Supporting Information for:

Selective C-4 Alkylation of Pyridine by Nickel/Lewis Acid Catalysis

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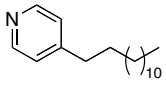
General. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique or in a dry box under an argon or nitrogen atmosphere. Flash column chromatography was performed using Kanto Chemical silica gel (spherical, 40–50 μm). Analytical thin layer chromatography (TLC) was performed on Merck Kieselgel 60 F_{254} (0.25 mm) plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline $KMnO_4$ solution followed by heating.

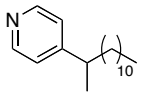
Apparatus. Proton and carbon nuclear magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on a Varian Mercury 400 spectrometer with Me_4Si or solvent resonance as the internal standard (^{1}H NMR, Me_4Si at 0 ppm or $CHCl_3$ at 7.26 ppm; ^{13}C NMR, Me_4Si at 0 ppm or $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), and integration. Melting points were determined using an OptiMelt MPA100. Elemental analyses were performed by Elemental Analysis Center of Kyoto University. High-resolution mass spectra were obtained with a JEOL JMS-700 (EI). Medium pressure chromatography was performed with a SHOKO Scientific Purif-espoir 2 chromatograph equipped with Purif-Pack (SHOKO Scientific, 20 mm x 60 mm, spherical, 30 μm). Preparative recycling silica gel chromatography was performed with a JAI LC-908 chromatograph equipped with COSMOSIL 5SL-II (Nacalai Tesque, 20 mm x 250 mm, spherical, 5 μm). GC analysis was performed on a Shimadzu GC 2014 equipped with an ENV-1 column (Kanto Chemical, 0.25 mm x 30 m, pressure = 31.7 kPa, detector = FID, 290 °C) with helium gas as a carrier.

Chemicals. Unless otherwise noted, commercially available chemicals were distilled and degassed before use. $Ni(cod)_2$, IPr, and IMes were purchased from Strem and used without further purification. Anhydrous toluene was purchased from Kanto Chemical and degassed by purging vigorously with argon for 20 min and further purified by passage through activated alumina under positive argon pressure as described by Grubbs et al.[1] Methylaluminum bis(2,6-di-*tert*-butyl-4-methylphenolate) (MAD) was prepared according to the literature procedure.[2]

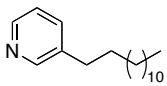
Nickel/Lewis Acid-catalyzed C-4 Alkylation of Pyridine. *A general procedure*. To a mixture of a pyridine derivative (1.0 mmol) and MAD (97 mg, 0.20 mmol) prepared in a 3 mL-vial was added a solution of $Ni(cod)_2$ (13.8 mg, 50 μmol) and IPr (19.4 mg, 50 μmol) in toluene (1.0 mL) in a dry box. After addition of an alkene (1.5 mmol) and undecane (internal standard, 39 mg, 0.25 mmol), the vial was sealed with a screw-cap, taken outside the dry box, and heated at 130 °C for the time specified in Table 1. The resulting mixture was filtered through a silica gel pad and then concentration *in vacuo*. The residue was purified by medium pressure chromatography to give the corresponding alkylpyridine products in yields listed in Table 1. Regioisomers were further separated by preparative silica gel chromatography to obtain independent spectra data.



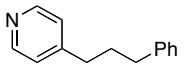
4-Tridecylpyridine (3aa). A pale yellow oil, R_f 0.61 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (dd, *J* = 4.5, 1.6 Hz, 2H), 7.08 (d, *J* = 5.9 Hz, 2H), 2.59 (t, *J* = 7.8 Hz, 2H), 1.61 (quint, *J* = 7.4 Hz, 2H), 1.37–1.20 (m, 20H), 0.88 (t, *J* = 6.8, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.7, 149.3, 123.8, 35.3, 32.0, 30.4, 29.8, 29.74, 29.71, 29.6, 29.5, 29.4, 29.3, 22.8, 14.3 (a signal for sp^3-carbon overlaps with others); Anal. Calcd for $C_{18}H_{31}N$: C, 82.69; H, 11.95. Found: C, 82.70; H, 12.09.



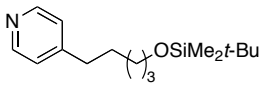
4-(Tridec-2-yl)pyridine (3'aa). A colorless oil, R_f 0.61 (hexane-ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 5.1 Hz, 2H), 7.10 (d, *J* = 6.0 Hz, 2H), 2.66 (sext, *J* = 7.1 Hz, 1H), 1.64–1.47 (m, 2H), 1.34–1.04 (m, 21H), 0.87 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.9, 149.6, 122.6, 39.4, 37.6, 31.9, 29.61, 29.59, 29.48, 29.3, 27.5, 22.7, 21.5, 14.1 (two signals for sp^3-carbons overlap with others); HRMS (EI) Calcd for $C_{18}H_{31}N$: M^+, 261.2457. Found: *m*/*z* 261.2451.



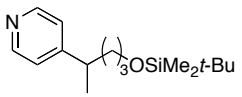
3-Tridecylpyridine. A pale yellow oil, R_f 0.68 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52–8.31 (m, 2H), 7.48 (d, *J* = 7.9 Hz, 1H), 7.20 (dd, *J* = 7.4, 4.9 Hz, 1H), 2.60 (t, *J* = 7.7 Hz, 2H), 1.62 (quint, *J* = 7.4 Hz, 2H), 1.39–1.17 (m, 20H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.7, 146.9, 137.9, 135.7, 123.1, 33.1, 32.0, 31.2, 29.8, 29.7, 29.6, 29.51, 29.45, 29.3, 22.8, 14.3 (two signals for sp^3-carbons overlap with others); HRMS (EI) Calcd for $C_{18}H_{31}N$: M^+, 261.2457. Found: *m*/*z* 261.2452.



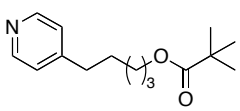
4-(3-Phenylpropyl)pyridine (3ab). A yellow oil, R_f 0.35 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 5.5 Hz, 2H), 7.34–7.01 (m, 7H), 2.76–2.50 (m, 4H), 2.04–1.90 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.0, 149.4, 141.4, 128.3, 128.2, 125.8, 123.8, 35.3, 34.7, 31.8; Anal. Calcd for $C_{14}H_{15}N$: C, 85.24; H, 7.66. Found: C, 85.44; H, 7.91.



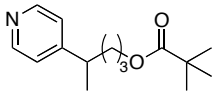
4-[5-(*tert*-Butyldimethylsilyloxy)pentyl]pyridine (3ac). A pale yellow oil, R_f 0.57 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 5.3 Hz, 2H), 7.13 (d, *J* = 5.1 Hz, 2H), 3.60 (t, *J* = 6.5 Hz, 2H), 2.62 (t, *J* = 7.7 Hz, 2H), 1.65 (quint, *J* = 7.7 Hz, 2H), 1.55 (quint, *J* = 7.0 Hz, 2H), 1.44–1.32 (m, 2H), 0.88 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.4, 149.3, 123.8, 63.0, 35.3, 32.6, 30.2, 26.1, 25.5, 18.5, –5.1; Anal. Calcd for $C_{16}H_{29}NOSi$: C, 68.76; H, 10.46. Found: C, 68.78; H, 10.42.



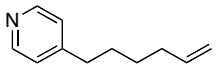
4-[5-(*tert*-Butyldimethylsilyloxy)pent-2-yl]pyridine (3'ac). A pale yellow oil, R_f 0.57 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 5.1 Hz, 2H), 7.12 (d, *J* = 6.0 Hz, 2H), 3.57 (t, *J* = 6.4 Hz, 2H), 2.70 (sext, *J* = 7.0 Hz, 1H), 1.64 (q, *J* = 7.7 Hz, 2H), 1.55–1.30 (m, 2H), 1.26 (d, *J* = 7.0 Hz, 3H), 0.89 (s, 9H), 0.03 (d, *J* = 1.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.6, 149.3, 122.5, 62.9, 39.3, 33.8, 30.8, 26.1, 21.7, 18.5, –5.1; HRMS (EI) Calcd for $C_{12}H_{20}NOSi$: [M–*t*Bu]$^+$, 222.1314. Found: *m*/*z* 222.1315.



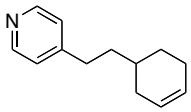
5-(Pyridin-4-yl)pentyl pivalate (3ad). A colorless oil, R_f 0.42 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 5.3 Hz, 2H), 7.13 (br, 2H), 4.05 (t, *J* = 6.6 Hz, 2H), 2.63 (t, *J* = 7.7 Hz, 2H), 1.73–1.57 (m, 4H), 1.45–1.35 (m, 2H), 1.18 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.6, 151.2, 149.6, 123.8, 64.0, 38.7, 35.0, 29.8, 28.3, 27.1, 25.4; Anal. Calcd for $C_{15}H_{23}NO_2$: C, 72.25; H, 9.30. Found: C, 72.01; H, 9.50.



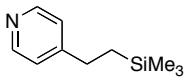
4-(Pyridin-4-yl)pentyl pivalate (3'ad). A pale yellow oil, R_f 0.42 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (br, 2H), 7.13 (br d, J = 4.2 Hz, 2H), 4.01 (t, J = 6.4 Hz, 2H), 2.77–2.66 (m, 1H), 1.72–1.44 (m, 4H), 1.26 (d, J = 7.0 Hz, 3H), 1.18 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 156.4, 149.5, 122.6, 63.9, 39.1, 38.7, 33.7, 27.2, 26.6, 21.5; HRMS (EI) Calcd for $C_{15}H_{23}NO_2$: M^+, 249.1729. Found: *m/z* 249.1730.



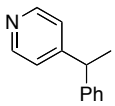
4-(Hex-5-enyl)pyridine (3ae). A pale yellow oil, R_f 0.40 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, J = 5.7 Hz, 2H), 7.10 (d, J = 5.9 Hz, 2H), 5.88–5.62 (m, 1H), 5.06–4.83 (m, 2H), 2.61 (t, J = 7.8 Hz, 2H), 2.08 (q, J = 7.1 Hz, 2H), 1.65 (quint, J = 7.8 Hz, 2H), 1.43 (quint, J = 7.6 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.5, 149.6, 138.4, 123.9, 114.7, 35.0, 33.4, 29.7, 28.3; Anal. Calcd for $C_{11}H_{15}N$: C, 81.94; H, 9.38. Found: C, 81.78; H, 9.66.



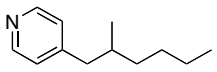
4-[2-(Cyclohex-3-enyl)ethyl]pyridine (3af). A pale yellow oil, R_f 0.45 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (dd, J = 4.5, 1.6 Hz, 2H), 7.11 (d, J = 4.4 Hz, 2H), 5.75–5.60 (m, 2H), 2.65 (t, J = 8.0 Hz, 2H), 2.26–1.94 (m, 3H), 1.88–1.46 (m, 5H), 1.38–1.15 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.8, 149.6, 127.1, 126.2, 123.8, 37.2, 33.0, 32.6, 31.7, 28.7, 25.0; Anal. Calcd for $C_{13}H_{17}N$: C, 83.37; H, 9.15. Found: C, 83.25; H, 9.29.



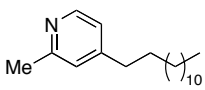
4-[2-(Trimethylsilyl)ethyl]pyridine (3ag). A pale yellow oil, R_f 0.47 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (dd, J = 4.5, 1.6 Hz, 2H), 7.13 (d, J = 4.5 Hz, 2H), 2.67–2.54 (m, 2H), 0.94–0.78 (m, 2H), 0.03 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.1, 149.6, 123.3, 29.5, 17.4, –1.8; Anal. Calcd for $C_{10}H_{17}NSi$: C, 66.97; H, 9.55. Found: C, 66.72; H, 9.69.



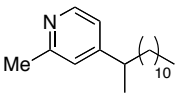
4-(1-Phenylethyl)pyridine (3'ah). A orange oil, R_f 0.45 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (dd, J = 4.6, 1.5 Hz, 2H), 7.33–7.15 (m, 5H), 7.12 (d, J = 4.6 Hz, 2H), 4.11 (q, J = 7.2 Hz, 1H), 1.63 (d, J = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.9, 149.5, 144.2, 128.5, 127.4, 126.5, 122.8, 44.3, 21.1; Anal. Calcd for $C_{13}H_{13}N$: C, 85.21; H, 7.15. Found: C, 85.44; H, 7.32.



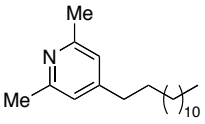
4-(2-Methylhexyl)pyridine (3ai). A pale yellow oil, R_f 0.49 (hexane–ethyl acetate = 1:2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (dd, J = 4.5, 1.6 Hz, 2H), 7.07 (d, J = 4.6 Hz, 2H), 2.62 (dd, J = 13.4, 6.0 Hz, 1H), 2.34 (dd, J = 13.4, 8.2 Hz, 1H), 1.81–1.67 (m, 1H), 1.41–1.09 (m, 6H), 0.88 (t, J = 7.1 Hz, 3H), 0.84 (d, J = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.7, 149.5, 124.6, 42.9, 36.3, 34.3, 29.2, 22.8, 19.3, 14.1; Anal. Calcd for $C_{12}H_{19}N$: C, 81.30; H, 10.80. Found: C, 81.12; H, 11.02.



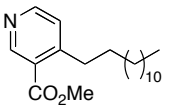
2-Methyl-4-tridecylpyridine (3ba). A colorless oil, R_f 0.63 (hexane–ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 4.9 Hz, 1H), 6.98 (s, 1H), 6.92 (d, J = 4.9 Hz, 1H), 2.55 (t, J = 7.7 Hz, 2H), 2.53 (s, 3H), 1.66–1.55 (m, 2H), 1.36–1.12 (m, 20H), 0.88 (t, J = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.8, 151.9, 148.6, 123.3, 120.9, 35.3, 32.0, 30.5, 29.8, 29.74, 29.73, 29.6, 29.51, 29.45, 29.3, 24.4, 22.8, 14.3 (a signal for sp^3-carbon overlaps with others); Anal. Calcd for $C_{19}H_{33}N$: C, 82.84; H, 12.07. Found: C, 82.82; H, 12.29.



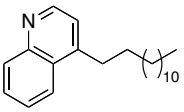
2-Methyl-4-(tridec-2-yl)pyridine (3'ba). A colorless oil, R_f 0.63 (hexane-ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, *J* = 5.1 Hz, 1H), 6.96 (s, 1H), 6.91 (d, *J* = 5.1 Hz, 1H), 2.62 (sext, *J* = 7.0 Hz, 1H), 2.54 (s, 3H), 1.62–1.47 (m, 2H), 1.35–1.05 (m, 21H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.8, 157.1, 148.6, 122.0, 119.6, 39.5, 37.7, 32.0, 29.7, 29.6, 29.4, 27.6, 24.5, 22.8, 21.6, 14.3 (three signals for sp^3-carbons overlap with others); HRMS (EI) Calcd for $C_{19}H_{33}N$: M^+, 275.2613. Found: *m/z* 275.2608.



2,6-Dimethyl-4-tridecylpyridine (3ca). A pale yellow oil, R_f 0.53 (hexane-ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.77 (s, 2H), 2.50 (t, *J* = 2.9 Hz, 2H), 2.48 (s, 6H), 1.66–1.48 (m, 2H), 1.46–1.19 (m, 20H), 0.87 (t, *J* = 7.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.3, 152.3, 120.5, 35.1, 31.9, 30.4, 29.7, 29.6, 29.5, 29.4, 29.34, 29.25, 24.3, 22.7, 14.1 (two signals for sp^3-carbons overlap with others); HRMS (EI) Calcd for $C_{20}H_{35}N$: M^+, 289.2770. Found: *m/z*. 289.2765.

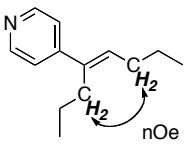


Methyl 4-tridecylnicotinate (3da). A colorless solid (mp = 48.6–50.1 °C), R_f 0.45 (hexane-ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.01 (s, 1H), 8.54 (d, *J* = 5.1 Hz, 1H), 7.15 (d, *J* = 5.1 Hz, 1H), 3.90 (s, 3H), 2.94 (t, *J* = 7.9 Hz, 2H), 1.57 (quint, *J* = 7.5 Hz, 2H), 1.43–1.14 (m, 20H), 0.85 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 153.8, 152.1, 151.6, 125.4, 125.3, 52.1, 33.7, 31.9, 30.7, 29.60, 29.58, 29.49, 29.35, 29.29, 22.6, 14.1 (three signals for sp^3-carbons overlap with others); Anal. Calcd for $C_{20}H_{33}NO_2$: C, 75.19; H, 10.41. Found: C, 75.31; H, 10.17.

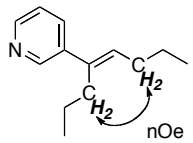


4-Tridecylquinoline (3ea). A colorless oil, R_f 0.68 (hexane-ethyl acetate = 1:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (d, *J* = 4.4 Hz, 1H), 8.11 (dd, *J* = 8.4, 0.7 Hz, 1H), 8.04 (dd, *J* = 8.4, 0.9 Hz, 1H), 7.70 (td, *J* = 7.7, 1.5 Hz, 1H), 7.56 (td, *J* = 7.7, 1.3 Hz, 1H), 7.31 (d, *J* = 4.4 Hz, 1H), 3.07 (t, *J* = 7.8 Hz, 2H), 1.76 (quint, *J* = 7.7 Hz, 2H), 1.52–1.16 (m, 20H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.1, 148.9, 148.2, 130.2, 129.0, 127.6, 126.2, 123.6, 120.7, 32.2, 31.9, 30.1, 29.69, 29.66, 29.63, 29.55, 29.4, 29.3, 22.7, 14.1 (two signals for sp^3-carbons overlap with others); Anal. Calcd for $C_{22}H_{33}N$: C, 84.83; H, 10.68. Found: C, 85.09; H, 10.61.

Nickel/Lewis Acid-catalyzed C-4 Alkenylation of Pyridine. To a mixture of pyridine (79 mg, 1.00 mmol) and a 1.8 M solution of $AlMe_3$ in hexane (111 μL, 0.20 mmol) prepared in a 3 mL-vial was added a solution of $Ni(cod)_2$ (13.8 mg, 50 μmol) and IMes (15.4 mg, 50 μmol) in toluene (1.0 mL) in a dry box. After addition of undecane (internal standard, 39 mg, 0.25 mmol), 4-octyne (0.33 g, 3.0 mmol) was added dropwise *via* a syringe pump at 110 °C for 3 h. The vial was sealed with a screw-cap, taken outside the dry box, and heated at 110 °C for additional 2 h. The resulting mixture was filtered through a silica gel pad, concentrated *in vacuo*, and purified by flush chromatography on silica gel (hexane–ethyl acetate = 5:1 to 3:1 as an eluant) to give **4** (100 mg, 53%) and **5** (29 mg, 15%).



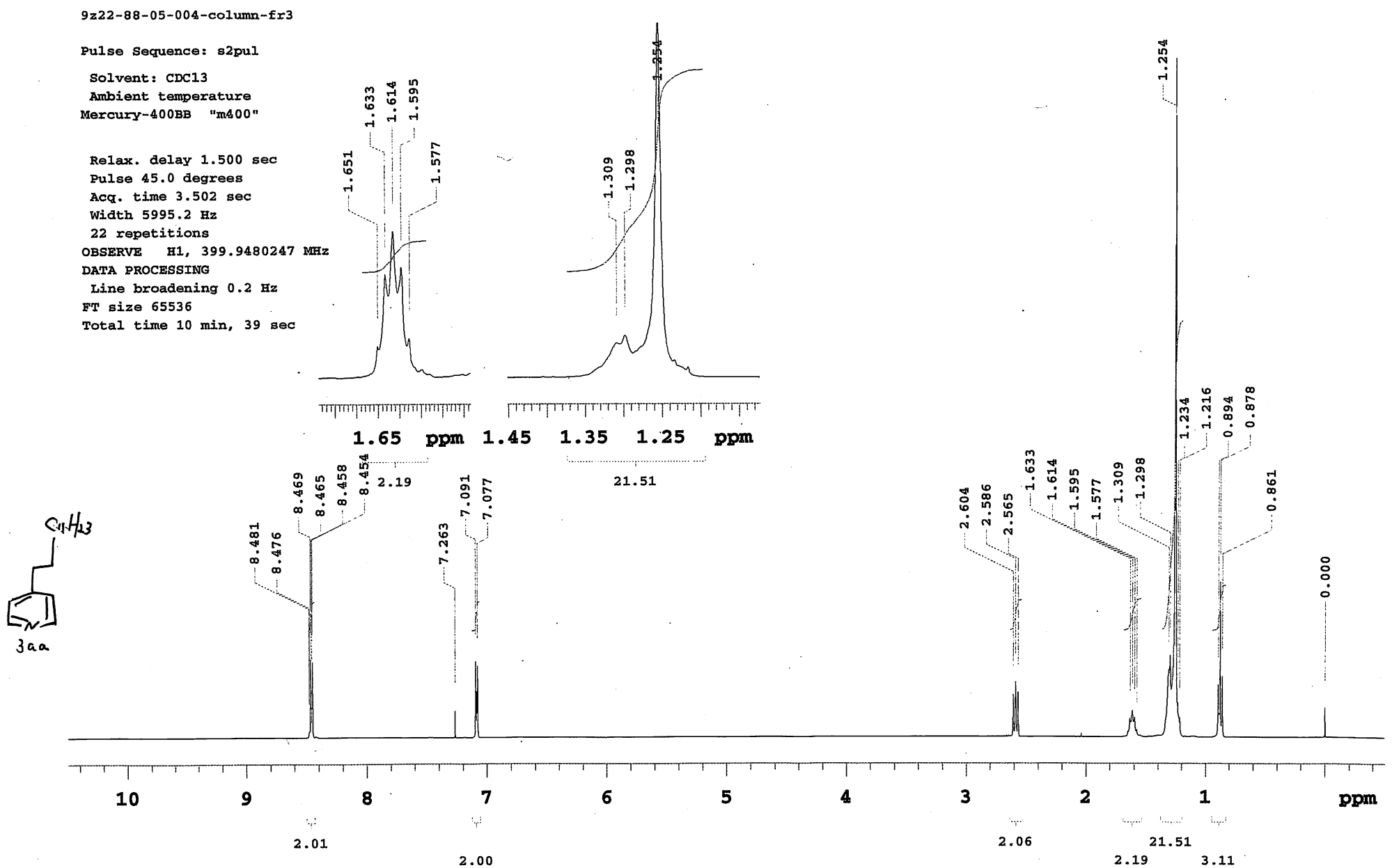
(*E*)-4-(Oct-4-en-4-yl)pyridine (4). A pale yellow oil, R_f 0.48 (hexane-ethyl acetate = 2:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 5.9 Hz, 2H), 7.23 (dd, *J* = 4.6, 1.6 Hz, 2H), 5.88 (t, *J* = 7.3 Hz, 1H), 2.46 (t, *J* = 7.7 Hz, 2H), 2.20 (q, *J* = 7.4 Hz, 2H), 1.48 (sext, *J* = 7.3 Hz, 2H), 1.36 (sext, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 149.7, 137.8, 132.4, 121.0, 30.74, 30.67, 22.8, 21.7, 13.92, 13.90; HRMS (EI) Calcd for $C_{13}H_{19}N$: M^+, 189.1517. Found: *m/z* 189.1522.



(*E*)-3-(Oct-4-en-4-yl)pyridine (5). A colorless oil, R_f 0.54 (hexane-ethyl acetate = 2:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (br s, 1H), 8.45 (br s, 1H), 7.61 (dt, *J* = 7.9, 1.8 Hz, 1H), 7.22 (dd, *J* = 7.8, 4.9 Hz, 1H), 5.69 (t, *J* = 7.2 Hz, 1H), 2.47 (t, *J* = 7.7 Hz, 2H), 2.19 (q, *J* = 7.3 Hz, 2H), 1.48 (sext, *J* = 7.4 Hz, 2H), 1.35 (sext, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.88 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 147.8, 147.5, 138.9, 137.0, 133.6, 131.2, 123.0, 31.4, 30.6, 22.9, 21.6, 13.9, 13.8; HRMS (EI) Calcd for $C_{13}H_{19}N$: M^+, 189.1517. Found: *m*/*z* 189.1517.

References

1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
2. Adachi, T.; Sugimoto, H.; Aida, T.; Inoue, S. *Macromolecules* **1993**, *26*, 1238.

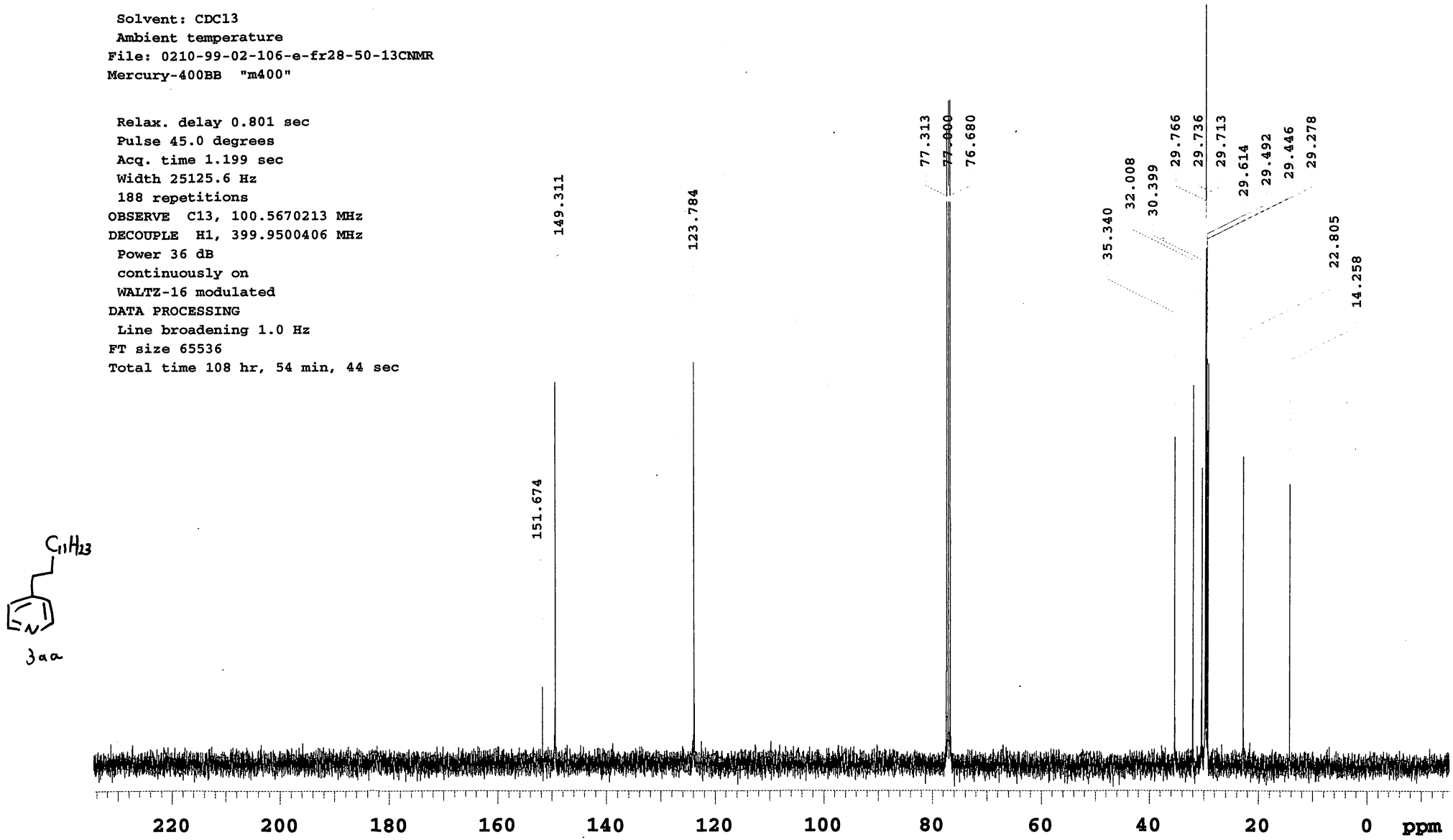

9z22-88-05-004-column-fr3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
22 repetitions
OBSERVE H1, 399.9480247 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 10 min, 39 sec
1.651
1.633
1.614
1.595
1.577
1.309
1.298
1.254
1.65 ppm 1.45 1.35 1.25 ppm
2.19
21.51
8.481
8.476
8.469
8.465
8.458
8.454
7.263
7.091
7.077
2.604
2.586
2.565
1.633
1.614
1.595
1.577
1.309
1.298
1.254
1.234
1.216
0.894
0.878
0.861
0.000
C11H23
3aa
10 9 8 7 6 5 4 3 2 1 ppm
2.01
2.00
2.06
21.51
2.19
3.11

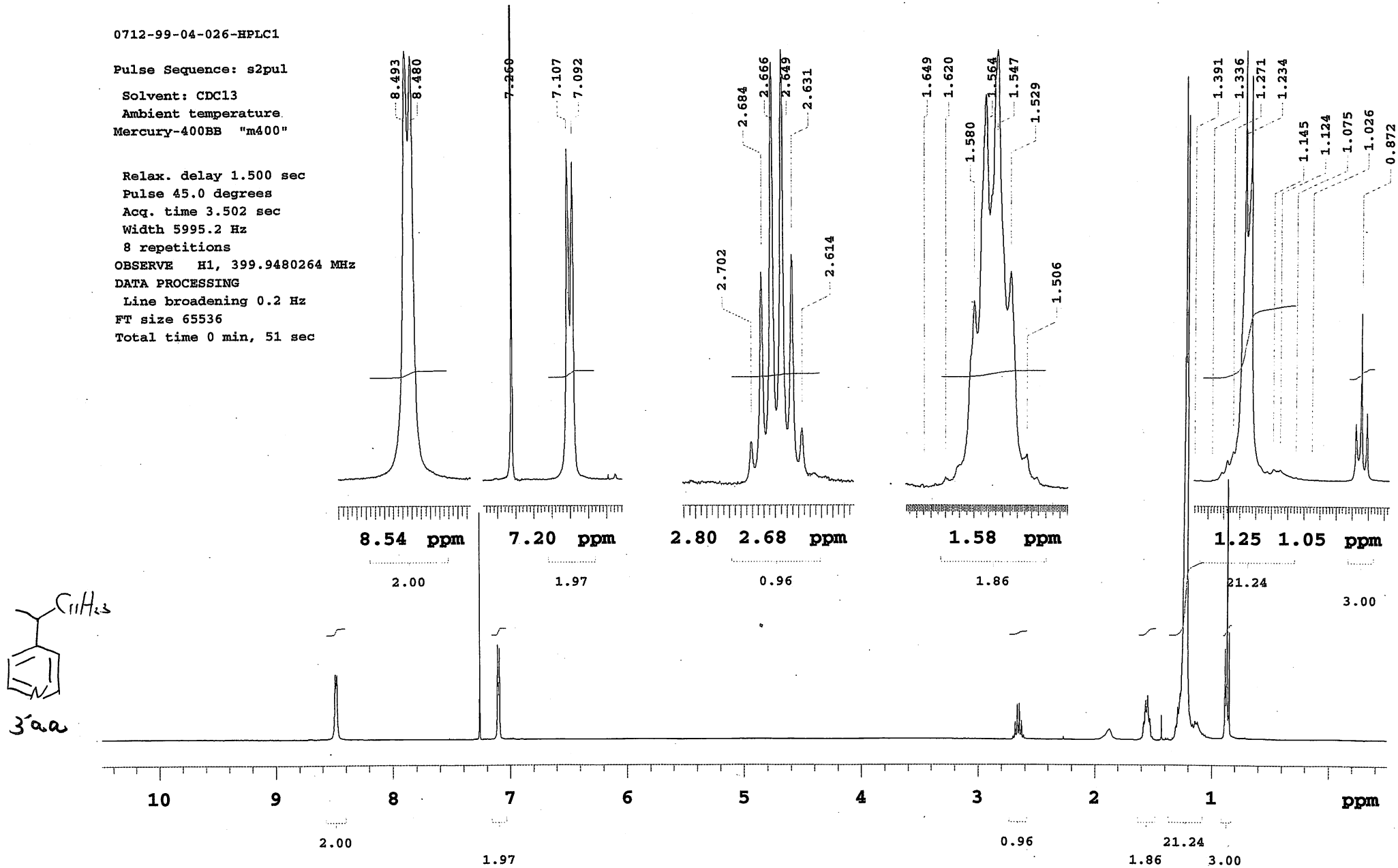
0210-99-02-106-e-fr28-50-13CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0210-99-02-106-e-fr28-50-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
188 repetitions
OBSERVE C13, 100.5670213 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec



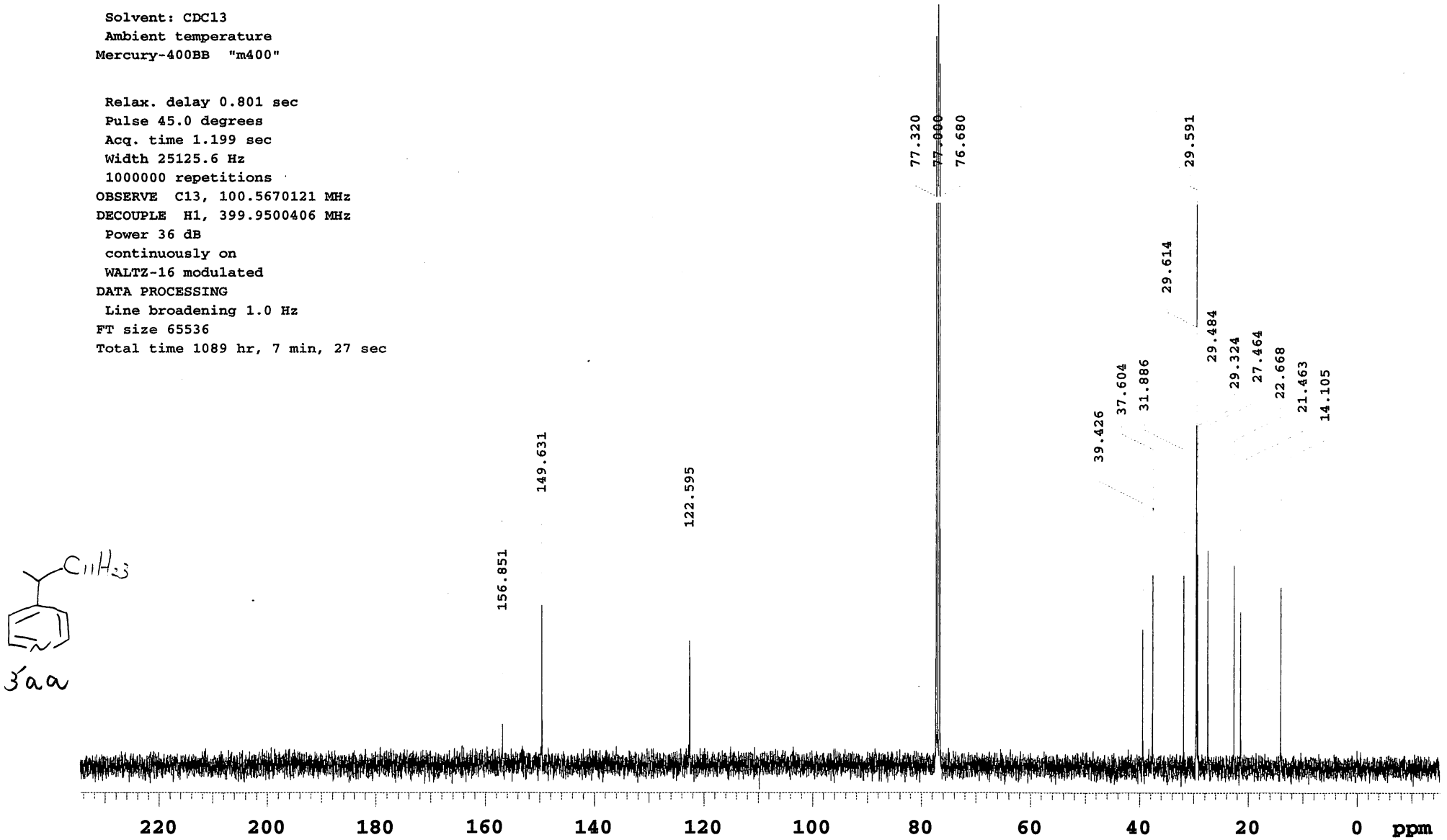

0712-99-04-026-HPLC1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480264 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.493
8.480
7.260
7.107
7.092
2.684
2.666
2.649
2.631
2.702
2.614
1.649
1.620
1.580
1.564
1.547
1.529
1.506
1.391
1.336
1.271
1.234
1.145
1.124
1.075
1.026
0.872
8.54 ppm
7.20 ppm
2.80 2.68 ppm
1.58 ppm
1.25 1.05 ppm
2.00
1.97
0.96
1.86
21.24
3.00
C11H23
3'aa
10
9
8
7
6
5
4
3
2
1
ppm

0712-99-04-026-HPLC1-13CNMR

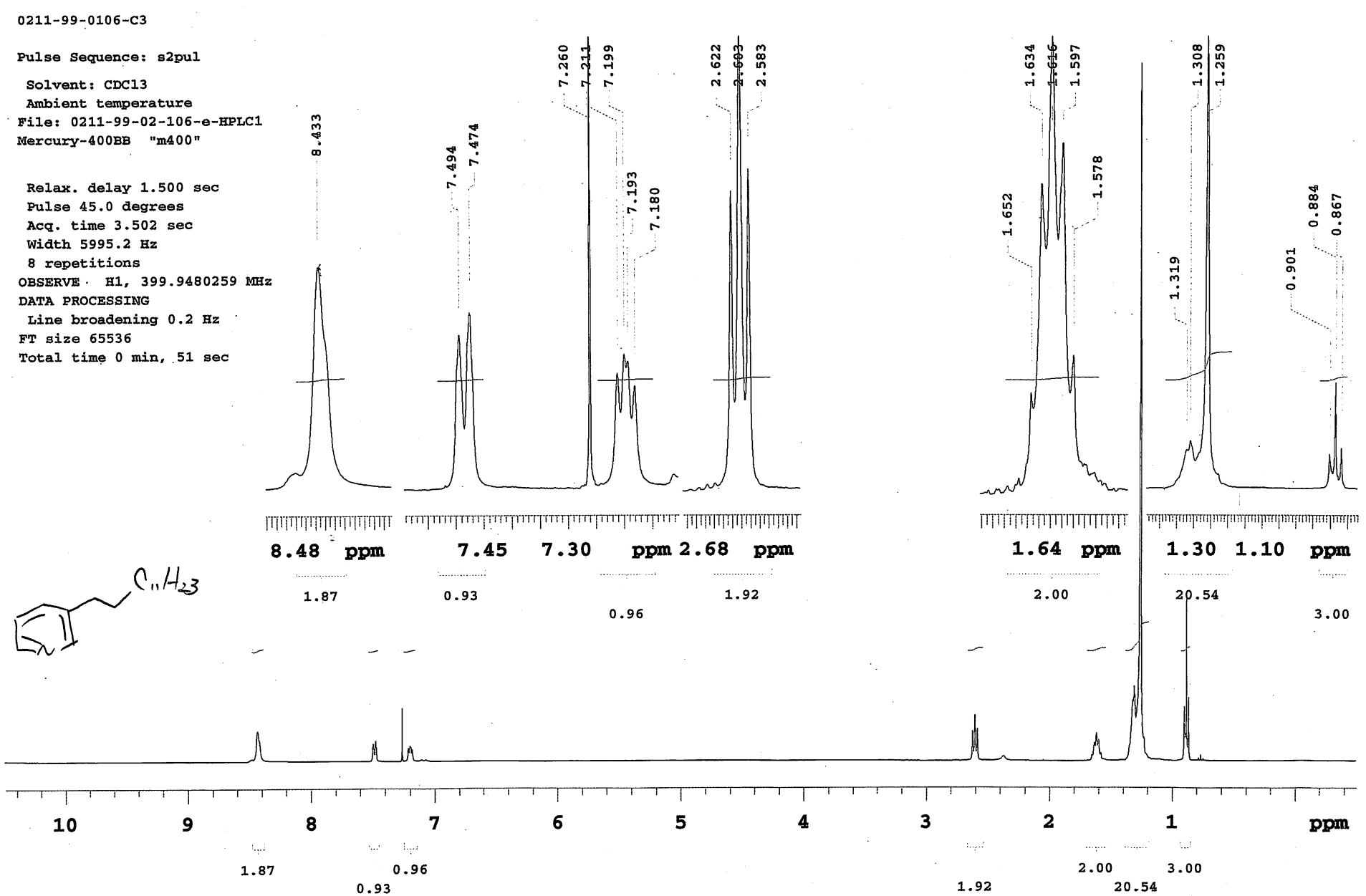
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1000000 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1089 hr, 7 min, 27 sec



3aa

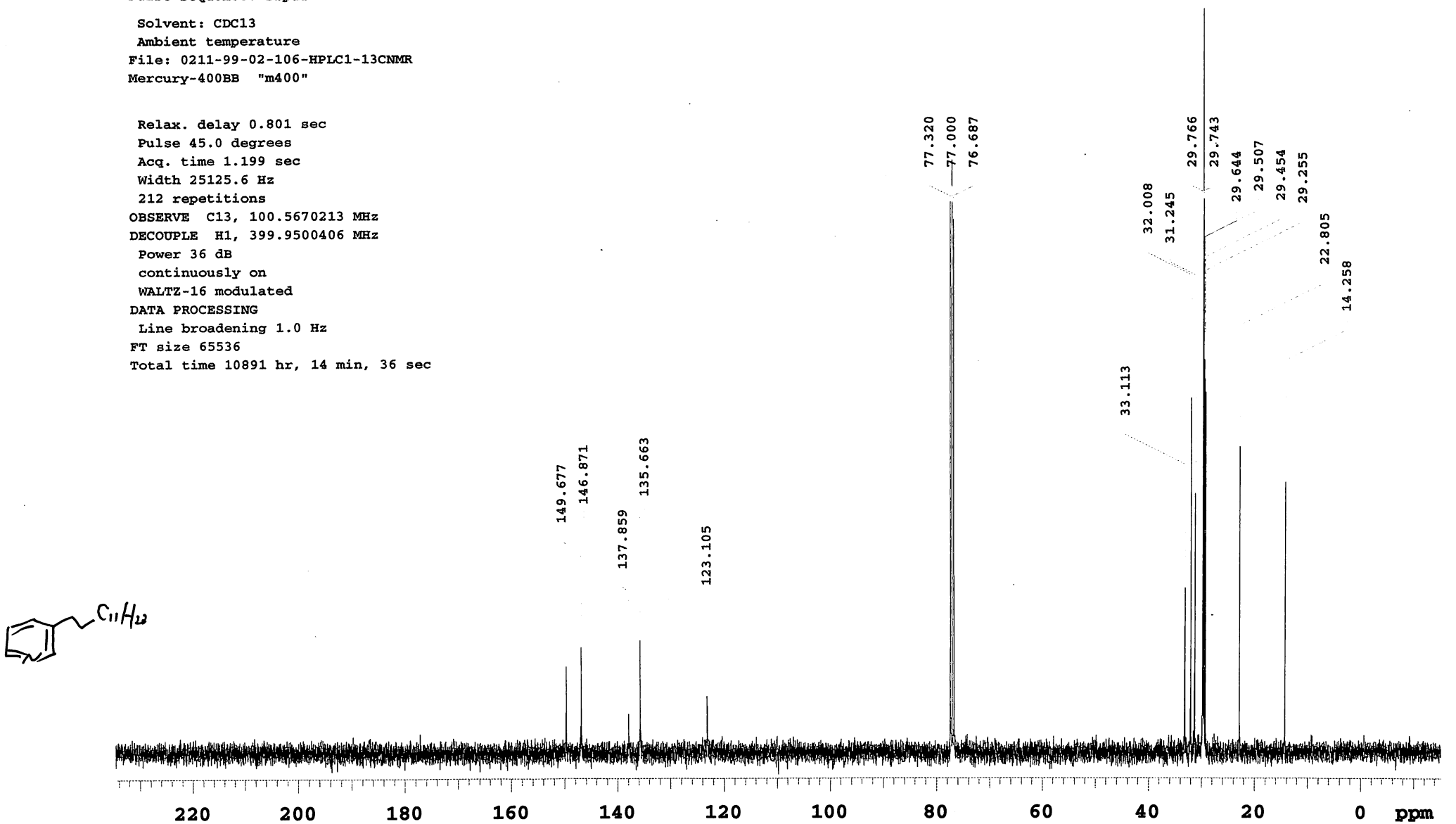

0211-99-0106-C3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0211-99-02-106-e-HPLC1
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480259 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.433
7.494
7.474
7.260
7.211
7.199
7.193
7.180
2.622
2.603
2.583
1.652
1.634
1.616
1.597
1.578
1.319
1.308
1.259
0.901
0.884
0.867
8.48 ppm
7.45 7.30 ppm
2.68 ppm
1.64 ppm
1.30 1.10 ppm
1.87
0.93
0.96
1.92
2.00
20.54
3.00
10 9 8 7 6 5 4 3 2 1 ppm

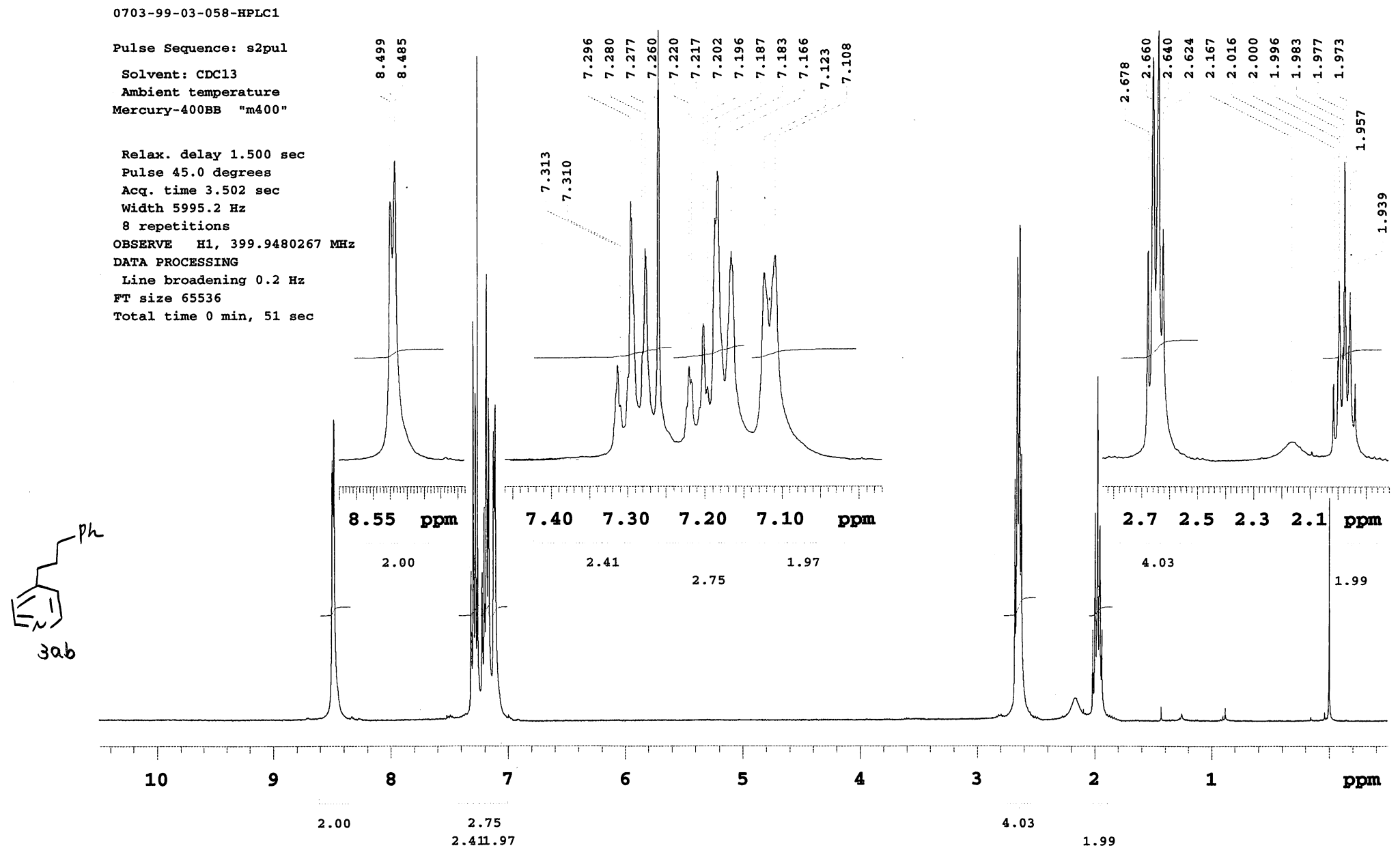
0211-99-106-C3-13CNMR

Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
File: 0211-99-02-106-HPLC1-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
212 repetitions
OBSERVE C13, 100.5670213 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec



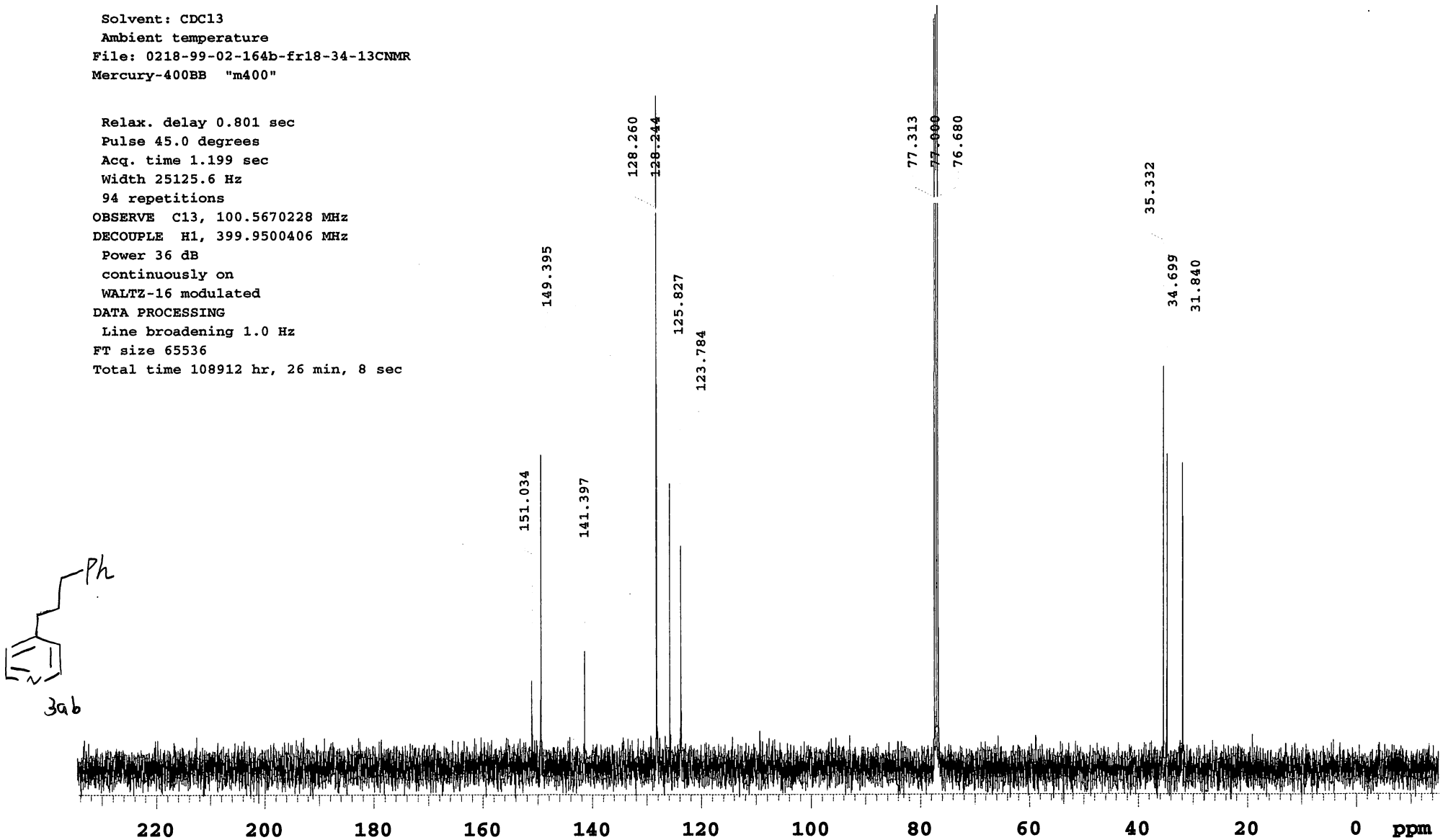

0703-99-03-058-HPLC1
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480267 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.499
8.485
7.313
7.310
7.296
7.280
7.277
7.260
7.220
7.217
7.202
7.196
7.187
7.183
7.166
7.123
7.108
2.678
2.660
2.640
2.624
2.167
2.016
2.000
1.996
1.983
1.977
1.973
1.957
1.939
8.55 ppm
7.40 7.30 7.20 7.10 ppm
2.7 2.5 2.3 2.1 ppm
2.00
2.41
2.75
1.97
4.03
1.99
10 9 8 7 6 5 4 3 2 1 ppm
2.00
2.75
2.41
1.97
4.03
1.99
Ph
3ab

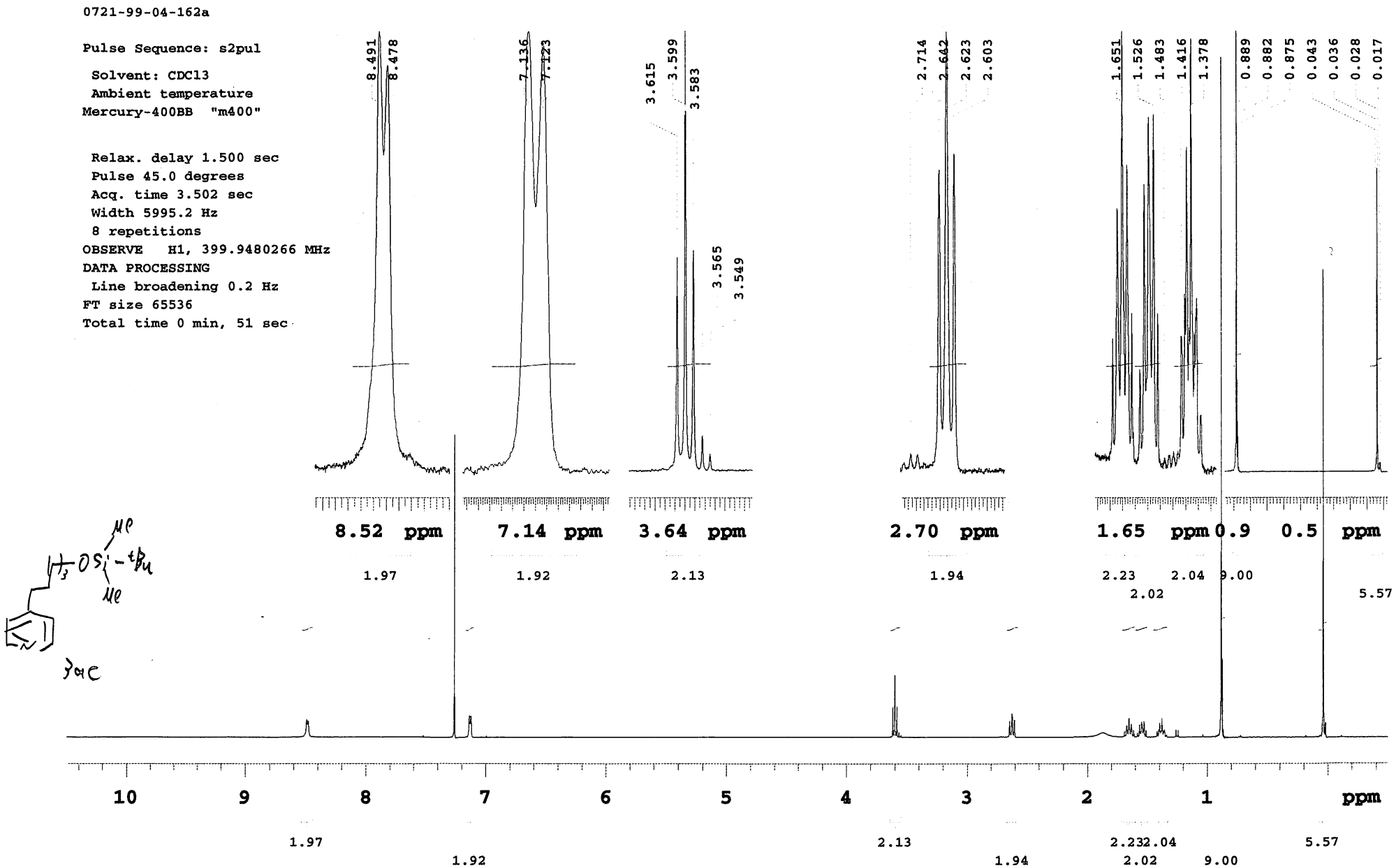
0218-99-02-164b-fr18-34-13CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0218-99-02-164b-fr18-34-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
94 repetitions
OBSERVE C13, 100.5670228 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108912 hr, 26 min, 8 sec



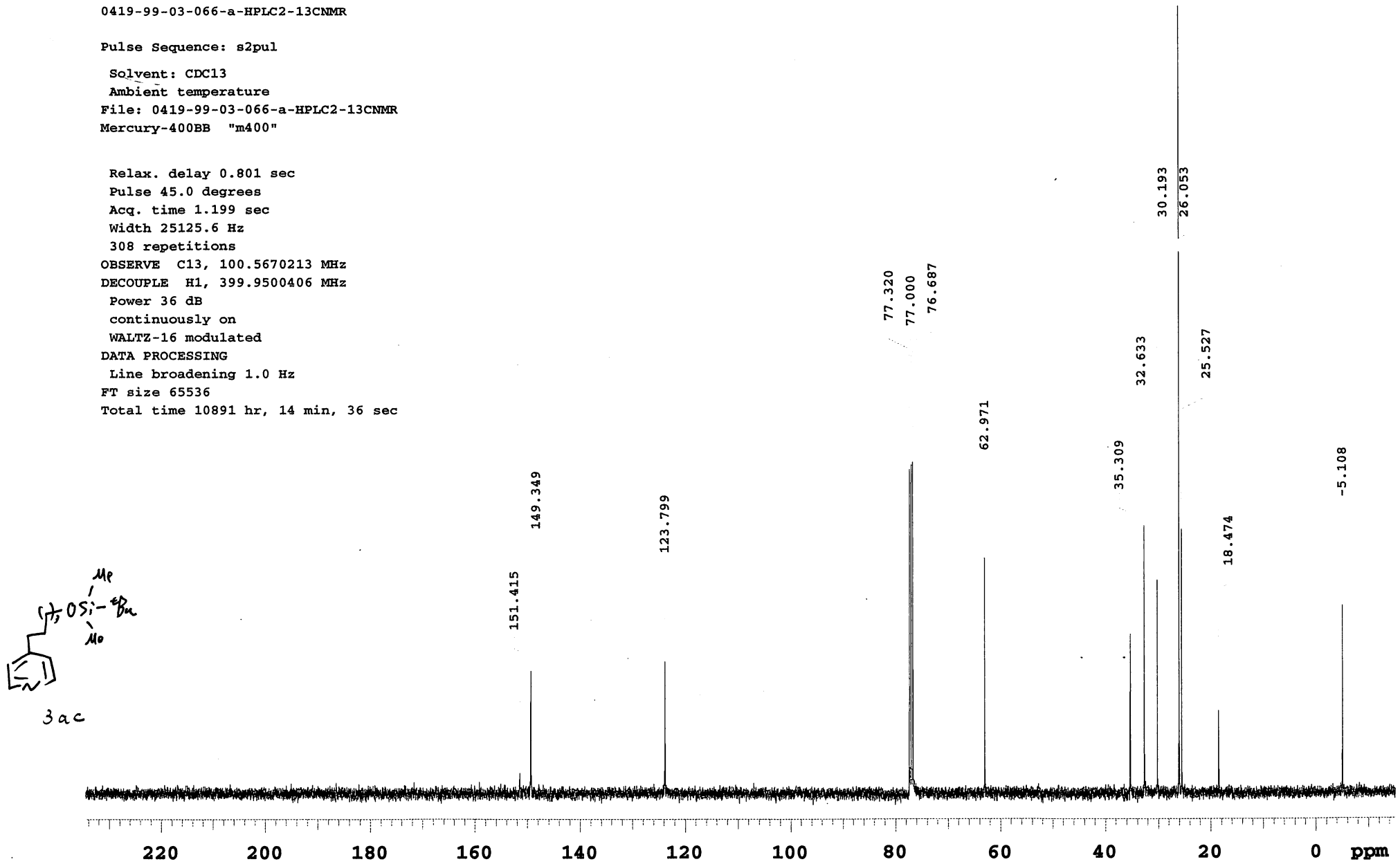

0721-99-04-162a
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.491
8.478
7.136
7.123
3.615
3.599
3.583
3.565
3.549
2.714
2.642
2.623
2.603
1.651
1.526
1.483
1.416
1.378
0.889
0.882
0.875
0.043
0.036
0.028
0.017
8.52 ppm
7.14 ppm
3.64 ppm
2.70 ppm
1.65 ppm
0.9
0.5 ppm
1.97
1.92
2.13
1.94
2.23
2.04
9.00
2.02
5.57
10
9
8
7
6
5
4
3
2
1
ppm
1.97
1.92
2.13
1.94
2.23
2.04
2.02
9.00
5.57

0419-99-03-066-a-HPLC2-13CNMR

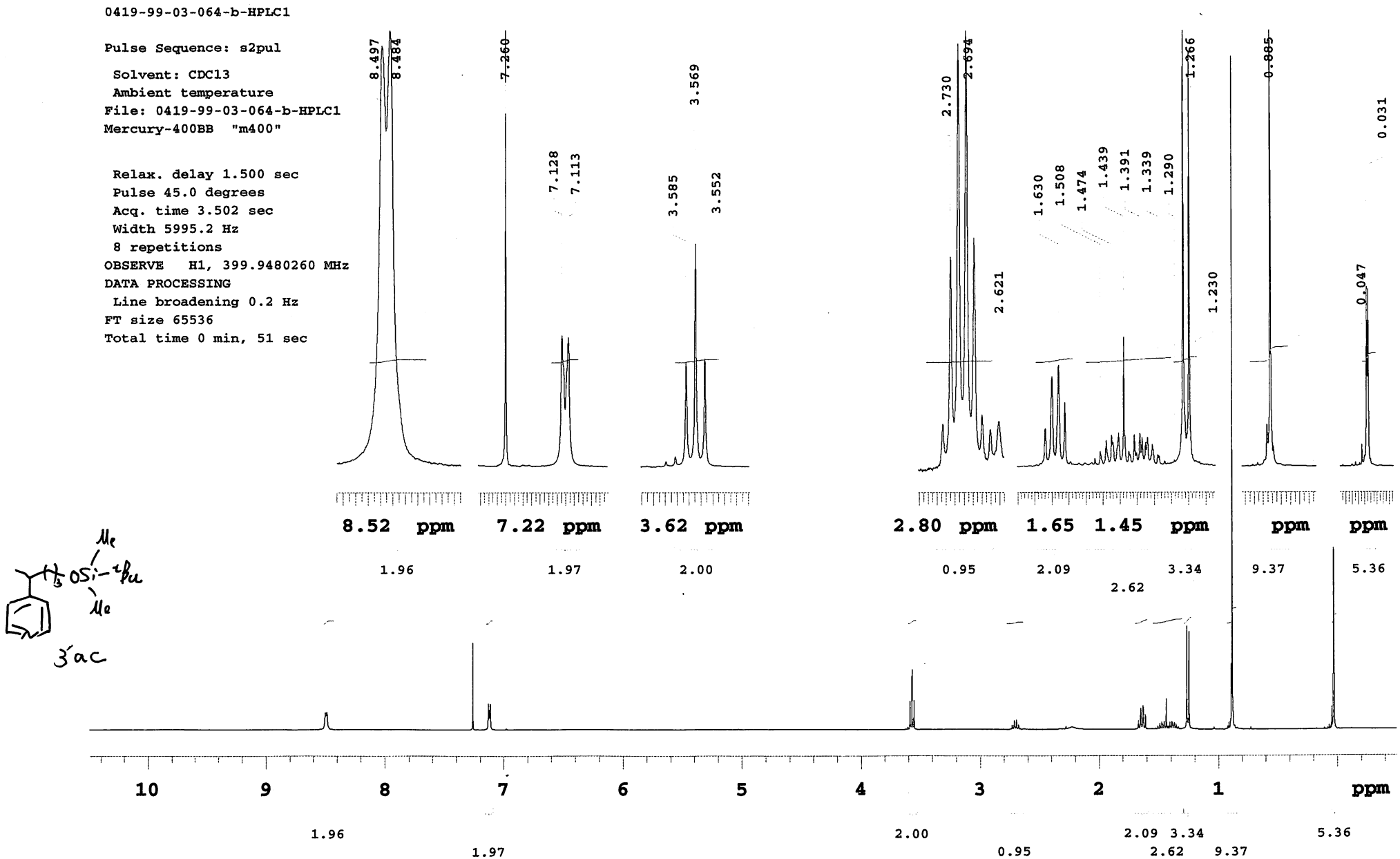
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0419-99-03-066-a-HPLC2-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
308 repetitions
OBSERVE C13, 100.5670213 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec



3ac

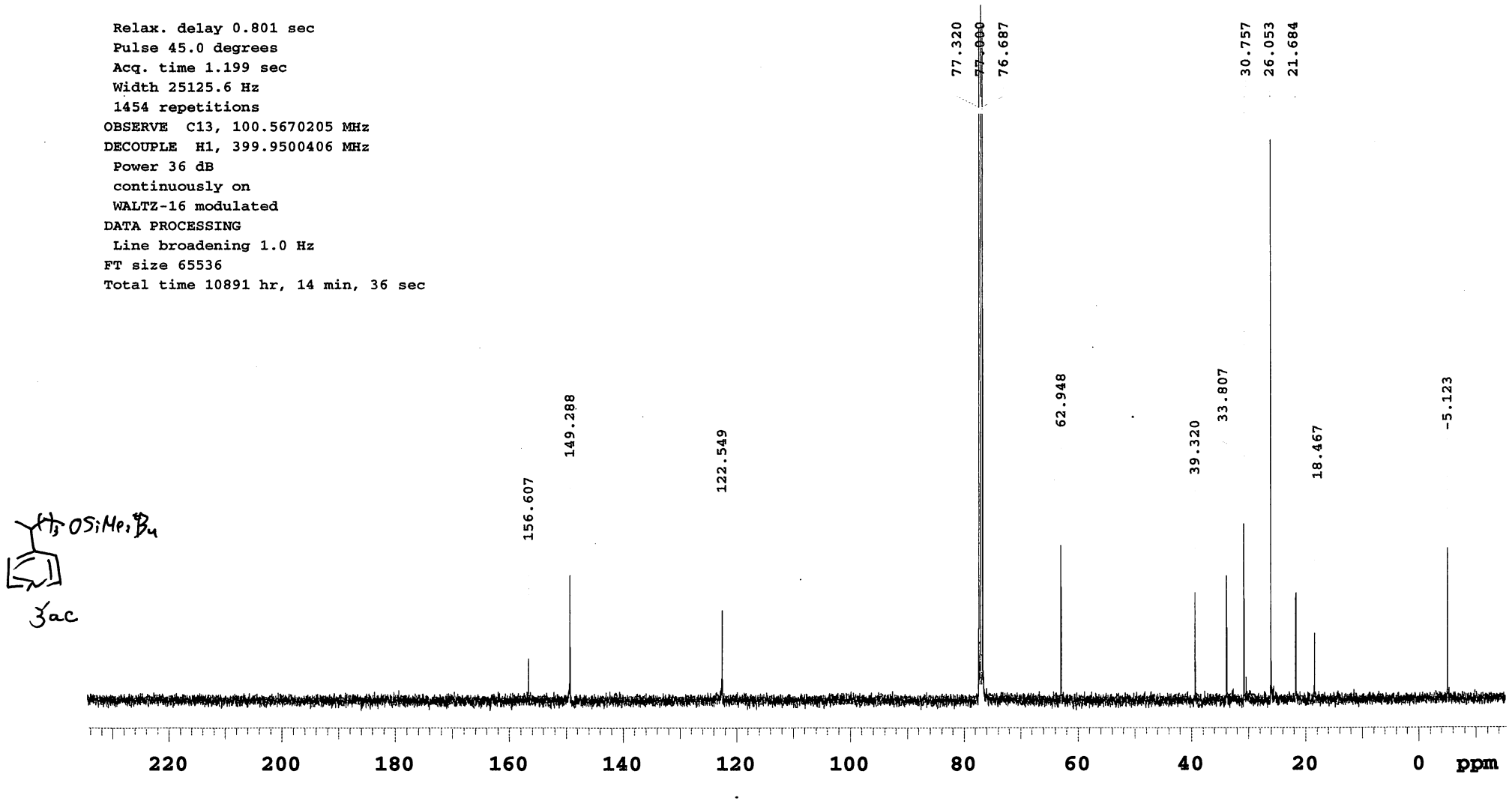

0419-99-03-064-b-HPLC1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0419-99-03-064-b-HPLC1
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480260 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.497
8.484
7.260
7.128
7.113
3.585
3.569
3.552
2.730
2.694
2.621
1.630
1.508
1.474
1.439
1.391
1.339
1.290
1.266
1.230
0.885
0.047
0.031
8.52 ppm
7.22 ppm
3.62 ppm
2.80 ppm
1.65 1.45 ppm
ppm
ppm
1.96
1.97
2.00
0.95
2.09
2.62
3.34
9.37
5.36
Me
OSi-tBu
Me
3ac
10
9
8
7
6
5
4
3
2
1
ppm
1.96
1.97
2.00
0.95
2.09
2.62
3.34
9.37
5.36

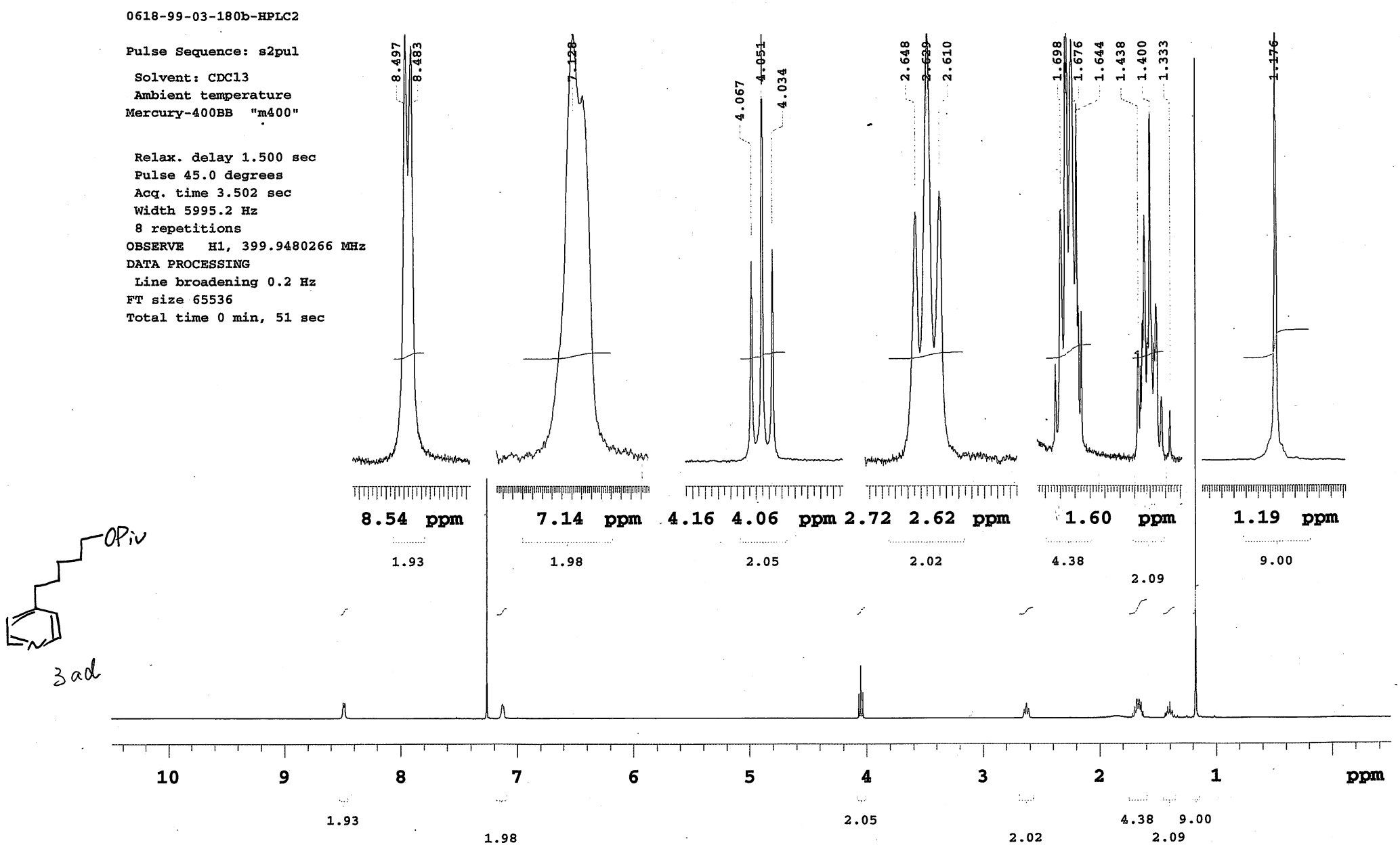
0420-99-03-064-b-HPLC1-13CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0420-99-03-064-b-HPLC1-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1454 repetitions
OBSERVE C13, 100.5670205 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec



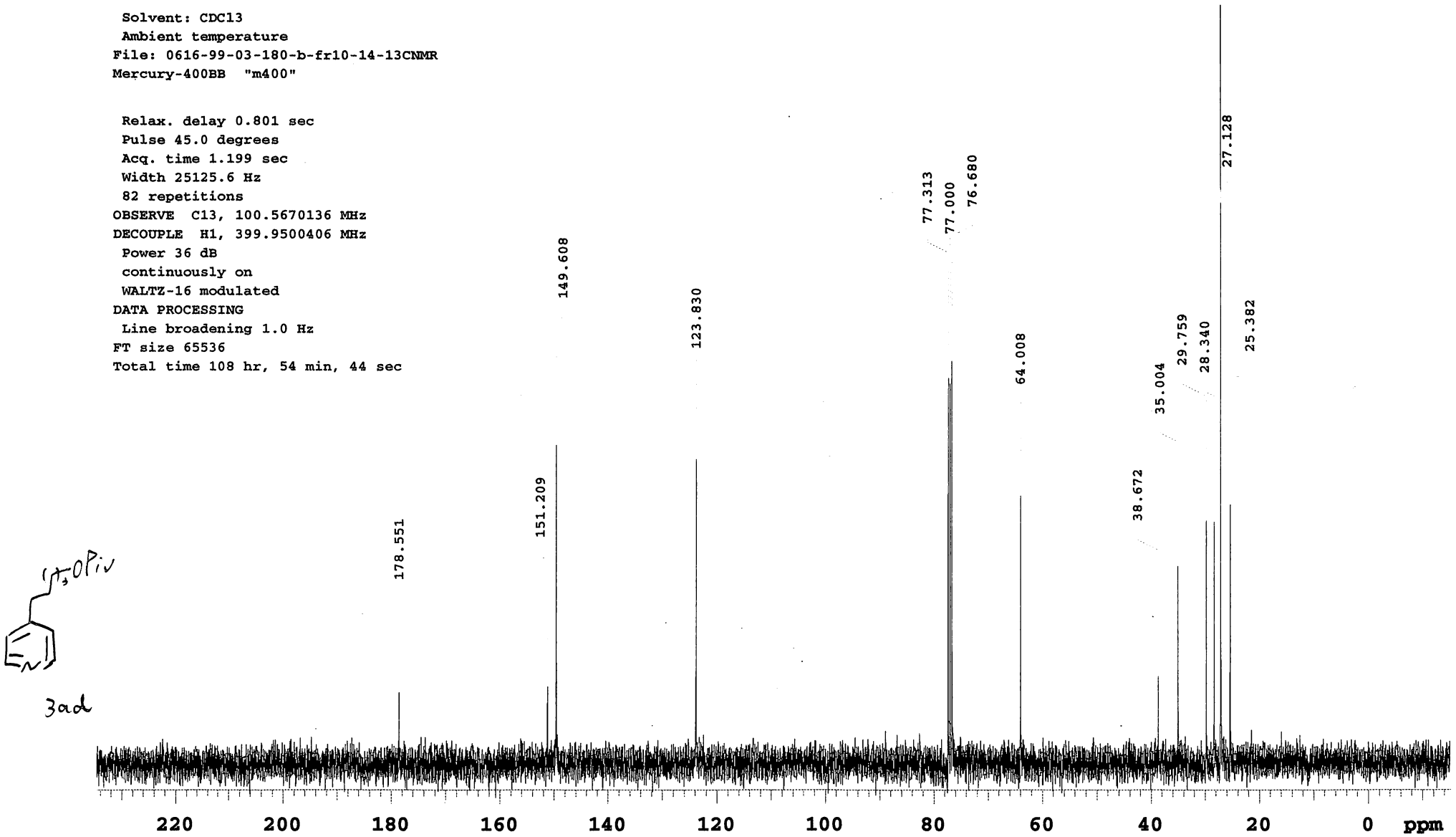

0618-99-03-180b-HPLC2
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.497
8.483
7.128
4.067
4.051
4.034
2.648
2.629
2.610
1.698
1.676
1.644
1.438
1.400
1.333
1.176
8.54 ppm
7.14 ppm
4.16 4.06 ppm
2.72 2.62 ppm
1.60 ppm
1.19 ppm
1.93
1.98
2.05
2.02
4.38
2.09
9.00
OPiv
3ad
10
9
8
7
6
5
4
3
2
1
ppm

0616-99-03-180-b-fr10-14-13CNMR

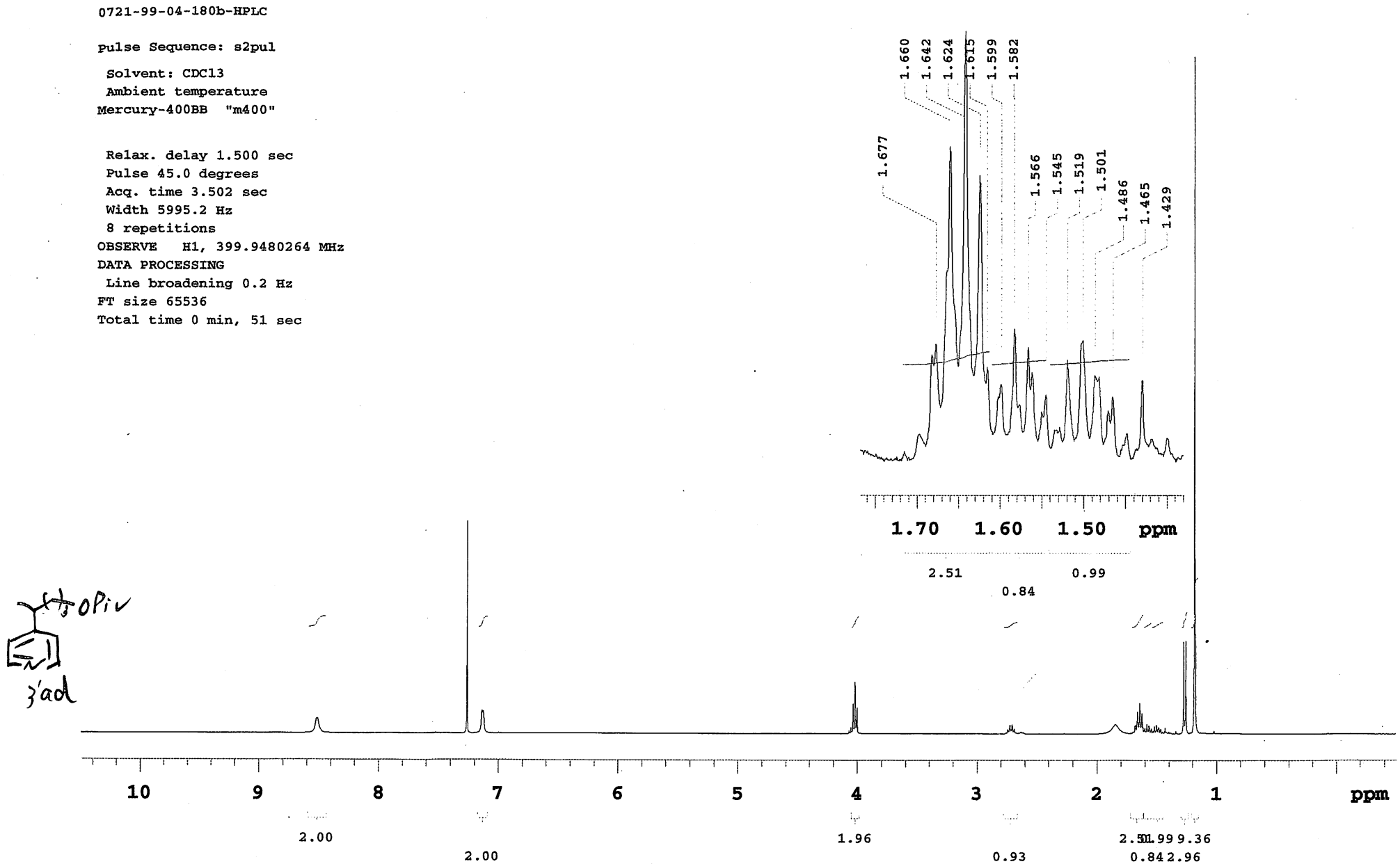
Pulse Sequence: s2pul

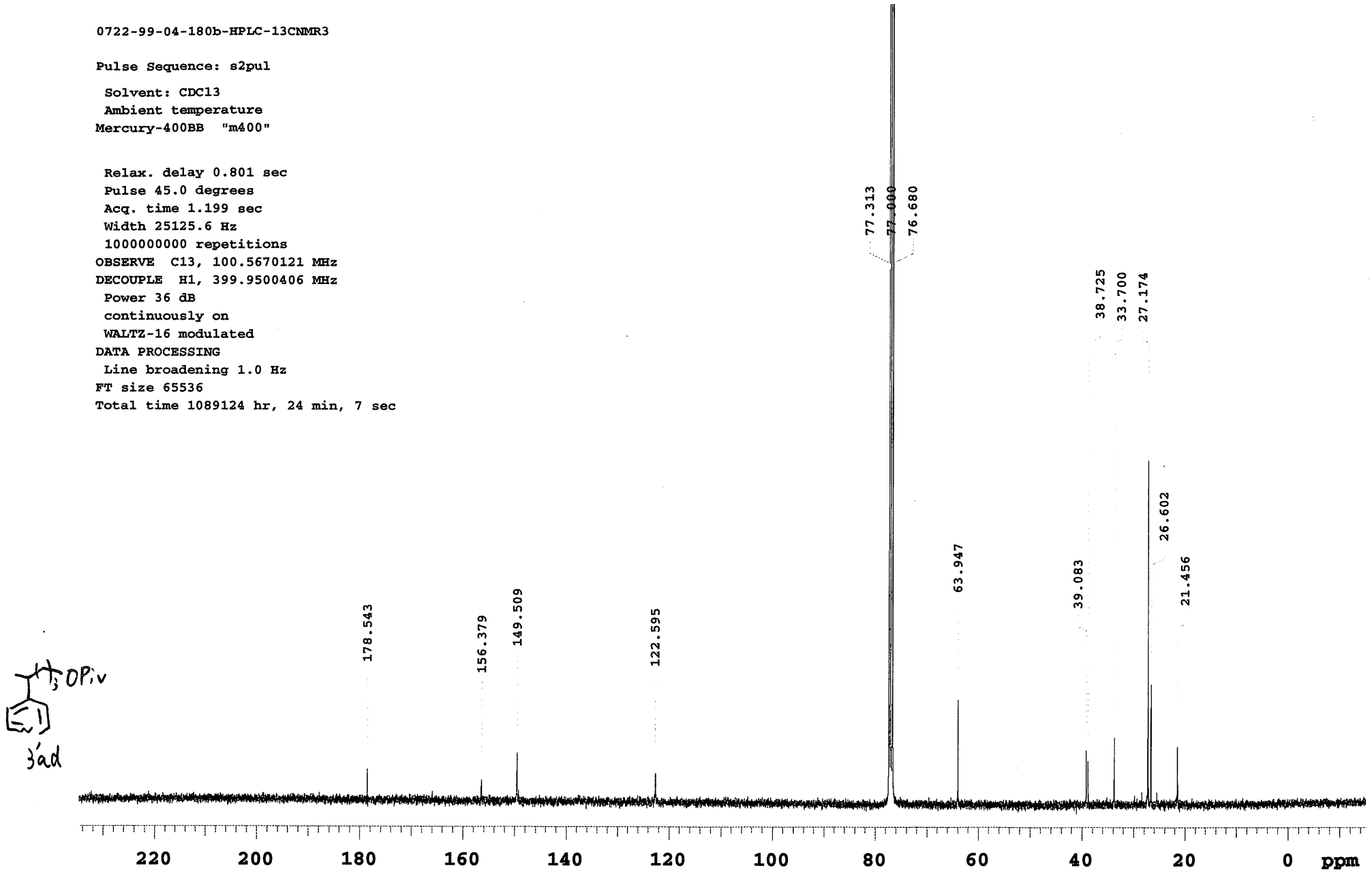
Solvent: CDCl3
Ambient temperature
File: 0616-99-03-180-b-fr10-14-13CNMR
Mercury-400BB "m400"

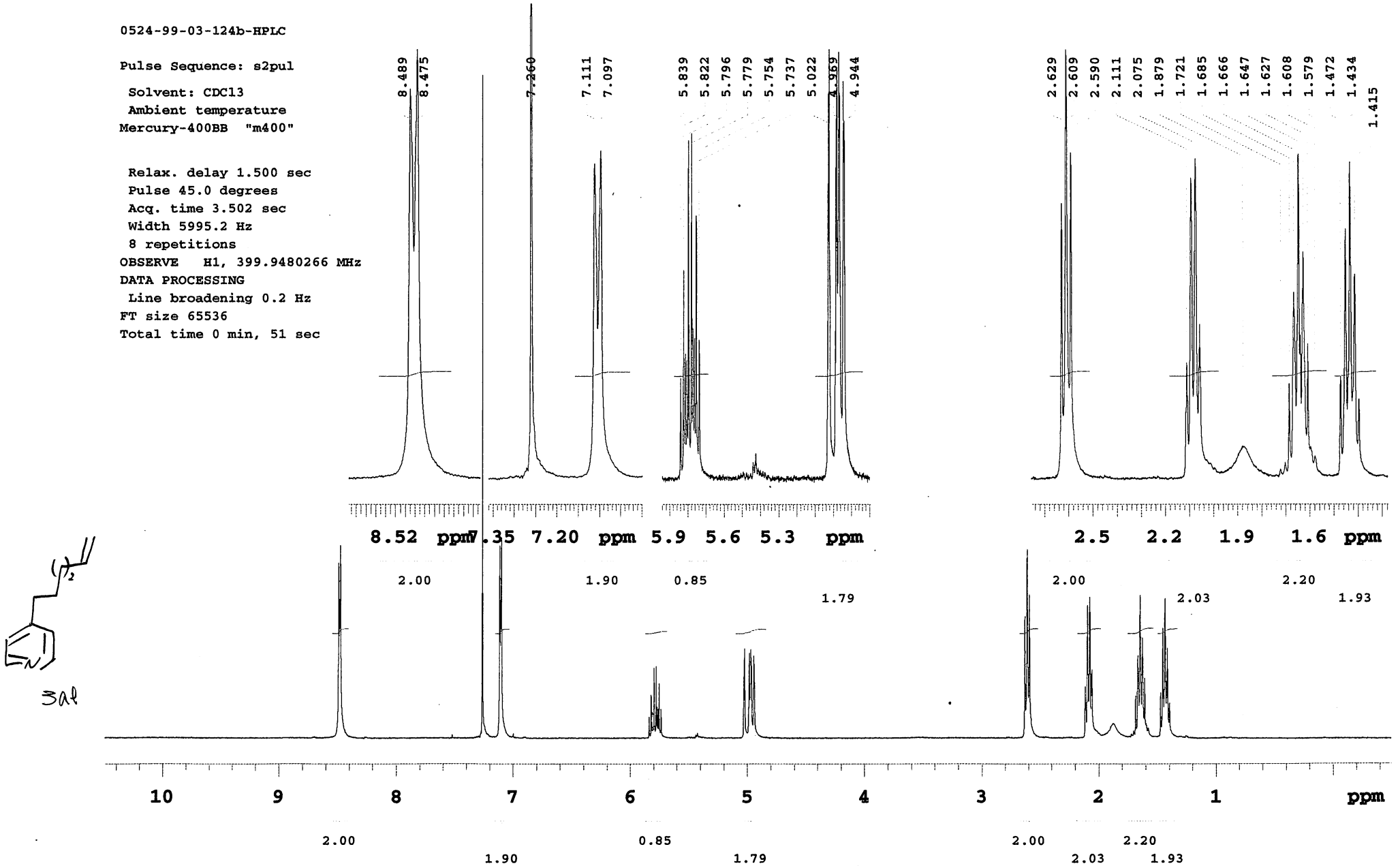
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
82 repetitions
OBSERVE C13, 100.5670136 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec



3ad


0721-99-04-180b-HPLC
pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480264 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
1.660
1.642
1.624
1.615
1.599
1.582
1.677
1.566
1.545
1.519
1.501
1.486
1.465
1.429
1.70
1.60
1.50
ppm
2.51
0.99
0.84
OPiv
3'ad
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.00
1.96
0.93
2.51
1.99
9.36
0.84
2.96


0722-99-04-180b-HPLC-13CNMR3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1000000000 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1089124 hr, 24 min, 7 sec
77.313
77.000
76.680
38.725
33.700
27.174
178.543
156.379
149.509
122.595
63.947
39.083
26.602
21.456
OPiv
3'ad
220
200
180
160
140
120
100
80
60
40
20
0
ppm

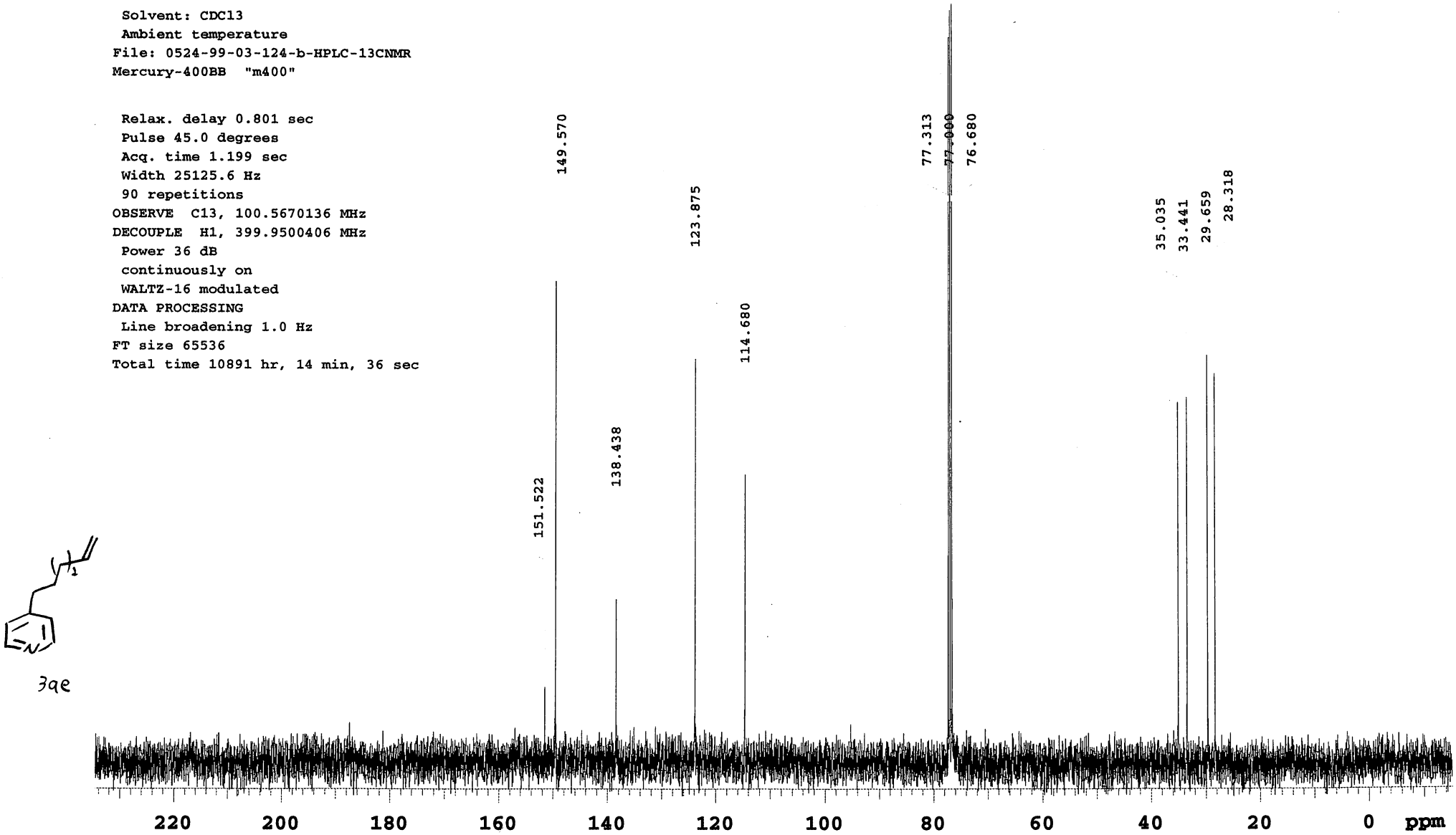

0524-99-03-124b-HPLC
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.489
8.475
7.260
7.111
7.097
5.839
5.822
5.796
5.779
5.754
5.737
5.022
4.969
4.944
2.629
2.609
2.590
2.111
2.075
1.879
1.721
1.685
1.666
1.647
1.627
1.608
1.579
1.472
1.434
1.415
8.52 ppm
7.35 7.20 ppm
5.9 5.6 5.3 ppm
2.5 2.2 1.9 1.6 ppm
2.00
1.90
0.85
1.79
2.00
2.03
2.20
1.93
10 9 8 7 6 5 4 3 2 1 ppm
2.00
1.90
0.85
1.79
2.00
2.03
2.20
1.93

0524-99-03-124-b-HPLC-13CNMR

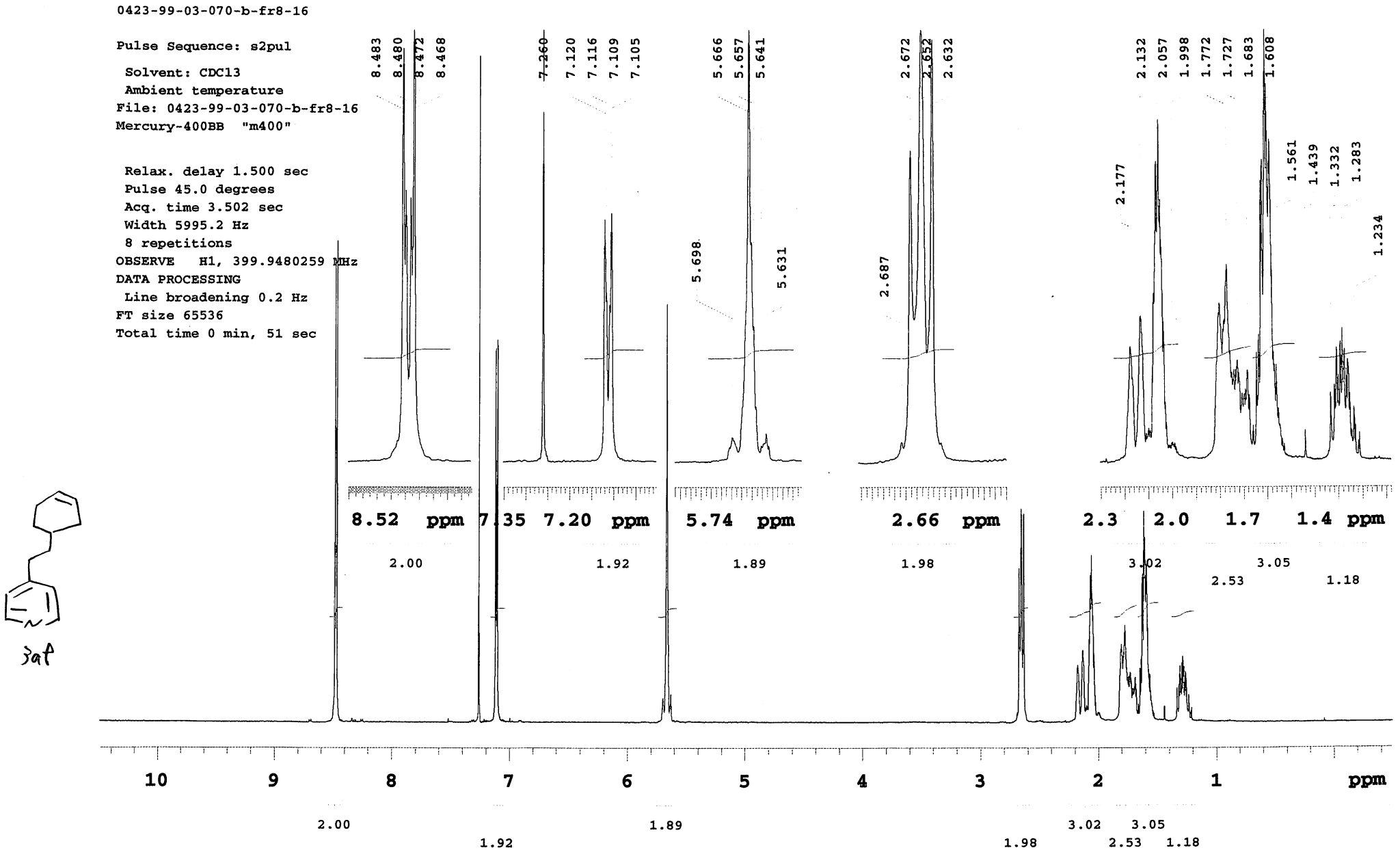
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0524-99-03-124-b-HPLC-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
90 repetitions
OBSERVE C13, 100.5670136 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec



3qe

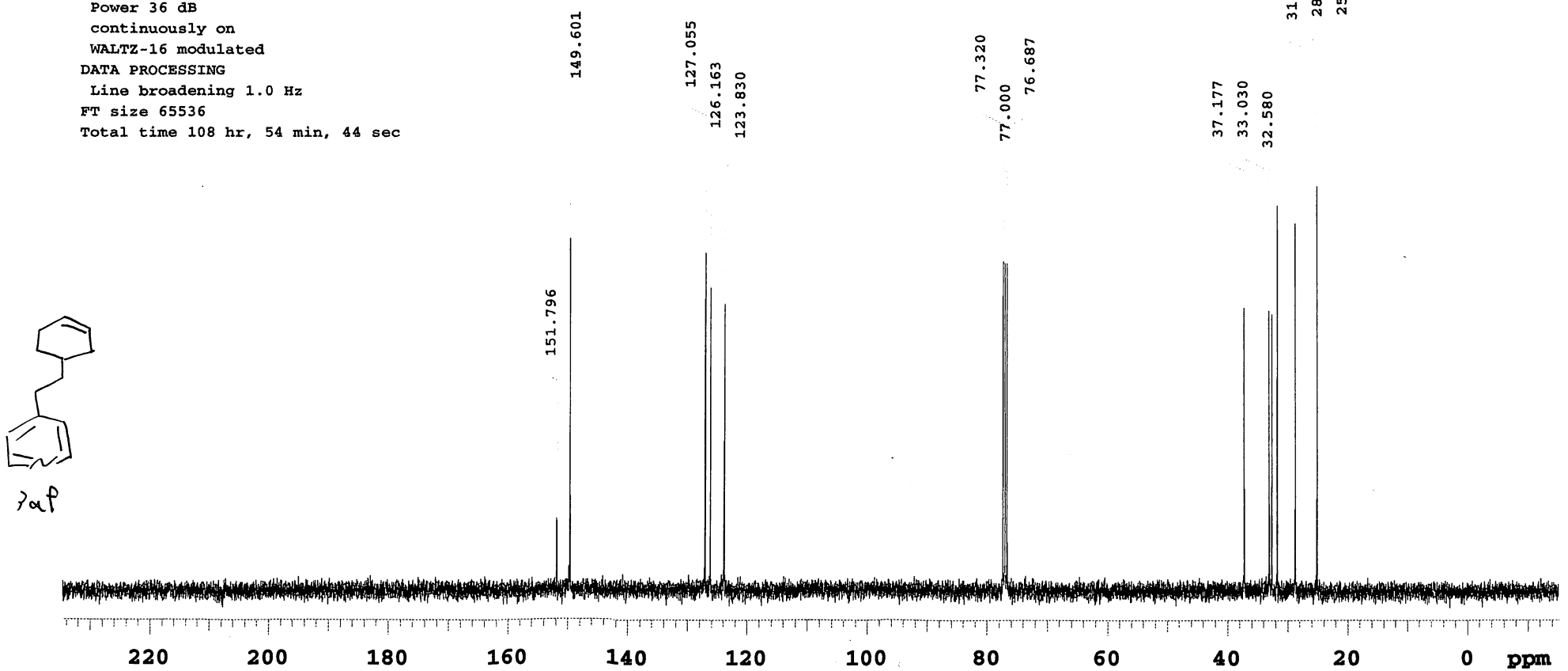

0423-99-03-070-b-fr8-16
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0423-99-03-070-b-fr8-16
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480259 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.483
8.480
8.472
8.468
7.260
7.120
7.116
7.109
7.105
5.666
5.657
5.641
5.698
5.631
2.672
2.652
2.632
2.687
2.177
2.132
2.057
1.998
1.772
1.727
1.683
1.608
1.561
1.439
1.332
1.283
1.234
8.52 ppm
7.35
7.20 ppm
5.74 ppm
2.66 ppm
2.3
2.0
1.7
1.4 ppm
2.00
1.92
1.89
1.98
3.02
3.05
2.53
1.18
10
9
8
7
6
5
4
3
2
1
ppm
3af

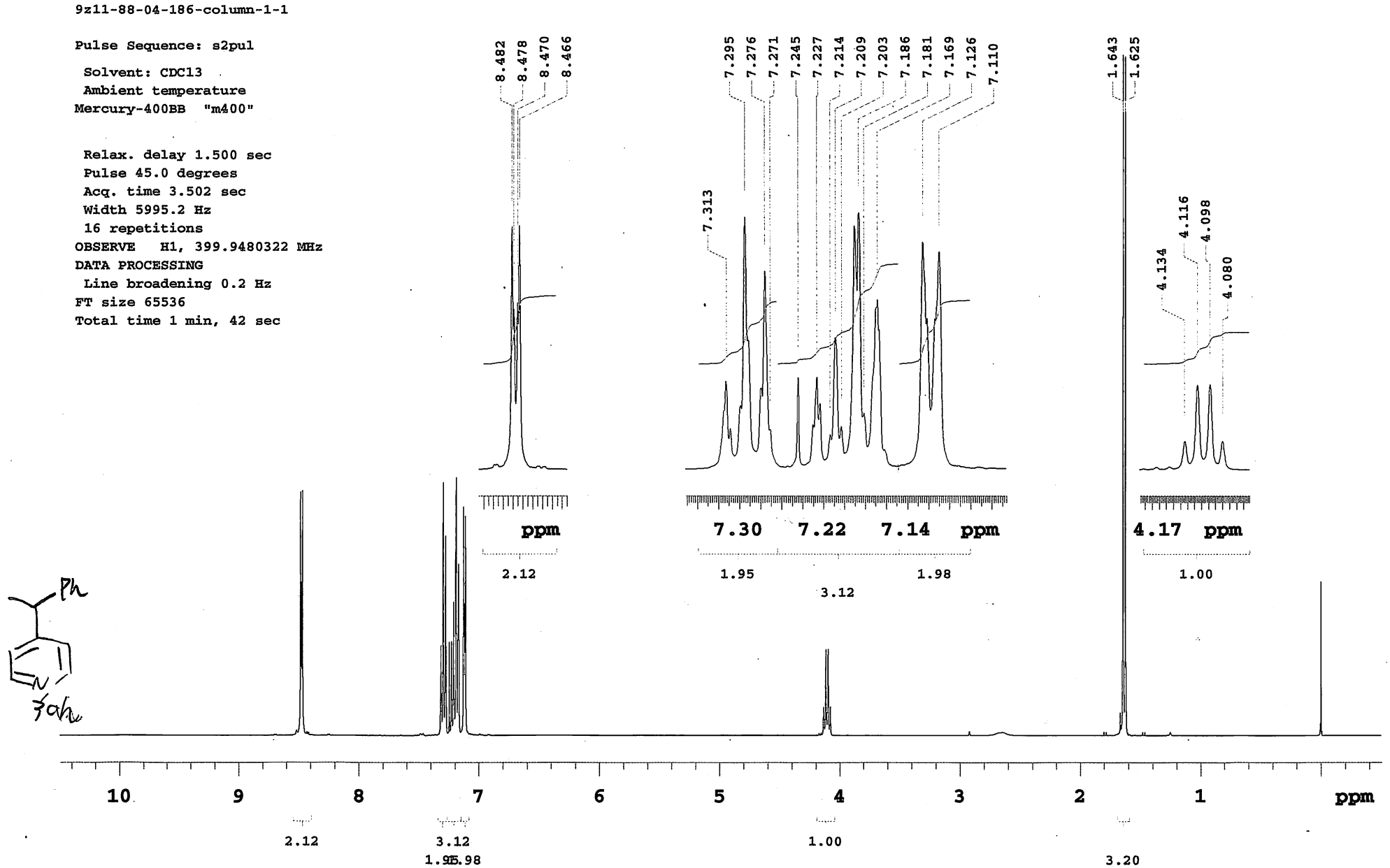
0628-99-03-070b-column1-13CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

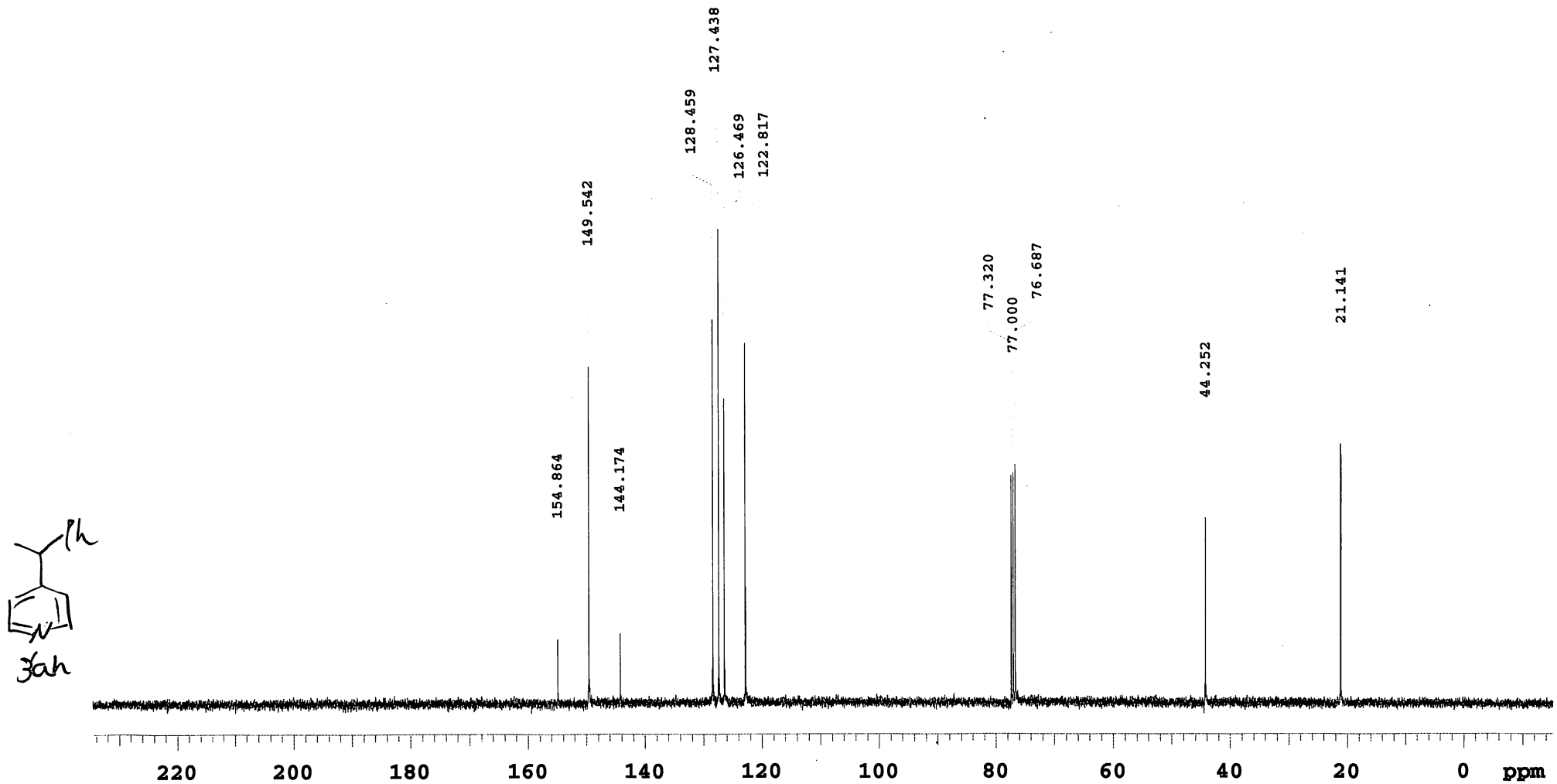
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
154 repetitions
OBSERVE C13, 100.5670144 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec

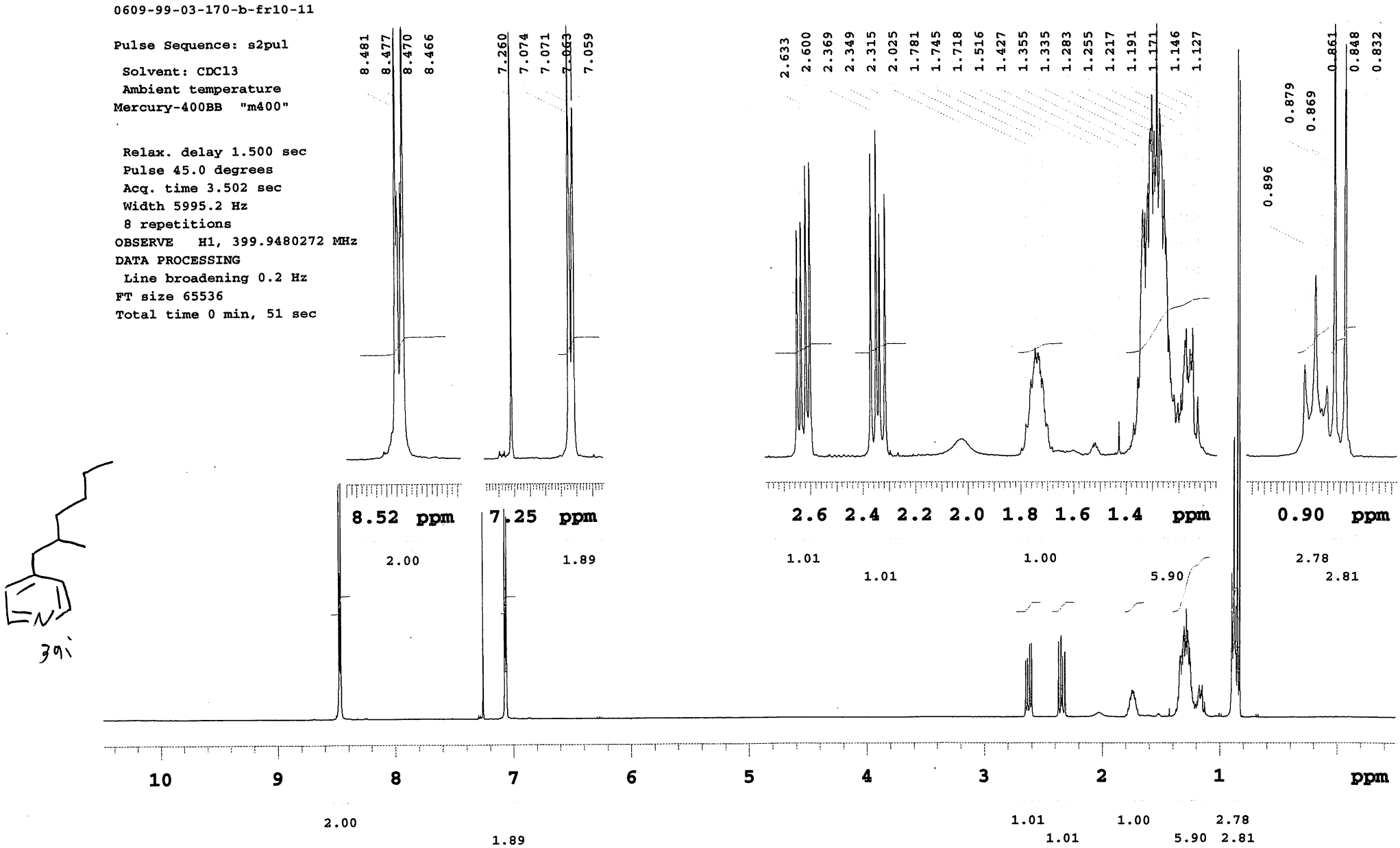



9z11-88-04-186-column-1-1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
16 repetitions
OBSERVE H1, 399.9480322 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 42 sec
8.482
8.478
8.470
8.466
7.313
7.295
7.276
7.271
7.245
7.227
7.214
7.209
7.203
7.186
7.181
7.169
7.126
7.110
1.643
1.625
4.134
4.116
4.098
4.080
ppm
7.30
7.22
7.14
ppm
4.17
ppm
2.12
1.95
3.12
1.98
1.00
Ph
Tah
10
9
8
7
6
5
4
3
2
1
ppm
2.12
3.12
1.00
3.20

9z11-88-04-186-column-1-1

Pulse Sequence: s2pul



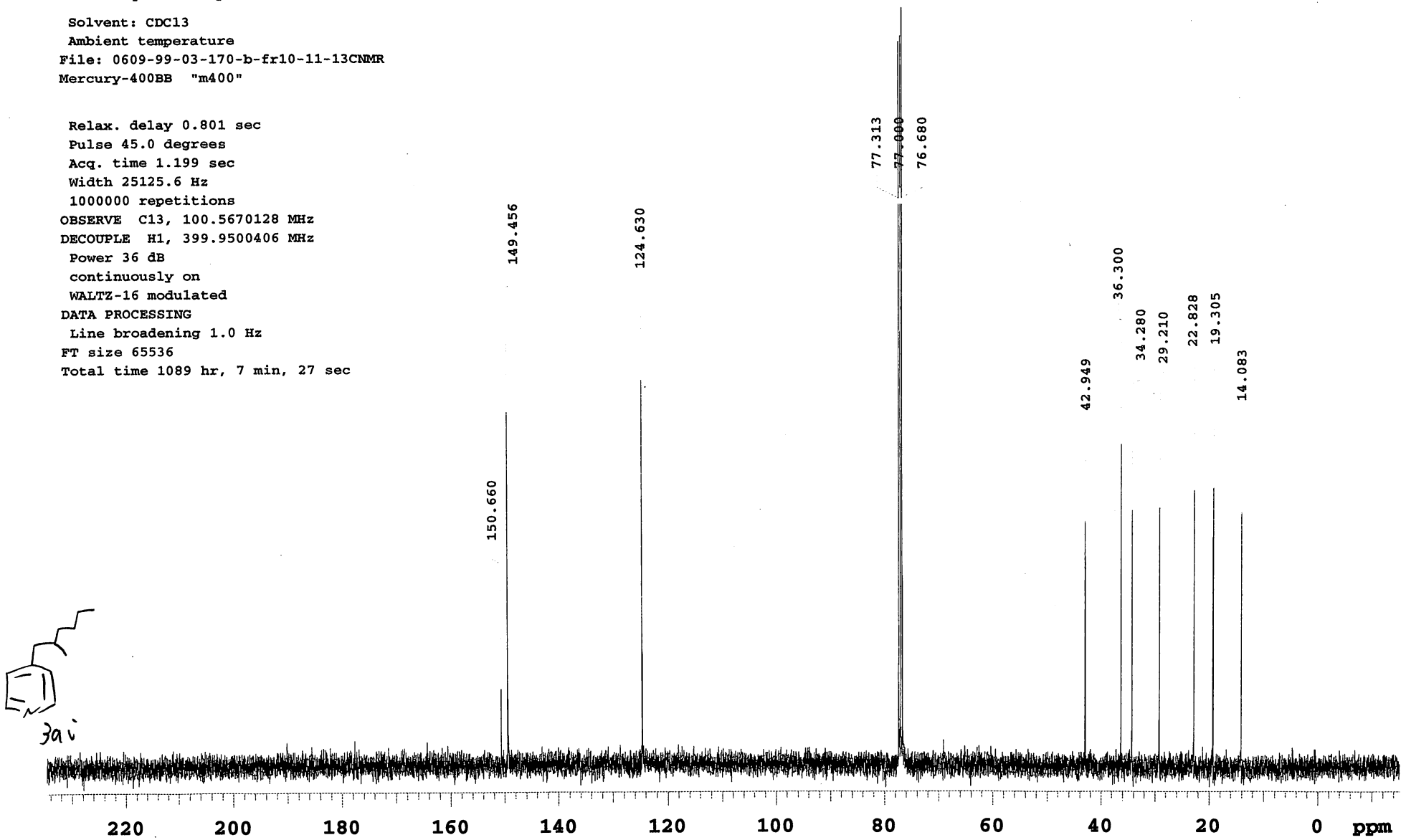

0609-99-03-170-b-fr10-11
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480272 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.481 8.477 8.470 8.466
7.260 7.074 7.071 7.063 7.059
2.633 2.600 2.369 2.349 2.315 2.025 1.781 1.745 1.718 1.516 1.427 1.355 1.335 1.283 1.255 1.217 1.191 1.171 1.146 1.127
0.896 0.879 0.869 0.861 0.848 0.832
8.52 ppm
7.25 ppm
2.6 2.4 2.2 2.0 1.8 1.6 1.4 ppm
0.90 ppm
2.00
1.89
1.01
1.01
1.00
5.90
2.78
2.81
10 9 8 7 6 5 4 3 2 1 ppm
2.00
1.89
1.01
1.01
1.00
5.90
2.78
2.81

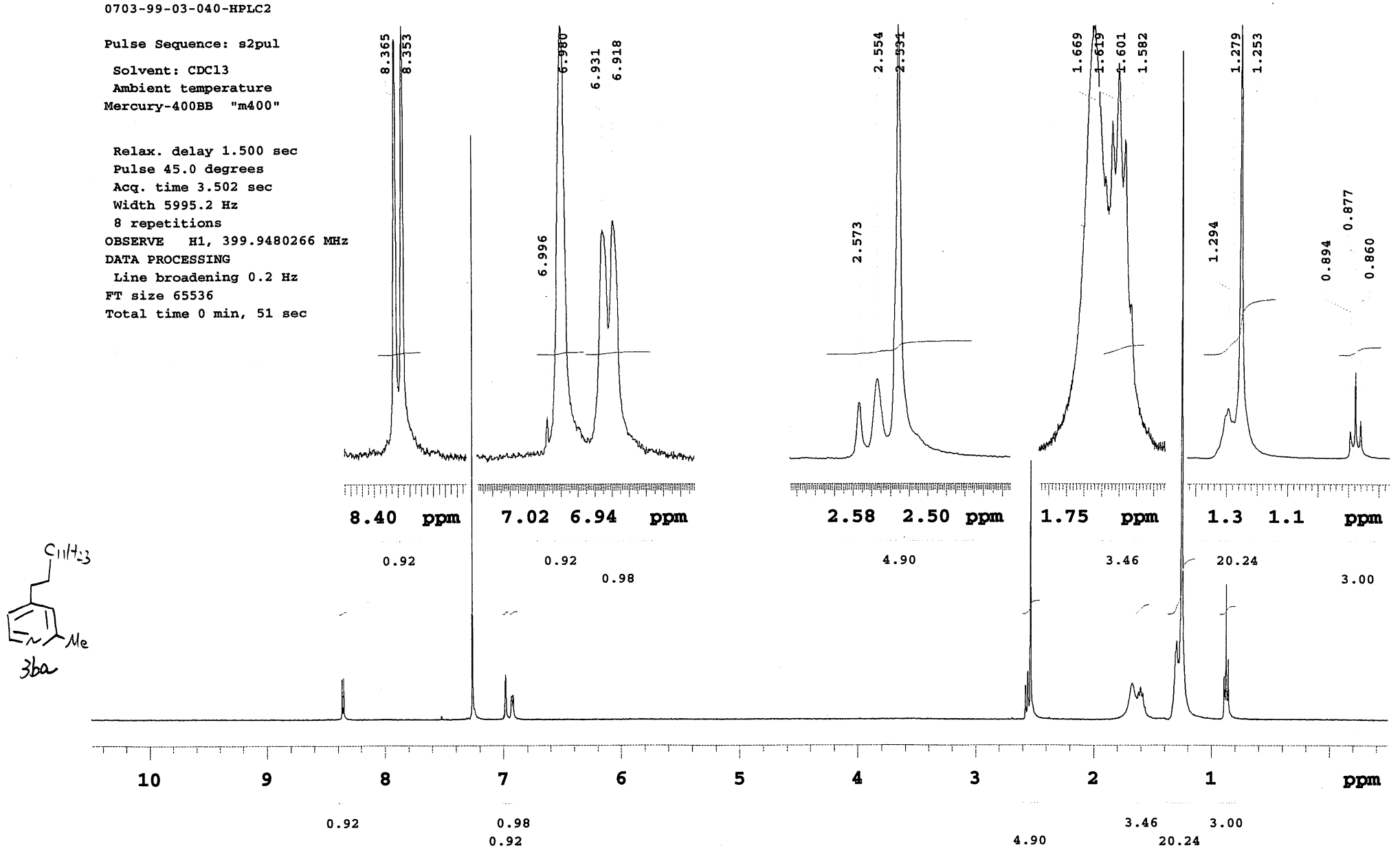
0609-99-03-170-b-fr10-11-13CNMR

Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
File: 0609-99-03-170-b-fr10-11-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
1000000 repetitions
OBSERVE C13, 100.5670128 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1089 hr, 7 min, 27 sec



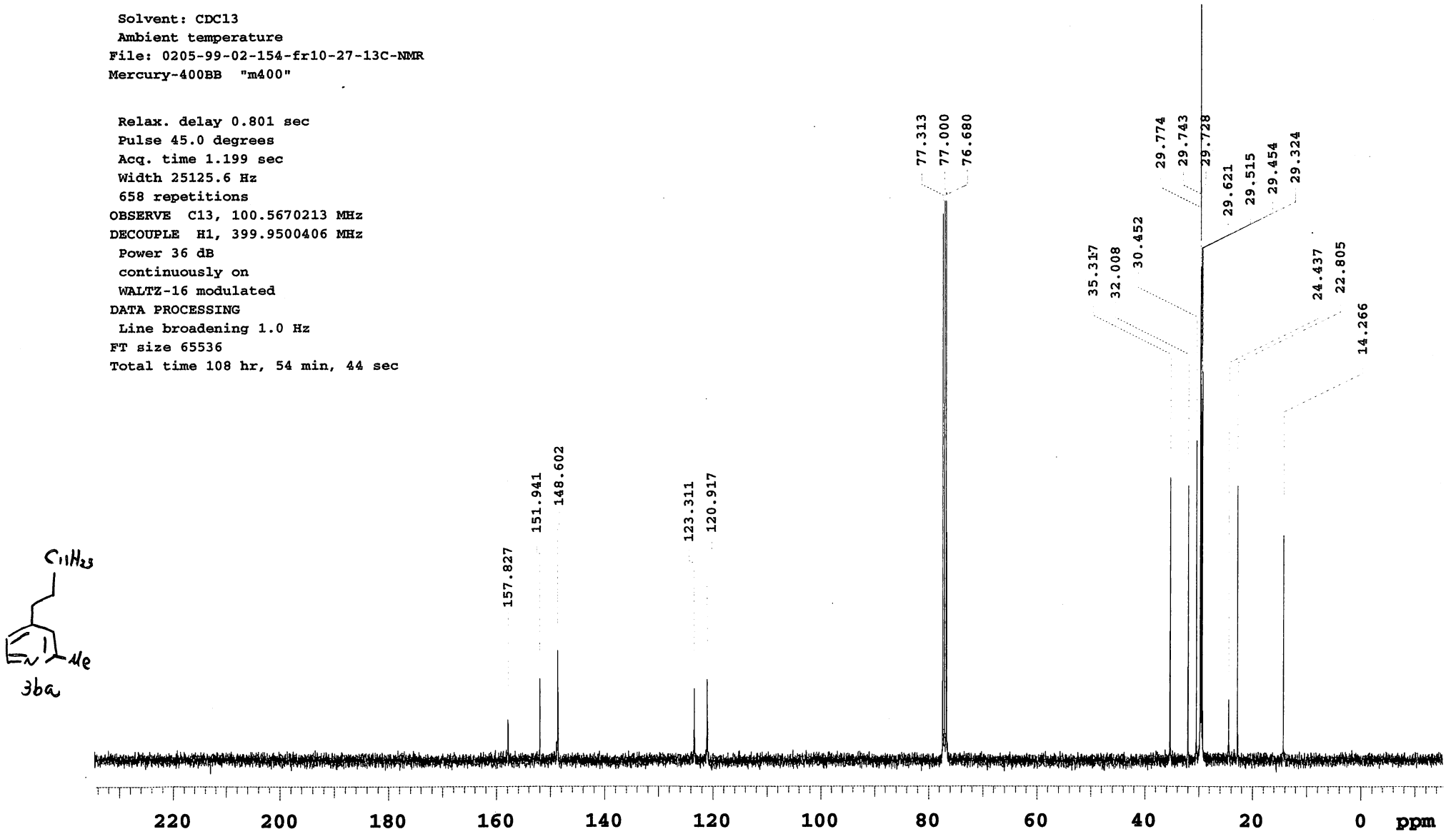

0703-99-03-040-HPLC2
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.365
8.353
6.980
6.996
6.931
6.918
2.573
2.554
2.534
1.669
1.619
1.601
1.582
1.294
1.279
1.253
0.894
0.877
0.860
8.40 ppm
7.02 6.94 ppm
2.58 2.50 ppm
1.75 ppm
1.3 1.1 ppm
0.92
0.92
0.98
4.90
3.46
20.24
3.00
10 9 8 7 6 5 4 3 2 1 ppm
0.92
0.98
0.92
4.90
3.46
20.24
3.00
C11H13
Me
3ba

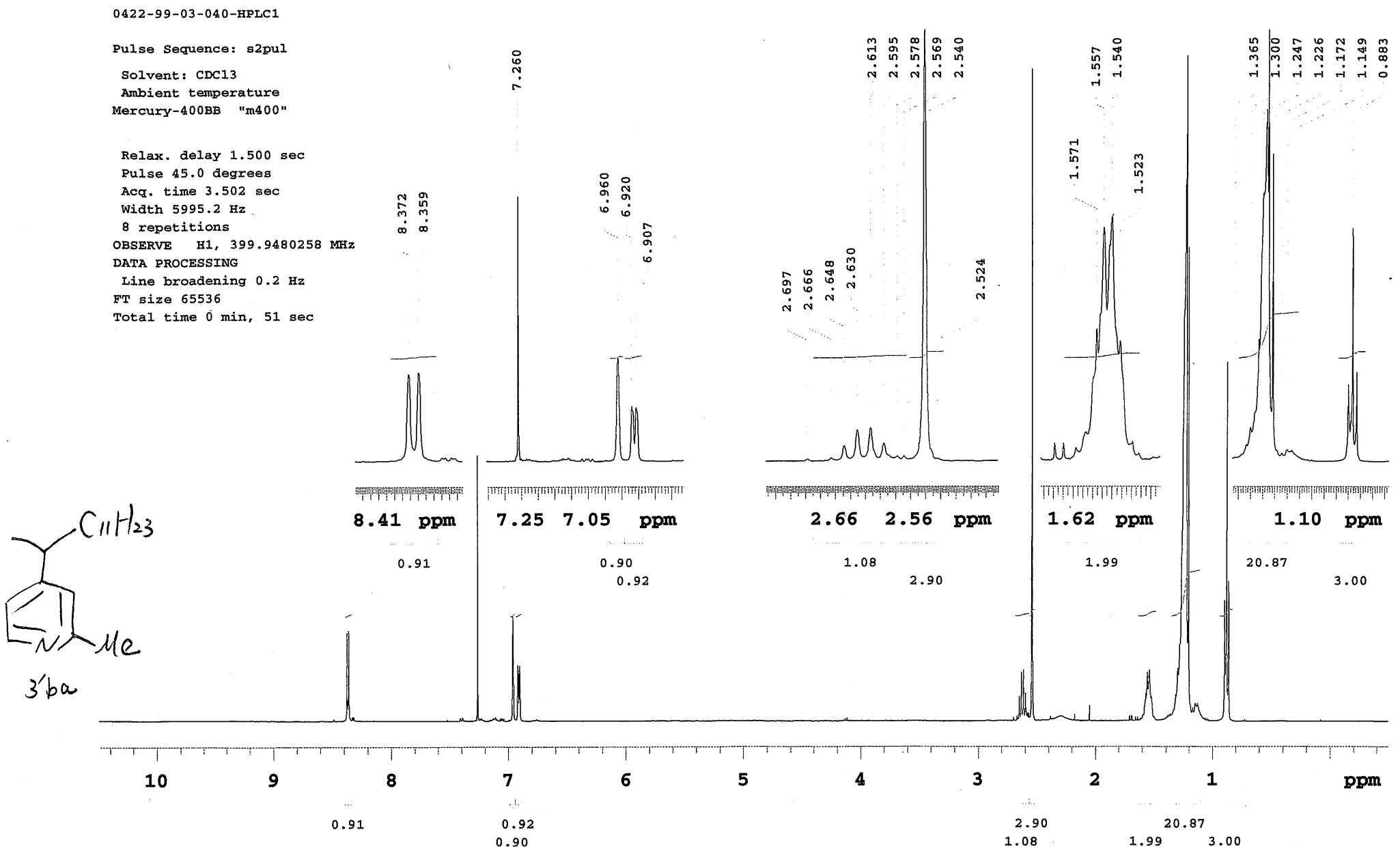
0205-99-02-154-fr10-27-13C-NMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0205-99-02-154-fr10-27-13C-NMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
658 repetitions
OBSERVE C13, 100.5670213 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec



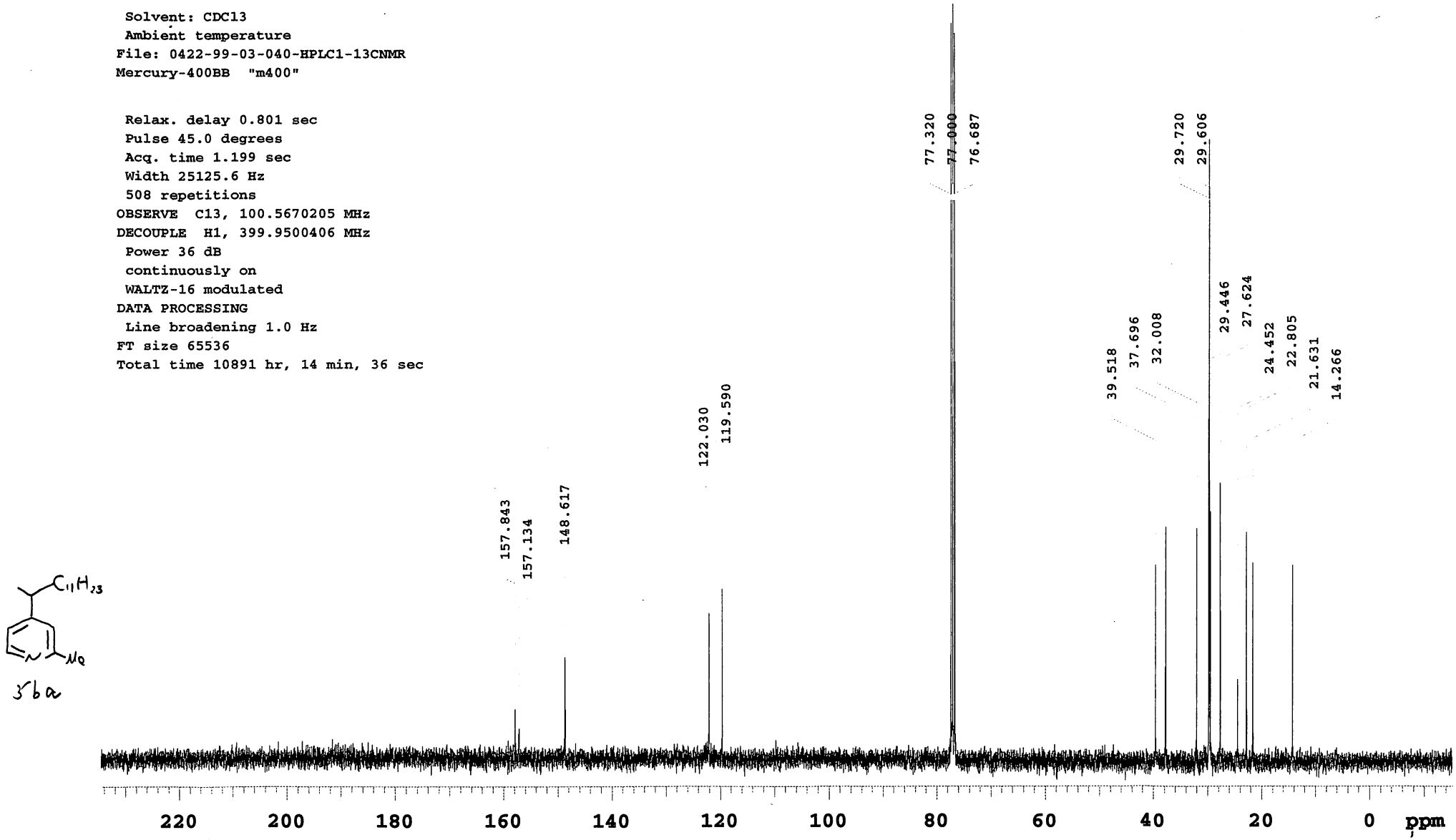

0422-99-03-040-HPLC1
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480258 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.372
8.359
7.260
6.960
6.920
6.907
2.697
2.666
2.648
2.630
2.613
2.595
2.578
2.569
2.540
2.524
1.571
1.557
1.540
1.523
1.365
1.300
1.247
1.226
1.172
1.149
0.883
8.41 ppm
7.25 7.05 ppm
2.66 2.56 ppm
1.62 ppm
1.10 ppm
0.91
0.90
0.92
1.08
2.90
1.99
20.87
3.00
C11H23
Me
3'ba
10
9
8
7
6
5
4
3
2
1
ppm

0422-99-03-040-HPLC1-13CNMR

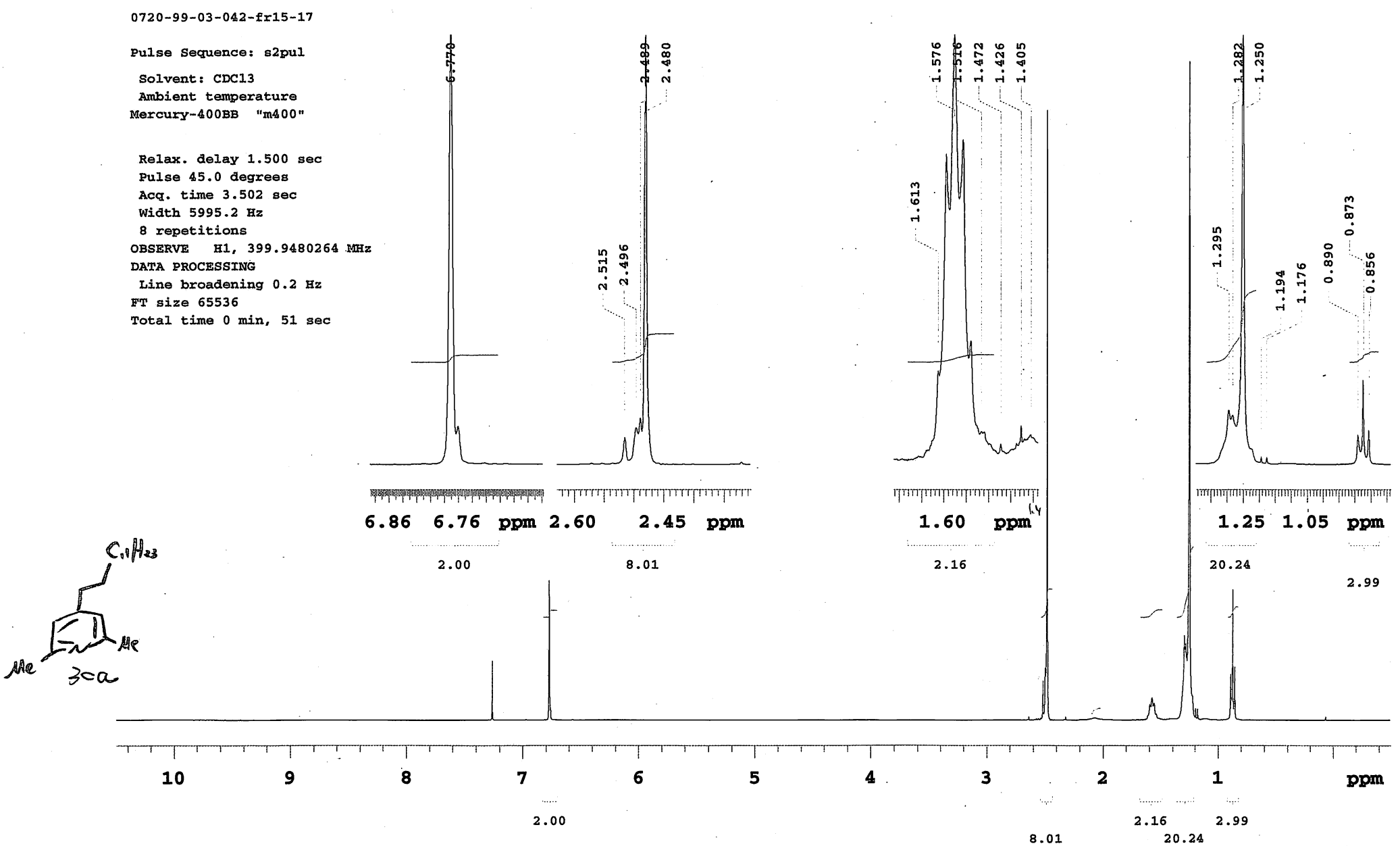
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0422-99-03-040-HPLC1-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
508 repetitions
OBSERVE C13, 100.5670205 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec



3ba

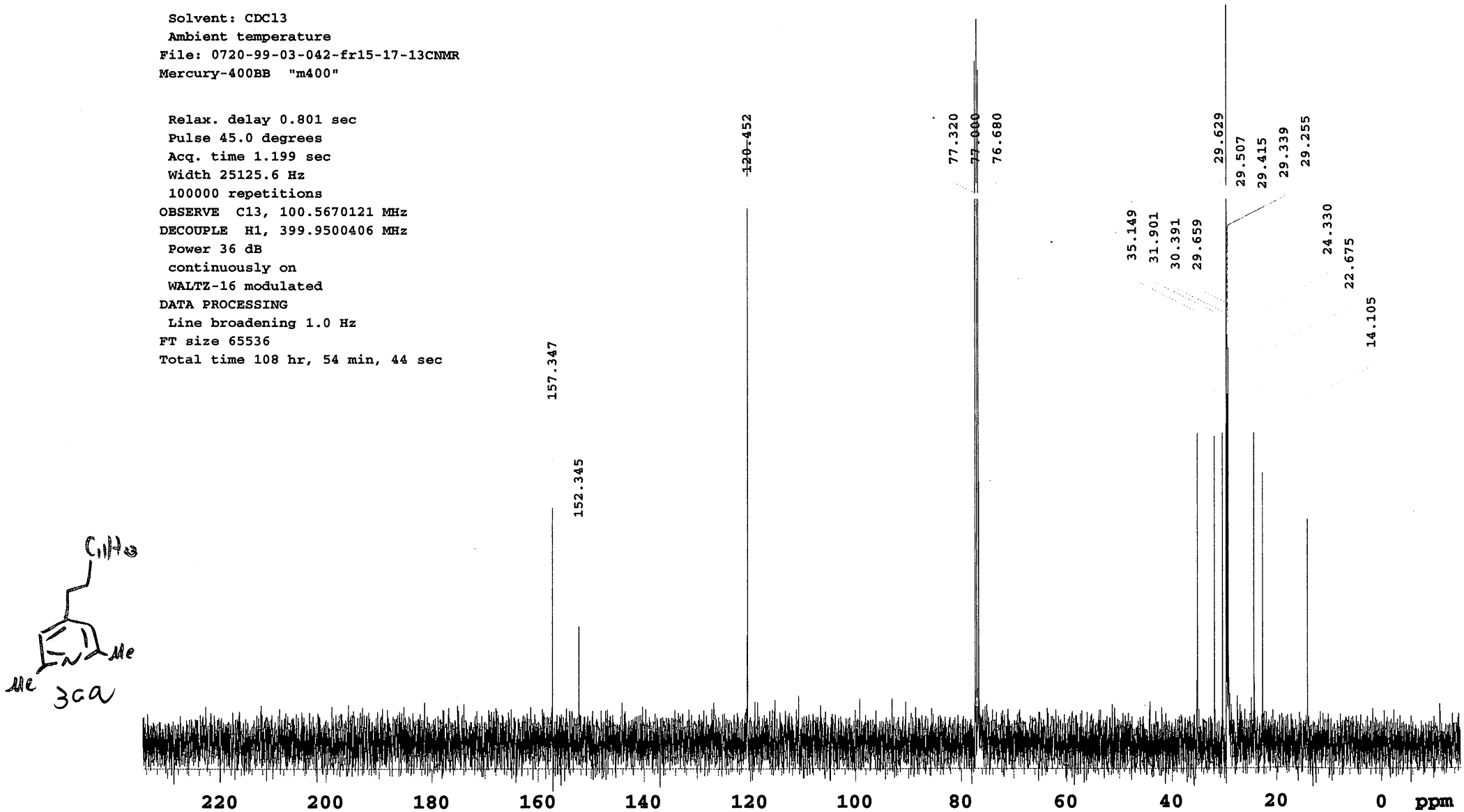

0720-99-03-042-fr15-17
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480264 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
6.770
2.515
2.496
2.489
2.480
1.613
1.576
1.516
1.472
1.426
1.405
1.295
1.282
1.250
1.194
1.176
0.890
0.873
0.856
6.86 6.76 ppm
2.60 2.45 ppm
1.60 ppm
1.25 1.05 ppm
2.00
8.01
2.16
20.24
2.99
10 9 8 7 6 5 4 3 2 1 ppm
C11H23
Me
Me
30a

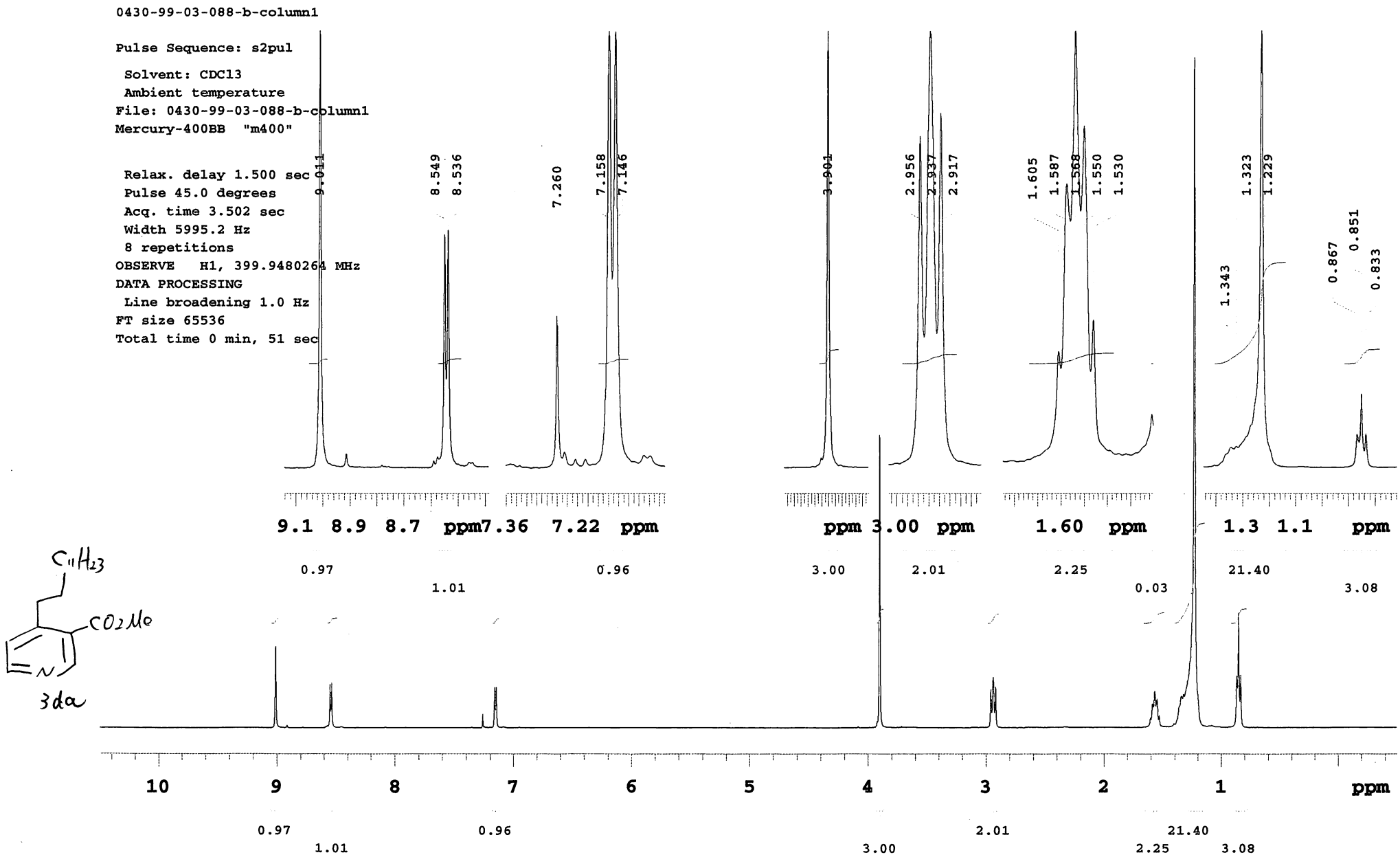
0720-99-03-042-fr15-17-13CNMR

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 0720-99-03-042-fr15-17-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
100000 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec



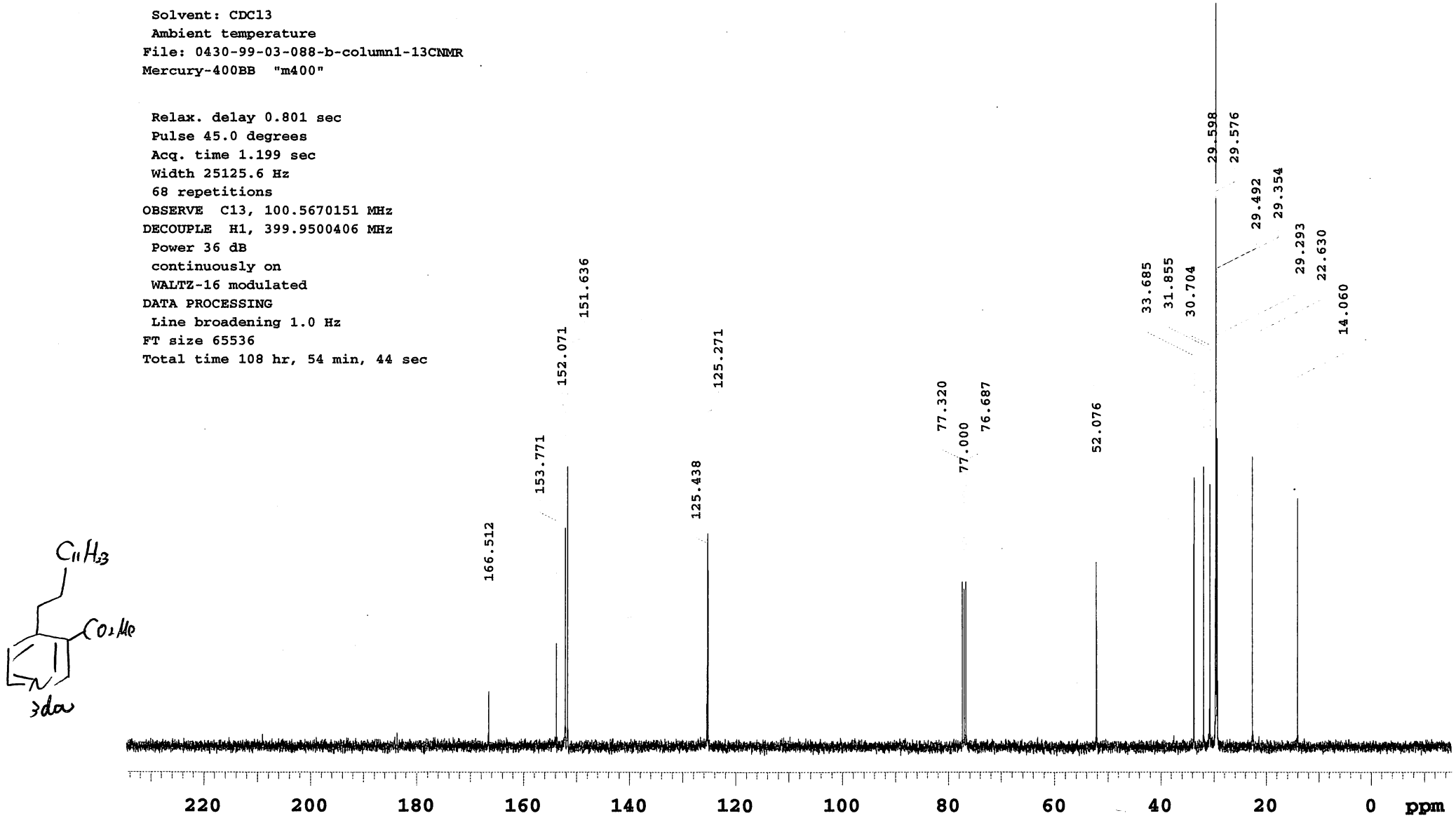

0430-99-03-088-b-column1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0430-99-03-088-b-column1
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480264 MHz
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 51 sec
9.011
8.549
8.536
7.260
7.158
7.146
3.001
2.956
2.937
2.917
1.605
1.587
1.568
1.550
1.530
1.343
1.323
1.229
0.867
0.851
0.833
9.1 8.9 8.7 ppm
7.36 7.22 ppm
ppm 3.00 ppm
1.60 ppm
1.3 1.1 ppm
0.97
1.01
0.96
3.00
2.01
2.25
0.03
21.40
3.08
C11H23
CO2Me
3da
10 9 8 7 6 5 4 3 2 1 ppm
0.97
1.01
0.96
3.00
2.01
2.25
21.40
3.08

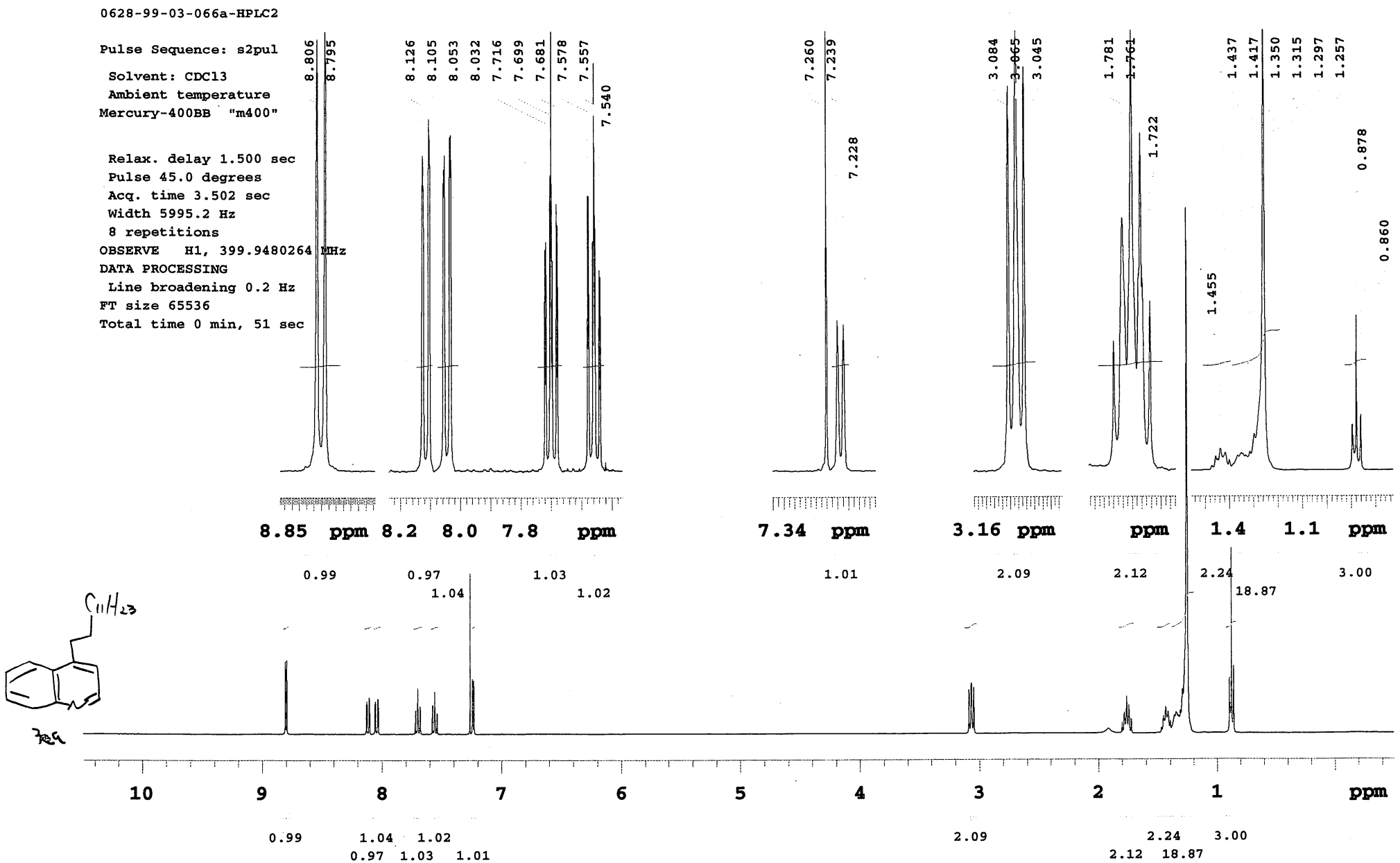
0430-99-03-088-b-column1-13CNMR

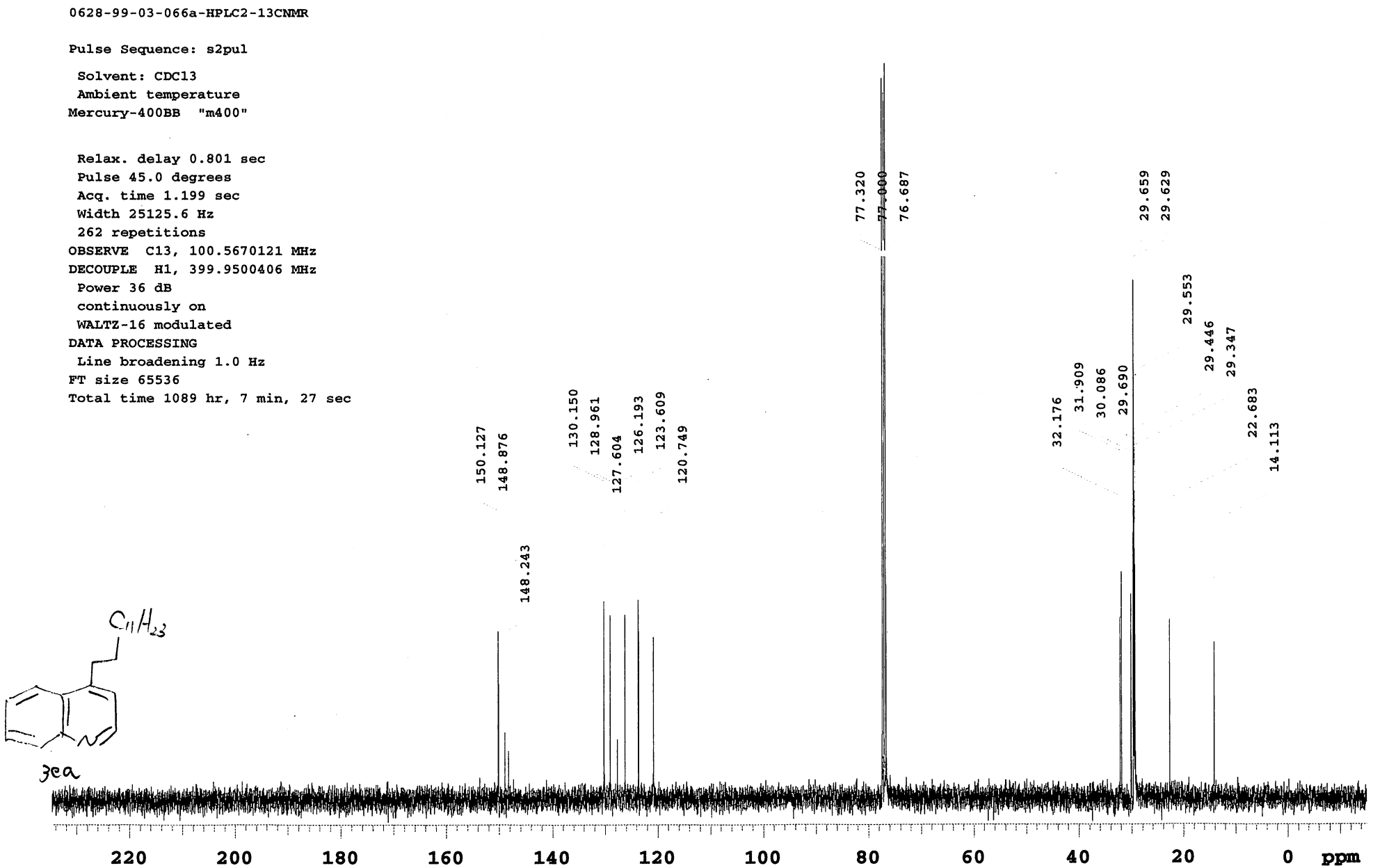
Pulse Sequence: s2pul

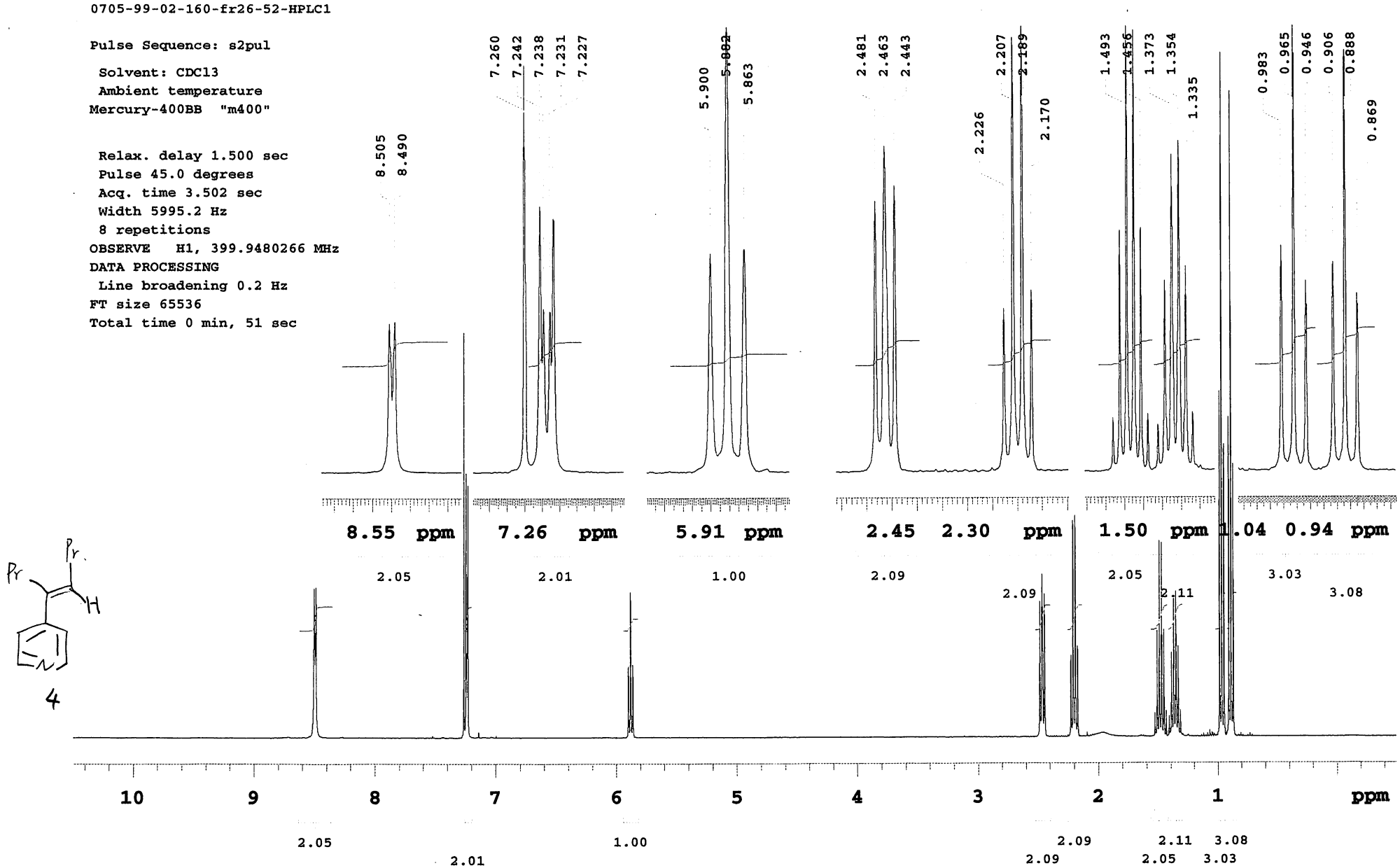
Solvent: CDCl3
Ambient temperature
File: 0430-99-03-088-b-column1-13CNMR
Mercury-400BB "m400"

Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
68 repetitions
OBSERVE C13, 100.5670151 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 108 hr, 54 min, 44 sec




0628-99-03-066a-HPLC2
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480264 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.806
8.795
8.126
8.105
8.053
8.032
7.716
7.699
7.681
7.578
7.557
7.540
7.260
7.239
7.228
3.084
3.065
3.045
1.781
1.761
1.722
1.455
1.437
1.417
1.350
1.315
1.297
1.257
0.878
0.860
8.85 ppm
8.2
8.0
7.8
ppm
7.34 ppm
3.16 ppm
ppm
1.4
1.1
ppm
0.99
0.97
1.04
1.03
1.02
1.01
2.09
2.12
2.24
18.87
3.00
C11H23
10
9
8
7
6
5
4
3
2
1
ppm
0.99
1.04
1.02
0.97
1.03
1.01
2.09
2.24
3.00
2.12
18.87


0628-99-03-066a-HPLC2-13CNMR
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
262 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1089 hr, 7 min, 27 sec
150.127
148.876
148.243
130.150
128.961
127.604
126.193
123.609
120.749
77.320
77.000
76.687
32.176
31.909
30.086
29.690
29.659
29.629
29.553
29.446
29.347
22.683
14.113
C11H23
3ea
220
200
180
160
140
120
100
80
60
40
20
0
ppm

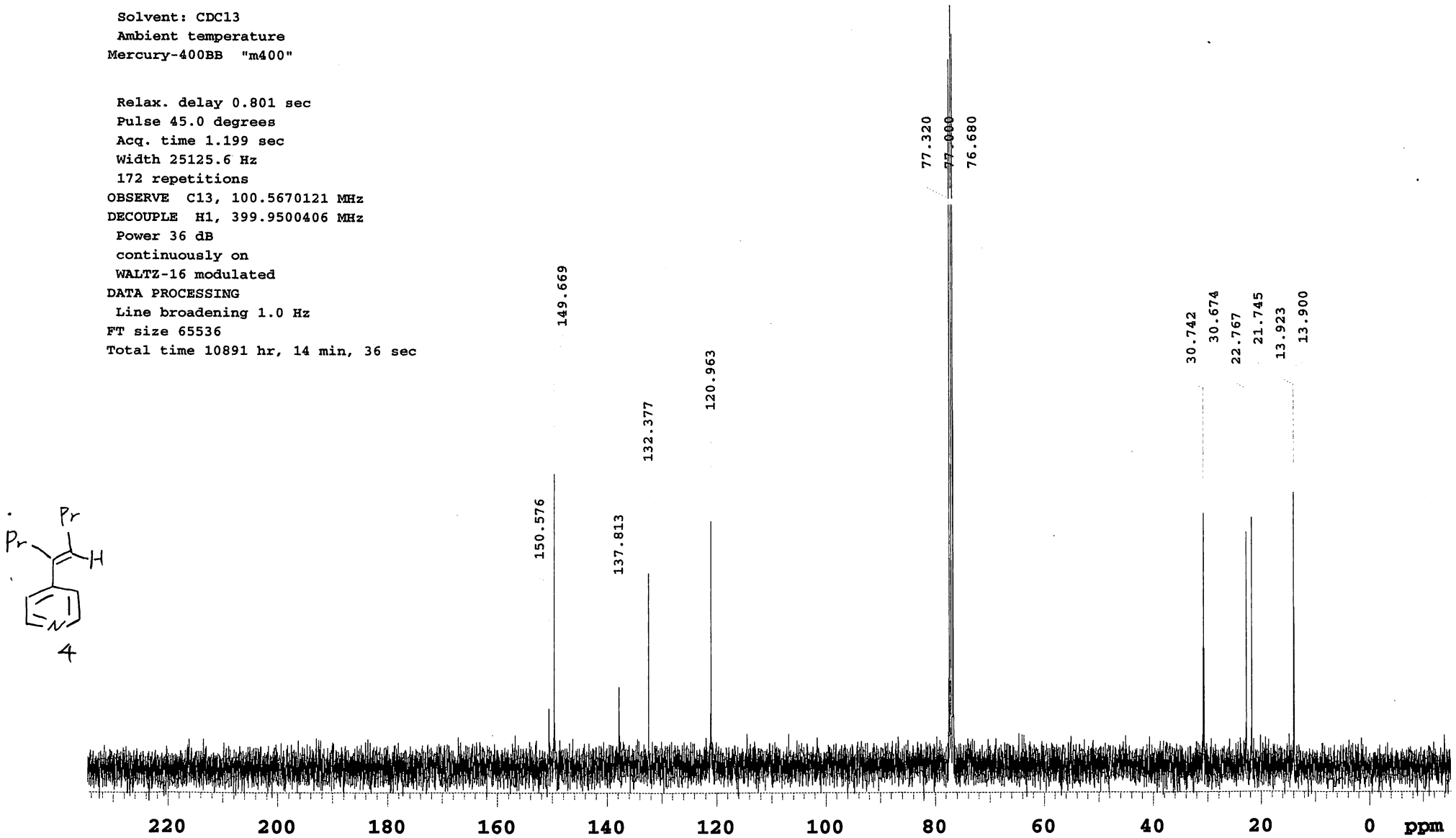

0705-99-02-160-fr26-52-HPLC1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.505
8.490
7.260
7.242
7.238
7.231
7.227
5.900
5.882
5.863
2.481
2.463
2.443
2.226
2.207
2.189
2.170
1.493
1.456
1.373
1.354
1.335
0.983
0.965
0.946
0.906
0.888
0.869
8.55 ppm
7.26 ppm
5.91 ppm
2.45 2.30 ppm
1.50 ppm
1.04 0.94 ppm
2.05
2.01
1.00
2.09
2.09
2.05
2.11
3.03
3.08
Pr
Pr
H
4
10
9
8
7
6
5
4
3
2
1
ppm
2.05
2.01
1.00
2.09
2.09
2.05
2.11
3.03
3.08

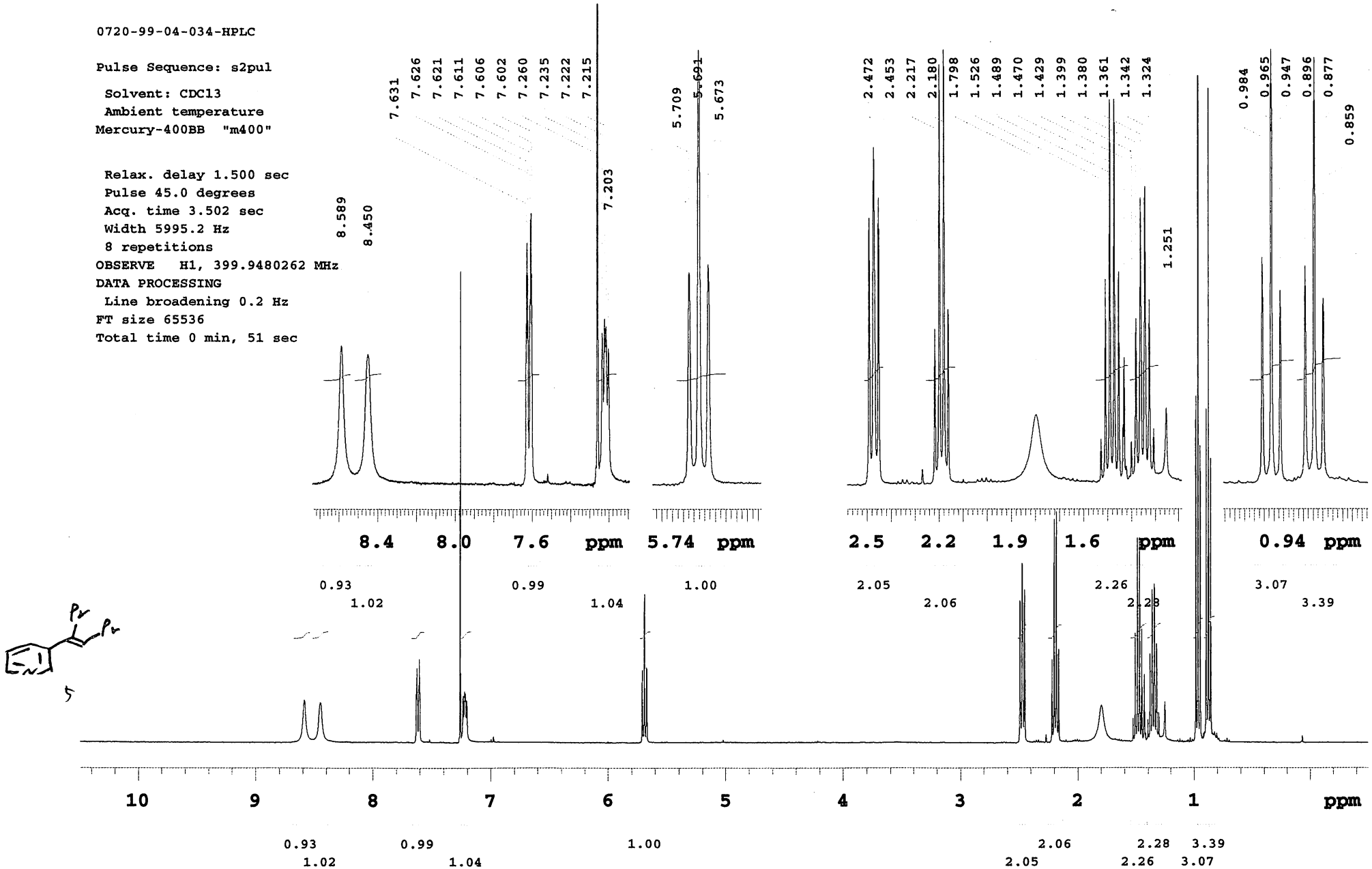
0212-99-02-160-fr26-52

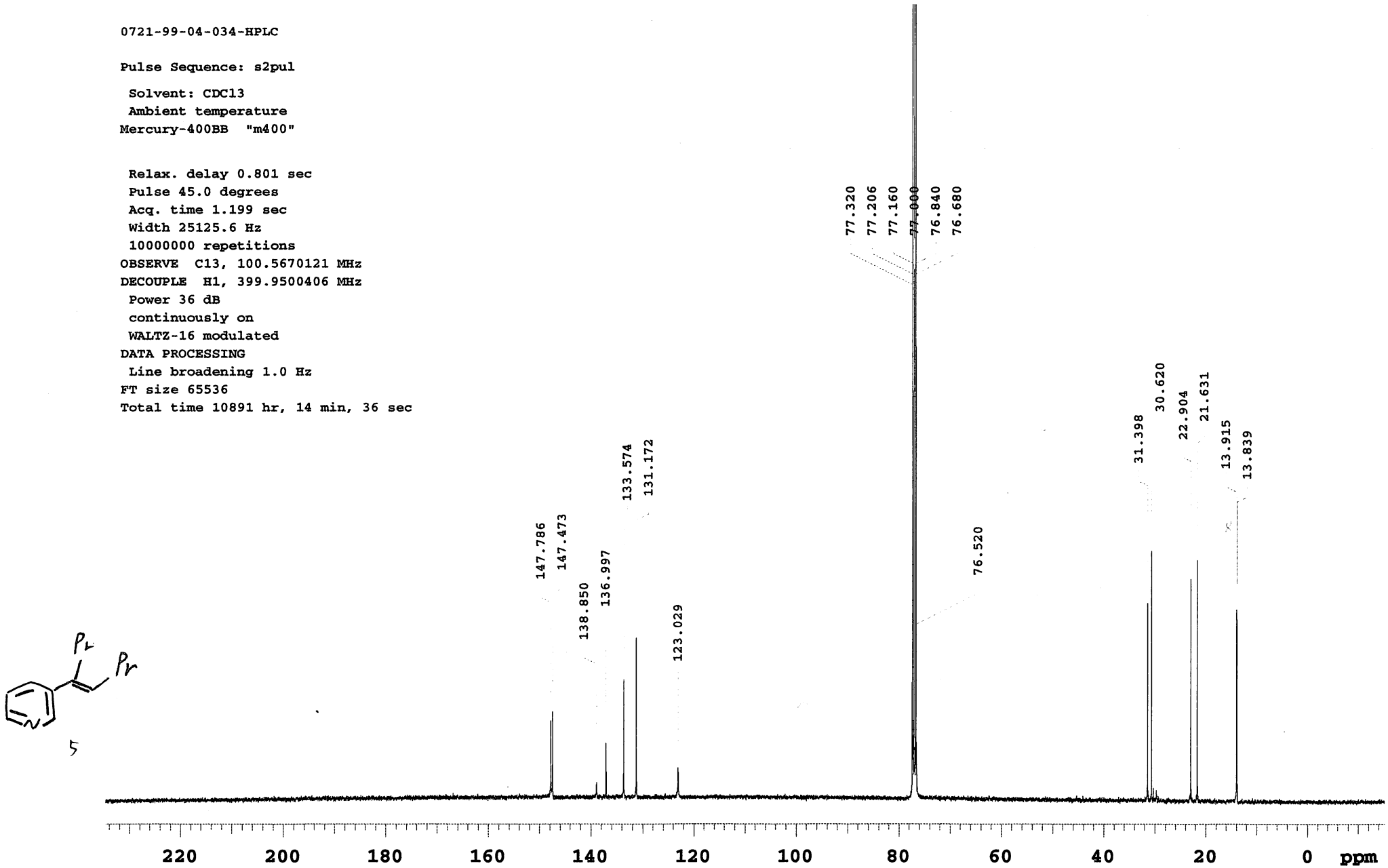
Pulse Sequence: s2pul

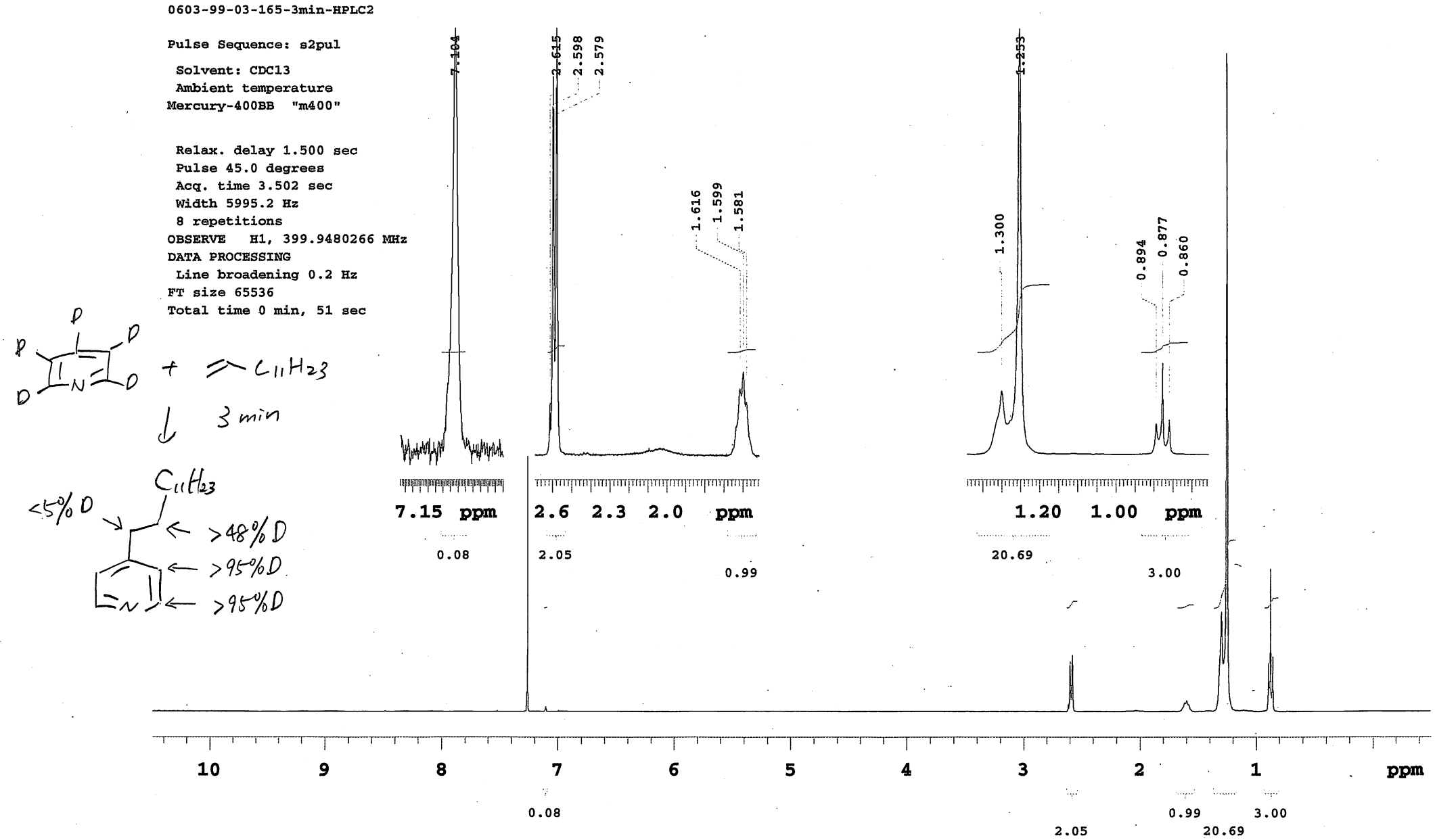
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"

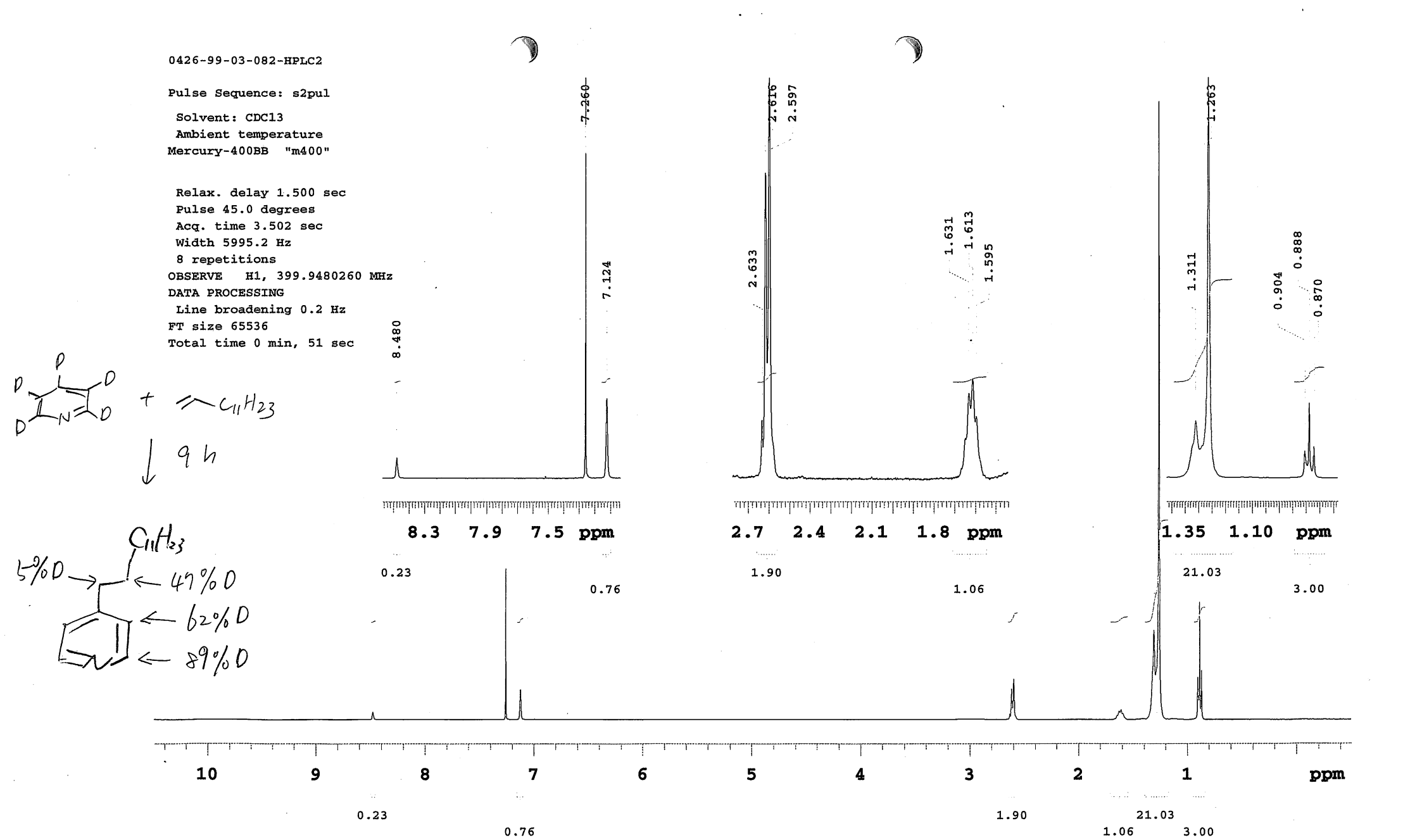
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
172 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec

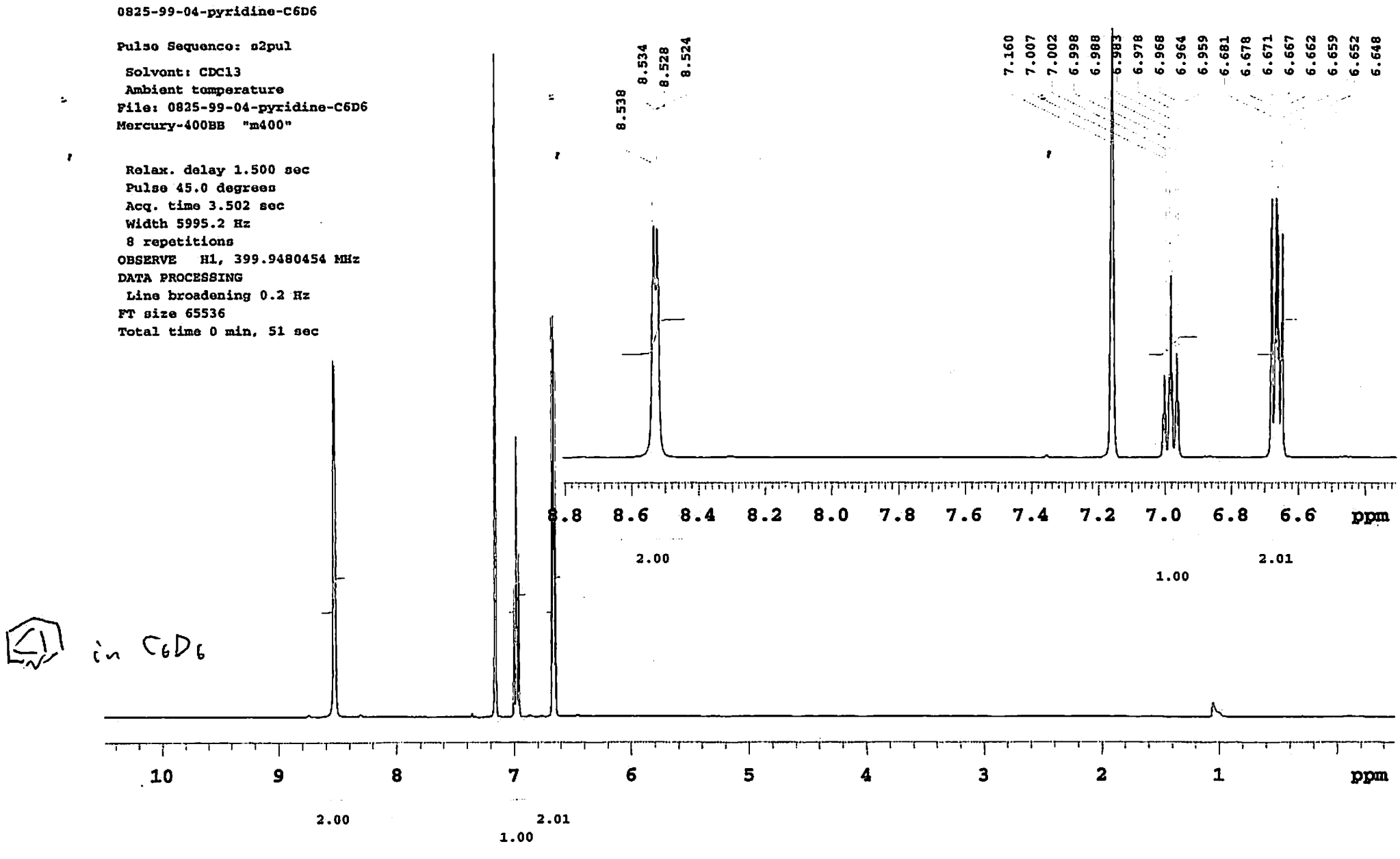


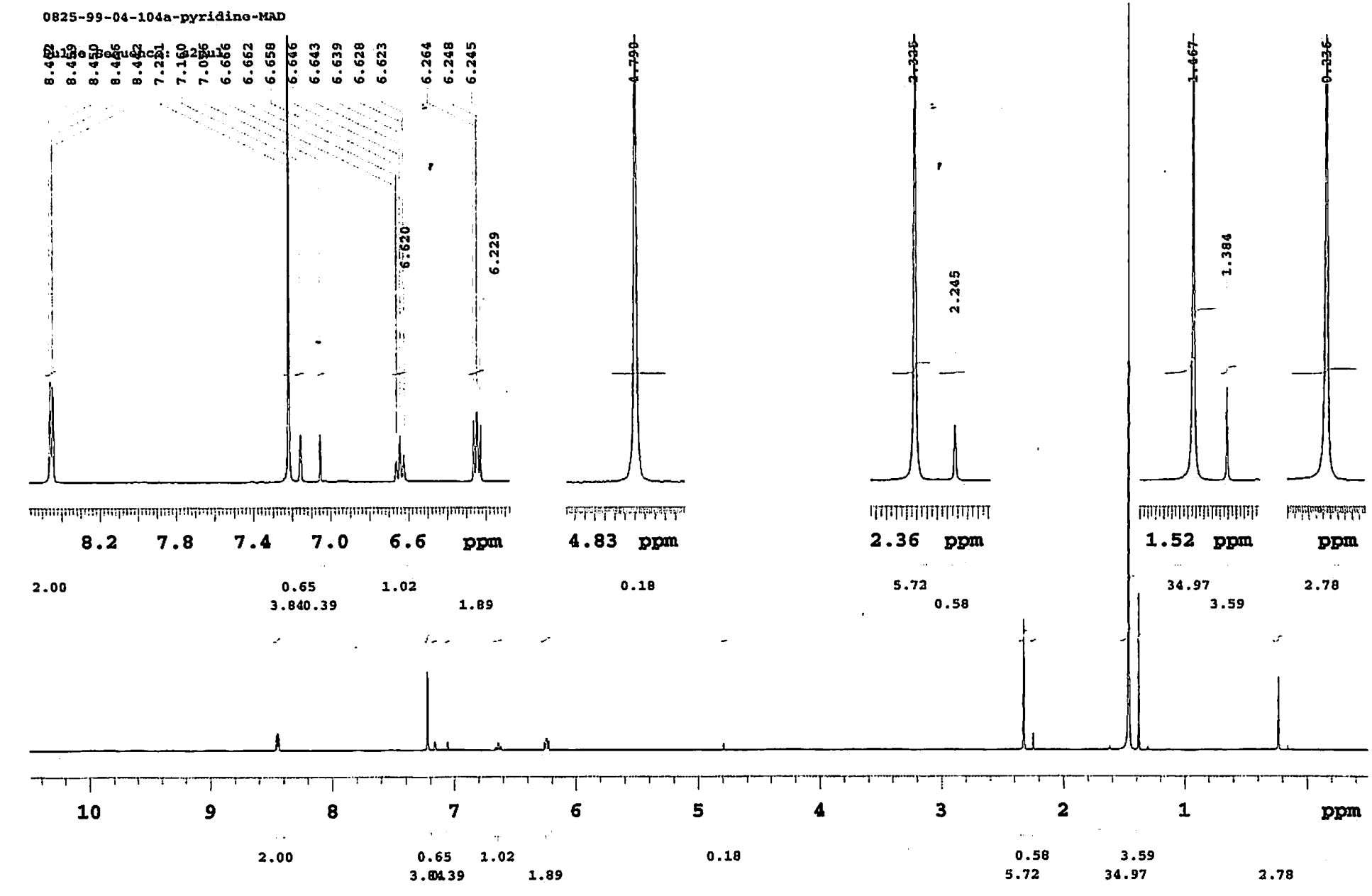

0720-99-04-034-HPLC
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480262 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.589
8.450
7.631
7.626
7.621
7.611
7.606
7.602
7.260
7.235
7.222
7.215
7.203
5.709
5.691
5.673
2.472
2.453
2.217
2.180
1.798
1.526
1.489
1.470
1.429
1.399
1.380
1.361
1.342
1.324
1.251
0.984
0.965
0.947
0.896
0.877
0.859
8.4
8.0
7.6
ppm
5.74
ppm
2.5
2.2
1.9
1.6
ppm
0.94
ppm
0.93
1.02
0.99
1.04
1.00
2.05
2.06
2.26
2.28
3.07
3.39
10
9
8
7
6
5
4
3
2
1
ppm
0.93
1.02
0.99
1.04
1.00
2.05
2.06
2.26
2.28
3.07
3.39
Pr
Pr
5

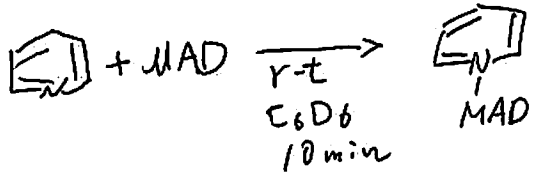

0721-99-04-034-HPLC
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 0.801 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25125.6 Hz
10000000 repetitions
OBSERVE C13, 100.5670121 MHz
DECOUPLE H1, 399.9500406 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10891 hr, 14 min, 36 sec
77.320
77.206
77.160
77.000
76.840
76.680
147.786
147.473
138.850
136.997
133.574
131.172
123.029
76.520
31.398
30.620
22.904
21.631
13.915
13.839
Pr
Pr
5
220
200
180
160
140
120
100
80
60
40
20
0
ppm

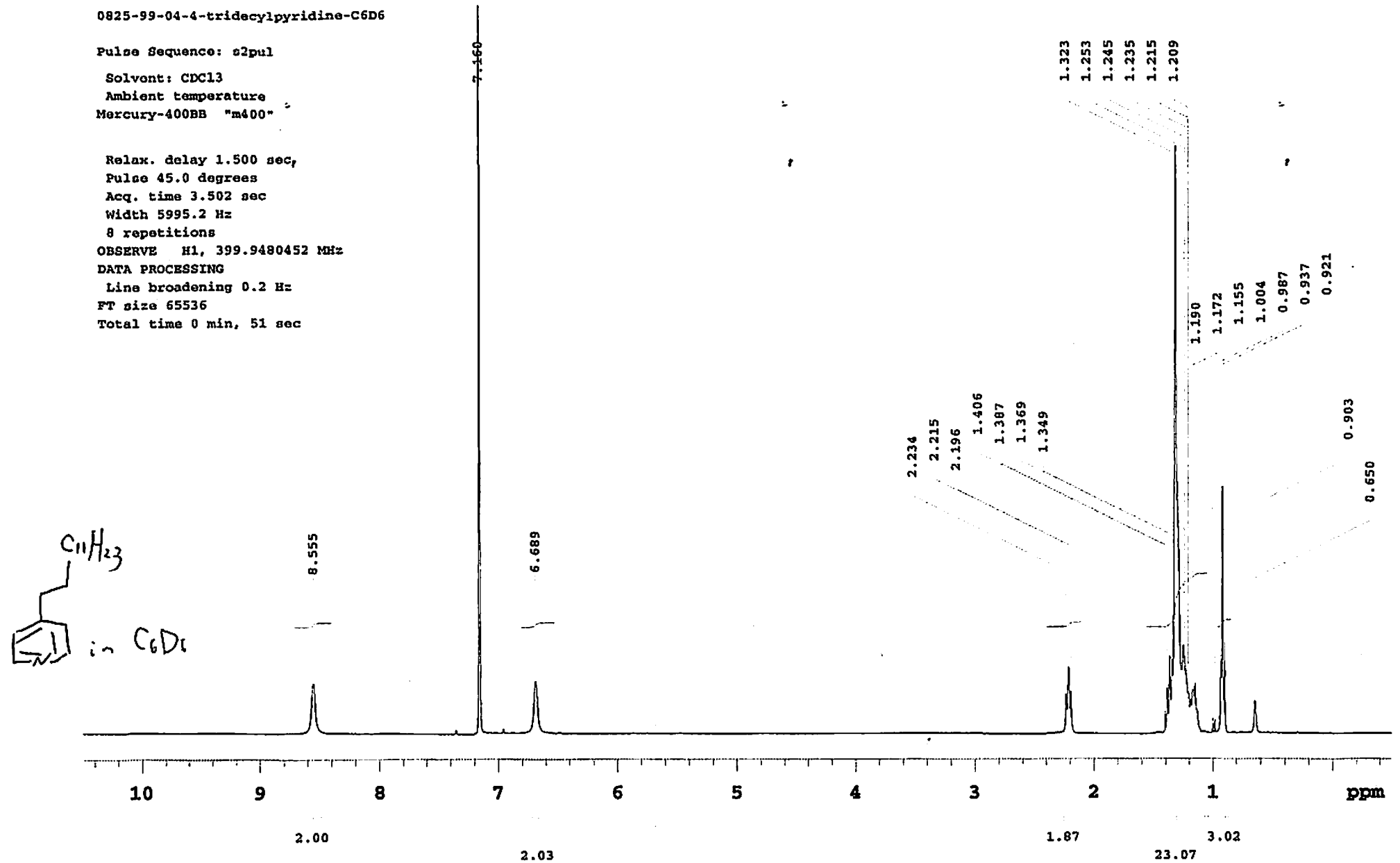

0603-99-03-165-3min-HPLC2
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
D
D
D
D
N
+
C11H23
3 min
C11H23
<5% D
>48% D
>95% D
>95% D
N
7.104
2.615
2.598
2.579
1.616
1.599
1.581
1.300
1.253
0.894
0.877
0.860
7.15 ppm
2.6 2.3 2.0 ppm
1.20 1.00 ppm
0.08
2.05
0.99
20.69
3.00
10 9 8 7 6 5 4 3 2 1 ppm
0.08
2.05
0.99
20.69
3.00

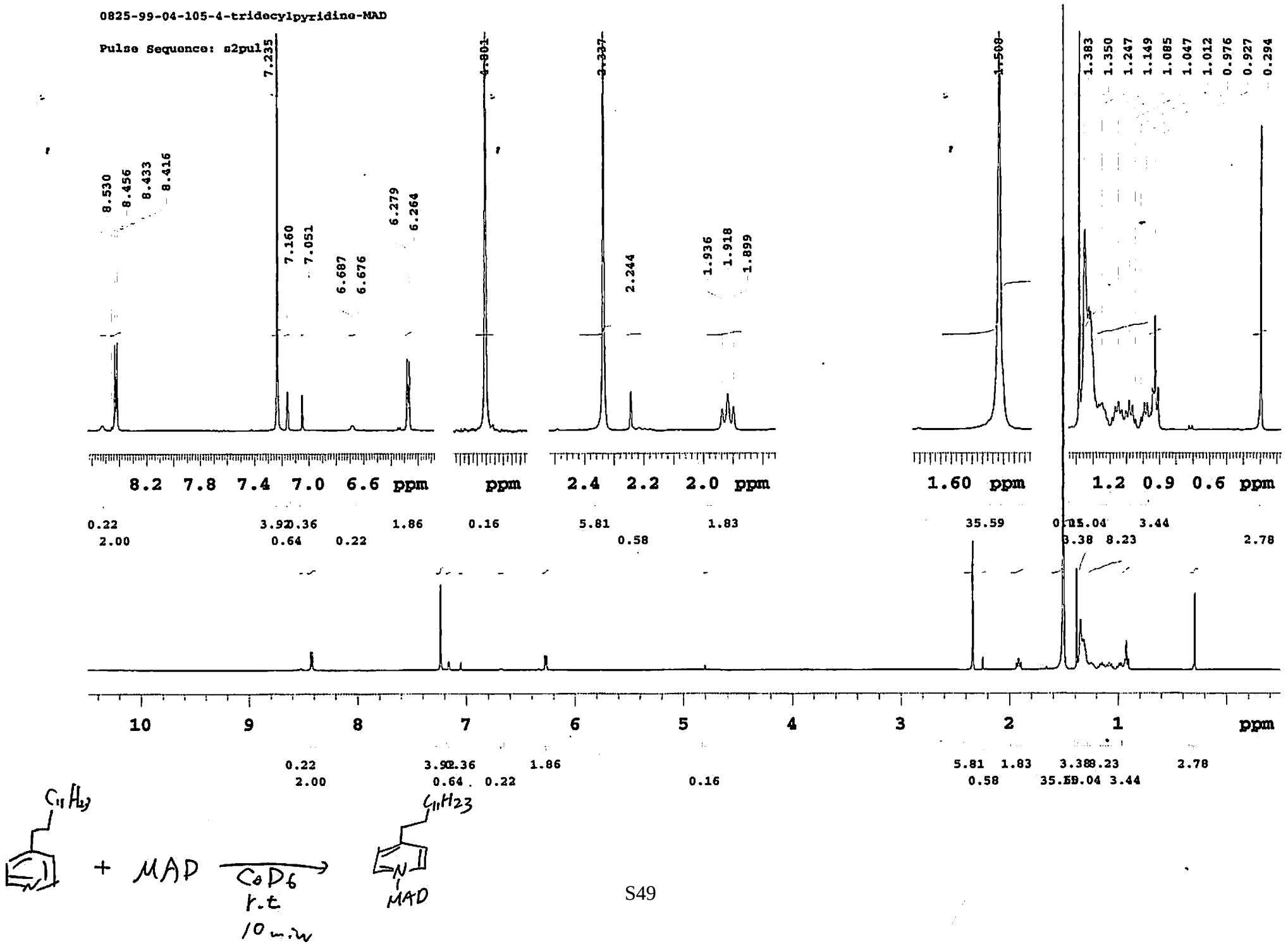

0426-99-03-082-HPLC2
Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480260 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
+ C11H23
9 h
C11H23
5% D
47% D
62% D
89% D
8.480
7.260
7.124
2.633
2.616
2.597
1.631
1.613
1.595
1.311
1.263
0.904
0.888
0.870
8.3 7.9 7.5 ppm
2.7 2.4 2.1 1.8 ppm
1.35 1.10 ppm
0.23
0.76
1.90
1.06
21.03
3.00
10 9 8 7 6 5 4 3 2 1 ppm

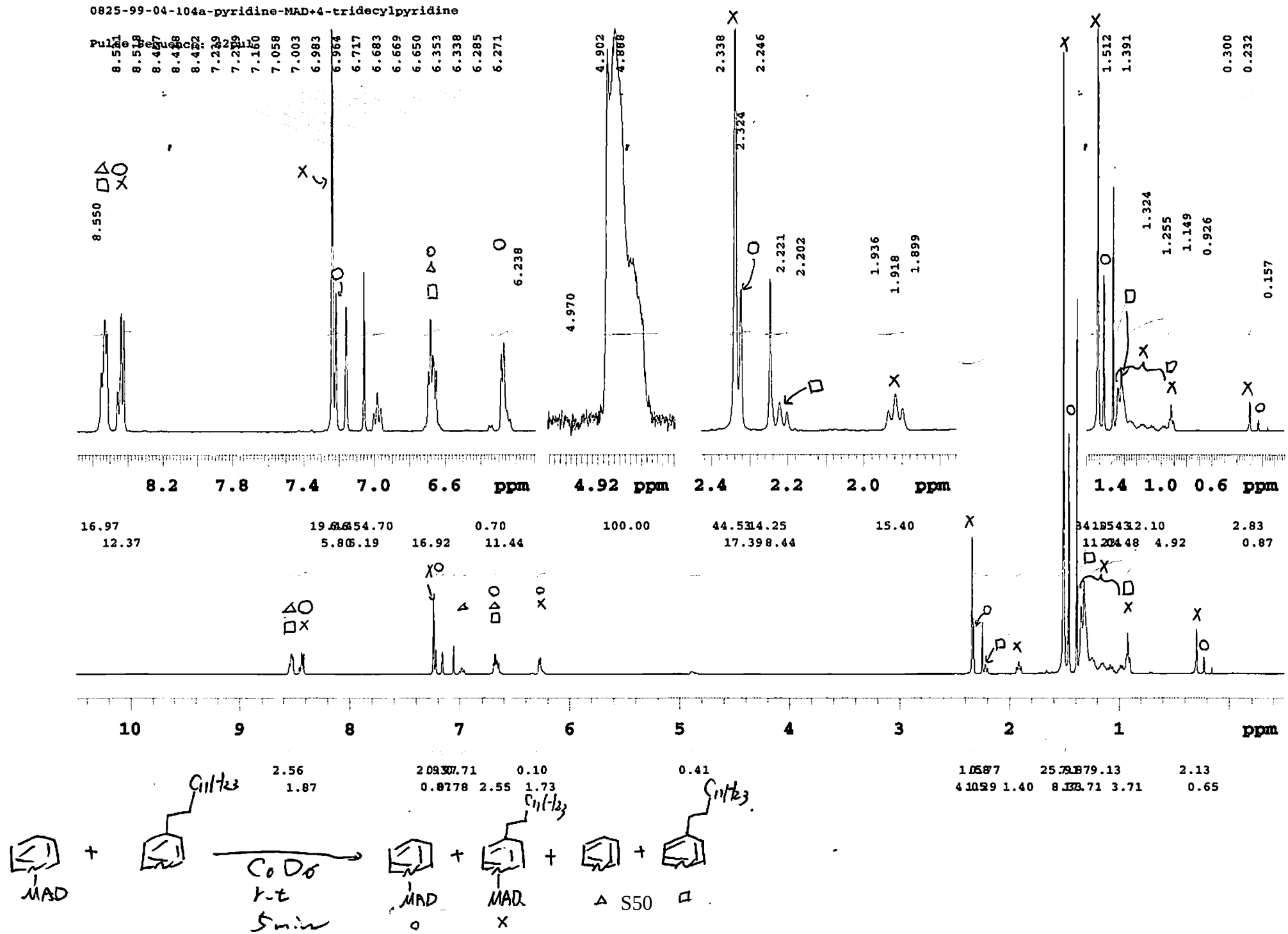

0825-99-04-pyridine-C6D6
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 0825-99-04-pyridine-C6D6
Mercury-400BB "m400"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480454 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
8.538
8.534
8.528
8.524
7.160
7.007
7.002
6.998
6.988
6.983
6.978
6.968
6.964
6.959
6.681
6.678
6.671
6.667
6.662
6.659
6.652
6.648
ppm
2.00
1.00
2.01
in C6D6

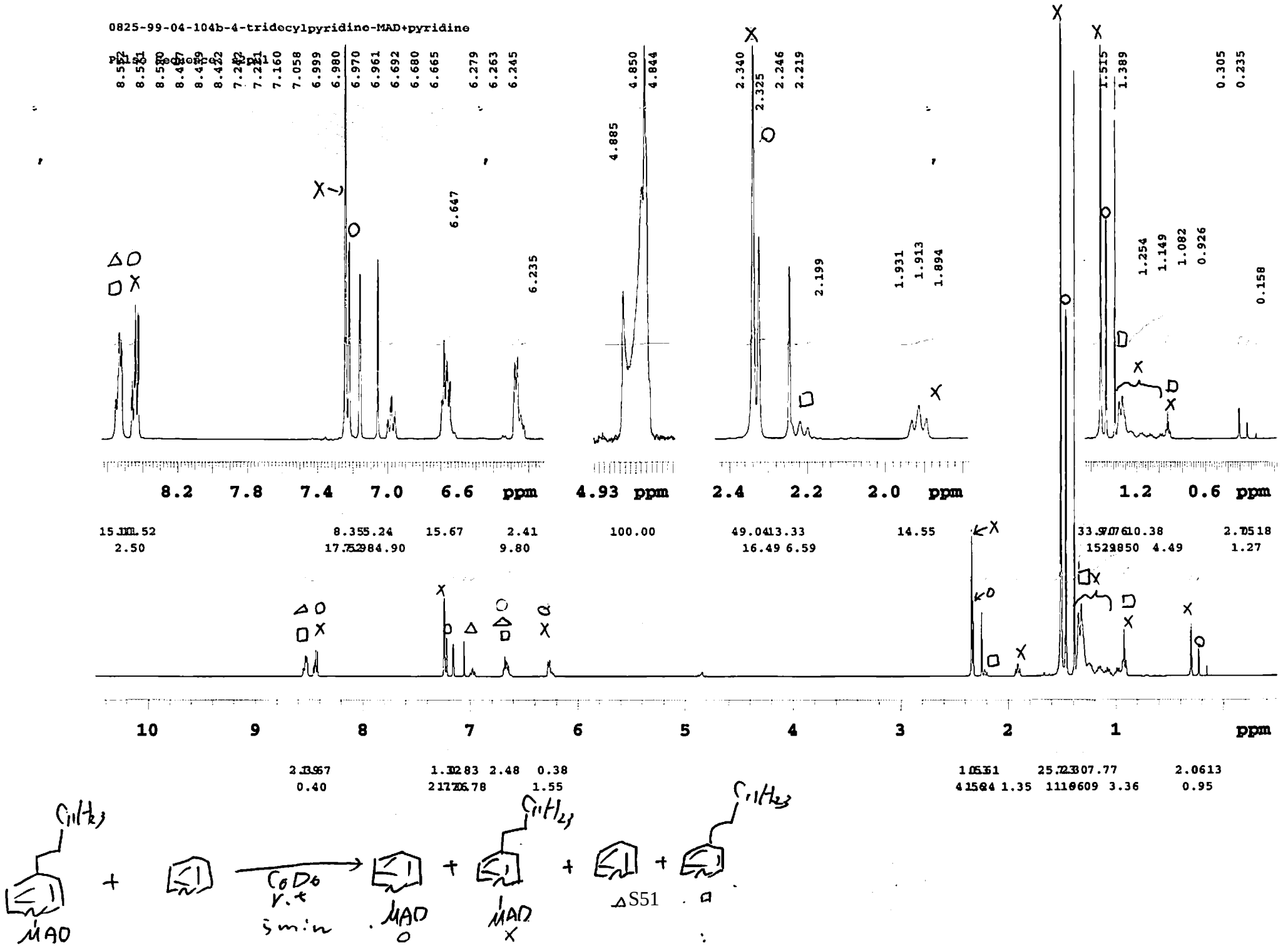

0825-99-04-104a-pyridine-MAD
8.482
8.459
8.450
8.442
7.221
7.160
7.075
6.666
6.662
6.658
6.646
6.643
6.639
6.628
6.623
6.264
6.248
6.245
6.620
6.229
4.790
2.335
2.245
1.467
1.384
0.336
8.2 7.8 7.4 7.0 6.6 ppm
4.83 ppm
2.36 ppm
1.52 ppm
ppm
2.00 0.65 1.02 0.18 5.72 0.58 34.97 3.59 2.78
3.84 0.39 1.89
10 9 8 7 6 5 4 3 2 1 ppm
2.00 0.65 1.02 0.18 0.58 3.59
3.84 0.39 1.89 5.72 34.97 2.78


+ MAD
r-t
C6D6
10 min
MAD


0825-99-04-4-tridecylpyridine-C6D6
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "m400"
Relax. delay 1.500 sec,
Pulse 45.0 degrees
Acq. time 3.502 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 399.9480452 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min, 51 sec
7.160
8.555
6.689
2.234
2.215
2.196
1.406
1.387
1.369
1.349
1.323
1.253
1.245
1.235
1.215
1.209
1.190
1.172
1.155
1.004
0.987
0.937
0.921
0.903
0.650
C11H23
in C6D6
10
9
8
7
6
5
4
3
2
1
ppm
2.00
2.03
1.87
23.07
3.02


0825-99-04-105-4-tridecylpyridine-NAD
Pulse Sequence: s2pul
8.530
8.456
8.433
8.416
7.235
7.160
7.051
6.687
6.676
6.279
6.264
4.801
3.337
2.244
1.936
1.918
1.899
1.508
1.383
1.350
1.247
1.149
1.085
1.047
1.012
0.976
0.927
0.294
8.2 7.8 7.4 7.0 6.6 ppm
ppm
2.4 2.2 2.0 ppm
1.60 ppm
1.2 0.9 0.6 ppm
10 9 8 7 6 5 4 3 2 1 ppm
+ MAD
C6D6
r.t
10 min
MAD


0825-99-04-104a-pyridine-MAD+4-tridecylpyridine
Pulse Sequence: zg30
8.550
6.964
6.717
6.683
6.669
6.650
6.353
6.338
6.285
6.271
6.238
4.902
4.888
4.970
2.338
2.324
2.246
2.221
2.202
1.936
1.918
1.899
1.512
1.391
1.324
1.255
1.149
0.926
0.300
0.232
0.157
8.2 7.8 7.4 7.0 6.6 ppm
4.92 ppm
2.4 2.2 2.0 ppm
1.4 1.0 0.6 ppm
16.97 19.66 54.70 0.70 100.00 44.53 14.25 15.40 12.10 2.83
12.37 5.80 5.19 16.92 11.44 17.39 8.44 11.20 4.48 4.92 0.87
10 9 8 7 6 5 4 3 2 1 ppm
2.56 2.09 3.07 0.71 0.10 0.41 10.87 25.79 9.13 2.13
1.87 0.87 2.55 1.73 4.10 1.40 8.33 3.71 0.65
C11H23
MAD
CD3OD
r.t
5min
MAD
MAD
S50


0825-99-04-104b-4-tridecylpyridine-MAD+pyridine
8.2 7.8 7.4 7.0 6.6 ppm
4.93 ppm
2.4 2.2 2.0 ppm
1.2 0.6 ppm
10 9 8 7 6 5 4 3 2 1 ppm
MAD
C6D6
r.t
5min
MAD
O
MAD
X
ΔS51
a