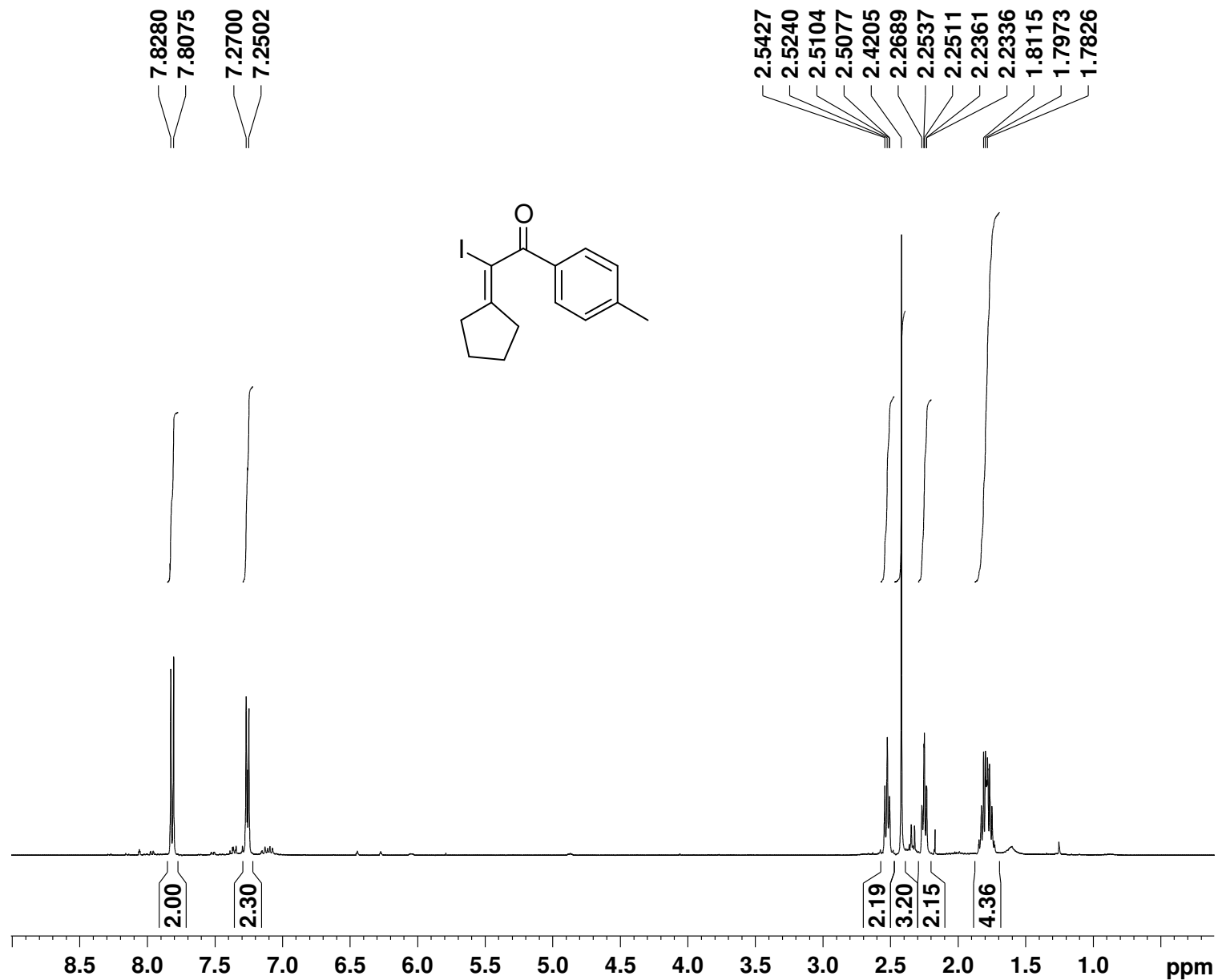
F2 - Acquisition Parameters
Date_     20110922
Time      14.53
INSTRUM   dpx400
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         11585.2
DW         20.850 usec
DE         6.00 usec
TE         292.5 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        8

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        -3.00 dB
SFO1       100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      84.00 usec
PL2        -3.00 dB
PL12       16.08 dB
PL13       18.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127338 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

¹H NMR spectrum for 2-cyclopentylidene-2-iodo-1-*p*-tolylethanone 4e



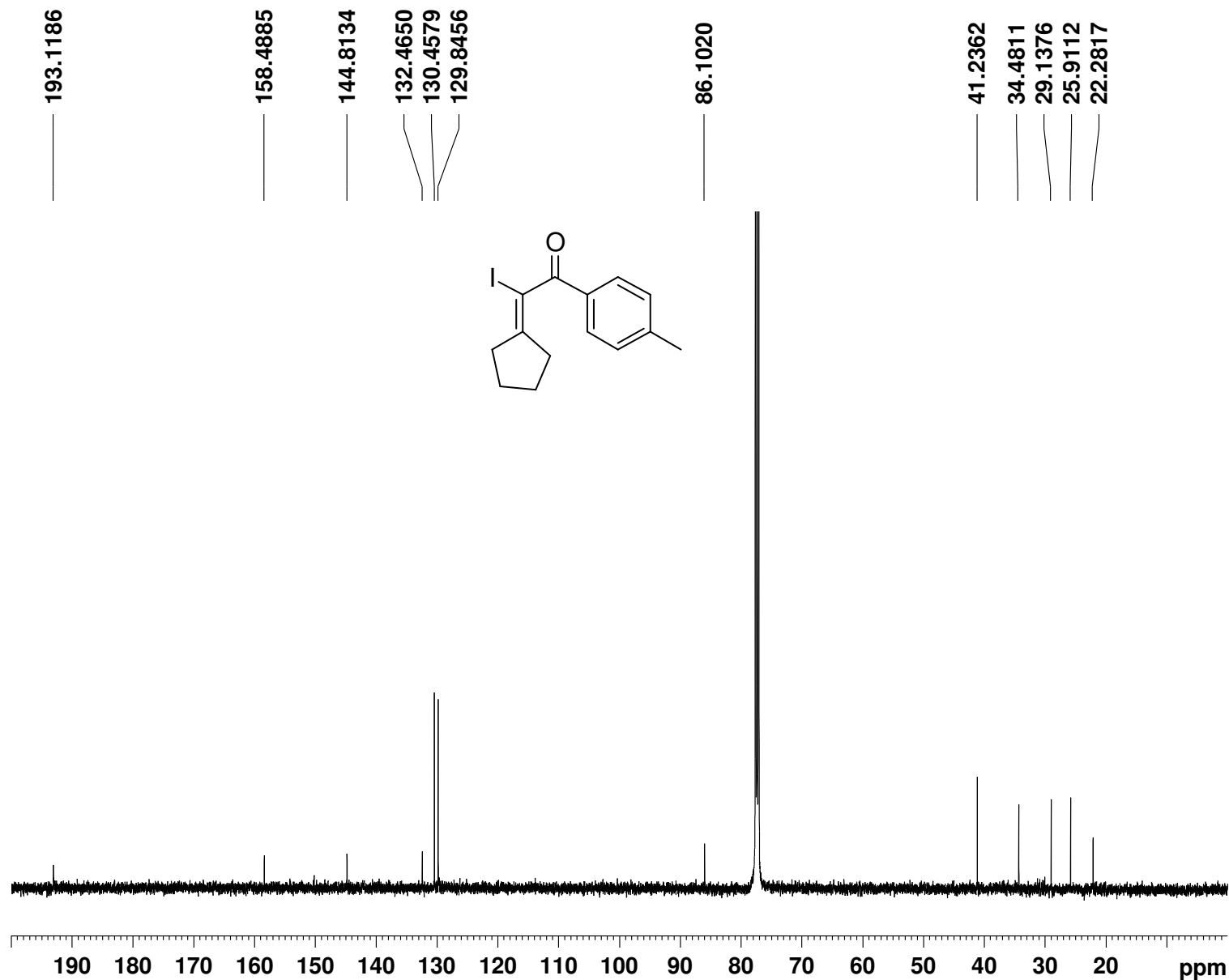
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Current Data Parameters
NAME      ARM-1380_500
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20111129
Time      13.19
INSTRUM   dpx400
PROBHD    5 mm QNP 1H/1
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         228.1
DW         60.400 usec
DE         6.00 usec
TE         293.0 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1        8.40 usec
PL1       -3.00 dB
SFO1      400.1324710 MHz

F2 - Processing parameters
SI         32768
SF         400.1300095 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```

¹³C NMR spectrum for 2-cyclopentylidene-2-iodo-1-*p*-tolylethanone 4e



```
Current Data Parameters
NAME      ARM-1252_500
EXPNO     3
PROCNO    1

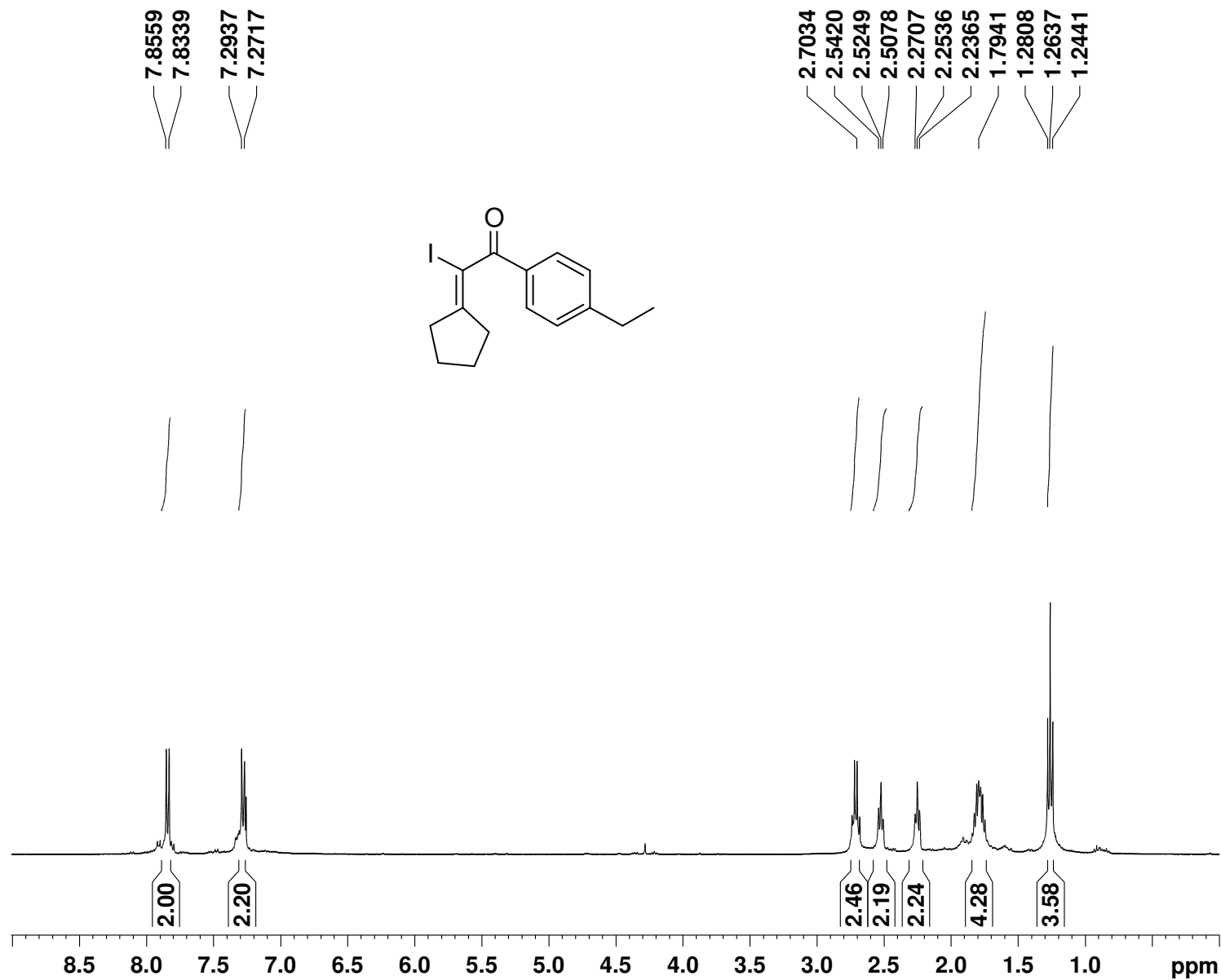
F2 - Acquisition Parameters
Date_     20111005
Time      18.07
INSTRUM   av500
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        9600
DS        4
SWH       30030.029 Hz
FIDRES    0.458222 Hz
AQ        1.0912244 sec
RG        16400
DW        16.650 usec
DE        6.00 usec
TE        303.6 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA     1.89999998 sec
TDO       300

===== CHANNEL f1 =====
NUC1      13C
P1        9.00 usec
PL1       0.00 dB
SFO1      125.7703637 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       0.00 dB
PL12      18.00 dB
PL13      18.00 dB
SFO2      500.1320005 MHz

F2 - Processing parameters
SI        32768
SF        125.7577384 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

¹H NMR spectrum for 2-cyclopentylidene-1-(4-ethylphenyl)-2-iodoethanone 4f



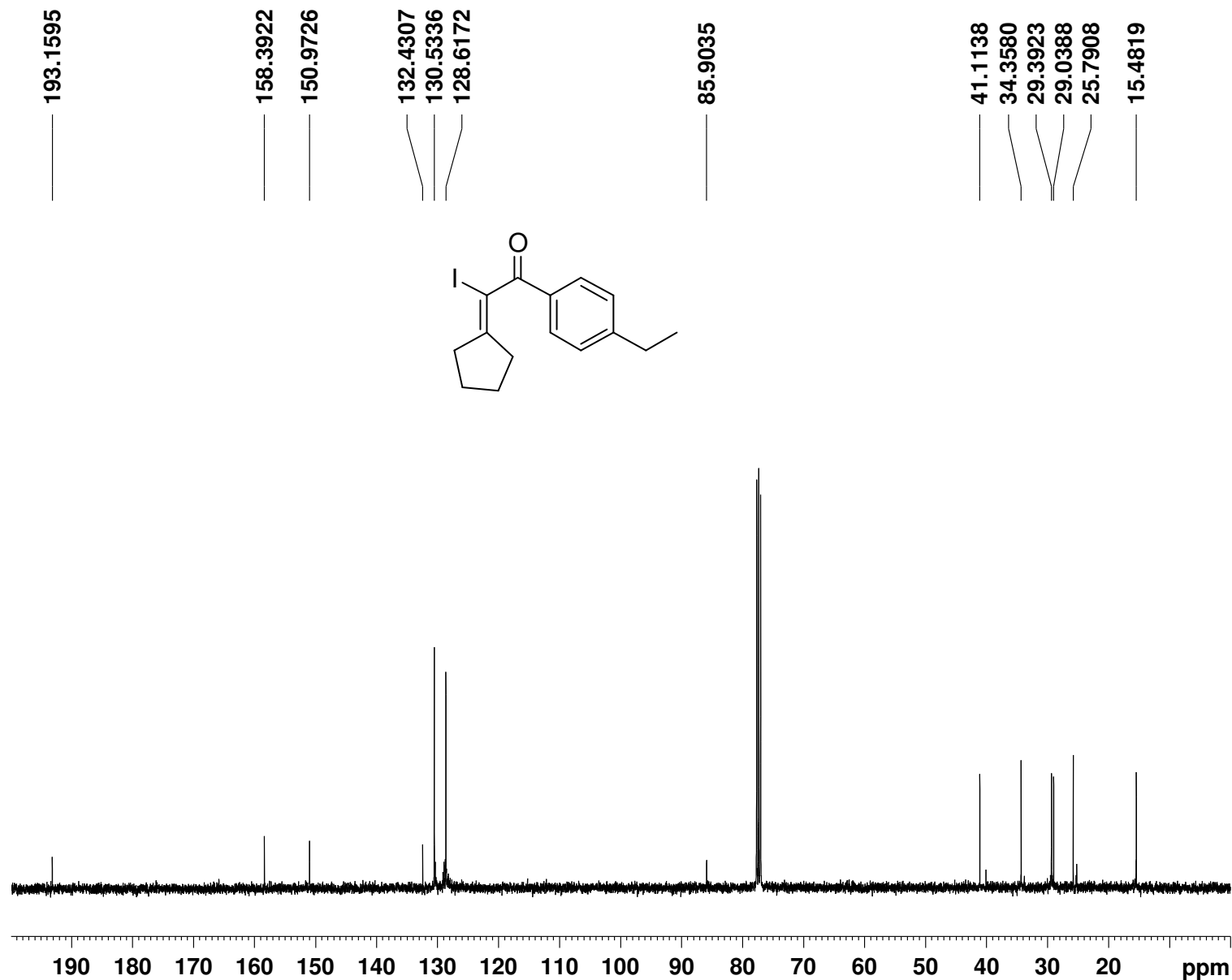
Current Data Parameters
NAME ARM-1376
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111107
Time 9.43
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 291.2 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 2-cyclopentylidene-1-(4-ethylphenyl)-2-iodoethanone 4f



Current Data Parameters
NAME ARM-1376
EXPNO 8
PROCNO 1

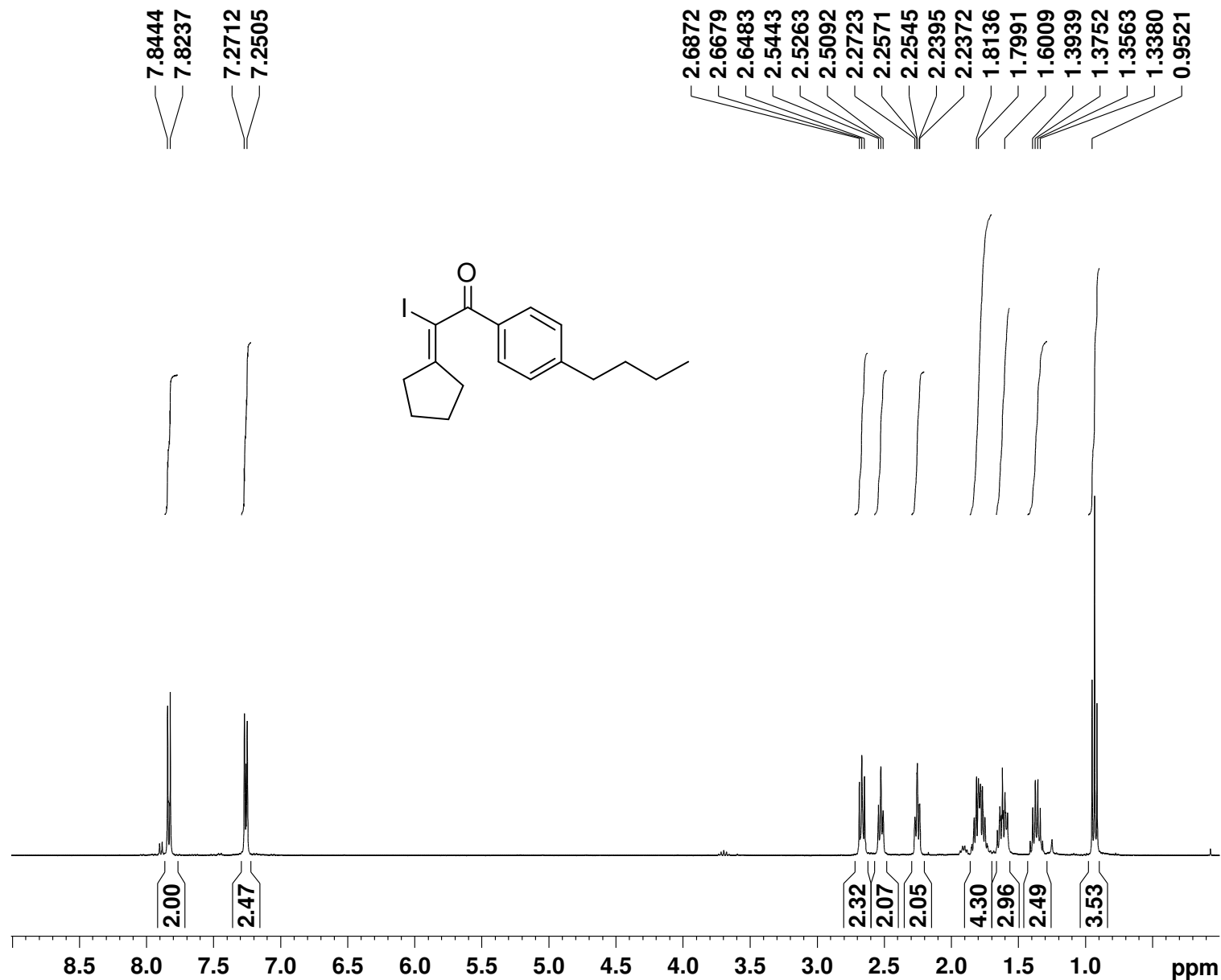
F2 - Acquisition Parameters
Date_ 20111107
Time 10.26
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TD 65536
SOLVENT CDCl3
NS 224
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 6502
DW 20.850 usec
DE 6.00 usec
TE 291.8 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 8

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127359 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-(4-butylphenyl)-2-cyclopentylidene-2-iodoethanone 4g



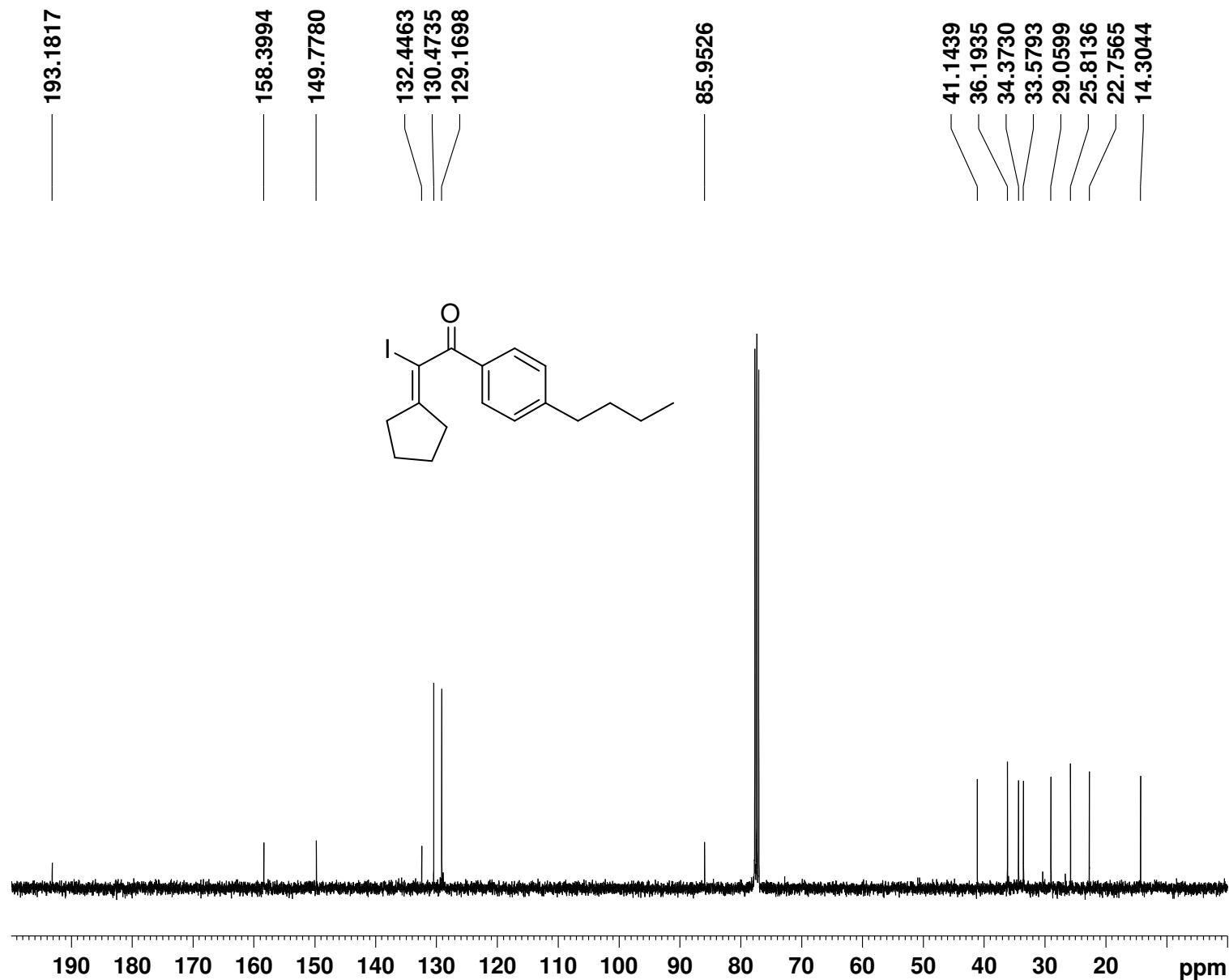
Current Data Parameters
NAME ARM-1300
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111020
Time 9.51
INSTRUM dp400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-(4-butylphenyl)-2-cyclopentylidene-2-iodoethanone 4g



Current Data Parameters
NAME ARM-1300
EXPNO 6
PROCNO 1

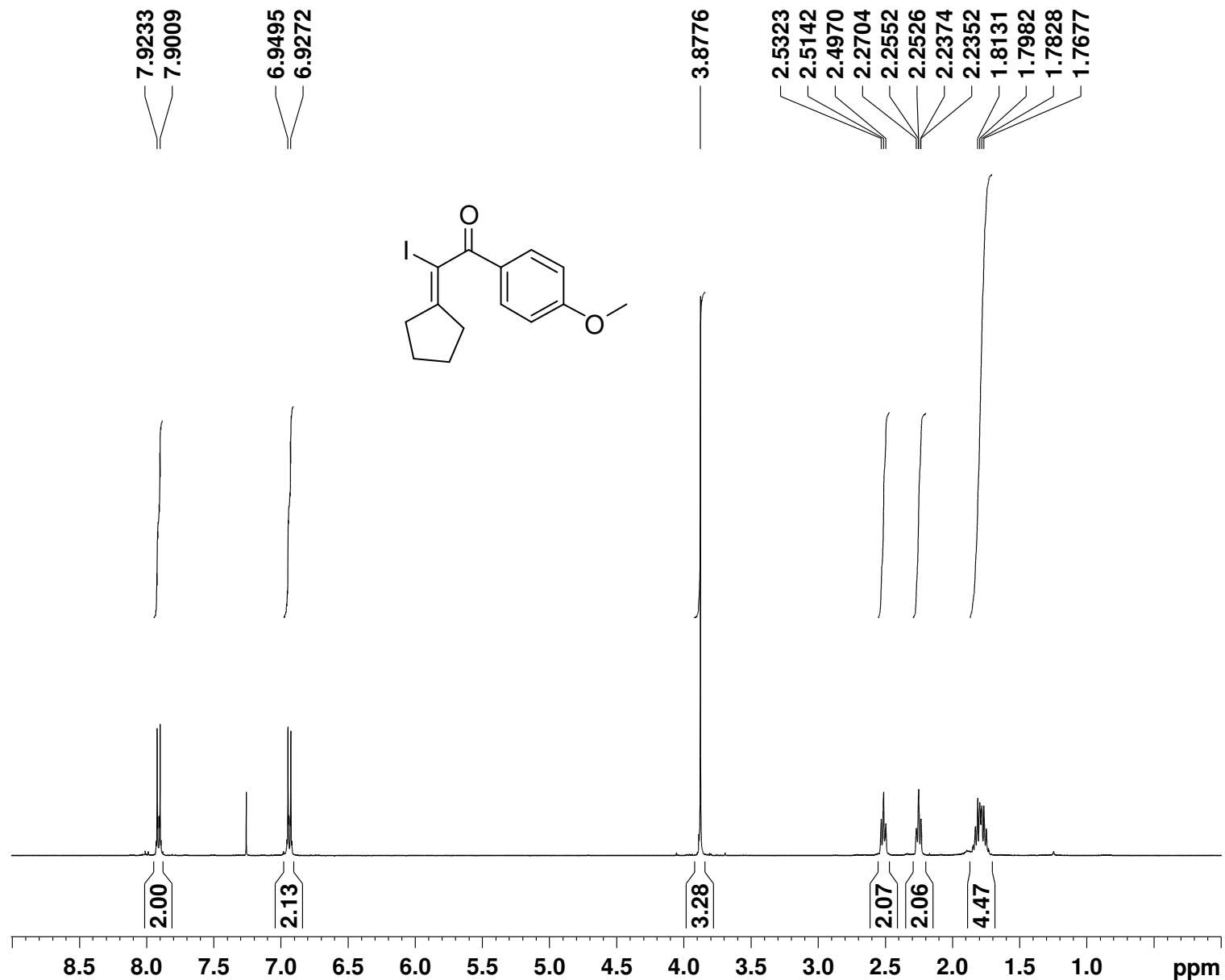
F2 - Acquisition Parameters
Date_ 20110929
Time 15.18
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 6502
DW 20.850 usec
DE 6.00 usec
TE 292.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127328 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 2-cyclopentylidene-2-iodo-1-(4-methoxyphenyl)ethanone 4h



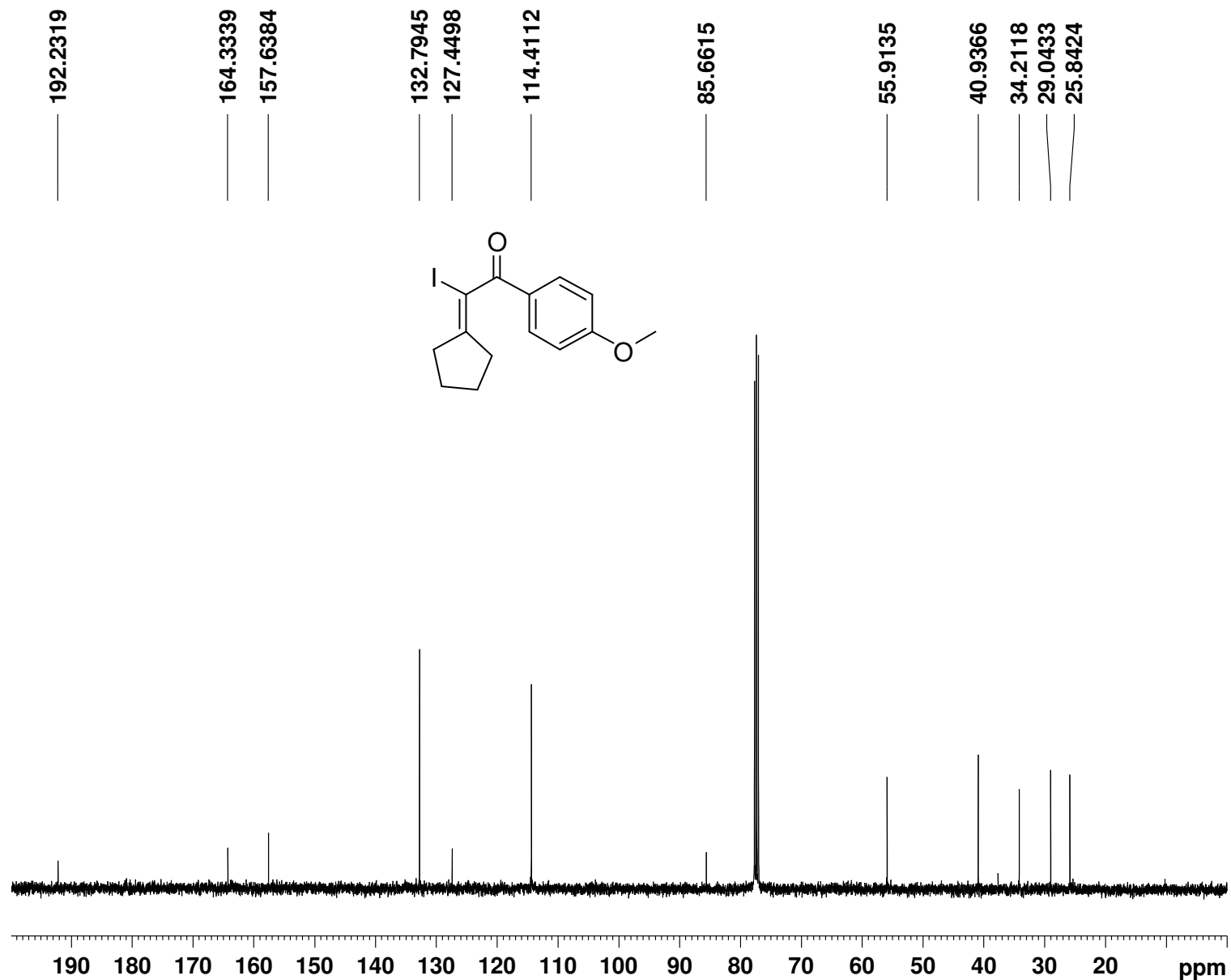
Current Data Parameters
NAME ARM-1301
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 2-cyclopentylidene-2-iodo-1-(4-methoxyphenyl)ethanone 4h



```
Current Data Parameters
NAME      ARM-1301
EXPNO    6
PROCNO   1

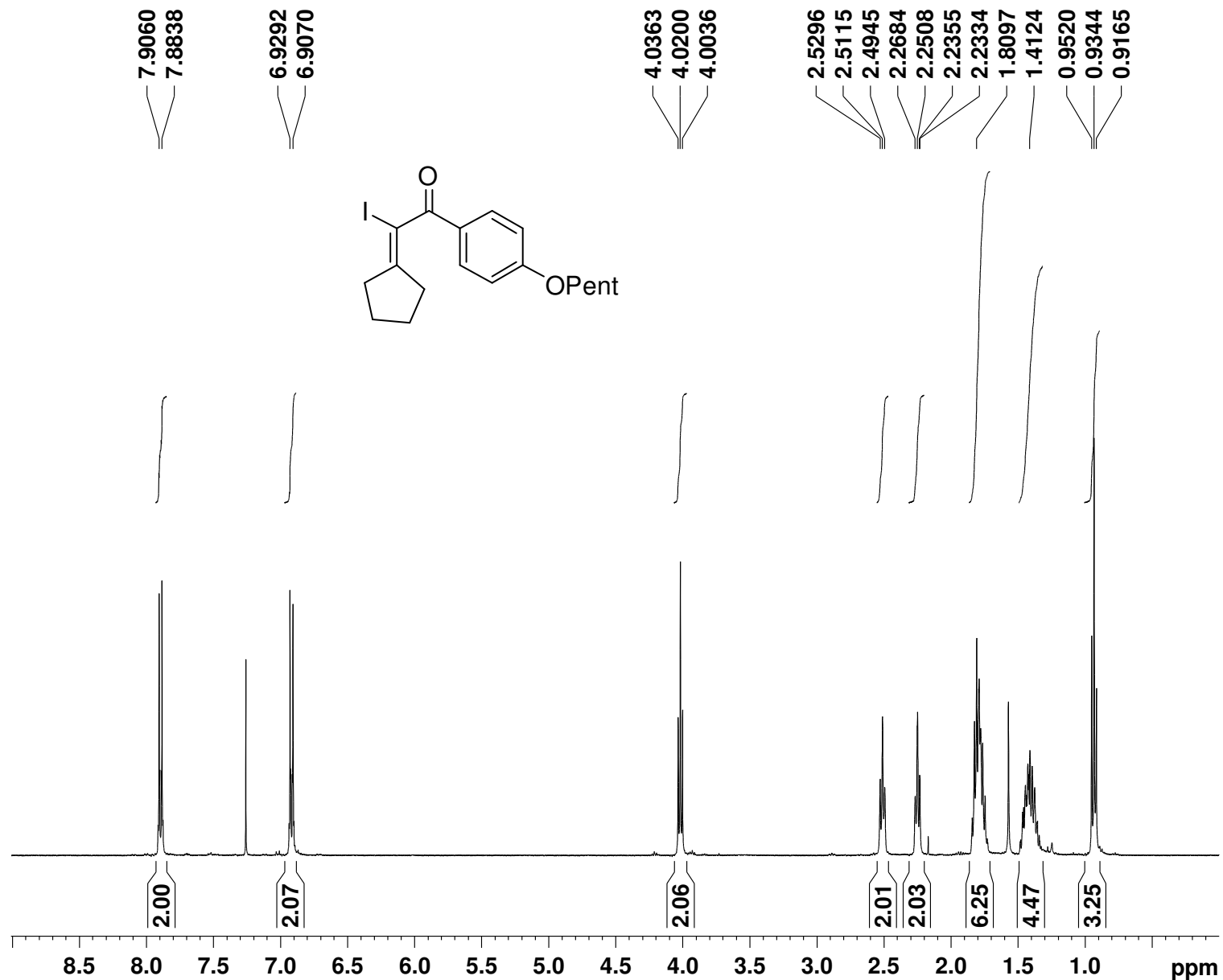
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Time     9.57
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PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       256
DS       4
SWH      23980.814 Hz
FIDRES   0.365918 Hz
AQ       1.3664756 sec
RG       18390.4
DW       20.850 usec
DE       6.00 usec
TE       292.0 K
D1       2.00000000 sec
d11      0.03000000 sec
DELTA    1.89999998 sec
TDO      8

===== CHANNEL f1 =====
NUC1     13C
P1       9.70 usec
PL1      -3.00 dB
SFO1     100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    84.00 usec
PL2      -3.00 dB
PL12     16.08 dB
PL13     18.00 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127329 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

¹H NMR spectrum for 2-cyclopentylidene-2-iodo-1-(4-(pentyloxy)phenyl)ethanone 4i



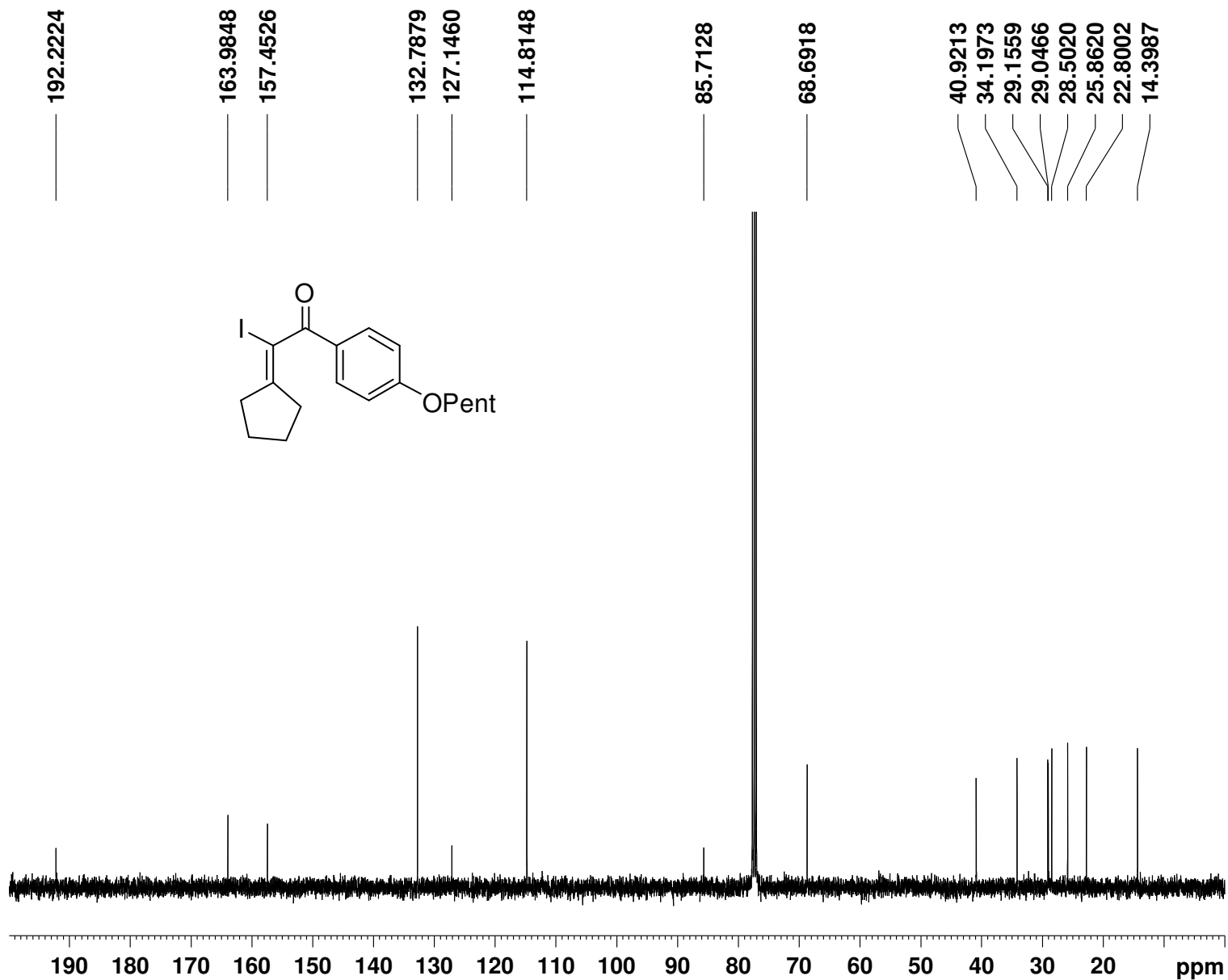
Current Data Parameters
NAME ARM-1327
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111020
Time 9.56
INSTRUM dp400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 2-cyclopentylidene-2-iodo-1-(4-(pentyloxy)phenyl)ethanone 4i



Current Data Parameters
NAME ARM-1327
EXPNO 5
PROCNO 1

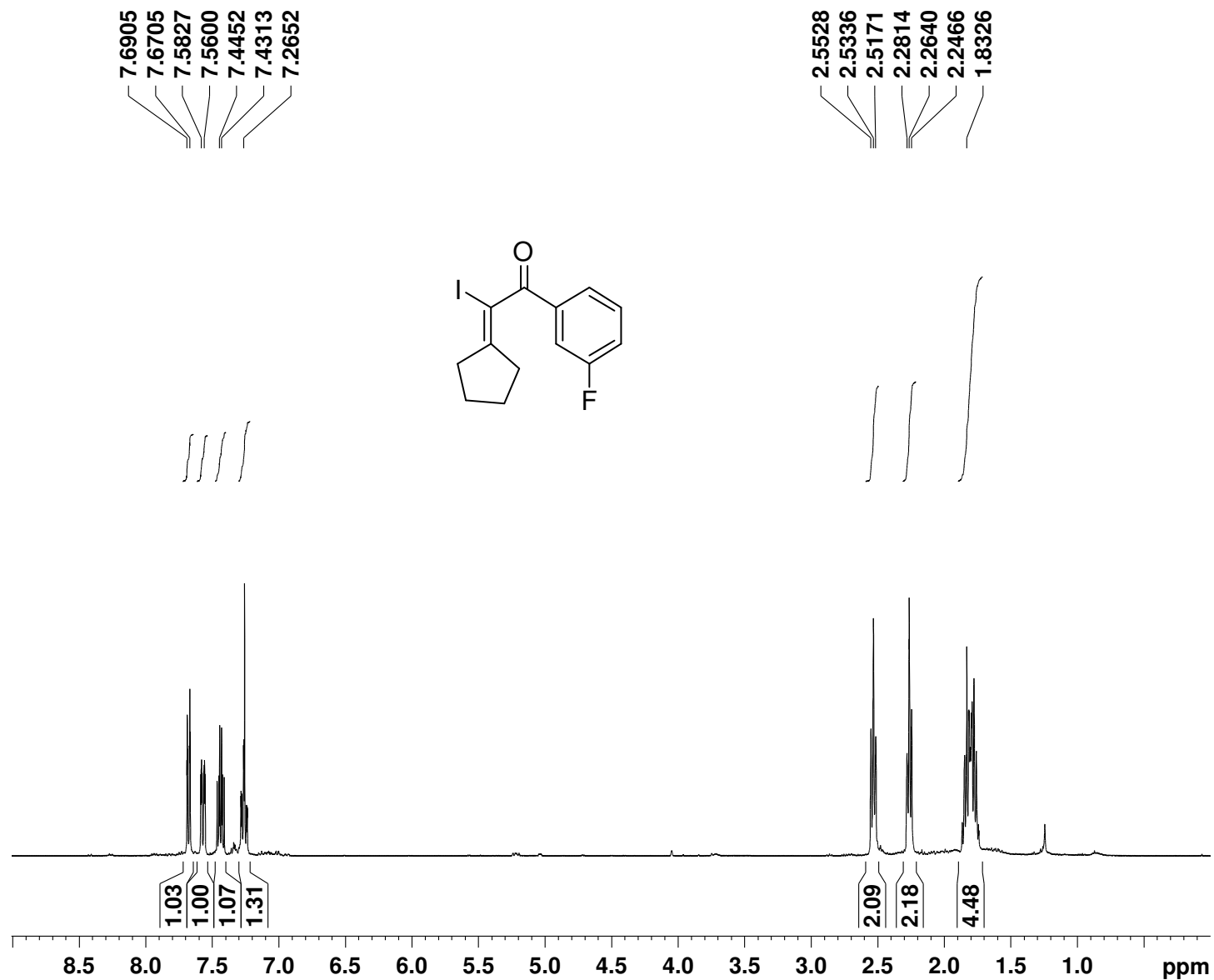
F2 - Acquisition Parameters
Date_ 20111020
Time 11.14
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 6.00 usec
TE 293.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 8

==== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127323 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 2-cyclopentylidene-1-(3-fluorophenyl)-2-iodoethanone 4j



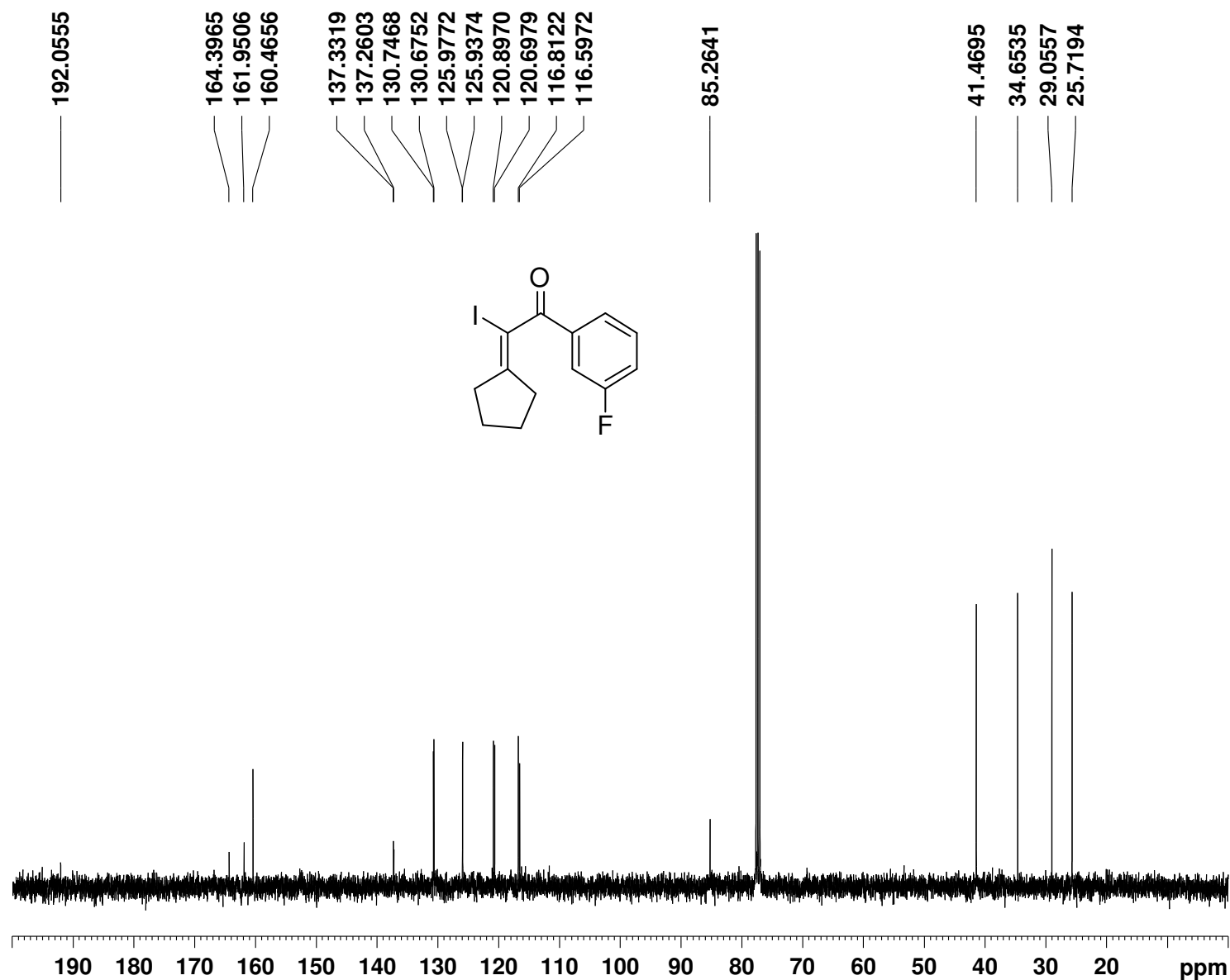
Current Data Parameters
NAME ARM-1369
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111027
Time 16.13
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 292.6 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 2-cyclopentylidene-1-(3-fluorophenyl)-2-iodoethanone 4j



```
Current Data Parameters
NAME      ARM-1369
EXPNO     7
PROCNO    1

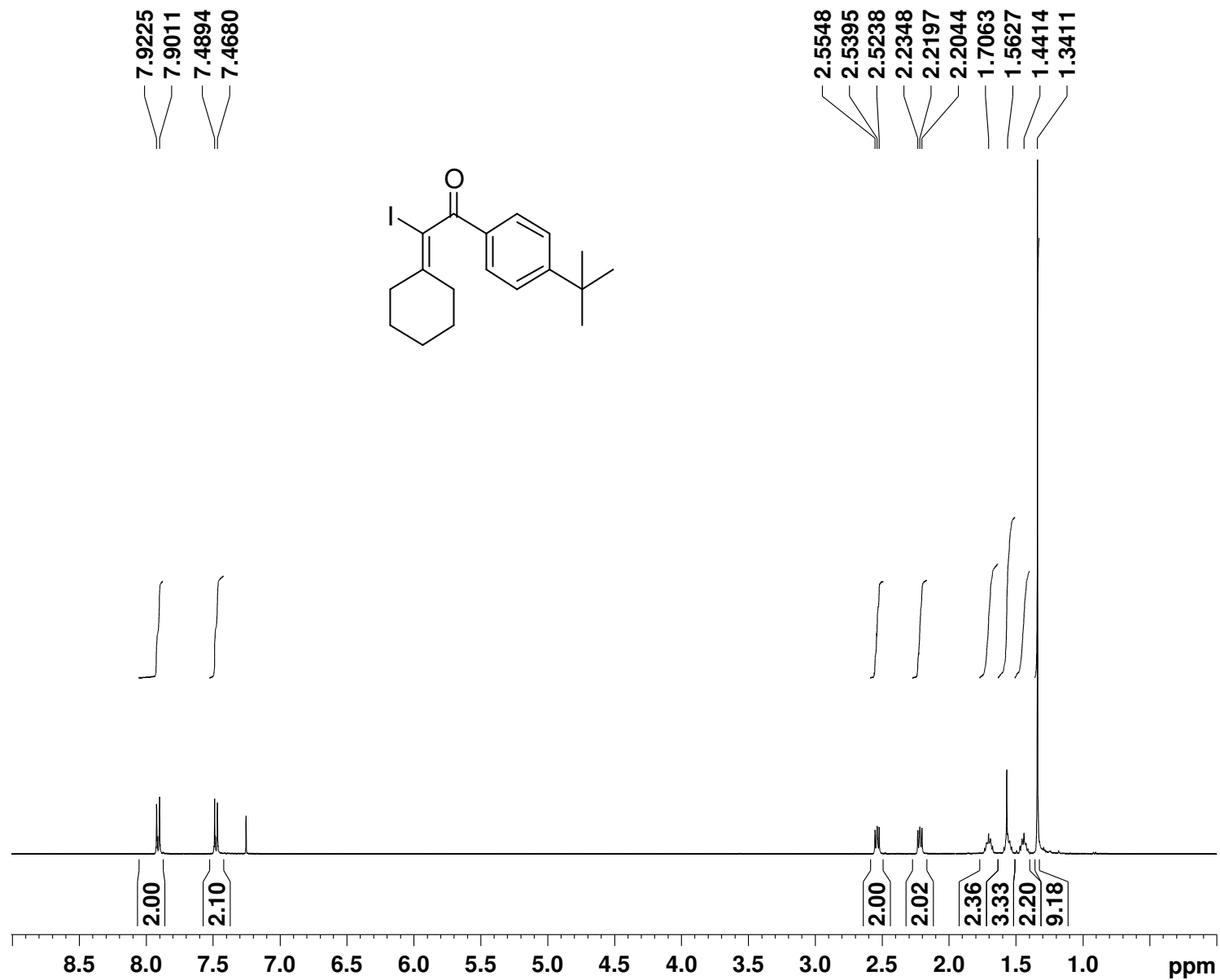
F2 - Acquisition Parameters
Date_     20111027
Time      16.16
INSTRUM   dpx400
PROBHD    5 mm QNP 1H/1
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        96
DS        4
SWH       23980.814 Hz
FIDRES    0.365918 Hz
AQ        1.3664756 sec
RG        6502
DW        20.850 usec
DE        6.00 usec
TE        293.1 K
D1        2.0000000 sec
d11       0.0300000 sec
DELTA    1.89999998 sec
TDO      8

===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1       -3.00 dB
SFO1     100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     84.00 usec
PL2       -3.00 dB
PL12     16.08 dB
PL13     18.00 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127349 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
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¹H NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclohexylidene-2-iodoethanone 4k



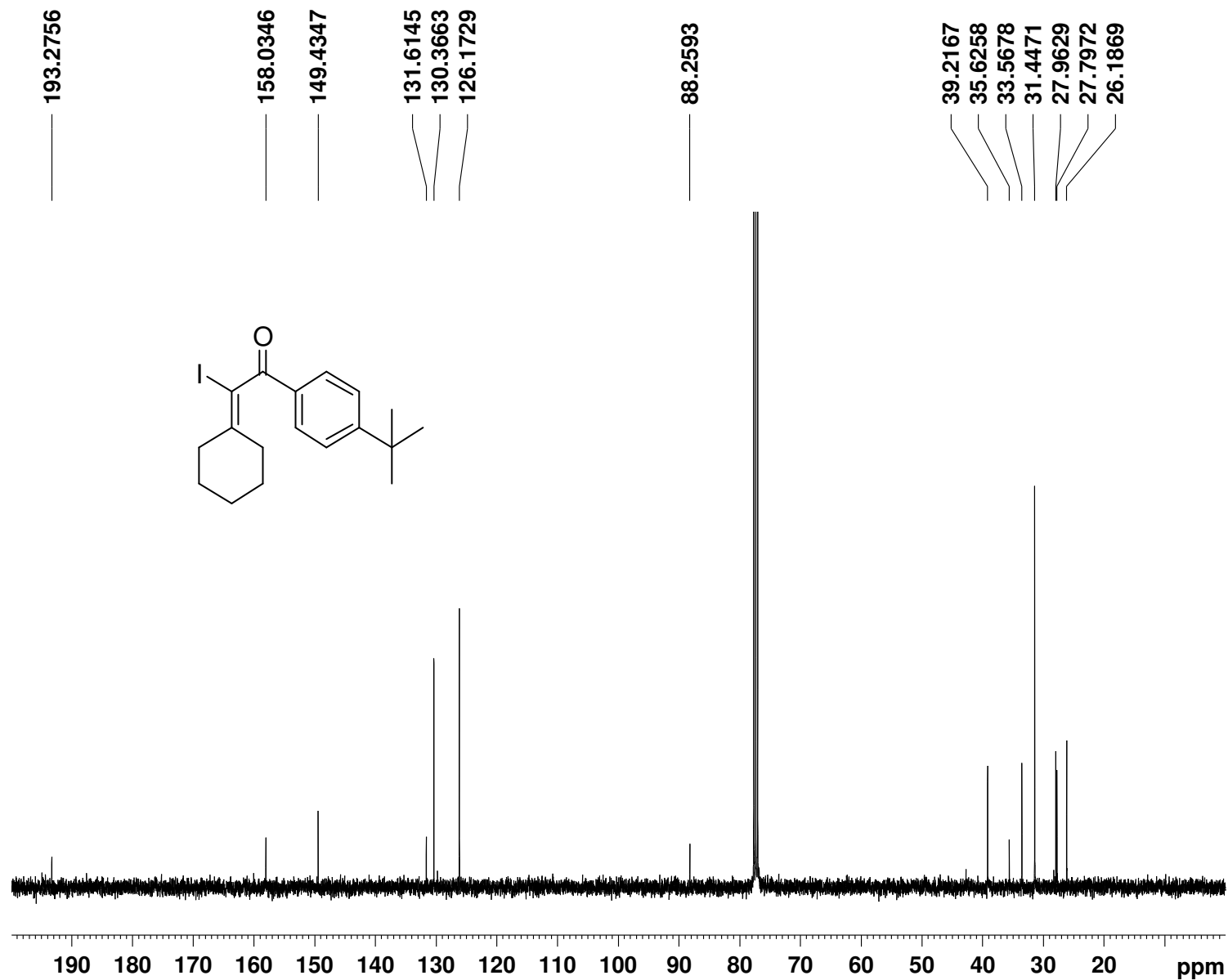
Current Data Parameters
NAME ARM-1341
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300119 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclohexylidene-2-iodoethanone 4k



Current Data Parameters
NAME ARM-1341
EXPNO 4
PROCNO 1

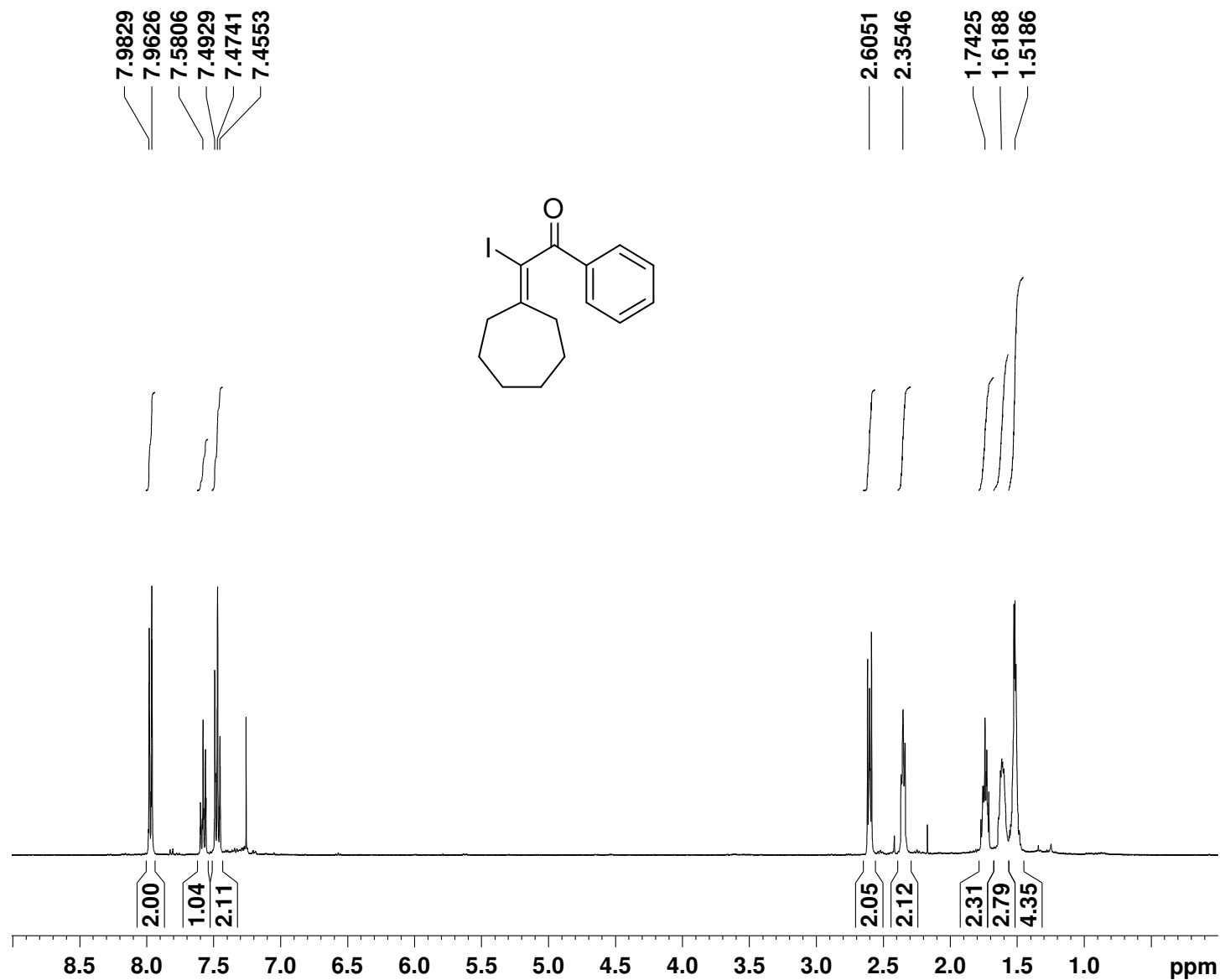
F2 - Acquisition Parameters
Date_ 20111012
Time 10.27
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.364756 sec
RG 11585.2
DW 20.850 usec
DE 6.00 usec
TE 293.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 8

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127332 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 2-cycloheptylidene-2-iodo-1-phenylethanone 4I



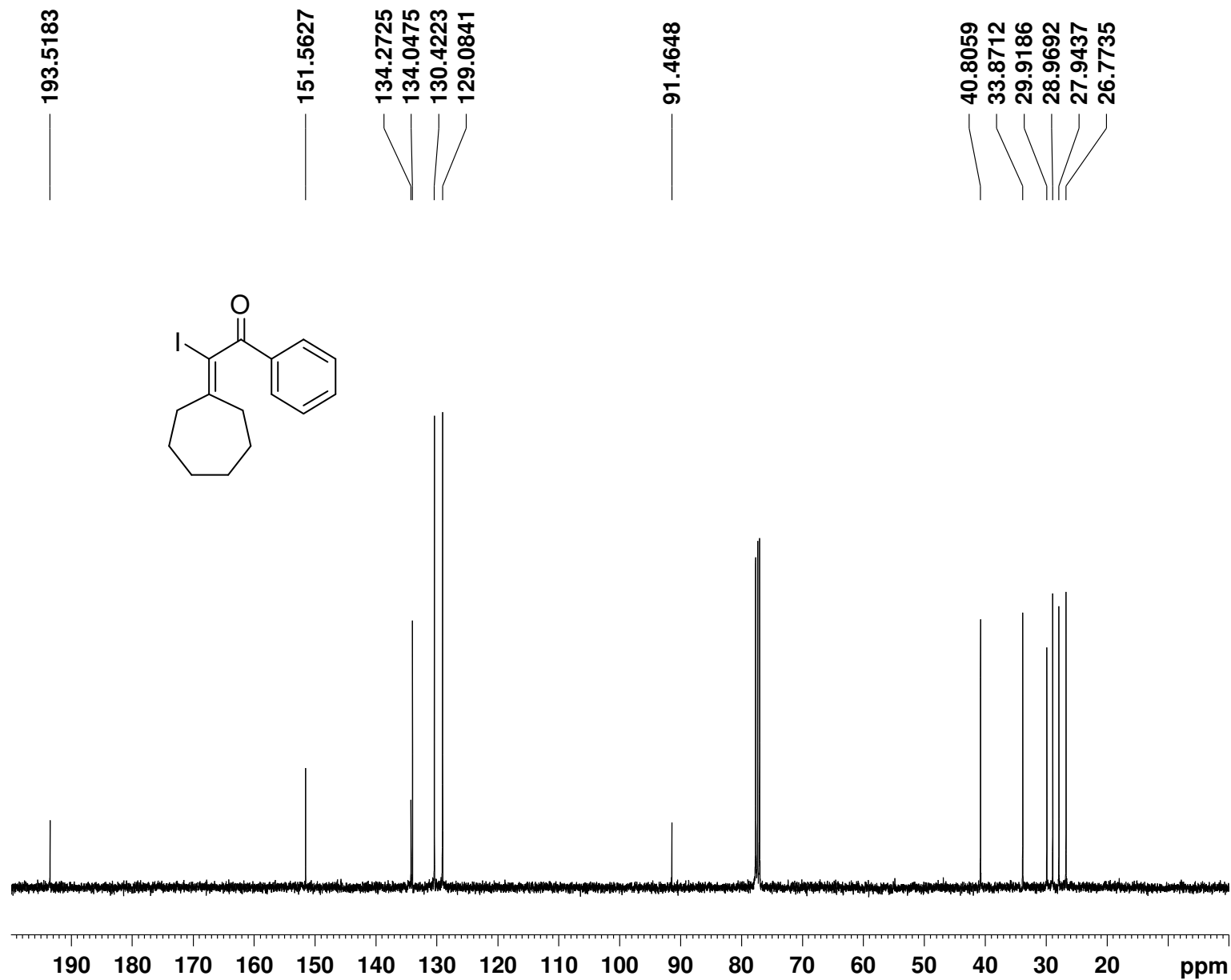
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NAME ARM-1381_500
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
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Time 15.06
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PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
RQ 60.400 usec
DW 2.919 usec
DE 6.00 usec
TE 291.9 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 2-cycloheptylidene-2-iodo-1-phenylethanone 4l



Current Data Parameters
NAME ARM-1329
EXPNO 5
PROCNO 1

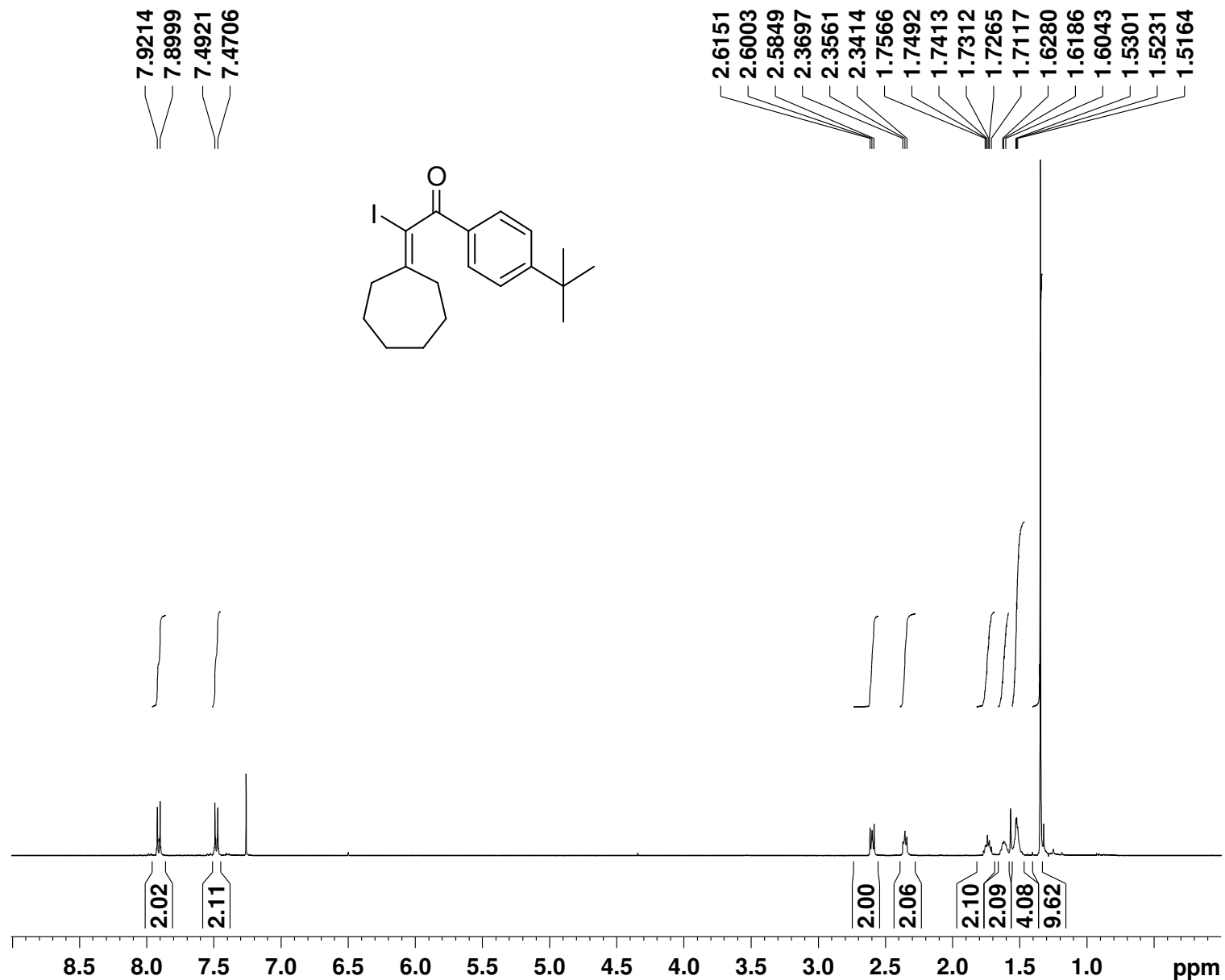
F2 - Acquisition Parameters
Date_ 20110923
Time 13.57
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 292.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 8

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
FPCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127365 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cycloheptylidene-2-iodoethanone 4m



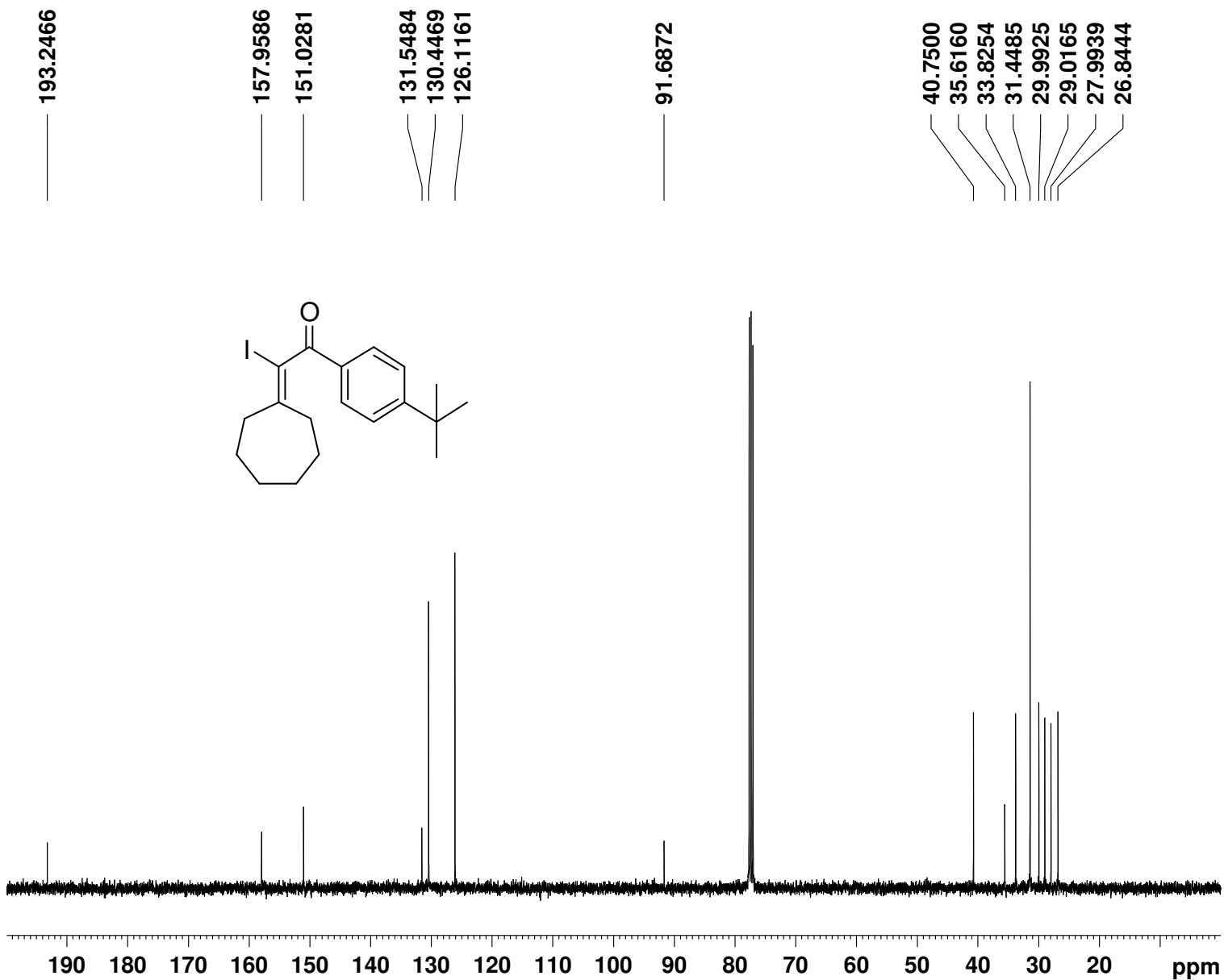
Current Data Parameters
NAME ARM-1332
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110927
Time 9.44
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cycloheptylidene-2-iodoethanone 4m



```
Current Data Parameters
NAME      ARM-1307
EXPNO     4
PROCNO    1

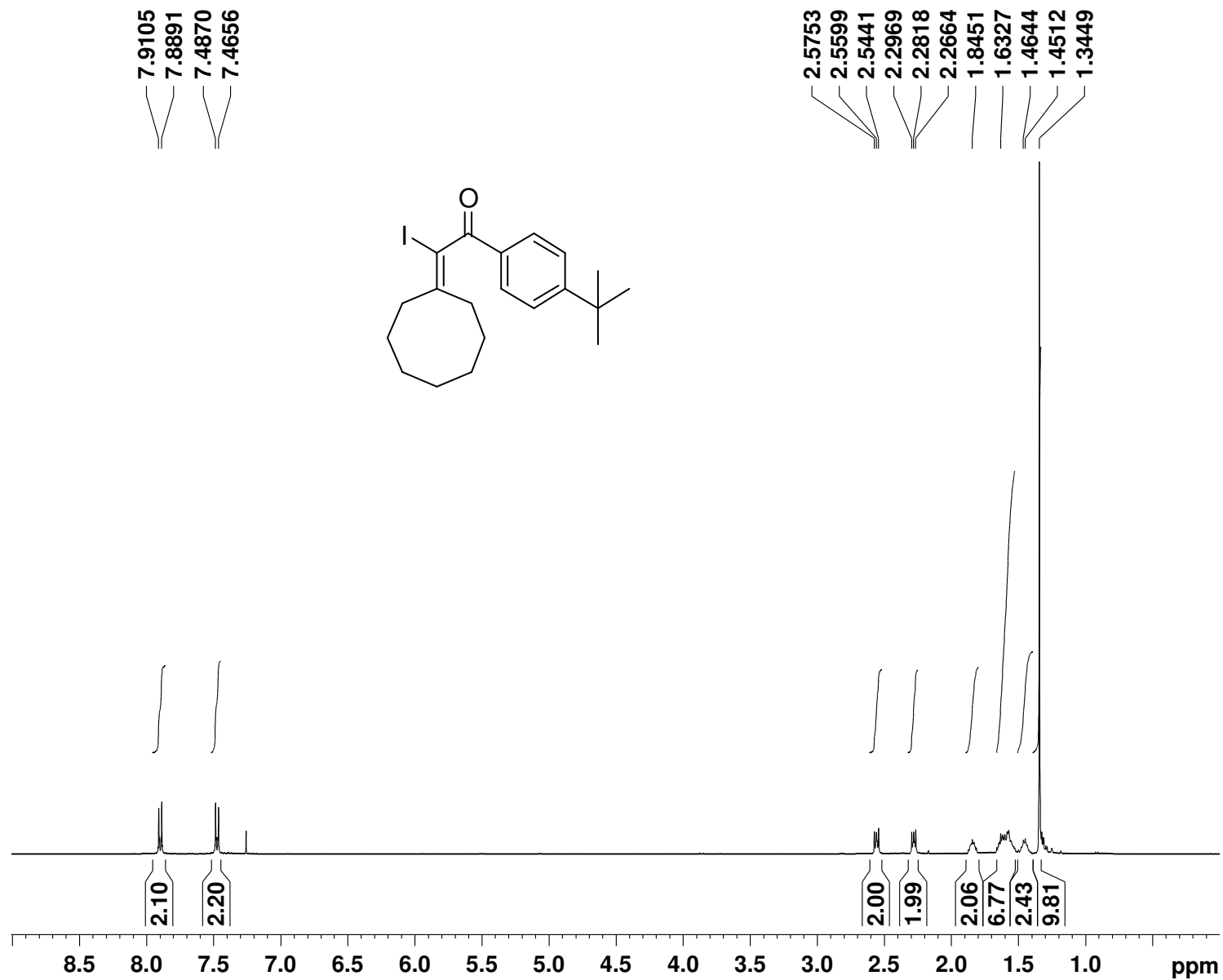
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Time      12.01
INSTRUM   dpx400
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PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         256
DS         4
SWH        23980.814 Hz
FIDRES     0.365918 Hz
AQ         1.3664756 sec
RG         20642.5
DW         20.850 usec
DE         6.00 usec
TE         293.5 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA     1.89999998 sec
TD0        8

===== CHANNEL f1 =====
NUC1       13C
P1         9.70 usec
PL1        -3.00 dB
SFO1       100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      84.00 usec
PL2        -3.00 dB
PL12       16.08 dB
PL13       18.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127338 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
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¹H NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclooctylidene-2-iodoethanone 4n



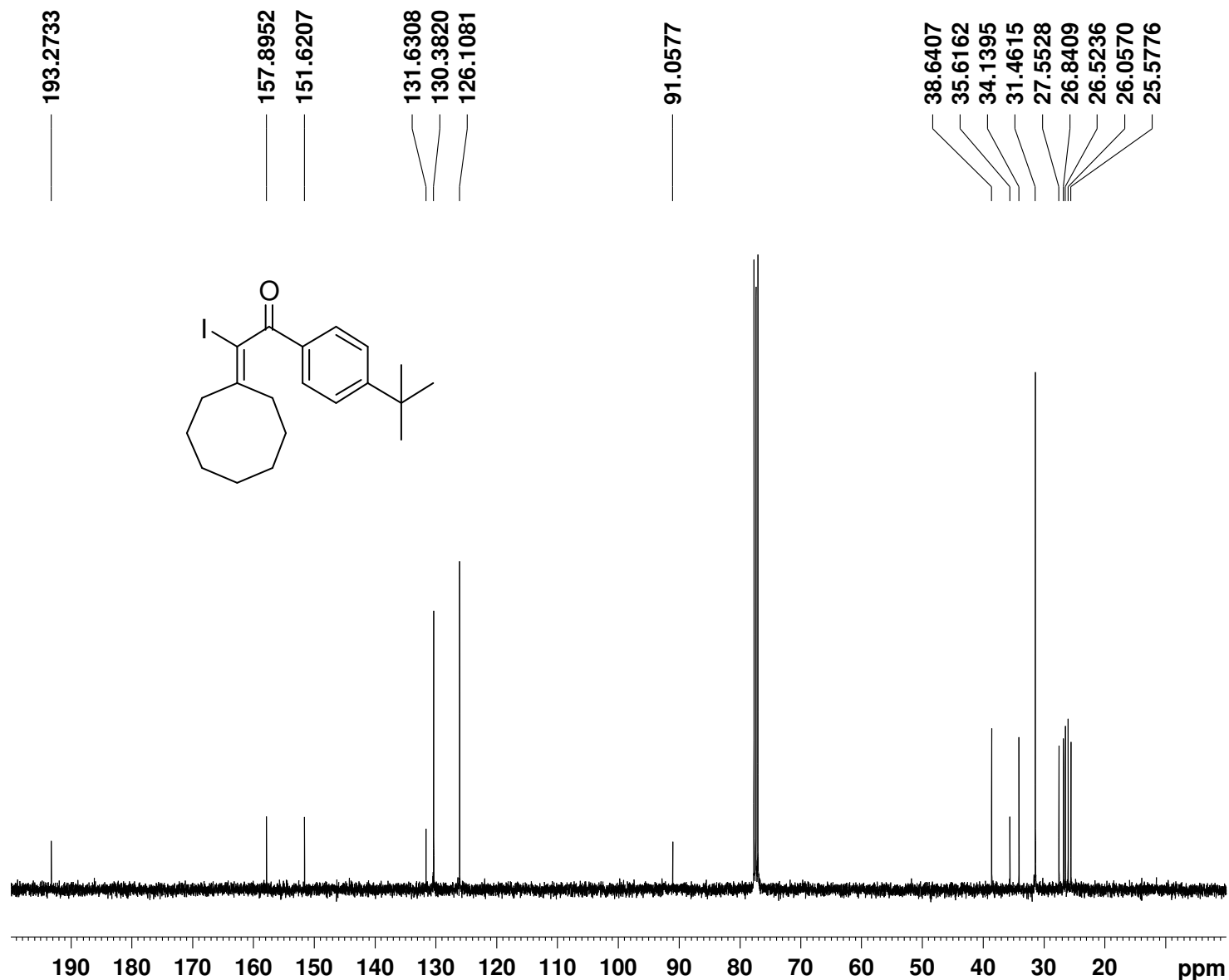
Current Data Parameters
NAME ARM-1334
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111020
Time 10.08
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 292.5 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.40 usec
PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 1-(4-*tert*-butylphenyl)-2-cyclooctylidene-2-iodoethanone 4n



Current Data Parameters
NAME ARM-1334
EXPNO 6
PROCNO 1

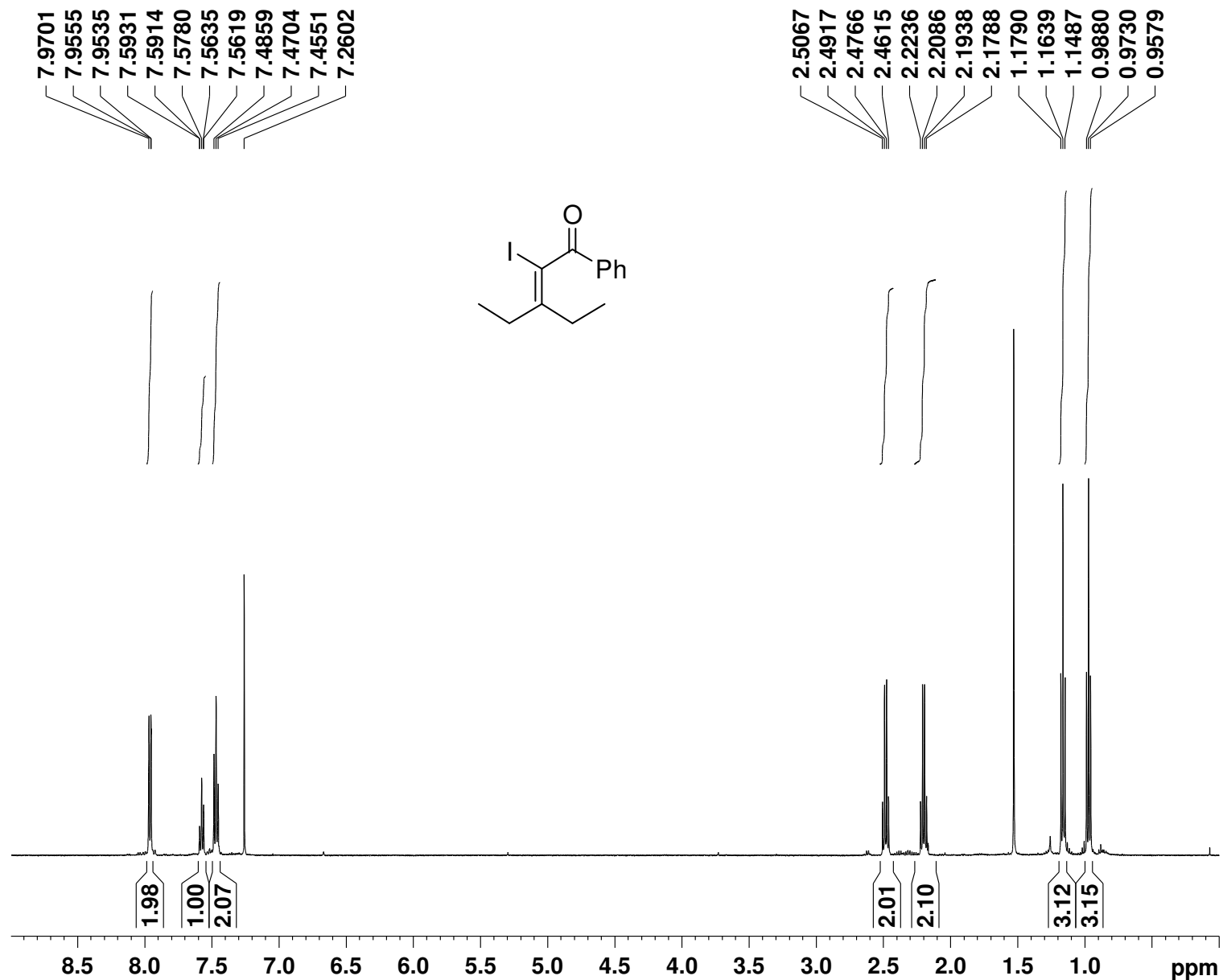
F2 - Acquisition Parameters
Date_ 20111020
Time 10.11
INSTRUM dpx400
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 9195.2
DW 20.850 usec
DE 6.00 usec
TE 292.9 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.89999998 sec
TDO 8

===== CHANNEL f1 =====
NUC1 13C
P1 9.70 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 -3.00 dB
PL12 16.08 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127331 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for 3-ethyl-2-iodo-1-phenylpent-2-en-1-one 4o



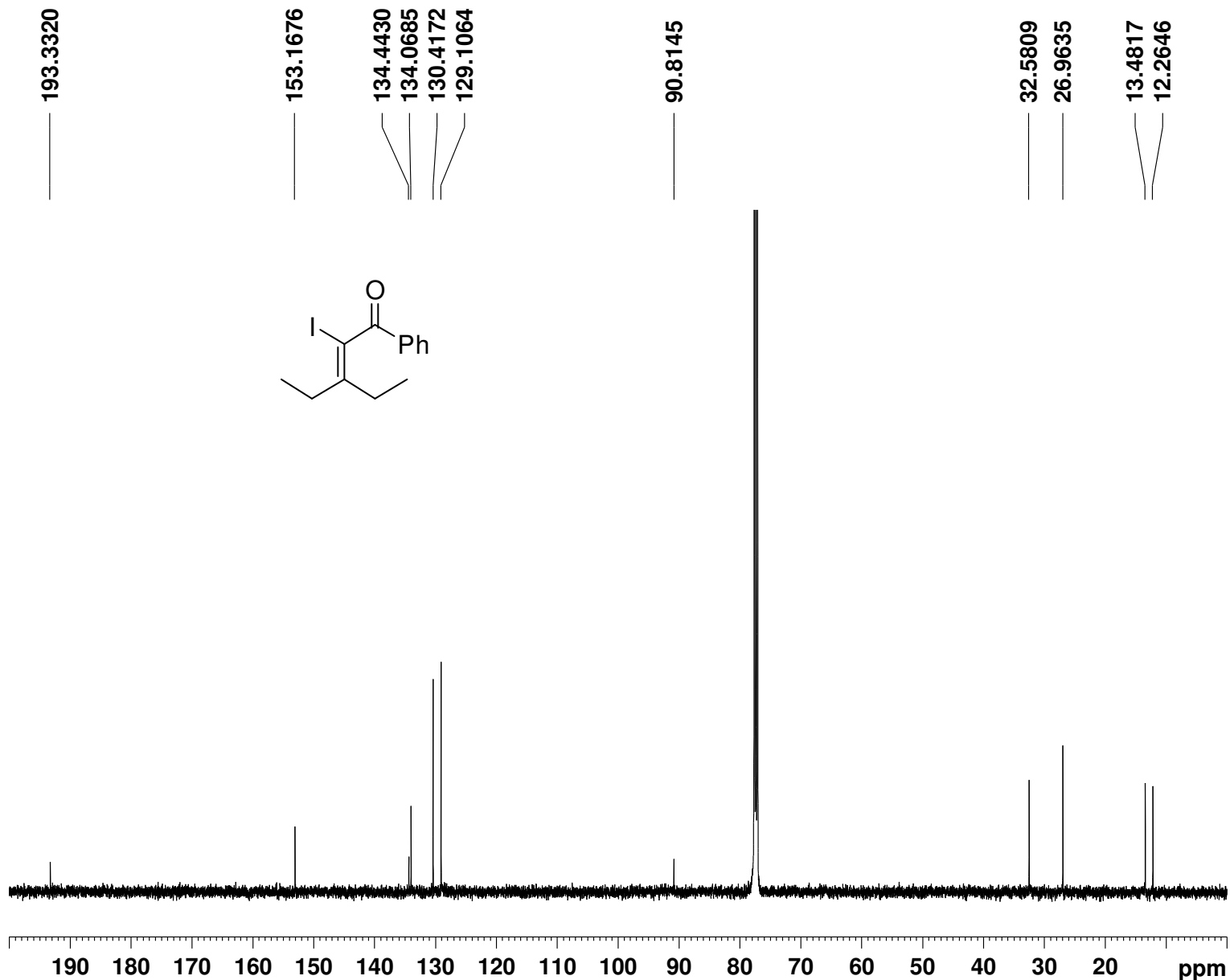
Current Data Parameters
NAME ARM-1330_500
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111018
Time 16.39
INSTRUM av500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 1150
DW 48.400 usec
DE 6.00 usec
TE 303.9 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.70 usec
PL1 0.10 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum for 3-ethyl-2-iodo-1-phenylpent-2-en-1-one 4o



Current Data Parameters
NAME ARM-1330_500
EXPNO 2
PROCNO 1

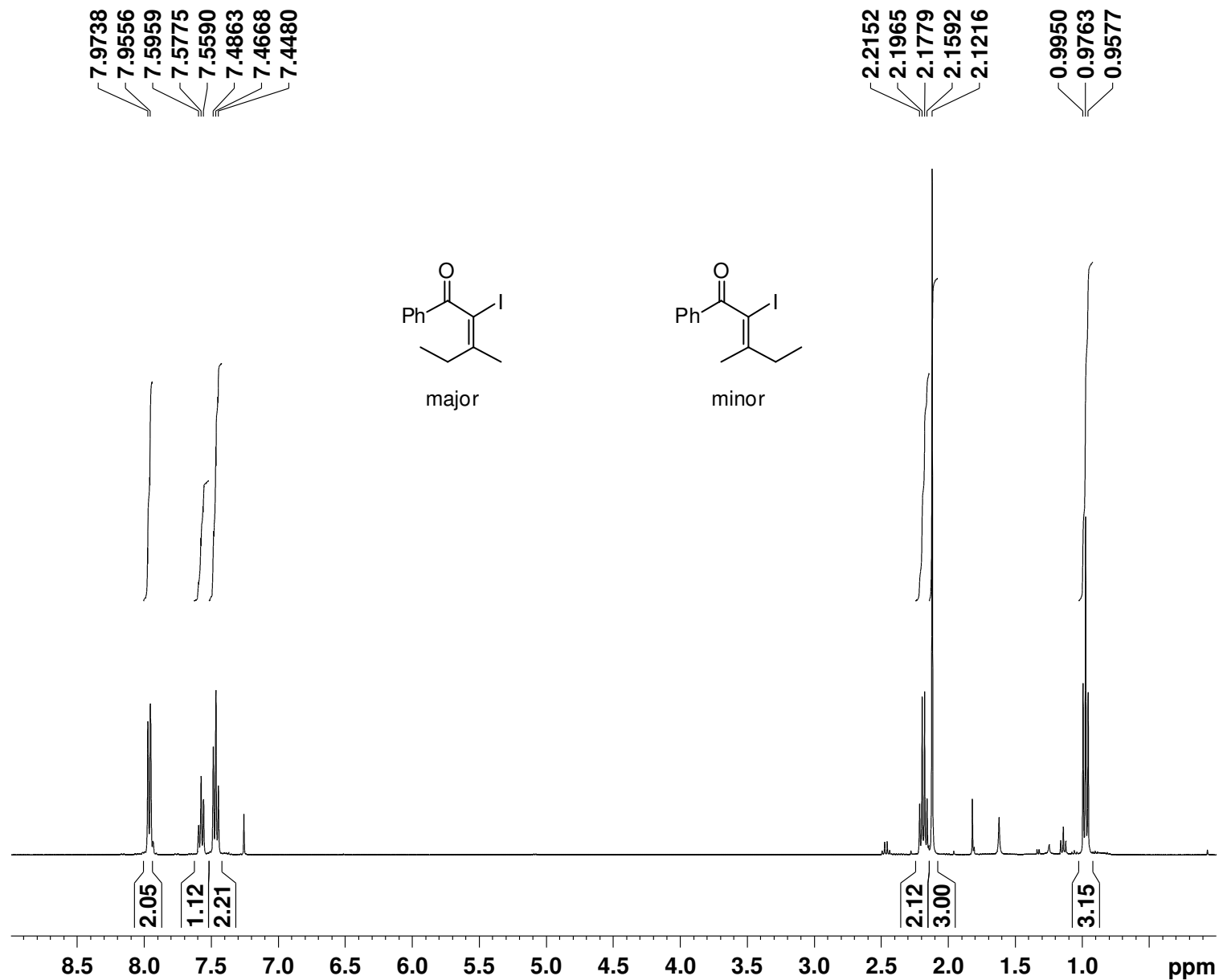
F2 - Acquisition Parameters
Date_ 20111018
Time 16.48
INSTRUM av500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8192
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 16400
DW 16.650 usec
DE 6.00 usec
TE 308.3 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.89999998 sec
TDO 256

==== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703637 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577364 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum for (*E*)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



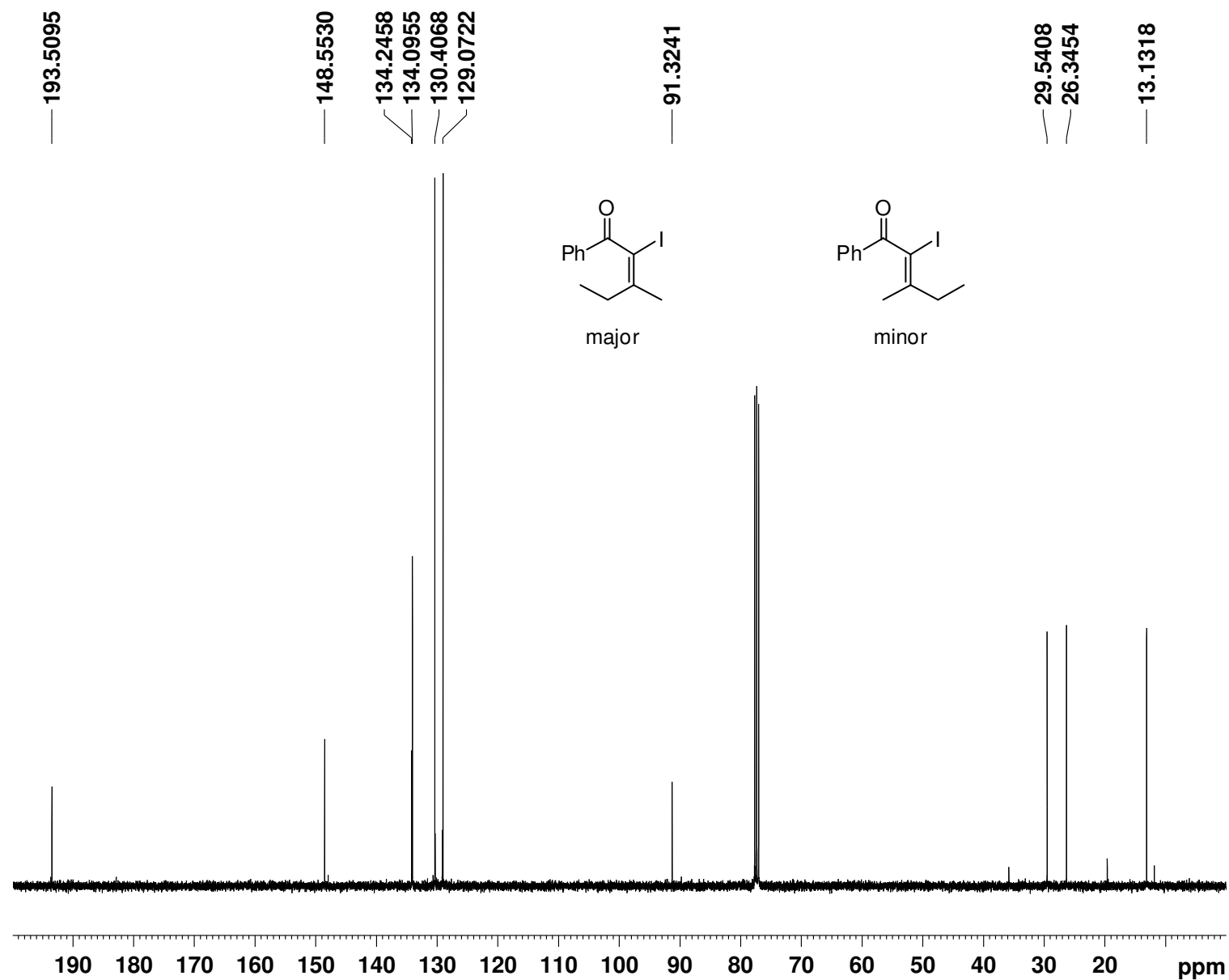
```
Current Data Parameters
NAME          WM-510
EXPNO         60
PROCNO        1

F2 - Acquisition Parameters
Date_         20120919
Time          17.39
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG            65.91
DW            60.800 usec
DE            10.69 usec
TE            294.2 K
D1            1.0000000 sec
TDO           1

===== CHANNEL f1 =====
SFO1          400.1324710 MHz
NUC1           1H
P1             8.00 usec
PLW1          24.00000000 W

F2 - Processing parameters
SI            131072
SF            400.1300109 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.50
```

¹³C NMR spectrum for (*E*)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



```
Current Data Parameters
NAME      WM-510
EXPNO     61
PROCNO    1

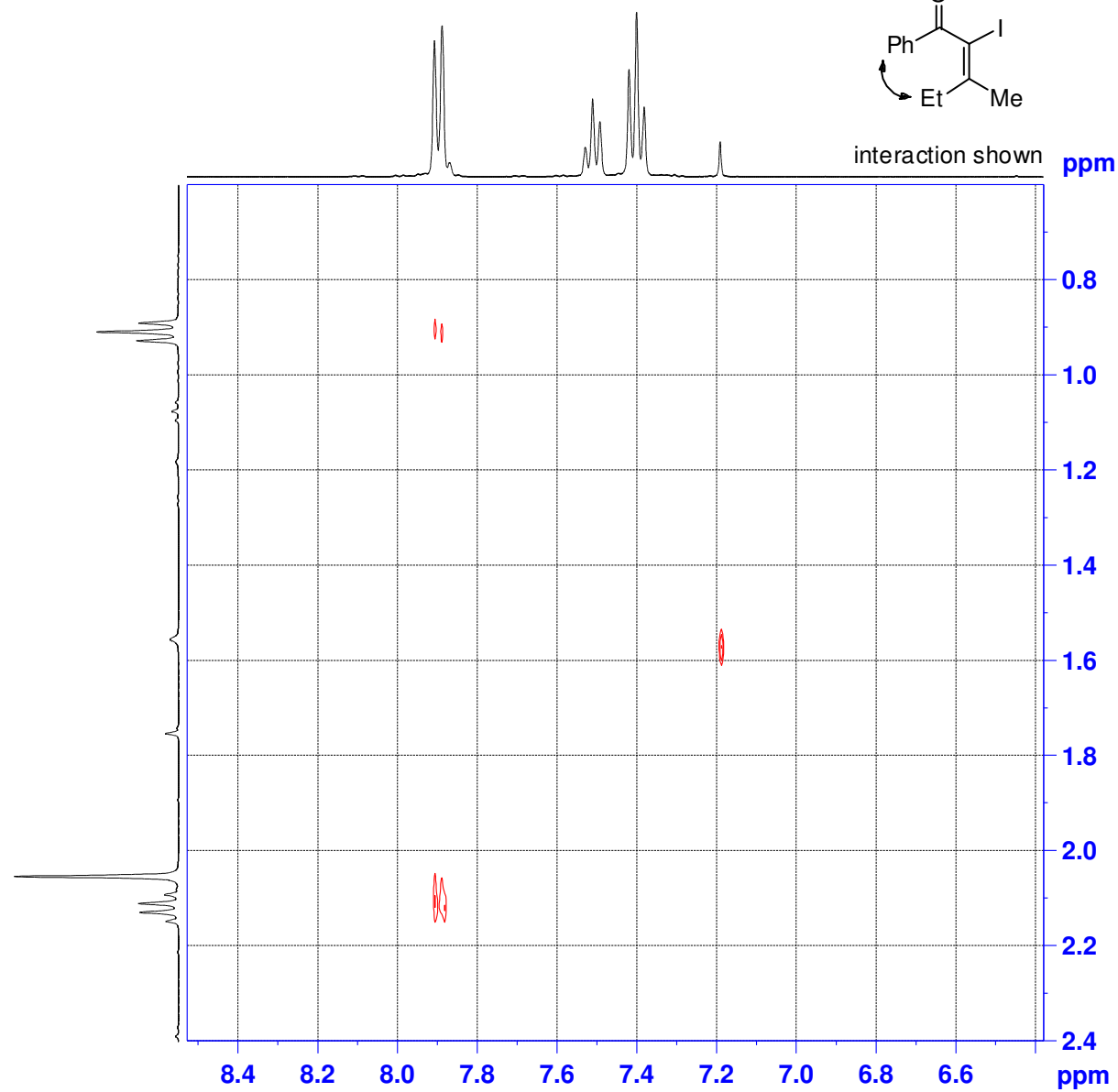
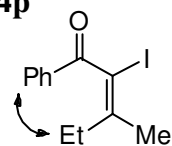
F2 - Acquisition Parameters
Date_     20120919
Time      17.54
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   zgpg30
TD        65536
SOLVENT   CDC13
NS        256
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631488 sec
RG        181.72
DW        20.800 usec
DE        8.18 usec
TE        294.5 K
D1        2.0000000 sec
D11       0.0300000 sec
TDO       1

===== CHANNEL f1 =====
SFO1      100.6228293 MHz
NUC1      13C
P1        9.00 usec
PLW1      77.0000000 W

===== CHANNEL f2 =====
SFO2      400.1316005 MHz
NUC2      1H
CPDPRG[2] waltz16
PCPD2     90.00 usec
PLW2      24.0000000 W
PLW12     0.17567000 W
PLW13     0.14229999 W

F2 - Processing parameters
SI        65536
SF        100.6127354 MHz
WDW       EM
SSB       0
LB        0.50 Hz
GB        0
PC        1.40
```

NOESY spectrum for (*E*)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



```
Current Data Parameters
NAME          WM-510
EXPNO         101
PROCNO        1

F2 - Acquisition Parameters
Date_         20120920
Time_         12.23
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       noesygpphpp
TD            2048
SOLVENT       CDCl3
NS            2
DS            32
SWH           3424.657 Hz
FIDRES        1.672196 Hz
AQ            0.2990080 sec
RG            25
DW            146.000 usec
DE            23.49 usec
TE            294.1 K
D0            0.00013581 sec
D1            1.93364406 sec
D8            0.50000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00029200 sec

===== CHANNEL f1 =====
SF01          400.1317910 MHz
NUC1          1H
P1            8.00 usec
P2            16.00 usec
P17           2500.00 usec
PLW1          24.00000000 W
PLW10         2.27220011 W

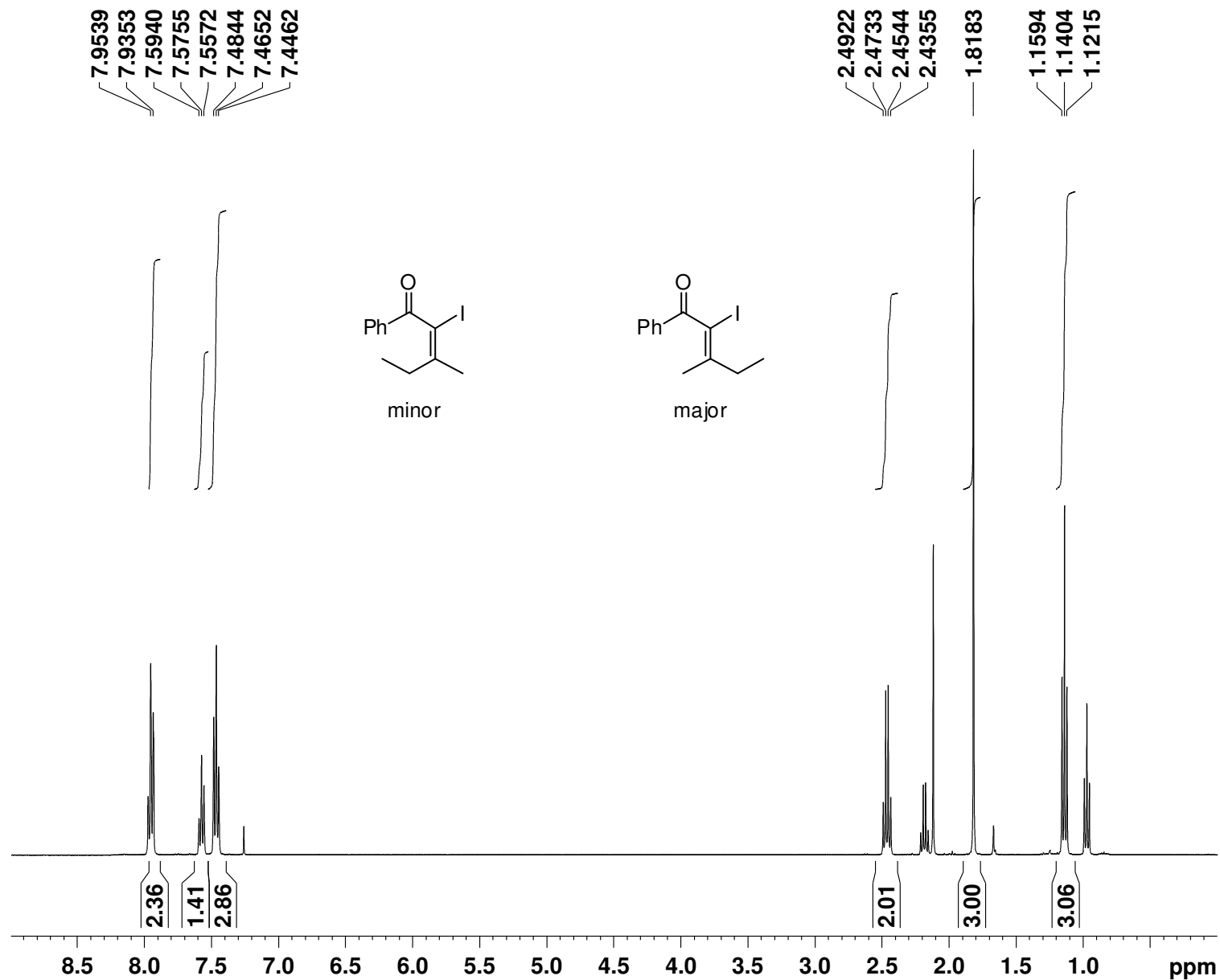
===== GRADIENT CHANNEL =====
GPNAM[1]     SMSQ10.100
GPZ1         40.00 %
P16          1000.00 usec

F1 - Acquisition parameters
TD           256
SF01         400.1318 MHz
FIDRES       13.377568 Hz
SW           8.559 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI           1024
SF           400.1300376 MHz
WDW          QSINE
SSB          2
LB           0 Hz
GB           0
PC           1.00

F1 - Processing parameters
SI           1024
MC2          States-TPPI
SF           400.1300376 MHz
WDW          QSINE
SSB          2
LB           0 Hz
GB           0
```

¹H NMR spectrum for (Z)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



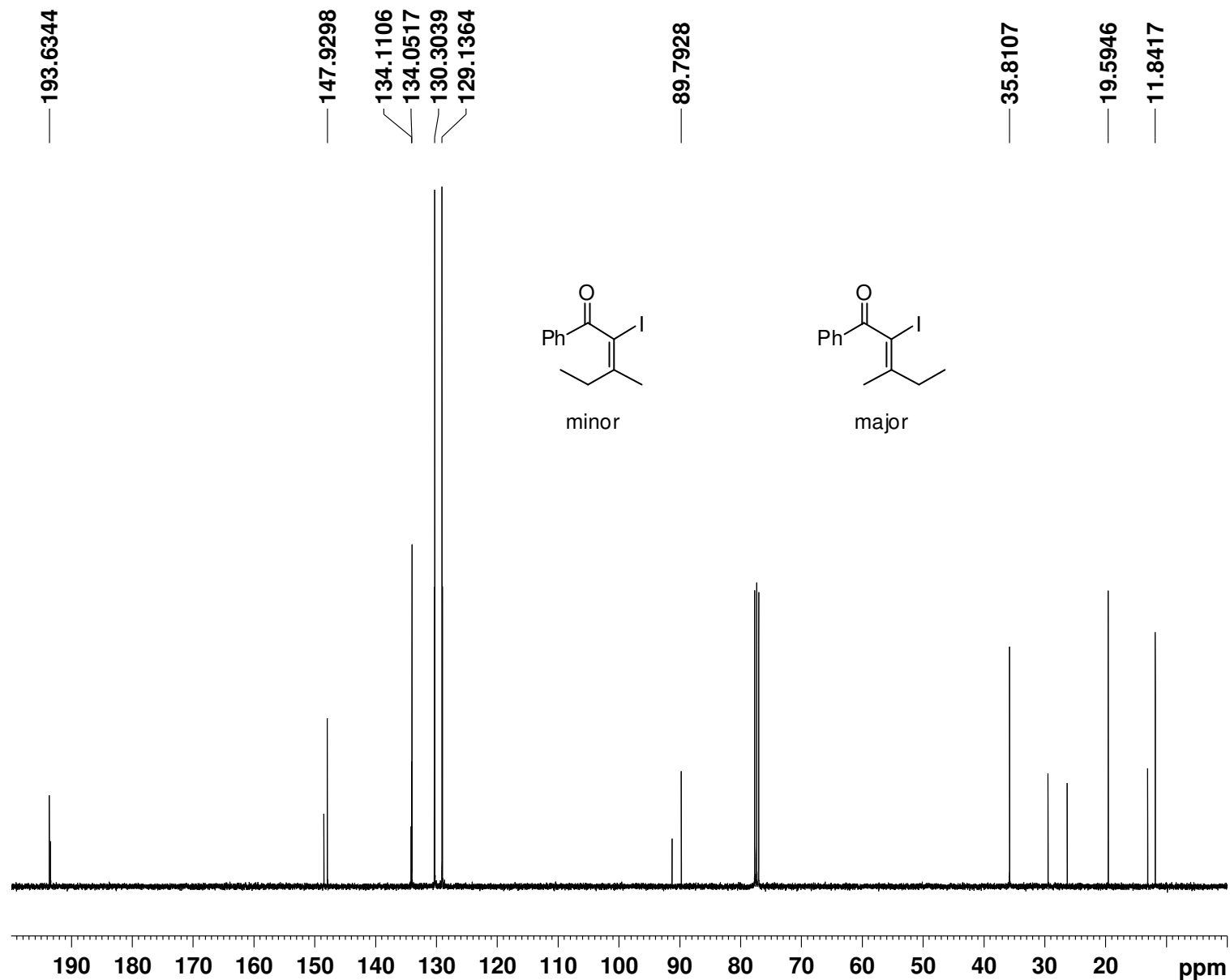
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Current Data Parameters
NAME          WM-510
EXPNO         40
PROCNO        1

F2 - Acquisition Parameters
Date_         20120919
Time          12.03
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9845889 sec
RG            46.39
DW            60.800 usec
DE            10.69 usec
TE            294.1 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SF01          400.1324710 MHz
NUC1           1H
P1             8.00 usec
PLW1          24.00000000 W

F2 - Processing parameters
SI            131072
SF            400.1300101 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.50
```

¹³C NMR spectrum for (Z)-2-iodo-3-methyl-1-phenylpent-2-en-1-one 4p



```
Current Data Parameters
NAME          WM-510
EXPNO         80
PROCNO        1

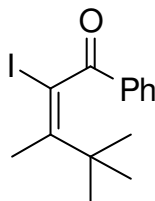
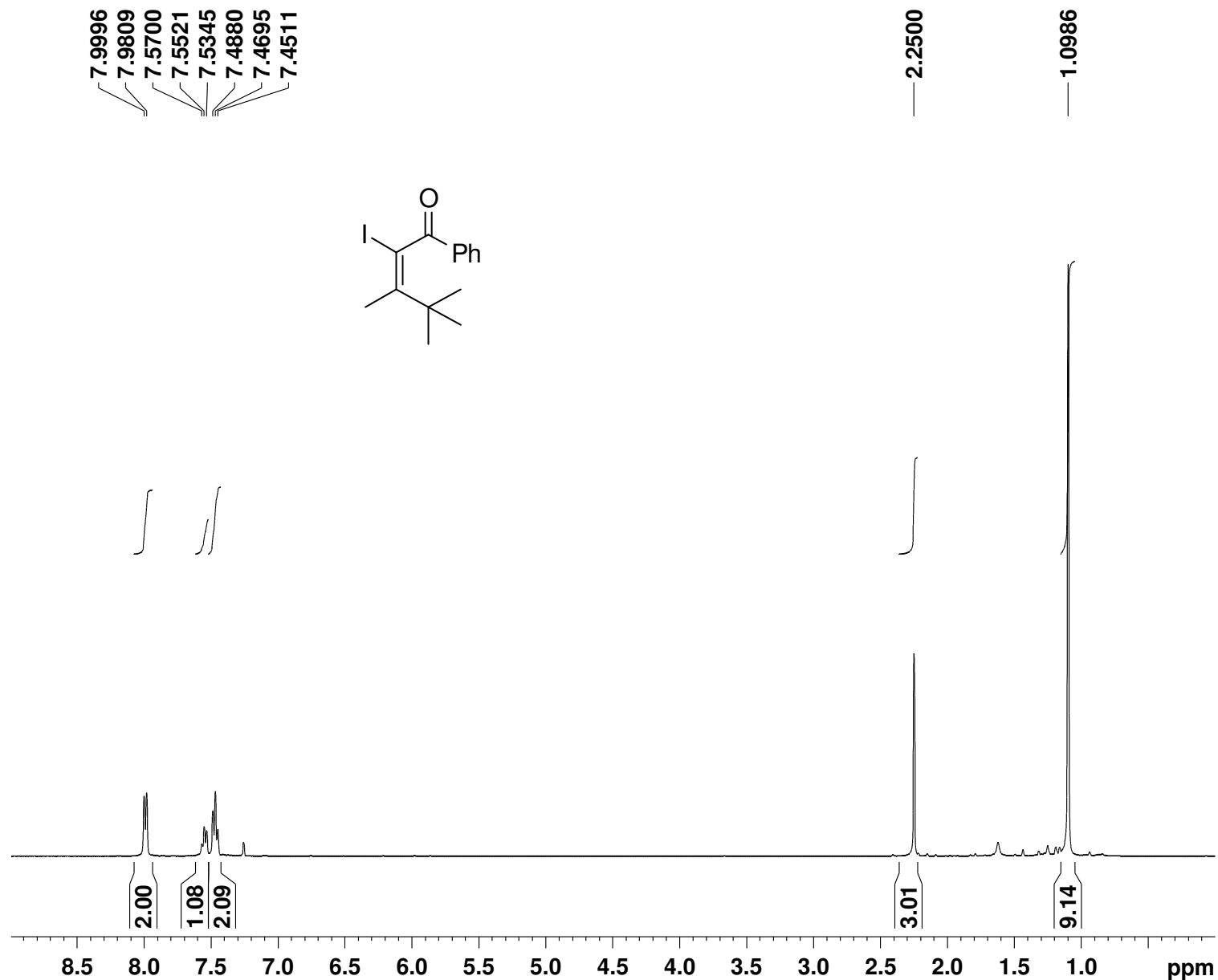
F2 - Acquisition Parameters
Date_         20120920
Time          11.16
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG            181.72
DW            20.800 usec
DE            8.18 usec
TE            294.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TDO           1

===== CHANNEL f1 =====
SF01          100.6228293 MHz
NUC1           13C
P1             9.00 usec
PLW1          77.0000000 W

===== CHANNEL f2 =====
SF02          400.1316005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2         90.00 usec
PLW2          24.0000000 W
PLW12         0.1756700 W
PLW13         0.14229999 W

F2 - Processing parameters
SI            65536
SF            100.6127392 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.40
```

¹H NMR spectrum for (*E*)-2-iodo-3,4,4-trimethyl-1-phenylpent-2-en-1-one 4q



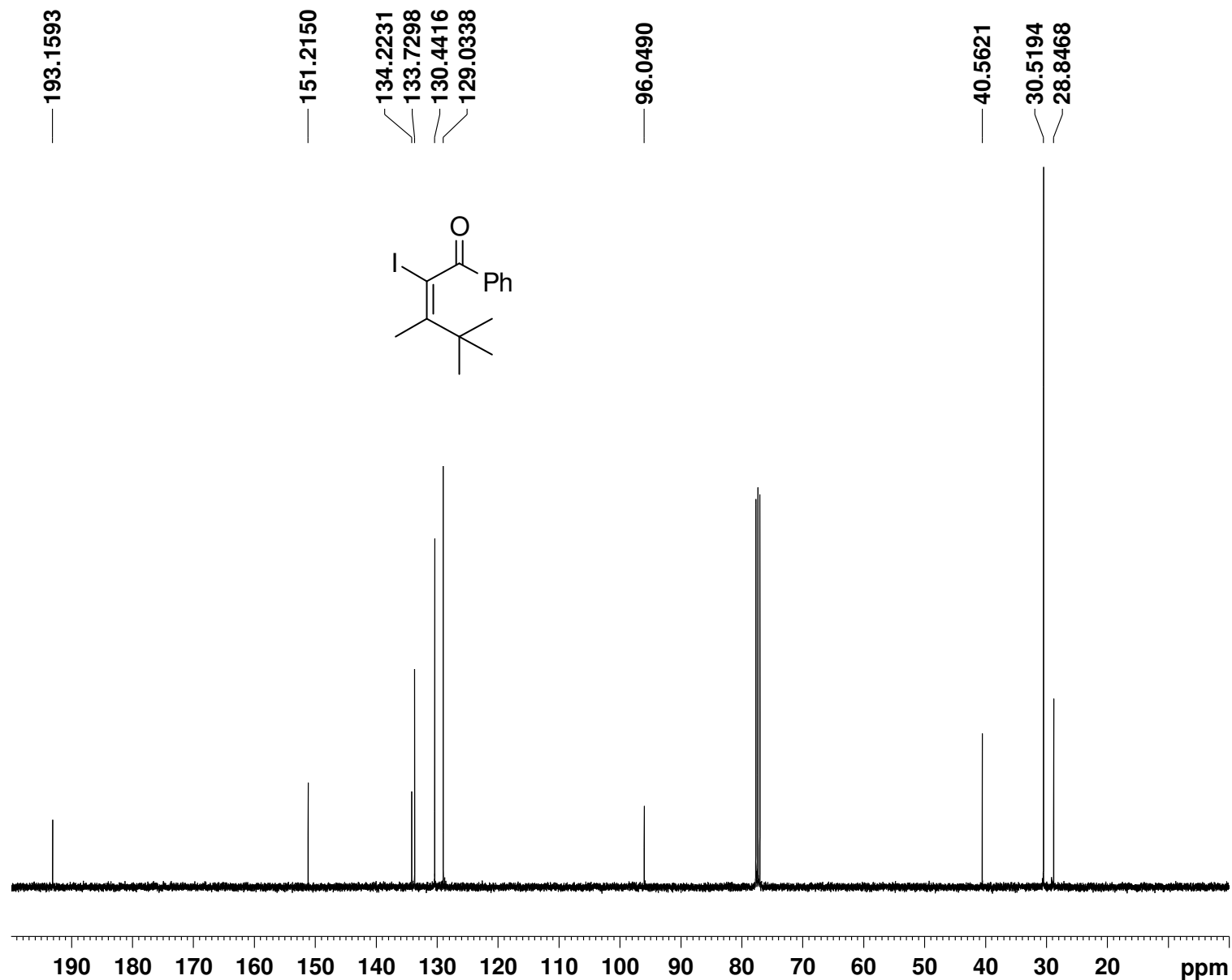
Current Data Parameters
NAME WM-507
EXPNO 90
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120906
Time 17.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 59.2
DW 60.800 usec
DE 10.69 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 400.1324710 MHz
NUC1 1H
P1 8.00 usec
PLW1 24.00000000 W

F2 - Processing parameters
SI 131072
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.50

¹³C NMR spectrum for (*E*)-2-iodo-3,4,4-trimethyl-1-phenylpent-2-en-1-one 4q



```
Current Data Parameters
NAME          WM-507
EXPNO         91
PROCNO        1

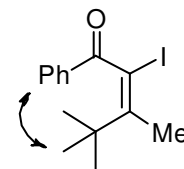
F2 - Acquisition Parameters
Date_         20120906
Time          17.42
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG            181.72
DW            20.800 usec
DE            8.18 usec
TE            294.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
SF01          100.6228293 MHz
NUC1           13C
P1             9.00 usec
PLW1          77.0000000 W

===== CHANNEL f2 =====
SF02          400.1316005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2         90.00 usec
PLW2          24.0000000 W
PLW12         0.1756700 W
PLW13         0.14229999 W

F2 - Processing parameters
SI            65536
SF            100.6127344 MHz
WDW           EM
SSB           0
LB            0.50 Hz
GB            0
PC            1.40
```

NOESY spectrum for (*E*)-2-iodo-3,4,4-trimethyl-1-phenylpent-2-en-1-one 4q



interaction shown

```
Current Data Parameters
NAME      WM507_500
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Date_     20120910
Time      23.11
INSTRUM   av500sgu
PROBHD    5 mm PABBO BB-
PULPROG   noesyph
TD         2048
SOLVENT   CDC13
NS         16
DS         4
SWH        4340.278 Hz
FIDRES     2.119276 Hz
AQ         0.2360448 sec
RG         64
DW         115.200 usec
DE         6.00 usec
TE         296.8 K
D0         0.00010285 sec
D1         1.96436501 sec
D8         0.69999999 sec
IN0        0.00023040 sec
```

```
===== CHANNEL f1 =====
NUC1      1H
P1         9.70 usec
PL1        0.10 dB
PL1W      22.82879257 W
SF01      500.1322275 MHz
```

```
F1 - Acquisition parameters
TD         256
SF01      500.1322 MHz
FIDRES     16.954210 Hz
SW         8.678 ppm
FnMODE     States-TPPI
```

```
F2 - Processing parameters
SI         1024
SF         500.1300480 MHz
WDW        QSINE
SSB         2
LB         0 Hz
GB         0
PC         1.00
```

```
F1 - Processing parameters
SI         1024
MC2        States-TPPI
SF         500.1300480 MHz
WDW        QSINE
SSB         2
LB         0 Hz
GB         0
```

