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Photoacylation of Alcohols in Neutral Medium

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We report here conditions which allow the photoacylation of primary, secondary *and* tertiary alcohols with *N*-acetyl-5,7-dinitroindoline under exceptionally mild conditions, at wavelengths harmless to most functional groups, including otherwise photosensitive ones.

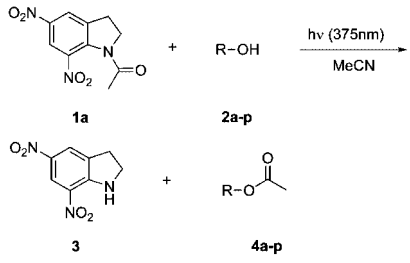
Introduction

The transformation of an alcohol into an ester is one of the most important reactions in organic synthesis, as attested by the countless preparative methods published to date.[1,2] However, in many instances, the existing methods require aggressive reagents (acid chlorides, strong bases or acids, activated alkylating agents) and are incompatible with sensitive functional groups. From this viewpoint, the photoinduced acylation of oxygen nucleophiles would represent an attractive alternative. While similar reactions have been successfully developed for the formation of amides and carbamates,[3–6] esters have proven to be more problematic targets, with the notable exception of methyl esters (when methanol is used as the solvent).[7] We report here conditions which allow the photoacylation of primary, secondary *and* tertiary alcohols with *N*-acetyl-5,7-dinitroindoline under exceptionally mild conditions, at wavelengths harmless to most functional groups, including otherwise photosensitive ones.

Results and Discussion

Our initial experiments, in which we used typical conditions that are suitable for the formation of amides (1 equiv. each of the nucleophile and acetyldinitroindoline in acetonitrile, irradiation at 350 nm), were unsuccessful and led only to deacetylated indoline, acetic acid and unreacted alcohol. Better conversion could only be obtained by using 3 equiv. of the acylating agent. These observations suggested that the photolysis irreversibly produced a species that rapidly decomposed to acetic acid and deacetylated indoline with trace amounts of moisture, if no powerful nucleophile (such as an amine) was available to react fast enough. A mechanistic study by time-resolved IR spectroscopy indeed showed a transient species with a lifetime of ca. 20 μs.[8,9]

We thus reasoned that anything capable of accelerating the bimolecular process required to form the desired ester would be beneficial. The addition of acyl transfer catalysts (such as *N*,*N*-4-dimethylaminopyridine) indeed increased the yields (from 22% to 56%). It was finally by increasing the concentration of the reactants, despite reaching saturation of the *N*-acetyl-5,7-dinitroindoline, that consistently good yields were obtained. Hence, irradiation of a 0.1 M solution of *N*-acetyl-5,7-dinitroindoline with 1 equiv. of an alcohol in strictly anhydrous acetonitrile at 375 nm for 16 h gave the corresponding acetate in good to excellent yields (Scheme 1, Table 1).



Scheme 1. Acetylation of alcohols with indoline **1a**.

The operating wavelength could be increased up to 405 nm, a wavelength where even very UV-photosensitive 3,5-dimethoxybenzoin ester **4p** is stable, without affecting the yields. This adds an interesting facet to our strategy of exploiting chromatic orthogonality[10] because both the esterification and the hydrolysis of **4p** can be carried out photochemically without the need for any external reagent.

The reaction is compatible with the presence of acetyl groups (Entry 13), conjugated and unconjugated alkenes alkynes (Entry 12), and functionalised phenols also react satisfactorily (Entry 7).

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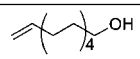
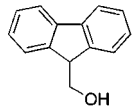
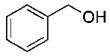
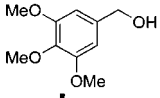
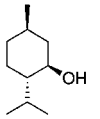
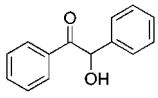
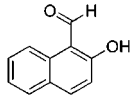
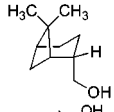
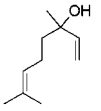
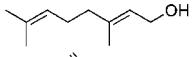
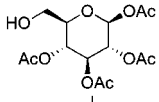
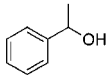
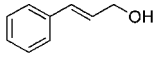
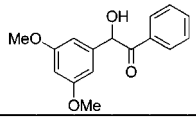
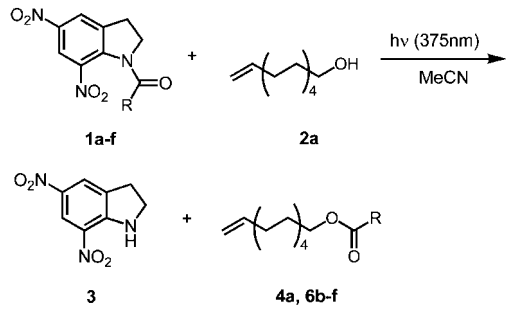
Table 1. Acetylation of alcohols with indoline **1a**.

Entry		Alcohol	Yield of **4**[a] [%]	Yield of **4**[b] [%]
1	**2a**		96	81
2	**2b**		76	63
3	**2c**		98	77
4	**2d**		95	83
5	**2e**		81	66
6	**2f**		76	67
7	**2g**		61	51
8	**2h**		90	72
9	**2i**		83	70
10	**2j**		57	47
11	**2k**		83	72
12	**2l**		70	64
13	**2m**		80	74
14	**2n**		77	69
15	**2o**		83	73
16	**2p**		76	68

[a] Determined in situ by ^{1}H NMR spectroscopy. [b] Isolated.

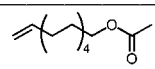
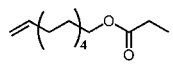
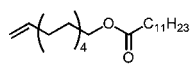
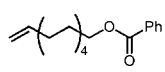
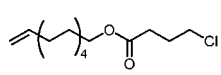
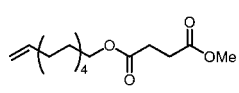
Other groups than simple acetyl can be transferred photochemically. To verify this, we successfully acylated 10-undecen-1-ol with a series of acylnitroindoles (**1a**–**f**), which were prepared in prior work[5] (Scheme 2, Table 2).

We emphasise the simplicity of the experiments, which can be carried out on a normal bench, without the need



Scheme 2. Acylation of alcohols with indolines **1a**–**f**.

Table 2. Acylation of alcohols with indolines **1a**–**f**.

Entry[a]	Indoline	R	Product	Yield[b] [%]	Yield[c] [%]
1	**1a**	CH_3	**4a** 	96	81
2	**1b**	CH_2CH_3	**6b** 	90	82
3	**1c**	$C_{11}H_{23}$	**6c** 	99	94
4	**1d**	C_6H_5	**6d** 	98	89
5	**1e**	$(CH_2)_3Cl$	**6e** 	80	74
6	**1f**	CH_2CH_2COOMe	**6f** 	83	78

[a] Conditions: 0.1 M solution in 1 mL of anhydrous acetonitrile for 16 h, mol ratio of *N*-acyl-5,7-dinitroindoline/alcohol is 1:1. [b] Determined by ^{1}H NMR spectroscopy. [c] Isolated yield.

of a mercury lamp, by using a small footprint LED-based photoreactor (Atlas Photonics Inc.) and quartz test tubes, under conventional magnetic stirring.

In conclusion, we extended the use of *N*-acyl-5,7-dinitrondoline for the photoacylation of alcohols with particularly mild conditions and a simple experimental protocol.

Experimental Section

General Methods: ^{1}H- and ^{13}C-NMR spectra were recorded with a Fourier transform Bruker-DRX-500 (500 MHz) or Bruker-DPX-360 (360 MHz) spectrometer with solvent used as a reference. For ^{13}C NMR, the number of hydrogen was determined by a DEPT sequence. IR spectra were recorded with a Fourier transform Mattson 5000 FTIR spectrometer, neat, in $CHCl_3$ (NaCl cell) or in KBr; absorption bands are in cm^{-1}. UV spectra were recorded with a Perkin Elmer Lambda 40 spectrometer; absorption bands are in nm. EI mass spectra were recorded with an HP 5988A Quadrupol spectrometer, with electron impact (70 eV) and ESI mass spectra with a Bruker FT/MS 4.7 T BioApex II spectrometer. Photochemical irradiations were carried out in a LUMOS 43 photoreactor (Atlas Photonics Inc.), in a quartz vessel, with 1 diode at 375, 385,

405 or 430 nm, or in a Srinivasan-Griffin (Rayonet-RPR-100) photoreactor, in a quartz vessel, with 16 lamps at 254, 300, 350 or 420 nm. Unless otherwise indicated, all commercial reagents were used without further purification.

Typical Procedure for the Preparation of 1-Acyl-5,7-dinitroindolines: A solution of 5,7-dinitroindoline (43.0 mg, 0.21 mmol),[6] aluminium trichloride (68.5 mg, 0.51 mmol) and acid chloride (0.41 mmol) was heated at reflux in 1,2-dichloroethane (5 mL) for 2 to 6 h. Extraction, followed by trituration in cyclohexane to remove the remaining acid chloride (if not volatile), gave the desired 1-acyl-5,7-dinitroindoline pure or with remaining 5,7-dinitroindoline.

1-Propionyl-5,7-dinitroindoline (1b): Pale brown solid (m.p. 144–147 °C). ^{1}H NMR (360 MHz, $CDCl_3$): δ = 8.58 (s, 1 H), 8.26 (s, 1 H), 4.37 (t, J = 8.3 Hz, 2 H), 3.38 (t, J = 8.2 Hz, 2 H), 2.56 (q, J = 7.3 Hz, 2 H), 1.26 (t, J = 7.4 Hz, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 172.3 (C), 143.1 (C), 139.8 (C), 139.1 (C), 138.1 (C), 123.0 (CH), 119.8 (CH), 49.9 (CH_2), 29.0 (CH_2), 29.0 (CH_2), 8.7 (CH_3) ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 3024, 1697, 1608, 1548, 1462, 1382, 1342, 1280 cm^{-1}. UV/Vis (51 μM soln in MeCN) λ (ε, M^{-1}cm^{-1}): 204 (14310), 226 (13140), 353 (11180) nm. MS: m/z (%) = 265 (2) $[M]^+$, 209 (90), 163 (7), 117 (9), 89 (8), 57 (100). HRMS: calcd. for $C_{11}H_{11}N_3O_5$ 265.0699; found 265.0674.

1-Lauroyl-5,7-dinitroindoline (1c): Very pale brown solid, purified from remaining 5,7-dinitroindoline by chromatography (m.p. 105–108 °C). ^{1}H NMR (360 MHz, $CDCl_3$): δ = 8.58 (s, 1 H), 8.26 (s, 1 H), 4.37 (t, J = 8.3 Hz, 2 H), 3.37 (t, J = 8.2 Hz, 2 H), 2.51 (t, J = 7.4 Hz, 2 H), 1.74 (quint, J = 7.5 Hz, 2 H), 1.4–1.2 (m, 16 H), 0.88 (t, J = 6.8 Hz, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.8 (C), 143.2(C), 139.8 (C), 139.2 (C), 138.0 (C), 123.0 (CH), 119.9 (CH), 50.0 (CH_2), 35.7 (CH_2), 31.9(CH_2), 29.5 (CH_2), 29.4 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 29.1 (CH_2), 28.5 (CH_2), 24.6 (CH_2), 22.6 (CH_2), 14.1 (CH_3) ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 2927, 2855, 1695, 1548, 1464, 1374, 1344, 1280 cm^{-1}. UV/Vis (51 μM soln in MeCN): λ (ε, M^{-1}cm^{-1}) = 226 (18240), 349 (13922) nm. MS: m/z (%) = 209 (96), 183 (18), 179 (18), 163 (5), 117 (8), 95 (13), 85 (23), 71 (38), 57 (100). HRMS: calcd. for $C_8H_7N_3O_4$, $C_{12}H_{23}O$ 209.0437, 183.1749; found 209.0437, 183.1749.

1-Benzoyl-5,7-dinitroindoline (1d): Yellow crystals, purified from remaining 5,7-dinitroindoline by recrystallisation (toluene/ethanol, 1:1) (m.p. 193–196 °C). ^{1}H NMR (360 MHz, $CDCl_3$): δ = 8.69 (s, 1 H), 8.31 (s, 1 H), 7.76 (d, J = 7.4 Hz, 2 H), 7.61 (t, J = 7.6 Hz, 1 H), 7.51 (t, J = 7.7 Hz, 2 H), 4.39 (t, J = 8.2 Hz, 2 H), 3.33 (t, J = 8.2 Hz, 2 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 169.9 (C), 143.7 (C), 141.3 (C), 139.2 (C), 138.8 (C), 133.3 (C), 132.8 (CH), 128.8 (CH), 128.8 (CH), 123.5 (CH), 120.0 (CH), 53.7 (CH_2), 29.1 (CH_2) ppm. IR (KBr): $\tilde{\nu}$ = 3100, 1661, 1600, 1545, 1528, 1439, 1372, 1338, 1306 cm^{-1}. UV (51 μM soln in MeCN): λ (ε, M^{-1}cm^{-1}) = 229 (19020), 353 (12550) nm. MS: m/z (%) = 313 (1) $[M]^+$, 105 (100), 77 (31). HRMS: calcd. for $C_{15}H_{11}N_3O_5$ 313.0699; found 313.0701.

1-(4-Chlorobutyryl)-5,7-dinitroindoline (1e): Yellow oil, purified from remaining 5,7-dinitroindoline by chromatography. ^{1}H NMR (360 MHz, $CDCl_3$): δ = 8.59 (s, 1 H), 8.28 (s, 1 H), 4.42 (t, J = 8.4 Hz, 2 H), 3.69 (t, J = 6.0 Hz, 2 H), 3.40 (t, J = 8.4 Hz, 2 H), 2.75 (t, J = 6.8 Hz, 2 H), 2.32 (quint, J = 6.4 Hz, 2 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.7 (C), 143.5 (C), 139.6 (C), 139.3 (C), 138.1 (C), 123.2 (CH), 120.0 (CH), 50.0 (CH_2), 44.2 (CH_2), 32.1 (CH_2), 28.6 (CH_2), 27.3 (CH_2) ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 3022, 1694, 1609, 1548, 1438, 1391, 1344, 1280 cm^{-1}. UV/Vis (48 μM soln in MeCN): λ (ε, M^{-1}cm^{-1}) = 202 (17708), 225 (11250), 350 (8542) nm. MS: m/z (%) = 313 (1) $[M]^+$, 209 (100), 179 (10), 163 (9), 117 (16), 107 (19), 105 (60), 77 (36). HRMS: calcd. for $C_{12}H_{12}N_3O_5$ 313.0466; found 313.0480.

1-(3-Methoxycarbonylpropionyl)-5,7-dinitroindoline (1f): Yellow crystals, purified from remaining 5,7-dinitroindoline by recrystallisation (toluene/ethanol, 1:1) (m.p. 126–130 °C). ^{1}H NMR (360 MHz, $CDCl_3$): δ = 8.53 (s, 1 H), 8.25 (s, 1 H), 4.49 (t, J = 8.2 Hz, 2 H), 3.68 (s, 3 H), 3.40 (t, J = 8.2 Hz, 2 H), 2.84–2.74 (m, 4 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 172.9 (C), 170.6 (C), 143.3 (C), 139.6 (C), 139.1 (C), 138.4 (C), 123.1 (CH), 119.8 (CH), 52.0 (CH_3), 50.1 (CH_2), 30.2 (CH_2), 28.7 (CH_2), 28.5 (CH_2) ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 3027, 1733, 1697, 1612, 1548, 1439, 1370, 1344, 1283, 1162 cm^{-1}. MS: m/z (%) = 323 (1) $[M]^+$, 292 (8), 209 (12), 163 (3), 115 (100), 87 (14), 59 (24), 55 (57). HRMS: calcd. for $C_{13}H_{13}N_3O_7$ 323.0754; found 323.0744.

Typical Procedure for the Photoacylation of Alcohols to Esters: All experiments were performed in anhydrous acetonitrile (dried by passing it, under an argon atmosphere, through a Grubbs purification system).[11] A mixture of 1-acyl-5,7-dinitroindoline (25.1 mg, 0.1 mmol), the alcohol (0.1 mmol, 1 equiv.) in dry MeCN (1 mL) was irradiated at 375 nm in a quartz tube for 16 h, under an argon atmosphere, with vigorous stirring. The reaction mixture was then concentrated under reduced pressure, and the yield of the ester was then estimated by ^{1}H NMR spectroscopy. 5,7-Dinitroindoline is insoluble in hexane and was removed by trituration and filtration, unless specified otherwise. Evaporation of the hexane filtrate gave the ester and (when applicable) some unreacted alcohol.

Undecen-10-enyl Acetate (4a): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^{1}H NMR (360 MHz, $CDCl_3$): δ = 5.87–5.75 (m, 1 H), 5.01–4.91 (m, 2 H), 4.05 (t, J = 6.8 Hz, 2 H), 2.06–2.01 (m, 5 H), 1.64 (quint, J = 6.8 Hz, 2 H), 1.39–1.28 (m, 12 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.4 (C), 139.4 (CH), 114.3 (CH), 64.8 (CH_2) 34.0 (CH_2), 29.6 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 29.2 (CH_2), 29.0 (CH_2), 28.7 (CH_2), 26.0 (CH_2), 21.2 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3078, 2928, 2856, 1743, 1641, 1464, 1441, 1388, 1366, 1240, 1039, 995, 910 cm^{-1}. MS (ESI): m/z (%) = 235.2 (100) $[M + Na]^+$. HRMS: calcd for $C_{13}H_{24}O_2$ 212.1776; found 212.1772.

9-Fluorenylmethyl Acetate (4b): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.78 (d, J = 7.2 Hz, 2 H), 7.61 (d, J = 7.2 Hz, 2 H), 7.42 (dd, J = 7.2 Hz, 2 H), 7.33 (dd, J = 7.2 Hz, 2 H), 4.37 (d, J = 7.2 Hz, 2 H), 4.22 (t, J = 7.3 Hz, 1 H), 2.16 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.2 (C), 143.9 (C), 141.4 (C), 127.9 (CH), 127.2 (CH), 125.2 (CH), 120.2 (CH), 66.6 (CH_2), 46.9 (CH), 21.2 (CH_3) ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 3067, 3041, 3021, 2951, 1741, 1449, 1382, 1363, 1245, 1036, 759, 740 cm^{-1}. MS (ESI): m/z (%) = 261.1 (100) $[M + Na]^+$. HRMS: calcd. for $C_{16}H_{14}O_2$ 238.0994; found 238.0992.

Benzyl Acetate (4c): Purification by flash column chromatography [SiO_2, hexane/EtOAc (3:1)]. ^{1}H NMR (360 MHz, $CDCl_3$): δ = 7.40–7.30 (m, 5 H), 5.11 (s, 2 H), 2.11 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.1 (C), 136.1 (C), 128.7 (2×CH), –128.4 (3×CH), 66.5 (CH_2), 21.2 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3066, 3035, 2956, 1743, 1498, 1456, 1381, 1363, 1233, 1028, 966, 750, 699 cm^{-1}. MS (ESI): m/z (%) = 173.0 (100) $[M + Na]^+$. HRMS: calcd. for $C_9H_{10}O_2$ 150.0681; found 150.0673.

3,4,5-Trimethoxybenzyl Acetate (4d): Purification by flash column chromatography [SiO_2, hexane/EtOAc (3:1)]. ^{1}H NMR (360 MHz, $CDCl_3$): δ = 6.59 (s, 2 H), 5.03 (s, 2 H), 3.87 (s, 6 H), 3.84 (s, 3 H), 2.11 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.1 (C), 153.5 (2×C), 138.1 (C), 131.6 (C), 105.7 (2×CH), 66.8 (CH_2),

61.0 (CH_3), 56.3 (2 × CH_3), 21.2 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2999, 2943, 1740, 1593, 1508, 1463, 1424, 1364, 1332, 1237, 1129, 1010 cm^{-1}. MS (ESI): *m/z* (%) = 263.1 (100) $[M + Na]^+$. HRMS: calcd. for $C_{12}H_{16}O_5$ 240.0998; found 240.0999.

(–)-Menthyl Acetate (4e): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 4.66 (td, *J* = 10.9, 4.1 Hz, 1 H), 2.03 (s, 3 H), 2.00–1.95 (m, 1 H), 1.92–1.79 (m, 1 H), 1.71–1.63 (m, 2 H), 1.55–1.41 (m, 1 H), 1.39–1.31 (m, 1 H), 1.11–0.94 (m, 2 H), 0.9–0.88 (m, 7 H), 0.77–0.75 (d, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.8 (C), 74.2 (CH), 47.0 (CH), 40.9 (CH_2), 34.2 (CH_2), 31.4 (CH), 26.3 (CH), 23.5 (CH_2), 22.0 (CH_3), 21.3 (CH_3), 20.7 (CH_3), 16.4 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2957, 2932, 2871, 1737, 1457, 1371, 1246, 1025 cm^{-1}. MS (ESI): *m/z* (%) = 221.1 (100) $[M + Na]^+$. HRMS: calcd. for $C_{12}H_{22}O_2$ 198.1620; found 198.1614.

2-Oxo-1,2-diphenylethyl Acetate (4f): The crude product was triturated with EtOAc. Evaporation of EtOAc gave the ester, the remaining alcohol and 5,7-dinitroindoline. Purification by flash column chromatography [SiO_2, hexane/EtOAc (6:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 7.93 (d, *J* = 7.2 Hz, 2 H), 7.53–7.32 (m, 8 H), 6.86 (s, 1 H), 2.21 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 193.7 (C), 170.5 (C), 134.6 (C), 133.6 (C), 133.5 (CH), 129.3 (CH), 129.1 (2 × CH), 128.8 (2 × CH), 128.7 (2 × CH), 128.6 (2 × CH), 77.6 (CH), 20.8 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 1740, 1697, 1598, 1451, 1374, 1231, 1182, 1056, 1005, 973, 932, 758, 699, 526 cm^{-1}. MS (ESI): *m/z* (%) = 277.1 (100) $[M + Na]^+$. HRMS: calcd for $C_{16}H_{14}O_3$ 254.0943; found 254.0942.

1-Formylnaphthalen-2-yl Acetate (4 g): The crude product was triturated with EtOAc. Evaporation of EtOAc gave the ester, the remaining alcohol and 5,7-dinitroindoline. Purification by flash column chromatography [SiO_2, hexane/EtOAc (6:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 10.71 (s, 1 H), 9.14 (d, *J* = 8.6 Hz, 1 H), 8.10 (d, *J* = 9.1 Hz, 1 H), 7.88 (d, *J* = 8.2 Hz, 1 H), 7.68 (t, *J* = 7.3 Hz, 1 H), 7.56 (t, *J* = 7.5 Hz, 1 H), 7.28 (d, *J* = 9.6 Hz, 1 H), 2.44 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 190.0 (C), 169.2 (C), 154.4 (C), 136.5 (CH), 131.8 (C), 131.1 (C), 129.6 (CH), 128.5 (CH), 126.7 (CH), 125.0 (CH), 121.6 (CH), 121.4 (C), 20.9 (CH_3) ppm. IR (KBr): $\tilde{\nu}$ = 1767, 1688, 1617, 1576, 1511, 1434, 1372, 1342, 1189, 1161, 1061, 1036, 1017, 979, 893, 857, 826, 763, 743, 706, 675, 510 cm^{-1}. MS (ESI): *m/z* (%) = 237.0 (100) $[M + Na]^+$. HRMS: calcd for $C_{13}H_{10}O_3$ 214.0630; found 214.0625.

Cyclohexyl Acetate (4h): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 4.76–4.70 (m, 1 H), 2.03 (s, 3 H), 1.87–1.82 (m, 2 H), 1.74–1.70 (m, 2 H) 1.57–1.52 (m, 1 H), 1.44–1.20 (m, 5 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.6 (C), 72.7 (CH), 31.6 (2 × CH_2), 25.4 (CH_2), 23.8 (2 × CH_2), 21.5 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2940, 2861, 1737, 1452, 1379, 1364, 1241, 1126, 1046, 1023, 968, 907, 735 cm^{-1}. MS (ESI): *m/z* (%) = 286.3 {2 × $[M + H]^+$}, 165.1 (40) $[M + Na]^+$.

***trans*-(–)-Myrtyl Acetate (4i):** Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 3.92–3.81 (m, 2 H), 2.34–2.25 (m, 1 H), 2.08–2.02 (m, 1 H), 2.03 (s, 3 H), 1.89–1.71 (m, 4 H), 1.67–1.59 (m, 1 H), 1.34–1.18 (m, 2 H), 1.21 (s, 3 H), 0.84 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$) δ = 171.4 (C), 68.0 (CH_2), 42.3 (CH), 40.8 (CH), 39.1 (C), 34.1 (CH), 26.6 (CH_2), 23.9 (CH_3), 23.3 (CH_2), 21.0 (CH_3), 20.1 (CH_3), 18.1 (CH_2) ppm. IR (neat): $\tilde{\nu}$ = 2979, 2945, 2917, 2870, 1743, 1463, 1386, 1367, 1236, 1031, 980 cm^{-1}. MS (ESI): *m/z* (%) = 219.1 (100) $[M + Na]^+$. HRMS: calcd. for $C_{12}H_{20}O_2$ 196.1463; found 196.1456.

(±)-Linaloyl Acetate (4j): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 6.01–5.93 (m, 1 H), 5.17,5.07 (m, 3 H), 2.01 (s, 3 H), 1.96 (t, *J* = 7.5 Hz, 2 H), 1.89–1.71 (m, 2 H), 1.67 (s, 3 H), 1.59 (s, 3 H), 1.53 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$) δ = 170.0 (C), 141.8 (CH), 131.8 (C), 123.8 (CH), 113.1 (CH_2), 82.9 (C), 39.7 (CH_2), 25.7 (CH_3), 23.6 (CH_3), 22.3 (CH_2), 22.2 (CH_3), 17.6 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2973, 2930, 2861, 1740, 1450, 1370, 1248, 1173, 1093, 1019, 922 cm^{-1}. HRMS: calcd. for $C_{12}H_{20}O_2$ 196.1463; found 196.1458.

Geranyl Acetate (4k): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.34 (t, *J* = 7.2 Hz, 1 H), 5.08 (t, *J* = 6.8 Hz, 1 H), 4.58 (d, *J* = 7.3 Hz, 2 H), 2.14–2.00 (m, 4 H), 2.05 (s, 3 H), 1.70 (s, 3 H), 1.68 (s, 3 H), 1.60 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 171.2 (C), 142.3 (C), 131.8 (C), 123.7 (CH), 118.2 (CH), 61.4 (CH_2), 39.5 (CH_2), 26.3 (CH_2), 25.7 (CH_3), 21.1 (CH_3), 17.7 (CH_3), 16.5 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2969, 2926, 2859, 1742, 1446, 1380, 1369, 1233, 1024, 982, 955 cm^{-1}. MS (ESI): *m/z* (%) = 219.1 (100) $[M + Na]^+$. HRMS: calcd. for $C_{12}H_{20}O_2$ 196.1463; found 196.1457.

1-Ethynylcyclohexyl Acetate (4l): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 2.60 (s, 1 H), 2.16–2.10 (m, 2 H), 2.05 (s, 3 H), 1.88–1.81 (m, 2 H), 1.65–1.59 (m, 4 H), 1.55–1.48 (m, 1 H), 1.38–1.27 (m, 1 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 169.3 (C), 83.7 (C), 75.1 (C), 74.2 (CH), 36.9 (2 × H_2), 25.1 (CH_2), 22.4 (2 × CH_2), 21.9 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3283, 2934, 2863, 2113, 1745, 1450, 1368, 1231, 1145, 1025, 957, 663 cm^{-1}. GC–MS: *m/z* (%) = 166.1 (2) $[M]^+$, 124.0 (58), 109 (40), 106 (50), 95 (74), 91 (10), 81 (92), 79 (68), 67 (52). HRMS: calcd. for $C_{10}H_{14}O_2$ 166.0994; found 160.0983.

1,2,3,4,6-Penta-*O*-acetyl-β-D-glucose (4m): The crude product was triturated with EtOAc. Evaporation of EtOAc gave the ester, the remaining alcohol and 5,7-dinitroindoline. Purification by flash column chromatography (SiO_2, CH_2Cl_2) to remove 5,7-dinitroindoline and [SiO_2, hexane/EtOAc (1:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.71 (d, *J* = 8.2 Hz, 1 H), 5.25 (dd, *J* = 9.3 Hz, 1 H), 5.15–5.10 (m, 2 H), 4.29 (dd, *J* = 12.5, 4.3 Hz, 1 H), 4.11 (dd, *J* = 12.5, 2.1 Hz, 1 H), 3.84 (dm, *J* = 10 Hz, 1 H), 2.11 (s, 3 H), 2.08 (s, 3 H), 2.03 (s, 6 H), 2.01 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.6 (C), 170.1 (C), 169.4 (C), 169.2 (C), 169.0 (C), 91.7 (CH), 72.7 (2 × CH), 70.2 (CH), 67.7 (CH), 61.4 (CH_2), 20.8 (CH_3), 20.7 (CH_3), 20.6 (3 × CH_3) ppm. IR (neat/$CHCl_3$): $\tilde{\nu}$ = 3025, 1760, 1370, 1222, 1080, 1040, 758, 669 cm^{-1}. HRMS: calcd. for $C_{16}H_{22}O_{11}$ 390.1162; found 390.1156.

(±)-1-Phenylethyl Acetate (4n): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 7.36–7.28 (m, 5 H), 5.88 (q, *J* = 6.8 Hz, 1 H), 2.08 (s, 3 H), 1.54 (d, *J* = 6.8 Hz, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.3 (C), 141.7 (C), 128.5 (2 × CH), 127.9 (CH), 126.1 (2 × CH), 72.3 (CH), 22.2 (CH_3), 21.4 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3065, 3034, 2983, 2936, 1744, 1495, 1453, 1372, 1242, 1210, 1065, 1027, 945, 761, 700, 539 cm^{-1}. HRMS: calcd. for $C_{10}H_{12}O_2$ 164.0837; found 164.0831.

Cinnamyl Acetate (4o): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 7.40 (d, *J* = 7.3 Hz; 2 H), 7.33 (dd, *J* = 7.3 Hz, 2 H), 7.26 (dd, *J* = 7.1 Hz, 1 H), 6.66 (d, *J* = 15.9 Hz, 1 H), 6.29 (dt, *J* = 15.9, 6.6 Hz, 1 H), 4.73 (d, *J* = 6.4 Hz, 2 H), 2.10 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 170.9 (C), 136.2 (C), 134.2 (CH), 128.6 (2 × CH), 128.1 (CH), 126.6 (2 × CH), 123.1 (CH), 65.1 (CH_2), 21.0 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3060, 3029, 2945, 1739,

1495, 1490, 1455, 1381, 1363, 1233, 1027, 967, 747, 694 cm^{-1}. HRMS: calcd. for $C_{11}H_{12}O_2$ 176.0837; found 176.0832.

3′,5′-Dimethoxy-2-oxo-1,2-diphenyl Acetate (4p): For this photosensitive ester, the working wavelength was 405 nm. The crude product was triturated with EtOAc and evaporation of EtOAc gave the ester, the remaining alcohol and 5,7-dinitroindoline. Purification by flash column chromatography [SiO_2, hexane/EtOAc (3:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 7.94 (d, J = 7.8 Hz, 2 H), 7.52 (t, J = 7.3 Hz, 1 H), 7.41 (t, J = 7.8 Hz, 2 H), 6.75 (s, 1 H), 6.59 (d, J = 1.8 Hz, 2 H), 6.42 (t, J = 1.8 Hz, 1 H), 3.76 (s, 6 H), 2.21 (s, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 193.5 (C), 170.4 (C), 161.2 (2 × C), 135.5 (C), 134.5 (C), 133.5 (CH), 128.8 (2 × CH), 128.6 (2 × CH), 106.7 (2 × CH), 101.2 (CH), 77.6 (CH), 55.4 (2 × CH_3), 20.8 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 3006, 2942, 2841, 1745, 1698, 1598, 1464, 1431, 1374, 1355, 1281, 1231, 1160, 1056, 1002, 839, 691 cm^{-1}. HRMS: calcd. for $C_{18}H_{18}O_5$ 314.1154; found 314.1147.

Undec-10-enyl Propionate (6b): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.85–5.77 (m, 1 H), 5.01–4.91 (m, 2 H), 4.05 (t, J = 6.8 Hz, 2 H), 2.32 (q, J = 7.7 Hz, 2 H), 2.06–2.00 (m, 2 H), 1.63–1.58 (m, 2 H), 1.37–1.28 (m, 12 H), 1.14 (t, J = 7.7 Hz, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 174.6 (C), 139.2 (CH), 114.3 (CH_2), 64.5 (CH_2) 33.8 (CH_2), 29.4 (CH_2), 29.4 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 28.9 (CH_2), 28.6 (CH_2), 27.6 (CH_2), 25.9 (CH_2), 9.2 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2979, 2929, 2856, 1741, 1464, 1350, 1274, 1187, 1084, 995, 910 cm^{-1}. HRMS: calcd. for $C_{16}H_{26}O_2$ 226.1933; found 226.1929.

Undec-10-enyl Lauroate (6c): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.85–5.77 (m, 1 H), 5.01–4.91 (m, 2 H), 4.05 (t, J = 6.8 Hz, 2 H), 2.28 (t, J = 7.5 Hz, 2 H), 2.06–2.01 (m, 2 H), 1.63–1.57 (m, 4 H), 1.37–1.25 (m, 28 H), 0.88 (t, J = 6.5 Hz, 3 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 174.0 (C), 139.2 (CH), 114.1 (CH_2), 64.4 (CH_2), 34.4 (CH_2), 33.8 (CH_2), 31.9 (CH_2), 29.6 (2 × CH_2), 29.5 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 29.3 (CH_2), 29.3 (CH_2), 29.2 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 28.9 (CH_2), 28.6 (CH_2), 25.9 (CH_2), 25.0 (CH_2), 22.7 (CH_2), 14.1 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2929, 2856, 1739, 1467, 1173, 909 cm^{-1}. HRMS: calcd. for $C_{23}H_{44}O_2$ 352.3341; found 352.3334.

Undec-10-enyl Benzoate (6d): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 8.05 (d, J = 7.3 Hz, 2 H), 7.55 (dd, J = 7.7, 7.3 Hz 1 H), 7.44 (dd, J = 8.2, 7.3 Hz 1 H), 5.87–5.75 (m, 1 H), 5.01–4.92 (m, 2 H), 4.31 (t, J = 6.8 Hz, 2 H), 2.04 (q, J = 7.2 Hz, 2 H), 1.80–1.73 (m, 2 H), 1.46–1.30 (m, 12 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 166.7 (C), 139.2 (CH), 132.8 (CH), 130.5 (C), 129.5 (2 × CH), 128.3 (2 × CH), 114.1 (CH_2), 65.1 (CH_2) 33.8 (CH_2), 29.5 (CH_2), 29.4 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 28.9 (CH_2), 28.7 (CH_2), 26.0 (CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2928, 2855, 1722, 1453, 1314, 1274, 1176, 1114, 1070, 1028, 910, 712 cm^{-1}. HRMS: calcd. for $C_{18}H_{26}O_2$ 274.1933; found 274.1929.

Undec-10-enyl 4-Chlorobutanoate (6e): Purification by flash column chromatography [SiO_2, hexane/EtOAc (9:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.85–5.77 (m, 1 H), 5.02–4.92 (m, 2 H), 4.07 (t, J = 6.8 Hz, 2 H), 3.60 (t, J = 6.4 Hz, 2 H), 2.50 (t, J = 7.2 Hz, 2 H), 2.13–2.01 (m, 4 H), 1.64–1.55 (m, 2 H), 1.42–1.29 (m, 12 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 172.8 (C), 139.2 (CH), 114.1 (CH_2), 64.8 (CH_2) 44.1 (CH_2), 33.8 (CH_2), 31.2 (CH_2), 29.4 (2 × CH_2), 29.2 (CH_2), 29.1 (CH_2), 28.9 (CH_2), 28.6 (CH_2), 27.7 (CH_2), 25.9 (CH_2) ppm. IR (neat): $\tilde{\nu}$ = 2930, 2856, 1737, 1466, 1299, 1240, 1206, 1176, 1147, 911 cm^{-1}. HRMS: calcd. for $C_{18}H_{26}O_2$ 274.1700; 274.1697.

Methyl Undec-10-enylsuccinate (6f): Purification by flash column chromatography [SiO_2, hexane/EtOAc (4:1)]. ^1H NMR (360 MHz, $CDCl_3$): δ = 5.85–5.75 (m, 1 H), 5.01–4.91 (m, 2 H), 4.08 (t, J = 6.8 Hz, 2 H), 3.69 (s, 3 H), 2.63 (br. s, 4 H), 2.06–2.01 (m, 2 H), 1.65–1.58 (m, 2 H), 1.39–1.28 (m, 12 H) ppm. ^{13}C NMR (90.55 MHz, $CDCl_3$): δ = 172.8 (C), 172.4 (C), 139.2 (CH), 114.1 (CH_2), 64.9 (CH_2), 51.8 (CH_3), 33.8 (CH_2), 29.4 (CH_2), 29.4 (CH_2), 29.2 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 28.9 (CH_2), 28.9 (CH_2), 28.6 (CH_2), 25.8(CH_2) ppm. IR (neat): $\tilde{\nu}$ = 2927, 2856, 1739, 1438, 1357, 1214, 1162, 997 cm^{-1}. HRMS: calcd. for $C_{16}H_{28}O_4$ 284.1988; found 284.1983.

Supporting Information (see footnote on the first page of this article): ^1H- and ^{13}C-NMR spectra for **1a**–**f**, **4a**–**p** and **6b**–**f**.

Acknowledgments

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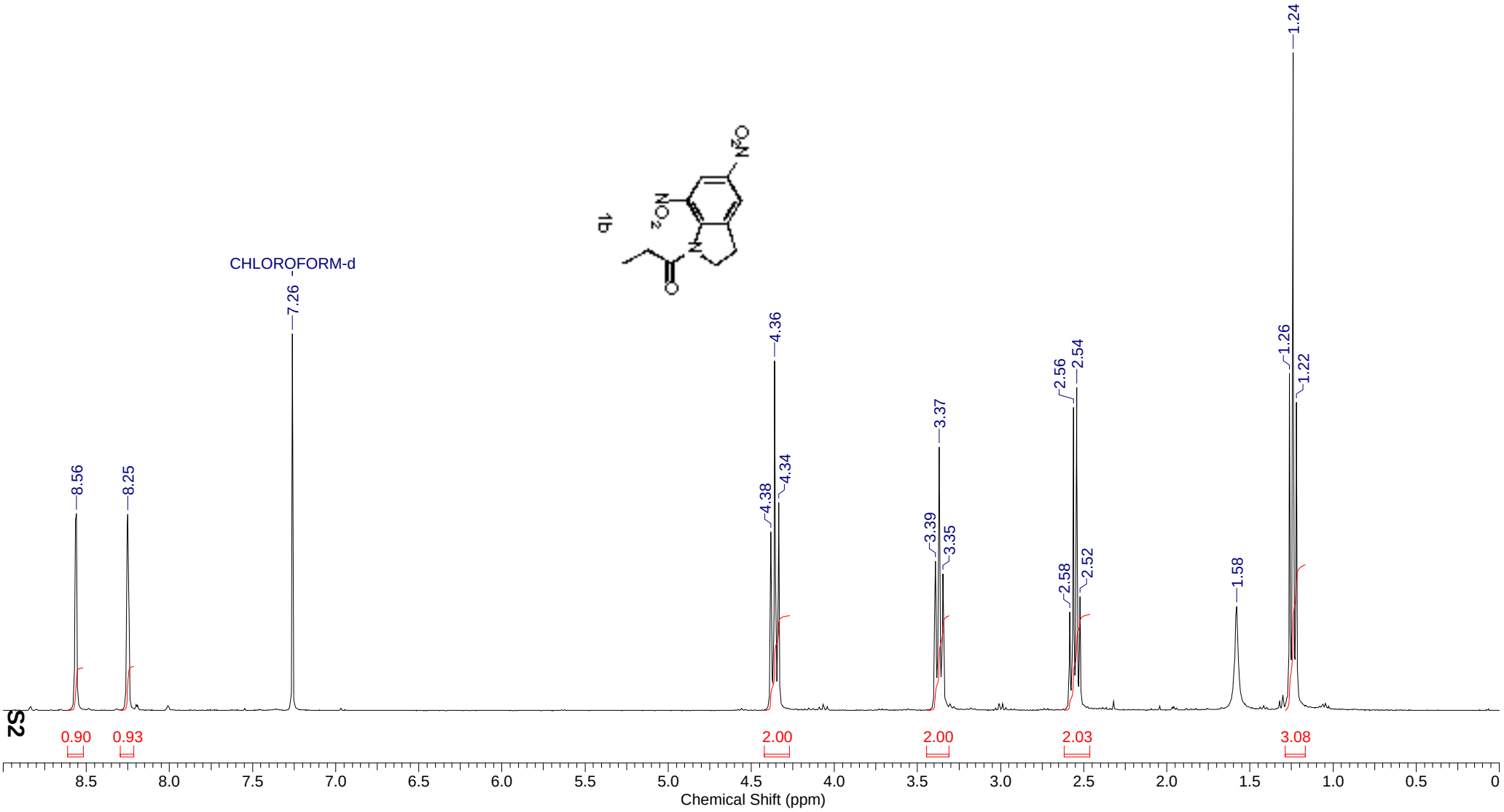
SUPPORTING INFORMATION

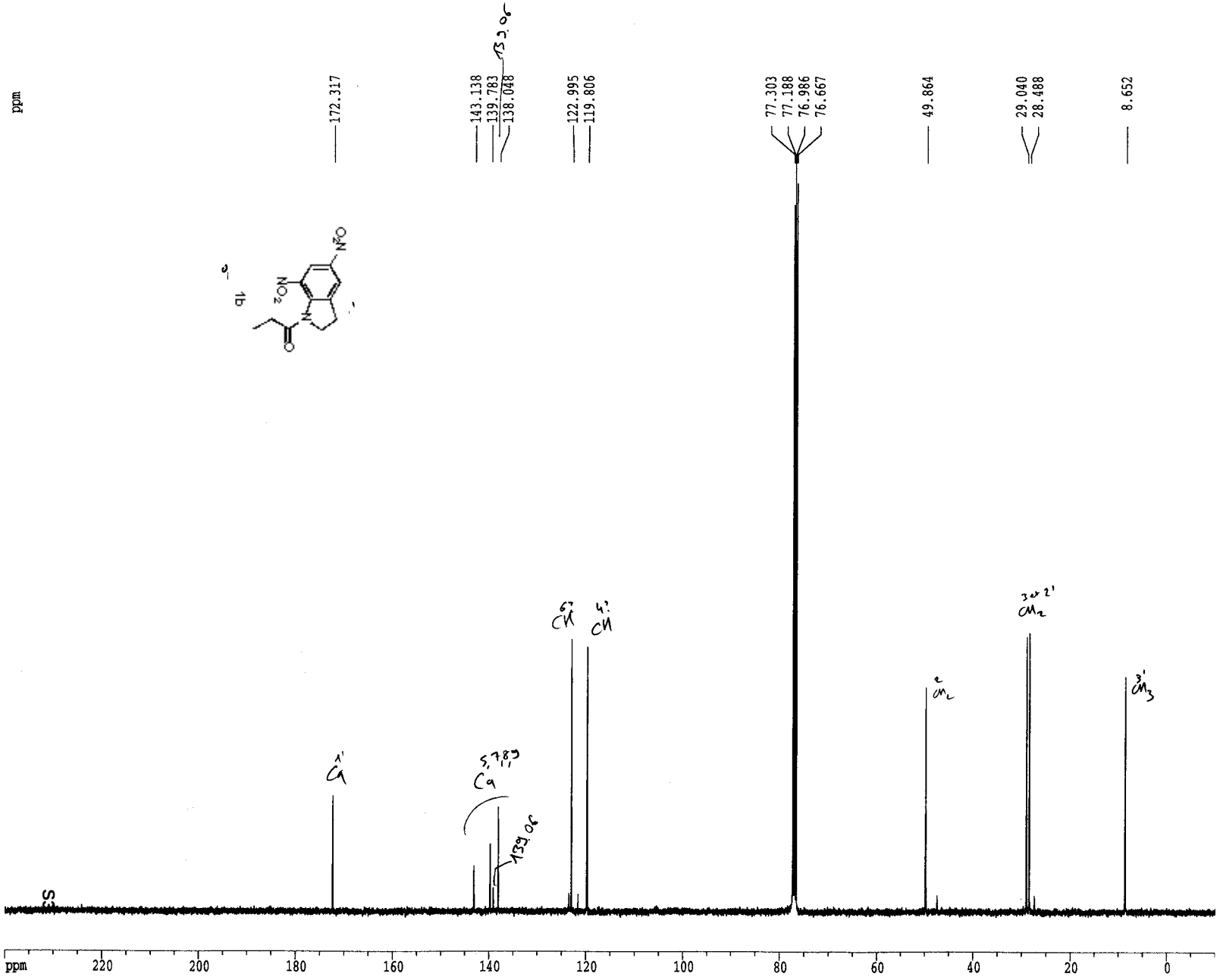
Title: Photoacylation of Alcohols in Neutral Medium
Author(s): Jean-Luc Débieux, Anne Cosandey, Céline Helgen, Christian G. Bochet*
Ref. No.: O200600790

1-Propionyl-5,7-dinitroindoline

Acquisition Time (sec)	2.2021	Comment	N-propyl, dinitroindoline PROTONNR CDCl3 u jld 2			Date	10 Mar 2006 11:58:56
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\dinitropropyl_001001r					Frequency (MHz)	360.13
Nucleus	1H	Number of Transients	32	Origin	dpx360	Original Points Count	16384
Owner	nmruser	Points Count	16384	Pulse Sequence	zg30	Receiver Gain	322.50
SW (cyclical) (Hz)	7440.48	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2210.5950
Sweep Width (Hz)	7440.02	Temperature (degree C)	27.000				



Celine
C13CPD CDCl3 u guest 6



Current Data Parameters

NAME	CH244B
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20010619
Time	7.34
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PROBHD	5 mm QNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	2
SWH	27173.912 Hz
FIDRES	0.414641 Hz
AQ	1.2059124 sec
RG	6502
DW	18.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	7.20 usec
PL1	-5.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-5.00 dB
PL12	13.00 dB
PL13	13.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

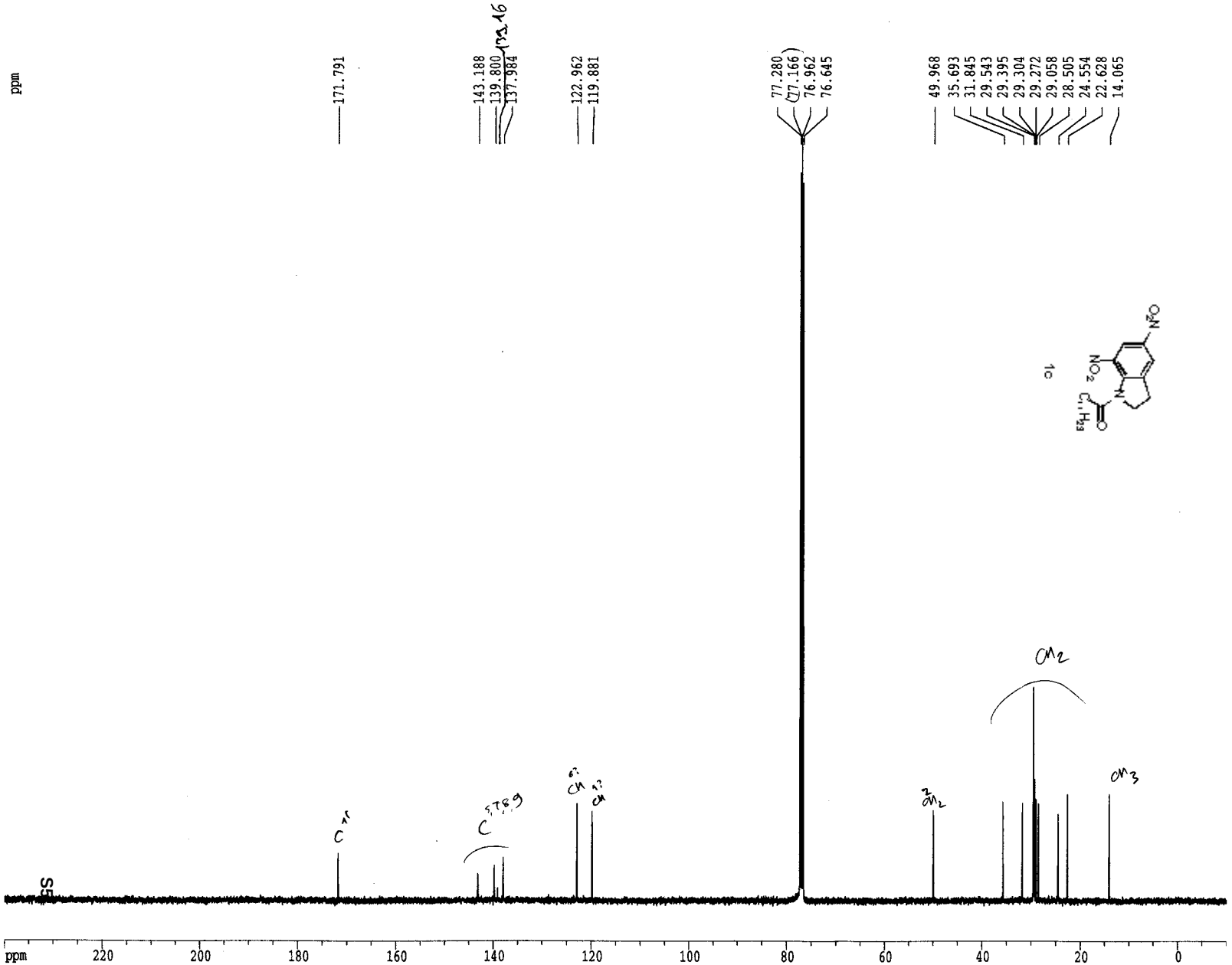
SI	32768
SF	100.6127786 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
F1P	240.000 ppm
F1	24147.07 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	8.33333 ppm/cm
HZCM	838.43982 Hz/cm

Celine□PROTON CDCl3 u guest 12□
*** Current Data Parameters ***
NAME : ch252g
EXPNO : 10
PROCNO : 1
8.5789
8.5732
8.2581
8.2556
8.2531
7.2688
4.3879
4.3677
4.3462
3.3890
3.3682
3.3480
2.5329
2.5146
2.4957
1.7721
1.7538
1.7355
1.7166
1.6976
1.5827
1.3870
1.3642
1.2658
0.9008
0.8844
0.8667
BRUKER
1e
H-C5
H-C5'
H-C2
H-C3
H-C2'
H-C3'
H-C12'
H-C4'-11'
Integral
1.0000
1.0370
2.1609
2.1674
2.1605
2.1994
17.959
3.2957
(ppm)

Celine
C13CPD CDCl3 u guest 12



Current Data Parameters

NAME	CH252G
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

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Time	1.26
INSTRUM	drx400
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PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	2
SWH	27173.912 Hz
FIDRES	0.414641 Hz
AQ	1.2059124 sec
RG	11585.2
DW	18.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	7.20 usec
PL1	-5.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-5.00 dB
PL12	13.00 dB
PL13	13.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

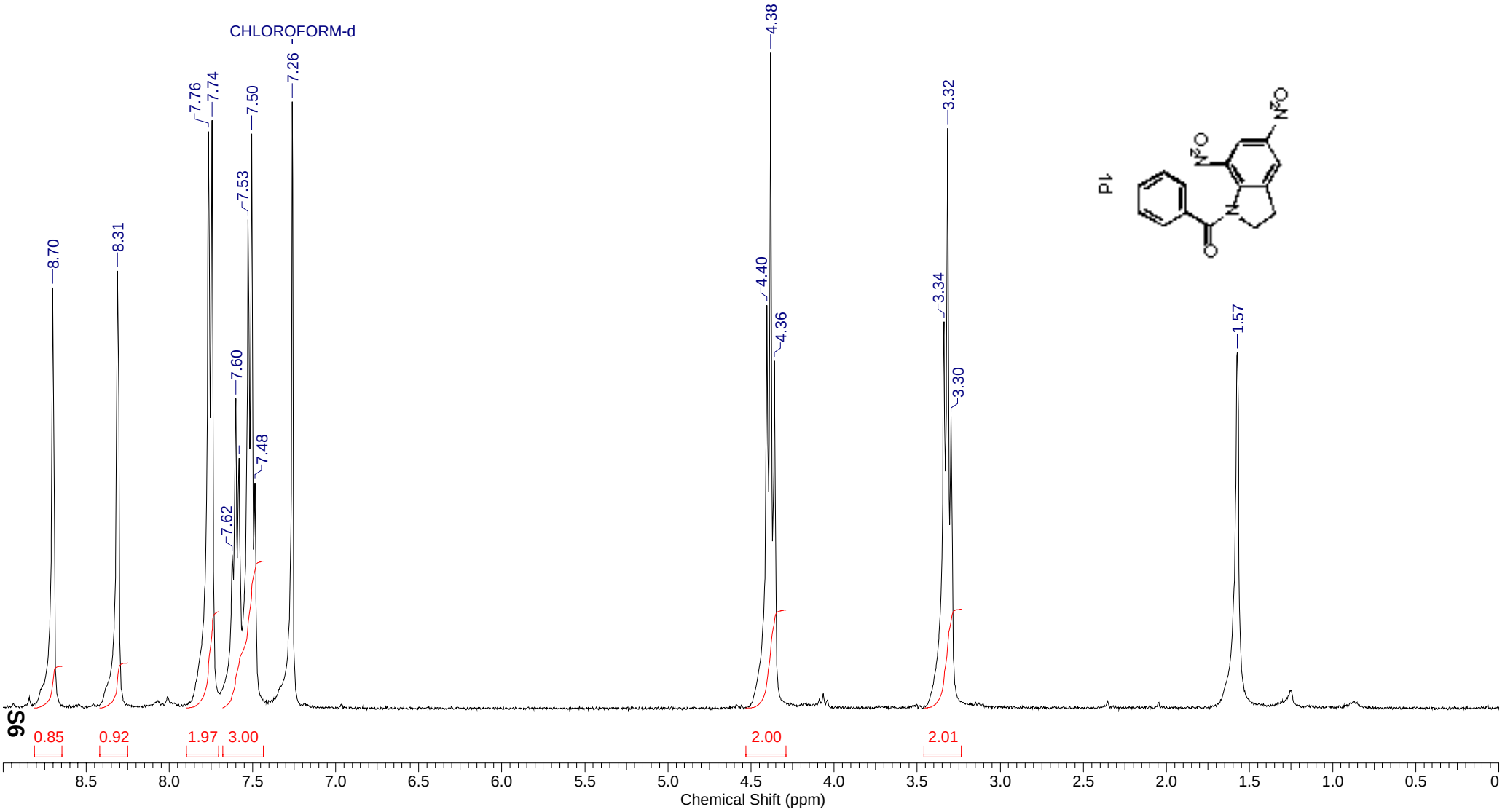
SI	32768
SF	100.6127786 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
F1P	240.000 ppm
F1	24147.07 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	8.33333 ppm/cm
HZCM	838.43982 Hz/cm

1-Benzoyl-5,7-dinitroindoline

Acquisition Time (sec)	2.2021	Comment	PROTONNR CDCl3 u jld 37			Date	16 Mar 2006 08:08:32
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\CH91_001001r					Frequency (MHz)	360.13
Nucleus	1H	Number of Transients	16	Origin	dpx360	Original Points Count	16384
Owner	nmruser	Points Count	16384	Pulse Sequence	zg30	Receiver Gain	406.40
SW (cyclical) (Hz)	7440.48	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2211.0491
Sweep Width (Hz)	7440.02	Temperature (degree C)	27.000				



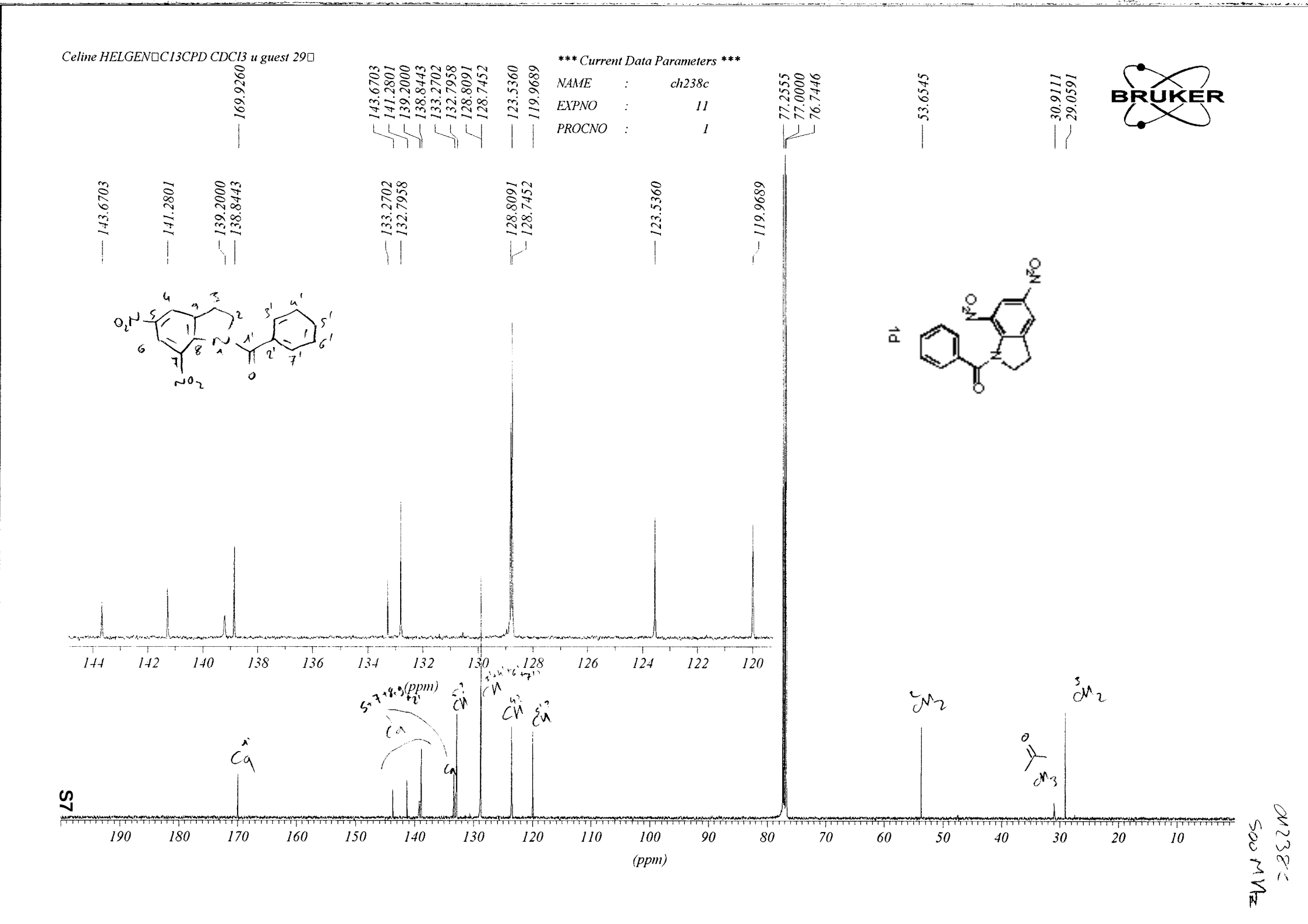
Celine HELGEN□C13CPD CDCl3 u guest 29□

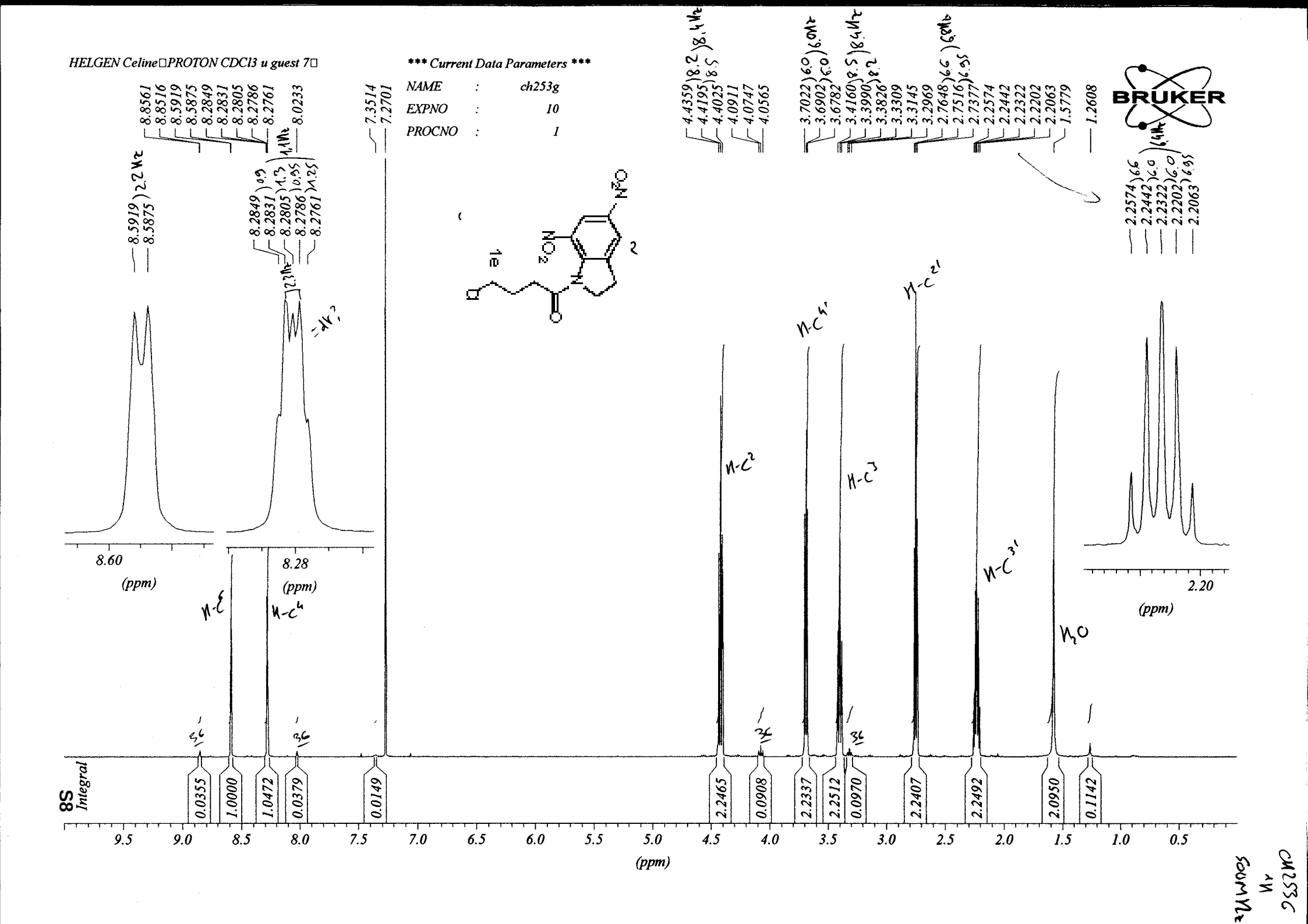
*** Current Data Parameters ***

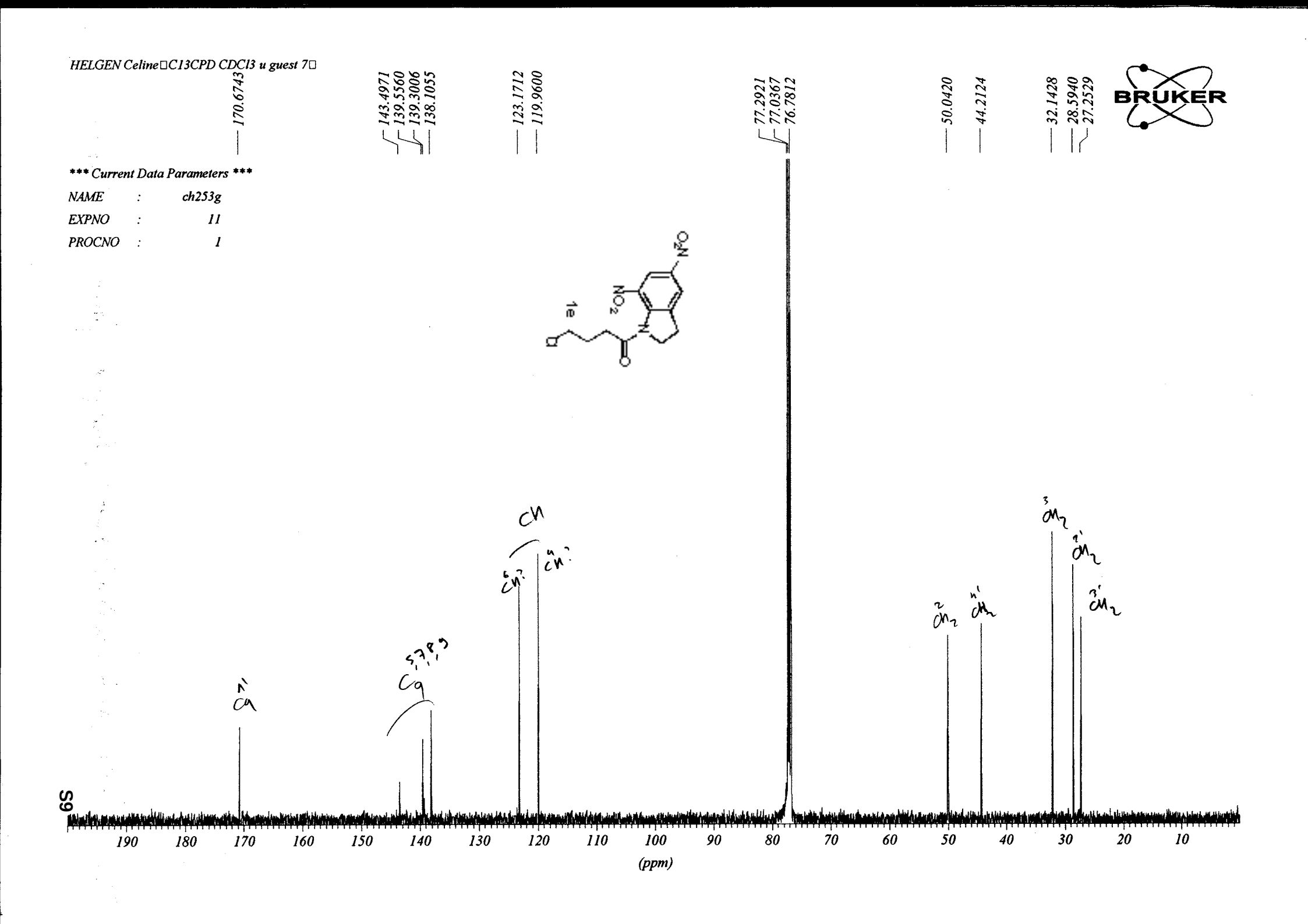
NAME : ch238c

EXPNO : 11

PROCNO : 1

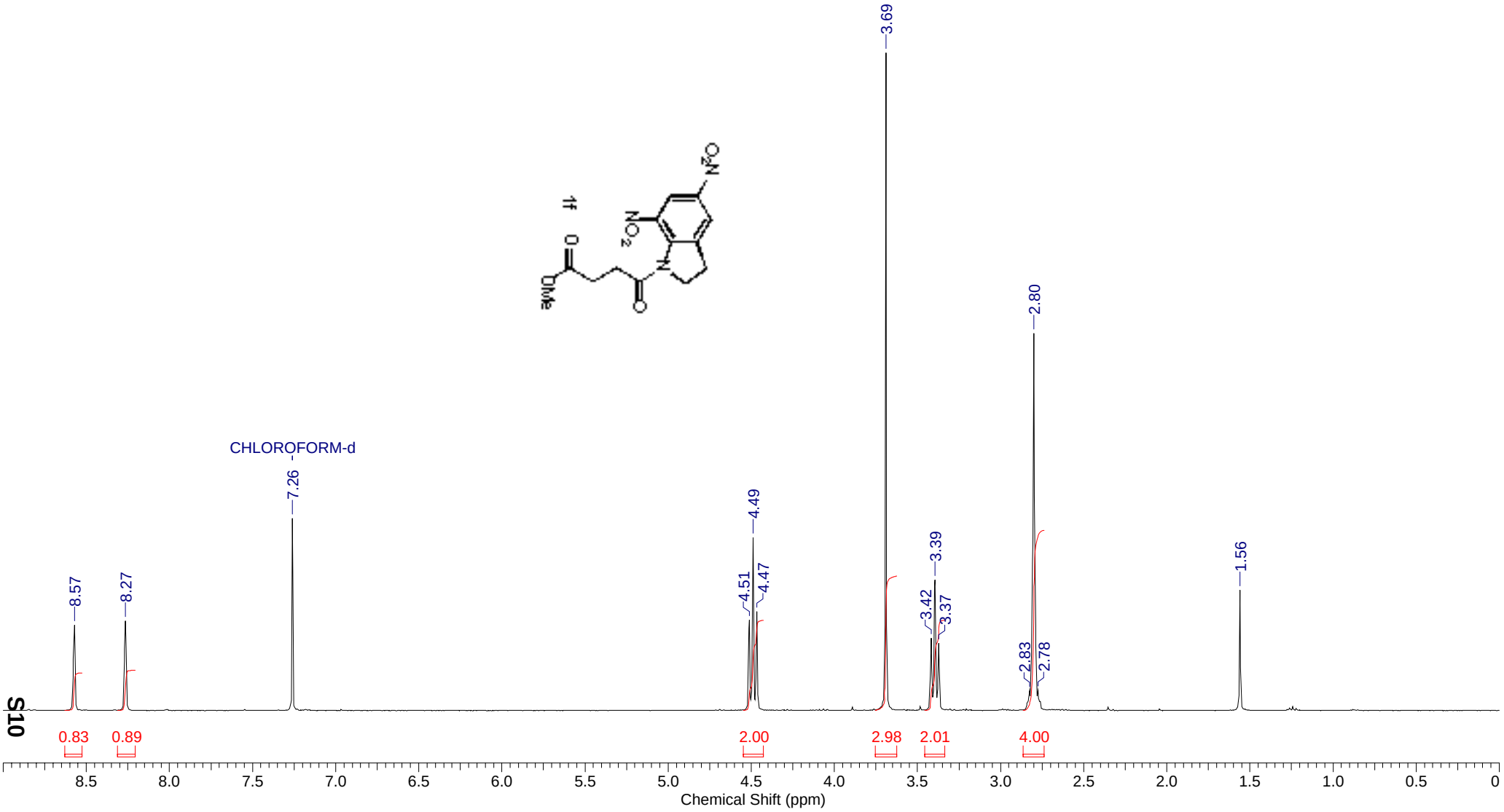



HELGEN Celine□PROTON CDCl3 u guest 7□
*** Current Data Parameters ***
NAME : ch253g
EXPNO : 10
PROCNO : 1
BRUKER
8.8561
8.8516
8.5919
8.5875
8.2849
8.2831
8.2805
8.2786
8.2761
8.0233
7.3514
7.2701
4.4359
4.4195
4.4025
4.0911
4.0747
4.0565
3.7022
3.6902
3.6782
3.4160
3.3990
3.3826
3.3309
3.3145
3.2969
2.7648
2.7516
2.7377
2.2574
2.2442
2.2322
2.2202
2.2063
1.5779
1.2608
8.60
8.28
2.20
(ppm)
H-C2
H-C3
H-C4
H-C4'
H-C2'
H-C3'
H-E
H2O
Integral
0.0355
1.0000
1.0472
0.0379
0.0149
2.2465
0.0908
2.2337
2.2512
0.0970
2.2407
2.2492
2.0950
0.1142
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

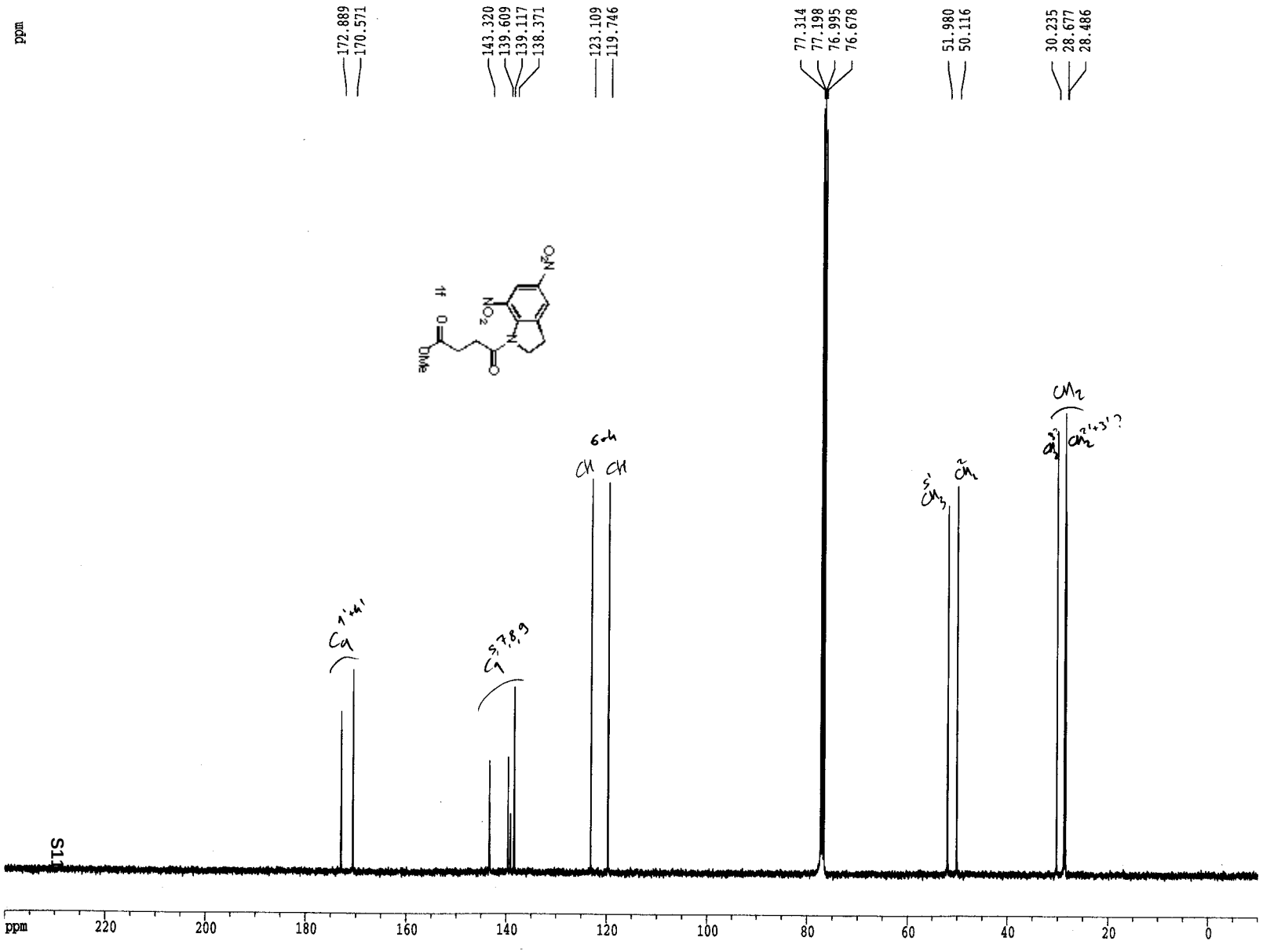

HELGEN Celine□C13CPD CDCl3 u guest 7□
170.6743
143.4971
139.5560
139.3006
138.1055
123.1712
119.9600
77.2921
77.0367
76.7812
50.0420
44.2124
32.1428
28.5940
27.2529
BRUKER
*** Current Data Parameters ***
NAME : ch253g
EXPNO : 11
PROCNO : 1
1e
NO2
NO2
S9
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
(ppm)

1-(3-Methoxycarbonyl-propionyl)-5,7-dinitroindoline

Acquisition Time (sec)	2.2021	Comment	PROTONNR CDCl3 u jld 6			Date	28 Apr 2006 13:07:12
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\ind-succin_001001r					Frequency (MHz)	360.13
Nucleus	1H	Number of Transients	16	Origin	dpx360	Original Points Count	16384
Owner	nmruser	Points Count	16384	Pulse Sequence	zg30	Receiver Gain	456.10
SW (cyclical) (Hz)	7440.48	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	2210.5947
Sweep Width (Hz)	7440.02	Temperature (degree C)	27.000				



Celine
C13CPD CDCl3 u guest 1



Current Data Parameters

NAME	CH280A
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20010927
Time	20.00
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PROBHD	5 mm QNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	3072
DS	2
SWH	27173.912 Hz
FIDRES	0.414641 Hz
AQ	1.2059124 sec
RG	4096
DW	18.400 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

========== CHANNEL f1 ==========

NUC1	13C
P1	7.20 usec
PL1	-5.00 dB
SFO1	100.6237959 MHz

========== CHANNEL f2 ==========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-5.00 dB
PL12	13.00 dB
PL13	13.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

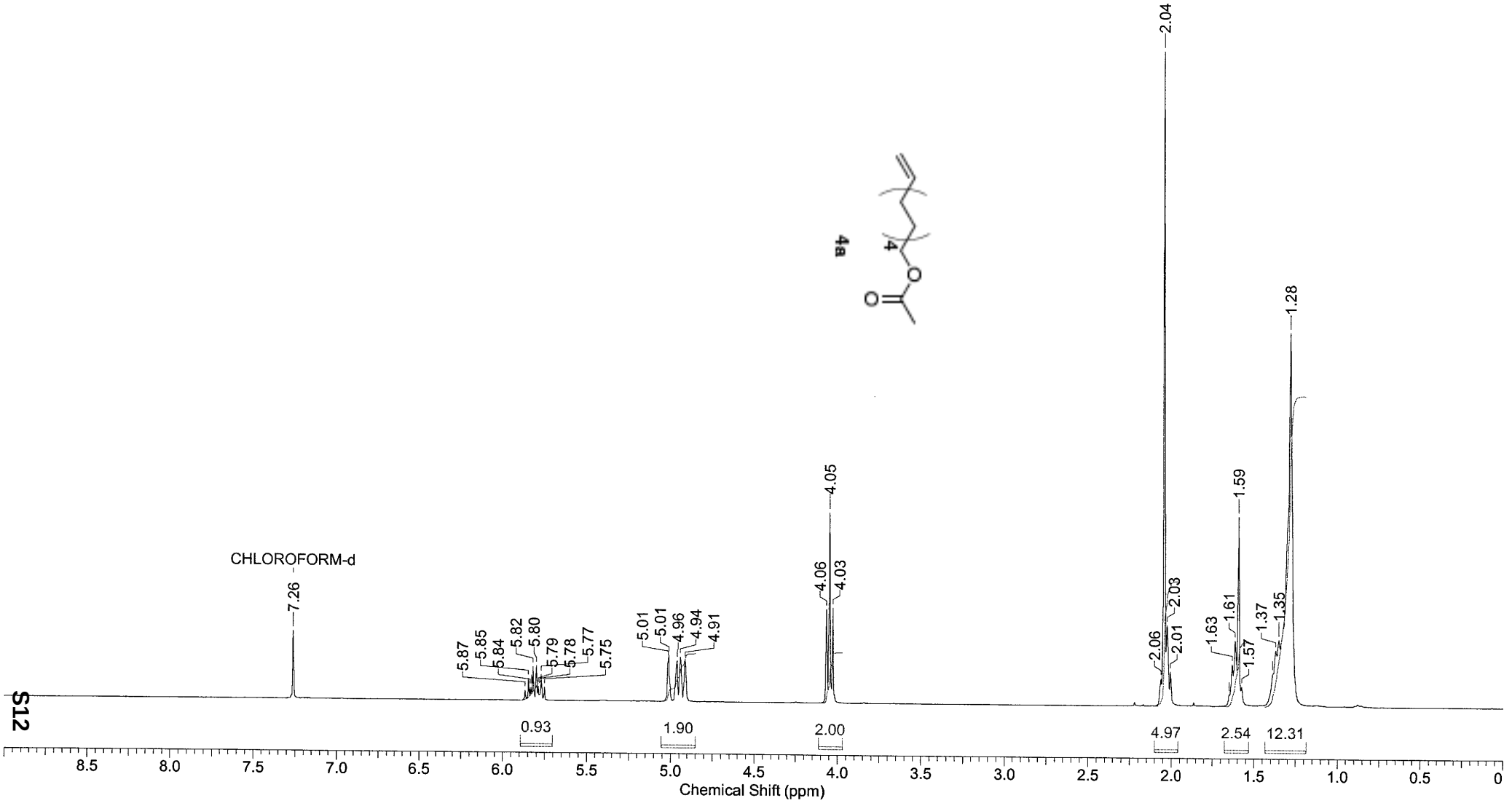
SI	32768
SF	100.6127786 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
F1P	240.000 ppm
F1	24147.07 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	8.33333 ppm/cm
HZCM	838.43982 Hz/cm

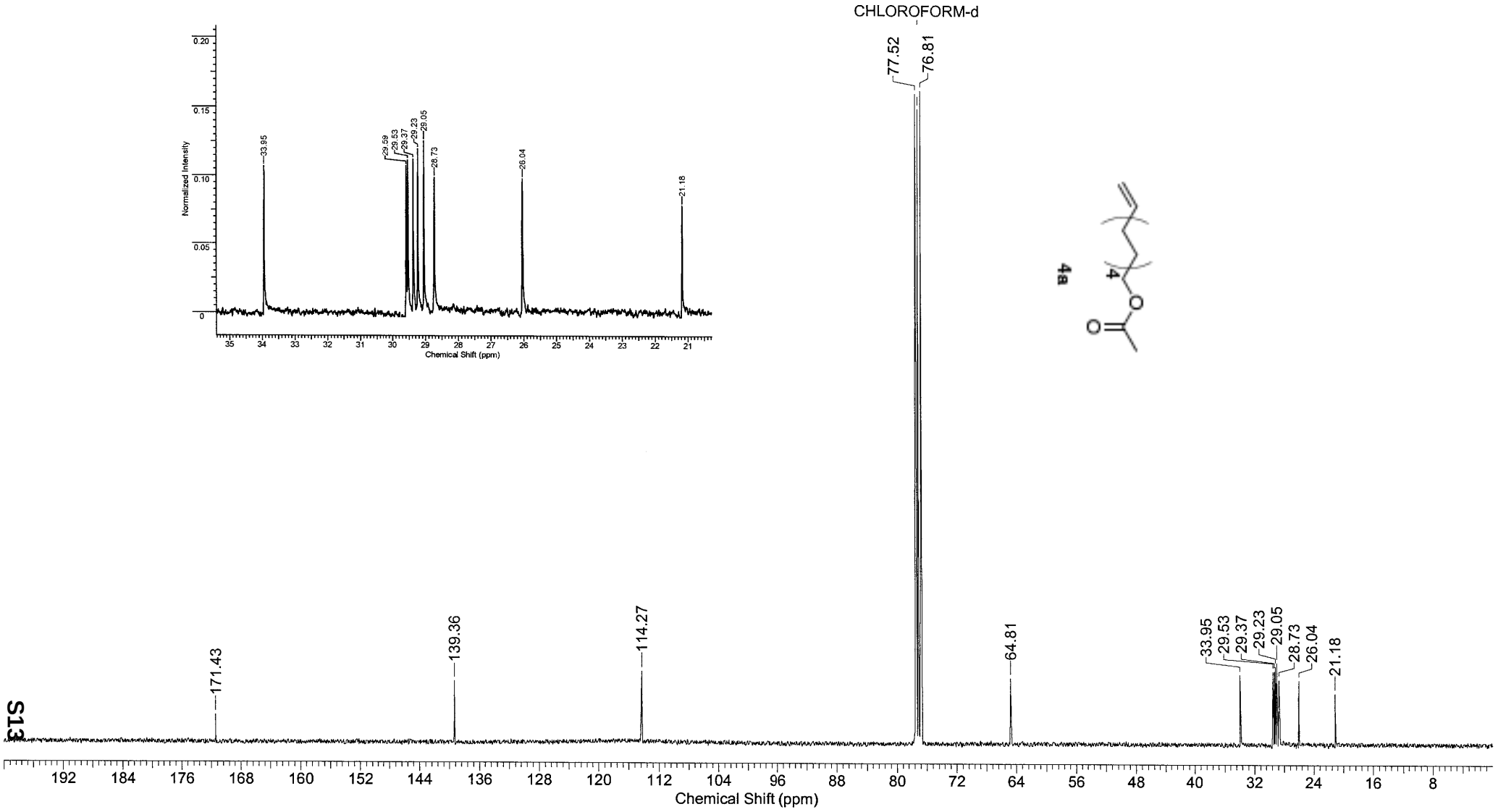
jld03

Acquisition Time (sec)	2.2021	***Comment***	jld03 PROTONNR CDCl3 u jld 3			***Date***	21 Jul 2005 11:56:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld03_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



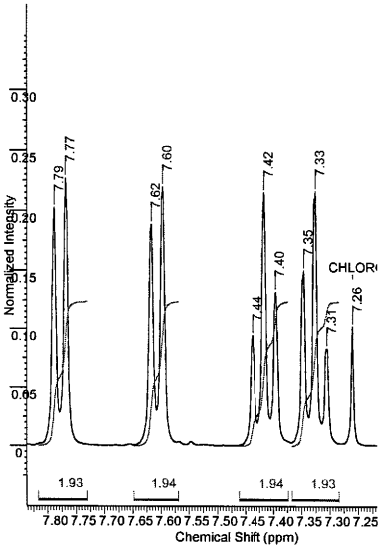
jld03, C13

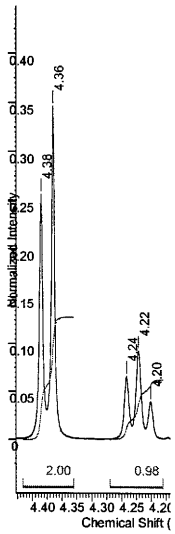
Acquisition Time (sec)	1.5139	***Comment***	jld03, C13 C13CPD CDCl3 u jld 3			***Date***	21 Jul 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld03-C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1625.50
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9030.6260
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				

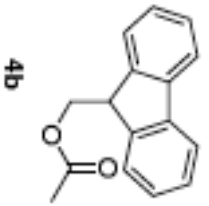


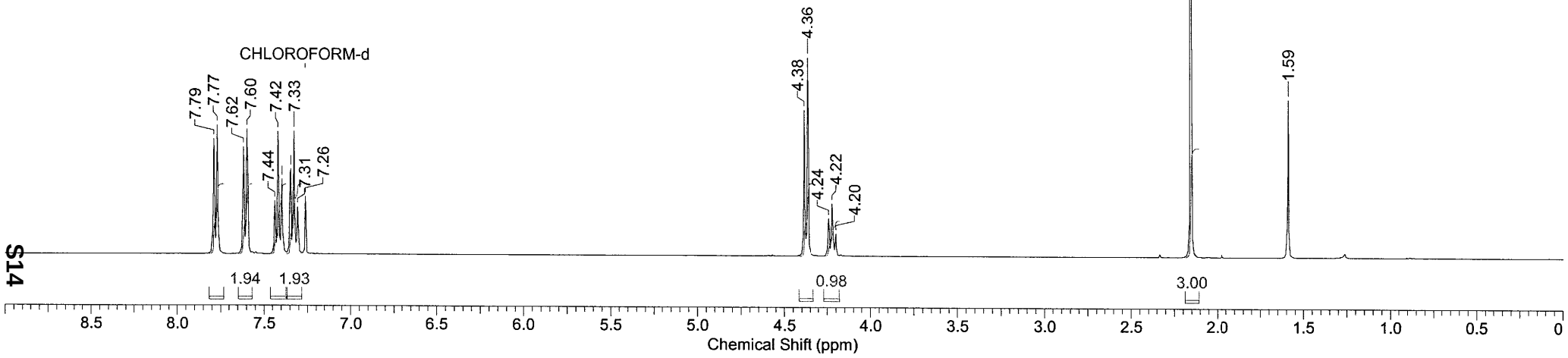
jld05-1

Acquisition Time (sec)	2.2021	***Comment***	jld05-1p pur PROTONNR CDCl3 u jld 9			***Date***	25 Jul 2005 12:07:28
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld05-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	203.20
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



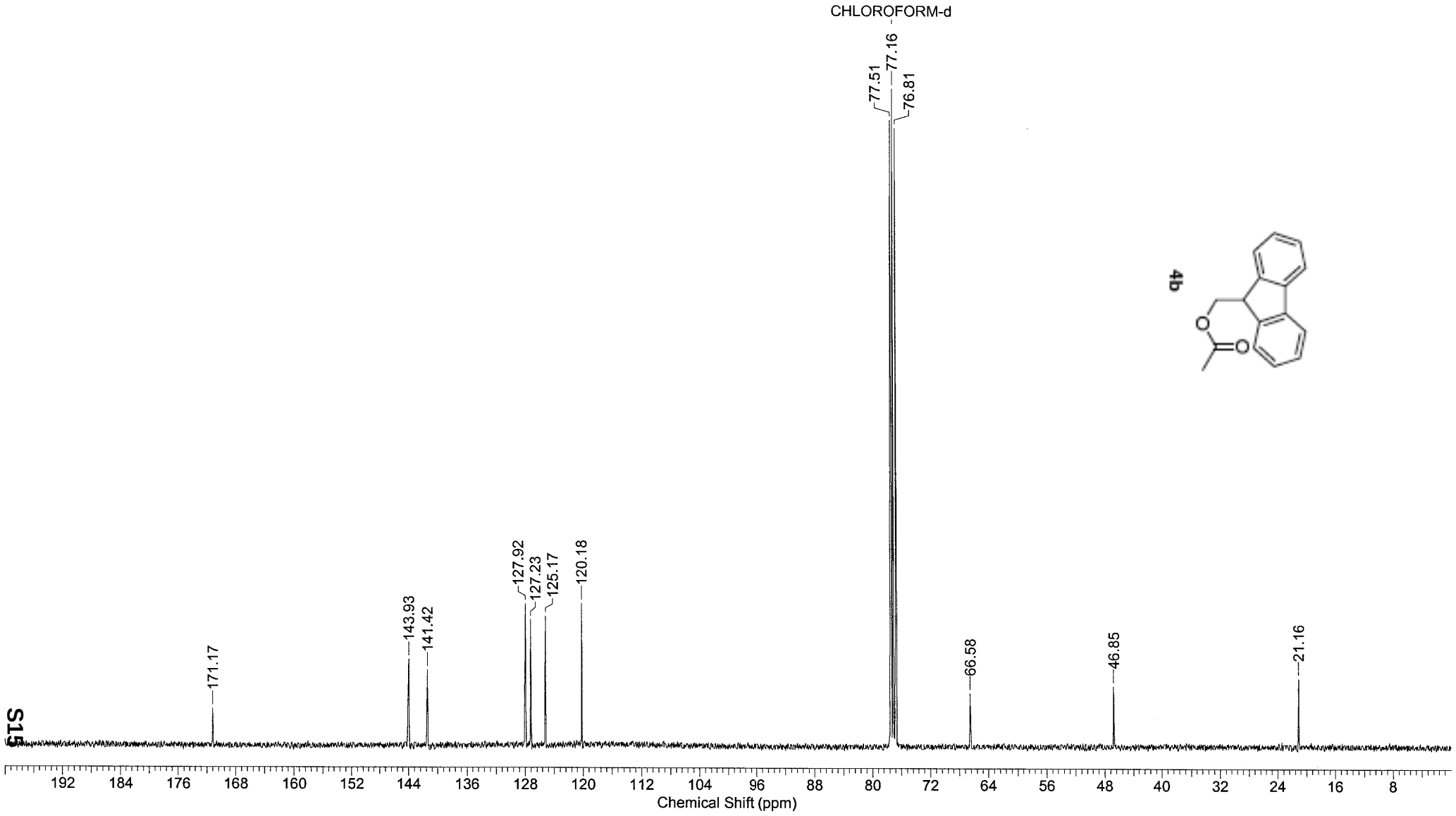






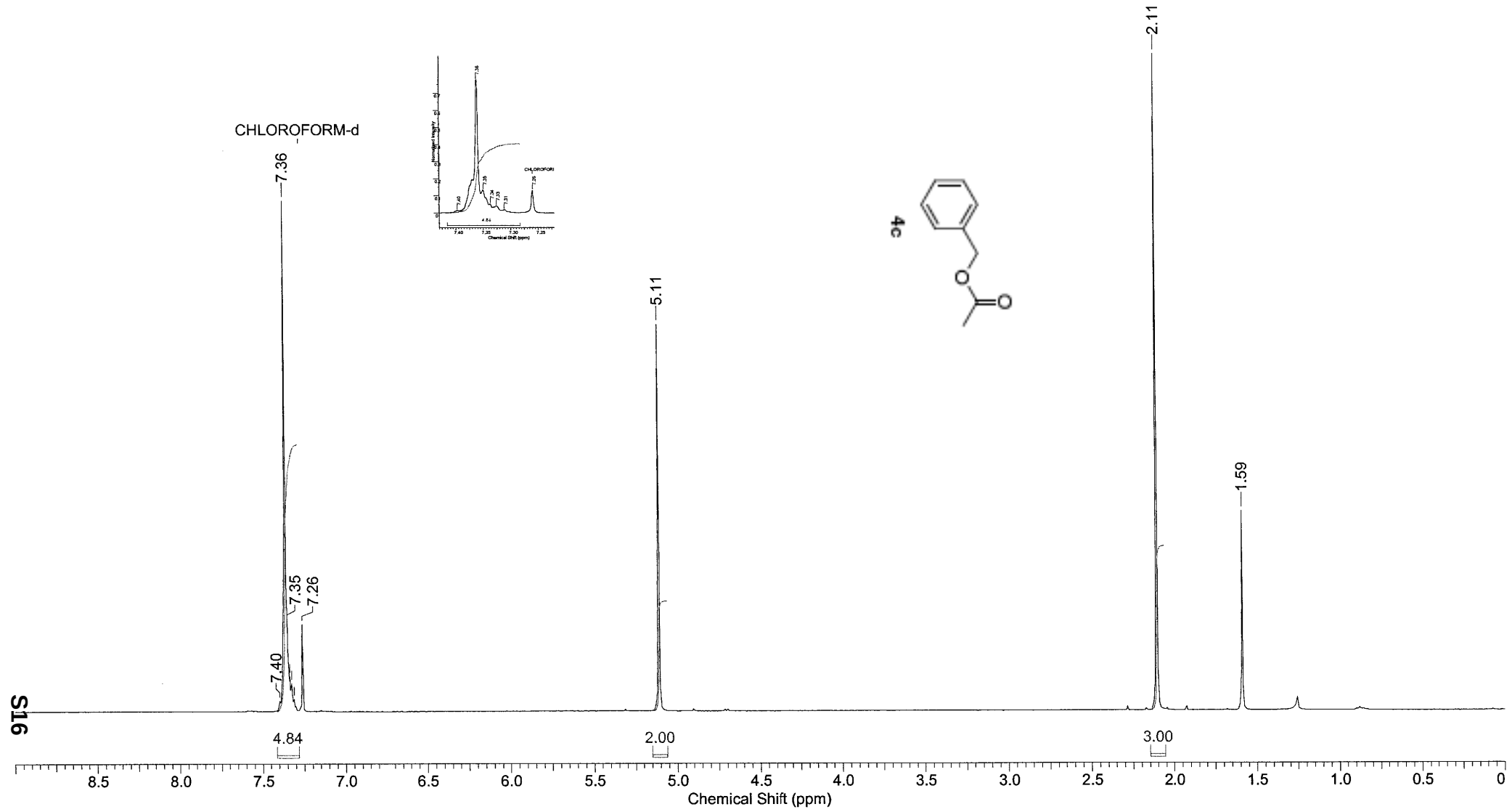
jld05-2

Acquisition Time (sec)	1.5139	***Comment***	jld05-1, C13 C13CPD CDCl3 u jld 9			***Date***	25 Jul 2005 23:19:28
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld05-1C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	2048	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1625.50
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9028.6436
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



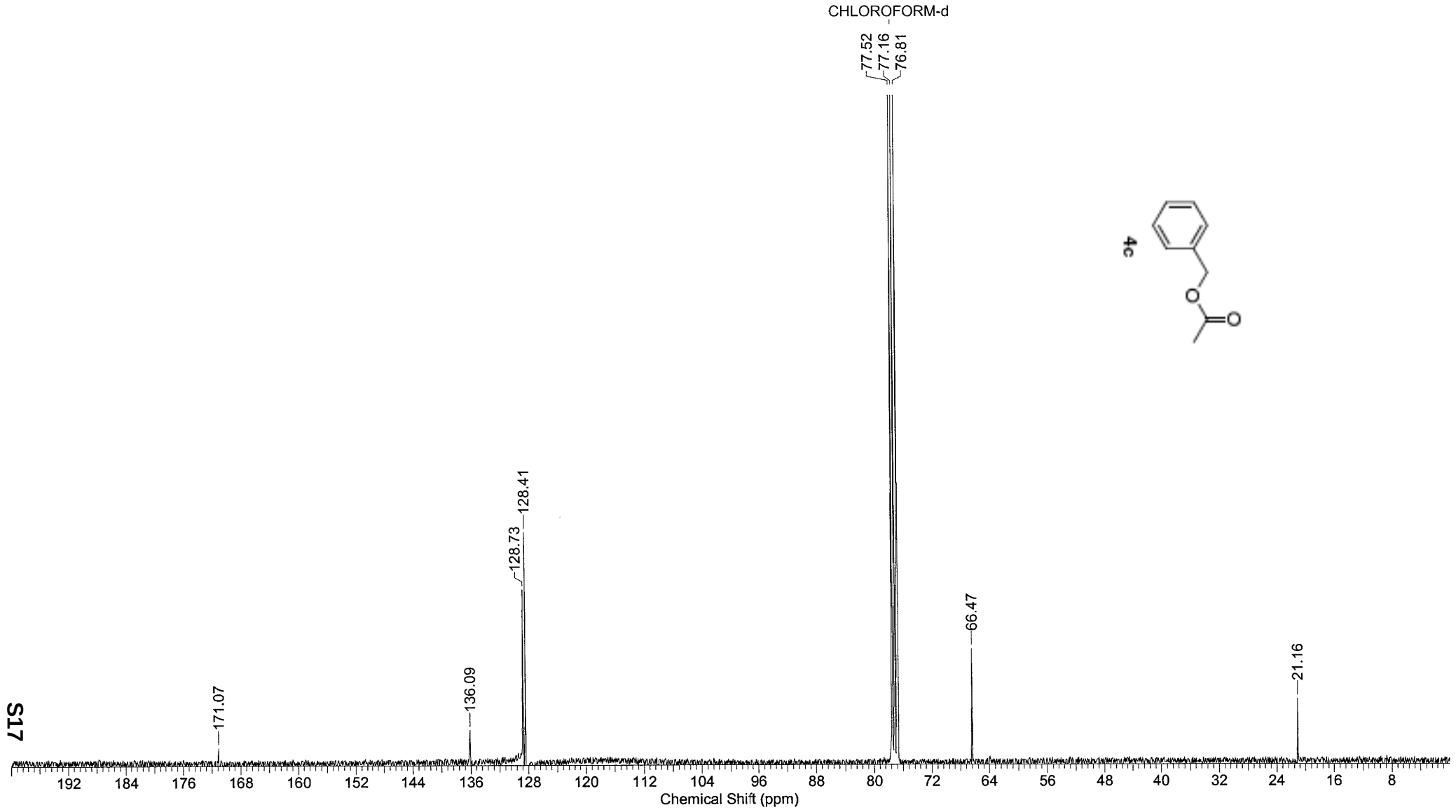
jld06

Acquisition Time (sec)	2.2021	***Comment***	jld06-2 PROTONNR CDCl3 u jld 6			***Date***	28 Jul 2005 08:12:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld06-2_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	256.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



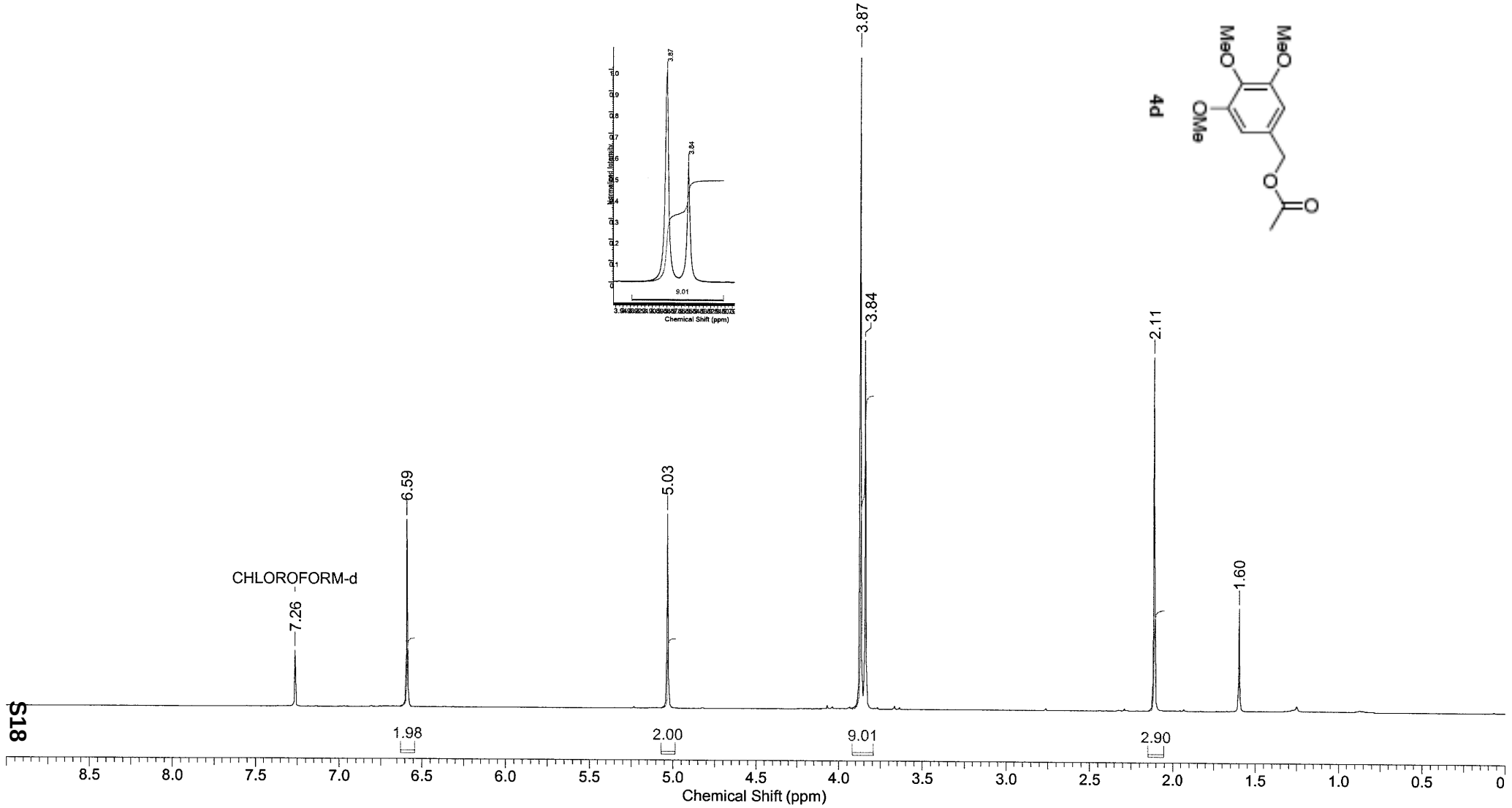
jld06, C13

Acquisition Time (sec)	1.5139	***Comment***	jld06-2, C13 C13CPD CDCl3 u jld 6			***Date***	28 Jul 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld06-2_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2896.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9030.6260
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



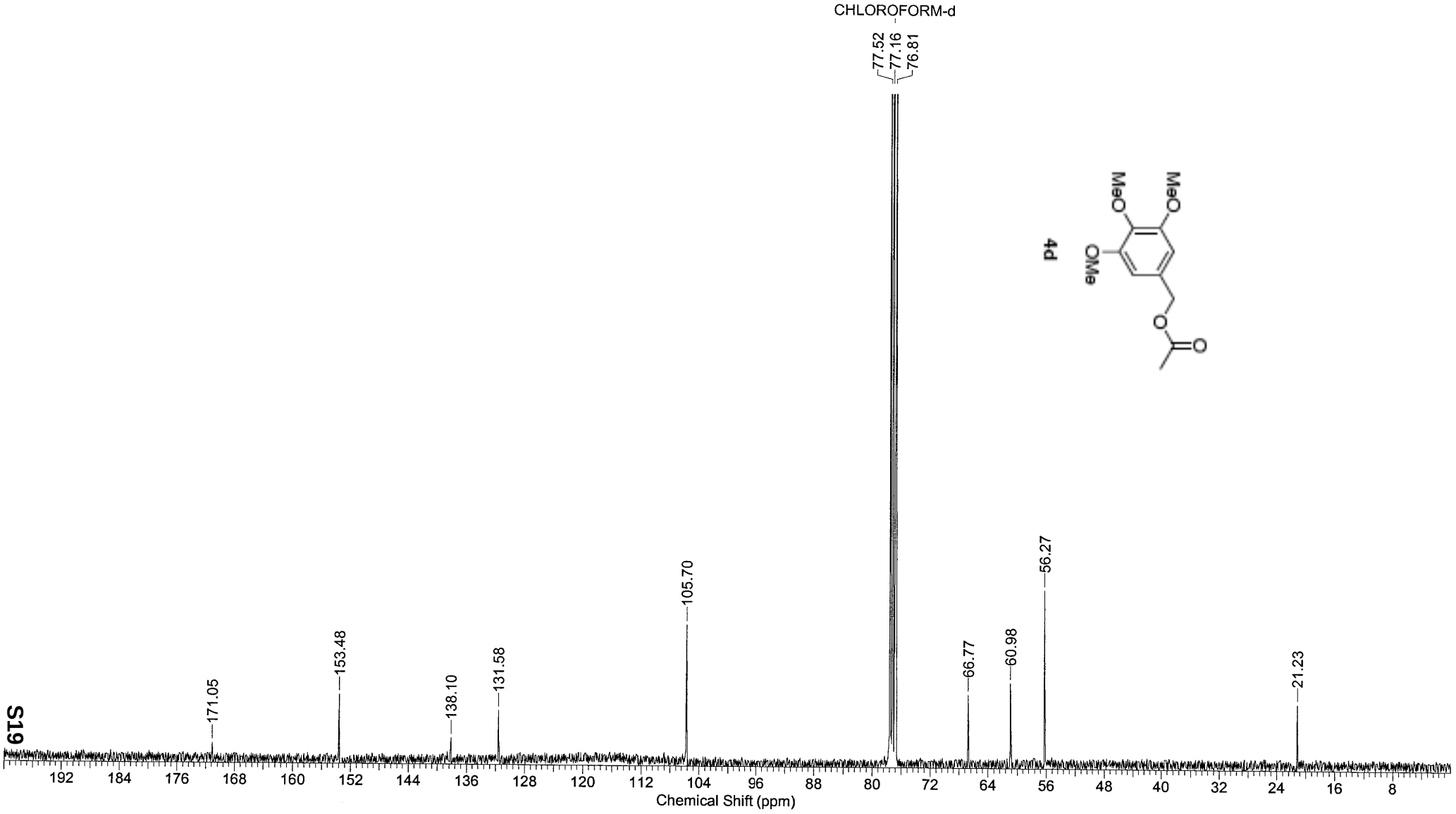
jld07

Acquisition Time (sec)	2.2021	***Comment***	jld07-1 PROTONNR CDCl3 u jld 7			***Date***	29 Jul 2005 06:28:16
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld07-1_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	228.10
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



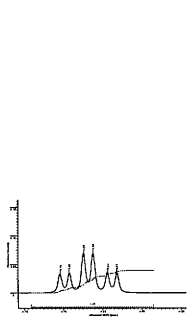
jld07, C13

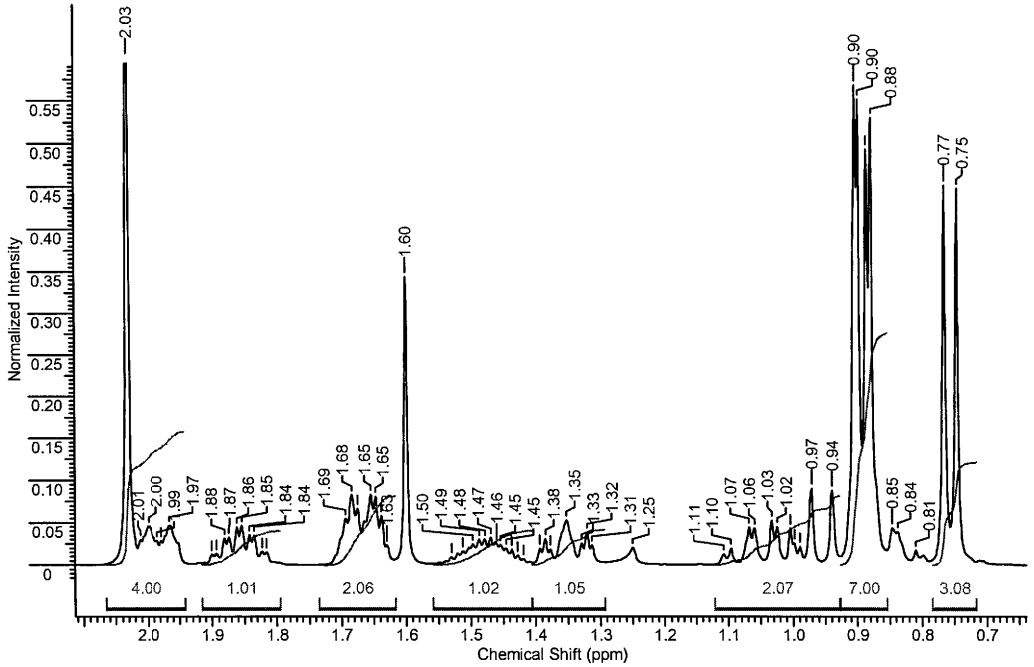
Acquisition Time (sec)	1.5139	***Comment***	jld07-1, C13 C13CPD CDCl3 u jld 7			***Date***	30 Jul 2005 01:12:32
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld07-1_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1448.20
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9029.9648
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				

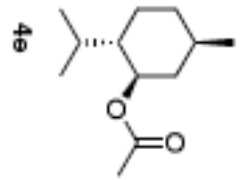


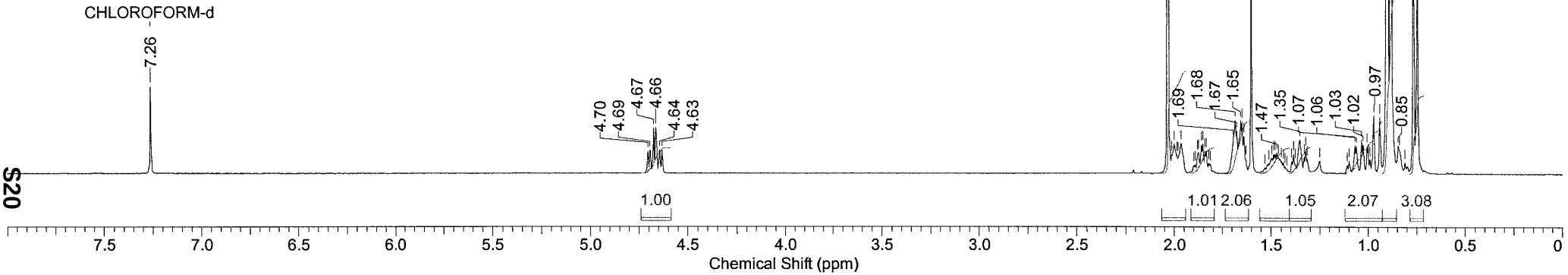
jld10

Acquisition Time (sec)	2.2021	***Comment***	jld10 PROTONNR CDCl3 u jld 8			***Date***	06 Aug 2005 08:12:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld10_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	143.70
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



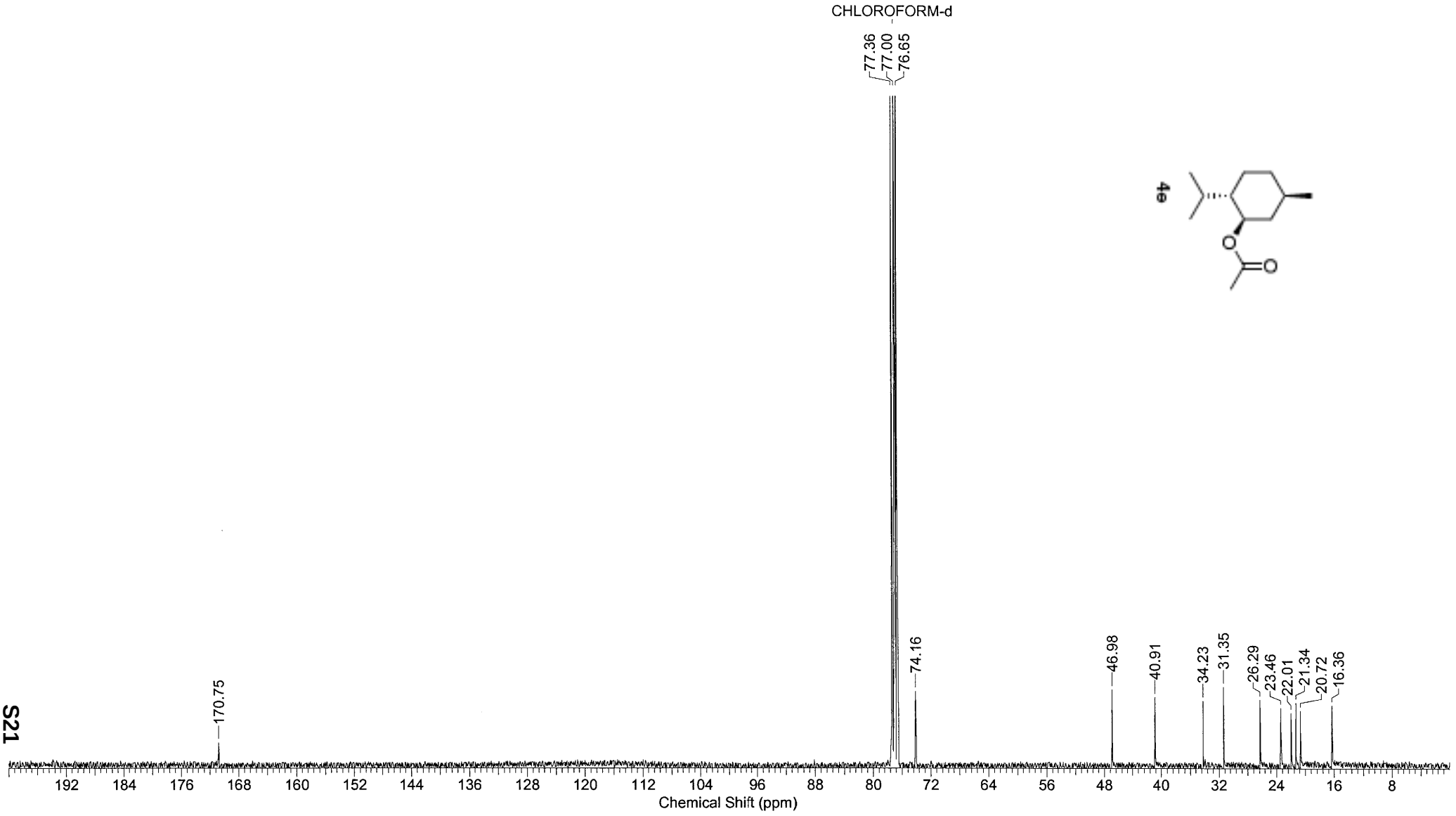






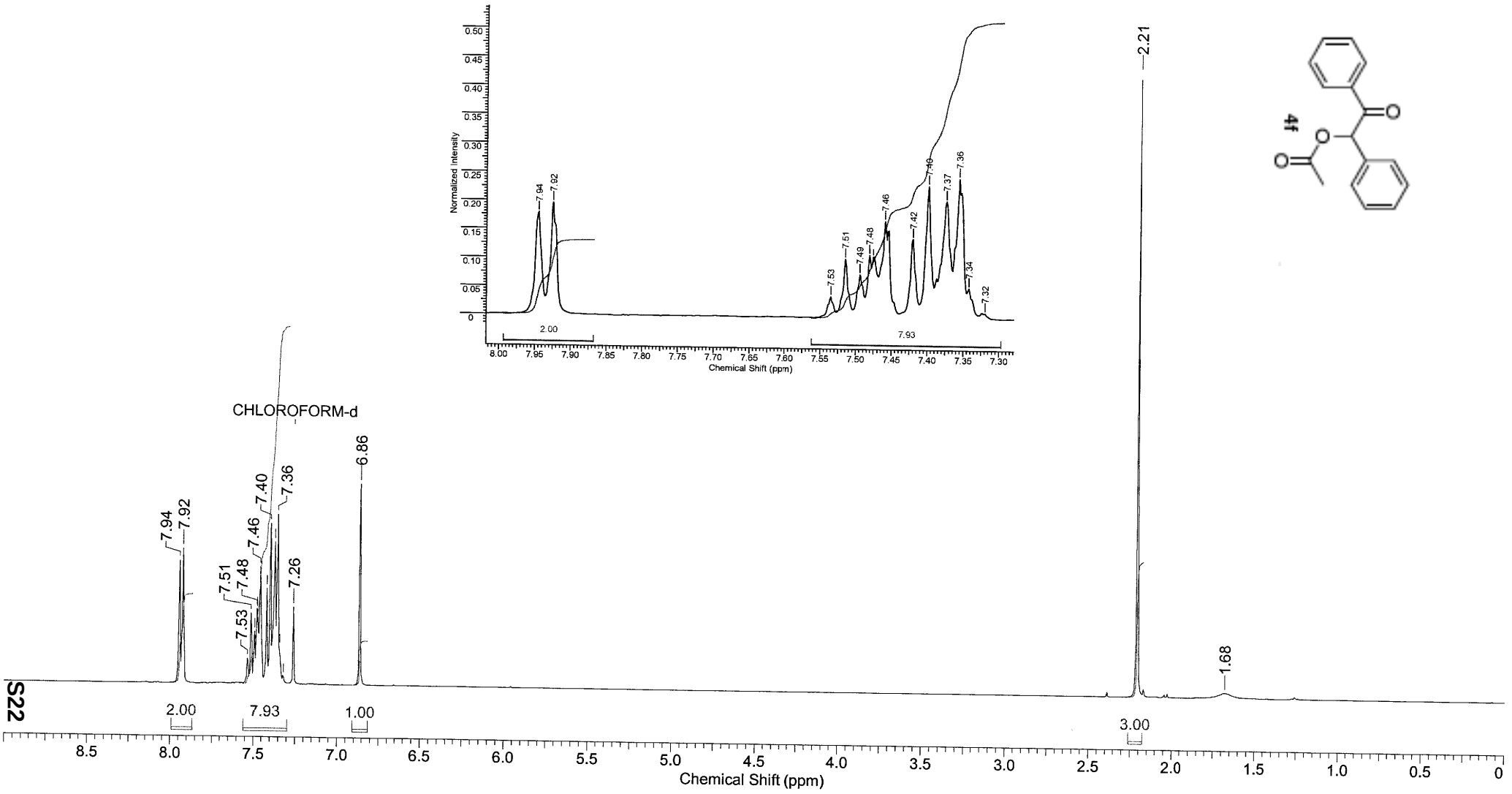
jld10, C13

Acquisition Time (sec)	1.5139	***Comment***	jld10, C13 C13CPD CDCl3 u jld 8			***Date***	06 Aug 2005 11:18:24
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld10_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	3072	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1824.60
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9016.1367
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



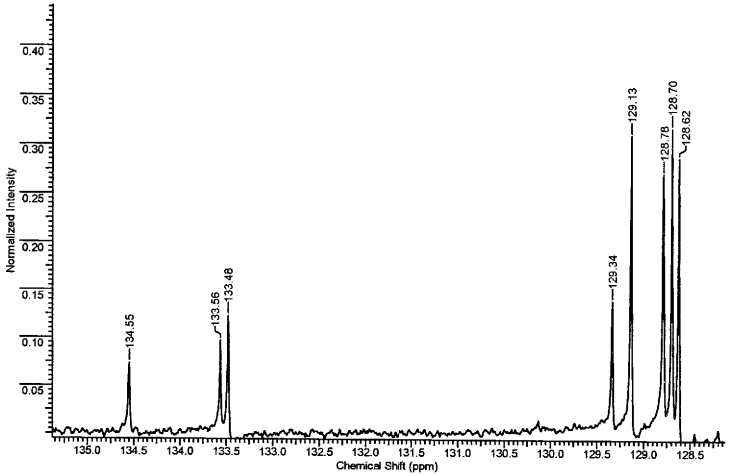
jld11-1

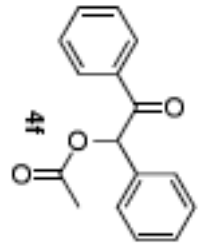
Acquisition Time (sec)	2.2021	***Comment***	jld11-1 PROTONNR CDCl3 u jld 11			***Date***	09 Aug 2005 11:50:24
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld11-1_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	181.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



jld11, C13

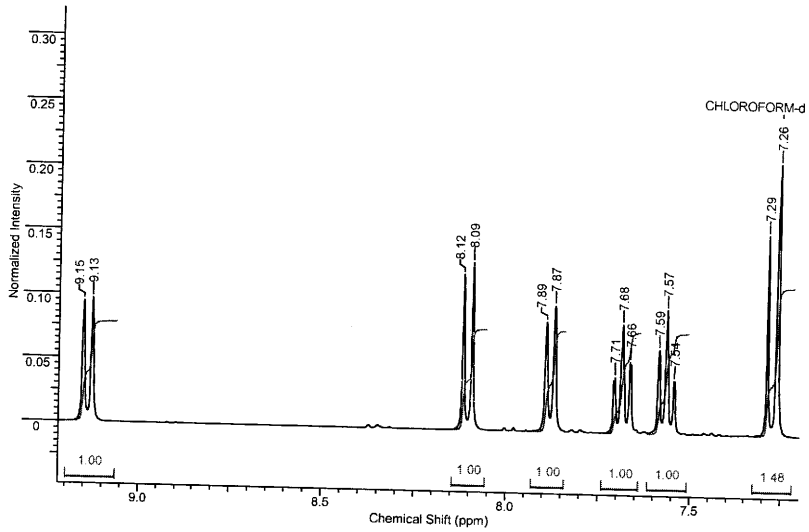
Acquisition Time (sec)	1.5139	***Comment***	jld11, C13 C13CPD CDCl3 u jld 11			***Date***	10 Aug 2005 21:05:04
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld11-1_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	3072	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	4096.00
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9014.8154
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				

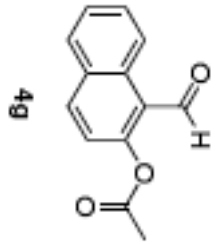




jld12

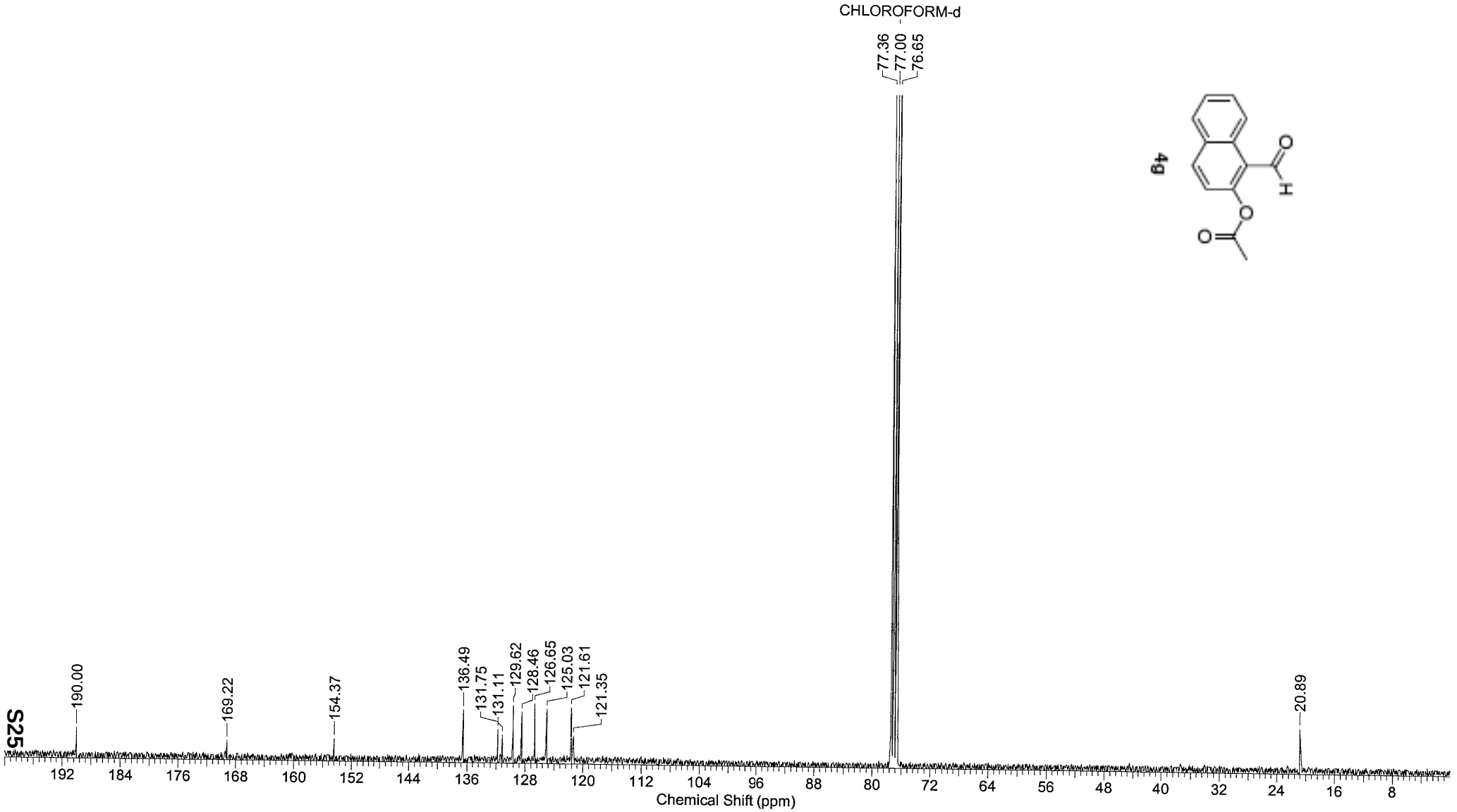
Acquisition Time (sec)	2.2021	***Comment***	jld12-2, pur PROTONNR CDCl3 u jld 12			***Date***	13 Aug 2005 09:36:00
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld12-2p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	362.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				





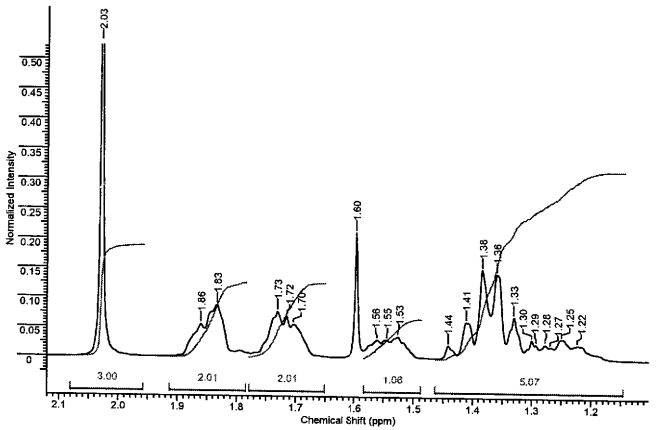
jld12, C13

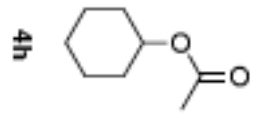
Acquisition Time (sec)	1.5139	***Comment***	jld12-2, pur C13CPD CDCl3 u jld 12			***Date***	13 Aug 2005 12:45:52
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld12_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	3072	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2896.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



jld13

Acquisition Time (sec)	2.2021	***Comment***	jld13-2, pur PROTONNR CDCl3 u jld 13			***Date***	18 Aug 2005 09:57:20
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld13-2p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	228.10
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				

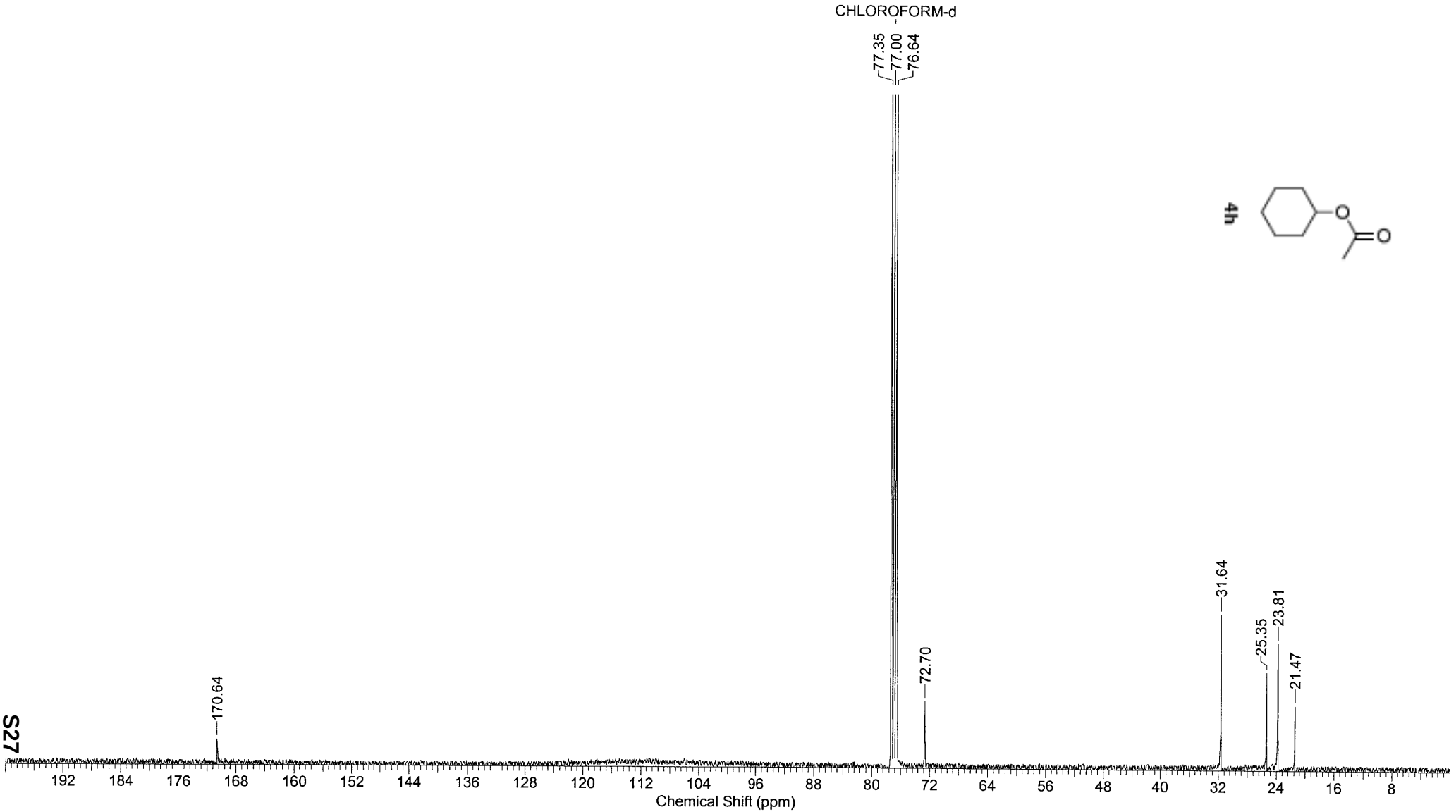




4h

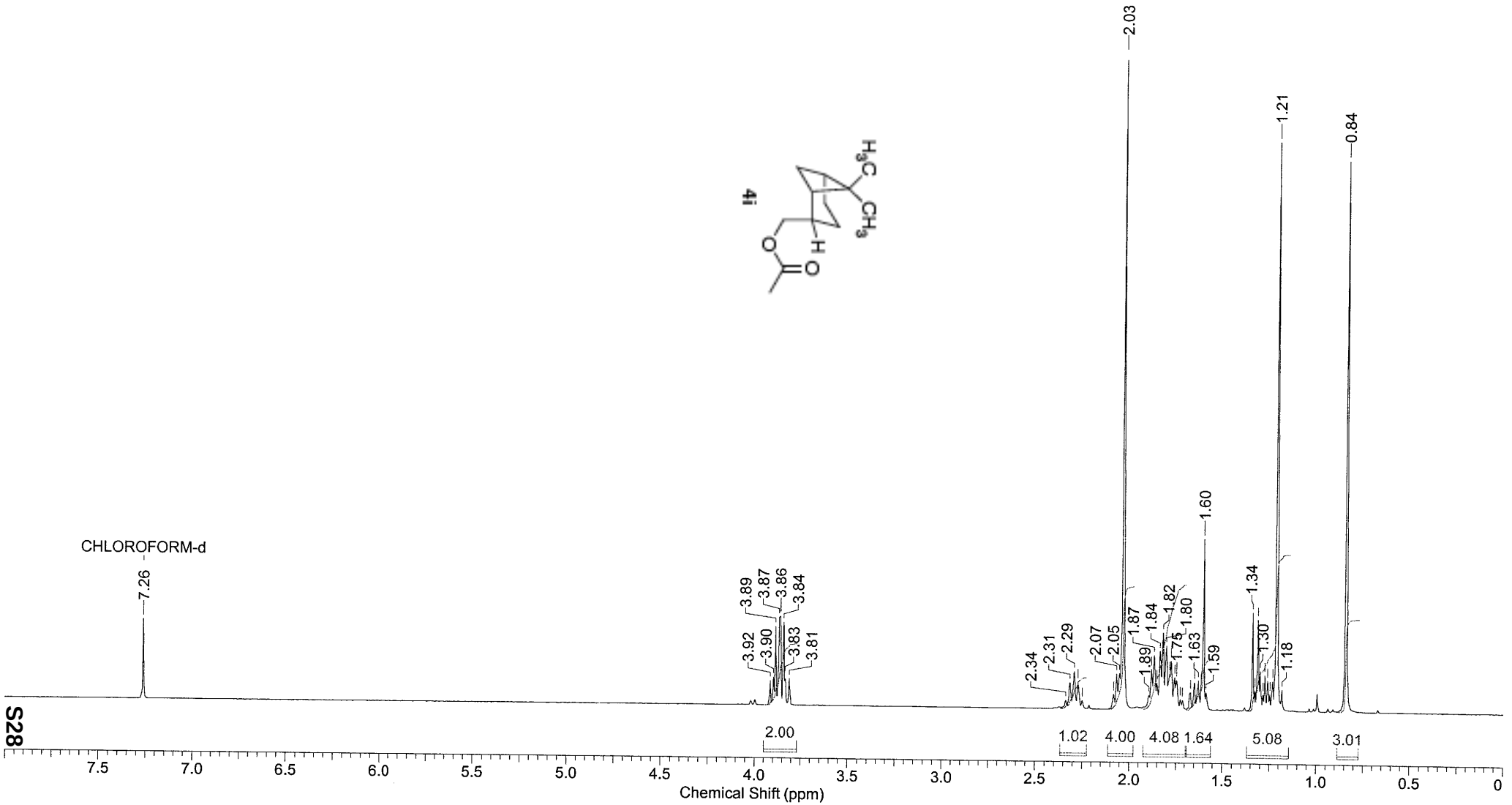
jld13, C13

Acquisition Time (sec)	1.5139	***Comment***	jld13, C13 C13CPD CDCl3 u jld 13			***Date***	18 Aug 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld13_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2896.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



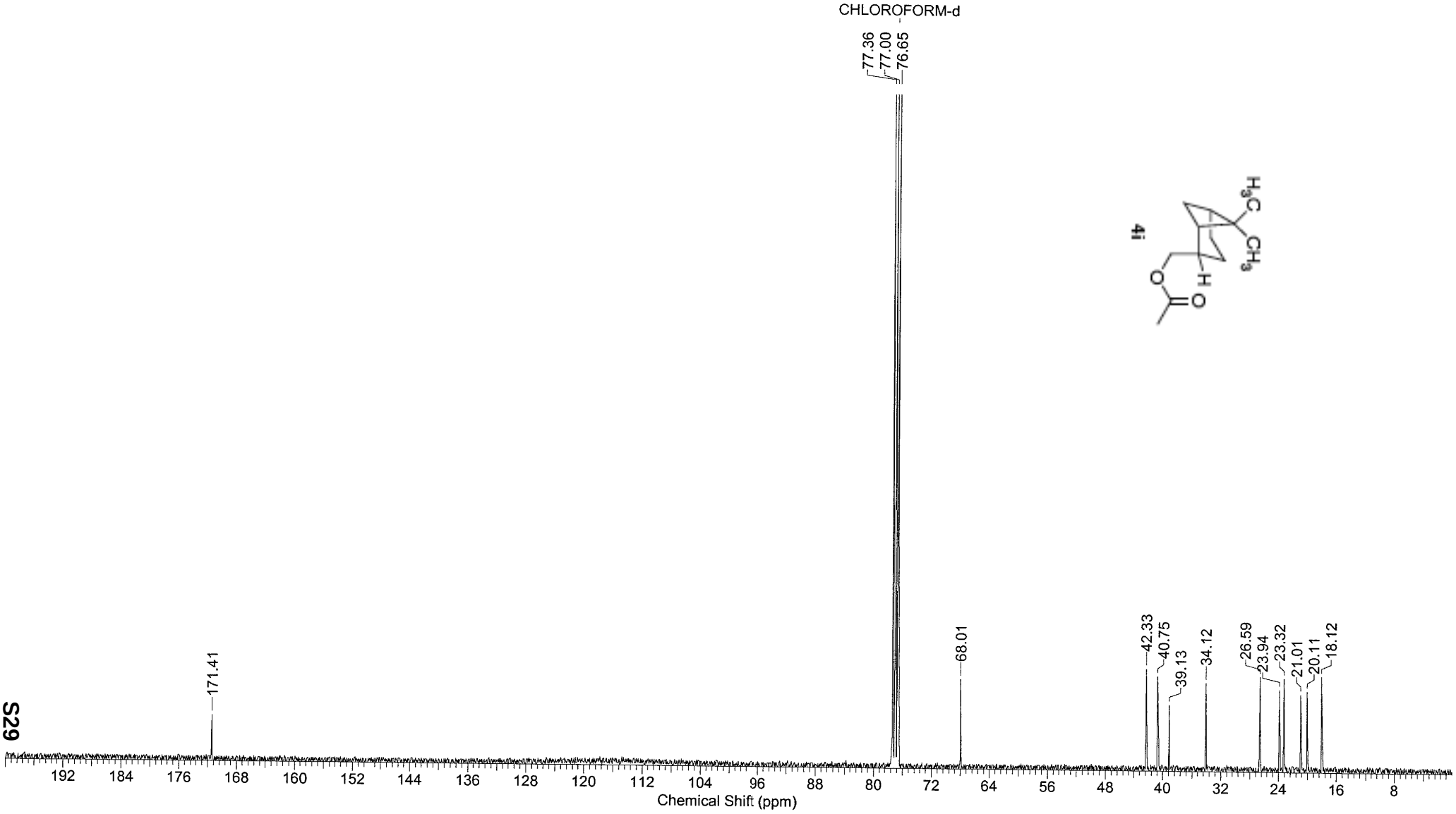
jld14

Acquisition Time (sec)	2.2021	***Comment***	jld14, pur PROTONNR CDCl3 u jld 12			***Date***	19 Aug 2005 12:43:44
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld14-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5947
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



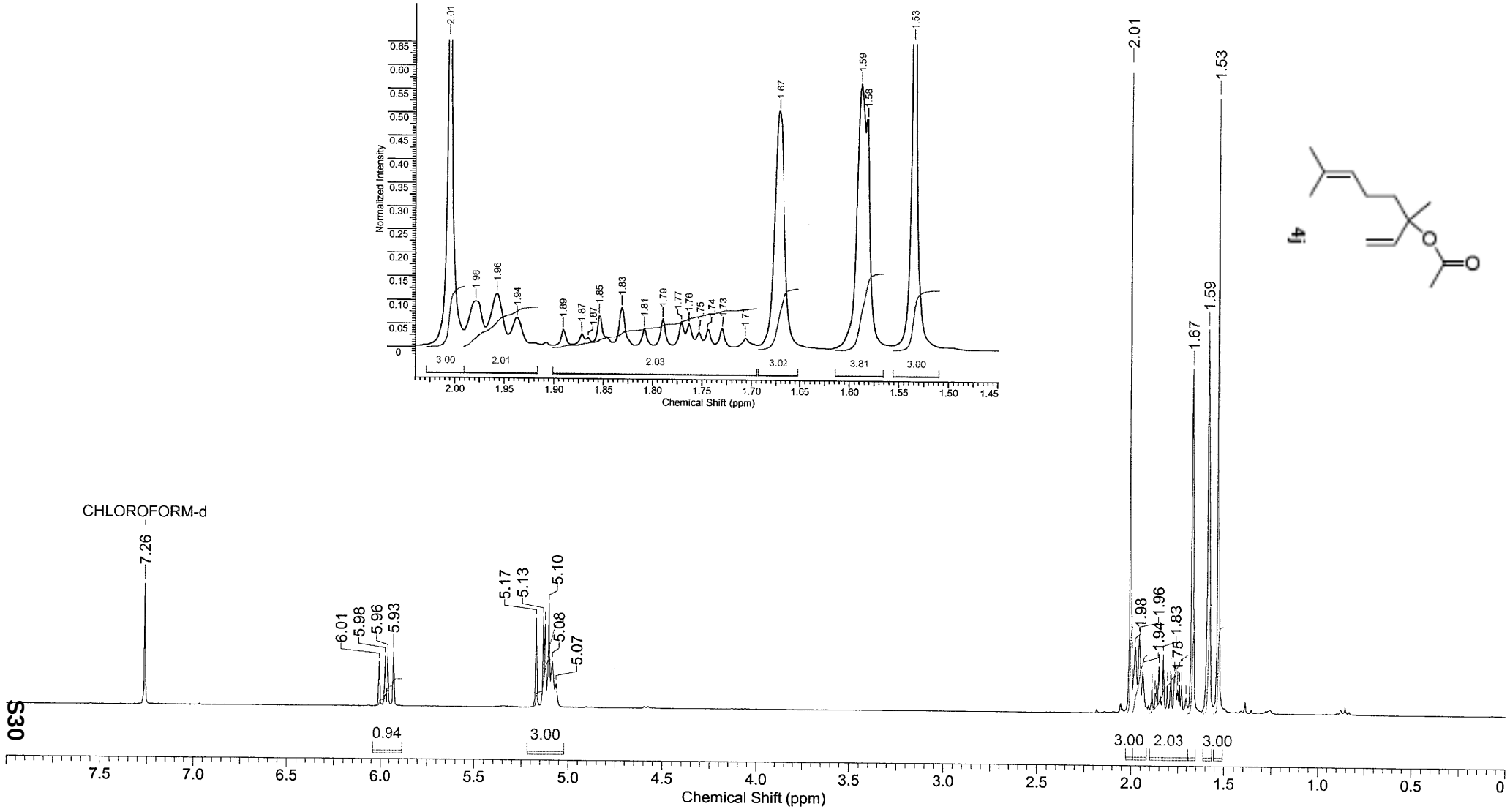
jld14, C13

Acquisition Time (sec)	1.5139	***Comment***	jld14_C13 C13CPD CDCl3 u jld 14			***Date***	19 Aug 2005 23:13:04
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld14_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	3072	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2580.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



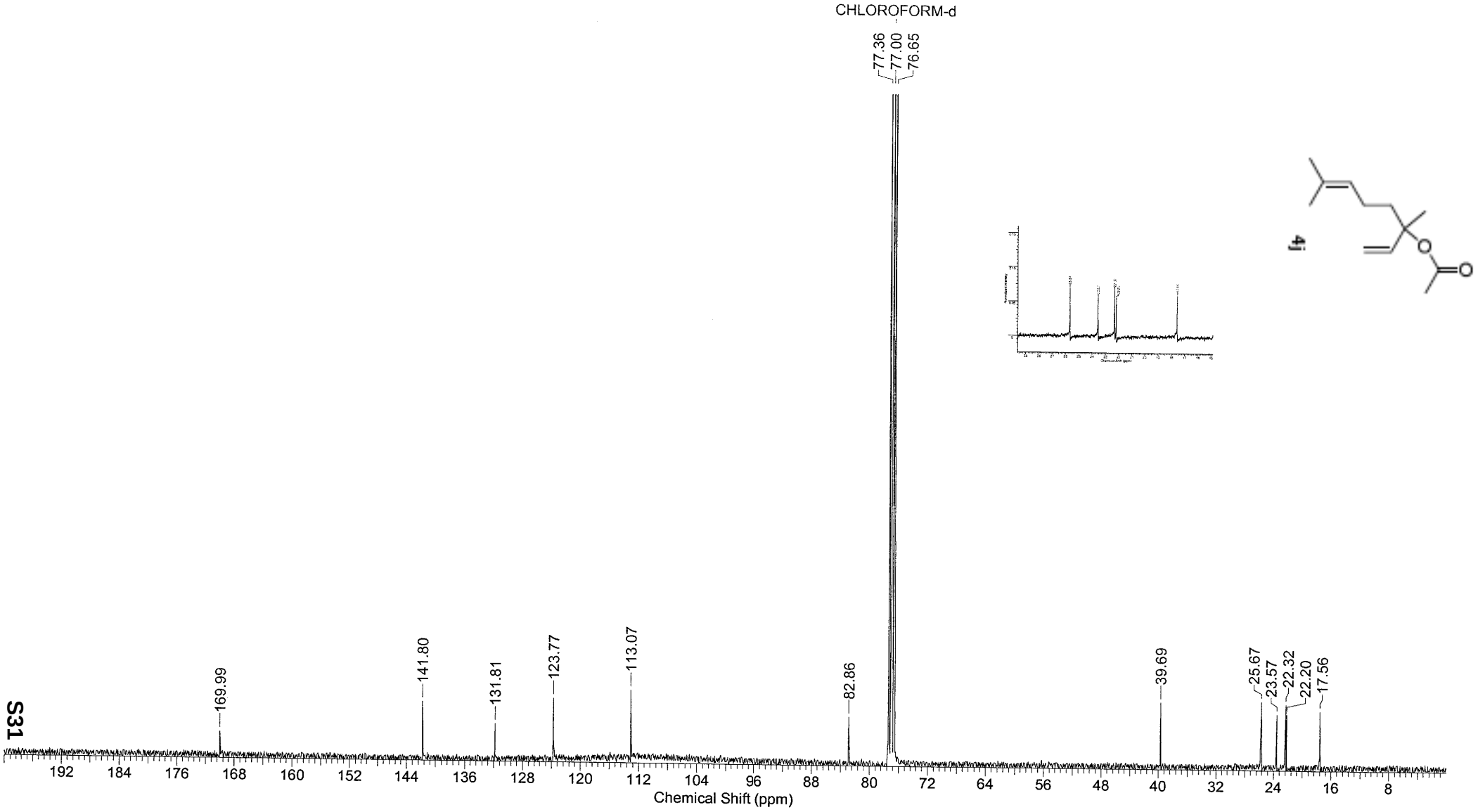
jld15-1

Acquisition Time (sec)	2.2021	***Comment***	jld15-1, pur PROTONNR CDCl3 u jld 15			***Date***	23 Aug 2005 09:23:12
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld15-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	203.20
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



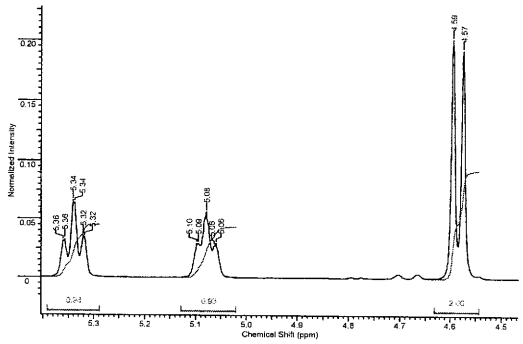
jld15, C13

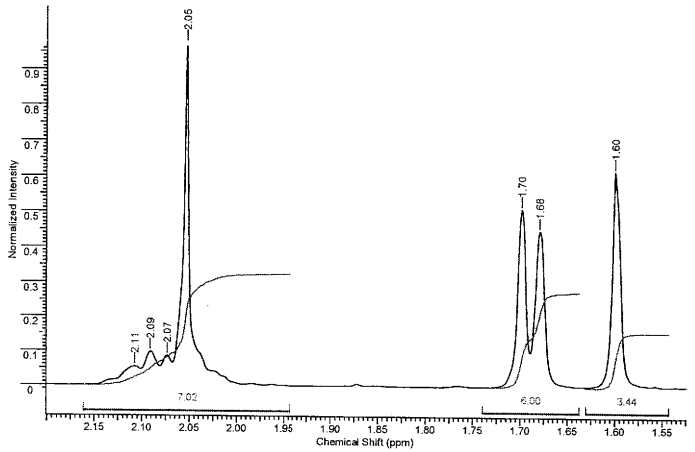
Acquisition Time (sec)	1.5139	***Comment***	jld15, C13 C13CPD CDCl3 u jld 15			***Date***	25 Aug 2005 02:12:16
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld15_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	3251.00
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				

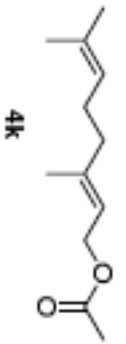


jld16

Acquisition Time (sec)	2.2021	***Comment***	jld16-1, pur PROTONNR CDCl3 u jld 16			***Date***	24 Aug 2005 10:59:12
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld16-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	128.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				

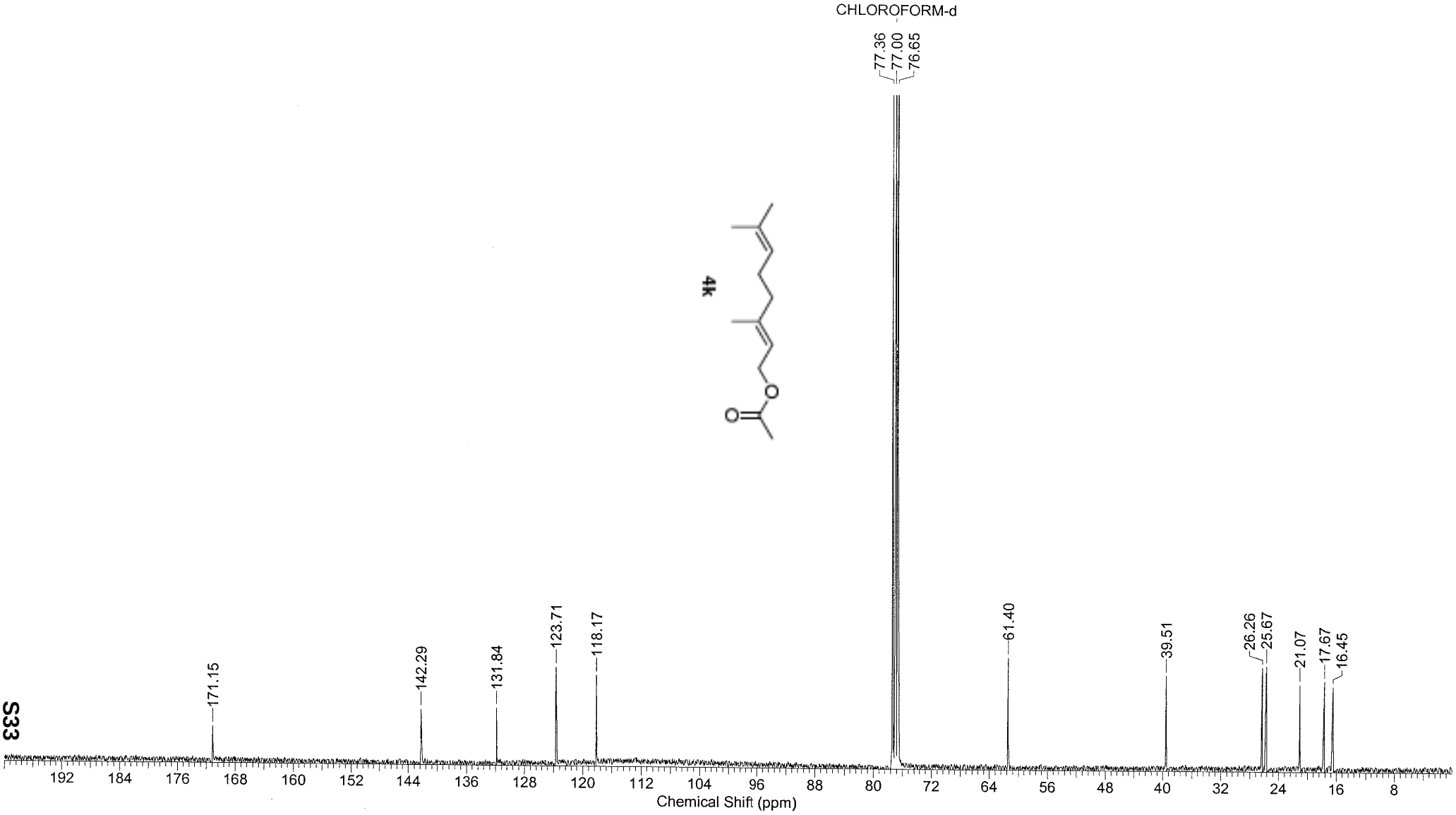






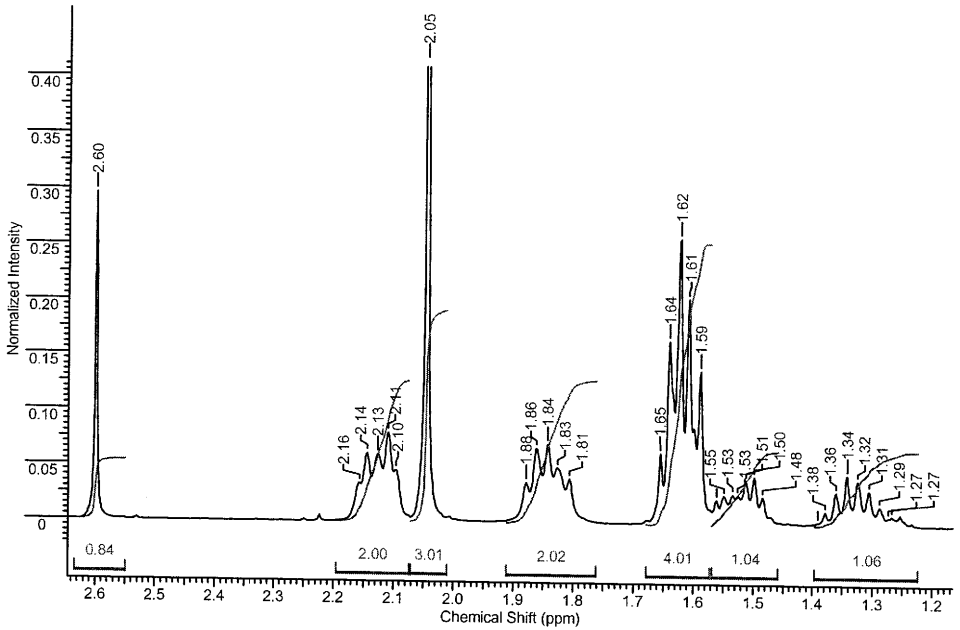
jld16, C13

Acquisition Time (sec)	1.5139	***Comment***	jld16, C13 C13CPD CDCl3 u jld 15			***Date***	25 Aug 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld16-1_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	4096.00
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4766
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



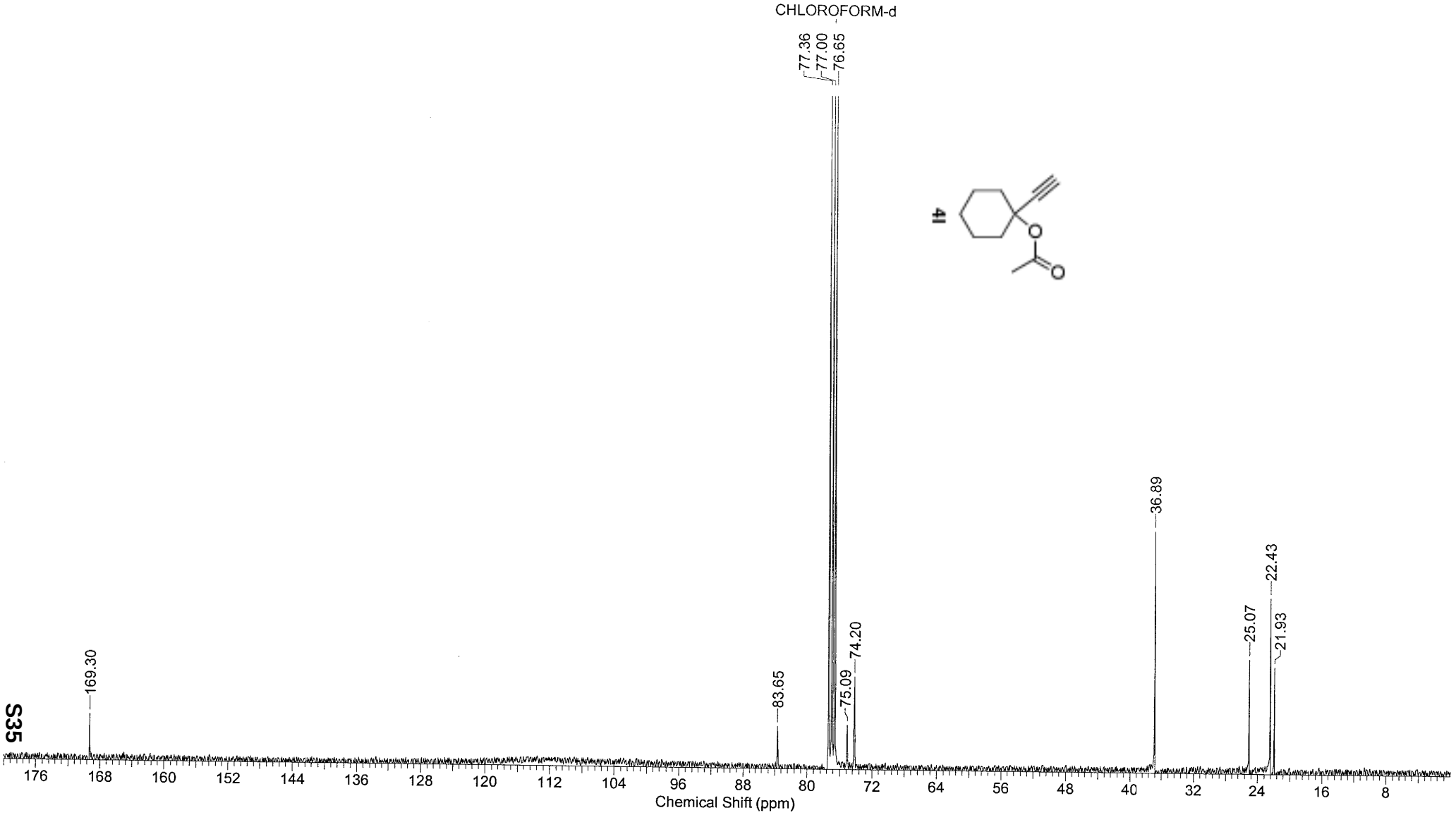
jld17

Acquisition Time (sec)	2.2021	***Comment***	jld17-1, pur PROTONNR CDCl3 u jld 16			***Date***	25 Aug 2005 12:18:08
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld17-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	161.30
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



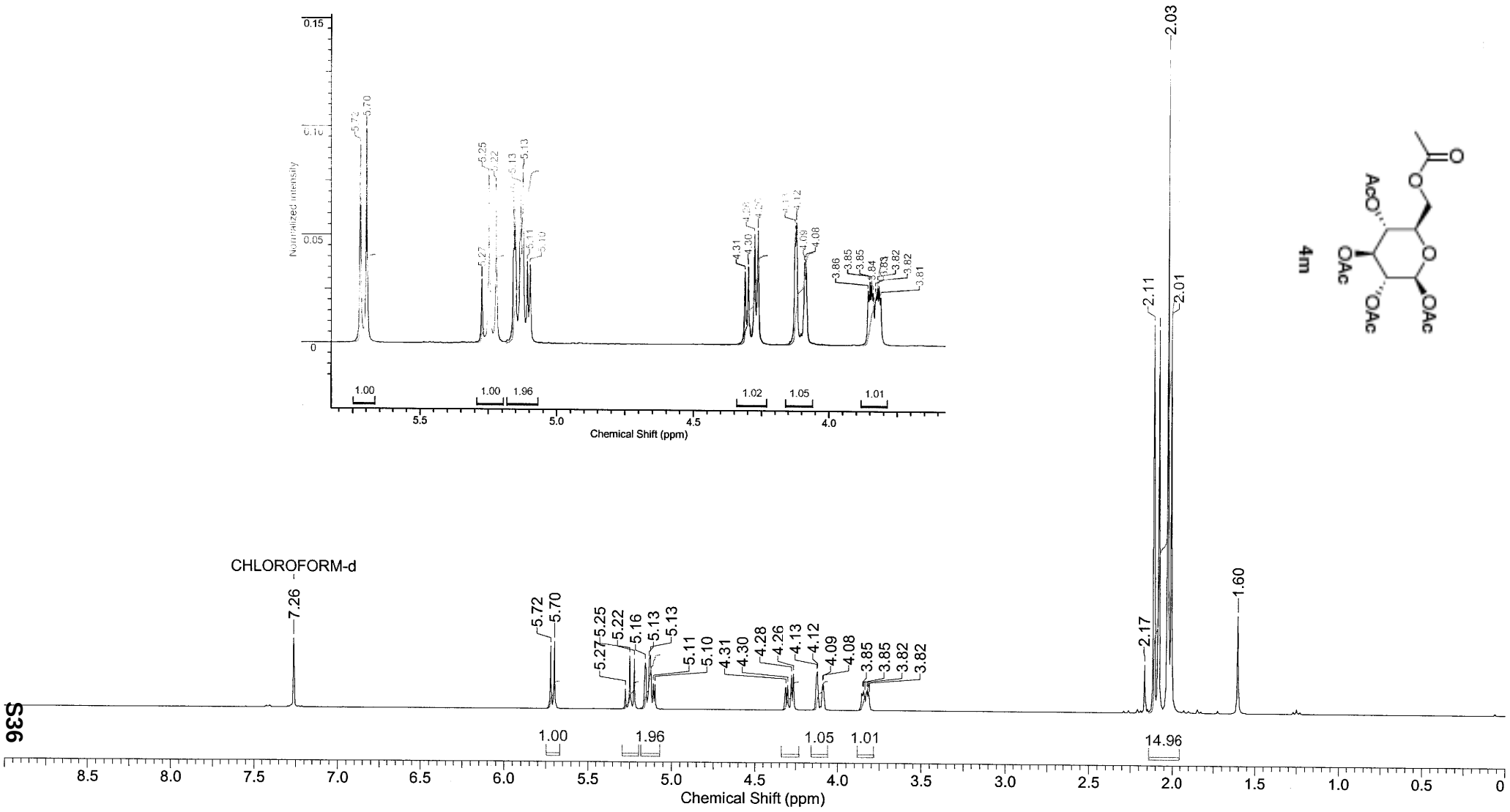
jld17, C13

Acquisition Time (sec)	1.5139	***Comment***	jld17, C13 C13CPD CDCl3 u jld 17			***Date***	26 Aug 2005 04:56:32
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld17_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1824.60
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



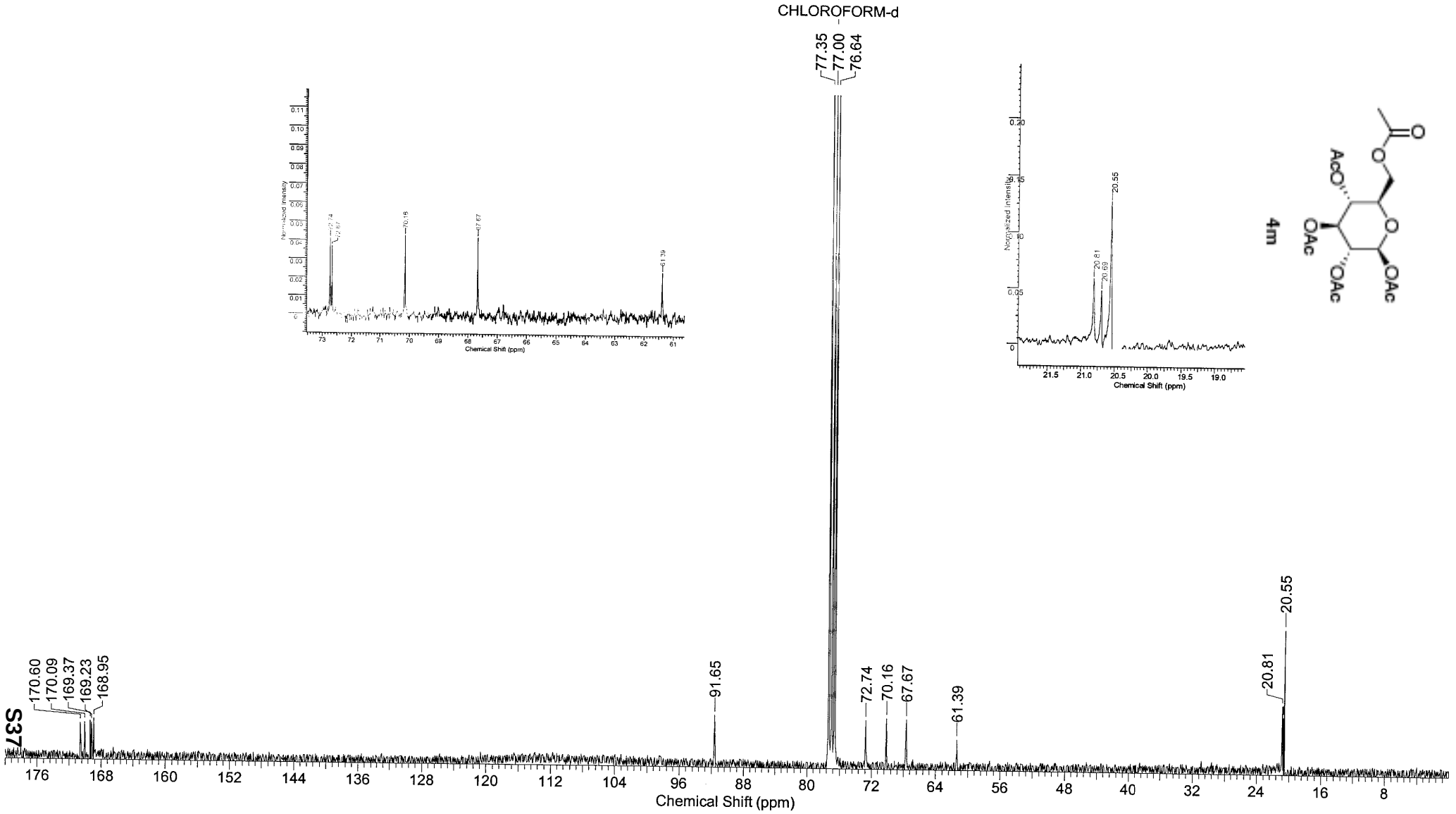
jld19

Acquisition Time (sec)	2.2021	***Comment***	jld19-2, pur PROTONNR CDCl3 u jld 19			***Date***	02 Sep 2005 15:30:08
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld19-2p2_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	203.20
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5947
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



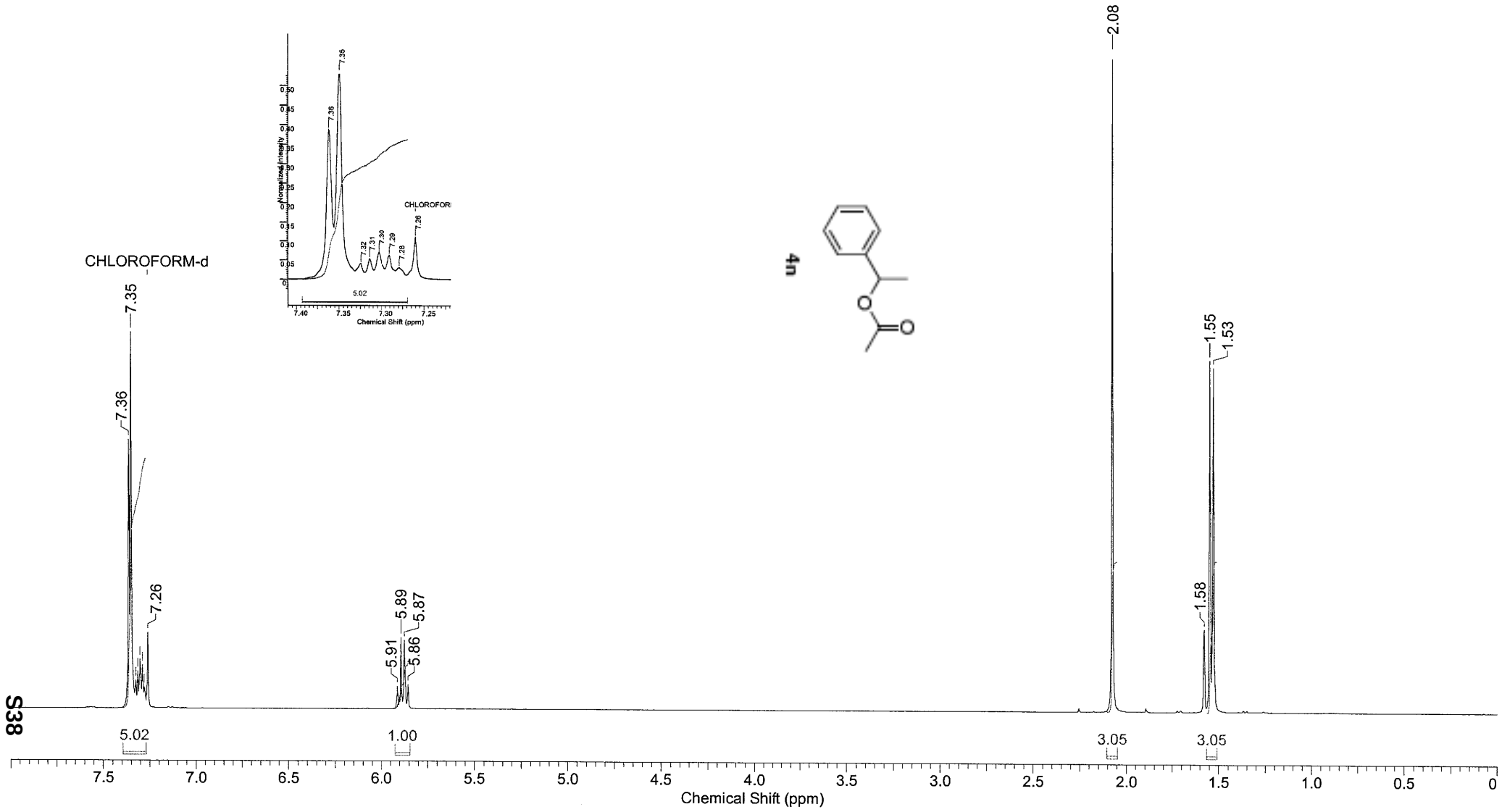
jld19, C13

Acquisition Time (sec)	1.5139	***Comment***	jld19, C13 C13CPD CDCl3 u jld 19			***Date***	03 Sep 2005 08:14:56
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld19_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	2048	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2580.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9014.8154
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



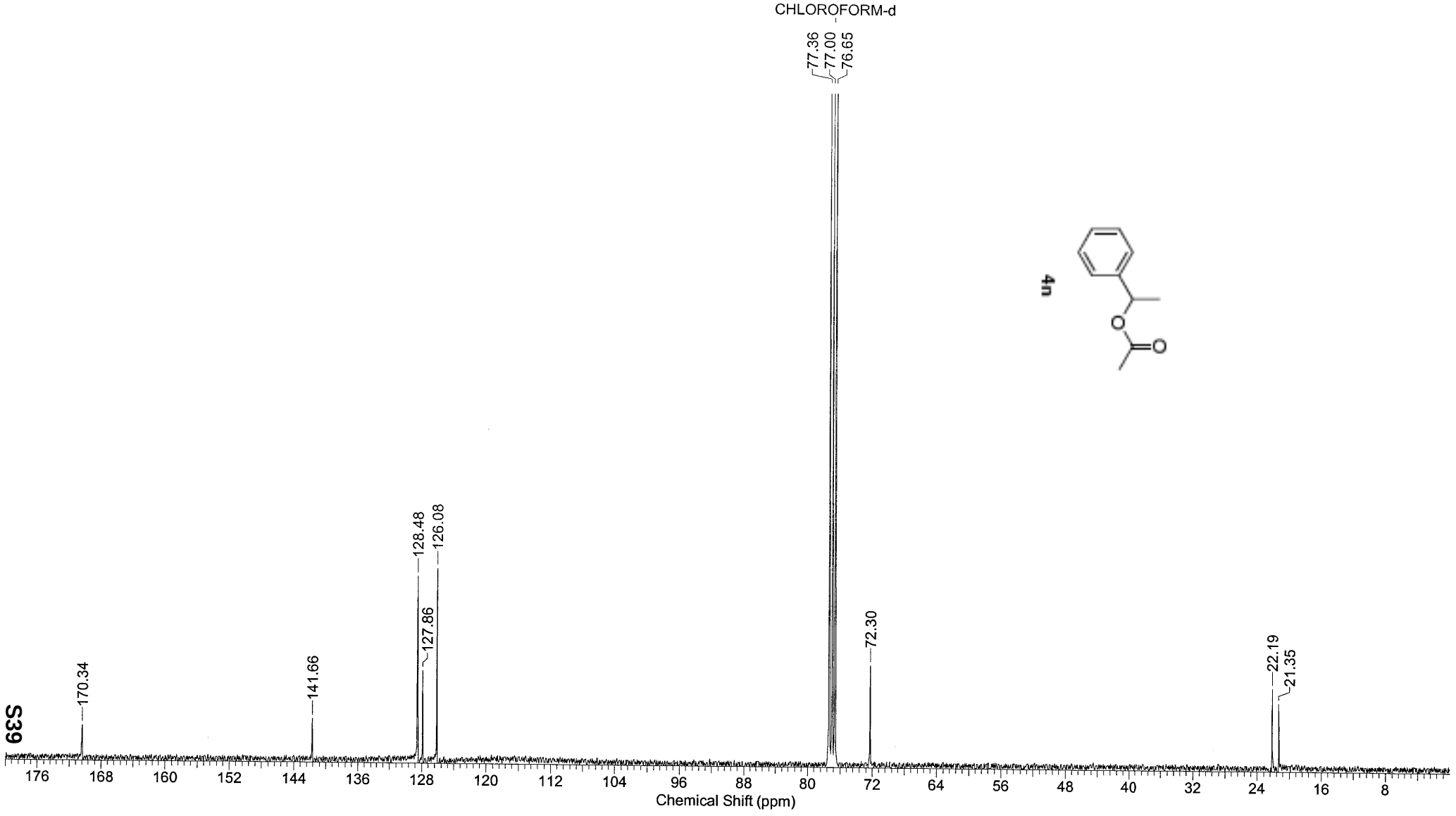
jld20

Acquisition Time (sec)	2.2021	***Comment***	jld20-1, pur PROTONNR CDCl3 u jld 20			***Date***	01 Sep 2005 09:44:32
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld20-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	228.10
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



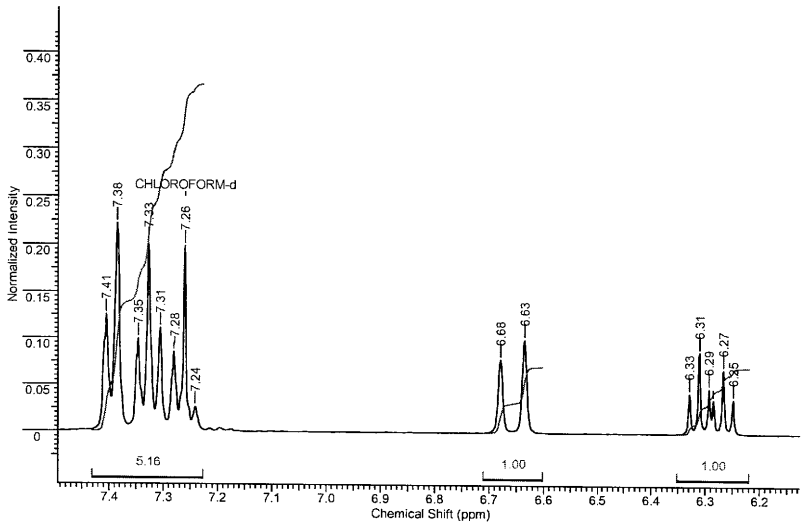
jld20, C13

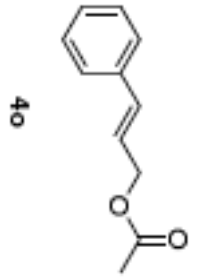
Acquisition Time (sec)	1.5139	***Comment***	jld20, C13 C13CPD CDCl3 u jld 20			***Date***	01 Sep 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld20_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	1824.60
SW(ppm) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9015.4756
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



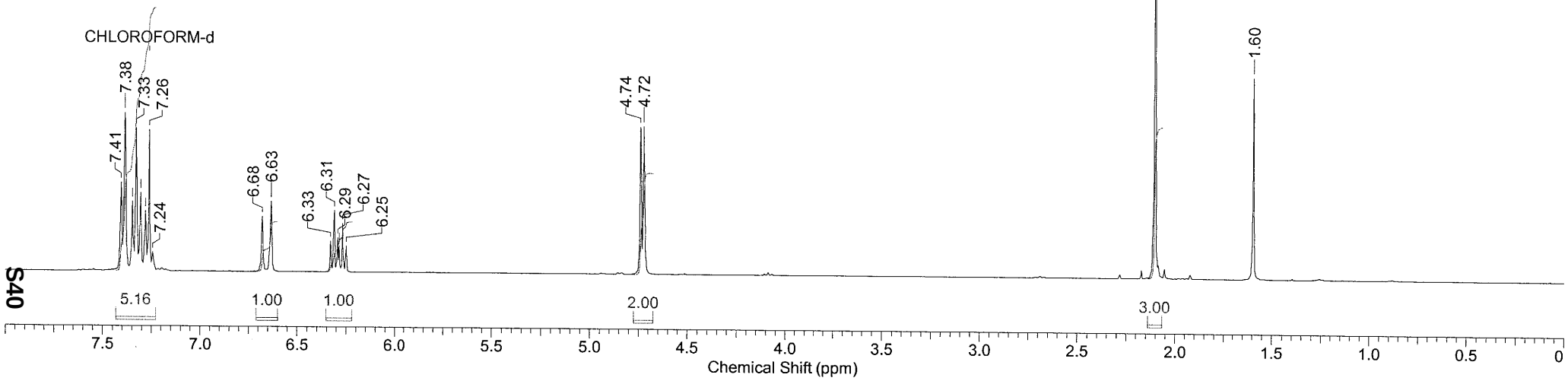
jld21

Acquisition Time (sec)	2.2021	***Comment***	jld21-1p PROTONNR CDCl3 u jld 26			***Date***	06 Sep 2005 10:44:16
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld21-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	203.20
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



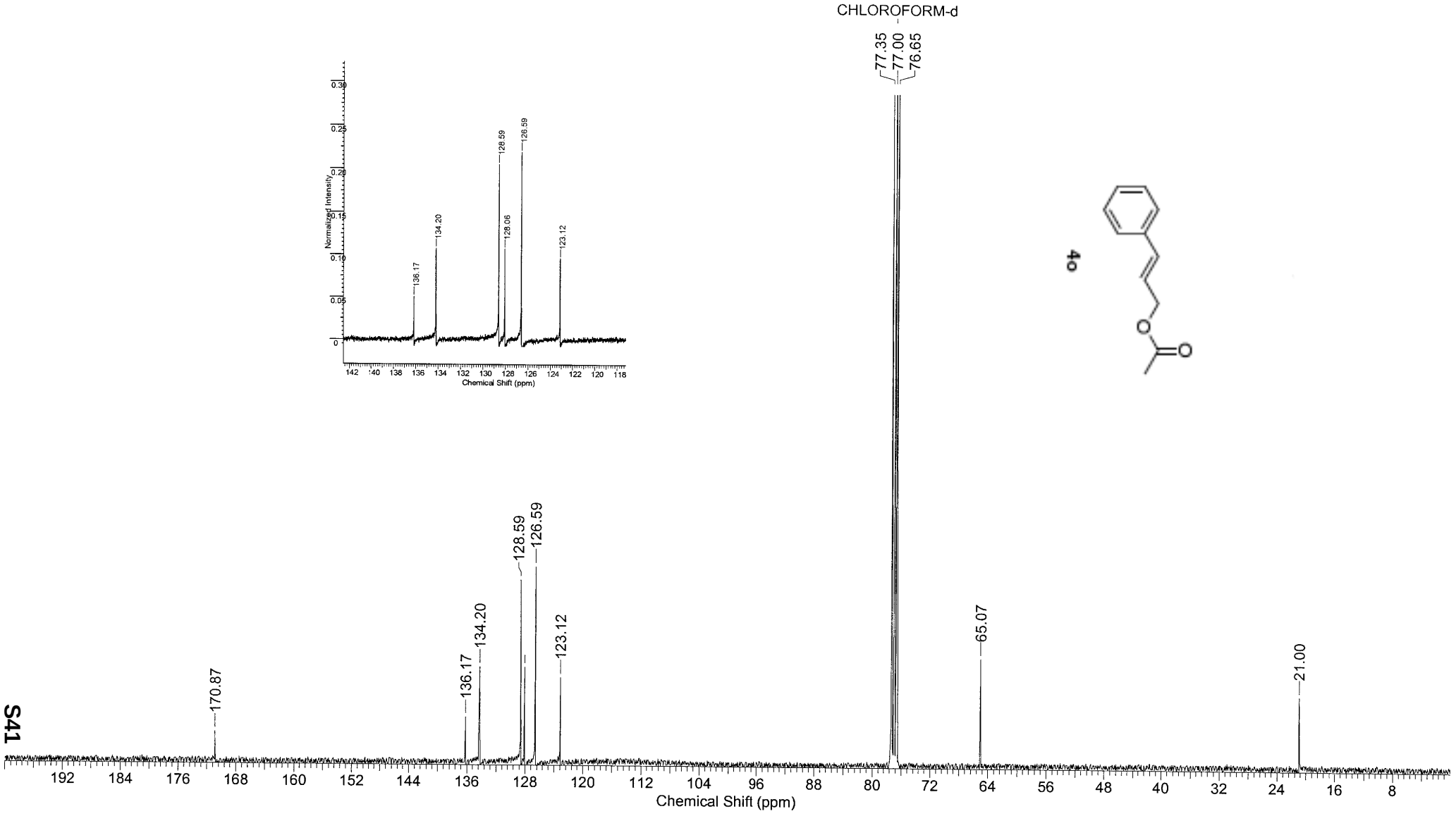


4o



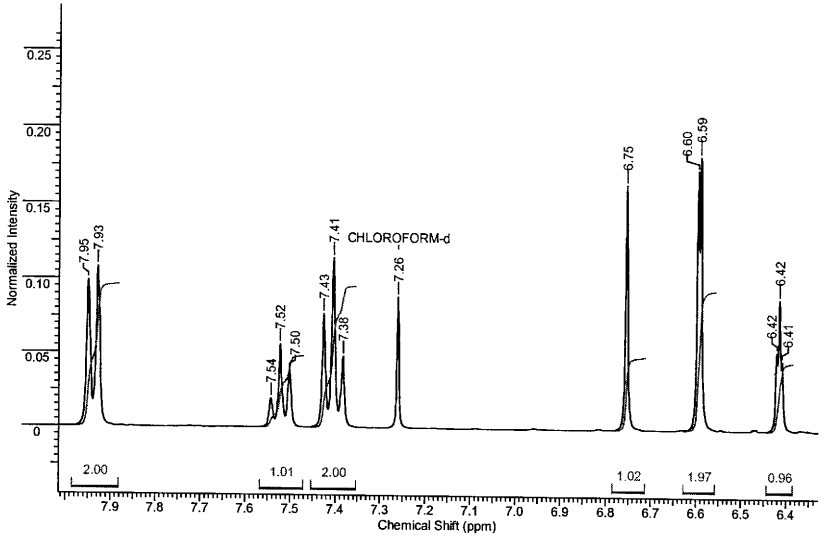
jld21, C13

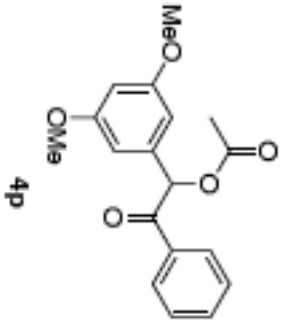
Acquisition Time (sec)	1.5139	***Comment***	jld21_C13 C13CPD CDCl3 u jld 26			***Date***	06 Sep 2005 22:04:48
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld21_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2580.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9014.8154
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



jld22

Acquisition Time (sec)	2.2021	***Comment***	jld22-1, pur PROTONNR CDCl3 u jld 21			***Date***	16 Sep 2005 09:42:24
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld22-1p2_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	228.10
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5950
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				

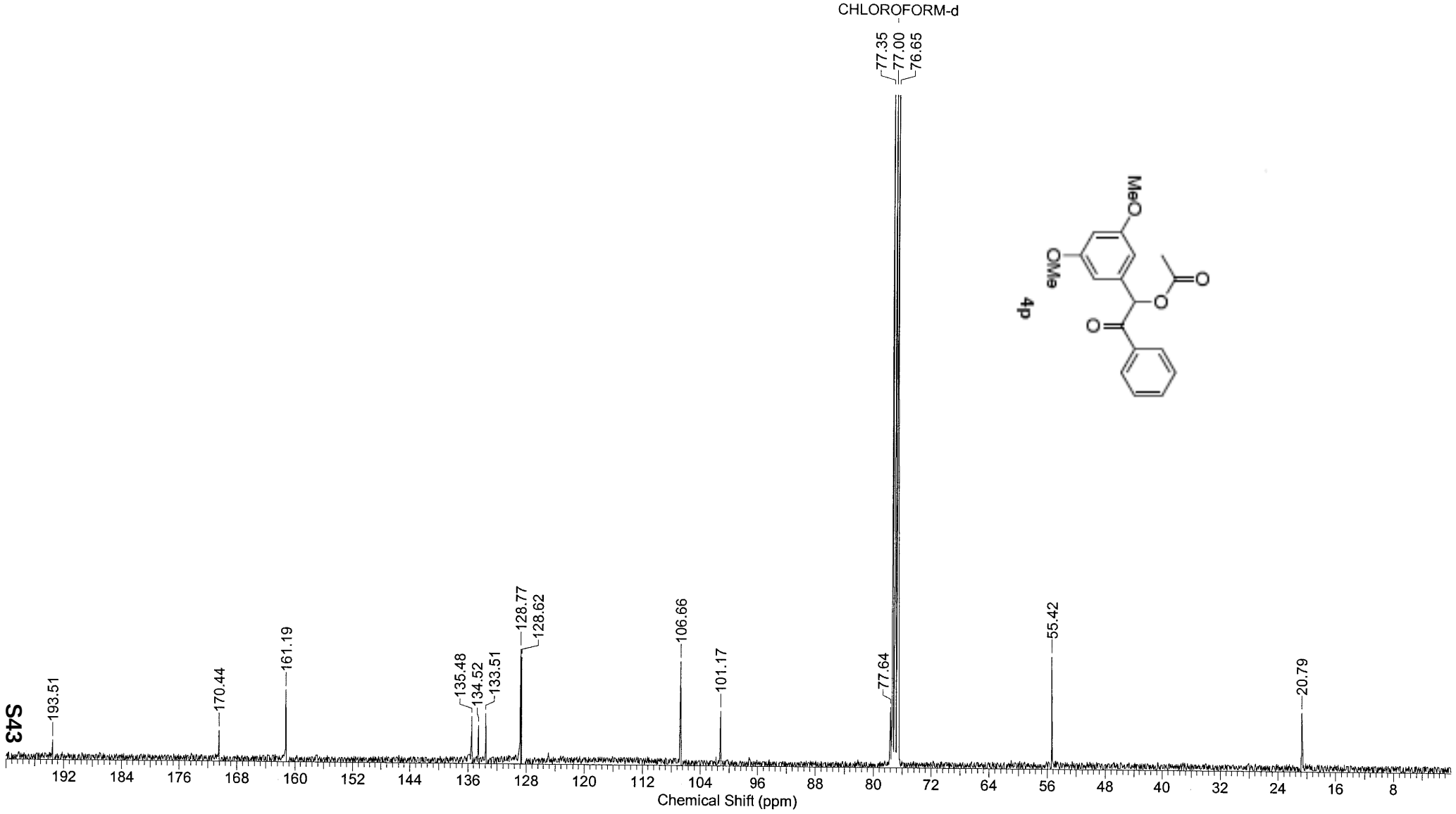




4p

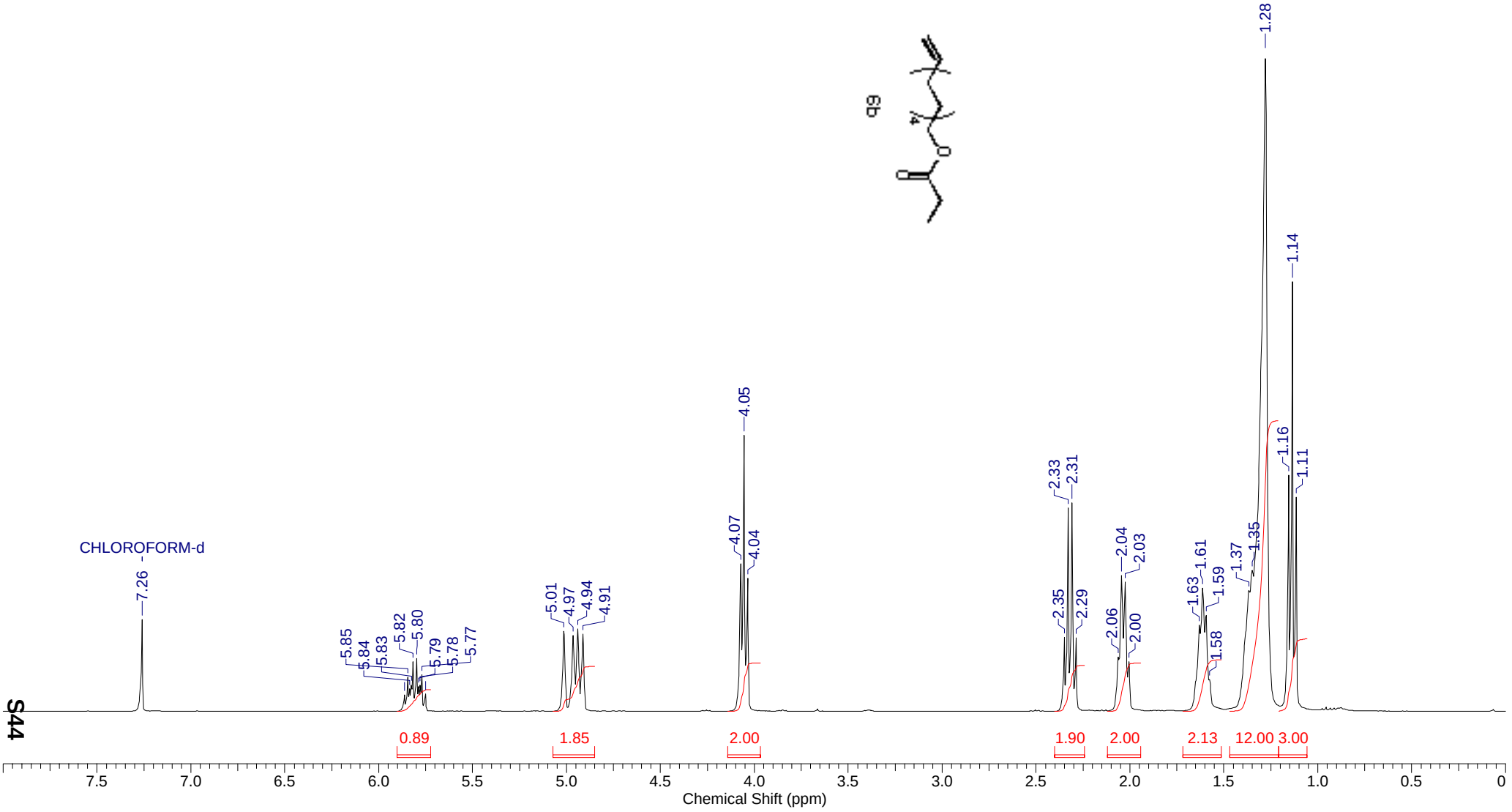
jld22, C13

Acquisition Time (sec)	1.5139	***Comment***	jld22-1, C13 C13CPD CDCl3 u jld 22			***Date***	16 Sep 2005 22:06:56
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld22_C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2896.30
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9014.8154
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



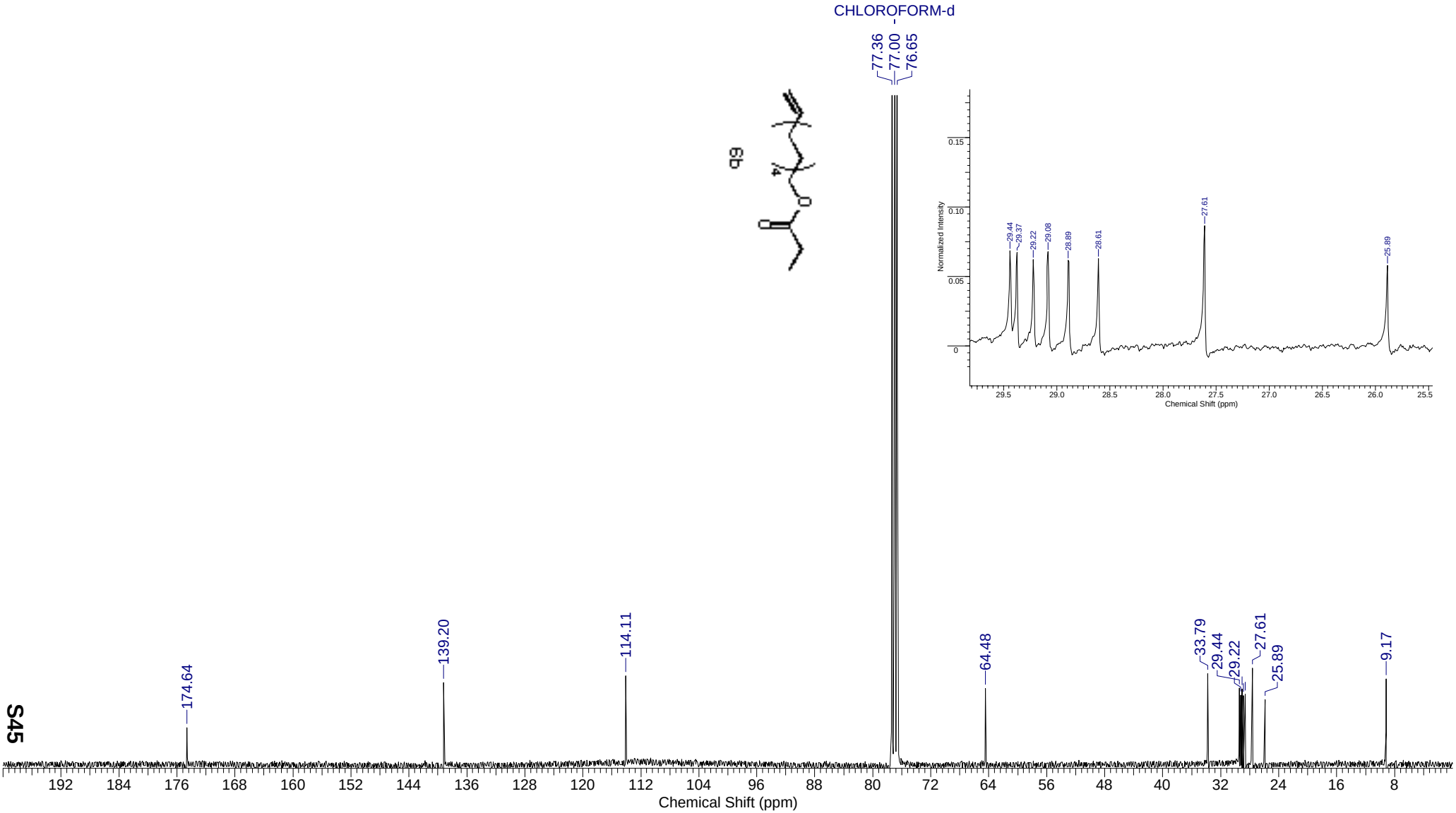
jld47-1

Acquisition Time (sec)	2.2021	***Comment***	PROTONNR CDCl3 u jld 27			***Date***	14 Mar 2006 16:57:36
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld47-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2211.0491
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



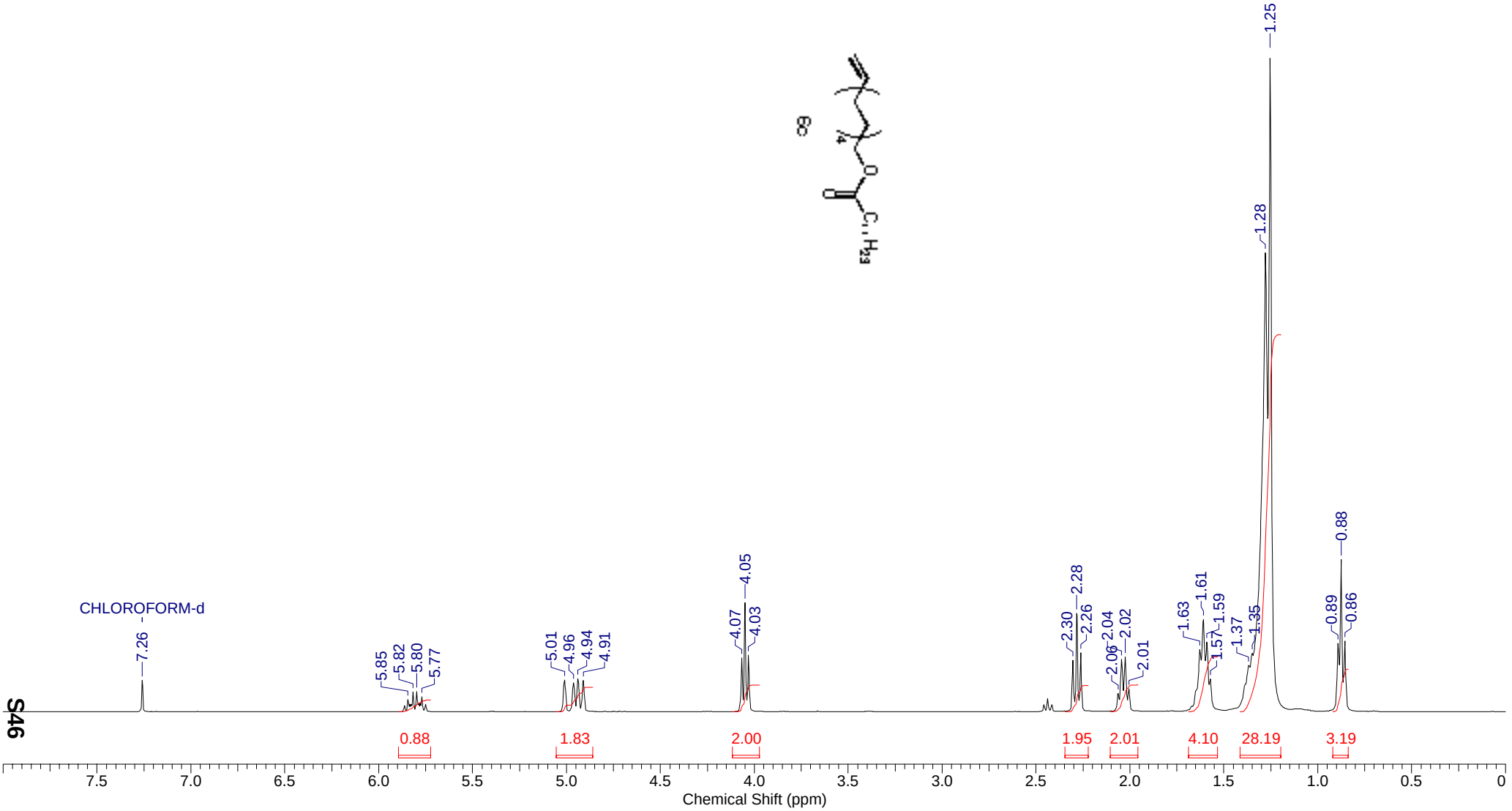
jld47 C13

Acquisition Time (sec)	1.5139	***Comment***	C13CPD CDCl3 u jld 27			***Date***	15 Mar 2006 01:21:04
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld47-1C13_001001r					***Frequency (MHz)***	90.55
Nucleus	13C	***Number of Transients***	4096	***Origin***	dpx360	***Original Points Count***	32768
Owner	nmruser	***Points Count***	32768	***Pulse Sequence***	zgpg30	***Receiver Gain***	2048.00
SW(cyclical) (Hz)	21645.02	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	9014.8154
Sweep Width (Hz)	21644.36	***Temperature (degree C)***	27.000				



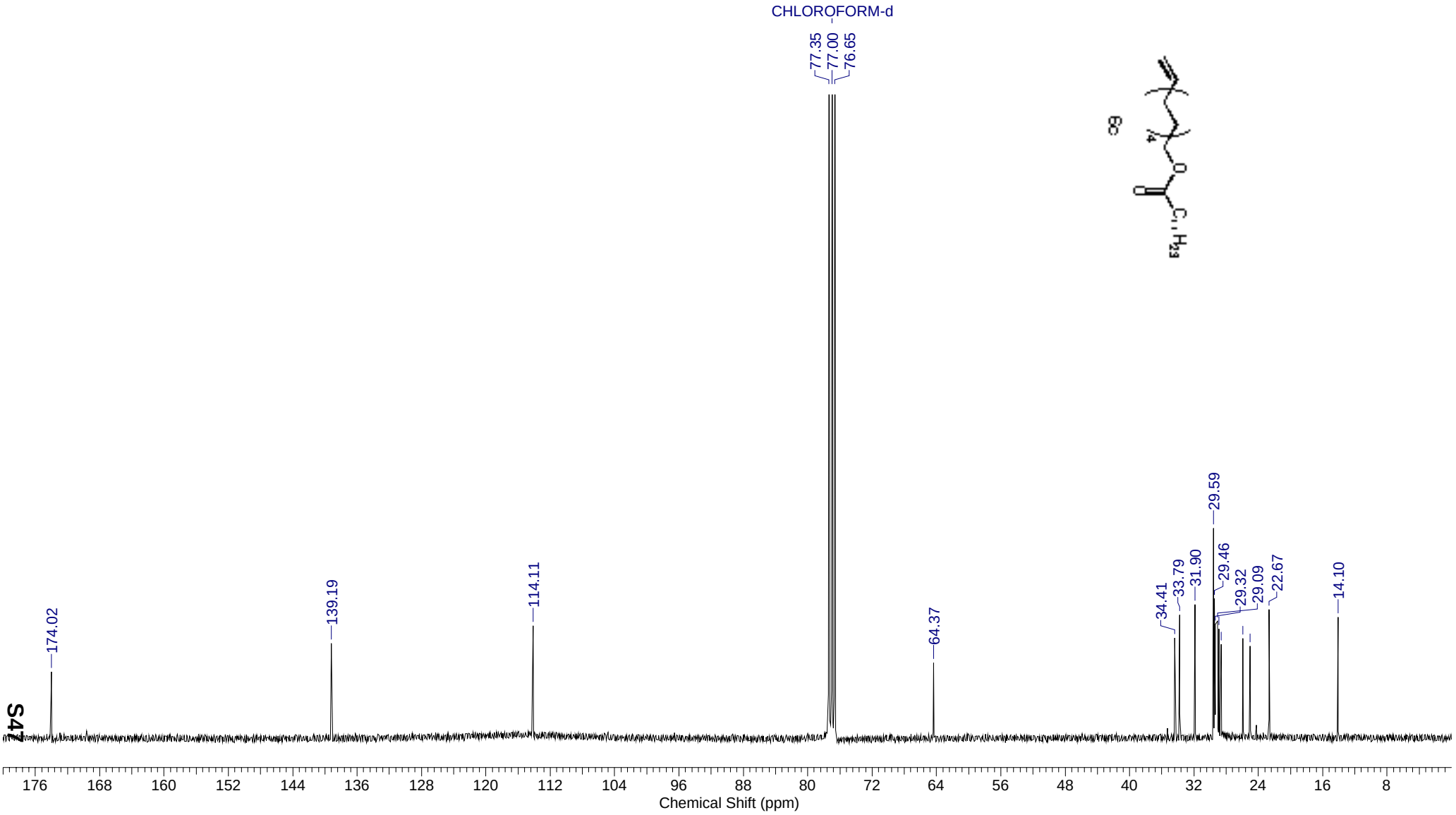
jld59-1

Acquisition Time (sec)	2.2021	***Comment***	PROTONNR CDCl3 u jld 59			***Date***	25 Apr 2006 12:52:16
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld59-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	71.80
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.1406
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



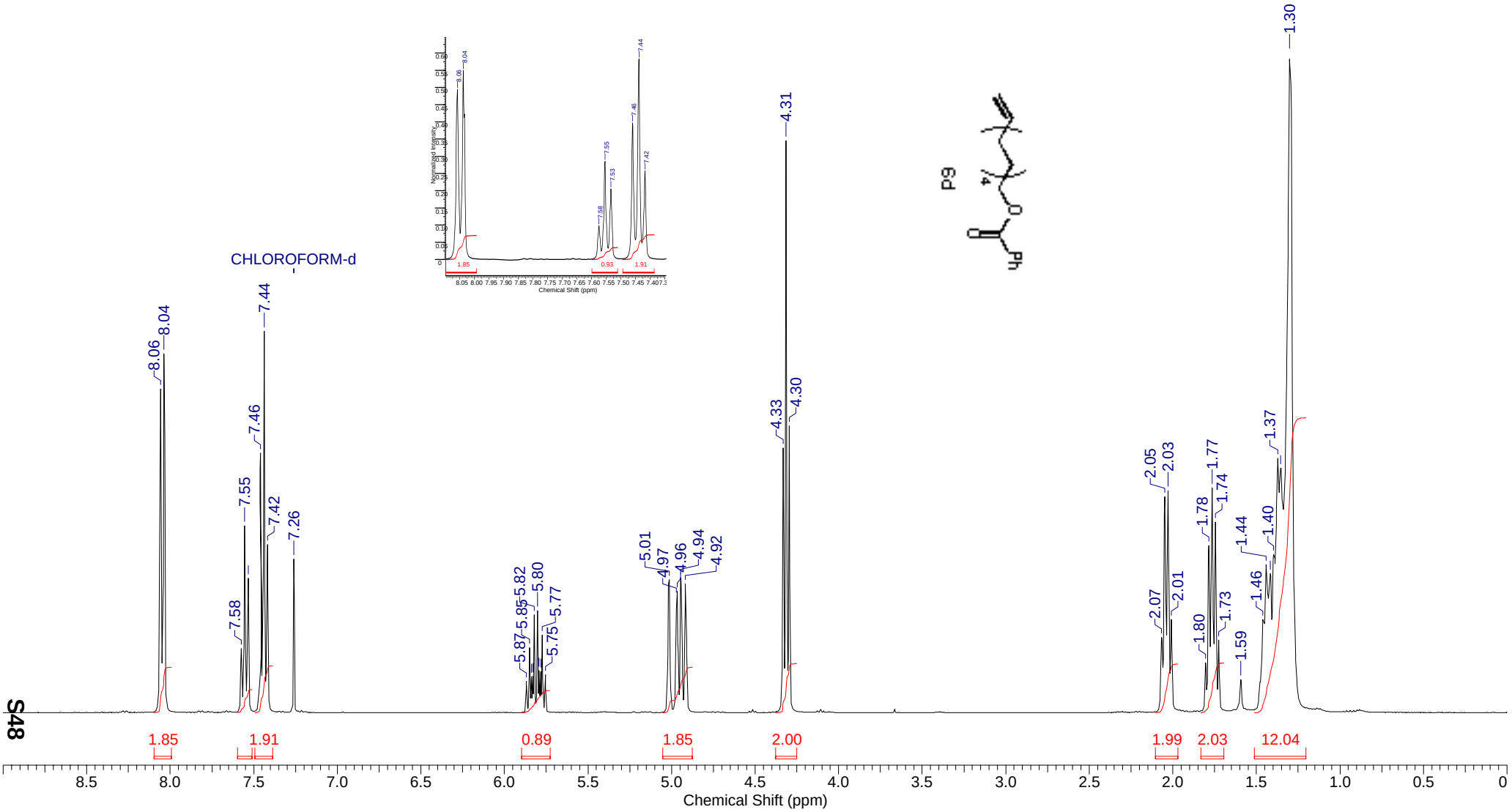
jld59, C13

Acquisition Time (sec)	1.5139	Comment	C13CPD CDCl3 u jld 59			Date	25 Apr 2006 22:06:56
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld59-1_C13_001001r					Frequency (MHz)	90.55
Nucleus	13C	Number of Transients	4096	Origin	dpx360	Original Points Count	32768
Owner	nmruser	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	1625.50
SW (cyclical) (Hz)	21645.02	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	9016.1367
Sweep Width (Hz)	21644.36	Temperature (degree C)	27.000				



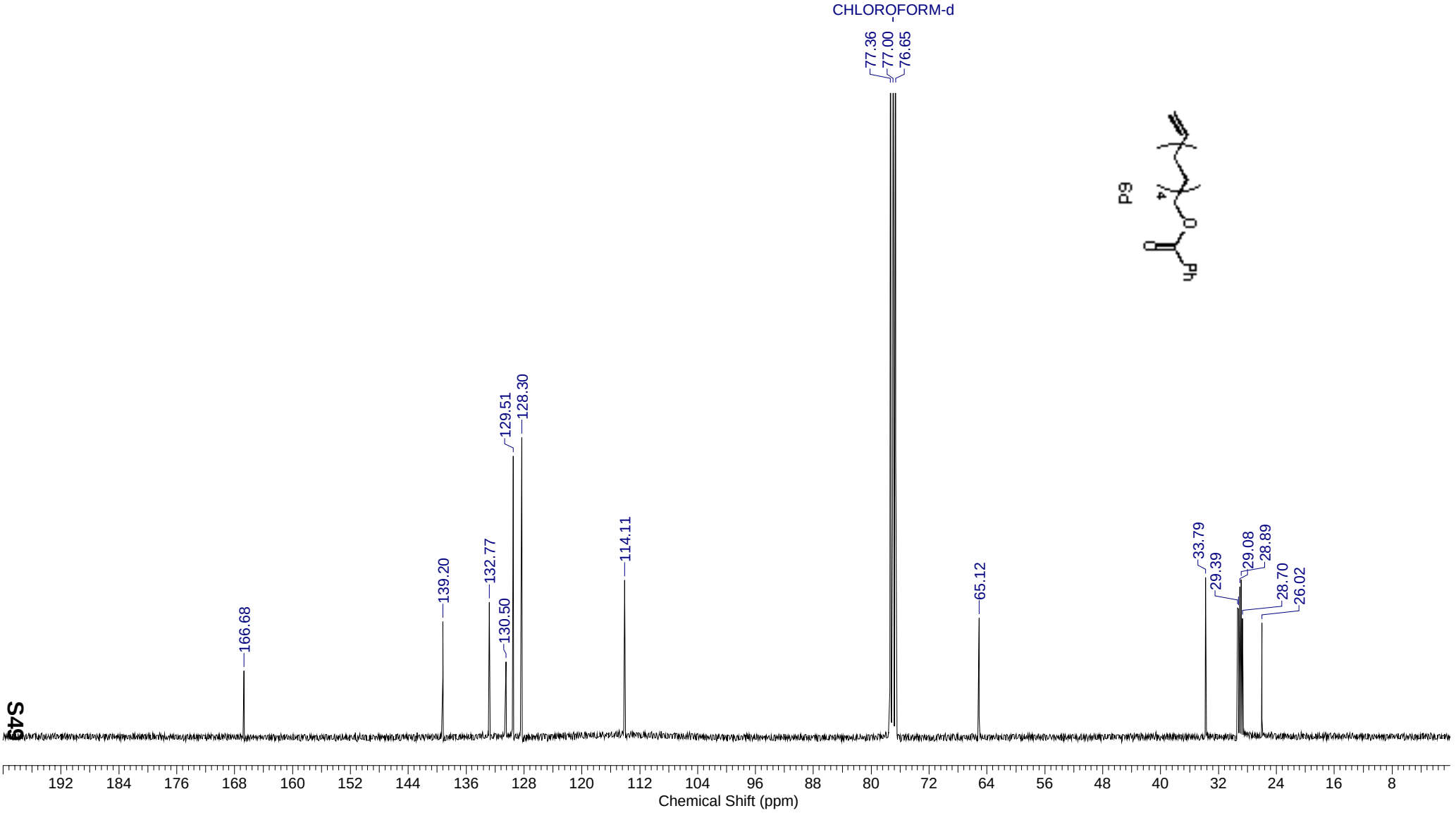
jld48

Acquisition Time (sec)	2.2021	***Comment***	PROTONNR CDCl3 u jld 48			***Date***	17 Mar 2006 13:09:20
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld48-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	32	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	90.50
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5947
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



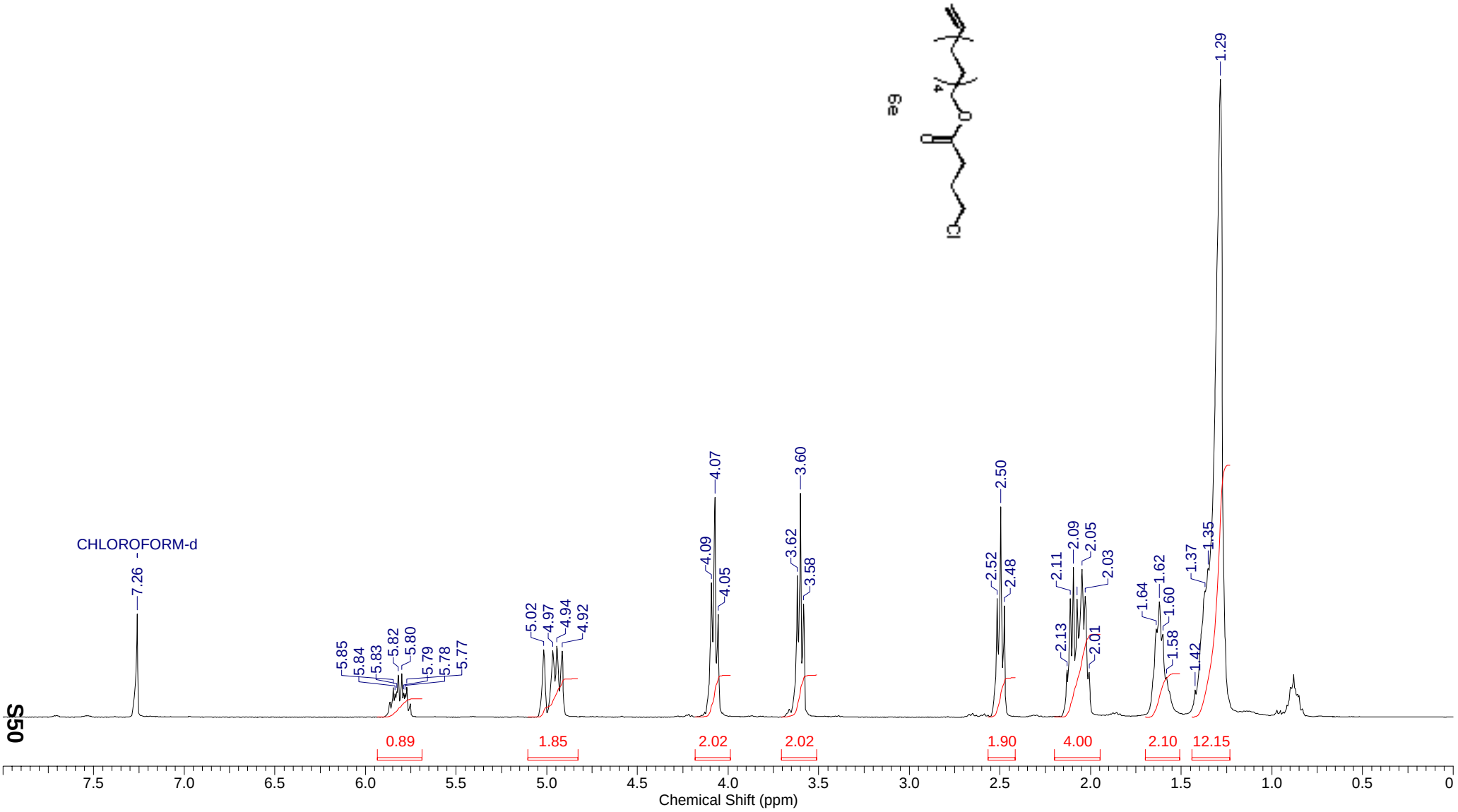
jld48 C13

Acquisition Time (sec)	1.5139	Comment	C13CPD CDCl3 u jld 48			Date	17 Mar 2006 23:04:32
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld48_C13_001001r					Frequency (MHz)	90.55
Nucleus	13C	Number of Transients	4098	Origin	dpx360	Original Points Count	32768
Owner	nmruser	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	2896.30
SW (cyclical) (Hz)	21645.02	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	9014.8154
Sweep Width (Hz)	21644.36	Temperature (degree C)	27.000				



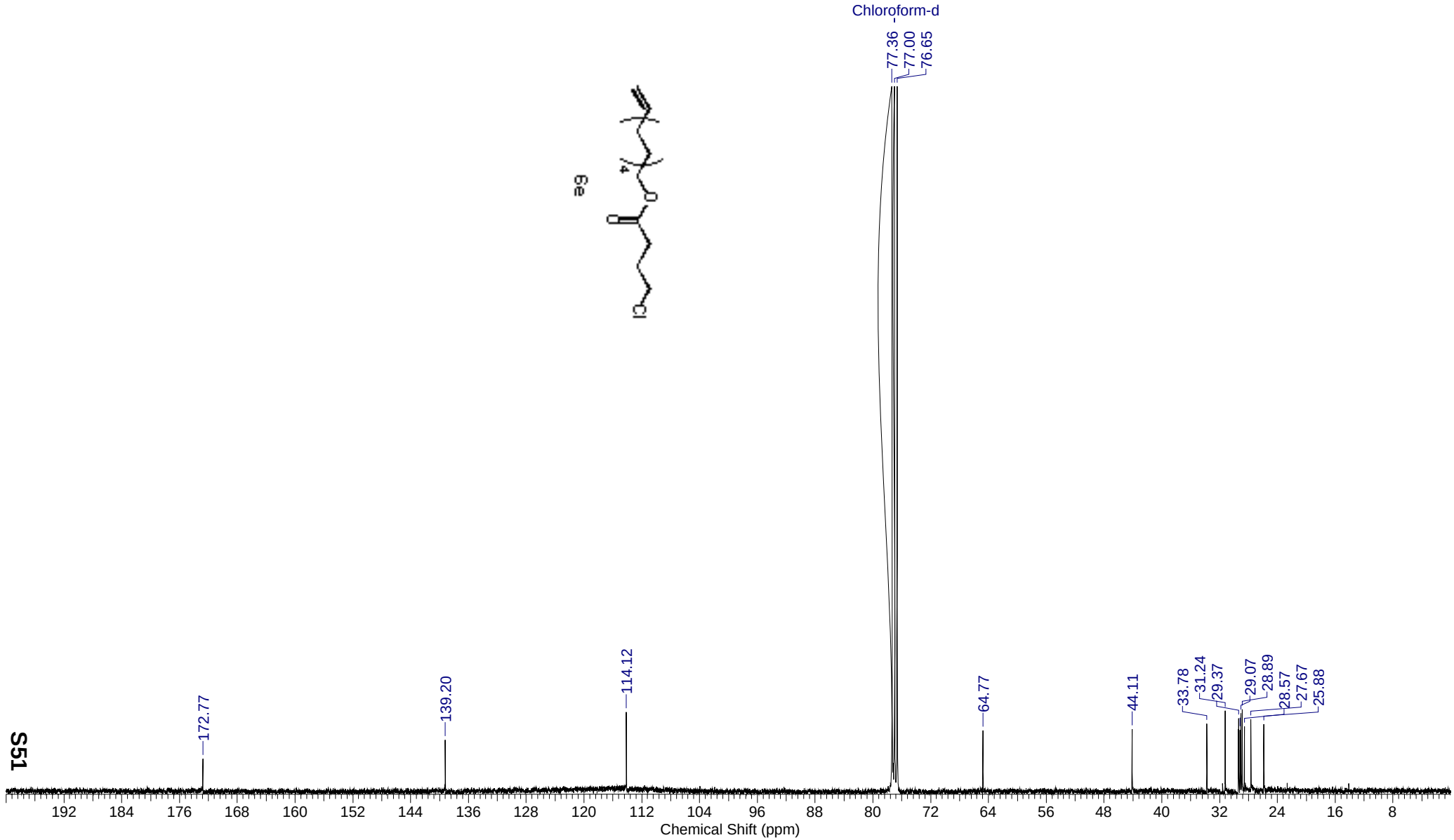
jld58

Acquisition Time (sec)	2.2021	***Comment***	PROTONNR CDCl3 u jld 58			***Date***	21 Apr 2006 12:03:12
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld58-2p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	143.70
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2211.0488
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



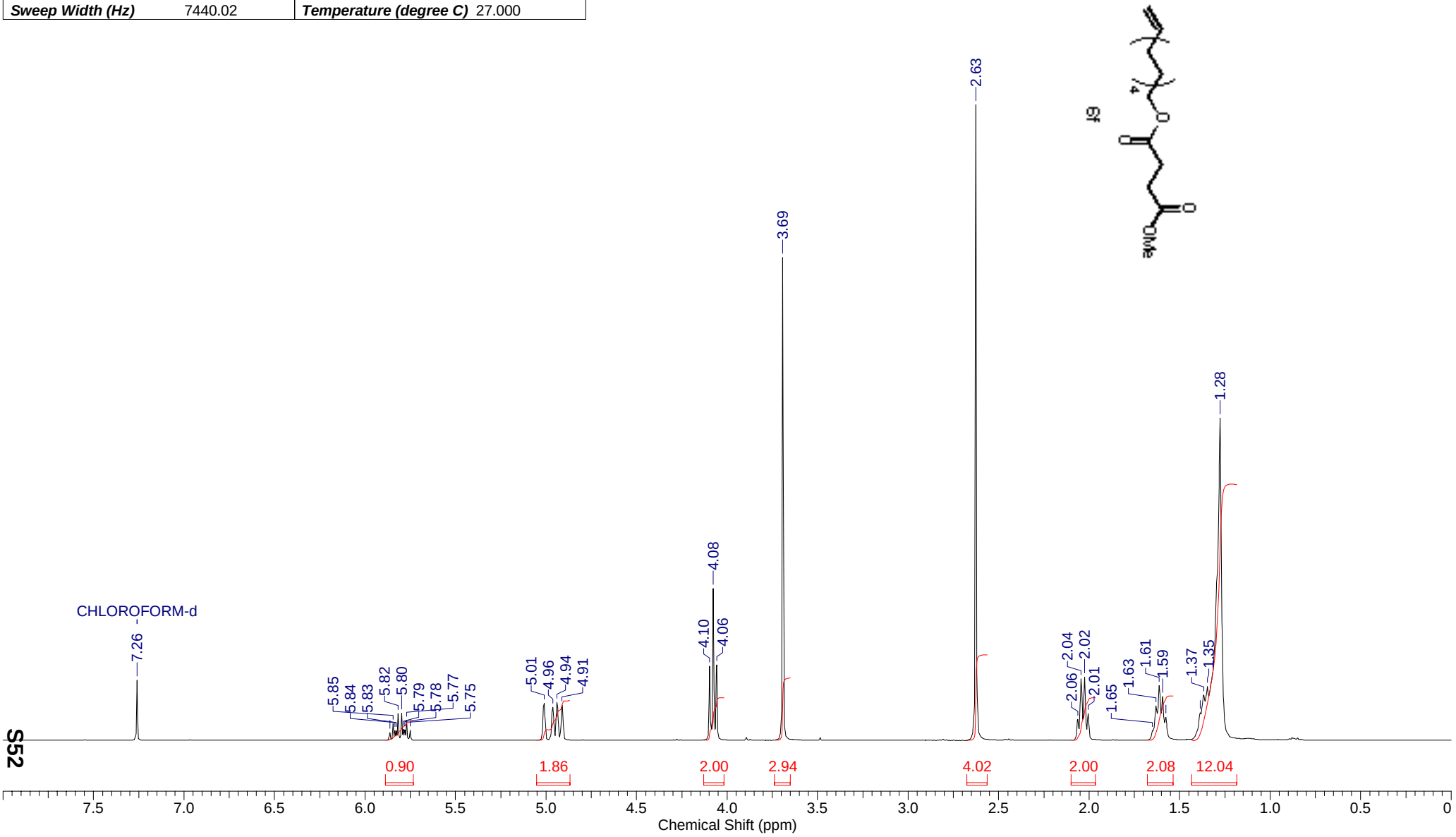
jld58, C13

Acquisition Time (sec)	1.5139	Comment	C13CPD CDCl3 u jld 58			Date	23 Apr 2006 17:27:40
File Name	C:\DOCUMENTS AND SETTINGS\NMR\DESKTOP\JLD58-2 \001001.1R					Frequency (MHz)	90.55
Nucleus	13C	Number of Transients	4096	Original Points Count	32768	Points Count	32768
Solvent	CHLOROFORM-D			Sweep Width (Hz)	21645.02	Temperature (degree C)	27.000



jld60

Acquisition Time (sec)	2.2021	***Comment***	PROTONNR CDCl3 u jld 60			***Date***	02 May 2006 08:53:20
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld60-1p_001001r					***Frequency (MHz)***	360.13
Nucleus	1H	***Number of Transients***	16	***Origin***	dpx360	***Original Points Count***	16384
Owner	nmruser	***Points Count***	16384	***Pulse Sequence***	zg30	***Receiver Gain***	143.70
SW(cyclical) (Hz)	7440.48	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2210.5947
Sweep Width (Hz)	7440.02	***Temperature (degree C)***	27.000				



jld60, C13

Acquisition Time (sec)	1.5139	Comment	C13CPD CDCl3 u jld 60			Date	03 May 2006 03:05:36
File Name	\\HOME\DebieuxJ\My Documents\Chimie\Doctorat\NMR\jld60-1_C13_001001r					Frequency (MHz)	90.55
Nucleus	13C	Number of Transients	4096	Origin	dpx360	Original Points Count	32768
Owner	nmruser	Points Count	32768	Pulse Sequence	zgpg30	Receiver Gain	2580.30
SW (cyclical) (Hz)	21645.02	Solvent	CHLOROFORM-d			Spectrum Offset (Hz)	9016.1367
Sweep Width (Hz)	21644.36	Temperature (degree C)	27.000