Supporting Information for

Phosphine-Mediated Olefination between Aldehydes and Allenes: An Efficient Synthesis of Trisubstituted 1,3-Dienes with High *E*-Selectivity

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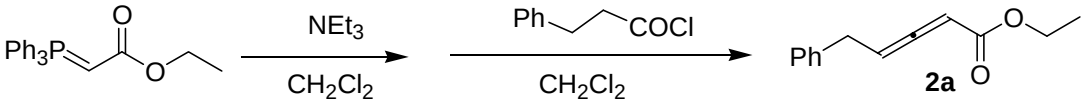
General Remarks

Unless otherwise mentioned, all reactions were carried out in nitrogen atmosphere under anhydrous conditions. ^{1}H and ^{13}C NMR spectra were recorded on a Variant 400 or a Bruker AV 300 spectrometer in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard. NOESY spectra were obtained on a Bruker AV 600 spectrometer in $CDCl_3$. Melting points were measured on a RY-I apparatus and uncorrected. High resolution ESI mass spectra were acquired with IonSpec QFT-ESI instrument. CHN microanalyses were measured with a Yanaco CHN Corder MT-3 automatic analyzer. X-ray crystallographic data were collected using a Nonius Kappa CCD diffractometer with Mo Kα radiation (λ = 0.7107 Å) at room temperature. Column chromatography was performed on silica gel (200-300 mesh) using a mixture of petroleum ether/ethyl acetate as eluant. Commercially available reagents were used without further purification. PTA was prepared from tetrahydroxymethylphosphonium sulfate according to a reported procedure.[1]

Preparation of Allenoates 2

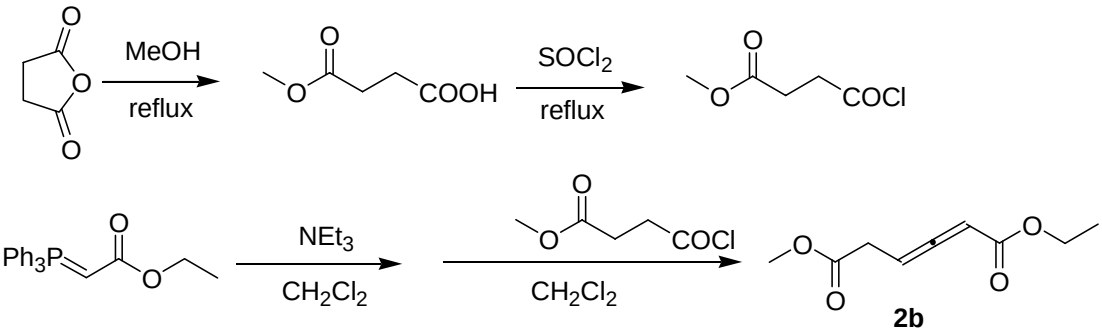
Synthesis of ethyl 5-phenylpenta-2, 3-dienoate[2] (2a)



Allenoate **2a** is a known compound and was synthesized according to a similar method developed by Hansen[3] and co-workers. To a solution of (ethoxycarbonylmethylene)triphenylphosphorane (50 mmol, 17.4 g) in dichloromethane (200 mL) was added 1.1 equiv of triethylamine (55 mmol, 5.6 g). After stirred for about 10 minutes, 1.1 equiv of 3-phenylpropanoyl chloride (55 mmol, 9.24 g) was dropwise added over 30 minutes at 0 ºC. Then the reaction mixture was allowed to be warmed up to room temperature and stirred overnight. The resulting mixture was carefully evaporated to remove most of the solvent, and the residue was extracted by petroleum ether (bp 30-60 ºC, 5 × 100 mL). The combined extracting

was concentrated and the crude product was subjected to column chromatography purification (eluant: 5% EtOAc in petroleum ether) to provide the allenoate **2a** as yellow oil (9.4 g, 93% yield). ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.36-7.15 (m, 5H), 5.78-5.73 (m, 1H), 5.62-5.59 (m, 1H), 4.24-4.15 (m, 2H), 3.49-3.44 (m, 2H), 1.30 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 212.7, 166.0, 138.5, 128.4, 126.5, 94.7, 88.6, 60.8, 34.0, 14.2.

Synthesis of 1-ethyl 6-methyl 2, 3-hexadienedioate (2b)



3-Carbomethoxypropionyl chloride was prepared according to a procedure described in literature[4]. Preparation of the allenoate **2b** was followed a similar procedure for **2a** described above.

Allenoate **2b** (colorless oil, 76% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 5.83-5.78 (m, 1H), 5.69-5.67 (m, 1H), 4.20 (q, J = 7.1 Hz, 2H), 3.73 (s, 3H), 3.23-3.18 (m, 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75MHz, TMS): δ = 212.3, 170.3, 165.2, 89.0, 88.6, 60.8, 51.9, 32.6, 14.0; HRMS calcd for $C_9H_{12}O_4Na^+$ requires 207.0628, found 207.0632.

General Olefination Procedure

Ph_3P-mediated olefination of allenoate 2a with aldehydes:

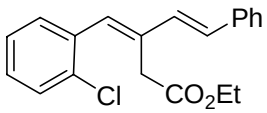
At room temperature and under nitrogen atmosphere, to a stirred solution of aldehyde (0.5 mmol) and Ph_3P (0.6 mmol, 157 mg) in dichloromethane (2 mL) was added allenoate **2a** (0.6 mmol, 121 mg) by the means of a microsyringe over 5 minutes. The resulting reaction mixture was further stirred at room temperature and monitored by TLC. When the aldehyde disappeared, the solvent was removed under reduced pressure and the residue was subjected to column chromatography on silica gel

(gradient eluant: petroleum ether/ethyl acetate 20:1–5:1) to give diene **3**.

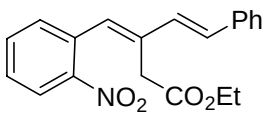
PTA-mediated olefination of allenoate 2a or 2b with aldehydes:

At room temperature and under nitrogen atmosphere, to a stirred solution of aldehyde (0.5 mmol) and PTA (0.6 mmol, 94 mg) in dichloromethane (5 mL) was added allenoate **2a** (0.6 mmol, 121 mg) or **2b** (0.6 mmol, 110 mg) by the means of a microsyringe over 5 minutes. The reaction mixture was further stirred at room temperature and monitored by TLC. When the aldehyde disappeared, water (15 mL) was added to dissolve the PTA oxide. The organic layer was separated and the aqueous layer was extracted with dichloromethane (3 × 10 mL). The combined extracting was dried over sodium sulfate and concentrated, and the residue was subjected to column chromatography on silica gel (gradient eluant: petroleum ether/ethyl acetate 20:1–5:1) to afford diene **3**.

Analytical Data for Dienes 3 and 4

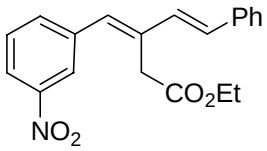


(3*E*,4*E*)-ethyl 3-(2-chlorobenzylidene)-5-phenylpent-4-enoate (3a) obtained from *o*-chlorobenzaldehyde (70 mg, 0.5 mmol) as a white solid (131 mg, 80% yield): mp 87-88 ºC; IR (thin film): ν_{max} 3024, 2987, 2897, 1741, 1467, 1448, 1319, 1184, 1151, 1028, 964, 831, 761, 751, 691, 657 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.56 (dd, J = 7.0, 2.3 Hz, 1H), 7.47-7.19 (m, 8H), 7.02 (d, J = 16.3 Hz, 1H), 6.93 (s, 1H), 6.69 (d, J = 16.3 Hz, 1H), 4.21 (q, J = 7.1 Hz, 2H), 3.47 (s, 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.4, 137.0, 135.2, 134.1, 134.0, 131.7, 131.6, 130.3, 129.4, 129.3, 128.7, 128.6, 127.7, 126.6, 126.5, 61.0, 34.3, 14.2; Anal. calcd for $C_{20}H_{19}ClO_2$: C, 73.50; H, 5.86%; found: C, 73.55; H, 5.95%.

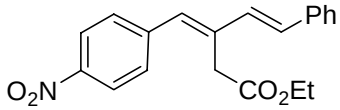


(3*E*,4*E*)-ethyl 3-(2-nitrobenzylidene)-5-phenylpent-4-enoate (3b) obtained from *o*-nitrobenzaldehyde (76 mg, 0.5 mmol) as a yellow solid (167 mg, 99% yield): mp

81-82 ºC; IR (thin film): ν_{max} 3022, 2985, 2922, 1721, 1514, 1335, 1239, 1195, 1140, 1025, 963, 870, 741, 685, 548 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.07 (d, J = 7.7 Hz, 1H), 7.68-7.59 (m, 2H), 7.48-7.22 (m, 6H), 7.13 (s, 1H), 7.00 (d, J = 16.3 Hz, 1H), 6.70 (d, J = 16.3 Hz, 1H), 4.18 (q, J = 7.1 Hz, 2H), 3.37 (s, 2H), 1.26 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.1, 148.1, 136.8, 134.1, 133.1, 132.3, 131.6, 131.0, 130.3, 129.9, 128.6, 128.3, 127.8, 126.6, 124.7, 61.0, 34.3, 14.1; Anal calcd for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15%; found: C, 71.07; H, 5.99; N, 3.99%.

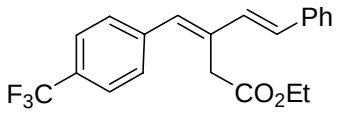


(3*E*,4*E*)-ethyl 3-(3-nitrobenzylidene)-5-phenylpent-4-enoate (3c) obtained from *m*-nitrobenzaldehyde (76 mg, 0.5 mmol) as a yellow solid (167 mg, 99% yield): mp 68-69 ºC; IR (thin film): ν_{max} 3072, 2976, 2902, 1726, 1533, 1351, 1305, 1199, 1130, 1068, 966, 810, 731, 696, 607, 549 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.32 (s, 1H), 8.11 (d, J = 8.2 Hz, 1H), 7.74 (d, J = 7.7 Hz, 1H), 7.54-7.44 (m, 3H), 7.36-7.23 (m, 3H), 6.96 (d, J = 16.2 Hz, 1H), 6.86 (s, 1H), 6.78 (d, J = 16.2 Hz, 1H), 4.26 (q, J = 7.1 Hz, 2H), 3.53 (s, 2H), 1.32 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.8, 148.3, 138.4, 136.7, 135.1, 134.7, 131.7, 131.2, 130.3, 129.2, 128.6, 127.9, 126.6, 123.4, 121.9, 61.3, 34.0, 14.1; Anal calcd for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15%; found: C, 70.96; H, 5.87; N, 4.12%.

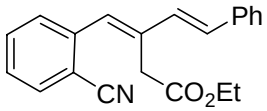


(3*E*,4*E*)-ethyl 3-(4-nitrobenzylidene)-5-phenylpent-4-enoate (3d) obtained from *p*-nitrobenzaldehyde (76 mg, 0.5 mmol) as a yellow solid (155 mg, 92% yield): mp 98-99 ºC; IR (thin film): ν_{max} 3021, 2977, 1727, 1591, 1514, 1446, 1334, 1197, 1107, 1030, 963, 889, 840, 741, 686, 634 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.16 (d, J = 8.6 Hz, 2H), 7.56 (d, J = 8.6 Hz, 2H), 7.44 (d, J = 7.4 Hz, 2H), 7.36-7.23 (m, 3H), 6.95 (d, J = 16.2 Hz, 1H), 6.86 (s, 1H), 6.78 (d, J = 16.2 Hz, 1H), 4.24 (q, J = 7.1 Hz, 2H), 3.54 (s, 2H), 1.30 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ =

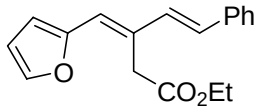
170.7, 146.5, 143.4, 136.6, 135.7, 132.1, 131.3, 130.6, 129.4, 128.6, 128.0, 126.6, 123.5, 61.2, 34.0, 14.1; Anal calcd for $C_{20}H_{19}NO_4$: C, 71.20; H, 5.68; N, 4.15%; found: C, 71.21; H, 5.29; N, 4.19%.



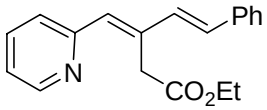
(3*E*,4*E*)-ethyl 3-(4-(trifluoromethyl)benzylidene)-5-phenylpent-4-enoate (3e) obtained from *p*-trifluoromethylbenzaldehyde (87 mg, 0.5 mmol) as a white solid (144 mg, 80% yield): mp 49-50 ºC; IR (thin film): ν_{max} 3027, 2989, 1728, 1608, 1444, 1324, 1252, 1163, 1114, 1066, 1027, 958, 889, 849, 749, 694, 598 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.61 (d, J = 8.2 Hz, 2H), 7.51 (d, J = 8.2 Hz, 2H), 7.45 (d, J = 7.2 Hz, 2H), 7.36-7.22 (m, 3H), 6.96 (d, J = 16.3 Hz, 1H), 6.87 (s, 1H), 6.73 (d, J = 16.3 Hz, 1H), 4.23 (q, J = 7.1 Hz, 2H), 3.53 (s, 2H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.1, 140.5, 137.0, 134.6, 133.1, 131.6, 129.8, 129.0, 128.7, 128.7 (q, J = 36.7 Hz, 1C), 127.9, 126.6, 125.3 (q, J = 3.8 Hz, 2C), 124.2 (q, J = 271.4 Hz, 1C), 61.1, 34.1, 14.2; Anal calcd for $C_{21}H_{19}F_3O_2$: C, 69.99; H, 5.31%; found: C, 70.13; H, 5.38%.



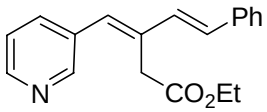
(3*E*,4*E*)-ethyl 3-(2-cyanobenzylidene)-5-phenylpent-4-enoate (3f) obtained from *o*-cyanobenzaldehyde (66 mg, 0.5 mmol) as a white solid (157 mg, 99% yield): mp 77-78 ºC; IR (thin film): ν_{max} 3028, 2981, 2933, 2220, 1723, 1593, 1478, 1446, 1364, 1324, 1195, 1142, 1026, 958, 841, 764, 684, 540 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.74 (d, J = 7.8 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.61-7.56 (m, 1H), 7.60 (d, J = 7.6 Hz, 2H), 7.39-7.23 (m, 4H), 7.02 (d, J = 16.3 Hz, 1H), 7.03 (s, 1H), 6.77 (d, J = 16.3 Hz, 1H), 4.23 (q, J = 7.1 Hz, 2H), 3.51 (s, 2H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.9, 140.2, 136.6, 136.3, 132.9, 132.5, 131.1, 130.7, 130.0, 129.3, 128.6, 128.0, 127.6, 126.7, 117.7, 112.4, 61.1, 34.2, 14.1; Anal calcd for $C_{21}H_{19}NO_2$: C, 79.47; H, 6.03; N, 4.41%; found: C, 79.18; H, 6.00; N, 4.44%.



(3*E*,4*E*)-ethyl 3-(furan-2-ylmethylene)-5-phenylpent-4-enoate (3g) obtained from 2-furylaldehyde (48 mg, 0.5 mmol) as yellow oil (102 mg, 72% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.42-7.17 (m, 6H), 6.90 (d, J = 16.1 Hz, 1H), 6.62 (d, J = 16.1 Hz, 1H), 6.52 (s, 1H), 6.45-6.39 (m, 2H), 4.17 (q, J = 7.1 Hz, 2H), 3.82 (s, 2H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.9, 152.5, 142.6, 137.1, 131.9, 129.9, 128.5, 128.3, 127.4, 126.3, 121.6, 111.6, 111.5, 60.7, 33.9, 14.1; Anal calcd for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43%; found: C, 76.50; H, 6.64%.

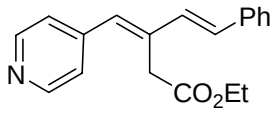


(3*E*,4*E*)-ethyl 5-phenyl-3-(pyridin-2-ylmethylene)pent-4-enoate (3h) obtained from 2-pyridylaldehyde (54 mg, 0.5 mmol) as colorless oil (103 mg, 70% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.55 (d, J = 4.7 Hz, 1H), 7.55 (dt, J = 7.7, 1.8 Hz, 1H), 7.44 (d, J = 7.3 Hz, 2H), 7.33-7.21 (m, 4H), 7.04-7.00 (m, 1H), 6.99 (d, J = 16.2 Hz, 1H), 6.72 (d, J = 16.2 Hz, 1H), 6.71 (s, 1H), 4.25 (s, 2H), 4.20 (q, J = 7.1 Hz, 2H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.3, 155.9, 148.9, 136.9, 136.3, 135.9, 132.6, 132.0, 129.7, 128.5, 127.6, 126.5, 125.1, 121.1, 60.4, 33.5, 14.1; Anal calcd for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77%; found: C, 77.72; H, 6.73; N, 4.71%.

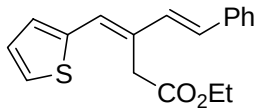


(3*E*,4*E*)-ethyl 5-phenyl-3-(pyridin-3-ylmethylene)pent-4-enoate (3i) obtained from 3-pyridylaldehyde (54 mg, 0.5 mmol) as colorless oil (111 mg, 76% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.54 (s, 1H), 8.38 (d, J = 4.6 Hz, 1H), 7.64 (d, J = 7.9 Hz, 1H), 7.33 (d, J = 7.2 Hz, 2H), 7.23-7.09 (m, 4H), 6.85 (d, J = 16.2 Hz, 1H), 6.67 (s, 1H), 6.61 (d, J = 16.2 Hz, 1H), 4.10 (q, J = 7.1 Hz, 2H), 3.41 (s, 2H), 1.61 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.8, 149.7, 148.0, 136.6, 135.4, 134.6, 132.4, 131.3, 130.5, 129.5, 128.4, 127.6, 126.4, 123.0, 60.9, 33.8, 14.0; Anal calcd for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77%; found: C, 77.68; H, 6.35; N,

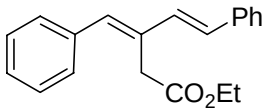
4.59%.



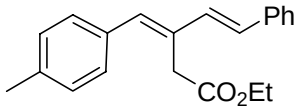
(3*E*,4*E*)-ethyl 5-phenyl-3-(pyridin-4-ylmethylene)pent-4-enoate (3j) obtained from 4-pyridylaldehyde (54 mg, 0.5 mmol) as colorless oil (111 mg, 76% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.48 (d, J = 5.6 Hz, 2H), 7.34 (d, J = 7.2 Hz, 2H), 7.25-7.11 (m, 5H), 6.83 (d, J = 16.1 Hz, 1H), 6.66 (d, J = 16.1 Hz, 1H), 6.63 (s, 1H), 4.12 (q, J = 7.1 Hz, 2H), 3.44 (s, 2H), 1.17 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.6, 149.7, 144.1, 136.5, 135.8, 131.5, 131.2, 130.4, 128.5, 128.9, 126.5, 123.1, 61.0, 33.9, 14.0; Anal calcd for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77%; found: C, 77.70; H, 6.60; N, 4.77%.



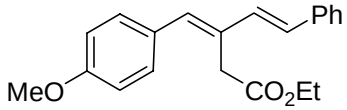
(3*E*,4*E*)-ethyl 5-phenyl-3-(thiophen-2-ylmethylene)pent-4-enoate (3k) obtained from 2-thiofurylaldehyde (56 mg, 0.5 mmol) as colorless oil (106 mg, 71% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.44-7.15 (m, 7H), 7.03 (m, 1H), 6.94 (d, J = 16.2 Hz, 1H), 6.91 (s, 1H), 6.63 (d, J = 16.2 Hz, 1H), 4.20 (q, J = 7.1 Hz, 2H), 3.75 (s, 2H), 1.26 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.5, 139.8, 137.2, 132.1, 130.8, 128.5, 128.4, 127.4, 127.3, 127.2, 126.4, 126.3, 126.2, 61.0, 34.2, 14.1; HRMS calcd for $C_{18}H_{18}O_2SNa^+$ requires 321.0920, found 321.0927.



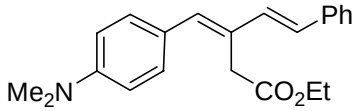
(3*E*,4*E*)-ethyl 3-benzylidene-5-phenylpent-4-enoate (3l) obtained from benzaldehyde (53 mg, 0.5 mmol) as colorless oil (133 mg, 91% yield); ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.45-7.20 (m, 10H), 6.96 (d, J = 16.2 Hz, 1H), 6.88 (s, 1H), 6.65 (d, J = 16.2 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.57 (s, 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.5, 137.2, 136.9, 134.9, 132.7, 132.2, 128.8, 128.6, 128.5, 128.3, 127.5, 127.3, 126.4, 60.9, 34.1, 14.2; Anal calcd for $C_{20}H_{20}O_2$: C, 82.16; H, 6.89%; found: C, 81.96; H, 6.89%.



(3*E*,4*E*)-ethyl 3-(4-methylbenzylidene)-5-phenylpent-4-enoate (3m) obtained from *p*-methylbenzaldehyde (60 mg, 0.5 mmol) as colorless oil (78 mg, 51% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.43 (d, J = 7.7 Hz, 2H), 7.34-7.16 (m, 7H), 6.96 (d, J = 16.2 Hz, 1H), 6.86 (s, 1H), 6.63 (d, J = 16.2 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.57 (s, 2H), 2.36 (s, 3H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 171.6, 137.2, 137.1, 135.0, 133.9, 132.3, 132.0, 129.0, 128.7, 128.5, 128.1, 127.4, 126.3, 60.9, 34.0, 21.2, 14.2; HRMS calcd for $C_{21}H_{22}O_2Na^+$ requires 329.1512, found 329.1518.

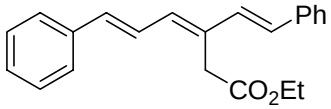


(3*E*,4*E*)-ethyl 3-(4-methoxybenzylidene)-5-phenylpent-4-enoate (3n) obtained from *p*-methoxybenzaldehyde (68 mg, 0.5 mmol) as a white solid (81 mg, 50% yield): mp. 49-51 ºC; IR (thin film): ν_{max} 3024, 2956, 2839, 1729, 1601, 1508, 1442, 1299, 1251, 1181, 1142, 1027, 961, 844, 749, 693, 528 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.43 (d, J = 7.4 Hz, 2H), 7.37-7.18 (m, 5H), 6.95 (d, J = 16.2 Hz, 1H), 6.90 (d, J = 8.7 Hz, 2H), 6.82 (s, 1H), 6.62 (d, J = 16.2 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.80 (s, 3H), 3.57 (s, 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 171.6, 158.8, 137.3, 134.7, 132.4, 131.2, 130.1, 129.3, 128.5, 127.7, 127.3, 126.3, 113.8, 60.9, 55.1, 34.0, 14.2; Anal calcd for $C_{21}H_{22}O_3$: C, 78.23; H, 6.88%; found: C, 78.34; H, 6.41%.

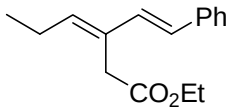


(3*E*,4*E*)-ethyl 3-(4-(dimethylamino)benzylidene)-5-phenylpent-4-enoate (3o) obtained from *p*-(*N*,*N*-dimethylamino)benzaldehyde (75 mg, 0.5 mmol) as a yellow solid (72 mg, 43% yield): mp.73-76 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.43 (d, J = 7.5 Hz, 2H), 7.34-7.17 (m, 5H), 6.97 (d, J = 16.2 Hz, 1H), 6.79 (s, 1H), 6.71 (d, J = 8.7 Hz, 2H), 6.57 (d, J = 16.2 Hz, 1H), 4.23 (q, J = 7.1 Hz, 2H), 3.62 (s, 2H), 2.97 (s, 6H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.9, 149.7,

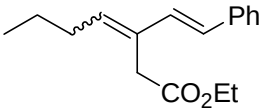
137.7, 135.5, 133.2, 130.1, 129.5, 128.5, 127.0, 126.7, 126.2, 125.0, 112.0, 60.8, 40.3, 34.2, 14.2; HRMS calcd for $C_{22}H_{25}NO_2H^+$ requires 336.1958, found 336.1961.



(3*E*,5*E*)-ethyl 6-phenyl-3-((*E*)-styryl)hexa-3,5-dienoate (3p) obtained from *trans*-cinnamaldehyde (66 mg, 0.5 mmol) as a yellow solid (48 mg, 30% yield): mp 71-74 ºC; ^{1}H NMR ($CDCl_3$, 300 MHz, TMS): δ = 7.46-7.21 (m, 10H), 7.16 (dd, J = 15.4, 11.3 Hz, 1H), 6.90 (d, J = 16.1 Hz, 1H), 6.69 (d, J = 15.4 Hz, 1H), 6.67 (d, J = 16.1 Hz, 1H), 6.54 (d, J = 11.3 Hz, 1H), 4.17 (q, J = 7.1 Hz, 2H), 3.58 (s, 2H), 1.24 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.7, 137.4, 137.3, 134.9, 134.5, 132.3, 131.7, 128.6, 128.5, 128.4, 127.8, 127.5, 126.6, 126.5, 124.7, 60.9, 33.4, 14.2; HRMS calcd for $C_{22}H_{22}O_2Na^+$ requires 341.1512, found 341.1517.

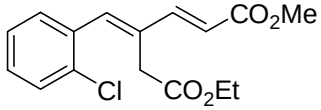


(*E*)-ethyl 3-((*E*)-styryl)hex-3-enoate (3q) obtained from propylaldehyde (29 mg, 0.5 mmol) as colorless oil (54 mg, 44% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.32-7.10 (m, 5H), 6.71 (d, J = 16.1 Hz, 1H), 6.42 (d, J = 16.1 Hz, 1H), 5.75 (t, J = 7.0 Hz, 1H), 4.05 (q, J = 7.0 Hz, 2H), 3.29 (s, 2H), 2.19-2.15 (m, 2H), 1.17 (t, J = 7.0 Hz, 3H), 0.98 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 171.2, 138.8, 137.5, 132.0, 130.6, 128.5, 127.0, 126.3, 126.2, 60.7, 32.9, 22.0, 14.2, 14.1; HRMS calcd for $C_{16}H_{20}O_2Na^+$ requires 267.1355, found 267.1358.

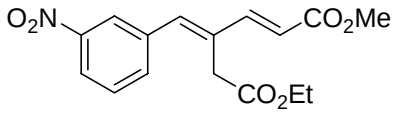


(*E*)-ethyl 3-((*E*)-styryl)hept-3-enoate (3r) obtained from *n*-butylaldehyde (36 mg, 0.5 mmol) as colorless oil (59 mg, 46% yield), contaminated by the minor product (*Z*,*E*)-isomer with a ratio of (*E*,*E*) : (*Z*,*E*) = 8 : 1; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.39 (d, J = 7.5 Hz, 2H), 7.31-7.17 (m, 3H), 6.79 (d, J = 16.3 Hz, 1H), 6.49 (d, J = 16.3 Hz, 1H), 5.85 (t, J = 7.4 Hz, 1H), 4.14 (q, J = 7.1 Hz, 2H), 3.38 (s, 2H), 2.24-2.18 (m, 2H), 1.53-1.43 (m, 2H),1.24 (t, J = 7.1 Hz, 3H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.2, 137.6, 137.1, 132.1, 131.3, 128.5, 127.0,

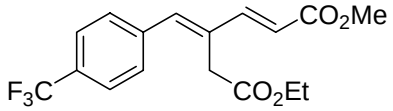
126.4, 126.2, 60.7, 33.0, 30.8, 22.5, 14.2, 13.8; HRMS calcd for $C_{17}H_{22}O_2\ Na^+$ requires 281.1512, found 281.1515.



(2*E*,4*E*)-6-ethyl 1-methyl 4-(2-chlorobenzylidene)hex-2-enedioate (3s) obtained from *o*-chlorobenzaldehyde (70 mg, 0.5 mmol) as a white solid (83 mg, 54% yield): mp 55-57 ºC; IR (thin film): ν_{max} 3060, 2972, 2947, 1730, 1709, 1619, 1464, 1434, 1316, 1251, 1224, 1197, 1003, 858, 764, 687, 459 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.46 (d, J = 15.9 Hz, 1H), 7.41 (m, 1H), 7.32 (m, 1H), 7.18 (m, 2H), 7.05 (s, 1H), 5.93 (d, J = 15.9 Hz, 1H), 4.10 (q, J = 7.1 Hz, 2H), 3.68 (s, 3H), 3.28 (s, 2H), 1.18 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.4, 167.0, 147.1, 138.3, 134.0, 132.3, 130.0, 129.5, 129.4, 126.6, 118.5, 61.0, 51.5, 33.8, 14.0; HRMS calcd for $C_{16}H_{17}ClO_4Na^+$ requires 331.0708, found 331.0702.

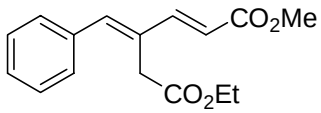


(2*E*,4*E*)-6-ethyl 1-methyl 4-(3-nitrobenzylidene)hex-2-enedioate (3t) obtained from *m*-nitrobenzaldehyde (76 mg, 0.5 mmol) as a white solid (116 mg, 73% yield): mp 69-70 ºC; IR (thin film): ν_{max} 3064, 2979, 2954, 1714, 1624, 1524, 1463, 1432, 1348, 1319, 1238, 1199, 1132, 1093, 991, 856, 810, 736, 711 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz, TMS): δ = 8.34 (s, 1H), 8.20 (d, J = 8.2 Hz, 1H), 7.75 (d, J = 7.6 Hz, 1H), 7.61-7.56 (m, 1H), 7.50 (d, J = 15.9 Hz, 1H), 7.09 (s, 1H), 6.12 (d, J = 15.9 Hz, 1H), 4.25 (q, J = 7.1 Hz, 2H), 3.80 (s, 3H), 3.44 (s, 2H), 1.32 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.1, 167.0, 148.4, 146.8, 138.3, 137.3, 134.8, 133.5, 129.6, 123.7, 123.0, 119.6, 61.6, 51.7, 33.9, 14.1; Anal calcd for $C_{16}H_{17}NO_6$: C, 60.18; H, 5.37; N, 4.39%; found: C, 59.94; H, 5.47; N, 4.25%.

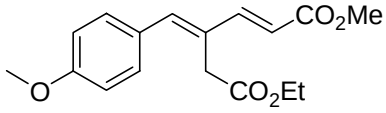


(2*E*,4*E*)-6-ethyl 1-methyl 4-(4-(trifluoromethyl)benzylidene)hex-2-enedioate: 6e (3u) obtained from *p*-trifluoromethylbenzaldehyde (87 mg, 0.5 mmol) as a white solid (120 mg, 70% yield): mp. 45-47 ºC; ^1H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.65 (d, J

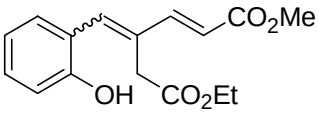
= 7.9 Hz, 2H), 7.53 (d, J = 7.9 Hz, 2H), 7.50 (d, J = 15.8 Hz, 1H), 7.09 (s, 1H), 6.06 (d, J = 15.8 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.78 (s, 3H), 3.44 (s, 2H), 1.30 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ =170.4, 167.1, 147.3, 139.8, 139.2, 132.7, 130.0 (q, J = 32.9 Hz, 1C), 129.1, 125.4(q, J = 3.3 Hz, 2C), 118.8, 61.4, 51.7, 33.7, 14.1; HRMS calcd for $C_{17}H_{17}F_3O_4Na^+$ requires 365.0971, found 365.0966.



(2*E*,4*E*)-6-ethyl 1-methyl 4-benzylidenehex-2-enedioate (3v) obtained from benzaldehyde (53 mg, 0.5 mmol) as colorless oil (86 mg, 63% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.51 (d, J = 15.8 Hz, 1H), 7.41-7.30 (m, 5H), 7.07 (s, 1H), 6.00 (d, J = 15.8 Hz, 1H), 4.21 (q, J = 7.1 Hz, 2H), 3.76 (s, 3H), 3.47 (s, 2H), 1.28 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 170.5, 167.1, 148.0, 141.6, 135.6, 130.9, 128.8, 128.4, 128.2, 117.6, 61.0, 51.4, 33.6, 14.0; HRMS calcd for $C_{16}H_{17}O_4Na^+$ requires 297.1097, found 297.1100.

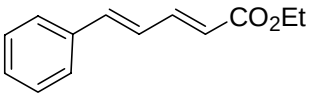


(2*E*,4*E*)-6-ethyl 1-methyl 4-(4-methoxybenzylidene)hex-2-enedioate (3w) obtained from *p*-methoxybenzaldehyde (68 mg, 0.5 mmol) as colorless oil (50 mg, 33% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.50 (d, J = 15.8 Hz, 1H), 7.37 (d, J = 8.5 Hz, 2H), 7.01 (s, 1H), 6.92 (d, J = 8.5 Hz, 2H), 5.96 (d, J = 15.8 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 3.83 (s, 3H), 3.77 (s, 3H), 3.49 (s, 2H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 170.9, 167.5, 159.7, 148.6, 141.7, 130.6, 129.2, 128.2, 116.6, 114.0, 61.2, 55.2, 51.6, 33.8, 14.1; HRMS calcd for $C_{17}H_{20}O_5Na^+$ requires 327.1203, found 327.1207.



(2*E*,4*E*)-6-ethyl 1-methyl 4-(2-hydroxybenzylidene)hex-2-enedioate (3x) obtained from salicylaldehyde (61 mg, 0.5 mmol) as colorless oil (103 mg, 71% yield), contaminated by the minor product (*Z*,*E*)-isomer with a ratio of (*E*,*E*) : (*Z*,*E*) = 8 : 1; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ = 7.56 (d, J = 15.9 Hz, 1H), 7.44 (br s, 1H),

7.27 (d, J = 7.7 Hz, 1H), 7.19-7.15 (m, 2H), 6.90-6.85 (m, 2H), 5.92 (d, J = 15.8 Hz, 1H), 4.18 (q, J = 7.1 Hz, 2H), 3.76 (s, 3H), 3.42 (s, 2H), 1.25 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS): δ = 171.5, 168.0, 154.4, 148.4, 138.2, 131.5, 130.0, 129.4, 122.6, 120.0, 116.8, 116.0, 61.3, 51.7, 33.9, 13.9; HRMS calcd for $C_{16}H_{18}O_5Na^+$ requires 313.1046, found 313.1038.



(2*E*,4*E*)-ethyl 5-phenylpenta-2,4-dienoate[5] **(4)** obtained from allenoate **2a** (101 mg, 0.5 mmol) as slightly yellow oil (79 mg, 78% yield); ^{1}H NMR ($CDCl_3$, 400 MHz, TMS) 7.45-7.41 (m, 3H), 7.36-7.29 (m, 3H), 6.91-6.82 (m, 2H), 5.98 (d, J = 15.3 Hz, 1H), 4.22 (q, J = 7.1 Hz, 2H), 1.31 (t, J = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ = 166.9, 144.4, 140.3, 136.0, 128.9, 128.7, 127.1, 126.2, 121.3, 60.2, 14.2.

Reference

1. Daigle, D. J. *Inorg. Synth.* **1998**, *32*, 40.

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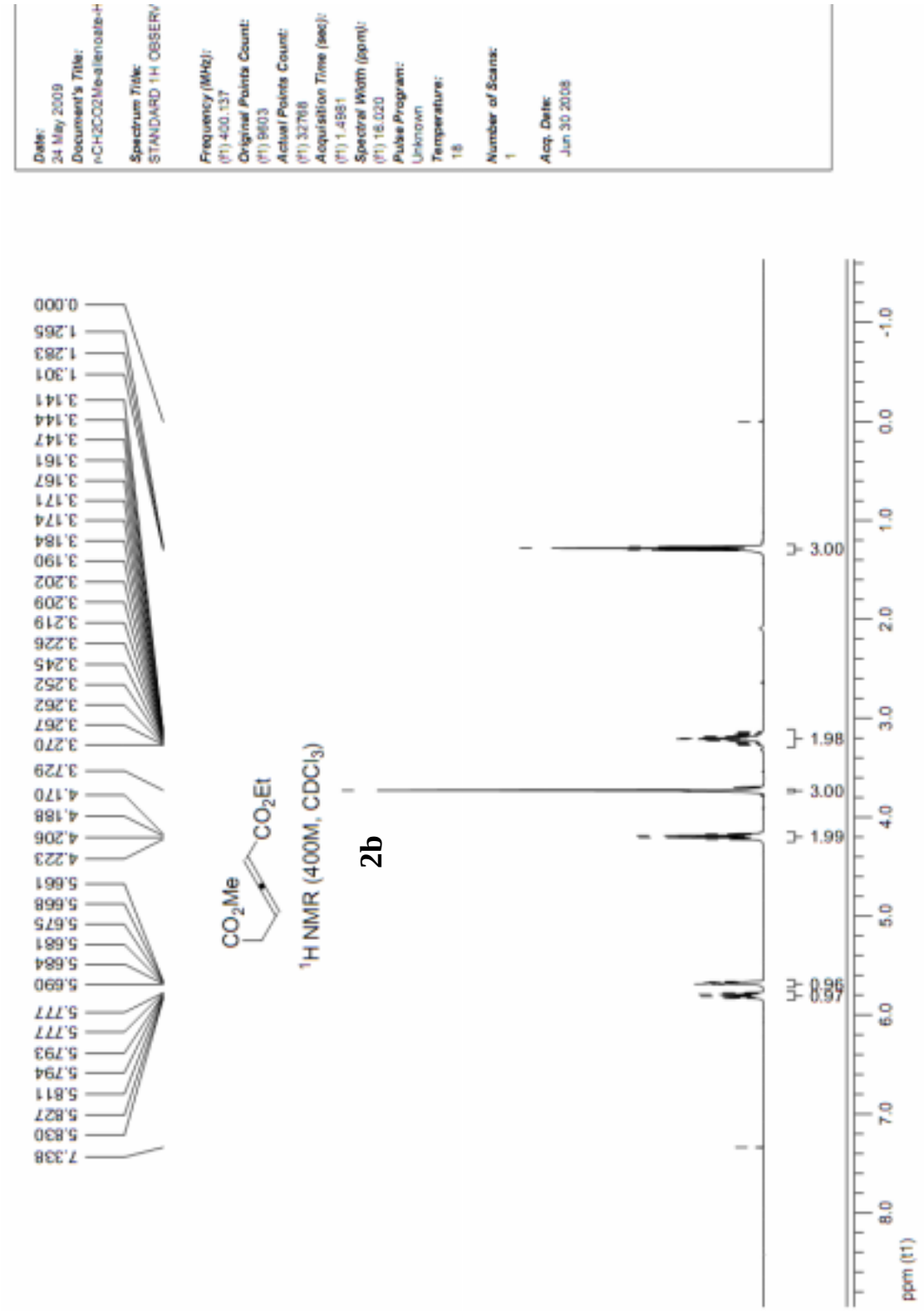
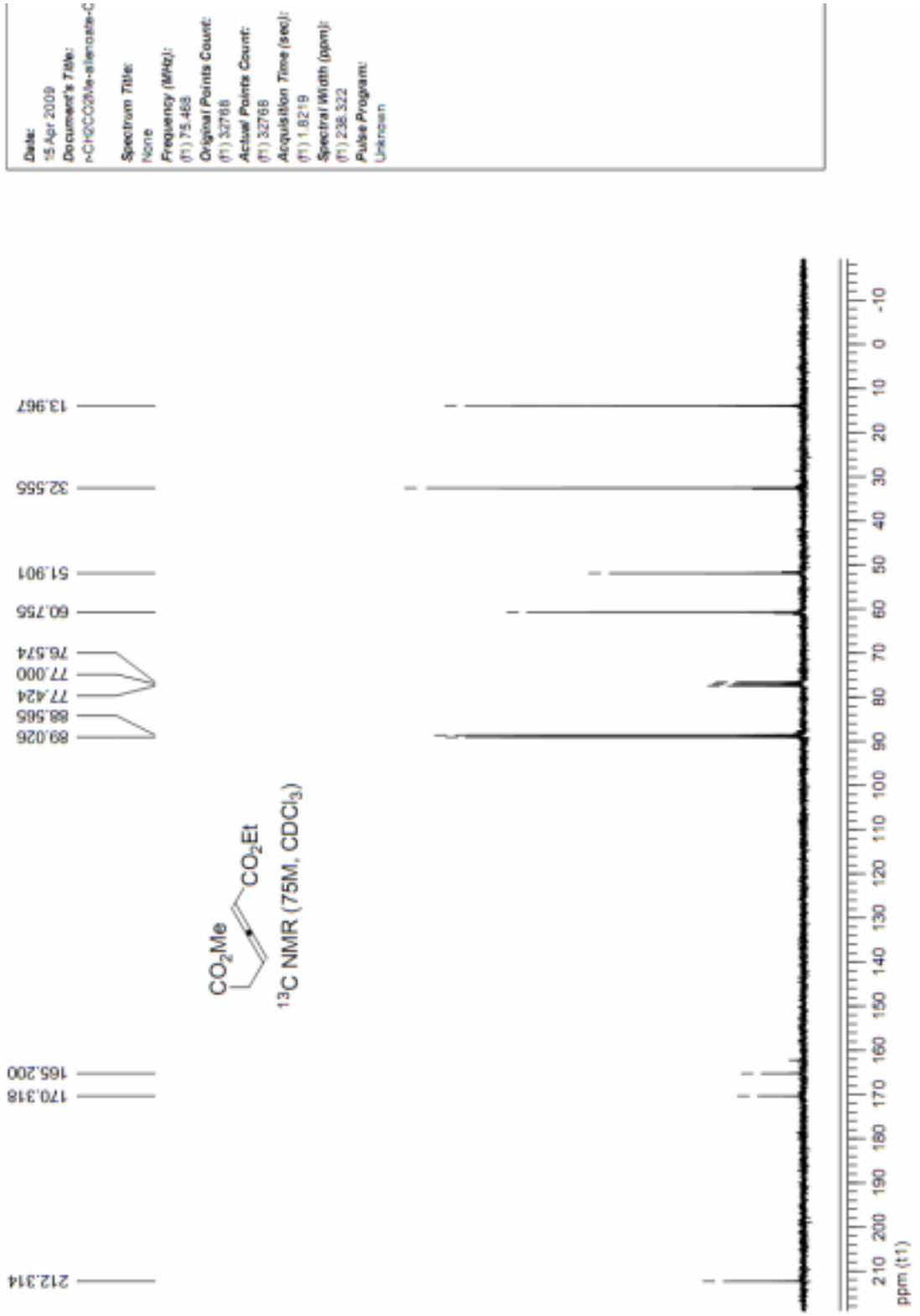
^{1}H and ^{13}C NMR Spectra

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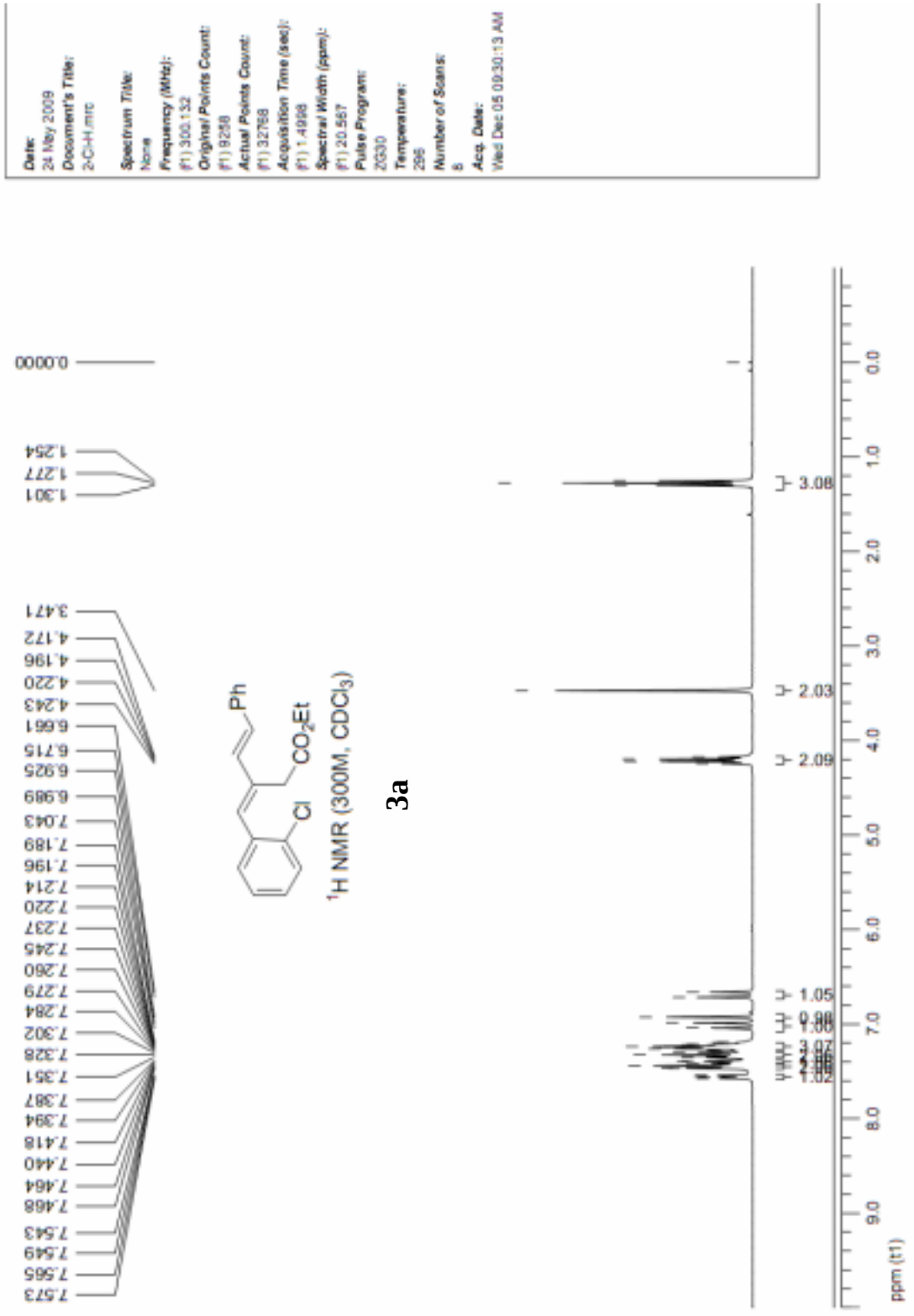
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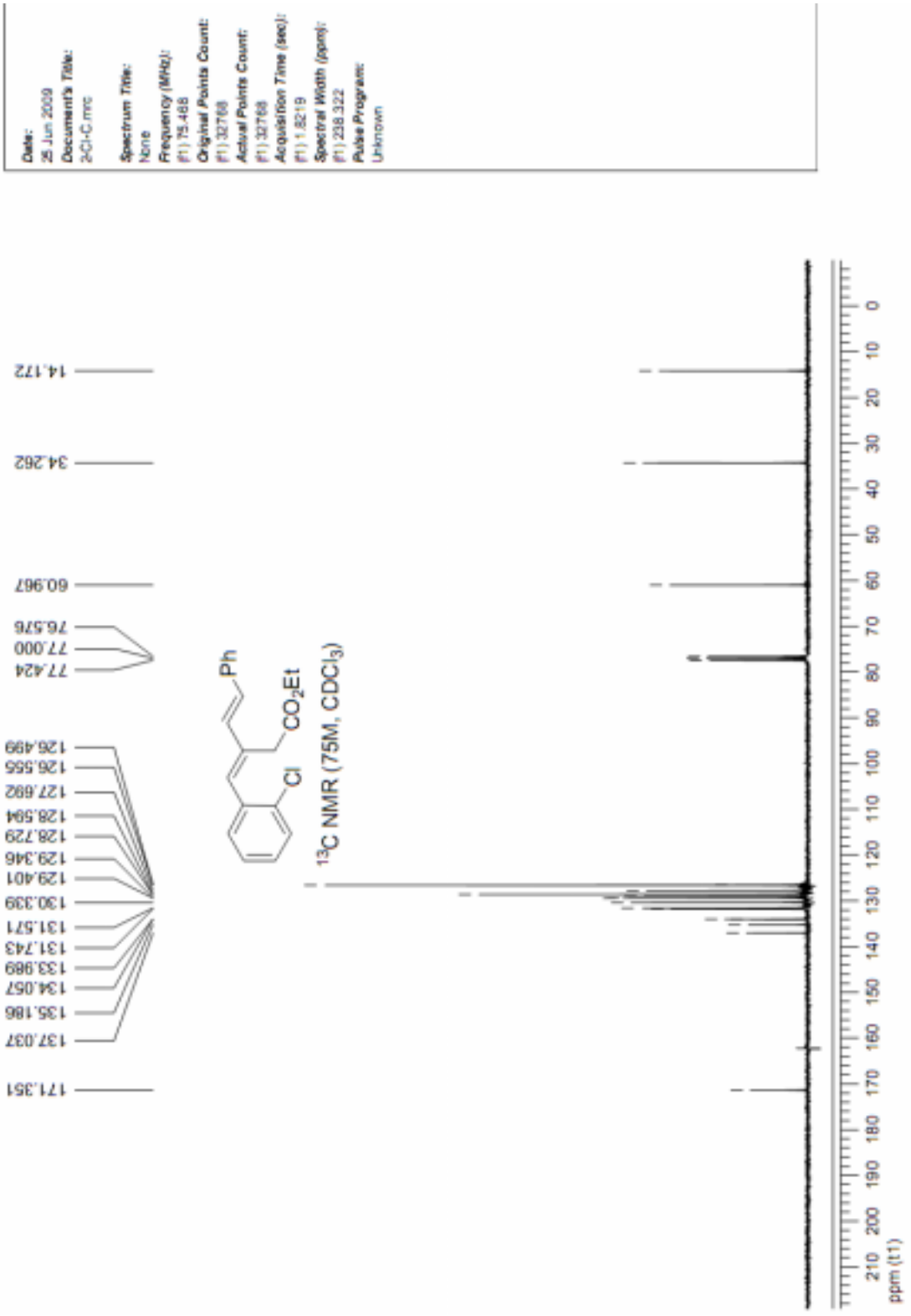
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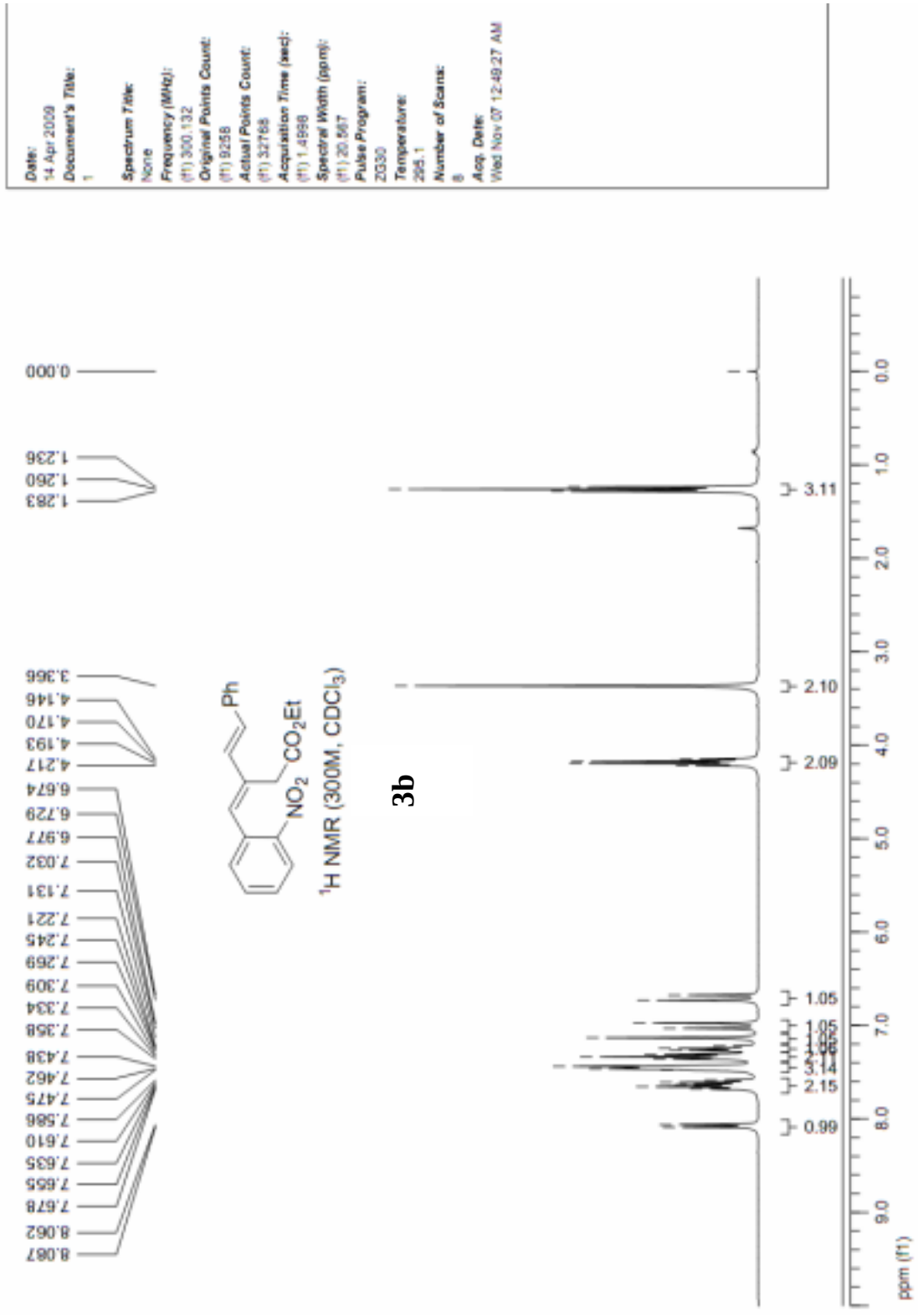
ppm (t1)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14 Apr 2020
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Actual Points Count:
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Acquisition Time (sec):
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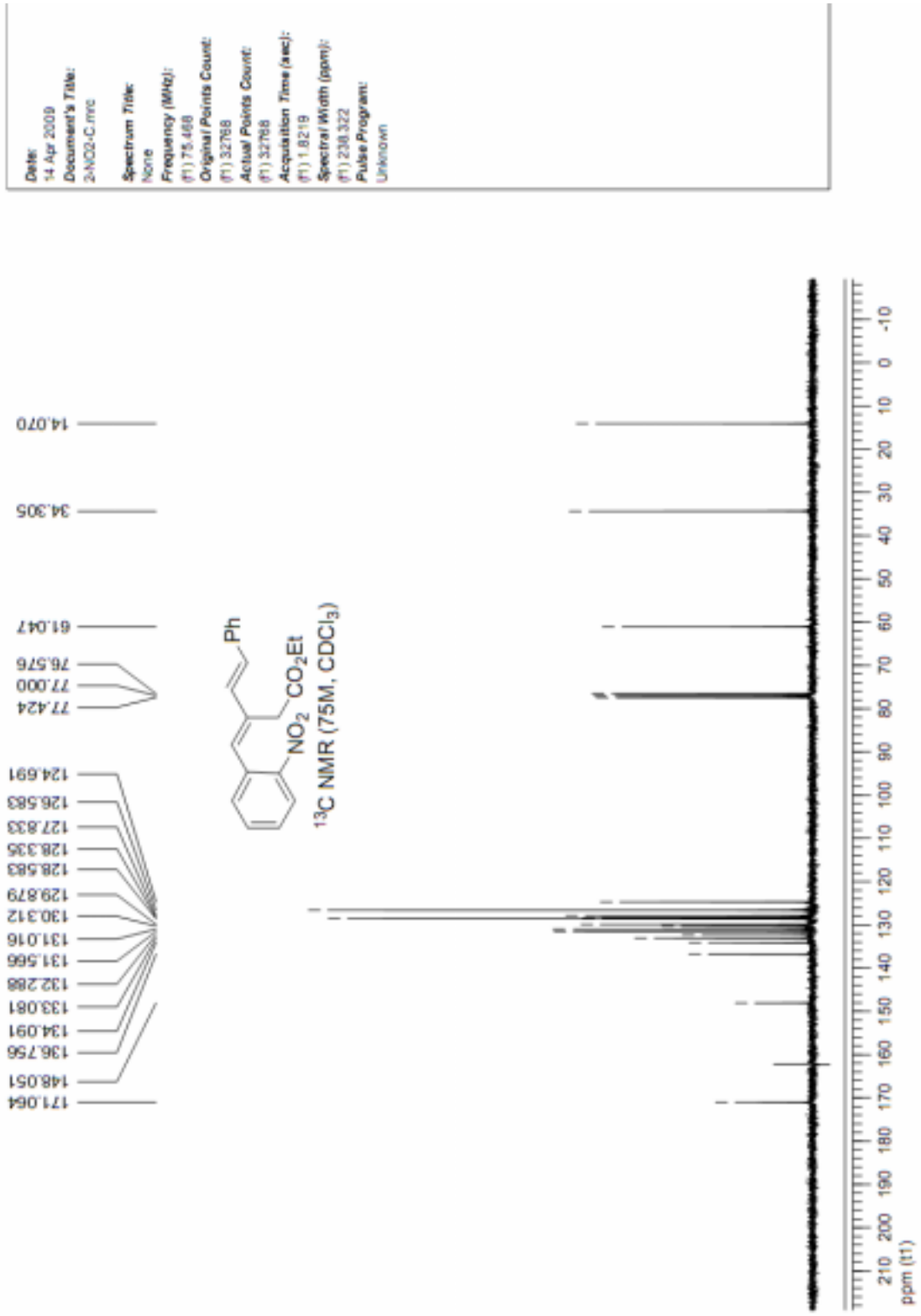
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
295.1
Number of Scans:
8
Acq. Date:
Wed Nov 07 12:49:27 AM
0.000
1.236
1.260
1.283
3.366
4.146
4.170
4.193
4.217
6.674
6.729
6.977
7.032
7.131
7.221
7.245
7.269
7.309
7.334
7.358
7.438
7.462
7.475
7.586
7.610
7.635
7.655
7.678
8.062
8.087
Ph
CO2Et
NO2
1H NMR (300M, CDCl3)
3b
3.11
2.10
2.09
1.05
1.05
3.14
2.15
0.99
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (f1)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2-NO2-C.mnr
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 238.322
Pulse Program:
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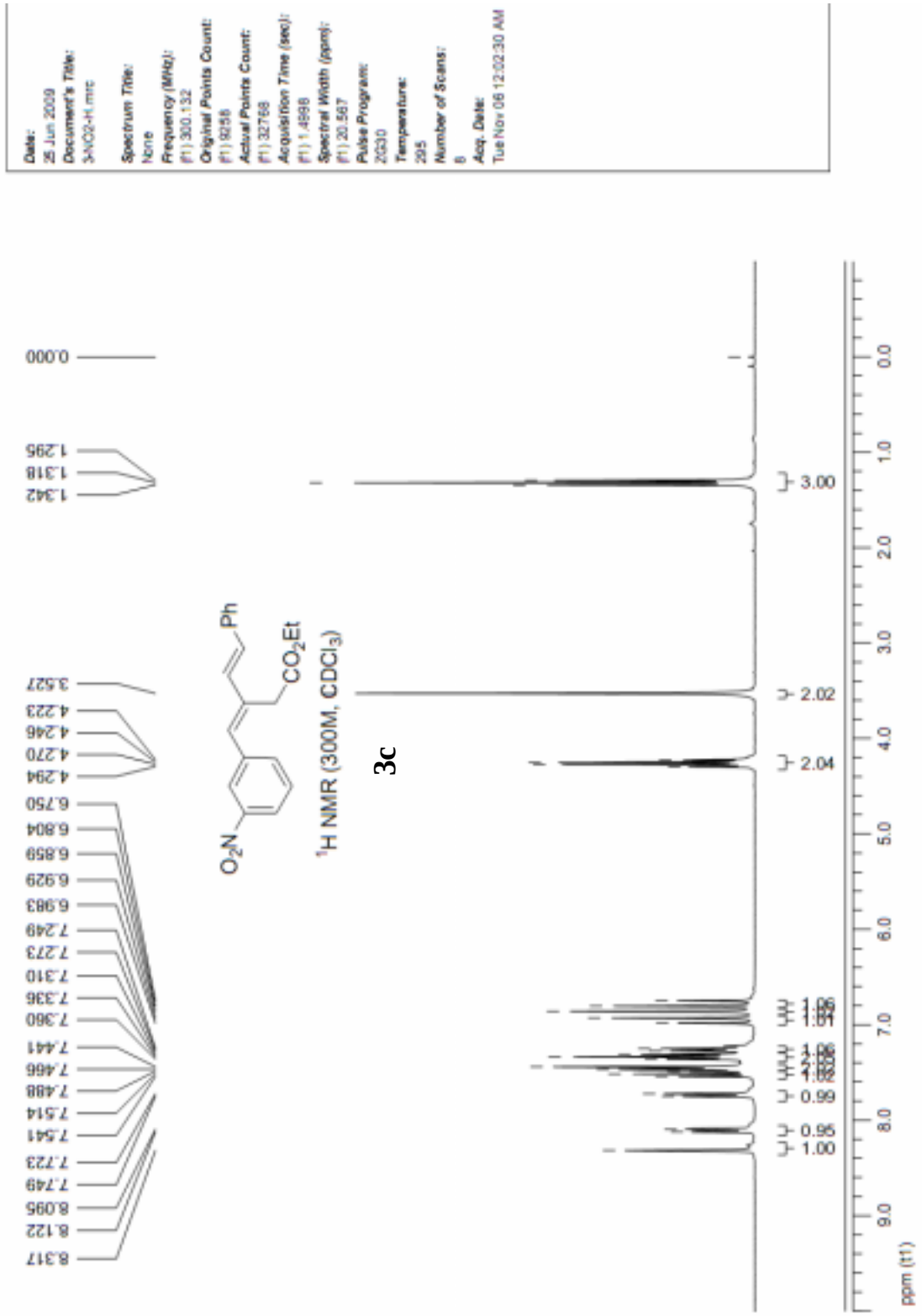
14.070
34.305
61.047
76.576
77.000
77.424
124.691
126.583
127.833
128.335
128.583
129.879
130.312
131.016
131.566
132.288
133.081
134.091
136.756
148.051
171.064
Ph
CO2Et
NO2
13C NMR (75M, CDCl3)
ppm (t1)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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2d30
Temperature:
295
Number of Scans:
8
Acq. Date:
Tue Nov 06 12:02:30 AM
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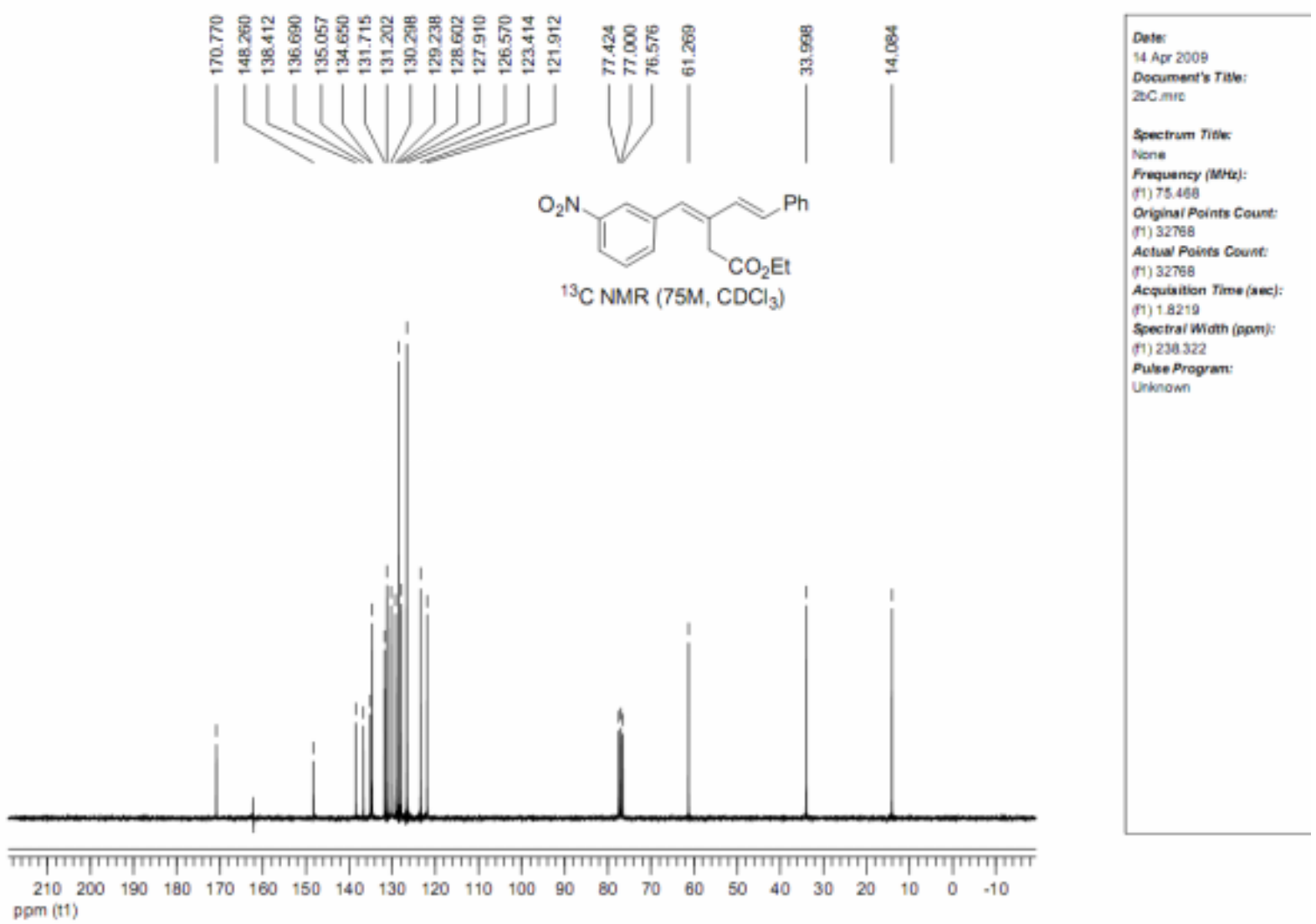
1.295
1.318
1.342
3.527
4.223
4.246
4.270
4.294
6.750
6.804
6.859
6.929
6.983
7.249
7.273
7.310
7.336
7.360
7.441
7.466
7.488
7.514
7.541
7.723
7.749
8.095
8.122
8.317
Ph
CO2Et
O2N
1H NMR (300M, CDCl3)
3c
3.00
2.02
2.04
0.99
0.96
1.00
ppm (t1)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148.260
138.412
136.690
135.057
134.650
131.715
131.202
130.298
129.238
128.602
127.910
126.570
123.414
121.912
77.424
77.000
76.576
61.269
33.998
14.084
O2N
Ph
CO2Et
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
14 Apr 2009
Document's Title:
2bC.mrc
Spectrum Title:
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Frequency (MHz):
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Original Points Count:
(f1) 32768
Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 238.322
Pulse Program:
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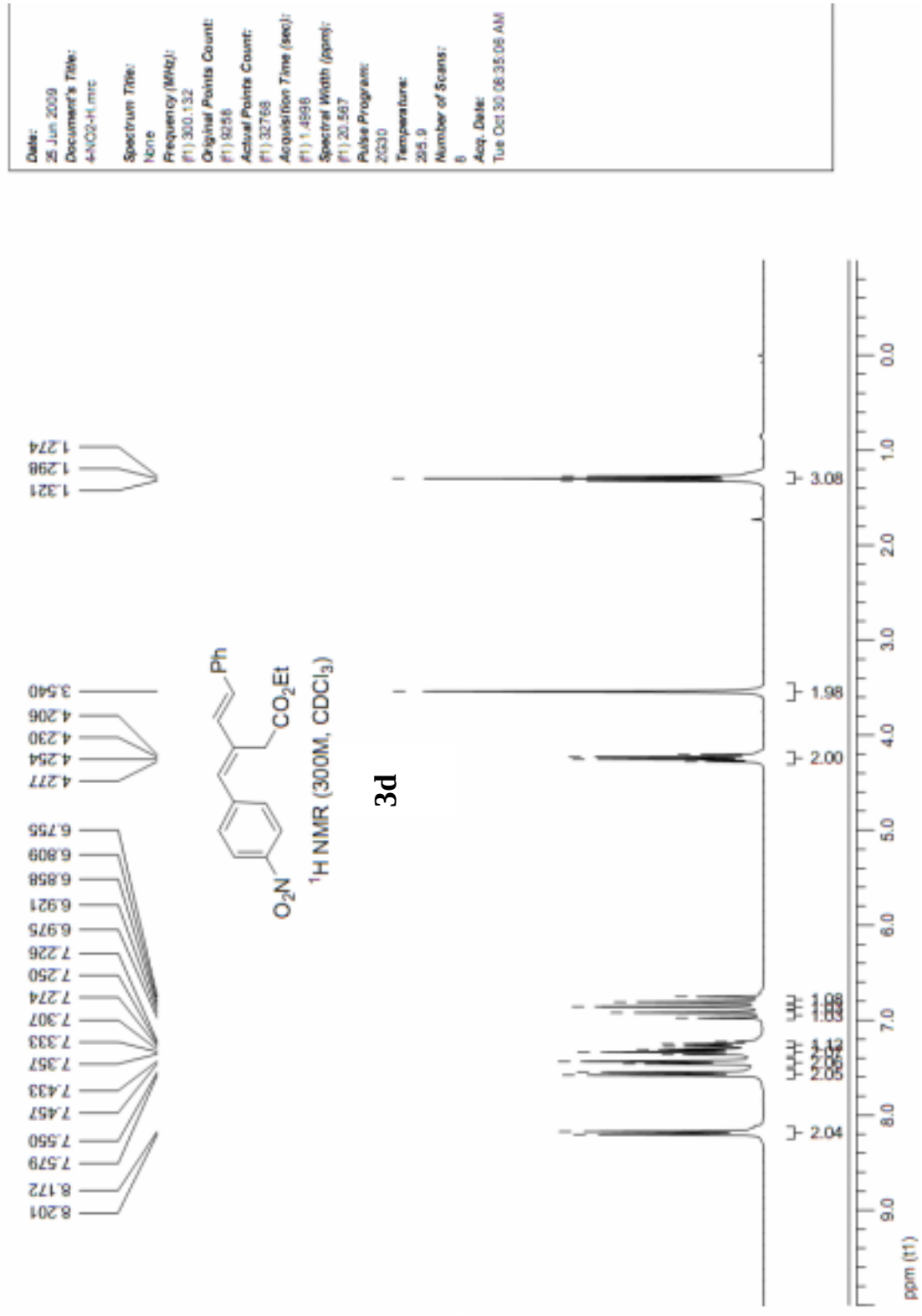
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Acquisition Time (sec):
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Spectral Width (ppm):
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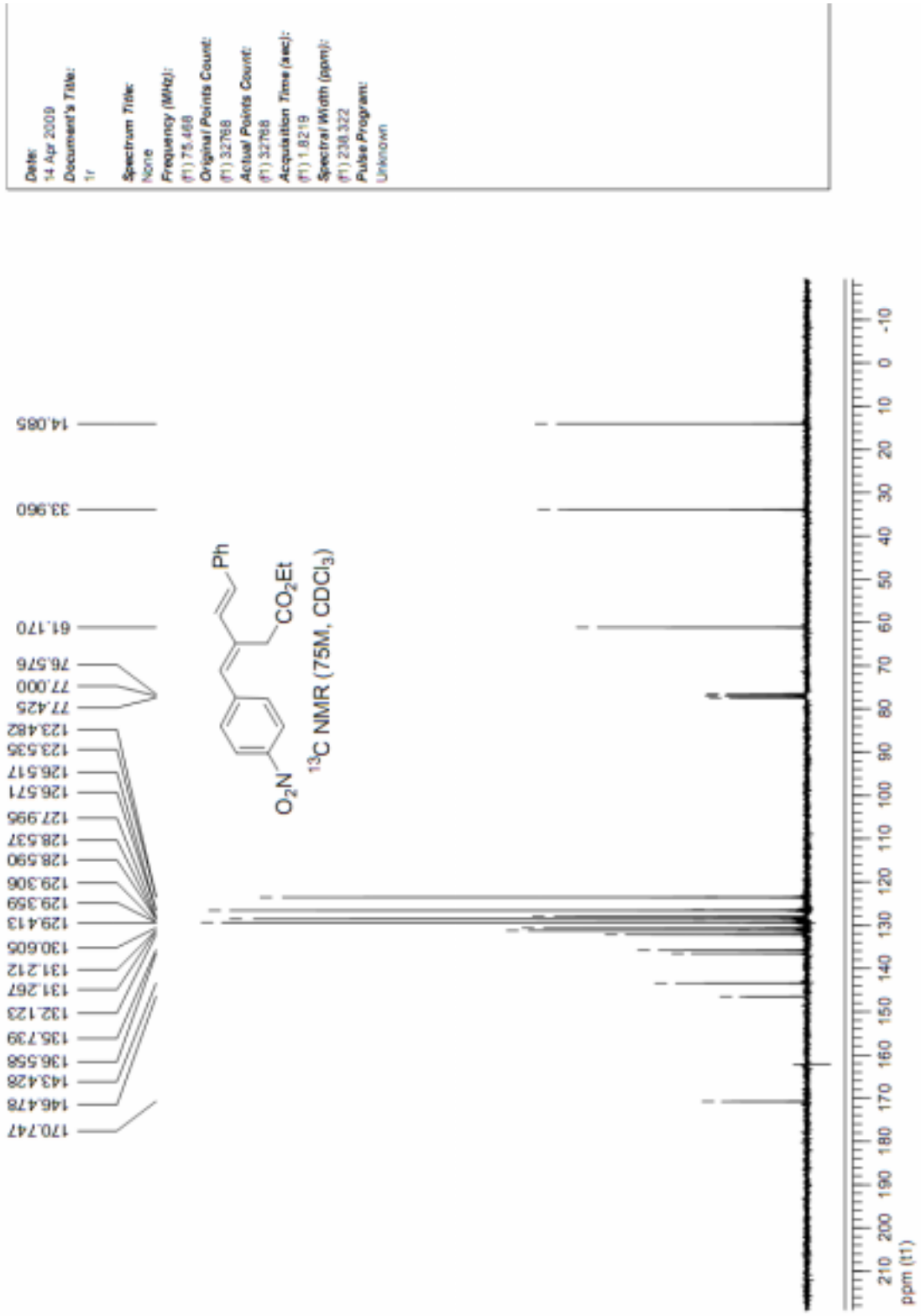
Pulse Program:
2d30
Temperature:
295.9
Number of Scans:
8
Acq. Date:
Tue Oct 30 08:35:06 AM
1.274
1.298
1.321
3.540
4.206
4.230
4.254
4.277
6.755
6.809
6.858
6.921
6.975
7.226
7.250
7.274
7.307
7.333
7.357
7.433
7.457
7.550
7.579
8.172
8.201
Ph
CO2Et
O2N
1H NMR (300M, CDCl3)
3d
3.08
1.98
2.00
2.04
ppm (t1)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14 Apr 2009
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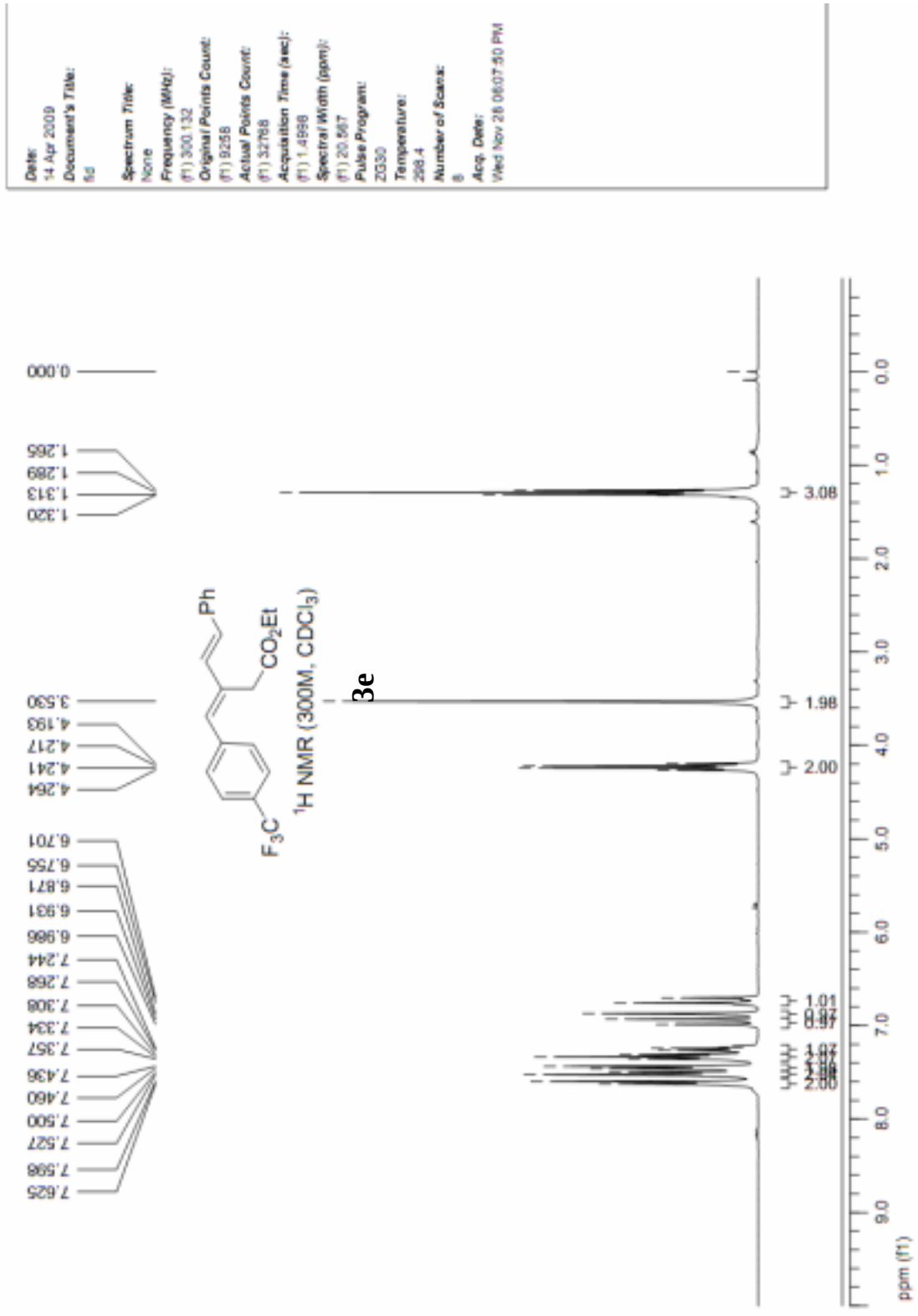
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
14.085
33.960
61.170
76.576
77.000
77.425
123.482
123.535
126.517
126.571
127.995
128.537
128.590
129.306
129.359
129.413
130.605
131.212
131.267
132.123
135.739
136.558
143.428
146.478
170.747
Ph
CO2Et
O2N
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)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14 Apr 2009
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Spectrum Title:
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Actual Points Count:
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Acquisition Time (sec):
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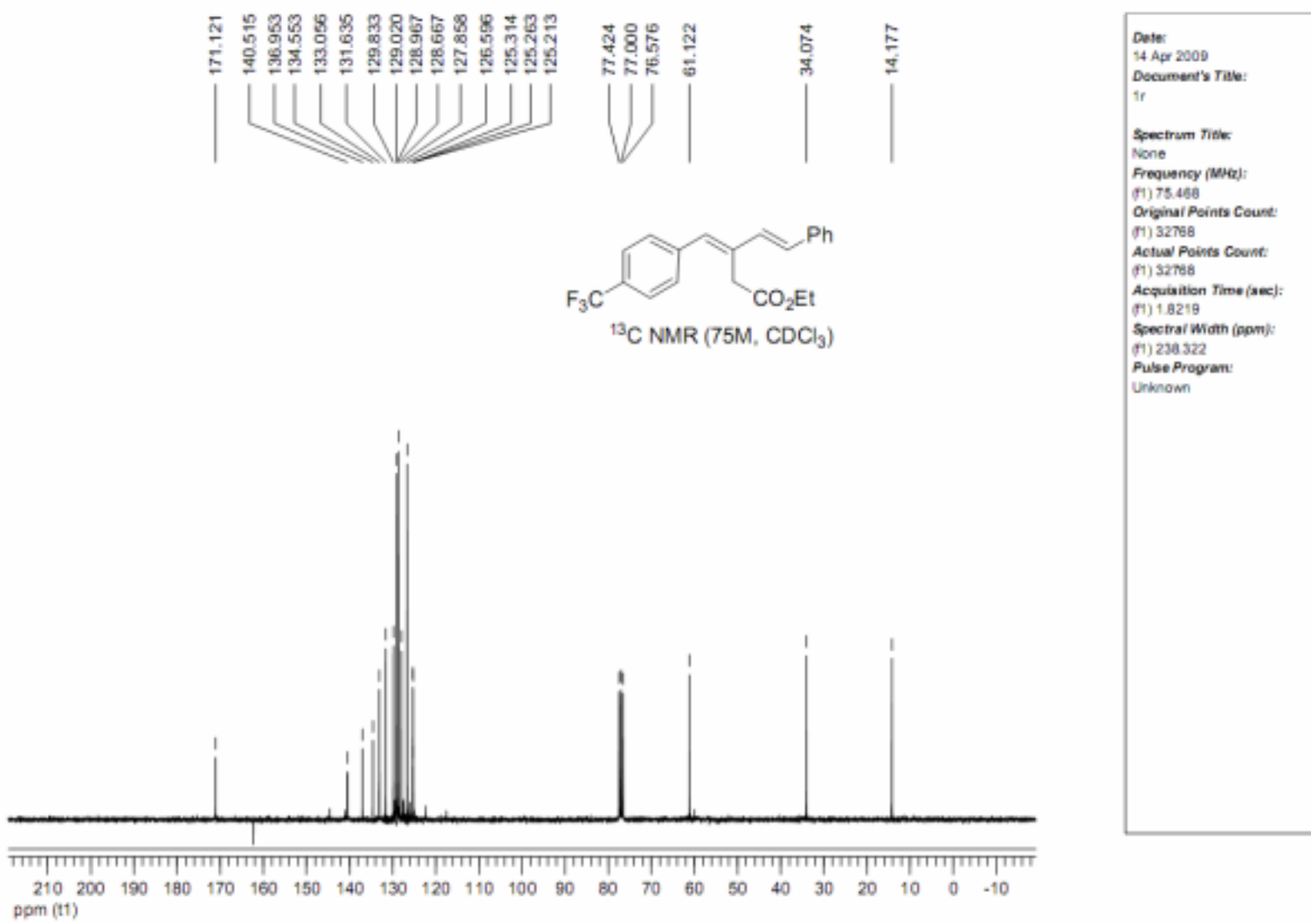
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
298.4
Number of Scans:
8
Acq. Date:
Wed Nov 26 08:07:50 PM
0.000
1.265
1.289
1.313
1.320
3.530
4.193
4.217
4.241
4.264
6.701
6.755
6.871
6.931
6.986
7.244
7.268
7.308
7.334
7.357
7.436
7.460
7.500
7.527
7.598
7.625
Ph
CO2Et
F3C
1H NMR (300M, CDCl3)
3e
3.08
1.98
2.00
1.01
0.97
ppm (f1)


171.121
140.515
136.953
134.553
133.056
131.635
129.833
129.020
128.967
128.667
127.858
126.596
125.314
125.263
125.213
77.424
77.000
76.576
61.122
34.074
14.177
Ph
F3C
CO2Et
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
14 Apr 2009
Document's Title:
1r
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
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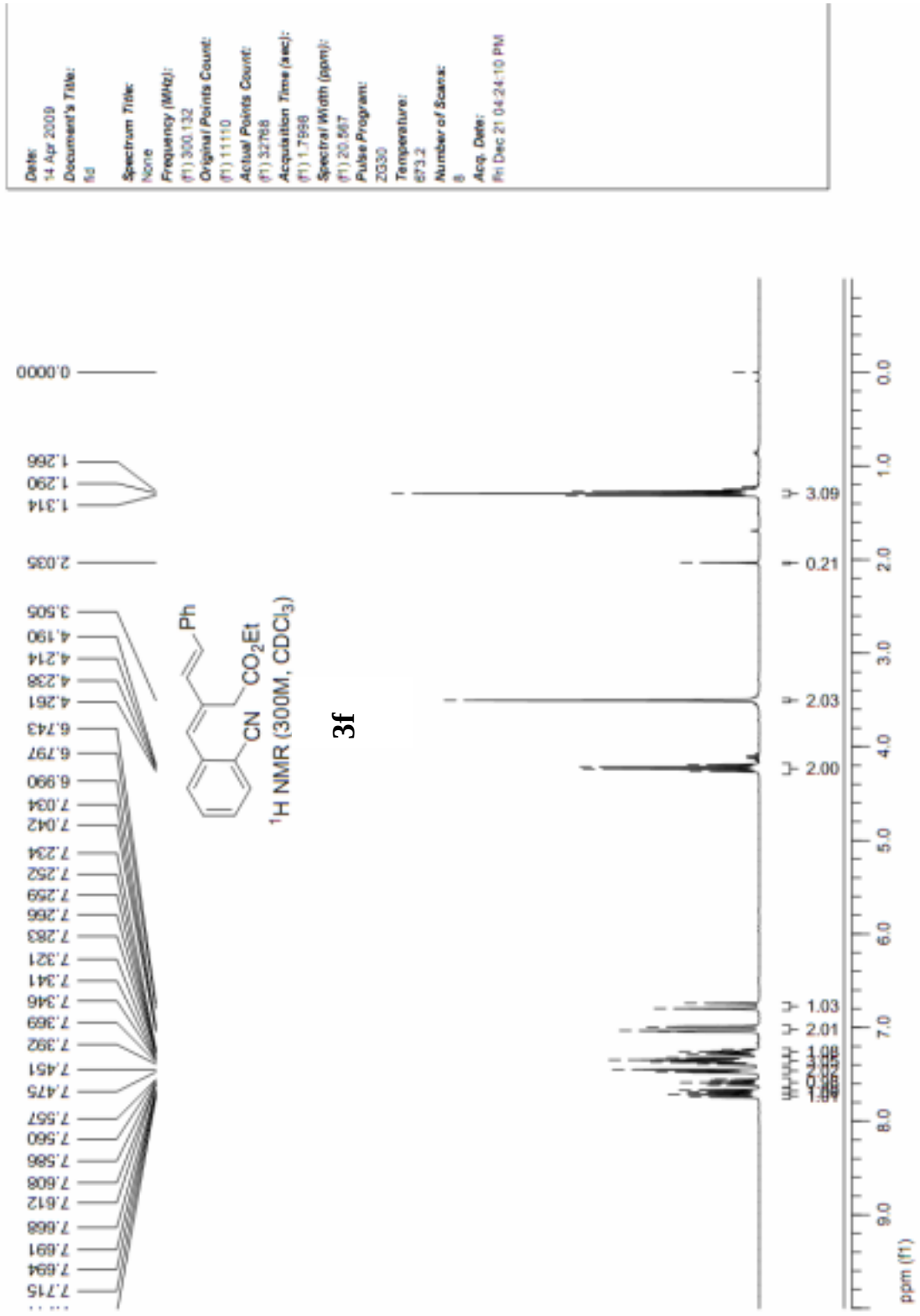
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown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Spectrum Title:
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Frequency (MHz):
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Original Points Count:
(f1) 11110
Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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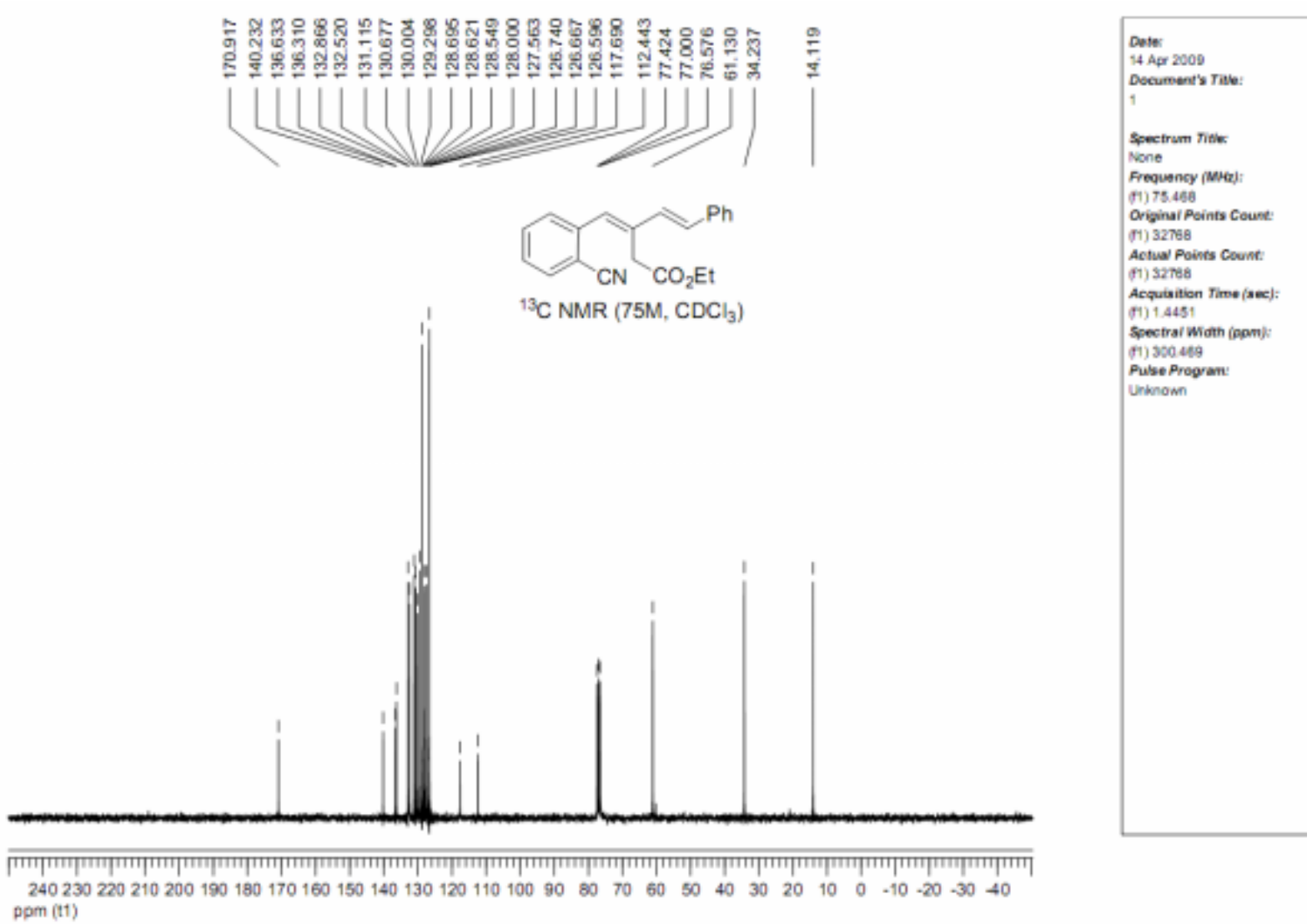
Pulse Program:
2D30
Temperature:
673.2
Number of Scans:
8
Acq. Date:
Fri Dec 21 04:24:10 PM
0.0000
1.266
1.290
1.314
2.035
3.505
4.190
4.214
4.238
4.261
6.743
6.797
6.990
7.034
7.042
7.234
7.252
7.259
7.266
7.283
7.321
7.341
7.346
7.369
7.392
7.451
7.475
7.557
7.560
7.586
7.608
7.612
7.668
7.691
7.694
7.715
Ph
CO2Et
CN
1H NMR (300M, CDCl3)
3f
3.09
0.21
2.03
2.00
1.03
2.01
ppm (f1)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140.232
136.633
136.310
132.866
132.520
131.115
130.677
130.004
129.298
128.695
128.621
128.549
128.000
127.563
126.740
126.667
126.596
117.690
112.443
77.424
77.000
76.576
61.130
34.237
14.119
Ph
CN
CO2Et
13C NMR (75M, CDCl3)
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
ppm (t1)
Date:
14 Apr 2009
Document's Title:
1
Spectrum Title:
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Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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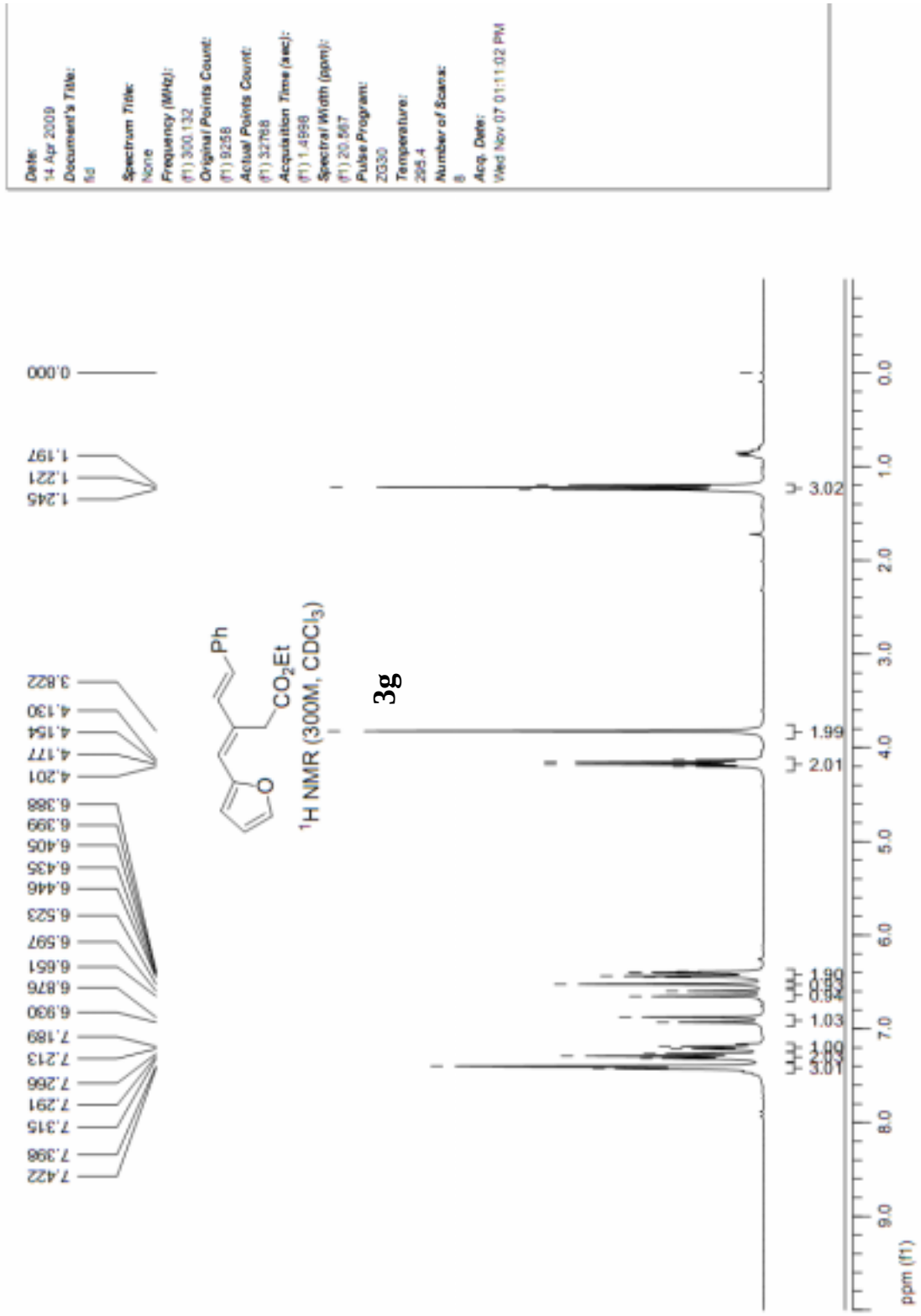
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Actual Points Count:
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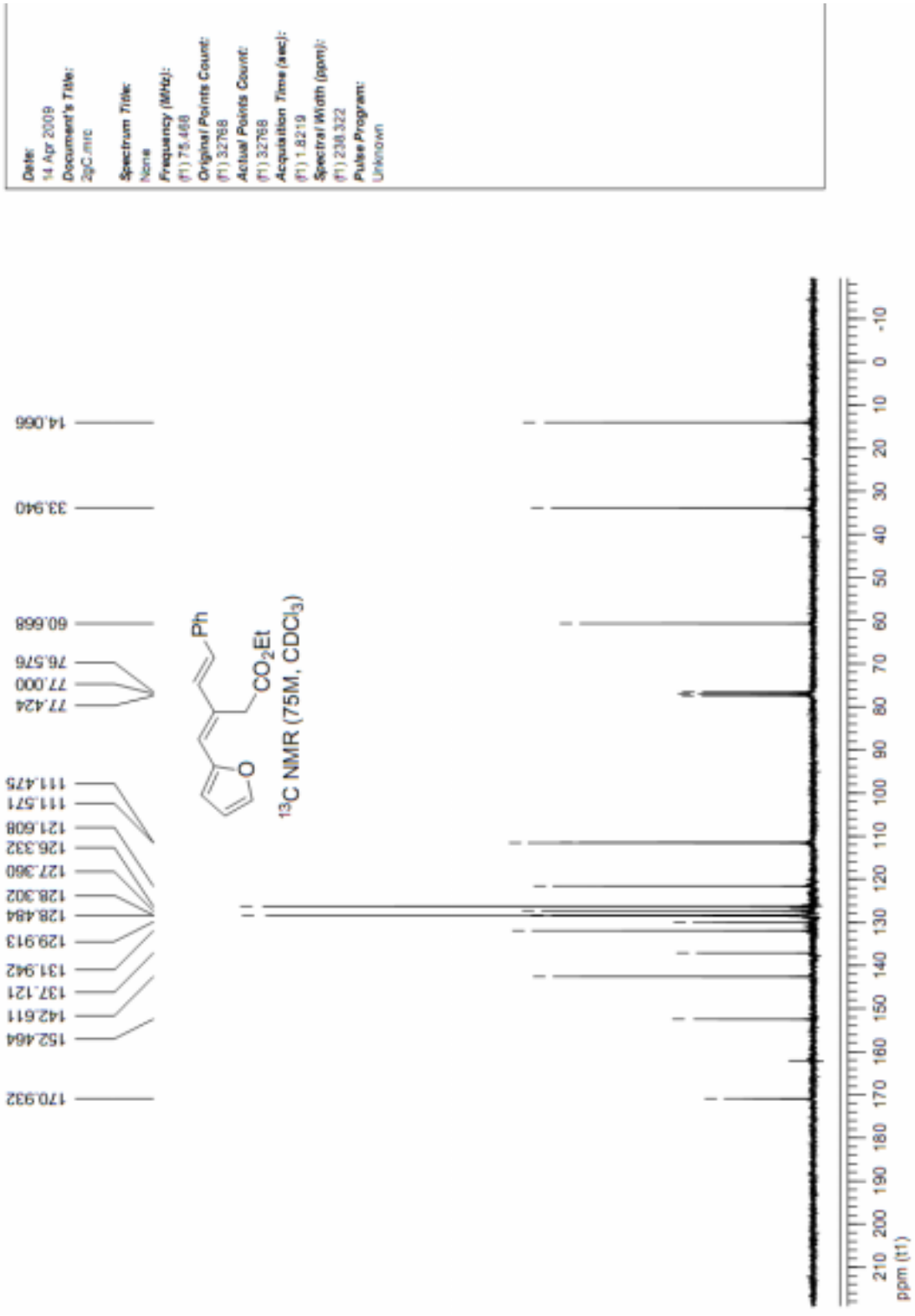
Acquisition Time (sec):
(f1) 1.4998
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
295.4
Number of Scans:
8
Acq. Date:
Wed Nov 07 01:11:02 PM
0.000
1.197
1.221
1.245
3.822
4.130
4.154
4.177
4.201
6.388
6.399
6.405
6.435
6.446
6.523
6.597
6.651
6.876
6.930
7.189
7.213
7.266
7.291
7.315
7.398
7.422
Ph
CO2Et
O
1H NMR (300M, CDCl3)
3g
3.02
1.99
2.01
1.03
3.01
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (f1)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Actual Points Count:
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Acquisition Time (sec):
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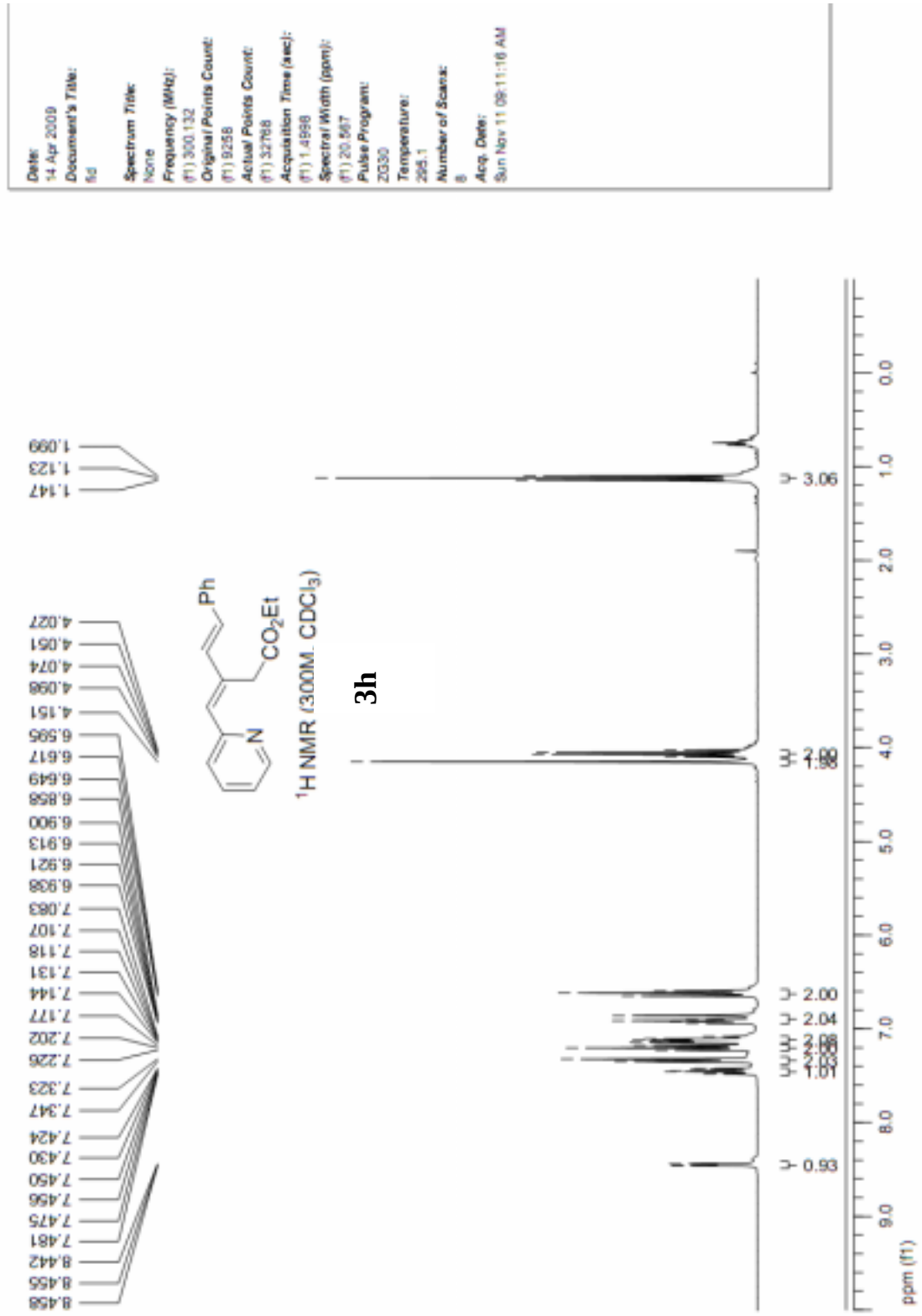
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
Ph
CO2Et
O
13C NMR (75M, CDCl3)
14.066
33.940
60.668
76.576
77.000
77.424
111.475
111.571
121.608
126.332
127.360
128.302
128.484
129.913
131.942
137.121
142.611
152.464
170.932
ppm (t1)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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
2D30
Temperature:
295.1
Number of Scans:
8
Acq. Date:
Sun Nov 11 09:11:16 AM
Ph
CO2Et
N
3h
1H NMR (300M, CDCl3)
1.099
1.123
1.147
4.027
4.051
4.074
4.098
4.151
6.596
6.617
6.649
6.858
6.900
6.913
6.921
6.938
7.083
7.107
7.118
7.131
7.144
7.177
7.202
7.226
7.323
7.347
7.424
7.430
7.450
7.456
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8.442
8.455
8.458
3.06
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2.04
1.01
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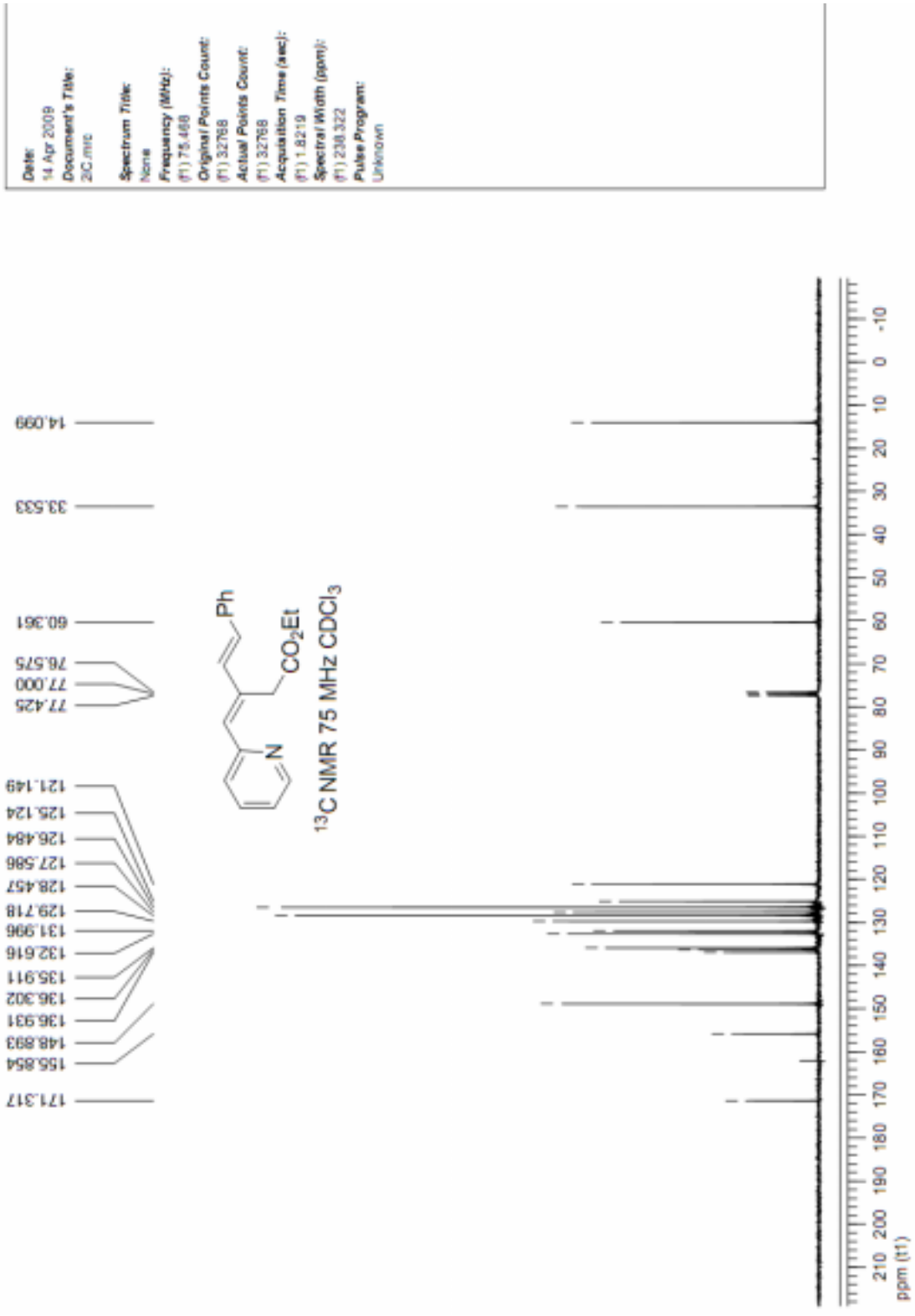
ppm (f1)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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 238.322
Pulse Program:
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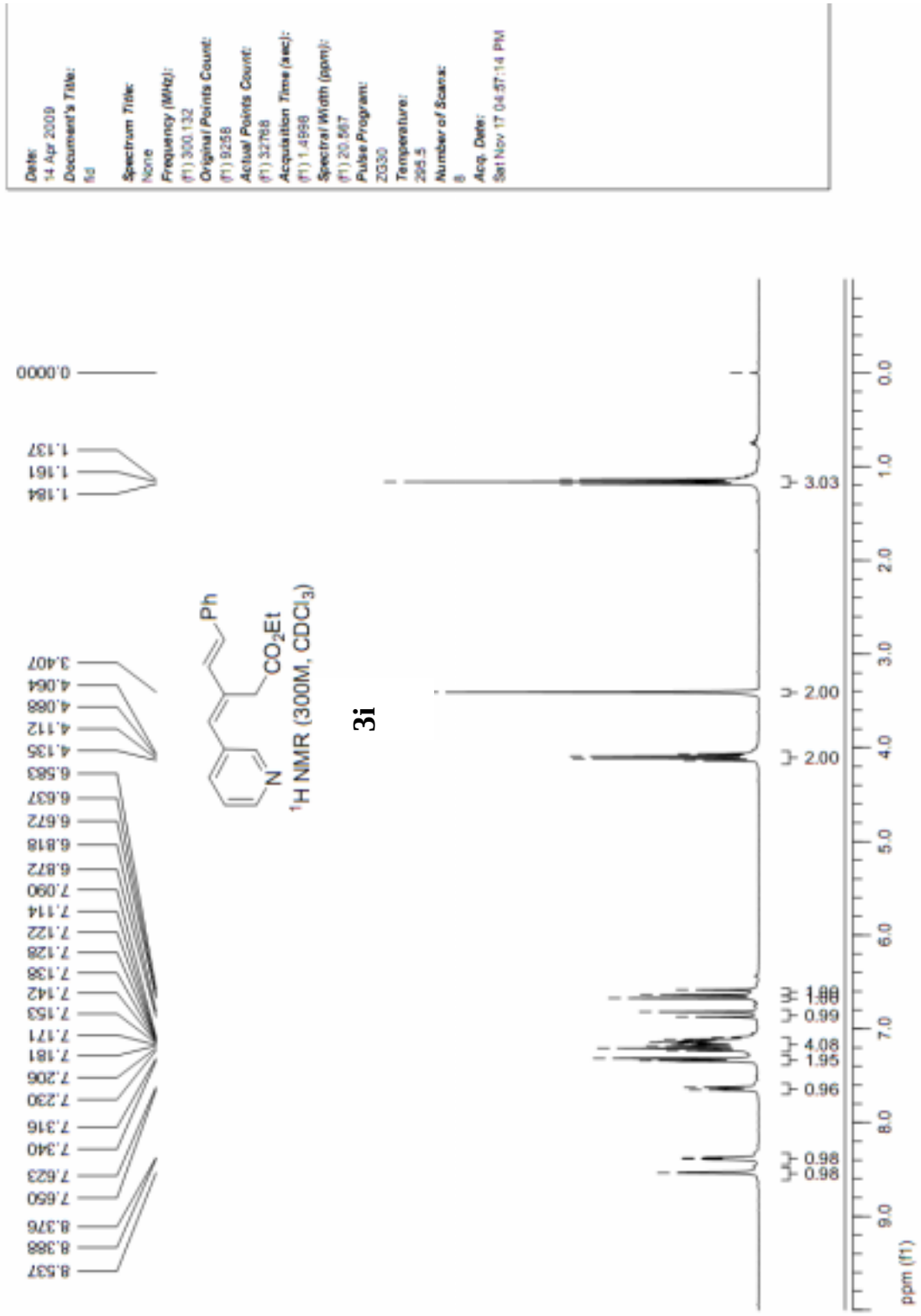
33.533
60.361
76.575
77.000
77.425
121.149
125.124
126.484
127.586
128.457
129.718
131.996
132.616
135.911
136.302
136.931
148.893
155.854
171.317
Ph
CO2Et
N
13C NMR 75 MHz CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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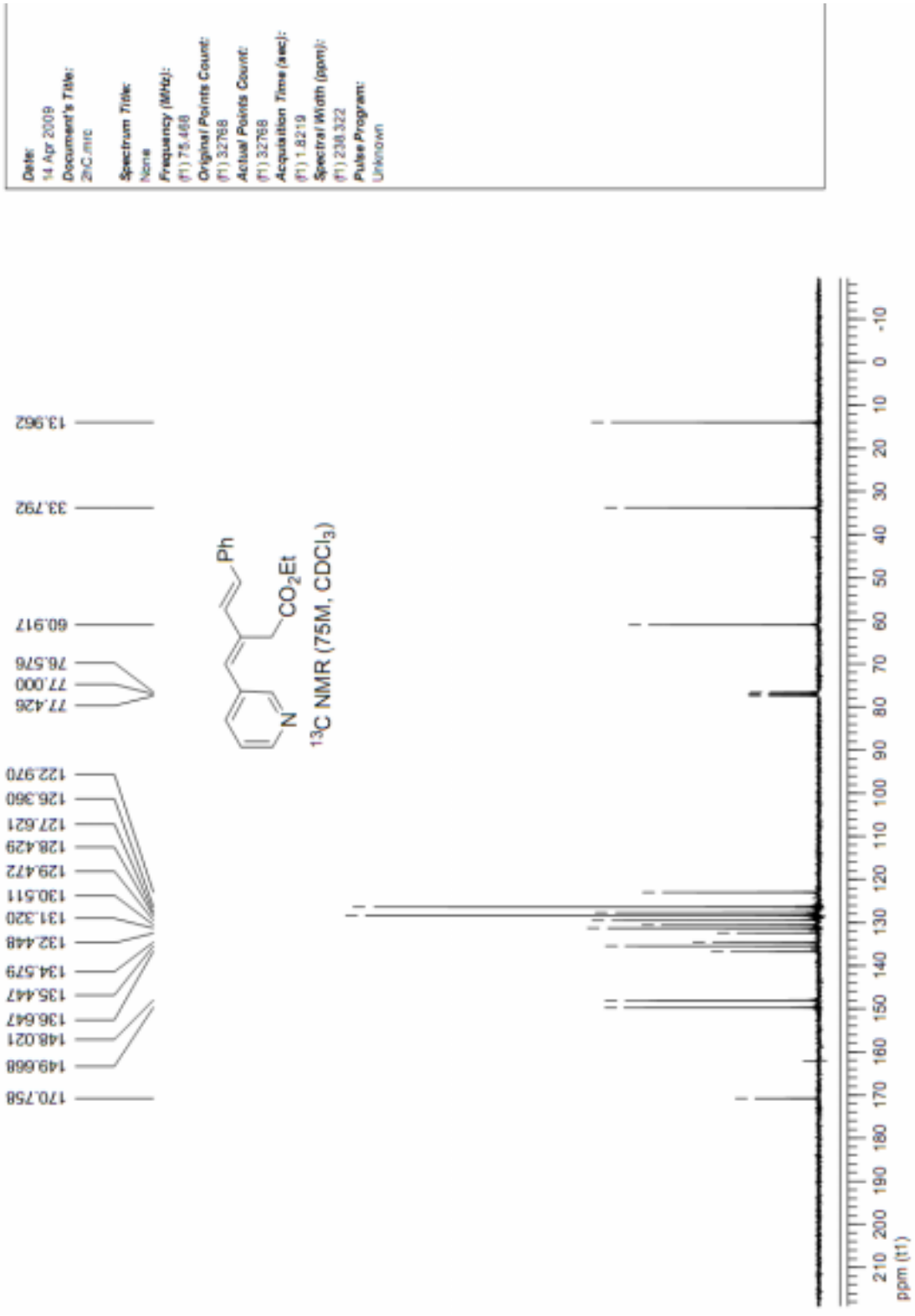
Pulse Program:
2D30
Temperature:
295.5
Number of Scans:
8
Acq. Date:
Sat Nov 17 04:57:14 PM
0.0000
1.137
1.161
1.184
3.407
4.064
4.088
4.112
4.135
6.583
6.637
6.672
6.818
6.872
7.090
7.114
7.122
7.128
7.138
7.142
7.153
7.171
7.181
7.206
7.230
7.316
7.340
7.623
7.650
8.376
8.388
8.537
Ph
CO2Et
N
1H NMR (300M, CDCl3)
3i
3.03
2.00
2.00
1.00
1.00
0.99
4.08
1.95
0.96
0.98
0.98
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (f1)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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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33.792
60.917
76.576
77.000
77.426
122.970
126.360
127.621
128.429
129.472
130.511
131.320
132.448
134.579
135.447
136.647
148.021
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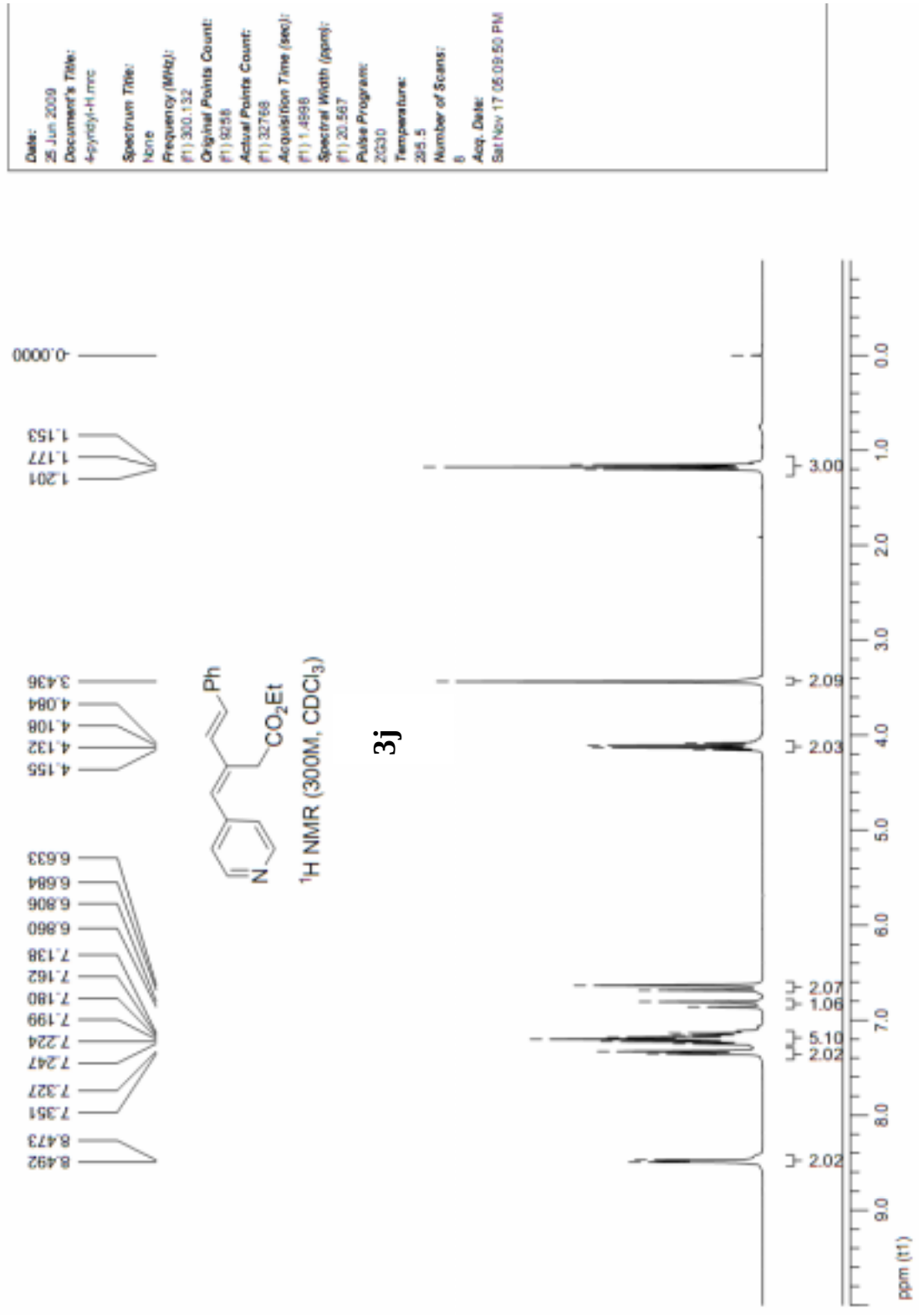
170.758
Ph
CO2Et
N
13C NMR (75M, CDCl3)
ppm (t1)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4-pyridyl-H.mnr
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
2d30
Temperature:
295.5
Number of Scans:
8
Acq. Date:
Sat Nov 17 05:09:50 PM
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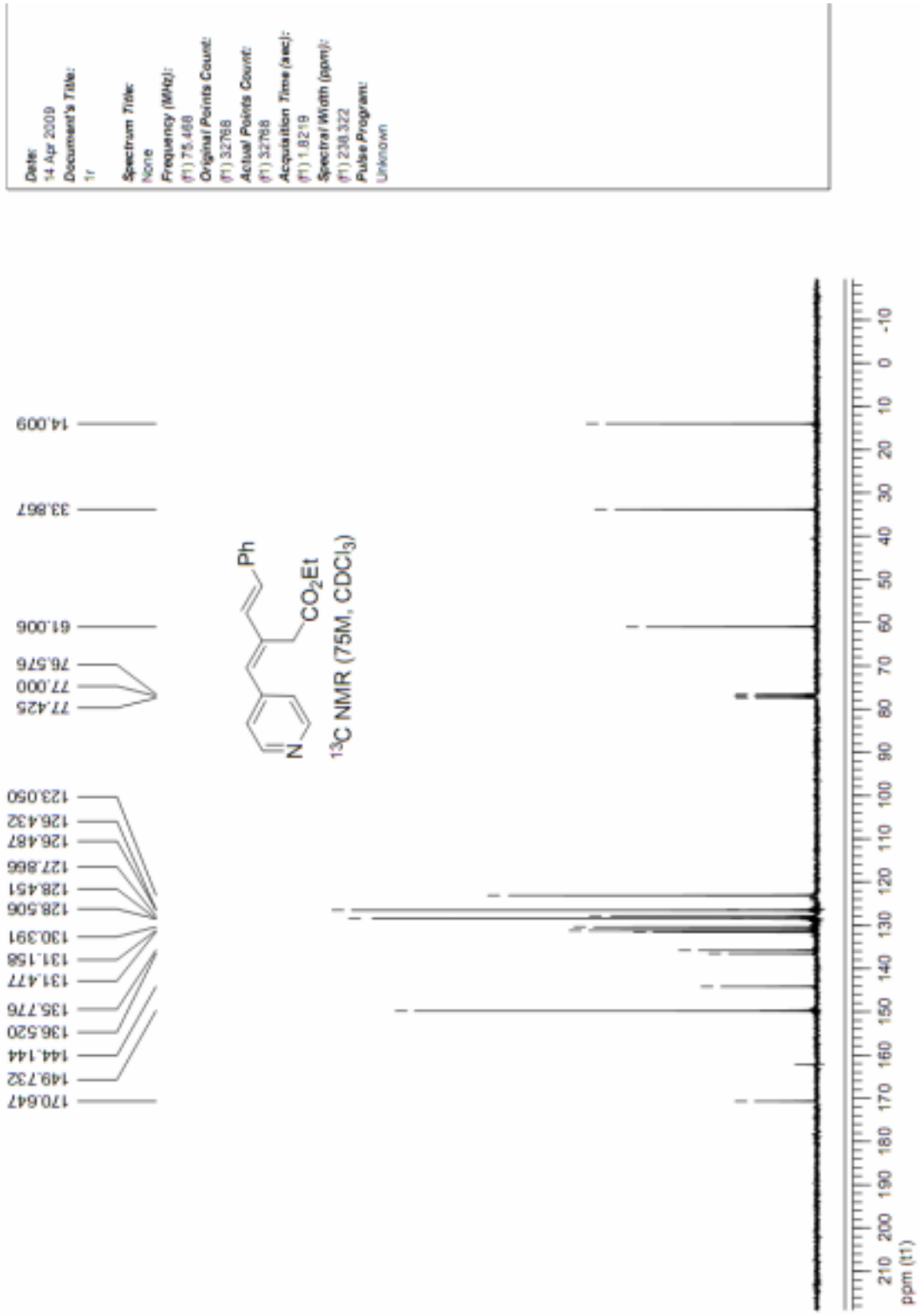
1.153
1.177
1.201
3.436
4.084
4.108
4.132
4.155
6.633
6.684
6.806
6.860
7.138
7.162
7.180
7.199
7.224
7.247
7.327
7.351
8.473
8.492
Ph
CO2Et
N
1H NMR (300M, CDCl3)
3j
3.00
2.09
2.03
2.07
1.06
5.10
2.02
2.02
ppm (t1)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1r
Spectrum Title:
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Frequency (MHz):
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Actual Points Count:
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Acquisition Time (sec):
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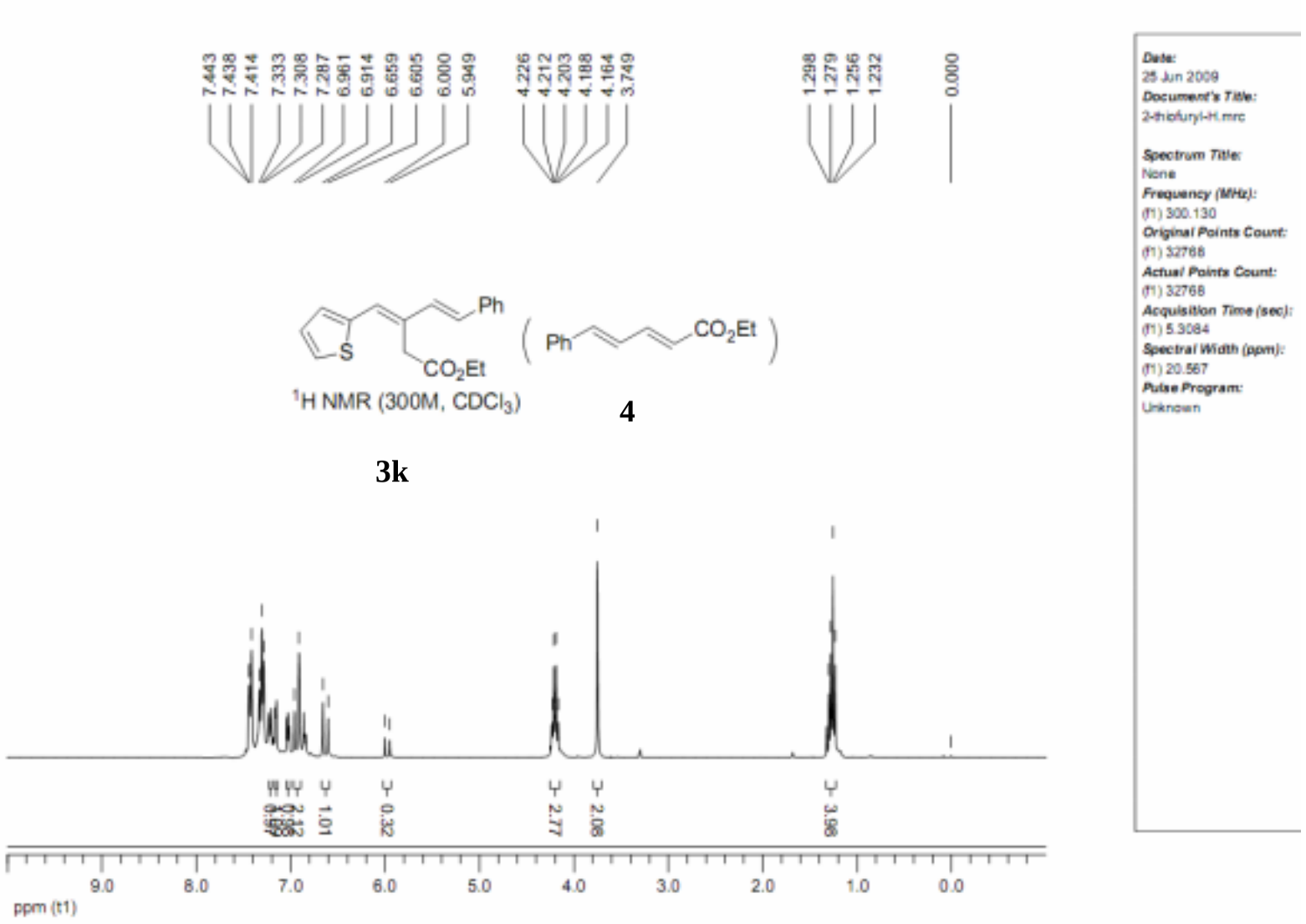
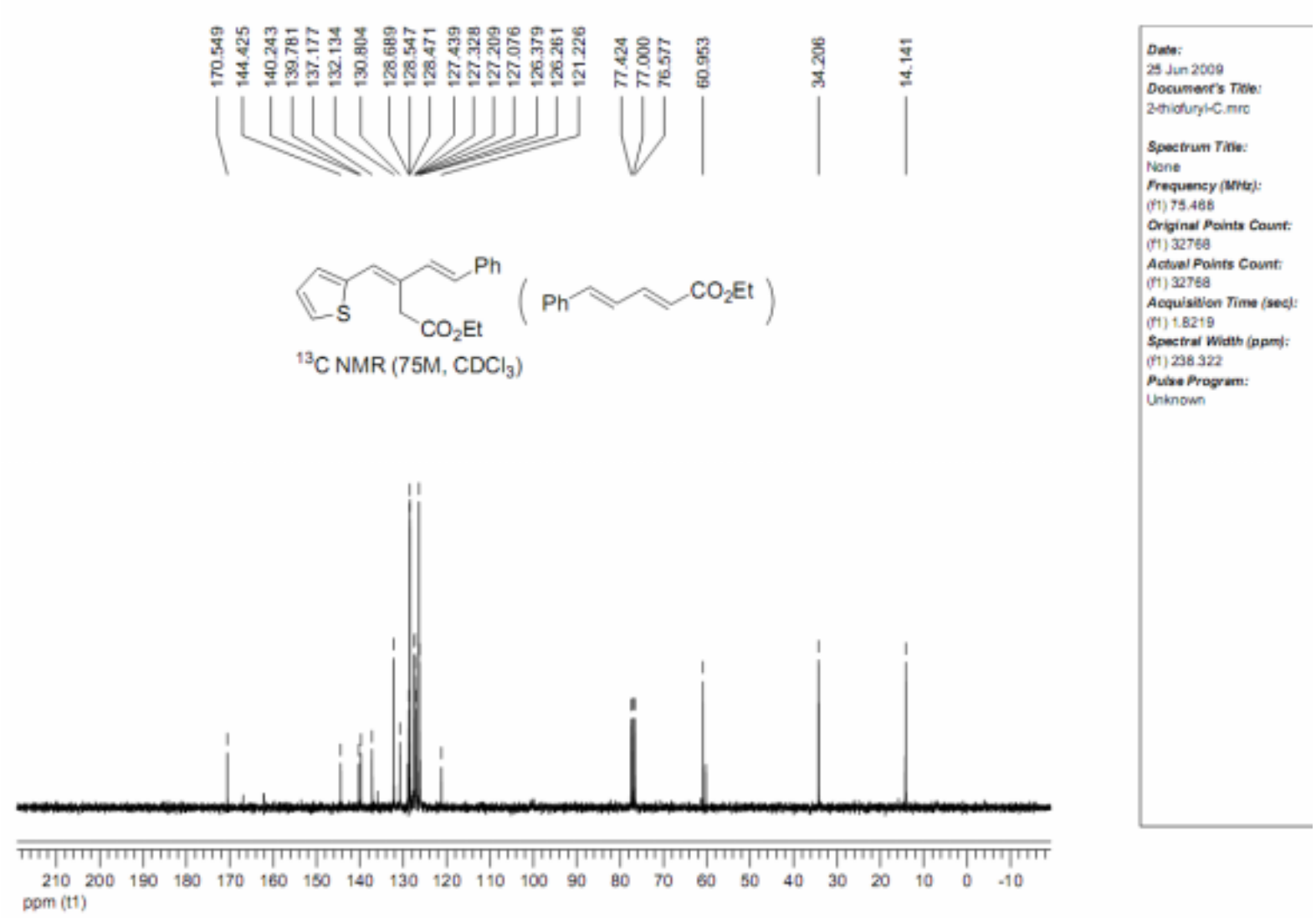
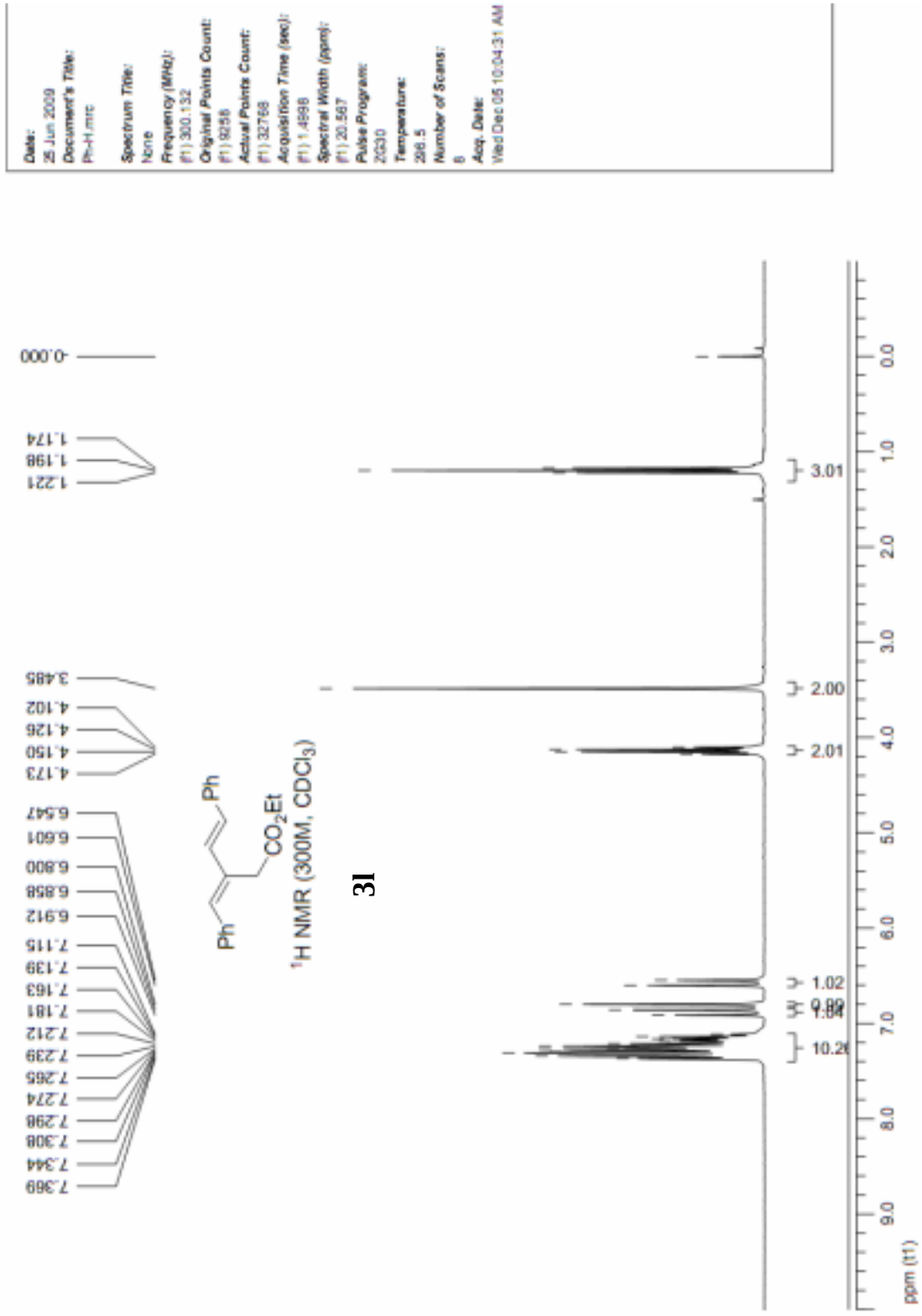
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
Ph
CO2Et
N
13C NMR (75M, CDCl3)
14.009
33.867
61.006
76.576
77.000
77.425
123.050
126.432
126.487
127.866
128.451
128.506
130.391
131.158
131.477
135.776
136.520
144.144
149.732
170.647
ppm (t1)


7.443
7.438
7.414
7.333
7.308
7.287
6.961
6.914
6.659
6.605
6.000
5.949
4.226
4.212
4.203
4.188
4.164
3.749
1.298
1.279
1.256
1.232
0.000
Ph
S
CO2Et
Ph
CO2Et
1H NMR (300M, CDCl3)
4
3k
1.01
0.32
2.77
2.08
3.98
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
Date:
25 Jun 2009
Document's Title:
2-thiofuryl-H.mrc
Spectrum Title:
None
Frequency (MHz):
(f1) 300.130
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 5.3084
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
Unknown


170.549
144.425
140.243
139.781
137.177
132.134
130.804
128.689
128.547
128.471
127.439
127.328
127.209
127.076
126.379
126.261
121.226
77.424
77.000
76.577
60.953
34.206
14.141
Ph
S
CO2Et
Ph
CO2Et
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
26 Jun 2009
Document's Title:
2-thiofuryl-C.mrc
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown


Data:
25 Jun 2009
Document's Title:
Ph-H.mnr
Spectrum Title:
None
Frequency (MHz):
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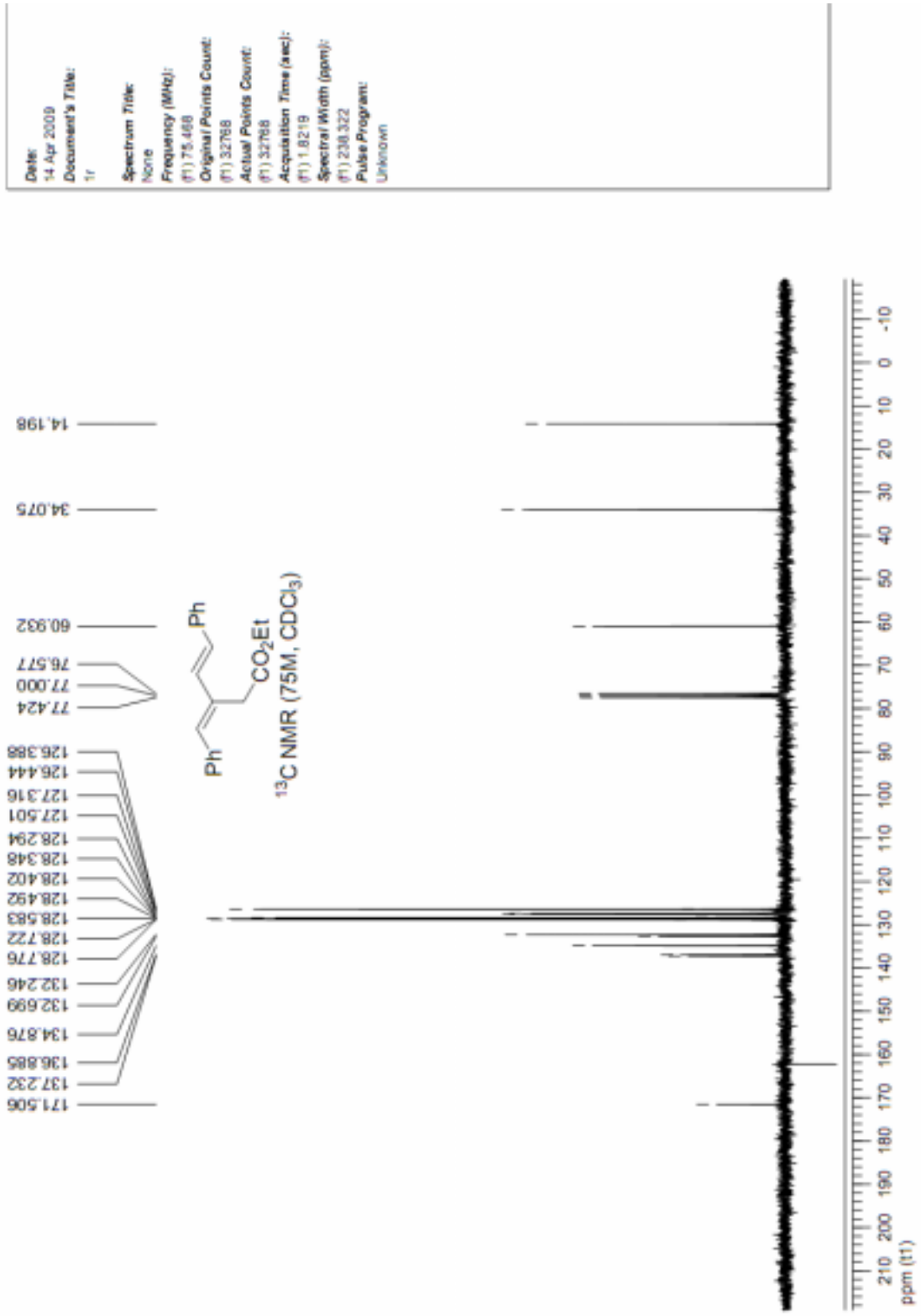
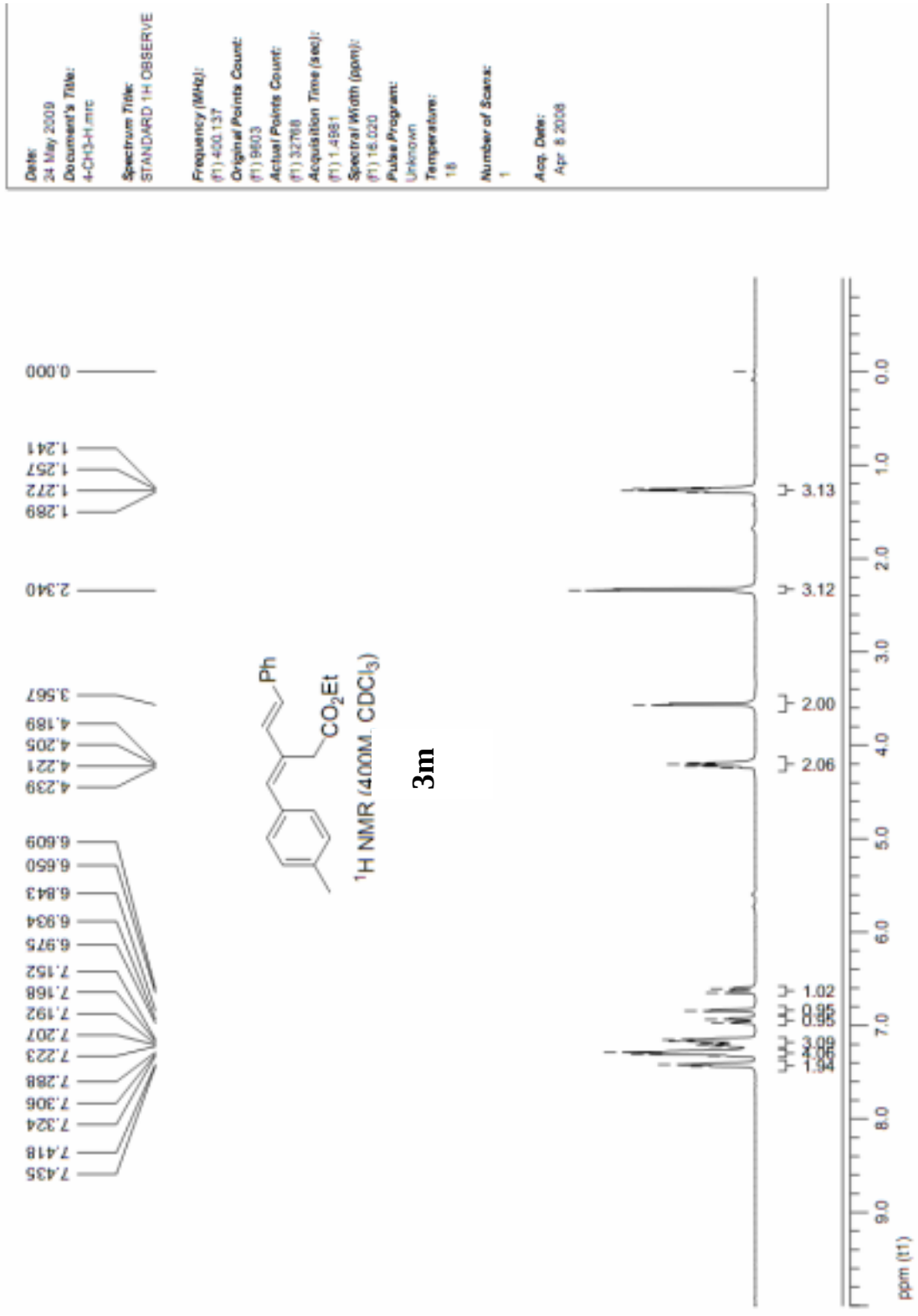
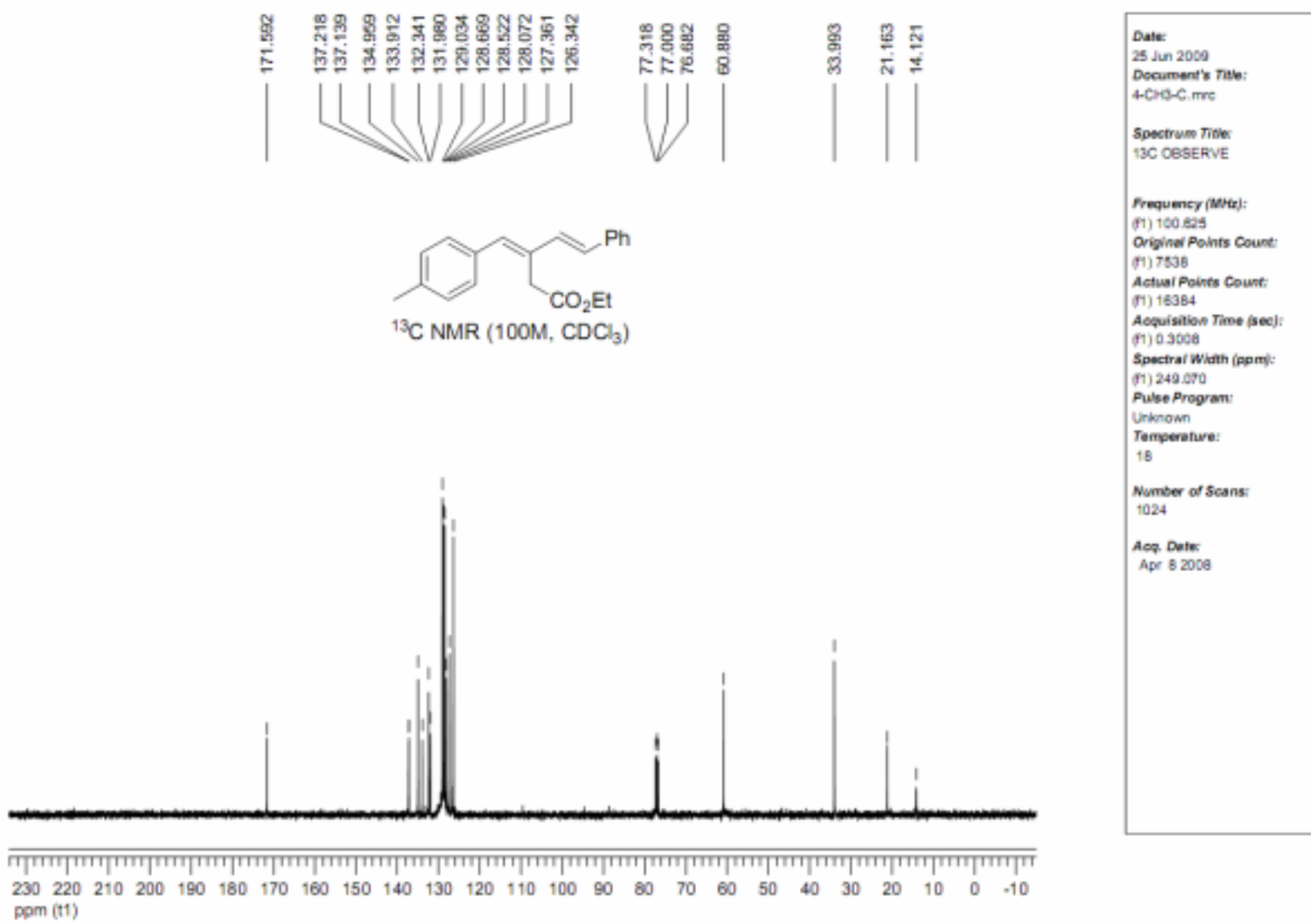
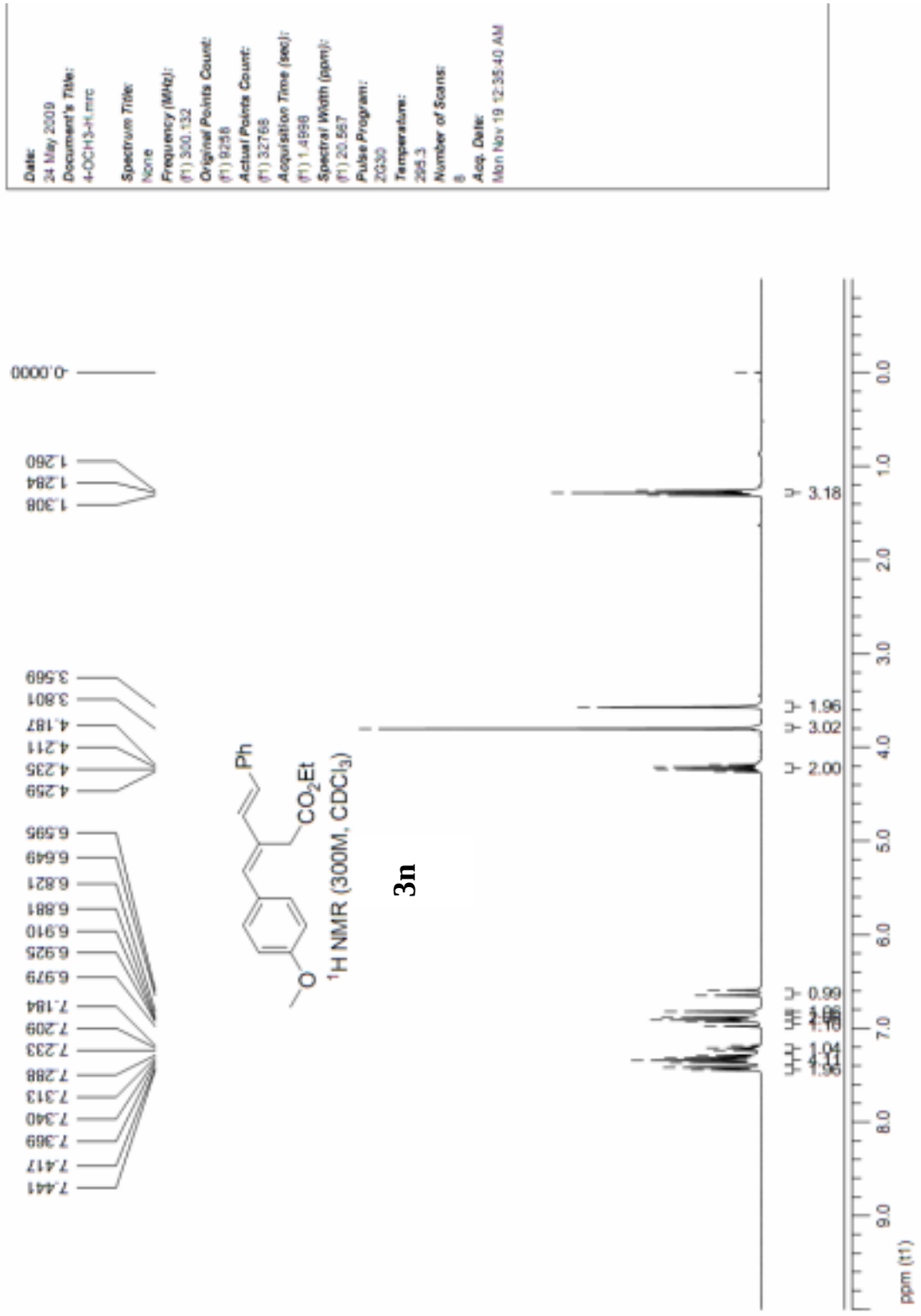
Original Points Count:
(f1) 9256
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4898
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2d30
Temperature:
298.5
Number of Scans:
8
Acq. Date:
Wed Dec 05 10:04:31 AM
0.000
1.174
1.198
1.221
3.485
4.102
4.126
4.150
4.173
6.547
6.601
6.800
6.858
6.912
7.115
7.139
7.163
7.181
7.212
7.239
7.265
7.274
7.298
7.308
7.344
7.369
Ph
CO2Et
Ph
1H NMR (300M, CDCl3)
3l
3.01
2.00
2.01
1.02
0.99
1.04
10.2
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)


Date:
14 Apr 2009
Document's Title:
1r
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
14.198
34.075
60.932
76.577
77.000
77.424
126.388
126.444
127.316
127.501
128.294
128.348
128.402
128.492
128.583
128.722
128.776
132.246
132.699
134.876
136.885
137.232
171.506
Ph
CO2Et
Ph
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)


Date:
24 May 2009
Document's Title:
4-CH3-H.mnr
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(F1) 400.137
Original Points Count:
(F1) 8803
Actual Points Count:
(F1) 32768
Acquisition Time (sec):
(F1) 1.4981
Spectral Width (ppm):
(F1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1
Acq. Date:
Apr 8 2008
0.000
1.241
1.257
1.272
1.289
2.340
3.567
4.189
4.205
4.221
4.239
6.809
6.650
6.843
6.934
6.975
7.152
7.168
7.192
7.207
7.223
7.288
7.306
7.324
7.418
7.435
Ph
CO2Et
1H NMR (400M, CDCl3)
3m
3.13
3.12
2.00
2.06
1.02
0.95
0.92
0.90
3.09
4.06
1.94
ppm (t1)


171.592
137.218
137.139
134.959
133.912
132.341
131.980
129.034
128.669
128.522
128.072
127.361
126.342
77.318
77.000
76.682
60.880
33.993
21.163
14.121
Ph
CO2Et
13C NMR (100M, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
25 Jun 2009
Document's Title:
4-CH3-C.mnr
Spectrum Title:
13C OBSERVE
Frequency (MHz):
(f1) 100.625
Original Points Count:
(f1) 7538
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 0.3008
Spectral Width (ppm):
(f1) 249.070
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1024
Acq. Date:
Apr 8 2008


Date:
24 May 2009
Document's Title:
4-OCH3-H.mnr
Spectrum Title:
None
Frequency (MHz):
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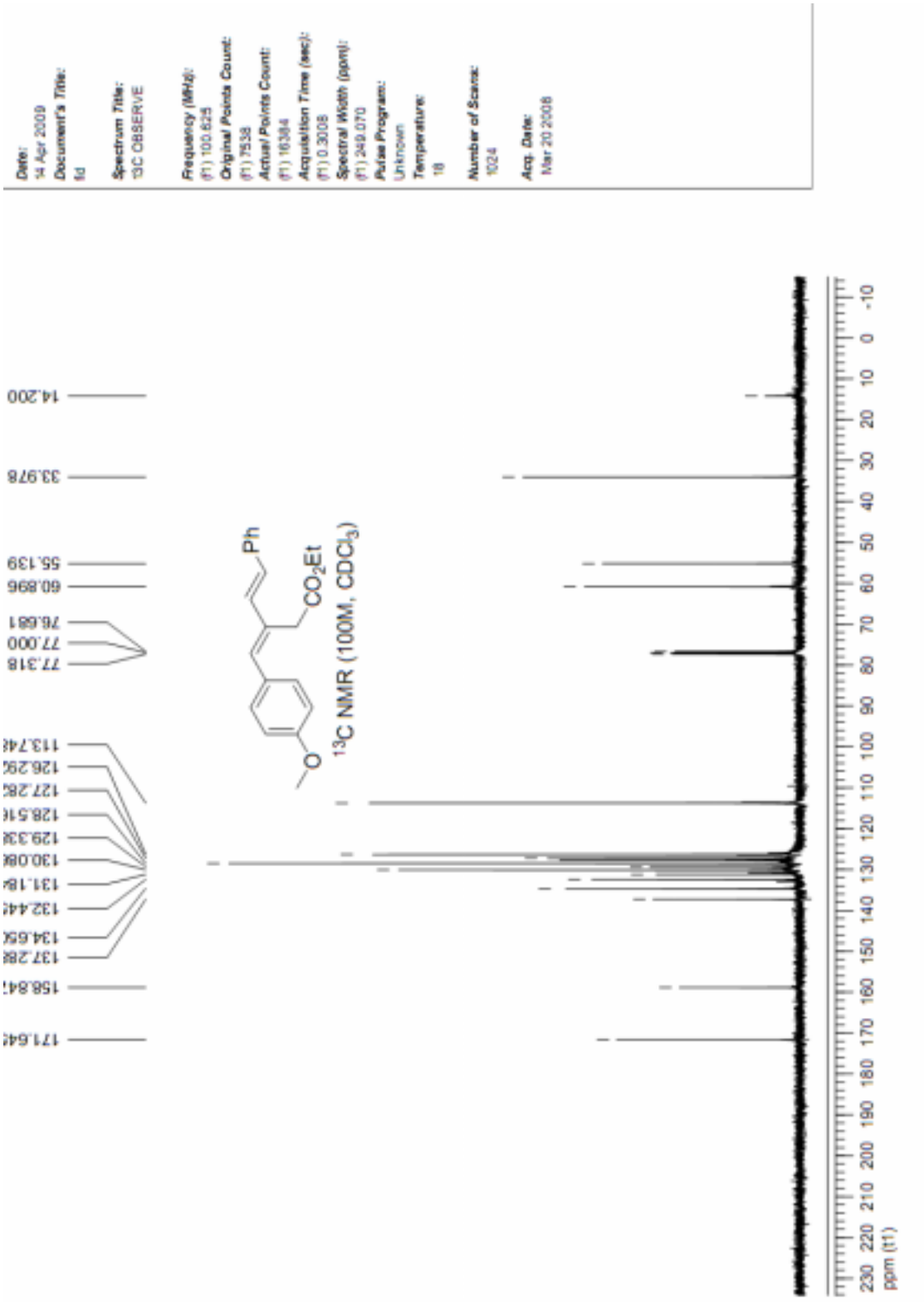
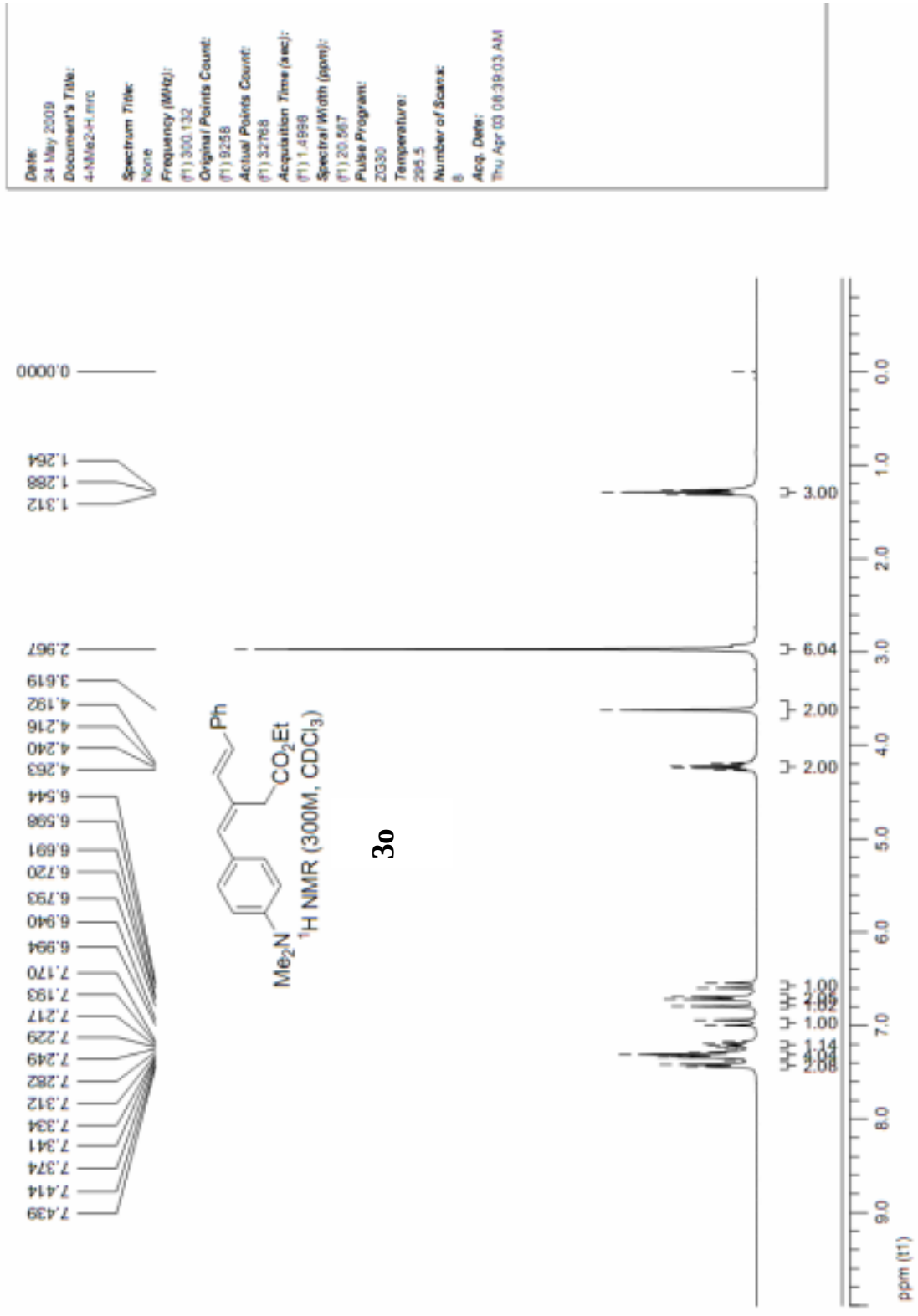
Original Points Count:
(f1) 8258
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4998
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
295.3
Number of Scans:
8
Acq. Date:
Mon Nov 19 12:35:40 AM
0.0000
1.260
1.284
1.308
3.569
3.801
4.187
4.211
4.235
4.259
6.595
6.649
6.821
6.881
6.910
6.925
6.979
7.184
7.209
7.233
7.288
7.313
7.340
7.369
7.417
7.441
Ph
CO2Et
O
1H NMR (300M, CDCl3)
3n
3.18
1.96
3.02
2.00
0.99
1.04
1.96
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)


Date:
14 Apr 2009
Document's Title:
fid
Spectrum Title:
13C OBSERVE
Frequency (MHz):
(f1) 100.625
Original Points Count:
(f1) 7533
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 0.3008
Spectral Width (ppm):
(f1) 249.070
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1024
Acq. Date:
Mar 20 2008
14.200
33.978
55.139
60.896
76.681
77.000
77.318
113.74
126.29
127.28
128.51
129.33
130.08
131.18
132.44
134.65
137.28
158.84
171.64
Ph
CO2Et
O
13C NMR (100M, CDCl3)
ppm (f1)


Date:
24 May 2009
Document's Title:
4-NMe2-H.mnr
Spectrum Title:
None
Frequency (MHz):
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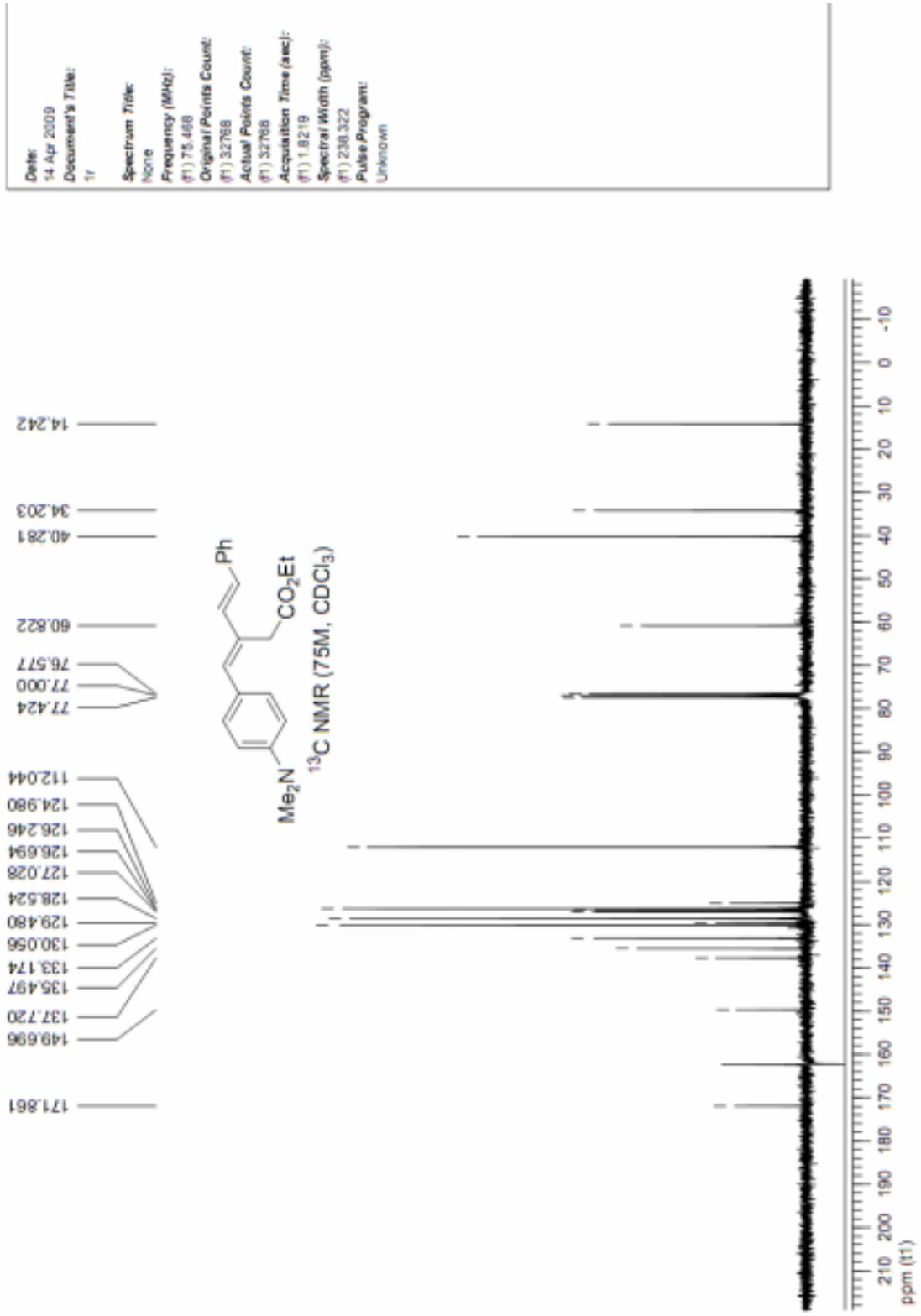
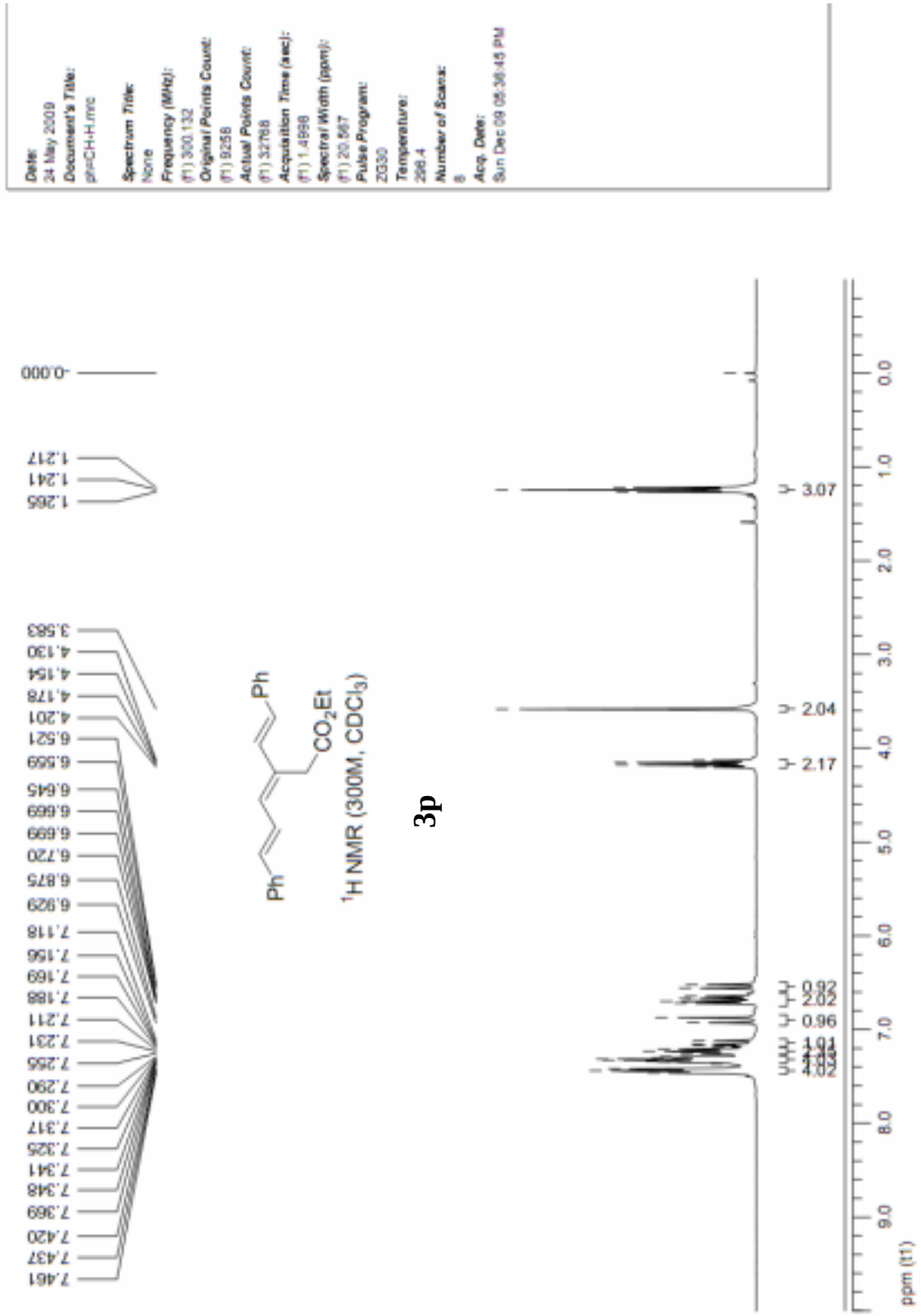
Original Points Count:
(f1) 9256
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4998
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
295.5
Number of Scans:
8
Acq. Date:
Thu Apr 03 08:39:03 AM
0.0000
1.264
1.288
1.312
2.967
3.619
4.192
4.216
4.240
4.263
6.544
6.598
6.691
6.720
6.793
6.940
6.994
7.170
7.193
7.217
7.229
7.249
7.282
7.312
7.334
7.341
7.374
7.414
7.439
3.00
6.04
2.00
2.00
1.00
1.00
1.14
2.00
Ph
CO2Et
Me2N
1H NMR (300M, CDCl3)
3o
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)


Date:
14 Apr 2009
Document's Title:
1r
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
14.242
34.203
40.281
60.822
76.577
77.000
77.424
112.044
124.980
126.246
126.694
127.028
128.524
129.480
130.056
133.174
135.497
137.720
149.696
171.861
Ph
CO2Et
Me2N
13C NMR (75M, CDCl3)
ppm (t1)


Date:
24 May 2009
Document's Title:
phsCH-H.mnr
Spectrum Title:
None
Frequency (MHz):
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Original Points Count:
(f1) 9256
Actual Points Count:
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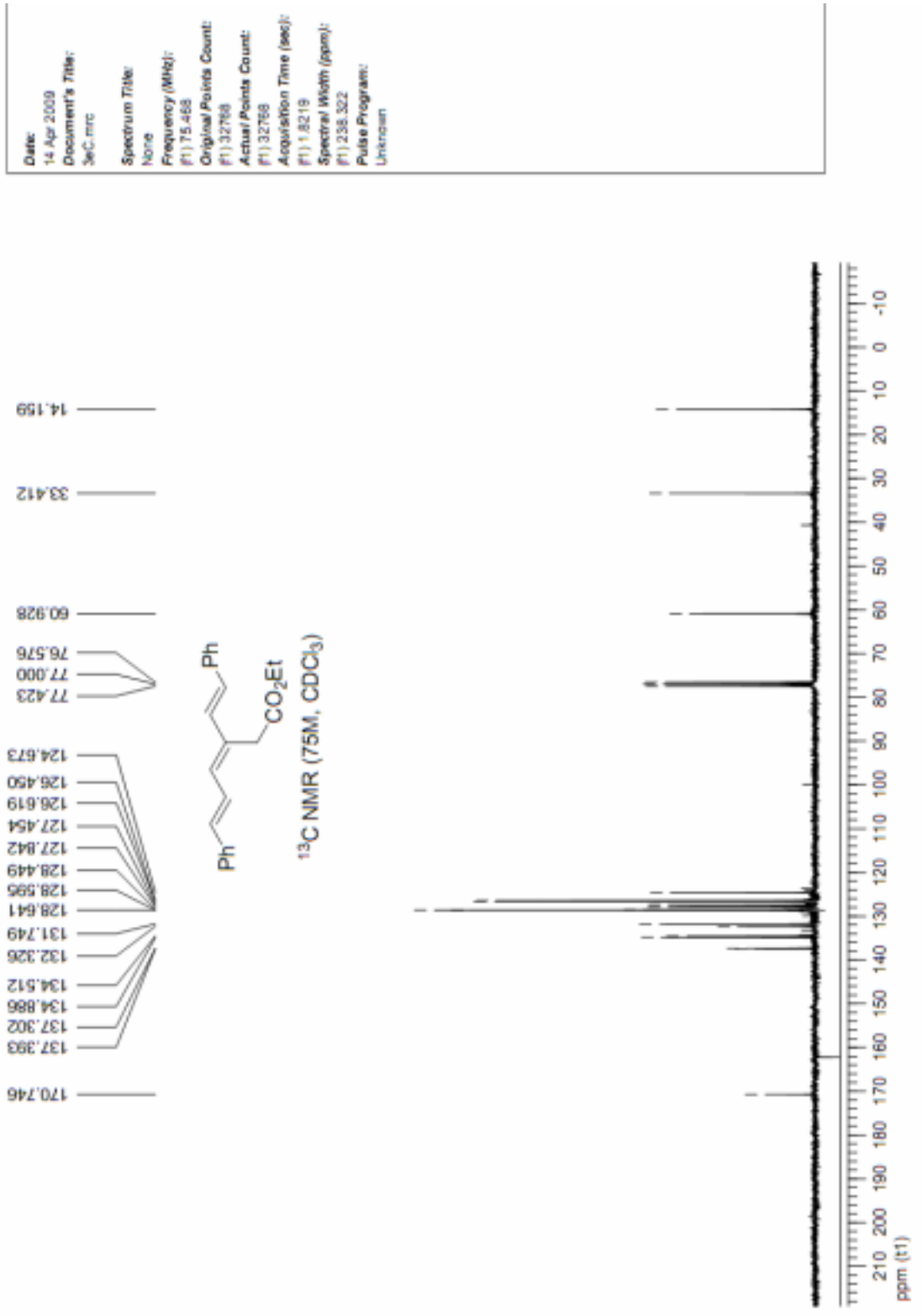
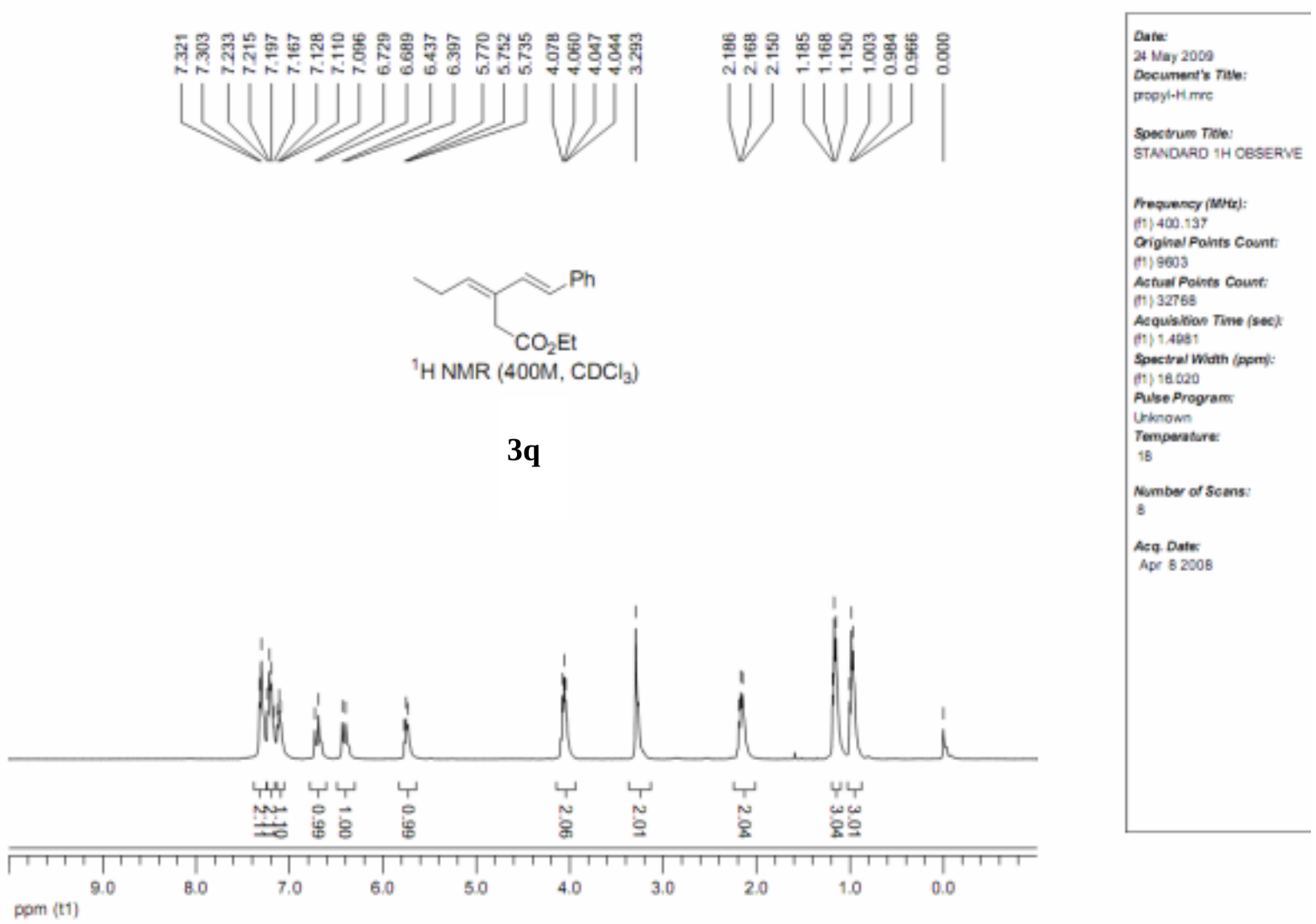
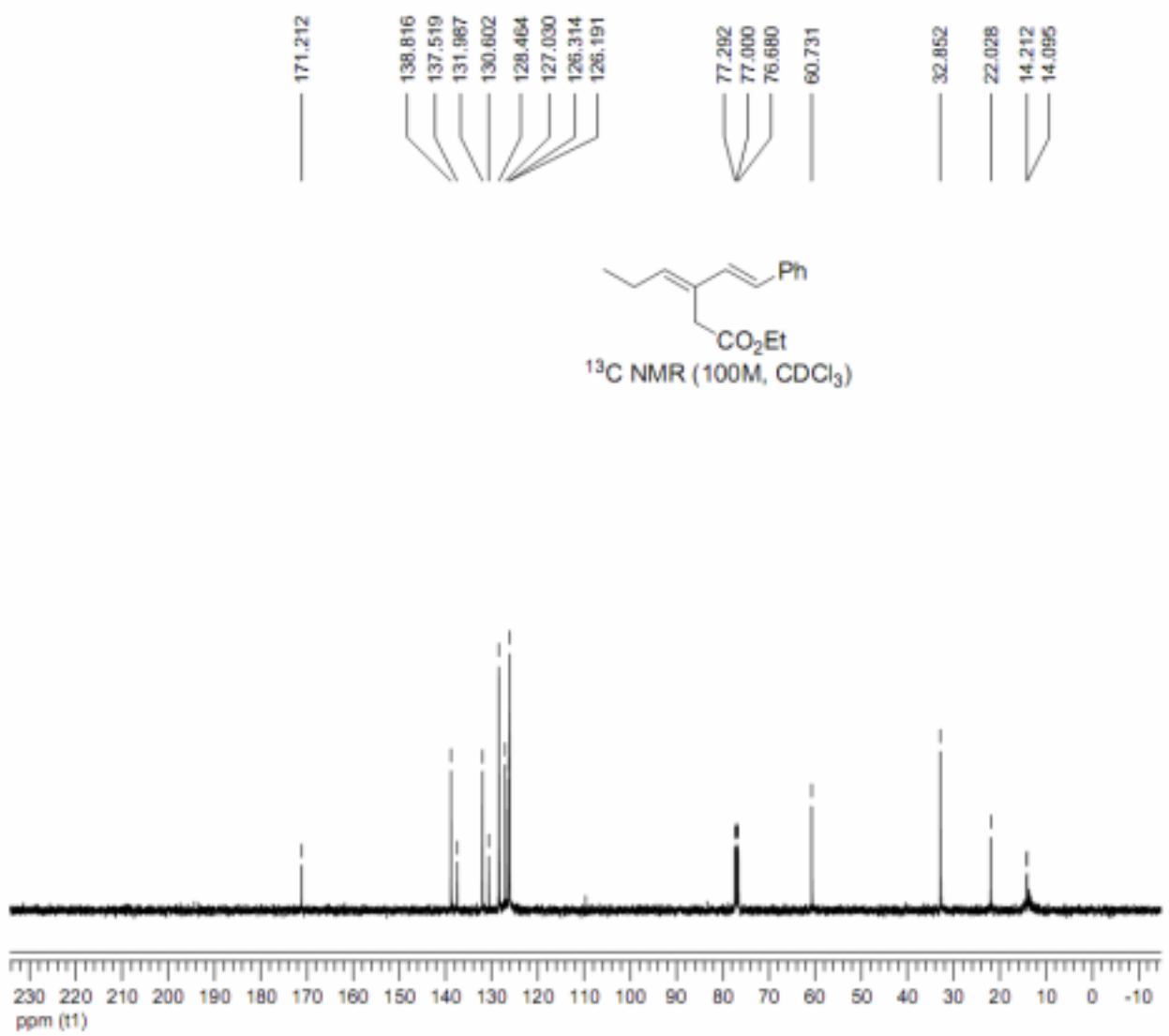
Acquisition Time (sec):
(f1) 1.4998
Spectral Width (ppm):
(f1) 20.567
Pulse Program:
2D30
Temperature:
298.4
Number of Scans:
8
Acq. Date:
Sun Dec 09 05:36:45 PM
-0.000
1.217
1.241
1.265
3.583
4.130
4.154
4.178
4.201
6.521
6.559
6.645
6.669
6.699
6.720
6.875
6.929
7.118
7.156
7.169
7.188
7.211
7.231
7.255
7.290
7.300
7.317
7.325
7.341
7.348
7.369
7.420
7.437
7.461
Ph
CO2Et
Ph
1H NMR (300M, CDCl3)
3p
3.07
2.04
2.17
0.92
2.02
0.96
1.91
4.02
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)


Date:
14 Apr 2009
Document's Title:
3eC.mnr
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown
14.159
33.412
60.928
76.576
77.000
77.423
124.673
126.450
126.619
127.454
127.842
128.449
128.595
128.641
131.749
132.326
134.512
134.886
137.302
137.393
170.746
Ph
CO2Et
Ph
13C NMR (75M, CDCl3)
ppm (t1)


7.321
7.303
7.233
7.215
7.197
7.167
7.128
7.110
7.096
6.729
6.689
6.437
6.397
5.770
5.752
5.735
4.078
4.060
4.047
4.044
3.293
2.186
2.168
2.150
1.185
1.168
1.150
1.003
0.984
0.966
0.000
Ph
CO2Et
1H NMR (400M, CDCl3)
3q
2.11
2.10
0.99
1.00
0.99
2.06
2.01
2.04
3.04
3.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
Date:
24 May 2009
Document's Title:
propyl-H.mrc
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 9603
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
8
Acq. Date:
Apr 8 2008



Date:
25 Jun 2009
Document's Title:
propyl-C.mrc

Spectrum Title:
13C OBSERVE

Frequency (MHz):
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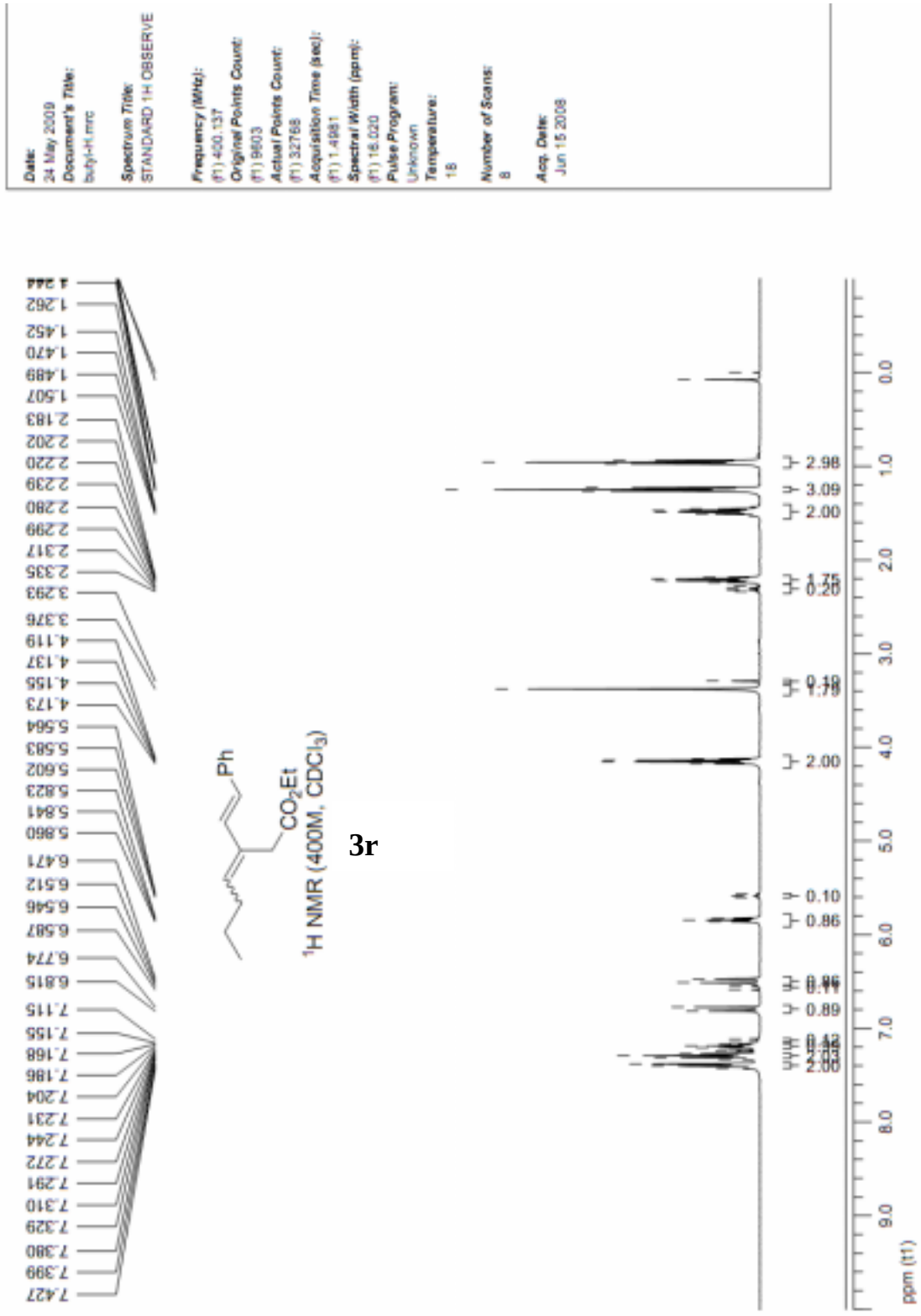
Original Points Count:
(f1) 7538
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 0.3008
Spectral Width (ppm):
(f1) 249.070
Pulse Program:
Unknown
Temperature:
18

Number of Scans:
1024

Acq. Date:
Apr 8 2008


Date:
24 May 2009
Document's Title:
butyl-H1.mnr
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
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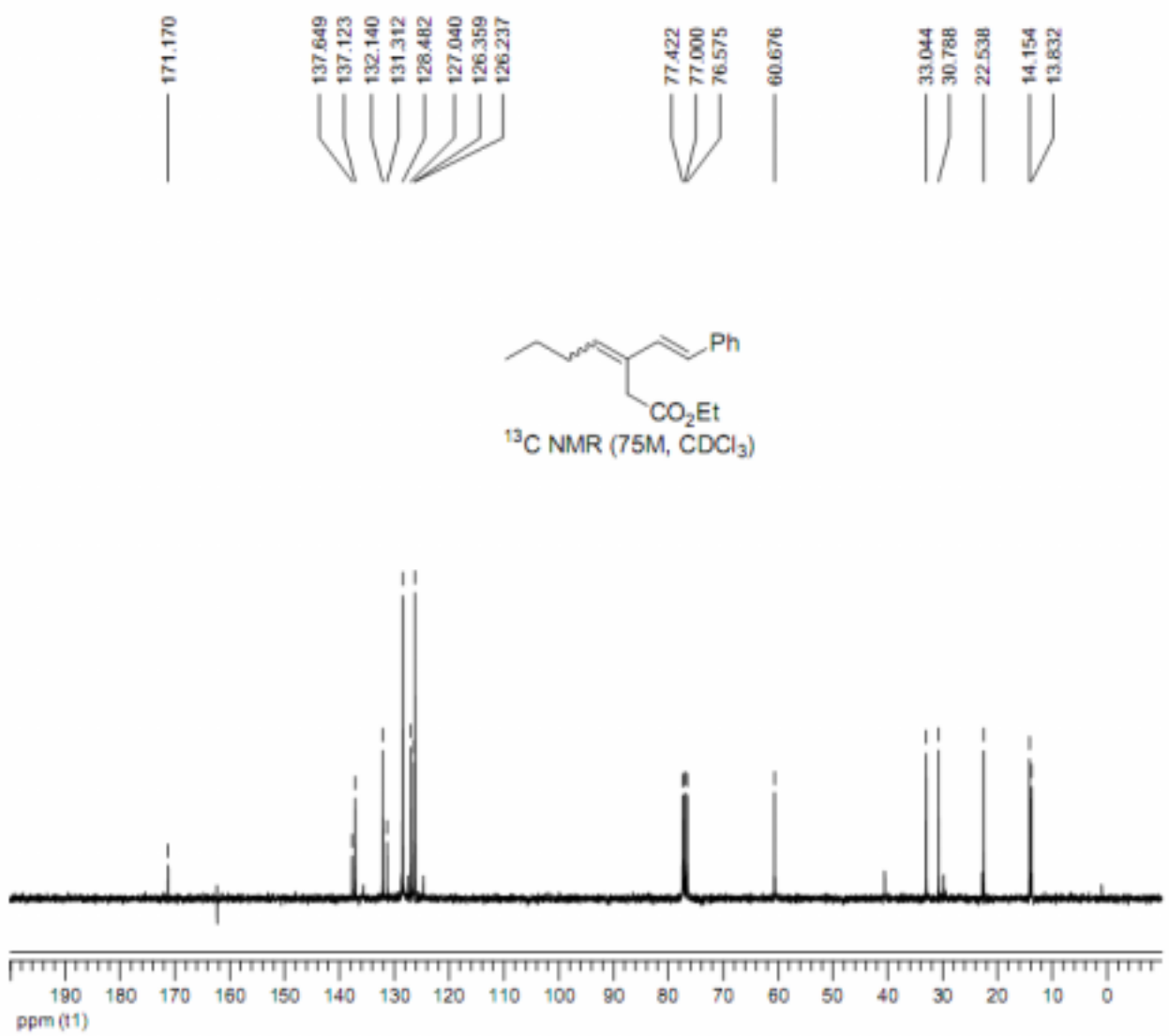
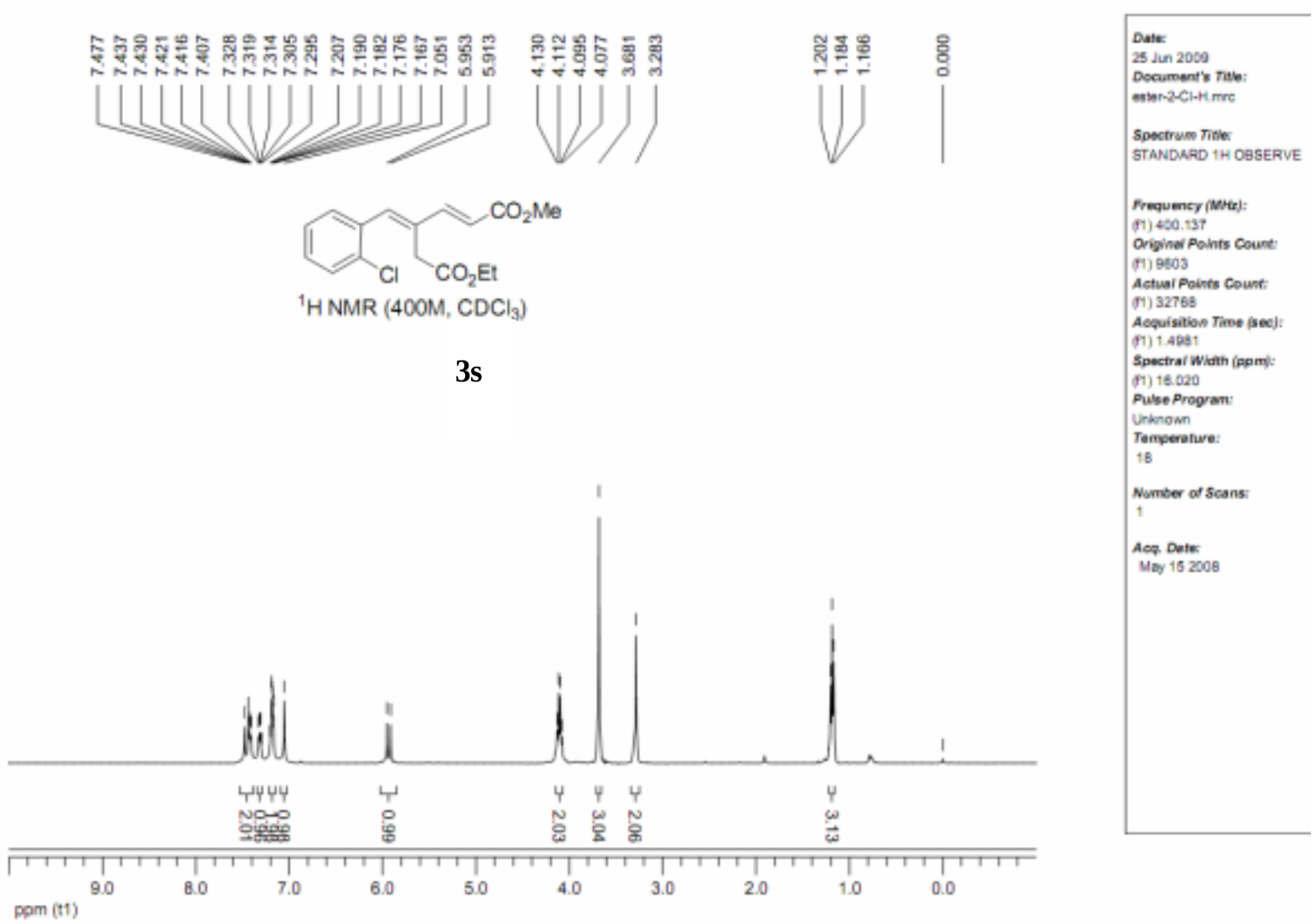
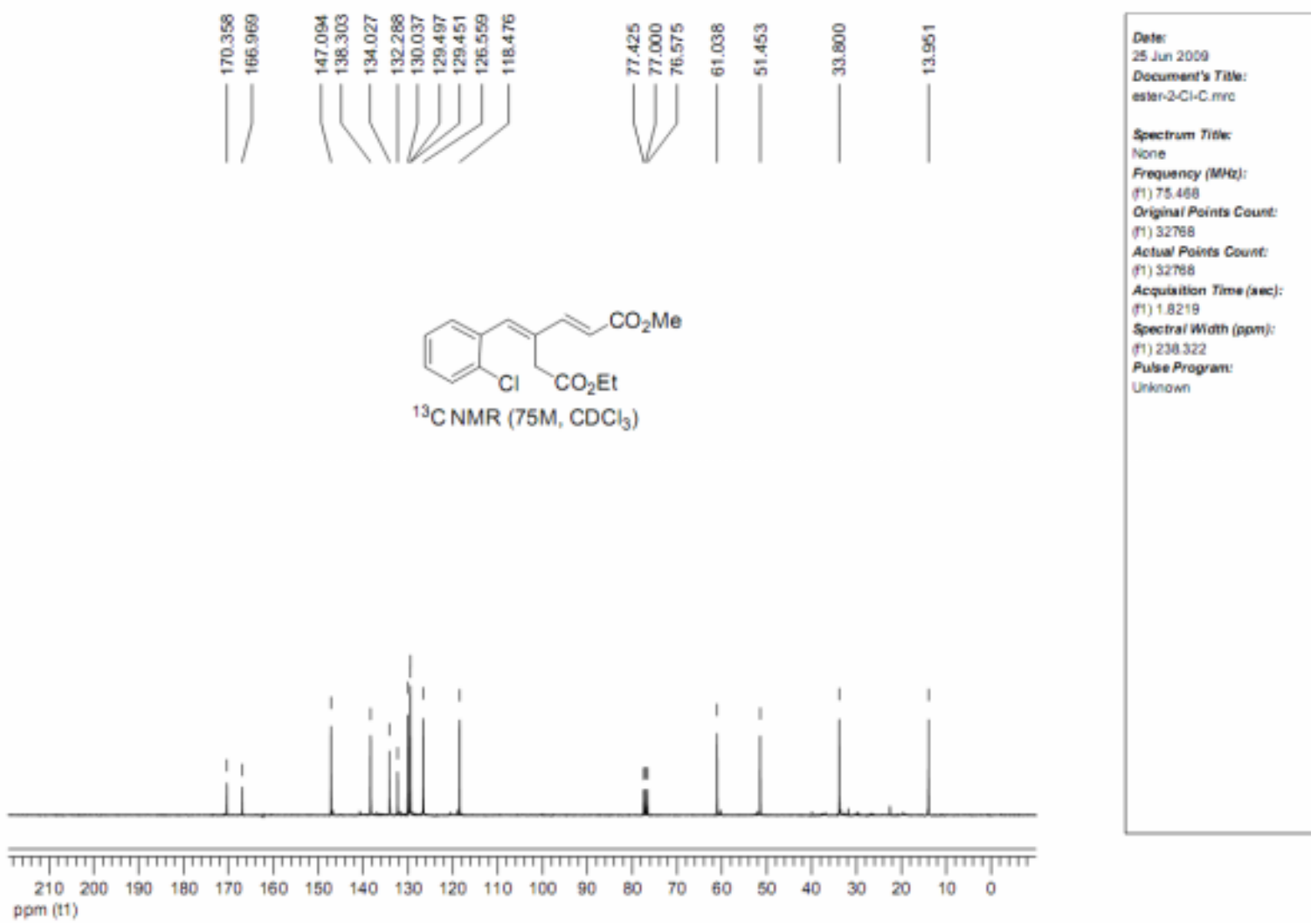
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
8
Acq. Date:
Jun 15 2008
Ph
CO2Et
1H NMR (400M, CDCl3)
3r
2.98
3.09
2.00
1.75
0.20
0.19
1.79
2.00
0.10
0.86
0.89
2.00
ppm (t1)



Date:
25 Jun 2009
Document's Title:
butyl-C.mrc

Spectrum Title:
None
Frequency (MHz):
(f1) 75.488
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown


7.477
7.437
7.430
7.421
7.416
7.407
7.328
7.319
7.314
7.305
7.295
7.207
7.190
7.182
7.176
7.167
7.051
5.953
5.913
4.130
4.112
4.095
4.077
3.681
3.283
1.202
1.184
1.166
0.000
CO2Me
Cl
CO2Et
1H NMR (400M, CDCl3)
3s
2.01
0.99
2.03
3.04
2.96
3.13
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-2-Cl-H.mrc
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 9603
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1
Acq. Date:
May 15 2008


170.358
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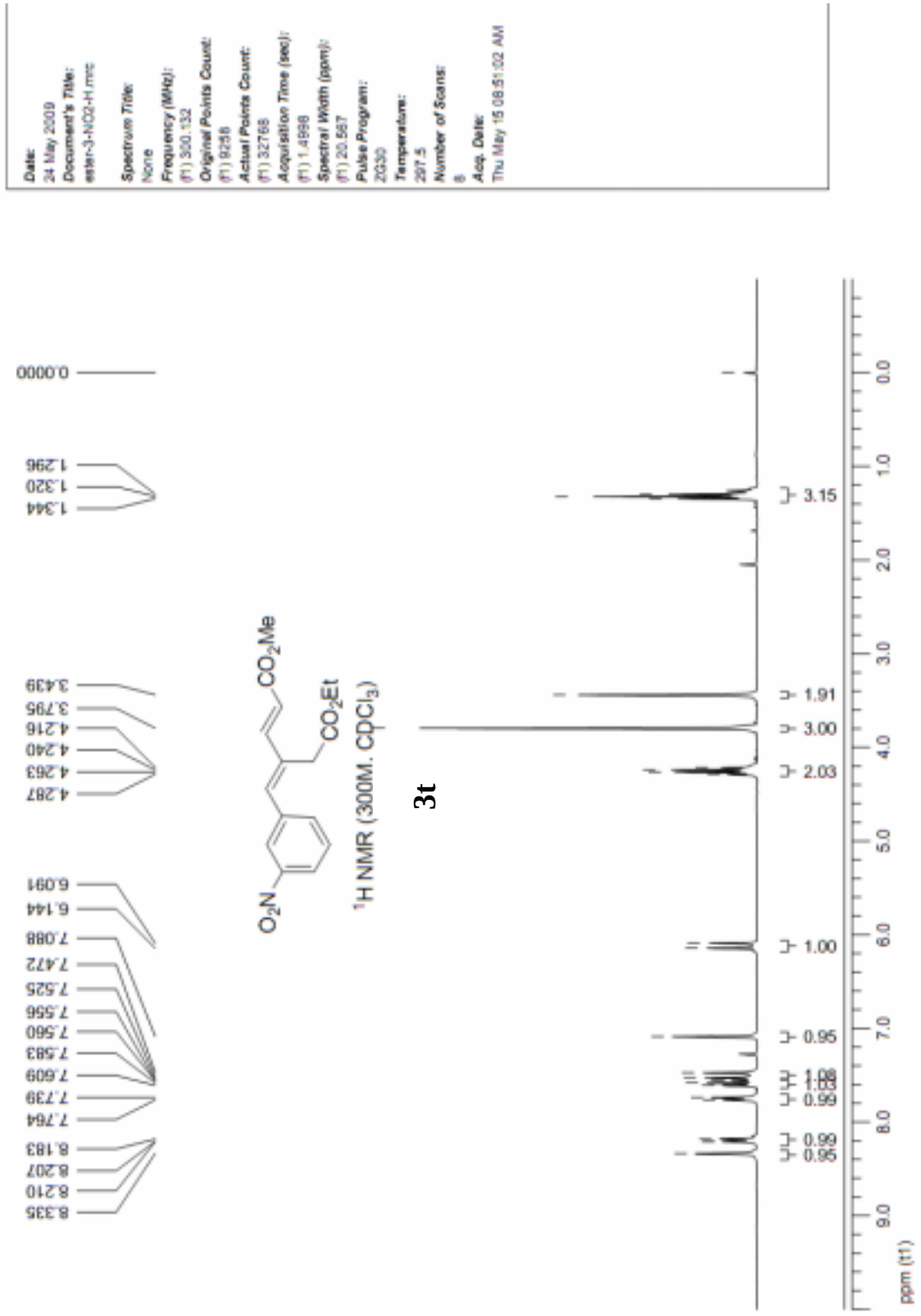
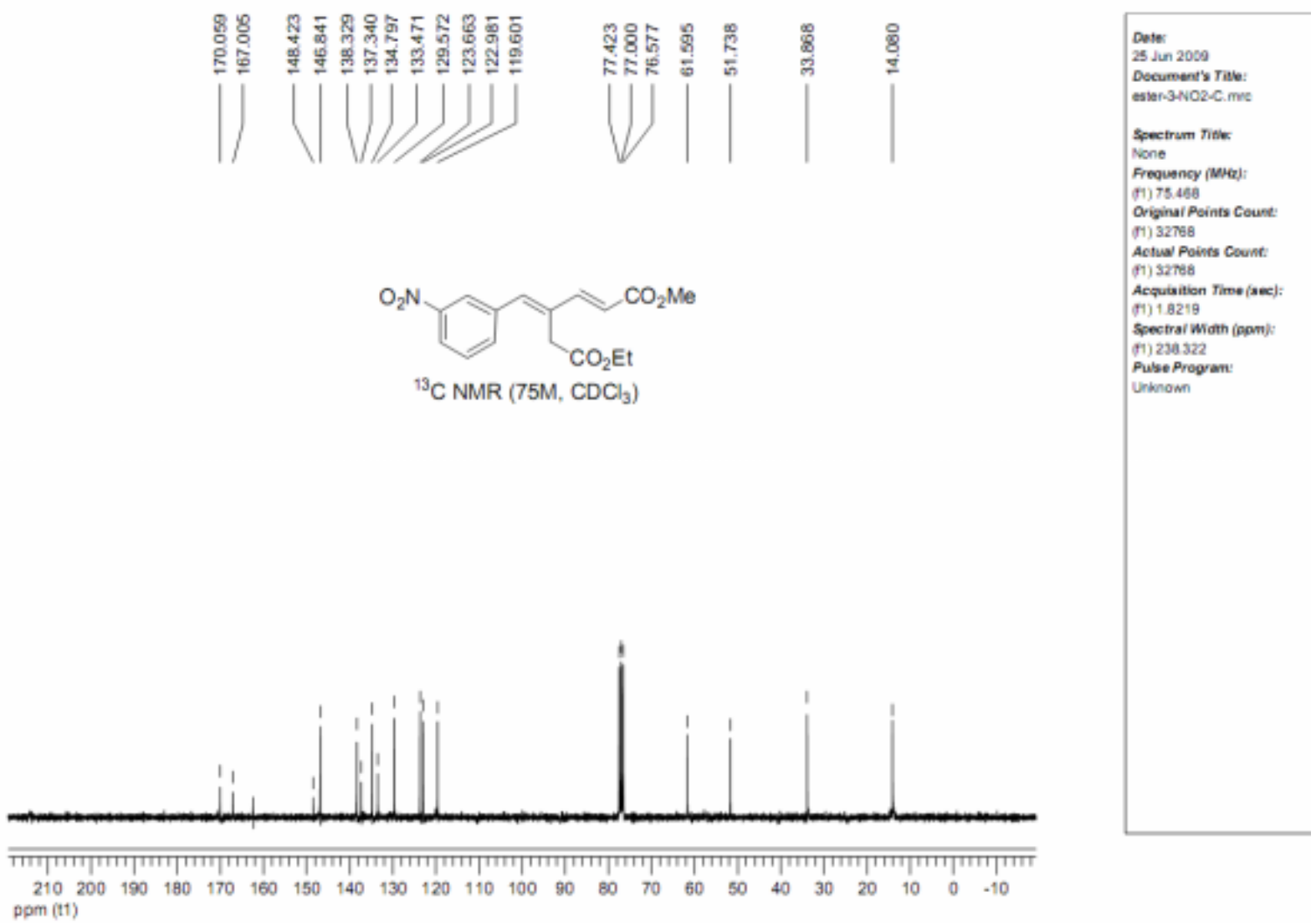
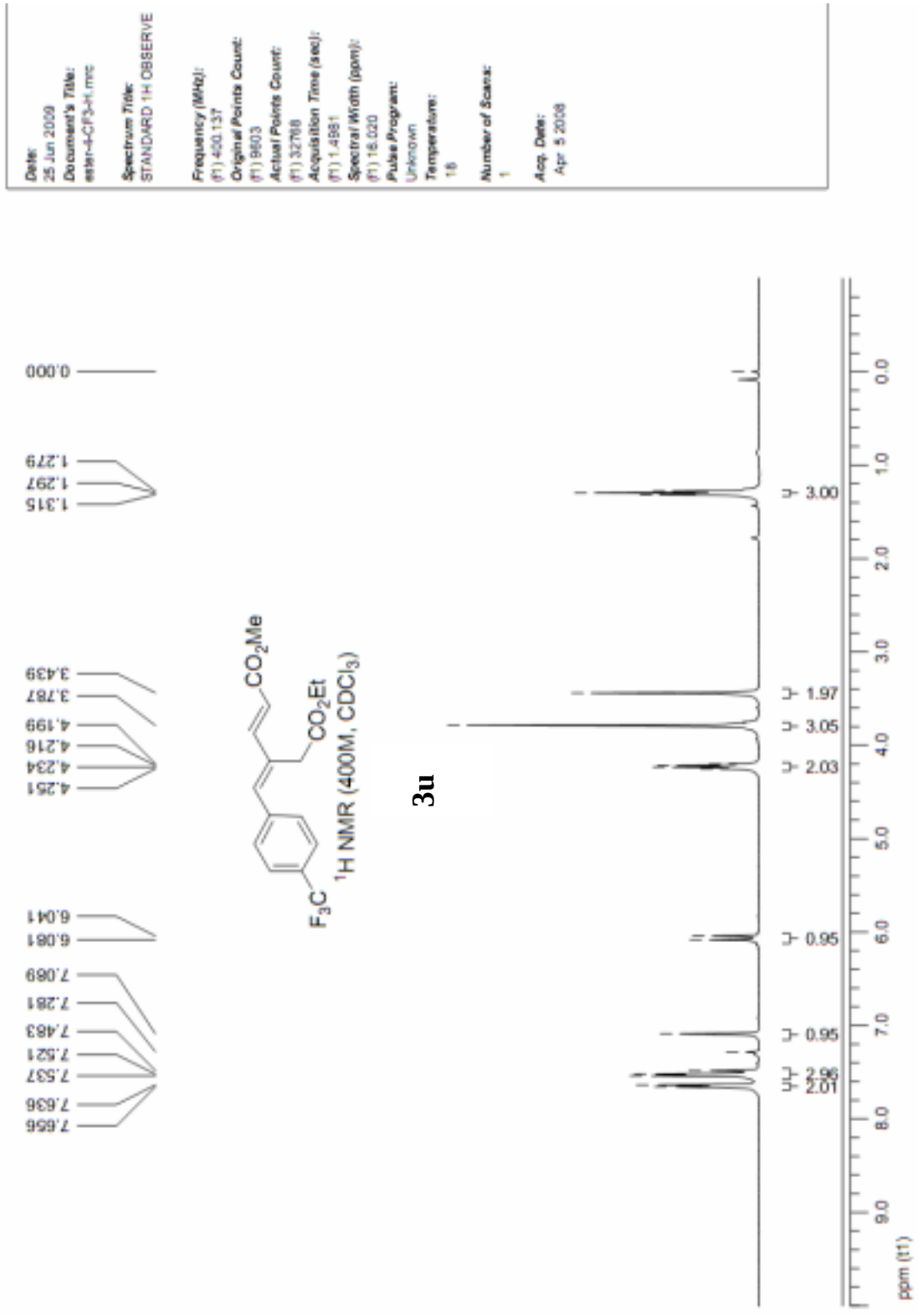
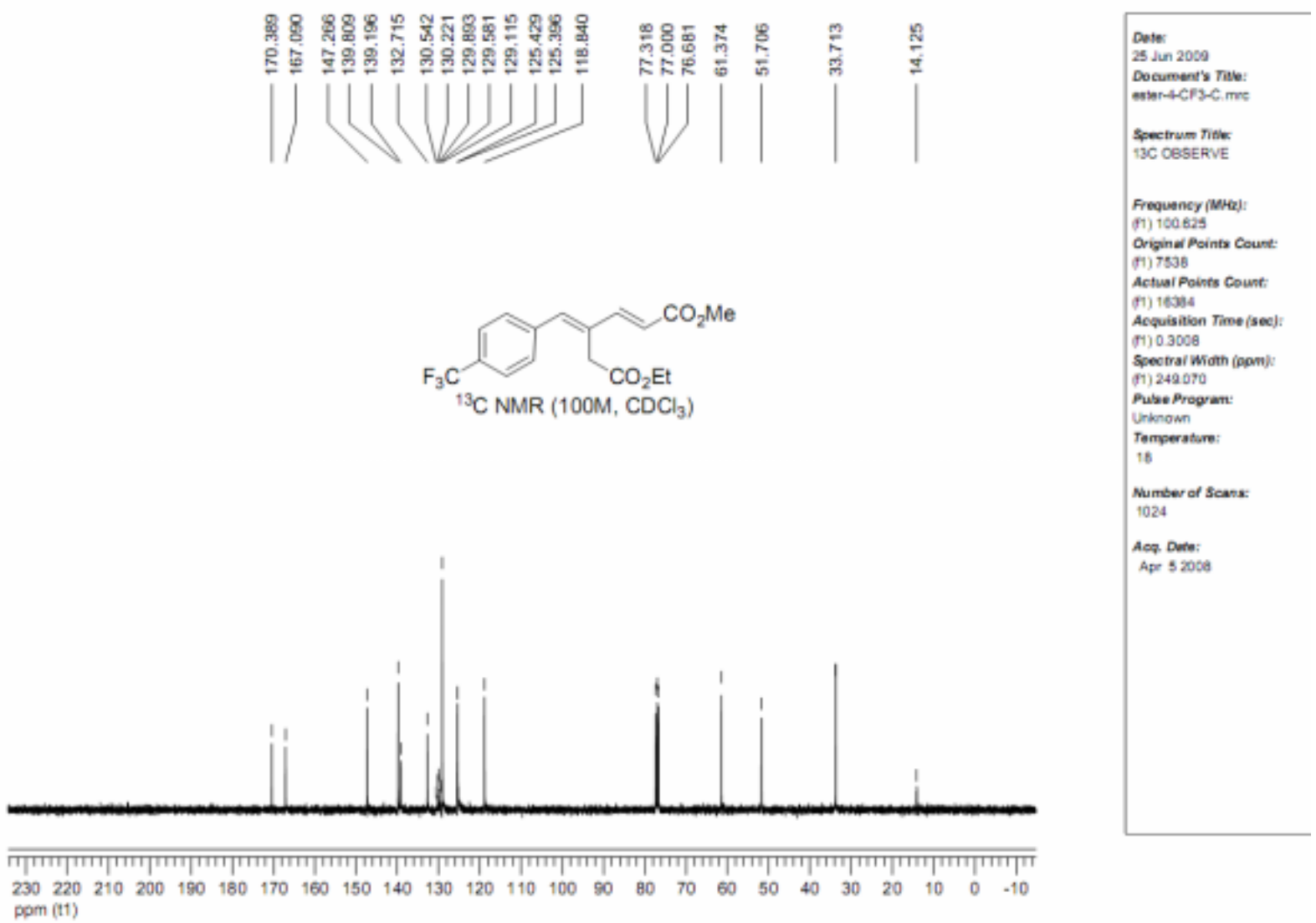
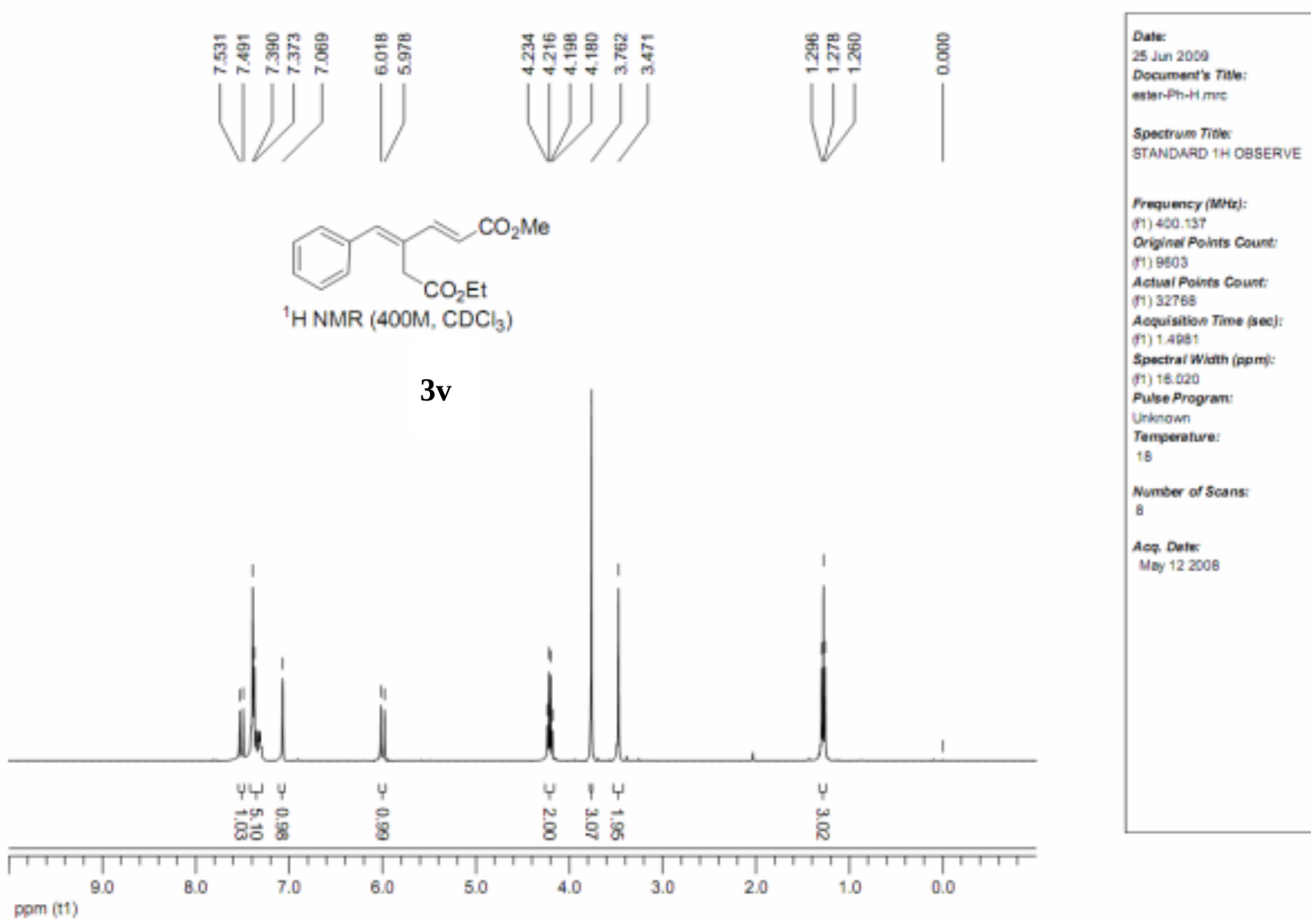
147.094
138.303
134.027
132.288
130.037
129.497
129.451
126.559
118.476
77.425
77.000
76.575
61.038
51.453
33.800
13.951
CO2Me
Cl
CO2Et
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-2-Cl-C.mrc
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown

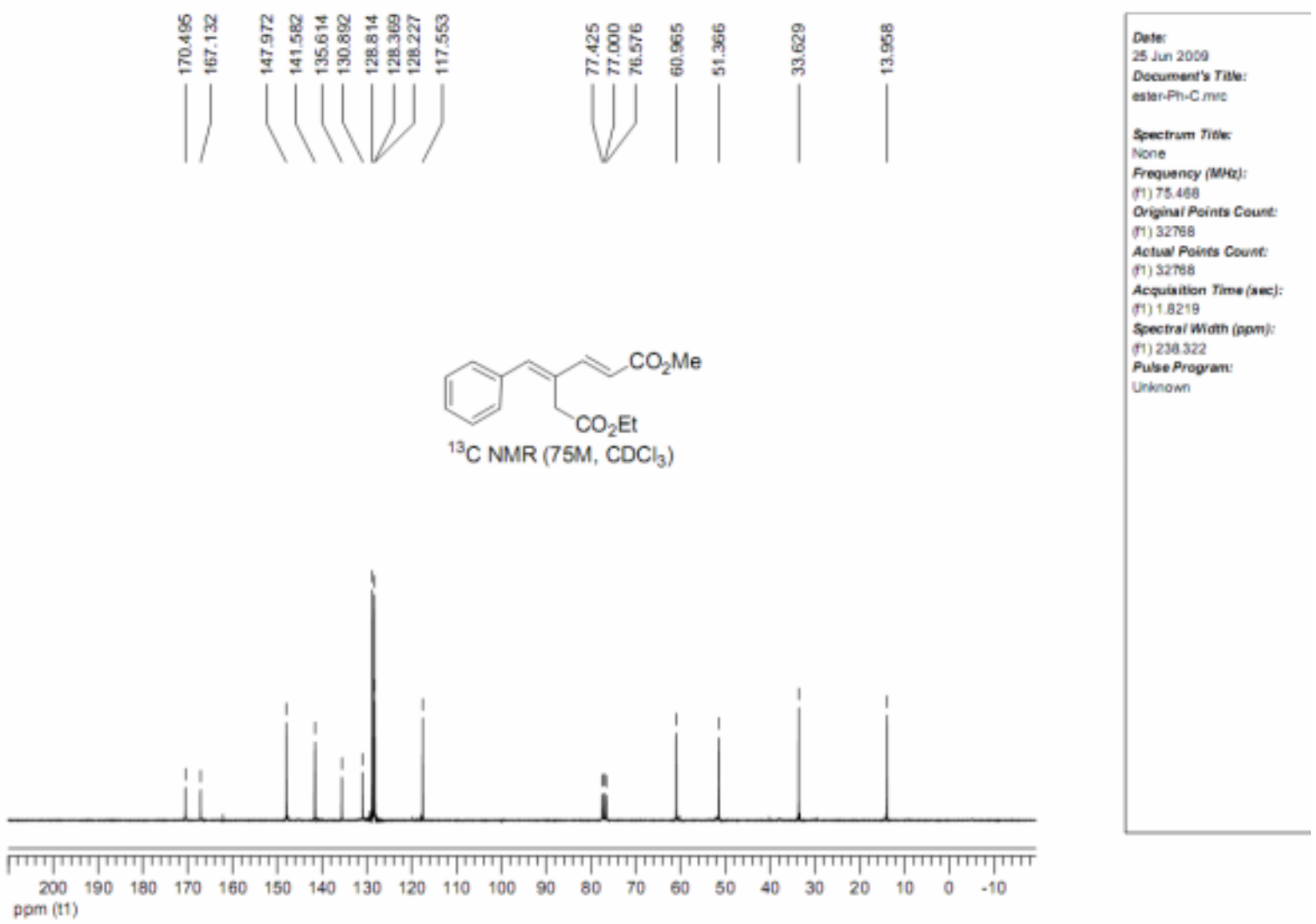

Date:
24 May 2009
Document's Title:
ester-3-NO2-H.mns
Spectrum Title:
None
Frequency (MHz):
(f1) 300.132
Original Points Count:
(f1) 9256
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4998
Spectral Width (ppm):
(f1) 20.587
Pulse Program:
2D30
Temperature:
297.5
Number of Scans:
8
Acq. Date:
Thu May 15 08:51:02 AM
0.0000
1.296
1.320
1.344
3.439
3.795
4.216
4.240
4.263
4.287
6.091
6.144
7.088
7.472
7.525
7.556
7.560
7.583
7.609
7.739
7.764
8.183
8.207
8.210
8.335
CO2Me
CO2Et
O2N
1H NMR (300M. CDCl3)
3t
3.15
1.91
3.00
2.03
1.00
0.95
1.08
1.03
0.99
0.99
0.95
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)

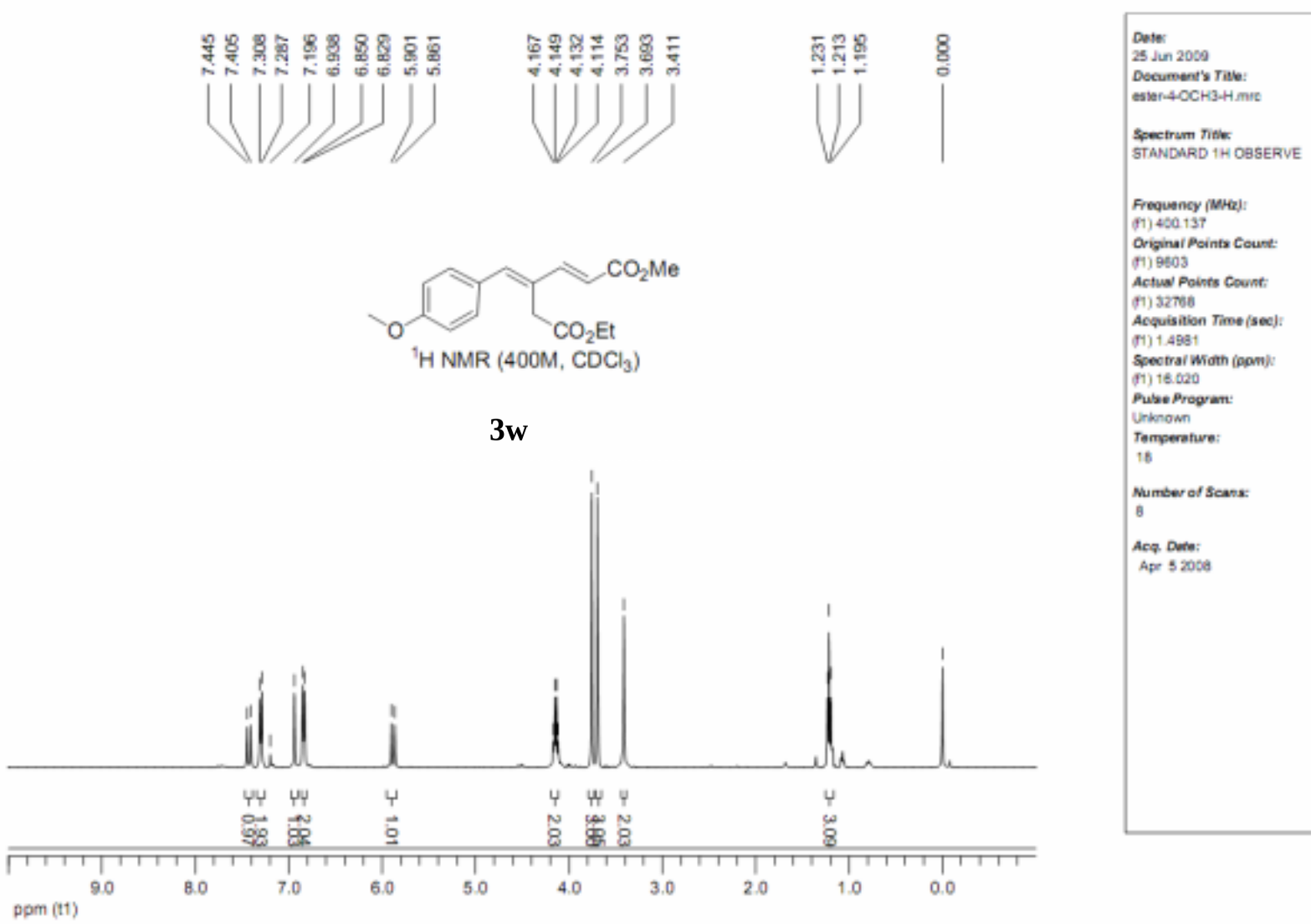

170.059
167.005
148.423
146.841
138.329
137.340
134.797
133.471
129.572
123.663
122.981
119.601
77.423
77.000
76.577
61.595
51.738
33.868
14.080
O2N
CO2Me
CO2Et
13C NMR (75M, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-3-NO2-C.mnr
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown

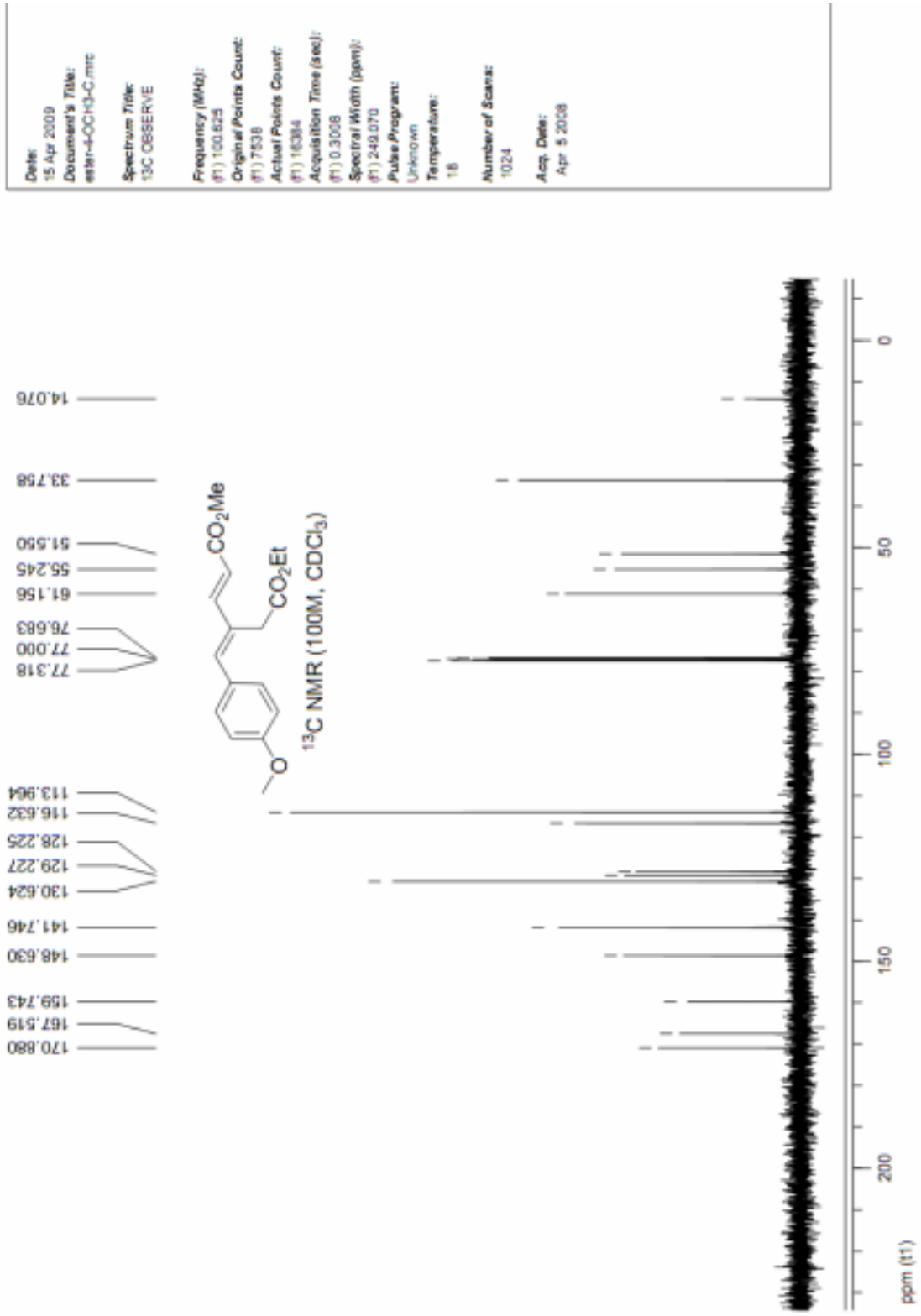

Date:
25 Jun 2020
Document's Title:
ester-4-CF3-H.mnr
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 8803
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1
Acq. Date:
Apr 5 2008
0.000
1.279
1.297
1.315
3.439
3.787
4.199
4.216
4.234
4.251
6.041
6.081
7.089
7.281
7.483
7.521
7.537
7.636
7.656
CO2Me
CO2Et
F3C
1H NMR (400M, CDCl3)
3u
3.00
1.97
3.05
2.03
0.95
0.95
2.96
2.01
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
ppm (t1)

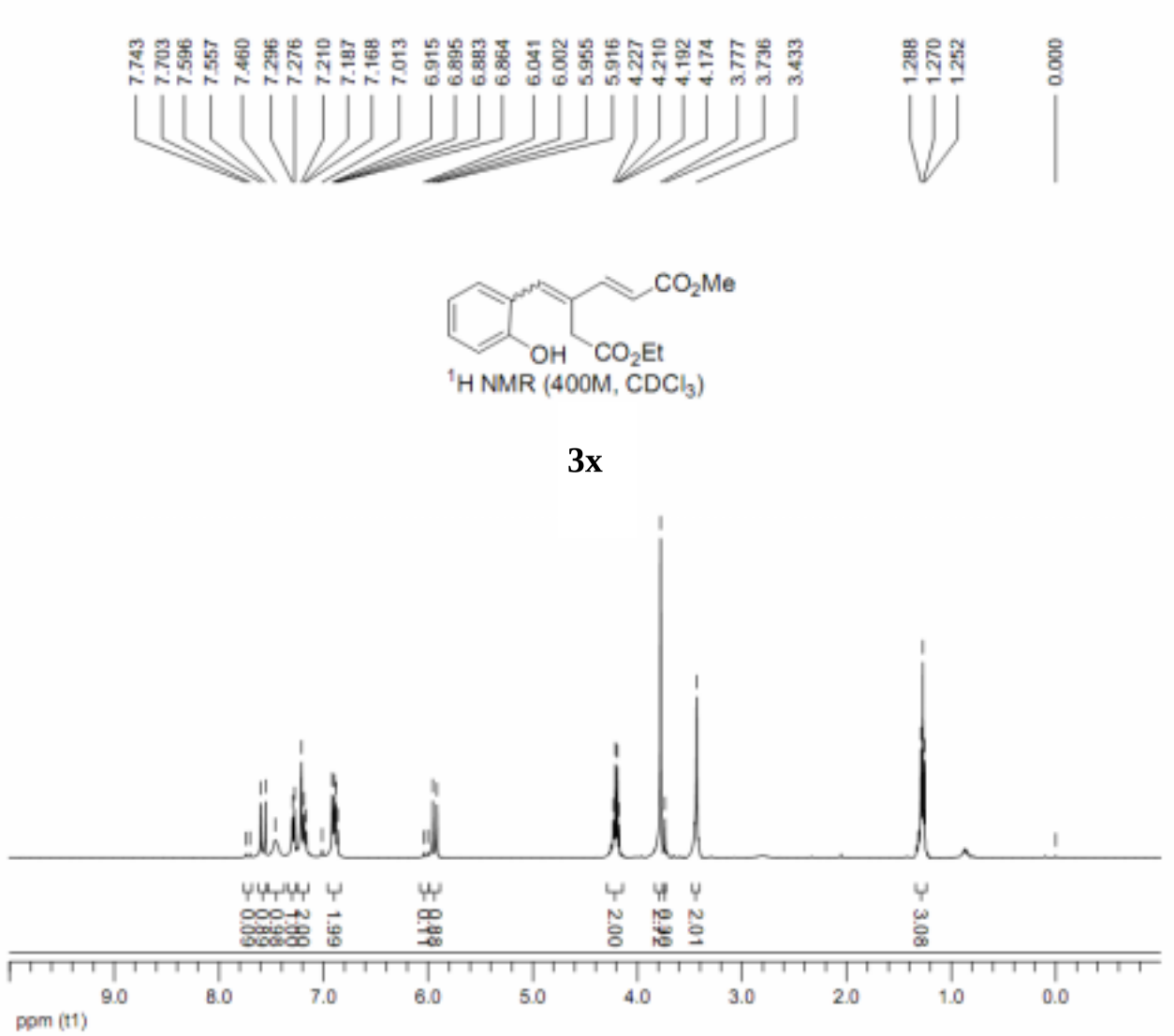

170.389
167.090
147.266
139.809
139.196
132.715
130.542
130.221
129.893
129.581
129.115
125.429
125.396
118.840
77.318
77.000
76.681
61.374
51.706
33.713
14.125
CO2Me
F3C
CO2Et
13C NMR (100M, CDCl3)
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-4-CF3-C.mnr
Spectrum Title:
13C OBSERVE
Frequency (MHz):
(f1) 100.625
Original Points Count:
(f1) 7538
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 0.3008
Spectral Width (ppm):
(f1) 249.070
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1024
Acq. Date:
Apr 5 2008


7.531
7.491
7.390
7.373
7.069
6.018
5.978
4.234
4.216
4.198
4.180
3.762
3.471
1.296
1.278
1.260
0.000
CO2Me
CO2Et
1H NMR (400M, CDCl3)
3v
1.03
5.10
0.98
0.99
2.00
3.07
1.96
3.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-Ph-H.mnr
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 9603
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
8
Acq. Date:
May 12 2008


170.495
167.132
147.972
141.582
135.614
130.892
128.814
128.369
128.227
117.553
77.425
77.000
76.576
60.965
51.366
33.629
13.958
CO2Me
CO2Et
13C NMR (75M, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-Ph-C.mnr
Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown


7.445
7.405
7.308
7.287
7.196
6.908
6.850
6.829
5.901
5.861
4.167
4.149
4.132
4.114
3.753
3.693
3.411
1.231
1.213
1.195
0.000
CO2Me
O
CO2Et
1H NMR (400M, CDCl3)
3w
0.97
1.83
1.03
2.04
1.01
2.03
3.00
2.03
3.09
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
Date:
25 Jun 2009
Document's Title:
ester-4-OCH3-H.mnr
Spectrum Title:
STANDARD 1H OBSERVE
Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 9603
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
8
Acq. Date:
Apr 5 2008


Date:
15 Apr 2009
Document's Title:
ester-4-OCH3-C.mnr
Spectrum Title:
13C OBSERVE
Frequency (MHz):
(f1) 100.625
Original Points Count:
(f1) 7536
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 0.3008
Spectral Width (ppm):
(f1) 249.070
Pulse Program:
Unknown
Temperature:
18
Number of Scans:
1024
Acq. Date:
Apr 5 2008
CO_2Me
CO_2Et
O
^{13}C NMR (100M, $CDCl_3$)
14.076
33.758
51.550
55.245
61.156
76.683
77.000
77.318
113.964
116.632
128.225
129.227
130.624
141.746
148.630
159.743
167.519
170.880
0
50
100
150
200
ppm (t1)



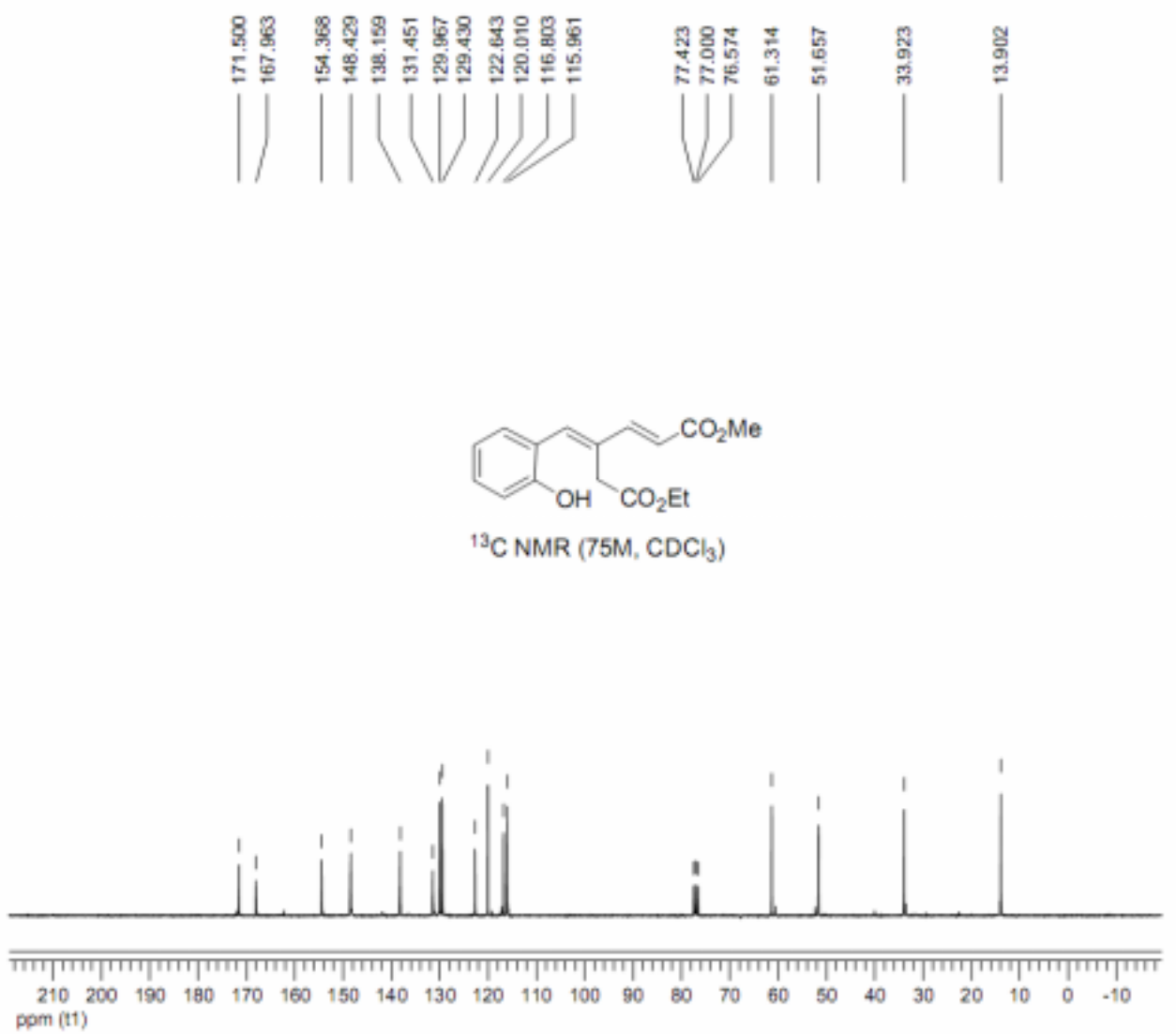
Date:
25 Jun 2009
Document's Title:
ester-2-OH-H.mnr

Spectrum Title:
STANDARD 1H OBSERVE

Frequency (MHz):
(f1) 400.137
Original Points Count:
(f1) 9603
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.4981
Spectral Width (ppm):
(f1) 16.020
Pulse Program:
Unknown
Temperature:
18

Number of Scans:
1

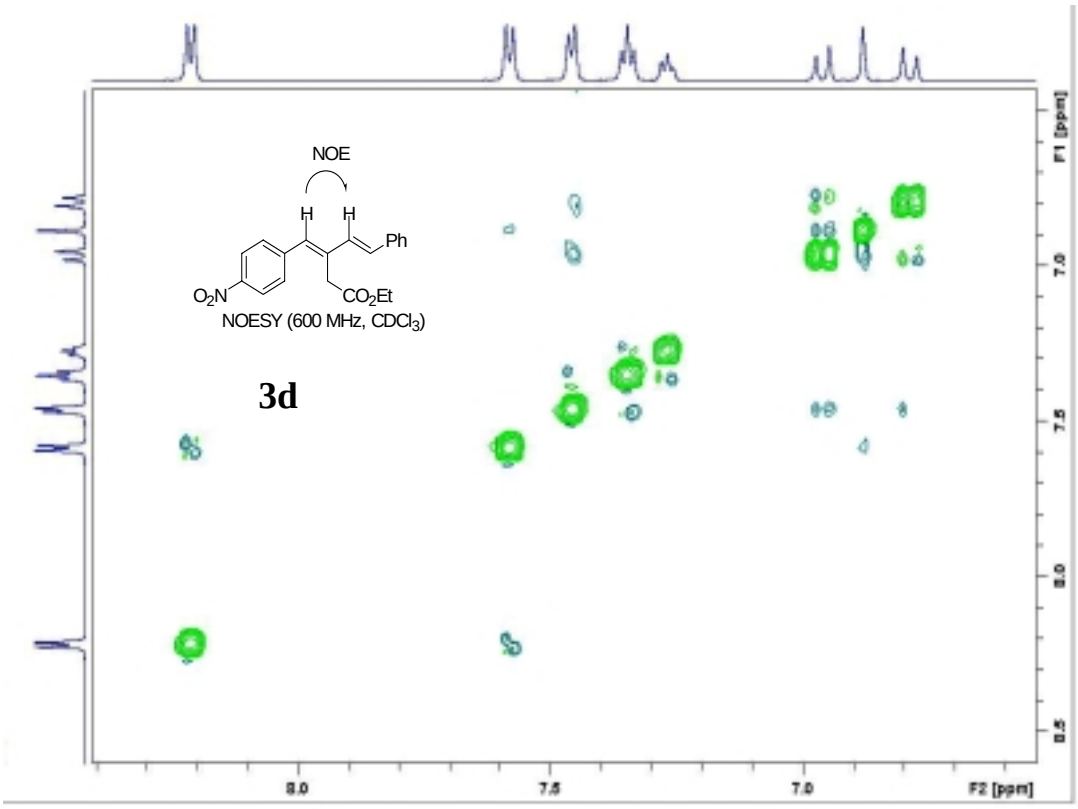
Acq. Date:
Jul 3 2008

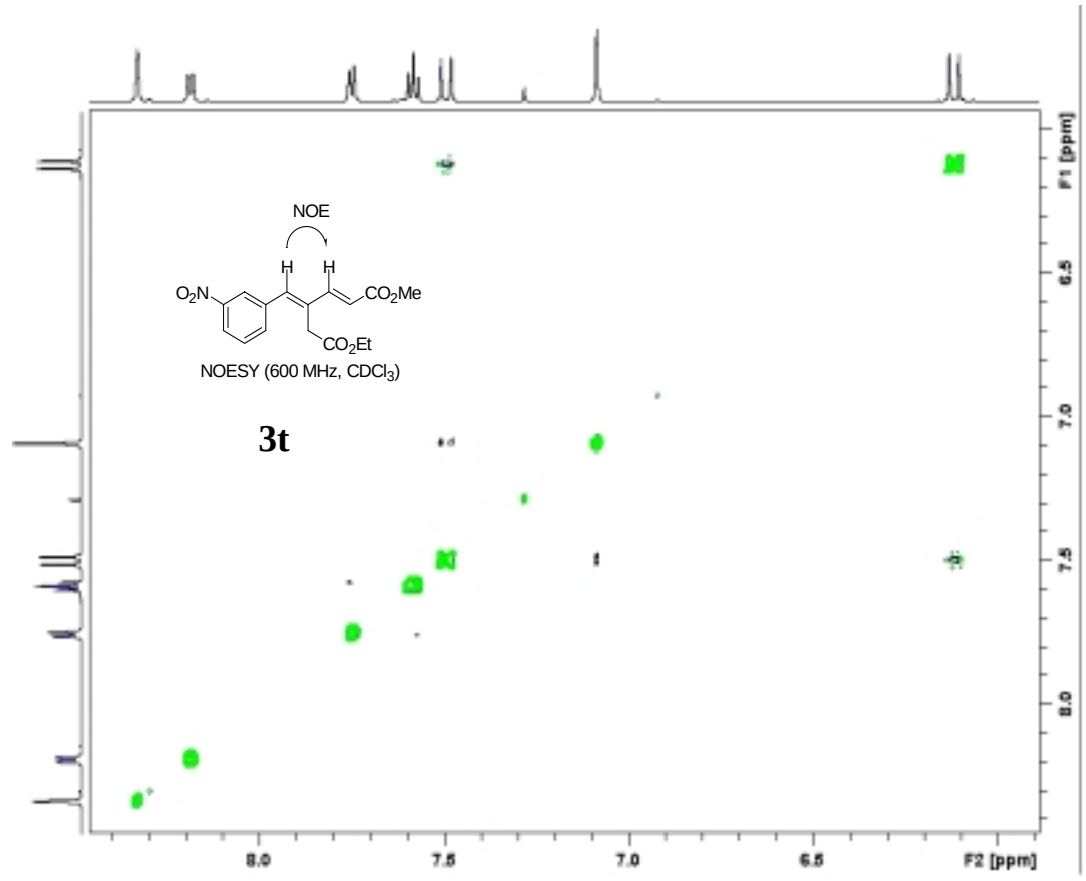


Date:
25 Jun 2009
Document's Title:
ester-2-OH-C.mrc

Spectrum Title:
None
Frequency (MHz):
(f1) 75.468
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.8219
Spectral Width (ppm):
(f1) 238.322
Pulse Program:
Unknown

NOSEY Spectra of 3d and 3t





ORTEP Representation of 3c and 3t

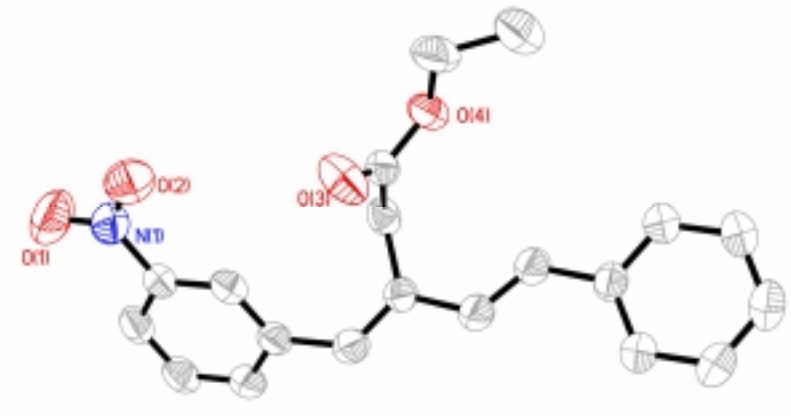


Table 1. Crystal Data and Structure Refinement for 3c

Identification code	**3c**
Empirical formula	C20H19NO4
Formula weight	337.36
Temperature	294(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2(1)/c
Unit cell dimensions	a = 6.854(2) Å alpha = 90°
	b = 7.879(2) Å beta = 90°
	c = 32.765(10) Å gamma = 90°
Volume	1769.4(10) $Å^3$
Z	4
Calculated density	1.266 Mg/m^3
Absorption coefficient	0.088 mm^{-1}
F(000)	712
Crystal size	0.26 x 0.22 x 0.20 mm^3
Theta range for data collection	1.24 to 26.42°
Limiting indices	-8<=h<=8, -9<=k<=7, -40<=l<=40
Reflections collected	9978
Independent reflections	3639 [R(int) = 0.0449]
Completeness to theta = 26.42°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9825 and 0.9774
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3639 / 0 / 227
Goodness-of-fit on F^2	0.998
Final R indices [I>2sigma(I)]	R1 = 0.0474, wR2 = 0.1033
R indices (all data)	R1 = 0.1189, wR2 = 0.1288
Largest diff. peak and hole	0.137 and -0.177 e. $Å^{-3}$

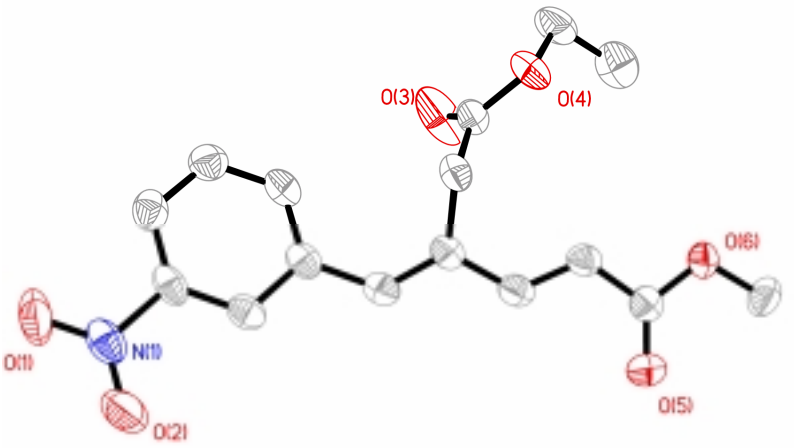


Table 2. Crystal Data and Structure Refinement for 3t

Identification code	**3t**
Empirical formula	C16H17NO6
Formula weight	319.31
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 8.2434(16) Å alpha = 82.63(3)° b = 9.3954(19) Å beta = 84.66(3)° c = 10.116(2) Å gamma = 80.19(3)°
Volume	763.6(3) $Å^3$
Z	2
Calculated density	1.389 Mg/m^3
Absorption coefficient	0.107 mm^{-1}
F(000)	336
Crystal size	0.16 x 0.12 x 0.08 mm^3
Theta range for data collection	2.04 to 25.01°
Limiting indices	-9<=h<=7, -11<=k<=11, -12<=l<=11
Reflections collected	5656
Independent reflections	2666 [R(int) = 0.0248]
Completeness to theta = 25.01°	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9915 and 0.9831
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2666 / 0 / 210
Goodness-of-fit on F^2	1.072
Final R indices [I>2sigma(I)]	R1 = 0.0327, wR2 = 0.0915
R indices (all data)	R1 = 0.0424, wR2 = 0.0962
Largest diff. peak and hole	0.169 and -0.228 e. $Å^{-3}$