Supplementary Information for

Fluorenes as new molecular scaffolds for carbon–carbon σ-bond cleavage reaction: acylfluorenylation of arynes

Hiroto Yoshida,* Takeshi Kishida, Masahiko Watanabe, and Joji Ohshita

Department of Applied Chemistry, Graduate School of Engineering, Hiroshima University, Higashi-Hiroshima 739-8527, Japan.

Contents

General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (^{1}H, 270 MHz; ^{13}C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (^{1}H, 400 MHz; ^{13}C, 99.5 MHz) spectrometer using residual chloroform (^{1}H, δ = 7.26) or $CDCl_3$ (^{13}C, δ = 77.0) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra were obtained with a JEOL JMS-SX102A spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. The preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (benzene or chloroform as an eluent). Column chromatography was carried out using Merck Kieselgel 60. Unless otherwise noted, commercially available reagents were used without purification. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h. THF was distilled from sodium/benzophenone ketyl. MeCN was distilled from phosphorus pentoxide.

Aryne Precursors. 2-(Trimethylsilyl)phenyl triflate (**1a**),[1] 3-(trimethylsilyl)-2-naphthyl triflate (**1b**),[2] 6-(trimethylsilyl)-5-indanyl triflate (**1c**),[3] 3-(trimethylsilyl)-5,6,7,8-tetrahydro-2-naphthyl triflate (**1d**),[2] 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1e**),[3] 1-(trimethylsilyl)-2-naphthyl triflate (**1f**),[4] 3-methoxy-2-(trimethylsilyl)phenyl triflate (**1g**)[5] and 4-methyl-2-(trimethylsilyl)phenyl triflate (**1h**)[6] were prepared according to literature procedures.

Fluorenes. Benzoyl fluorene (**2b**) was prepared according to a literature method.[7] Other fluorenyl ketones, except for **2g**, were synthesized in a similar manner as the preparation of **2b**. Ethyl (**2i**) or isopropyl (**2j**) fluorene-9-carboxylate was prepared by standard esterification of fluorene-9-carboxylic acid. Ethyl esters of substituted fluorenecarboxylic acids (**2k**–**2m**) were prepared from the respective substituted fluorenes according to a literature method.[8] 2,7-Bis(phenylethynyl)fluorene was synthesized by the Sonogashira coupling of 2,7-dibromofluorene and phenylacetylene.[9]

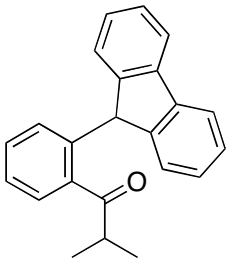
Preparation of 2g. To a THF solution (10 mL) of fluorene (1.66 g, 10.0 mol) was added dropwise *n*-BuLi (1.57 M in hexane, 7.00 mL, 11.0 mmol) at 0 °C, and the resulting solution was stirred at 0 °C for 1 h before addition of anhydrous $MgCl_2$ (1.05 g, 11.0 mmol) at 0 °C. After the mixture was stirred at 0 °C for 0.5 h, methyl *o*-toluate (4.05 g, 27.0 mmol) was added at 0 °C, and stirring was continued for 2 h at room temperature. The mixture was quenched with saturated aqueous NH_4Cl solution, and extracted with ethyl acetate. The

combined organic layer was dried over $MgSO_4$, and concentrated. Silica gel column chromatography (hexane/dichloromethane as an eluent) followed by recrystallization (hexane/dichloromethane) gave **2g** as a yellow solid.

A General Procedure for Acylfluorenylation of Arynes.

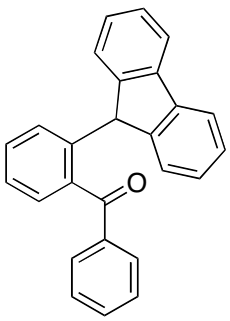
A Schlenk tube equipped with a magnetic stirring bar was charged with KF (0.60 mmol) and 18-crown-6 (0.60 mmol). The tube was evacuated at room temperature for 1 h with stirring before addition of a fluorenes (0.20 mmol), THF (10 mL) and an aryne precurosor (0.30 mmol). The resulting mixture was stirred at room temperature for the period as specified in Table 1 or Scheme 2. The mixture was diluted with ethyl acetate, filtered through a Celite plug, washed three times with brine and dried over $MgSO_4$. Evaporation of the solvent followed by silica gel column chromatography (hexane/dichloromethane as an eluent) or gel permeation chromatography gave the corresponding product.

2-(9*H*-Fluoren-9-yl)phenyl isopropyl ketone (3aa).



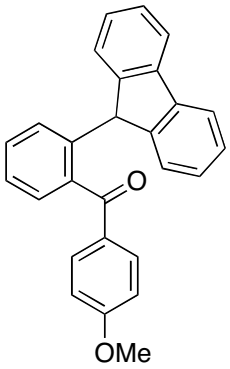
Isolated in 97% yield as a white solid: m.p. 140–142 °C; ^{1}H NMR ($CDCl_3$) δ 1.35 (d, J = 6.5 Hz, 6H), 3.57 (sept, J = 6.5 Hz, 1H), 5.44 (s, 1H), 6.49 (d, J = 7.6 Hz, 1H), 7.15 (t, J = 7.3 Hz, 1H), 7.22-7.33 (m, 3H), 7.34-7.46 (m, 4H), 7.60 (d, J = 7.6 Hz, 1H), 7.83 (d, J = 7.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.6, 39.4, 50.1, 119.7, 125.5, 126.3, 126.6, 127.2, 127.3, 129.4, 130.8, 140.2, 140.9, 141.1, 148.9, 209.5; HRMS Calcd for $C_{23}H_{20}O$: M^+, 312.1514. Found: *m*/*z* 312.1518.

2-(9*H*-Fluoren-9-yl)benzophenone (3ab).



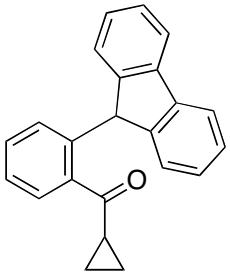
Isolated in 90% yield as a white solid: m.p. 170–172 °C; ^{1}H NMR ($CDCl_3$) δ 5.18 (s, 1H), 6.45 (brs, 1H), 6.95-7.17 (m, 4H), 7.17-7.56 (m, 8H), 7.63 (d, *J* = 7.3 Hz, 2H), 7.88 (brs, 2H); ^{13}C NMR ($CDCl_3$) δ 50.3, 119.7, 120.1, 125.5, 125.9, 127.3, 128.2, 128.5, 128.9, 130.3, 130.6, 133.5, 137.8, 139.6, 140.8, 141.0, 148.3, 198.4; HRMS Calcd for $C_{26}H_{18}O$: M^+, 346.1358. Found: *m*/*z* 346.1360.

2-(9*H*-Fluoren-9-yl)-4'-methoxybenzophenone (3ac).



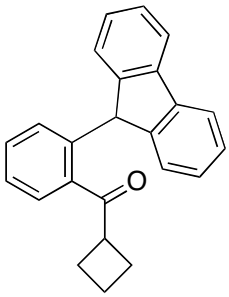
Isolated in 74% yield as a white solid: m.p. 155–159 °C; ^{1}H NMR ($CDCl_3$) δ 3.90 (s, 3H), 5.29 (s, 1H), 6.58 (brs, 1H), 7.00 (d, *J* = 8.1 Hz, 2H), 7.17-7.43 (m, 9H), 7.77 (d, *J* = 8.1 Hz, 2H), 8.00 (brs, 2H); ^{13}C NMR ($CDCl_3$) δ 50.5, 55.5, 113.7, 119.8, 125.5, 126.0, 127.2, 127.3, 127.8, 128.8, 130.2, 130.8, 132.7, 140.1, 140.5, 141.1, 148.3, 164.0, 197.1; HRMS Calcd for $C_{27}H_{20}O_2$: M^+, 376.1463. Found: *m*/*z* 376.1468.

2-(9*H*-Fluoren-9-yl)phenyl cyclopropyl ketone (3ad).



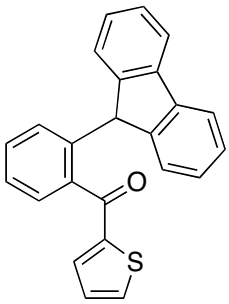
Isolated in 71% yield as a white solid: m.p. 139–142 °C; ^{1}H NMR ($CDCl_3$) δ 1.19 (brs, 2H), 1.41 (brs, 2H), 2.70 (brs, 1H), 5.75 (s, 1H), 6.52 (brs, 1H), 7.10-7.48 (m, 8H), 7.76-7.94 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 12.5, 21.4, 49.8, 119.8, 125.5, 126.4, 127.1, 127.3, 127.9, 129.0, 131.1, 140.3, 141.0, 141.1, 148.6, 205.4; HRMS Calcd for $C_{23}H_{18}O$: M^+, 310.1358. Found: *m*/*z* 310.1360.

2-(9*H*-Fluoren-9-yl)phenyl cyclobutyl ketone (3ae).



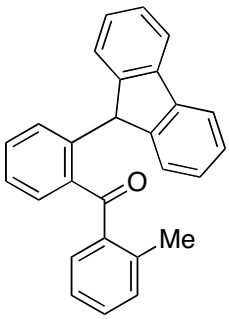
Isolated in 69% yield as a white solid: m.p. 116–119 °C; ^{1}H NMR ($CDCl_3$) δ 1.80-2.22 (m, 2H), 2.22-2.66 (m, 4H), 4.02-4.30 (m, 1H), 5.85 (s, 1H), 6.52 (d, *J* = 7.2 Hz, 1H), 7.08-7.52 (m, 8H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 17.8, 25.3, 44.8, 49.8, 119.8, 125.5, 126.3, 127.1, 127.3, 127.7, 129.5, 131.3, 138.3, 141.1, 141.5, 148.8, 205.8; HRMS Calcd for $C_{24}H_{20}O$: M^+, 324.1514. Found: *m*/*z* 324.1528.

2-(9*H*-Fluoren-9-yl)phenyl 2-thienyl ketone (3af).



Isolated in 58% yield as a yellow solid: m.p. 186–190 °C; ^{1}H NMR ($CDCl_3$) δ 5.40 (s, 1H), 6.59 (brs, 1H), 7.16-7.50 (m, 9H), 7.59 (d, *J* = 7.3 Hz, 1H), 7.64-7.90 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 50.3, 119.7, 125.6, 125.9, 127.2, 127.3, 127.9, 128.3, 129.0, 130.7, 135.4, 135.7, 139.5, 140.6, 141.1, 145.3, 148.3, 190.2; HRMS Calcd for $C_{24}H_{16}OS$: M^+, 352.0922. Found: *m*/*z* 352.0912.

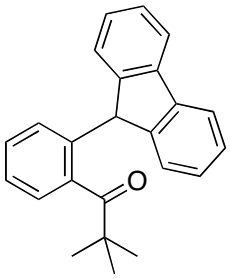
2-(9*H*-Fluoren-9-yl)-2'-methylbenzophenone (3ag).



Isolated in 63% yield as a white solid: m.p. 124–127 °C; ^{1}H NMR ($CDCl_3$) δ 2.63 (s, 3H), 5.59 (s, 1H), 6.55 (brs, 1H), 7.19-7.62 (m, 13H), 7.79 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.3, 50.0, 119.8, 125.5, 126.1, 127.2, 127.4, 129.1, 129.4, 129.5, 131.1, 131.4,

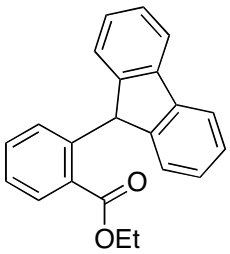
131.75, 131.81, 137.6, 138.4, 139.2, 140.5, 141.1, 148.6, 200.7; HRMS Calcd for $C_{27}H_{20}O$: M^+, 360.1514. Found: *m*/*z* 360.1515.

2-(9*H*-Fluoren-9-yl)phenyl *t*-butyl ketone (3ah).



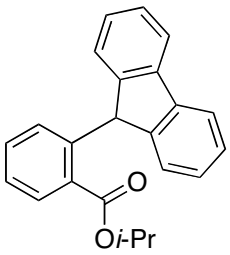
Isolated in 52% yield as a white solid: m.p. 168–171 °C; ^{1}H NMR ($CDCl_3$) δ 1.43 (s, 9H), 4.81 (s, 1H), 6.42 (d, *J* = 8.1 Hz, 1H), 7.08 (t, *J* = 6.9 Hz, 1H), 7.15-7.51 (m, 8H), 7.80 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 27.6, 45.3, 51.4, 119.8, 124.5, 125.6, 125.8, 127.4, 127.5, 128.6, 129.2, 138.7, 141.0, 141.7, 148.3, 214.6; HRMS Calcd for $C_{24}H_{22}O$: M^+, 326.1671. Found: *m*/*z* 326.1677.

Ethyl 2-(9*H*-fluoren-9-yl)benzoate (3ai).



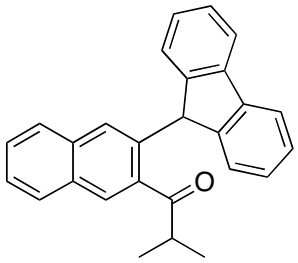
Isolated in 93% yield as a white solid: m.p. 86–89 °C; ^{1}H NMR ($CDCl_3$) δ 1.48 (t, *J* = 7.1 Hz, 3H), 4.52 (q, *J* = 7.1 Hz, 2H), 6.23 (s, 1H), 6.52 (d, *J* = 7.5 Hz, 1H), 7.15-7.41 (m, 8H) , 7.84 (d, *J* = 7.5 Hz, 2H), 8.00 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 50.0, 61.3, 119.8, 125.5, 126.4, 127.1, 127.3, 129.0, 130.1, 131.1, 132.0, 141.1, 142.8, 148.4, 168.3; HRMS Calcd for $C_{22}H_{18}O_2$: M^+, 314.1307. Found: *m*/*z* 314.1302.

Isopropyl 2-(9*H*-fluoren-9-yl)benzoate (3aj).



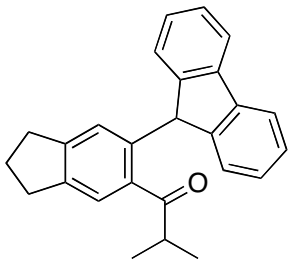
Isolated in 88% yield as a white solid: m.p. 76–78 °C; ^{1}H NMR ($CDCl_3$) δ 1.47 (d, *J* = 6.2 Hz, 6H), 5.42 (sept, *J* = 6.2 Hz, 1H), 6.20 (s, 1H), 6.51 (d, *J* = 7.6 Hz, 1H), 7.05-7.45 (m, 8H), 7.84 (d, *J* = 7.6 Hz, 2H), 7.97 (d, *J* = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 22.0, 50.1, 68.9, 119.8, 125.5, 126.4, 127.1, 127.3, 128.9, 130.0, 131.7, 131.9, 141.1, 142.6, 148.5, 167.9; HRMS Calcd for $C_{23}H_{20}O_2$: M^+, 328.1463. Found: *m*/*z* 328.1465.

3-(9*H*-Fluoren-9-yl)-2-naphthyl isopropyl ketone (3ba).



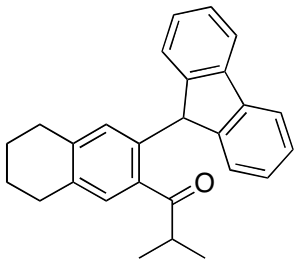
Isolated in 83% yield as a white solid: m.p. 125–128 °C; ^{1}H NMR ($CDCl_3$) δ 1.38 (d, *J* = 6.9 Hz, 6H), 3.75 (sept, *J* = 6.9 Hz, 1H), 5.56 (s, 1H), 6.98 (s, 1H), 7.20-7.25 (m, 2H), 7.35-7.44 (m, 7H), 7.82-7.88 (m, 3H), 8.15 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 18.7, 39.5, 50.1, 119.8, 125.6, 126.3, 127.1, 127.3, 127.4, 127.5, 128.2, 128.5, 129.0, 131.1, 134.3, 137.9, 138.4, 141.0, 149.4, 209.4; HRMS Calcd for $C_{27}H_{22}O$: M^+, 362.1671. Found: *m*/*z* 362.1663.

6-(9*H*-Fluoren-9-yl)-5-indanyl isopropyl ketone (3ca).



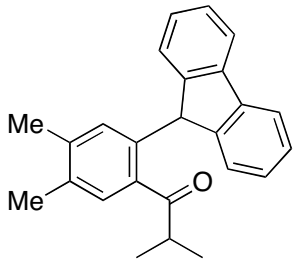
Isolated in 81% yield as a white solid: m.p. 127–131 °C; ^{1}H NMR ($CDCl_3$) δ 1.34 (d, *J* = 6.9 Hz, 6H), 1.99 (quint, *J* = 7.2 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 2.90 (t, *J* = 7.3 Hz, 2H), 3.57 (sept, *J* = 6.9 Hz, 1H), 5.46 (s, 1H), 6.32 (s, 1H), 7.23-7.48 (m, 7H),7.82 (d, *J* = 8.2Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.8, 25.2, 32.4, 32.6, 39.2, 50.1, 119.7, 122.5, 124.9, 125.6, 127.0, 127.3, 128.3, 138.4, 141.1, 142.3, 147.7, 149.3, 209.4; HRMS Calcd for $C_{26}H_{24}O$: M^+, 352.1827. Found: *m*/*z* 352.1835.

3-(9*H*-Fluoren-9-yl)-5,6,7,8-tetrahydro-2-naphthyl isopropyl ketone (3da).



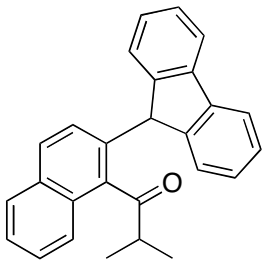
Isolated in 74% yield as a white solid: m.p. 118–121 °C; ^{1}H NMR ($CDCl_3$) δ 1.32 (d, *J* = 6.9 Hz, 6H), 1.67-1.74 (m, 4H), 2.42 (t, *J* = 5.9 Hz, 2H), 2.77 (t, *J* = 5.9 Hz, 2H), 3.57 (sept, *J* = 6.9 Hz, 1H), 5.46 (s, 1H), 6.17 (s, 1H), 7.22-7.40 (m, 7H), 7.79-7.82 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 18.8, 22.7, 22.9, 29.01, 29.04, 39.0, 49.8, 119.6, 125.6, 127.0, 127.2, 127.6, 129.6, 135.2, 137.4, 137.9, 140.7, 141.0, 149.2, 209.4; HRMS Calcd for $C_{27}H_{26}O$: M^+, 366.1984. Found: *m*/*z* 366.1991.

2-(9*H*-Fluoren-9-yl)-4,5-dimethylphenyl isopropyl ketone (3ea).



Isolated in 59% yield as a white solid: m.p. 112–117 °C; ^{1}H NMR ($CDCl_3$) δ 1.33 (d, *J* = 6.6 Hz, 6H), 1.98 (s, 3H), 2.25 (s, 1H), 3.57 (sept, *J* = 6.6 Hz, 1H), 5.47 (s, 1H), 6.23 (s, 1H), 7.22-7.41 (m, 7H),7.82 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.8, 19.5, 19.6, 39.1, 49.8, 119.7, 125.5, 127.0, 127.3, 128.0, 130.2, 134.7, 137.7, 138.4, 140.1, 141.1, 149.1, 209.4; HRMS Calcd for $C_{25}H_{24}O$: M^+, 340.1827. Found: *m*/*z* 340.1821.

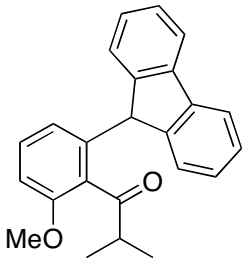
2-(9*H*-Fluoren-9-yl)-1-naphthyl isopropyl ketone (3fa).



Isolated in 80% yield as a white solid: m.p. 143–145 °C; ^{1}H NMR ($CDCl_3$) δ 1.47 (d, *J* = 6.9 Hz, 6H), 3.54 (sept, *J* = 6.9 Hz, 1H), 5.07 (s, 1H), 6.46 (d, *J* = 8.9 Hz, 1H), 7.26-7.61 (m, 9H), 7.73-7.89 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 18.3, 43.8, 51.3, 119.9, 124.8, 125.3, 125.4,

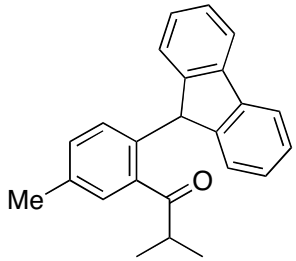
126.0, 126.8, 127.55, 127.63, 128.3, 129.5, 129.7, 132.3, 135.5, 139.2, 147.7, 213.6; Anal. Calcd for $C_{27}H_{22}O$: C, 89.47; H, 6.12. Found: C, 89.19; H, 6.46.

2-(9*H*-Fluoren-9-yl)-6-methoxyphenyl isopropyl ketone (3ga).



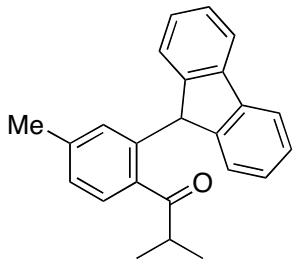
Isolated in 55% yield as a white solid: m.p. 159–162 °C; ^{1}H NMR ($CDCl_3$) δ 1.33 (d, *J* = 6.9 Hz, 6H), 3.38 (sept, *J* = 6.9 Hz, 1H), 3.87 (s, 3H), 4.83 (s, 1H), 6.01 (d, *J* = 7.9 Hz, 1H), 6.75 (d, *J* = 8.2 Hz, 1H), 7.02 (t, *J* = 8.2 Hz, 1H), 7.26 (t, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 2H), 7.46 (d, *J* = 7.3 Hz, 2H), 7.79 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.1, 42.1, 50.8, 55.6, 108.6, 119.7, 120.4, 125.6, 127.3, 128.3, 130.2, 132.1, 140.3, 141.0, 148.2, 156.1, 211.8; Anal. Calcd for $C_{24}H_{22}O_2$: C, 84.18; H, 6.48. Found: C, 83.96; H, 6.59.

2-(9*H*-Fluoren-9-yl)-5-methylphenyl isopropyl ketone (3ha).



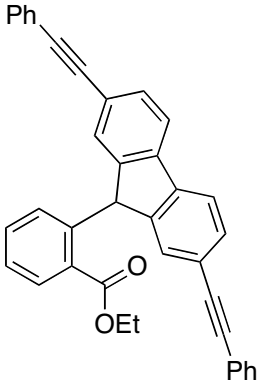
Isolated in 38% yield as a white solid: m.p. 98–101 °C; ^{1}H NMR ($CDCl_3$) δ 1.33 (d, *J* = 6.6 Hz, 6H), 2.34 (s, 3H), 3.56 (sept, *J* = 6.8 Hz, 1H), 5.37 (s, 1H), 6.37 (d, *J* = 7.9 Hz, 1H), 6.96 (d, *J* = 7.9 Hz, 1H), 7.25 (t, *J* = 7.3 Hz, 2H), 7.34-7.42 (m, 5H), 7.82 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.6, 21.0, 39.4, 49.9, 119.7, 125.5, 127.0, 127.1, 127.3, 129.2, 131.7, 135.9, 137.8, 140.3, 141.1, 149.0, 209.7; HRMS Calcd for $C_{24}H_{22}O$: M^+, 326.1671. Found: *m*/*z* 326.1674.

2-(9*H*-Fluoren-9-yl)-4-methylphenyl isopropyl ketone (3'ha).



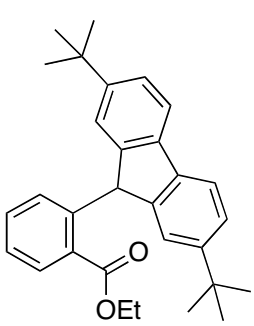
Isolated in 36% yield as a white solid: m.p. 106–111 °C; ^{1}H NMR ($CDCl_3$) δ 1.33 (d, *J* = 6.6 Hz, 6H), 2.08 (s, 3H), 3.56 (sept, *J* = 6.6 Hz, 1H), 5.53 (s, 1H) , 6.28 (s, 1H), 7.06 (d, *J* = 7.9 Hz, 1H), 7.26 (t, *J* = 7.2 Hz, 2H), 7.33-7.45 (m, 4H), 7.54 (d, *J* = 7.9 Hz, 1H), 7.82 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 18.7, 21.2, 39.1, 50.0, 119.7, 125.5, 127.1, 127.2, 127.3, 129.7, 137.3, 141.1, 141.3, 149.0, 209.2; HRMS Calcd for $C_{24}H_{22}O$: M^+, 326.1671. Found: *m*/*z* 326.1670.

Ethyl 2-[2,7-bis(phenylethynyl)-9*H*-fluoren-9-yl]benzoate (3ak).



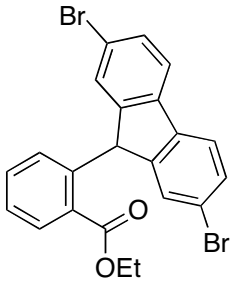
Isolated in 64% yield as a yellow solid: m.p. 147–150 °C; ^{1}H NMR ($CDCl_3$) δ 1.52 (t, *J* = 7.3 Hz, 3H), 4.55 (q, *J* = 7.3 Hz, 2H), 6.33 (s, 1H), 6.57 (d, *J* = 7.7 Hz, 1H), 7.24-7.40 (m, 9H), 7.52-7.63 (m, 7H), 7.80 (d, *J* = 7.7 Hz, 2H), 8.05 (d, *J* = 7.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 49.7, 61.4, 89.9, 90.0, 120.1, 122.3, 123.2, 126.8, 128.1, 128.3, 128.7, 129.1, 130.3, 131.0, 131.5, 132.3, 140.6, 141.9, 148.9, 168.0; HRMS Calcd for $C_{36}H_{26}O_2$: M^+, 514.1933. Found: *m*/*z* 514.1939.

Ethyl 2-(2,7-di-*t*-butyl-9*H*-fluoren-9-yl)benzoate (3al).



Isolated in 61% yield as a white solid: m.p. 166–171 °C; ^{1}H NMR ($CDCl_3$) δ 1.30 (s, 18H), 1.47 (t, *J* = 7.3 Hz, 3H), 4.52 (q, *J* = 7.3 Hz, 2H), 6.09 (s, 1H), 6.55 (d, *J* = 7.9 Hz, 1H), 7.16-7.42 (m, 6H), 7.70 (d, *J* = 7.9 Hz, 2H), 7.97 (d, *J* = 6.3 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.4, 31.5, 34.8, 50.3, 61.4, 119.0, 122.3, 124.2, 126.3, 129.1, 130.0, 131.3, 132.0, 138.6, 143.2, 148.4, 150.1, 168.7; HRMS Calcd for $C_{30}H_{34}O$: M^+, 426.2559. Found: *m*/*z* 426.2552.

Ethyl 2-(2,7-dibromo-9*H*-fluoren-9-yl)benzoate (3am).



Isolated in 32% yield as a white solid: m.p. 135–138 °C; ^{1}H NMR ($CDCl_3$) δ 1.47 (t, *J* = 7.3 Hz, 3H), 4.49 (q, *J* = 7.2 Hz, 2H), 6.23 (s, 1H), 6.47 (d, *J* = 7.6 Hz, 1H), 7.21-7.36 (m, 2H), 7.45-7.52 (m, 4H), 7.63 (d, *J* = 8.2 Hz, 2H), 8.01 (dd, *J* = 7.6, 1.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 49.9, 61.5, 121.2, 121.5, 127.1, 128.3, 129.0, 130.5, 130.6, 130.8, 132.4, 139.1, 141.3, 150.1, 167.8; Anal. Calcd for $C_{22}H_{16}Br_2O_2$: C, 55.96; H, 3.42. Found: C, 56.00; H, 3.54.

References

(1) Y. Himeshima, T. Sonoda and H. Kobayashi, *Chem. Lett*., 1983, 1211.

(2) H. Yoshida, T. Terayama, J. Ohshita and A. Kunai, *Chem. Commun*., 2004, 1980.

(3) H. Yoshida, S. Sugiura and A. Kunai, *Org. Lett*., 2002, **4**, 2767.

(4) D. Peña, D. Pérez, E. Guitián and L. Castedo, *J. Org. Chem*., 2000, **65**, 6944.

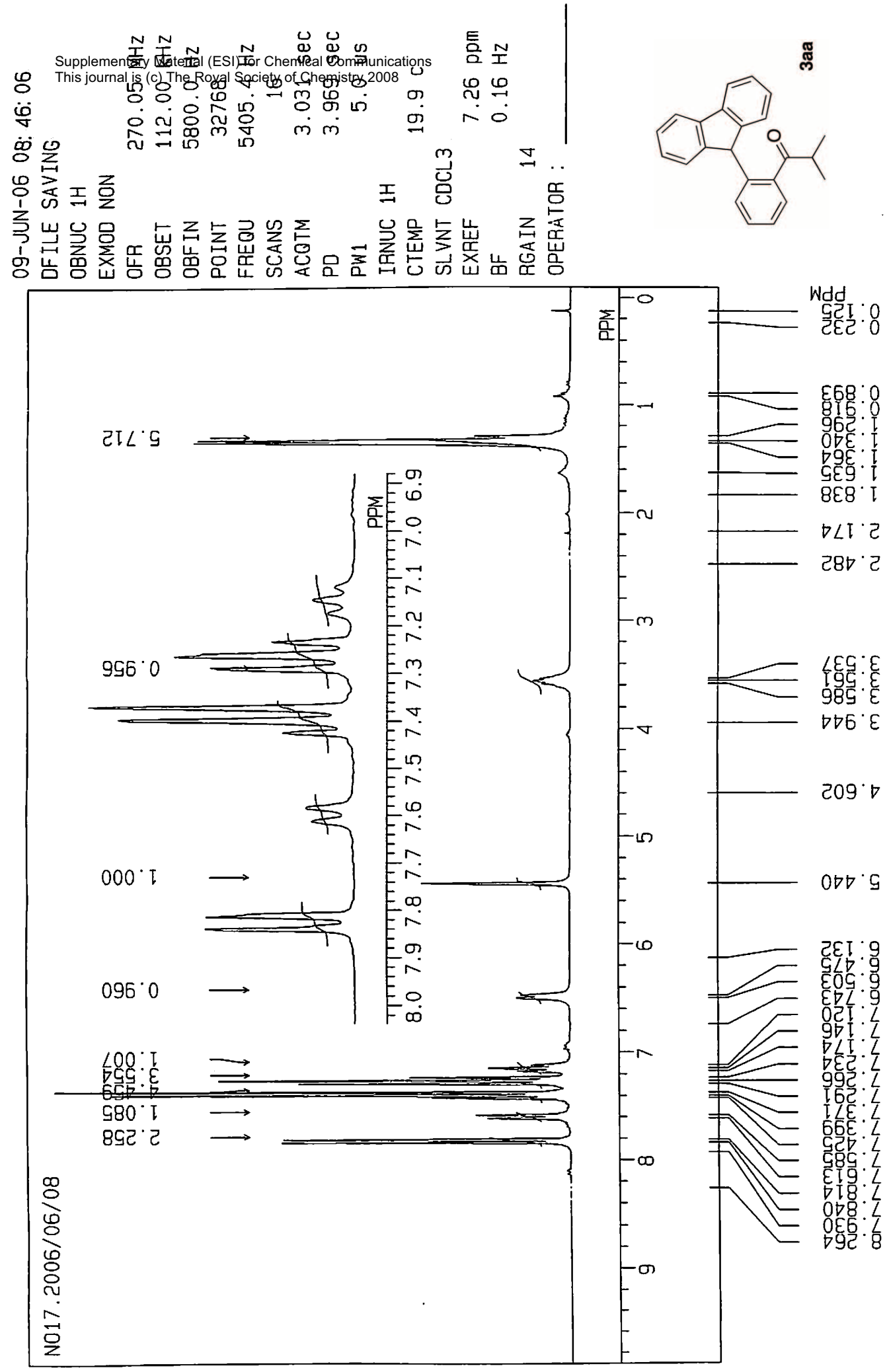
(5) D. Peña, D. Pérez, E. Guitián and L. Castedo, *J. Am. Chem. Soc*., 1999, **121**, 5827.

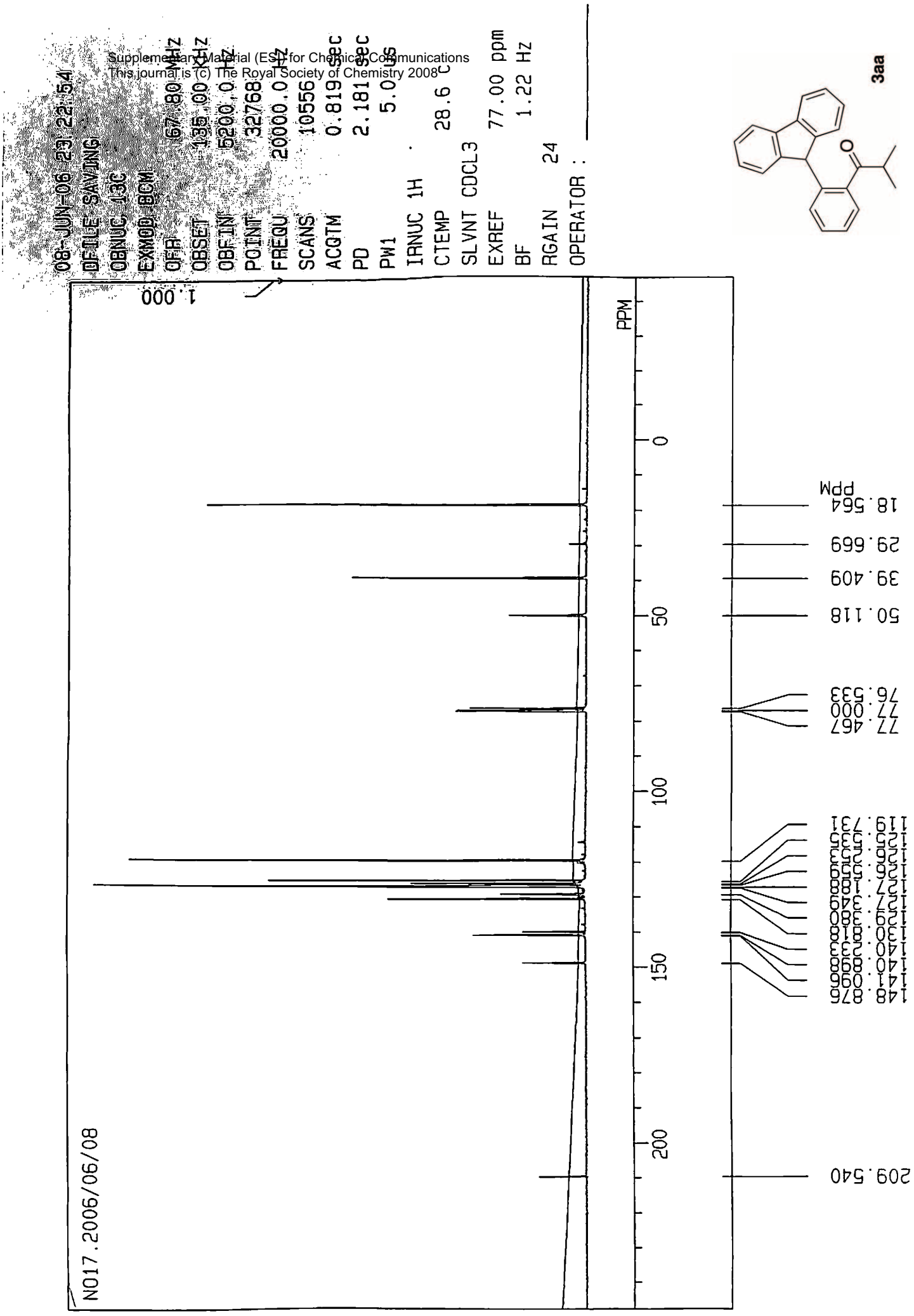
(6) E. Yoshikawa, K. V. Radhakrishnan and Y. Yamamoto, *J. Am. Chem. Soc*., 2000, **122**, 7280.

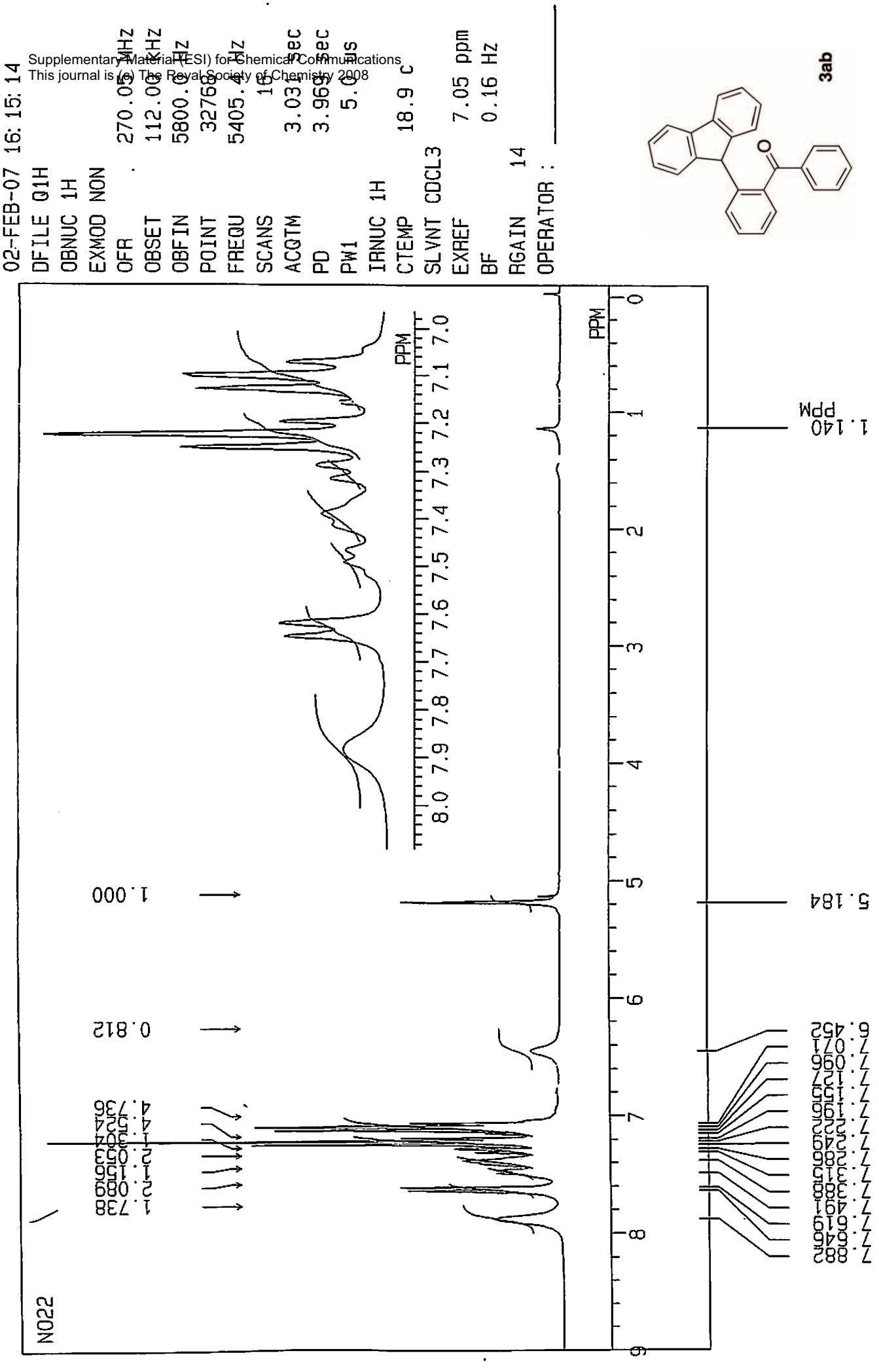
(7) W. S. Matthews, J. E. Bares, J. E. Bartmess, F. G. Bordwell, F. J. Cornforth, G. E. Drucker, Z. Margolin, R. J. McCallum, G. J. McCollum and N. R. Vanier, *J. Am. Chem. Soc*., 1975, **97**, 7006.

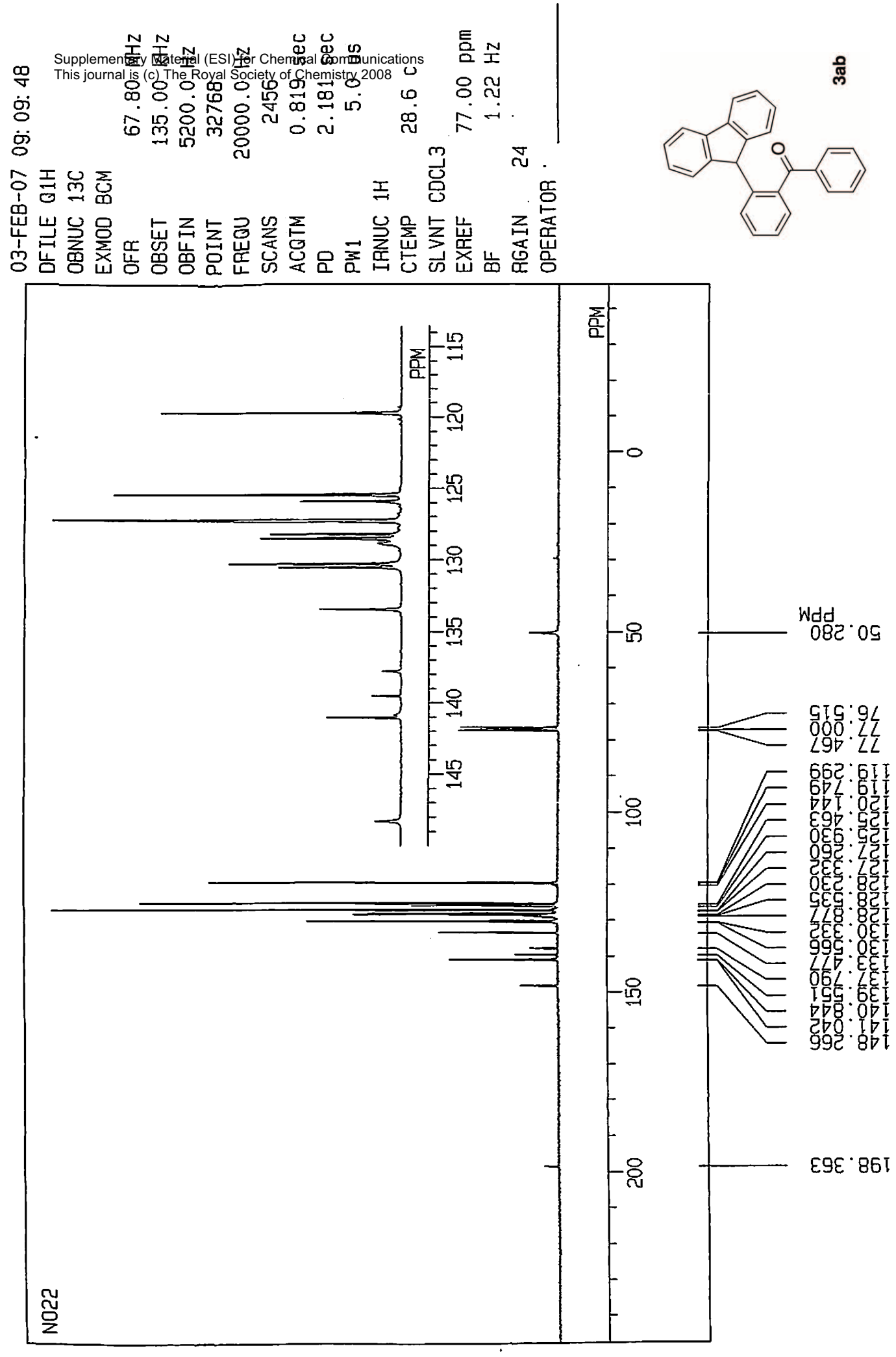
(8) M. Ranger and M. Leclerc, *Macromolecules*, 1999, **32**, 3306.

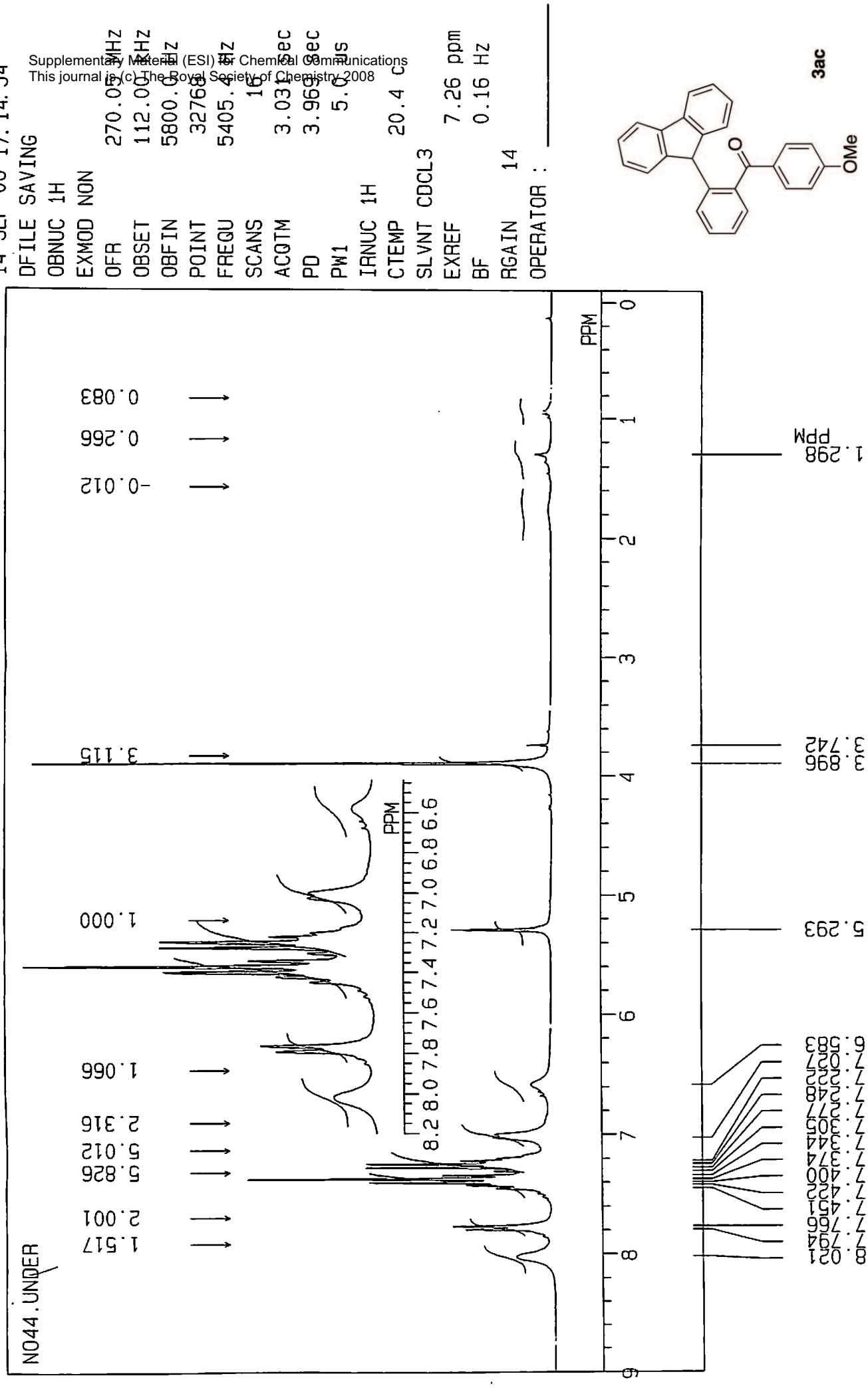
(9) E. Birckner, U. -W. Grummt, A. H. Göller, T. Pautzsch, D. A. M. Egbe, M. Al-Higari and E. Klemm, *J. Phys. Chem. A*, 2001, **105**, 10307.

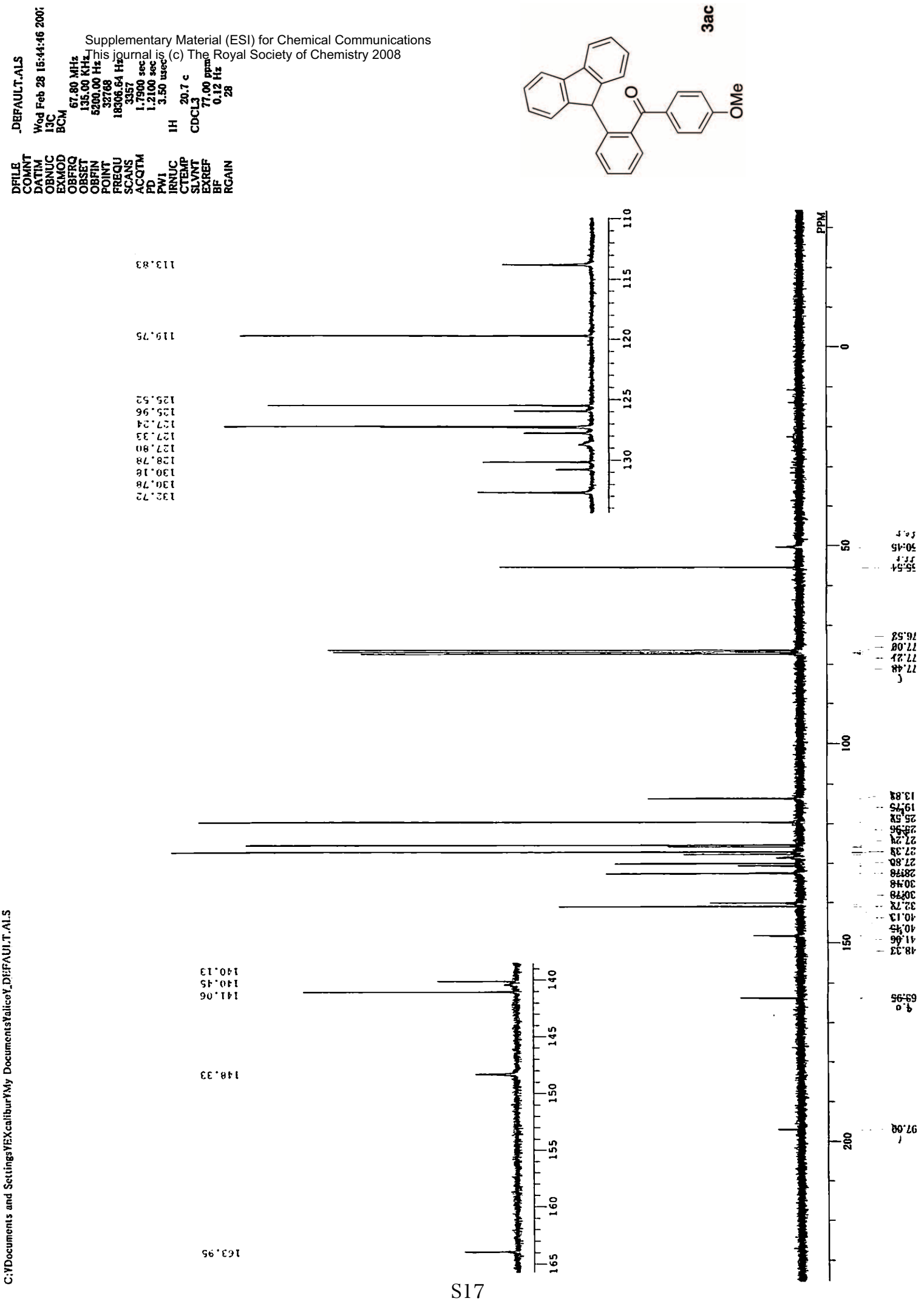

09-JUN-06 08: 46: 06
DFILE SAVING
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.00 MHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 5.0 us
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 14
OPERATOR :
3aa
NO17.2006/06/08
PPM
5.712
0.956
1.000
0.960
2.258
1.085
3.554
1.007
PPM
PPM


08-JUN-06 23:22:54
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 MHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 10556
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 28.6 °C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 24
OPERATOR :
3aa
1.000
PPM
0
50
100
150
200
18.564
29.669
39.409
50.118
76.533
77.000
77.467
119.731
148.876
209.540
N017.2006/06/08


02-FEB-07 16: 15: 14
DFILE Q1H
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.0G kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 5.0 us
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 7.05 ppm
BF 0.16 Hz
RGAIN 14
OPERATOR :
3ab
N022
PPM
1.000
0.812
1.140
5.184

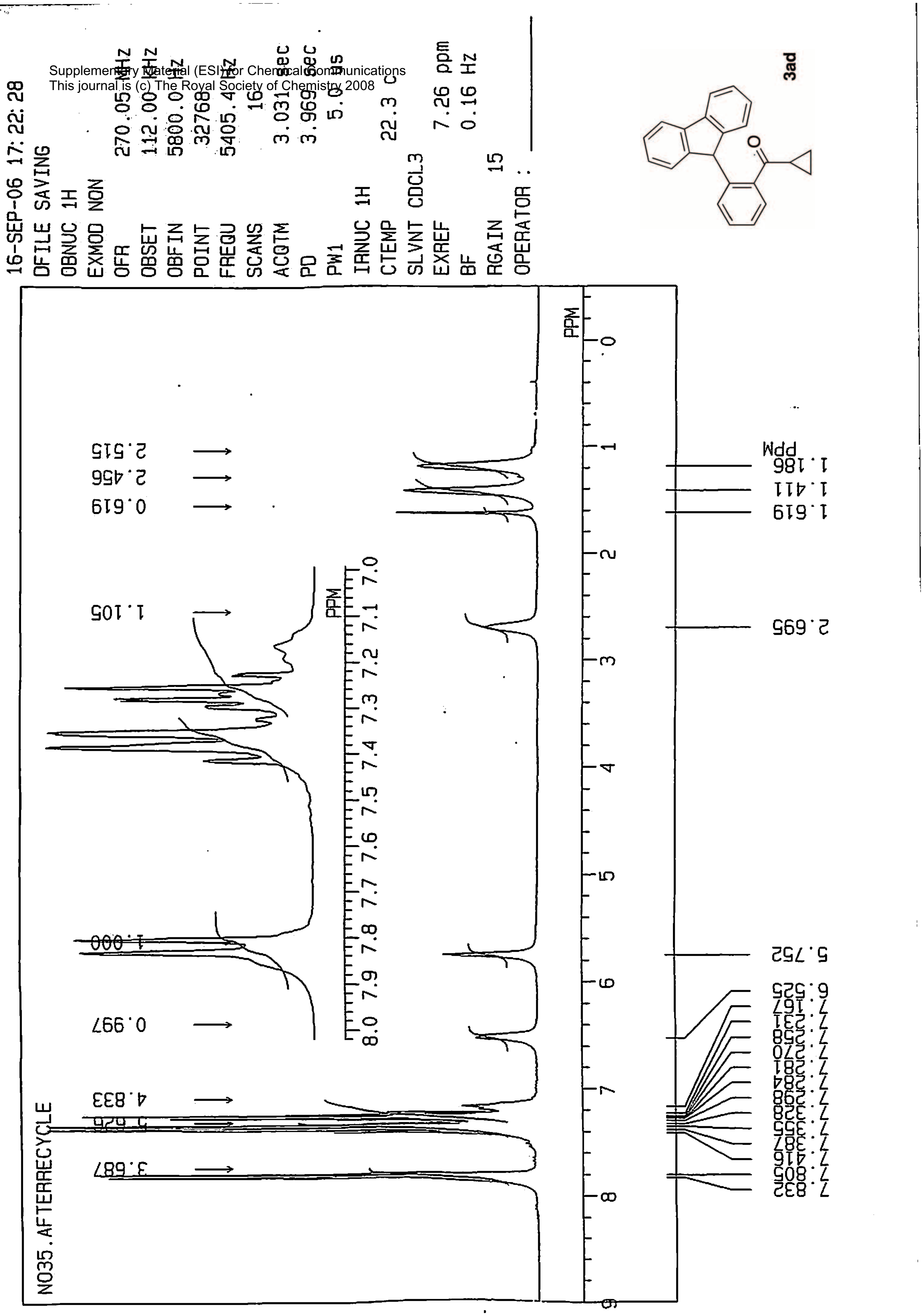

03-FEB-07 09:09:48
DFILE Q1H
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 MHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 2456
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 28.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 24
OPERATOR
3ab
N022
PPM
50.280
76.515
77.000
77.467
119.299
119.749
120.144
125.463
125.930
127.360
127.732
128.332
128.830
128.835
128.877
130.332
130.566
133.477
137.790
139.551
140.844
141.042
148.266
198.363


14-SEP-06 17:14:54
DFILE SAVING
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.00 MHz
OBFIN 5800.00 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.0315 sec
PD 3.9635 sec
PW1 5.00 us
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 14
OPERATOR :
3ac
OMe
O
NO44.UNDER
PPM
1.298
3.742
3.896
5.293
7.261
7.400
7.422
7.451
7.766
7.794
8.021

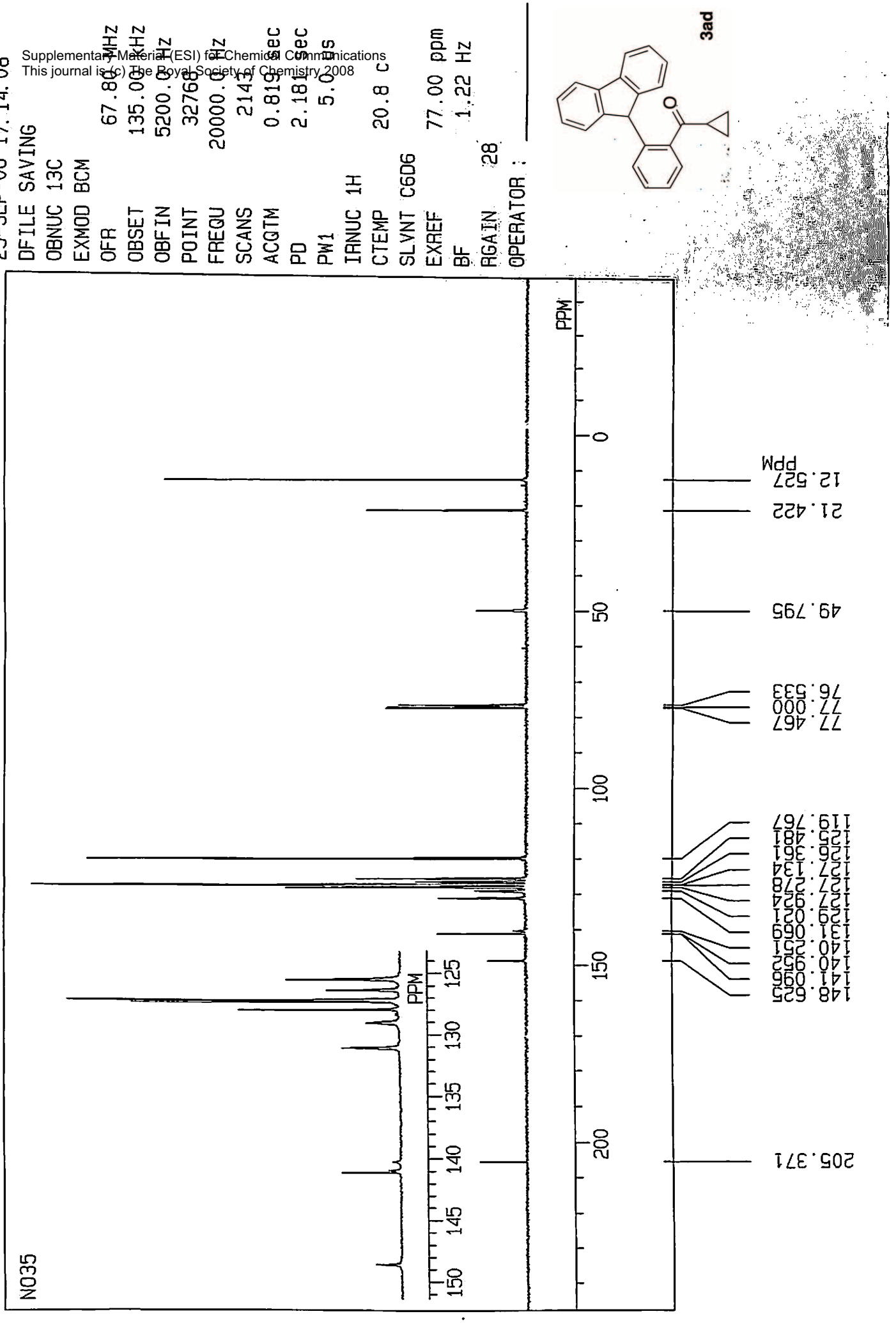


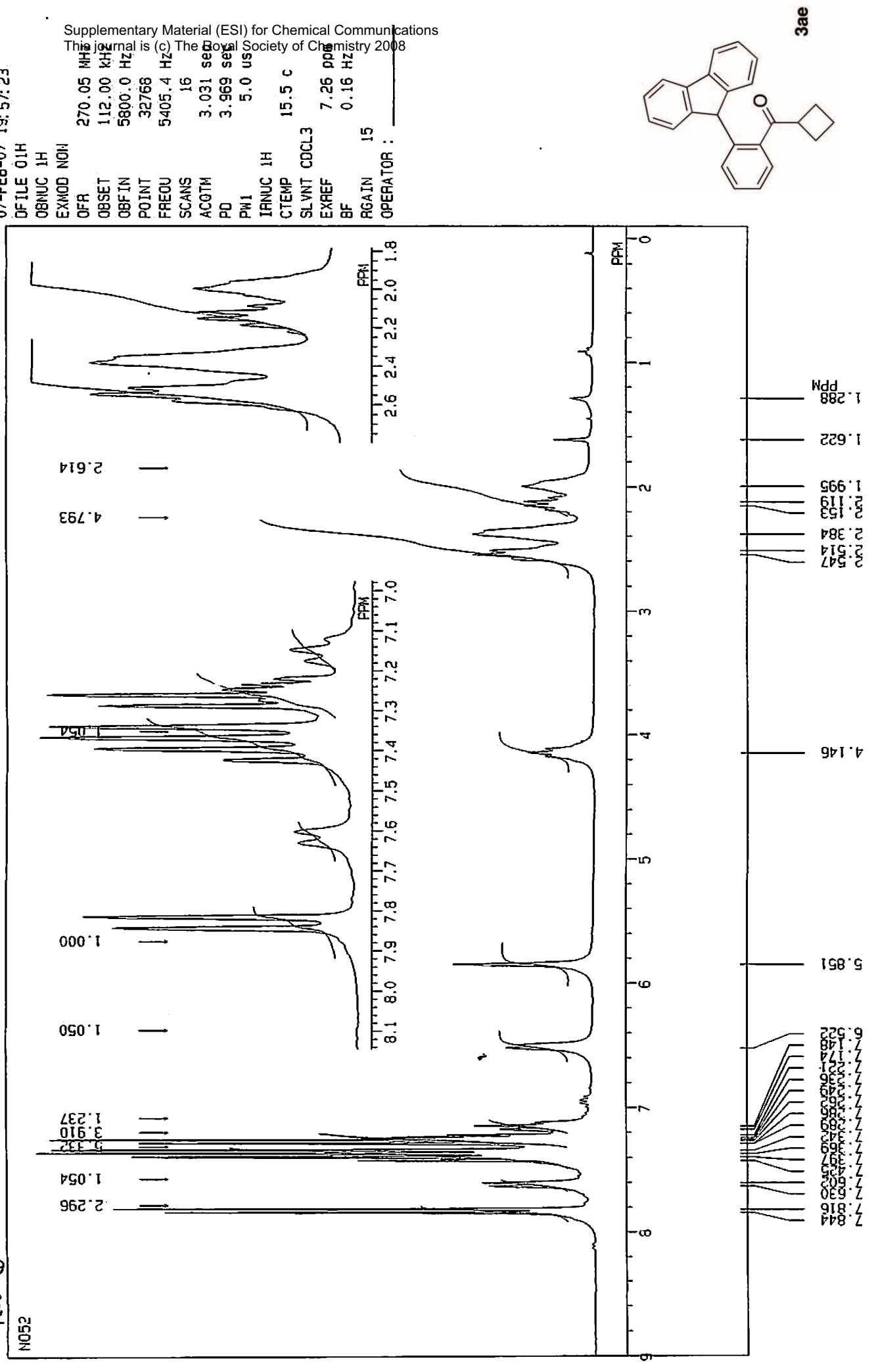
C:\Documents and Settings\Excalibur\My Documents\Alice_DEFAULT.ALS

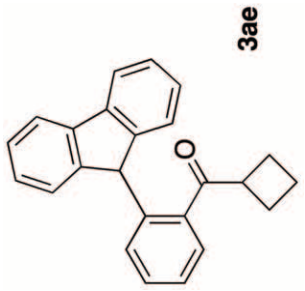
N054 @



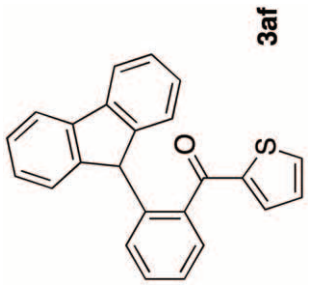
3ad


3ad
25-SEP-06 17:14:08
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 2143
ACQTM 0.8192 sec
PD 2.1811 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.8 c
SLVNT C6D6
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 28
OPERATOR :
N035
PPM
12.527
21.422
49.795
76.533
77.000
77.467
119.767
125.481
125.736
125.961
127.134
127.278
127.924
129.021
131.069
140.257
140.952
141.096
148.625
205.371

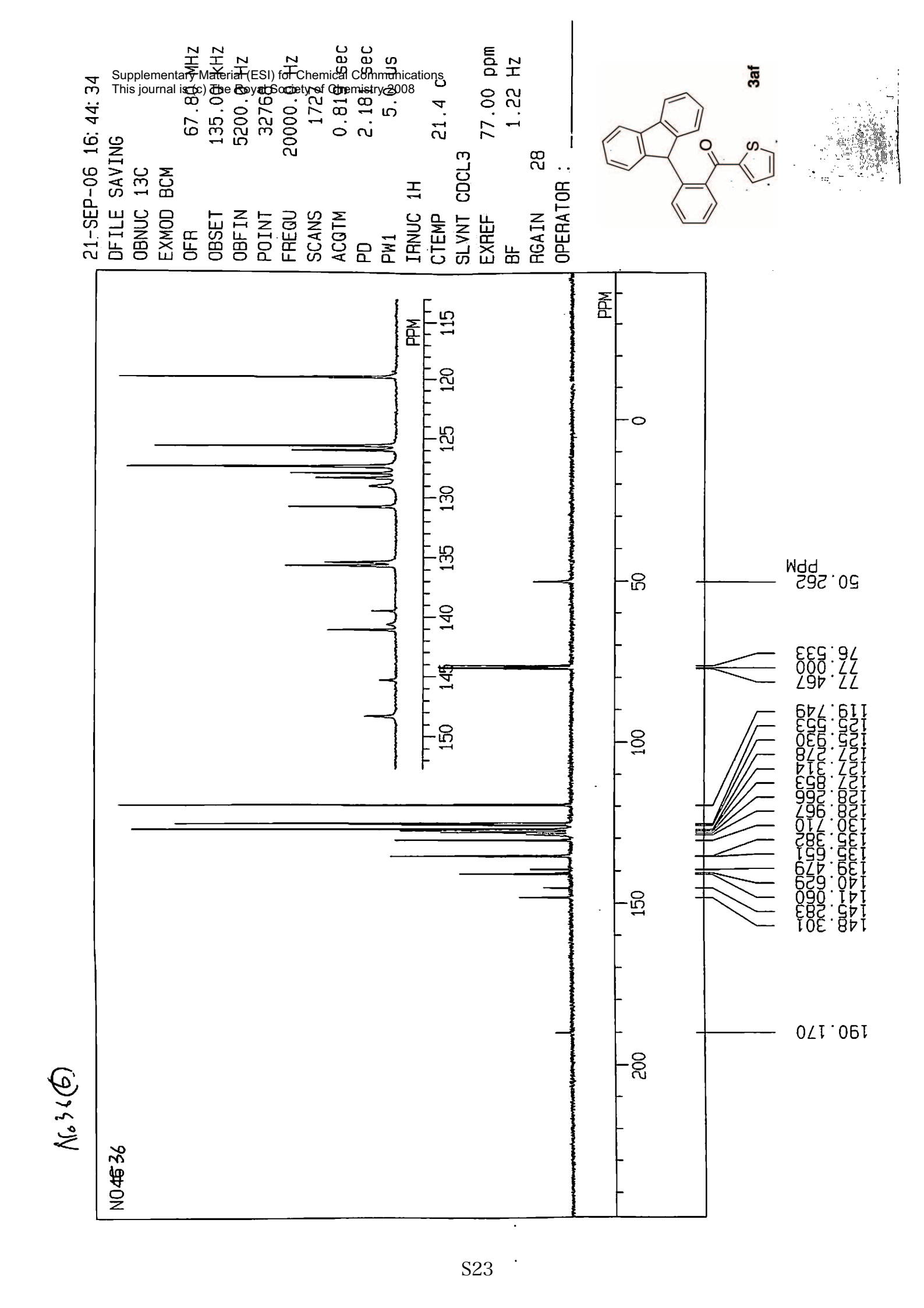

3ae
07-FEB-07 19:57:23
DFILE 01H
OBNUC 1H
EXMOD NOM
OFA 270.05 MHz
OBSET 112.00 kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 5.0 usec
IRNUC 1H
CTEMP 15.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 15
OPERATOR :
NO52
PPM
2.296
1.054
5.132
3.910
1.237
1.050
1.000
1.054
4.793
2.614
1.288
1.622
1.995
2.153
2.119
2.384
2.514
2.547
4.146
5.851
6.522
7.148
7.174
7.816
7.844

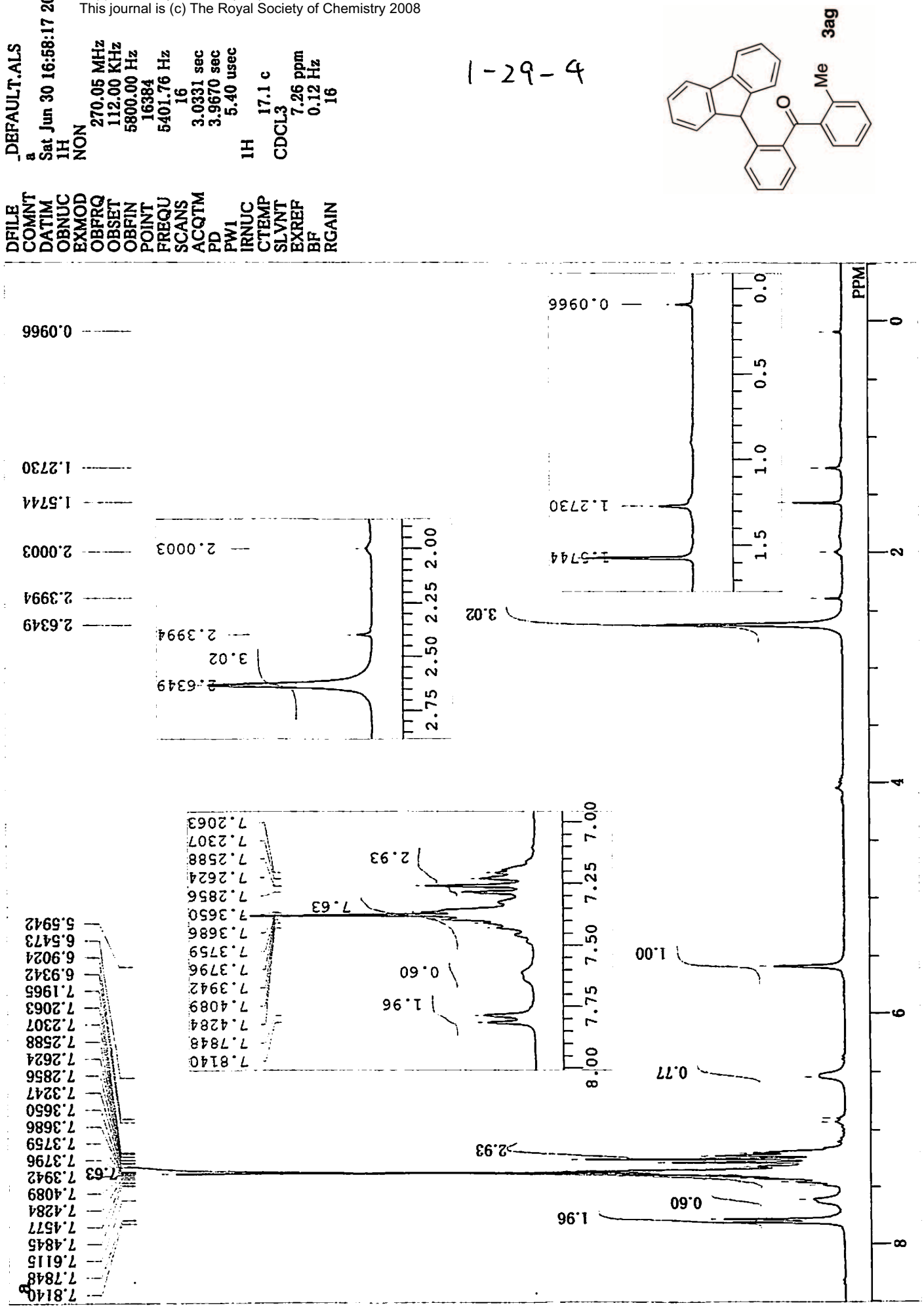

3ae
O

07-FEB-07 14:25:05
DFILE 01H
OBNUC 13C
EXMOD BCM
OFA 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 1807
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 28.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 24
OPERATOR :
N052
PPM
17.846
25.285
44.763
49.849
76.915
77.000
77.467
119.767
148.805
205.821

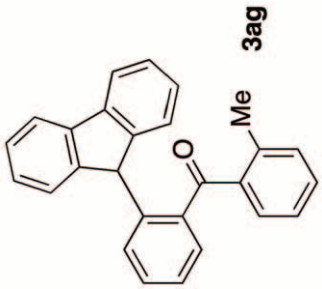

3af
O
S

05-FEB-07 23:46:00
DFILE Q1H
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.00 kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.969 sec
PW1 5.0 us
IRNUC 1H
CTEMP 16.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 18
OPERATOR :
NO51
PPM
1.000
0.892
12.742
6.484
PPM
6.574

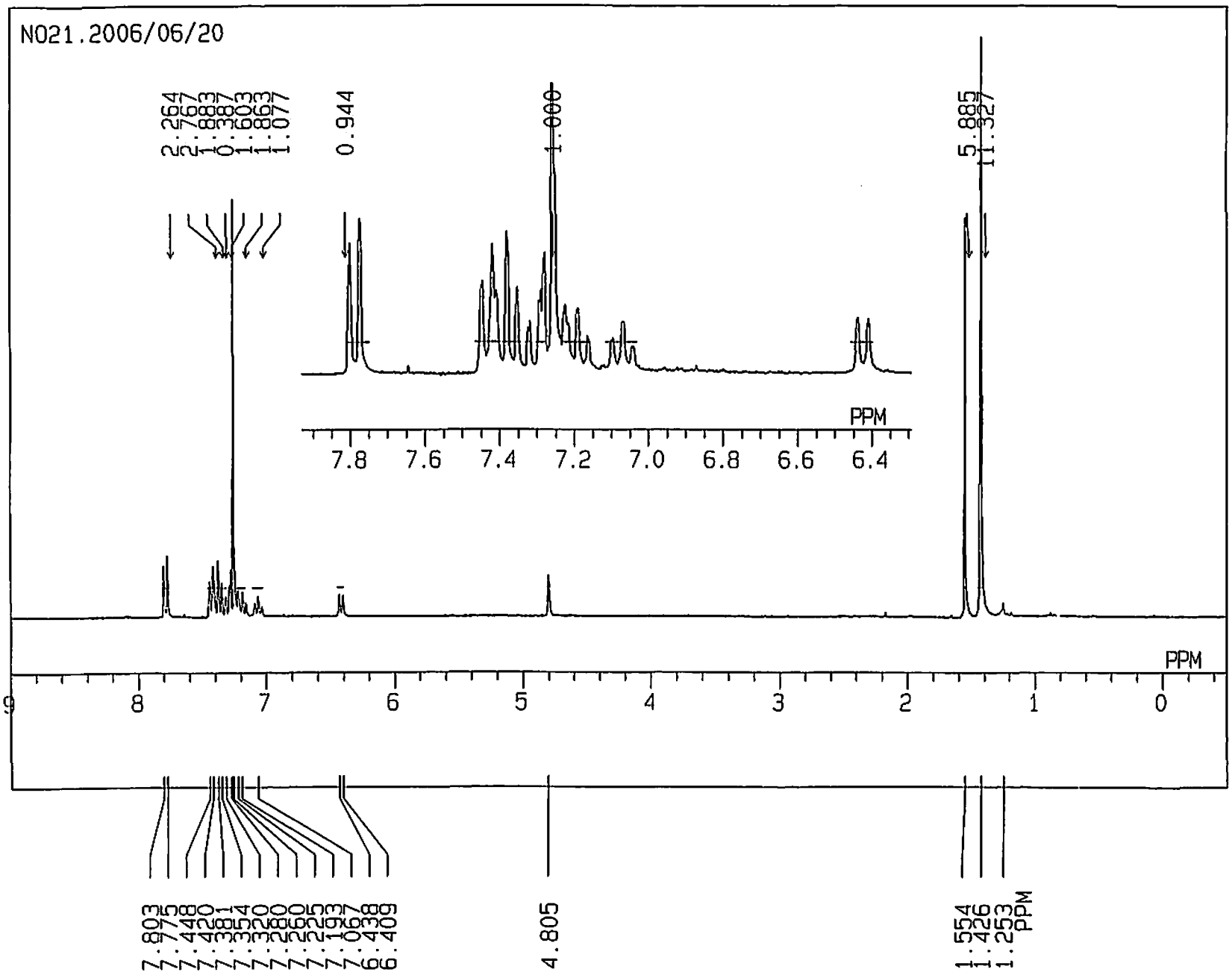

21-SEP-06 16: 44: 34
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 172
ACQTM 0.819 Sec
PD 2.18 Sec
PW1 5.0 us
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 28
OPERATOR :
3af
PPM
190.170
77.467
77.000
76.533
50.262
NO4536


DFILE _DEFAULT.ALS
COMNT a
DATIM Sat Jun 30 16:58:17 2007
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 16
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 17.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 16
1-29-4
3ag
Me
O
PPM
0.0966
1.2730
1.5744
2.0003
2.3994
2.6349
3.02
1.00
0.77
2.93
0.60
1.96
7.63

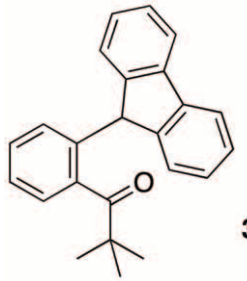
1-24-9


3ag
Me
O

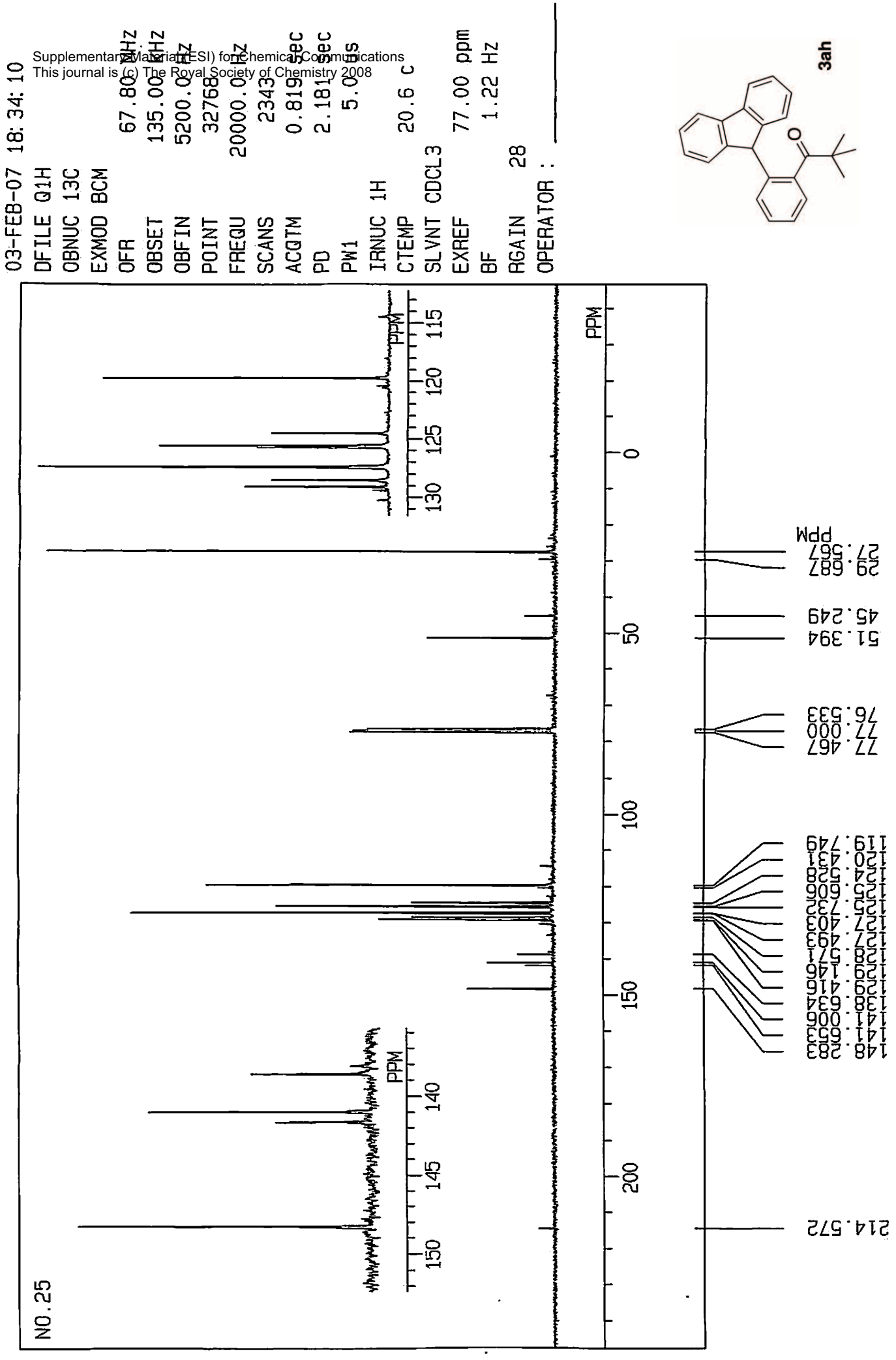
DFILE _DEFAULT.ALS
COMNT
DATIM Tue Jul 10 18:27:48 2007
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18306.64 Hz
SCANS 4034
ACQTM 1.7900 sec
PD 1.2100 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
PPM
21.2505
26.0121
49.9353
67.2876
76.5312
77.0000
77.1974
77.4688
114.4432
119.7969
119.9039
120.5782
125.4961
126.0882
127.2313
127.2806
127.3793
129.0899
129.1804
129.2708
129.3942
129.4189
129.4846
131.1376
131.3844
131.7544
131.8120
132.0094
138.4240
139.2052
140.5375
141.1214
148.6380
200.6950

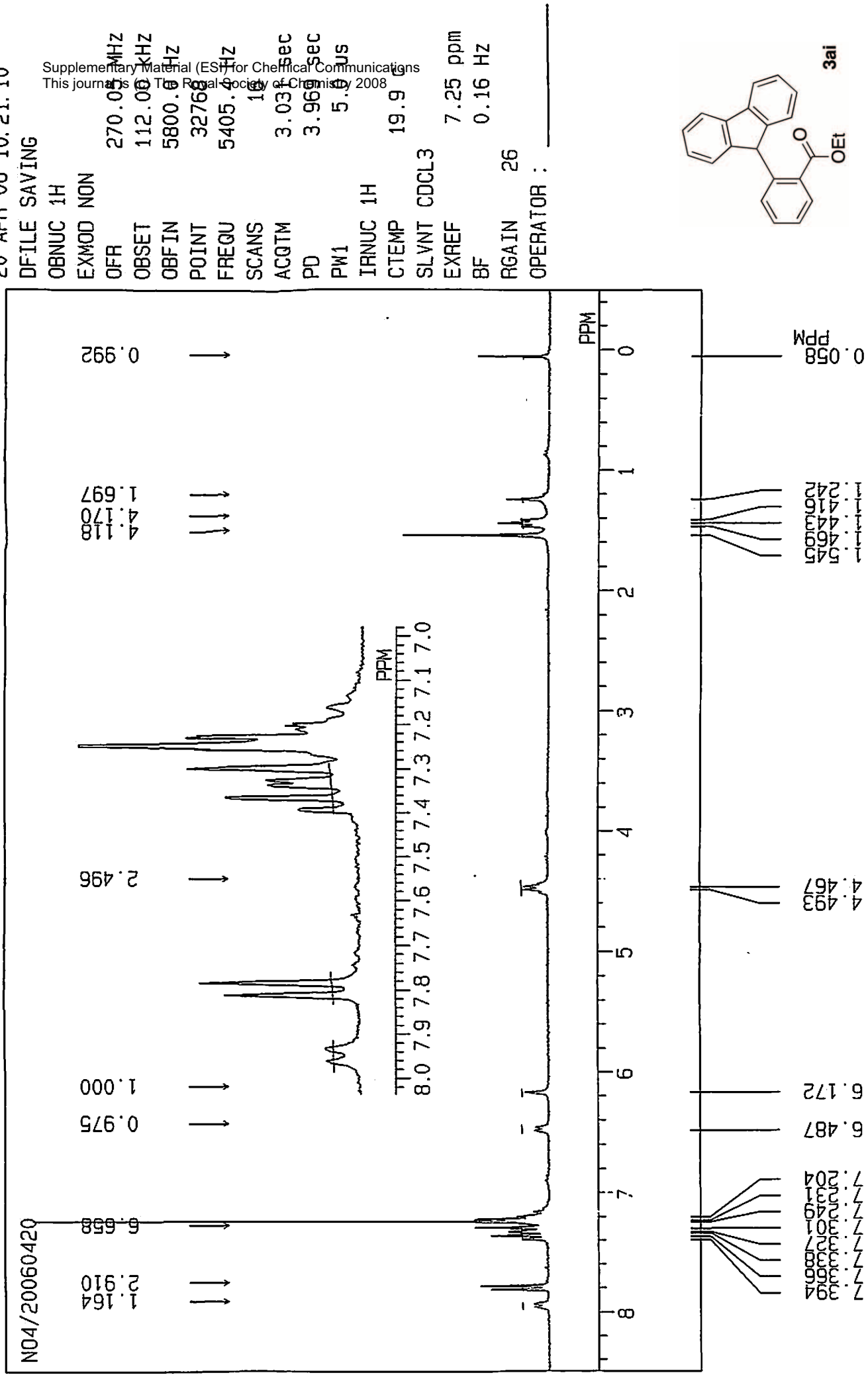


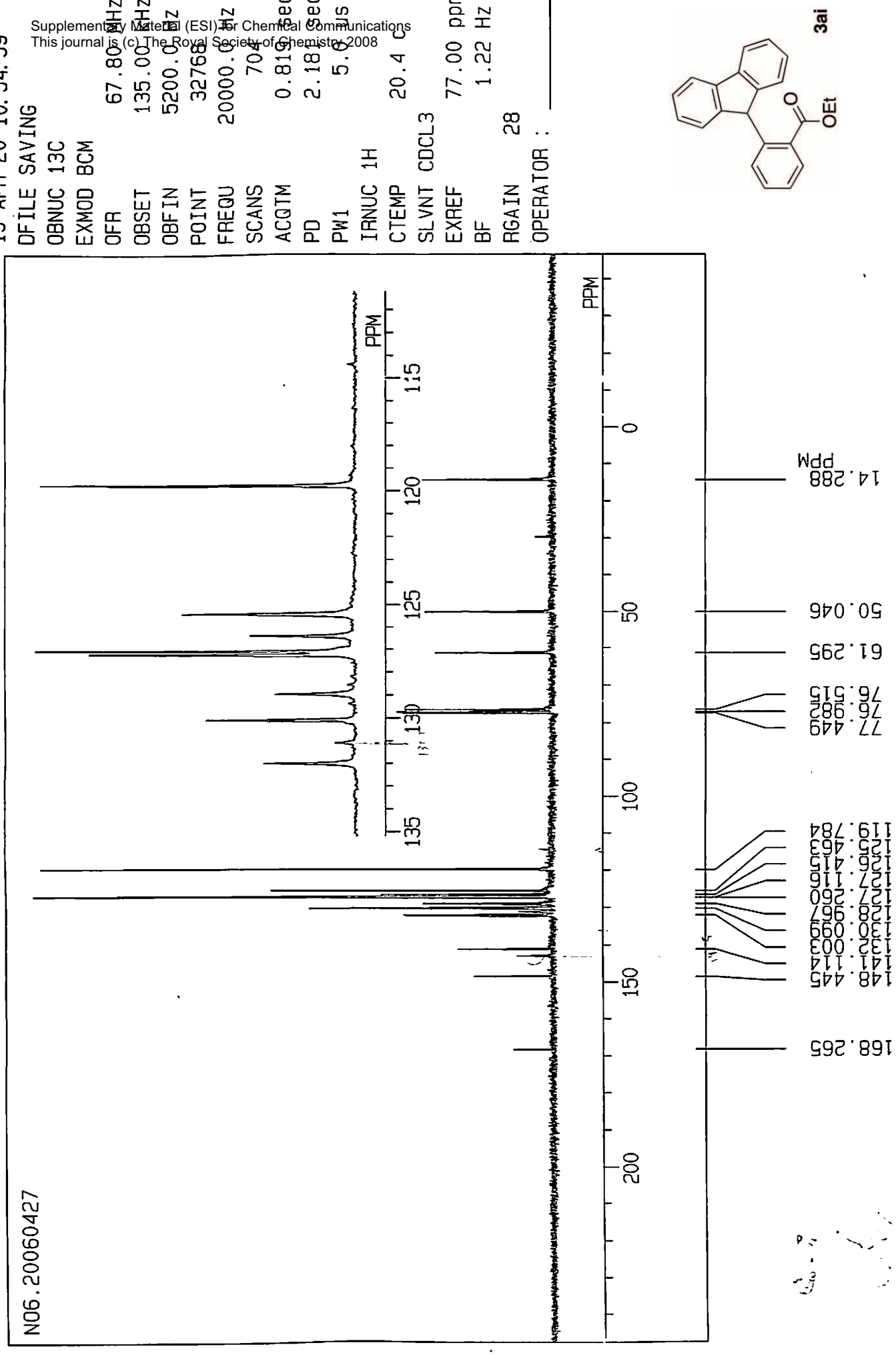
20-JUN-06 17: 43: 49
DFILE SAVING
OBNUC 1H
EXMOD NON
OFR 270.0[illegible] MHz
OBSET 112.0[illegible] kHz
OBFIN 5800.[illegible] Hz
POINT 32768
FREQU 5405.4[illegible] Hz
SCANS 1[illegible]
ACQTM 3.03[illegible] sec
PD 3.96[illegible] sec
PW1 5.[illegible] us
IRNUC 1H
CTEMP 19.0 C
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 25
OPERATOR : _____

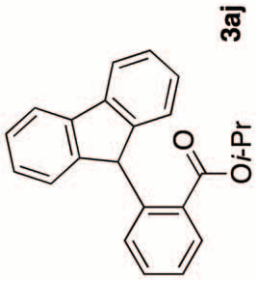


3ah

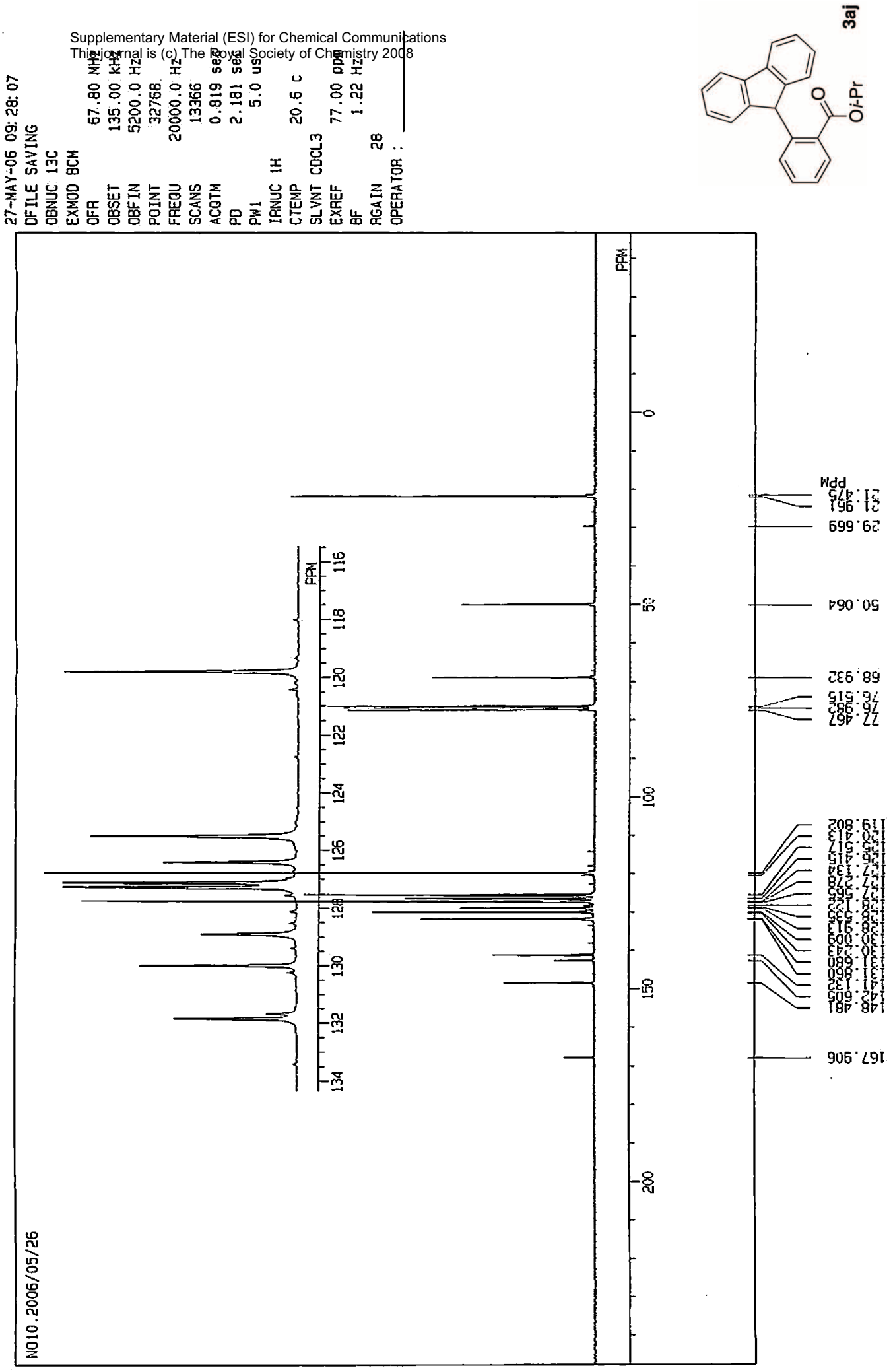

03-FEB-07 18:34:10
DFILE Q1H
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 MHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 20000.00 Hz
SCANS 2343
ACQTM 0.8192 sec
PD 2.1810 sec
PW1 5.00 us
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 28
OPERATOR :
3ah
NO.25
PPM
214.572
148.283
141.653
141.006
138.634
138.416
129.146
128.571
127.493
127.403
125.733
125.606
124.528
120.431
119.749
77.467
77.000
76.533
51.394
45.249
29.687
27.567


20-APR-06 10:21:10
DFILE SAVING
OBNUC 1H
EXMOD NON
OFR 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.6 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.03 sec
PD 3.96 sec
PW1 5.0 us
IRNUC 1H
CTEMP 19.9 C
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.16 Hz
RGAIN 26
OPERATOR :
3ai
OEt
O
N04/20060420
PPM
0.992
1.697
4.118
4.170
2.496
1.000
0.975
6.658
2.164
2.910
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
0.058
1.245
1.410
1.444
1.469
1.545
4.467
4.493
6.172
6.487
7.204
7.233
7.249
7.301
7.327
7.338
7.365
7.394

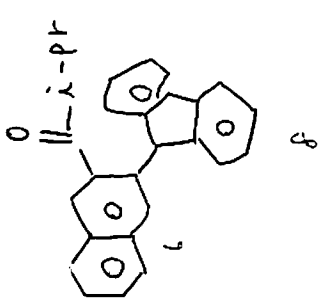

13-APR-20 10:54:39
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 MHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 704
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.4 C
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 28
OPERATOR :
3ai
OEt
O
N06.20060427
PPM
14.288
50.046
61.295
76.515
76.980
77.449
119.784
125.465
125.615
127.116
127.260
128.967
130.099
132.003
141.114
148.445
168.265

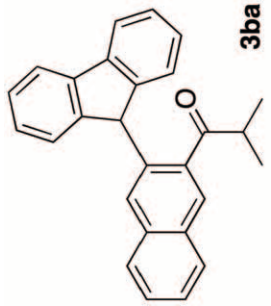

3aj
O
O-i-Pr

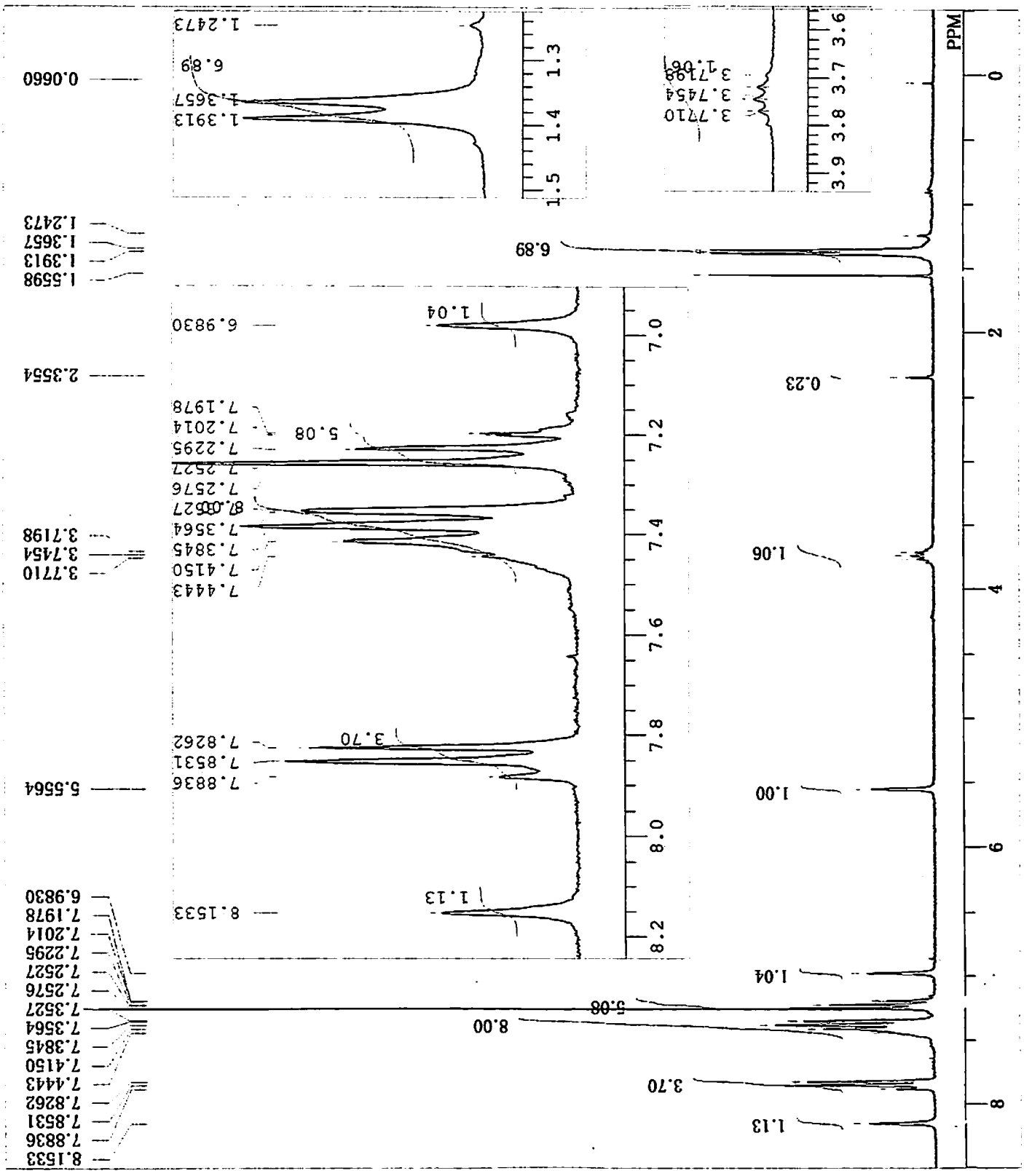
02-FEB-07 14:05:36
OFILE 01H
OBNUC 1H
EXMOD NOM
OFR 270.05 MHz
OBSET 112.00 kHz
OBFIN 5800.0 Hz
POINT 32768
FREQU 5405.4 Hz
SCANS 16
ACQTM 3.031 sec
PD 3.959 sec
PW1 5.0 us
TNUC 1H
CTEMP 18.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.16 Hz
RGAIN 14
OPERATOR :
6.604
1.003
1.000
1.029
4.380
4.365
4.350
1.467
0.826
0.812
0.797
PPM

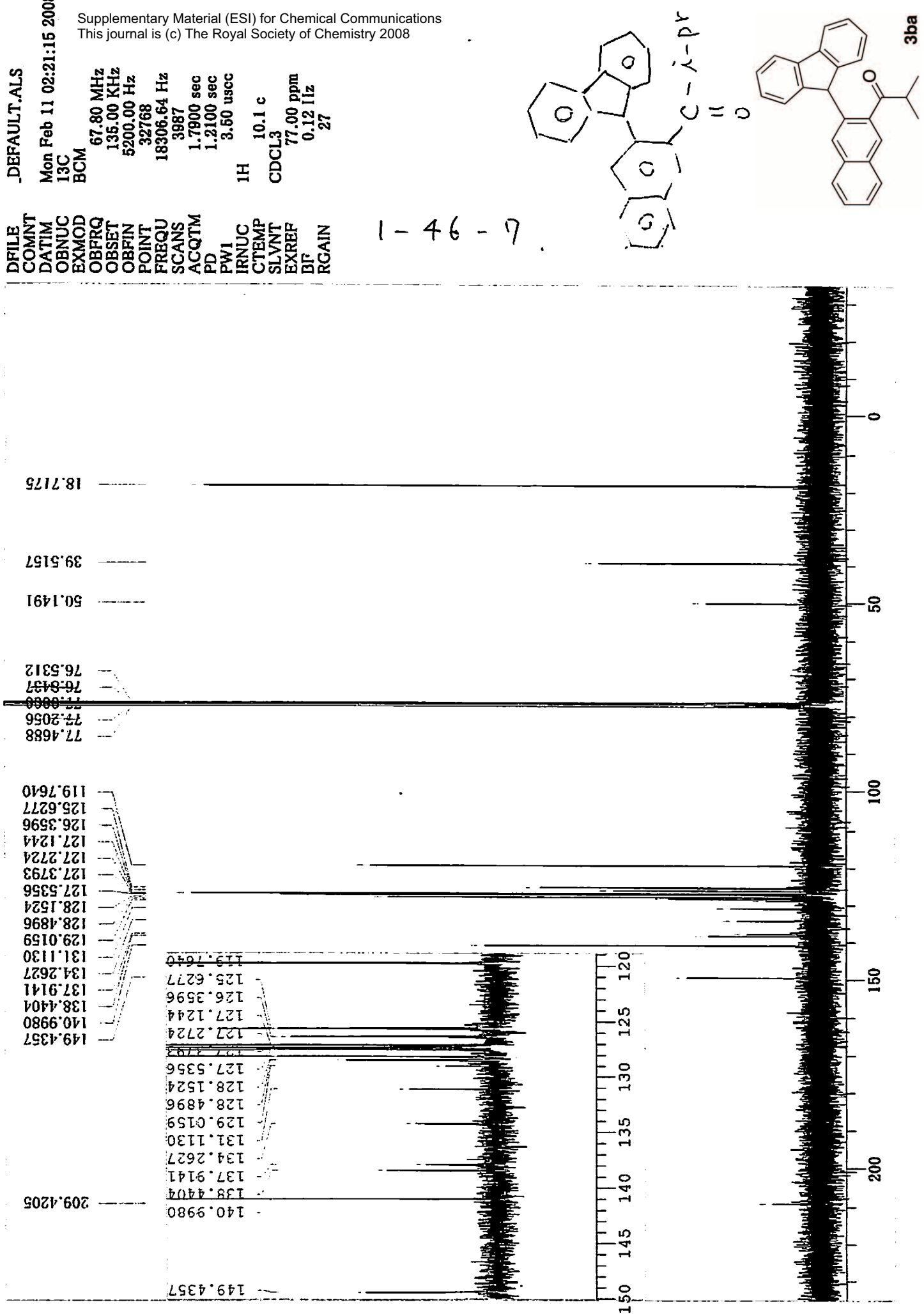

3aj
O
O-i-Pr
27-MAY-06 09:28:07
DFILE SAVING
OBNUC 13C
EXMOD BCM
OFR 67.80 MHz
OBSET 135.00 kHz
OBFIN 5200.0 Hz
POINT 32768
FREQU 20000.0 Hz
SCANS 13366
ACQTM 0.819 sec
PD 2.181 sec
PW1 5.0 us
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.22 Hz
RGAIN 28
OPERATOR :
NO10.2006/05/26
PPM
PPM
29.669
50.064
68.932
77.467
167.906

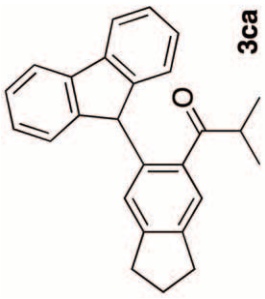
1-46-6




3ba

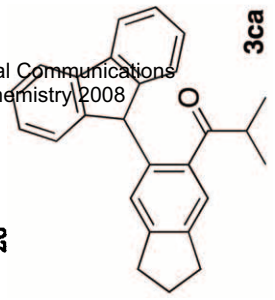

_DEFAULT.ALS
Thu Feb 07 16:33:22 2008
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
8
3.0331 sec
3.9670 sec
5.40 usec
1H
12.4 c
CDCL3
7.26 ppm
0.12 Hz
23
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM


_DEFAULT.ALS
Mon Feb 11 02:21:15 2008
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
3987
1.7900 sec
1.2100 sec
3.50 usec
1H
10.1 c
CDCL3
77.00 ppm
0.12 Hz
27
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
1-46-7
3ba
18.7175
39.5157
50.1491
76.5312
76.8437
77.2056
77.4688
119.7640
125.6277
126.3596
127.1244
127.2724
127.3793
127.5356
128.1524
128.4896
129.0159
131.1130
134.2627
137.9141
138.4404
140.9980
149.4357
209.4205

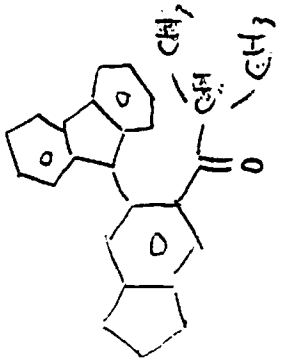

3ca
O

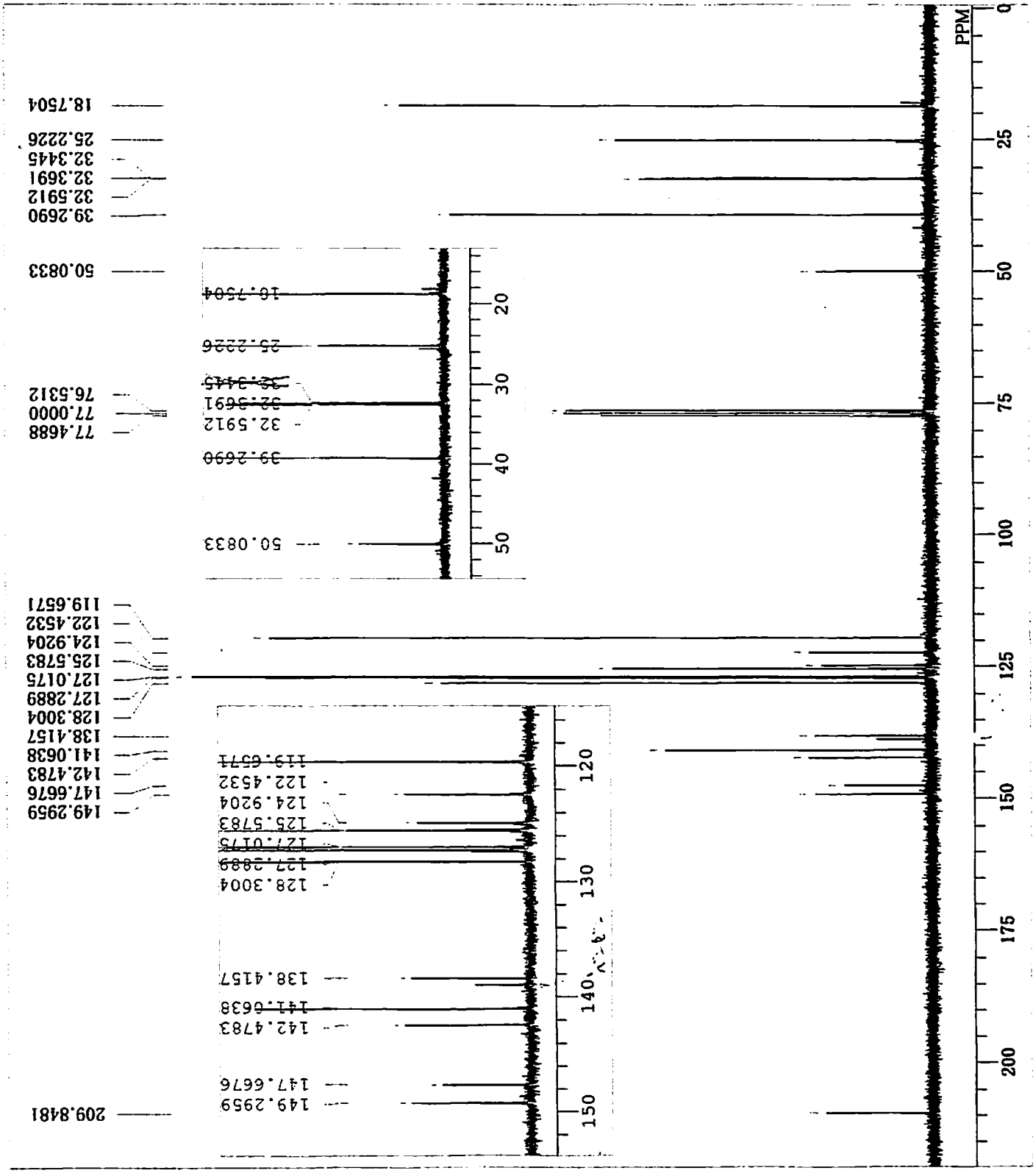
2-13-4.
(60-4)

_DEFAULT.ALS
Sat Sep 08 15:56:38 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
17.3 c
CDCL3
7.26 ppm
0.12 Hz
16
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
2.32
2.26
2.64
1.08
1.03
1.00
2.71
1.21
2.60
5.4563
6.3203
8.03

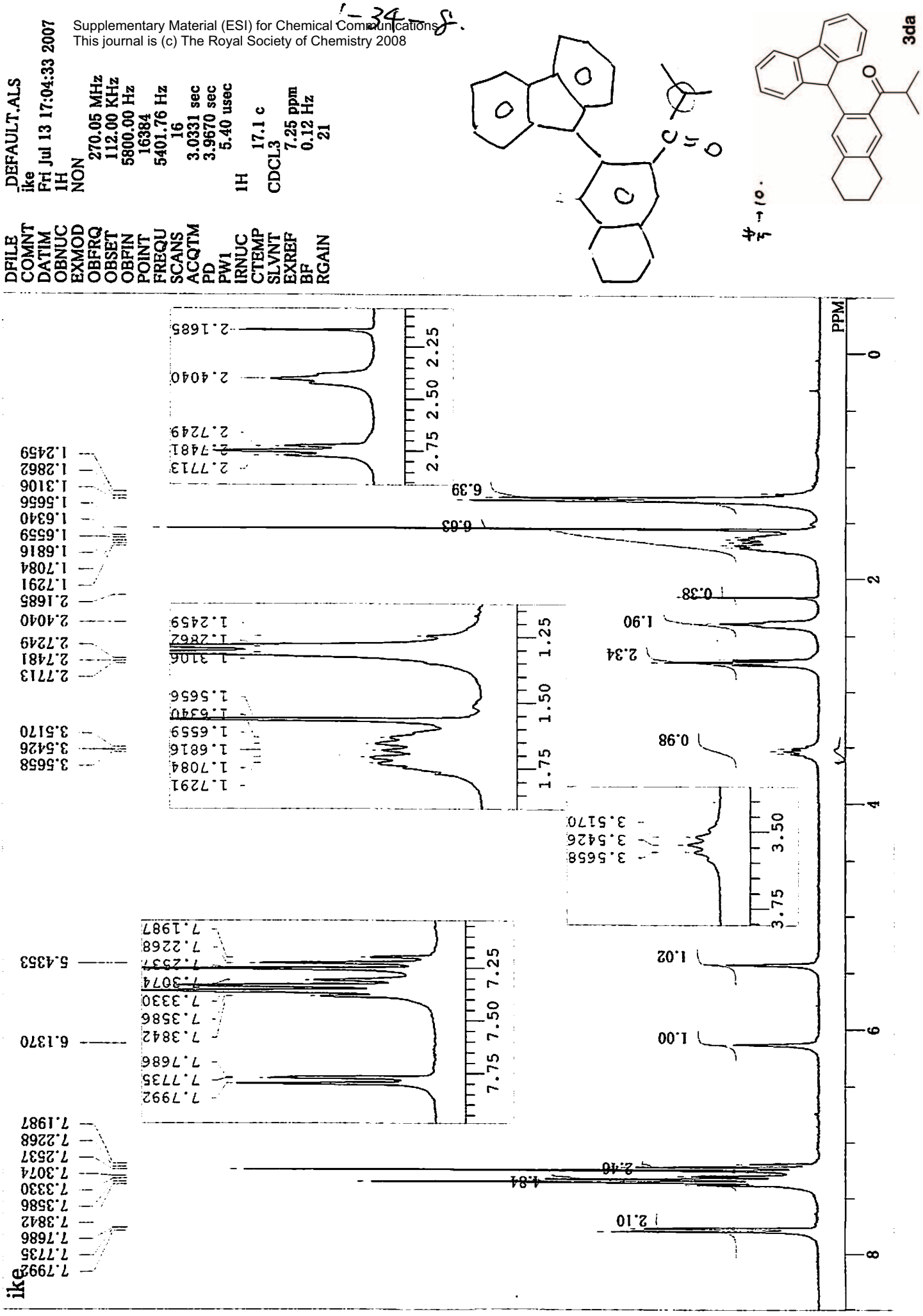

3ca
O

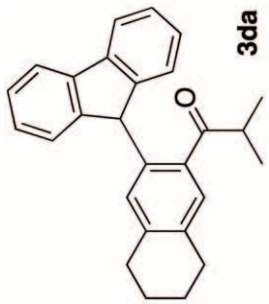
2-13-5.


CH3
CH3
O


DEFAULT.ALS
Sat Sep 08 16:55:59 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
1088
1.7900 sec
1.2100 sec
3.50 usec
1H
18.8 c
CDCL3
77.00 ppm
0.12 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
PREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
18.7504
25.2226
32.3445
32.3691
32.5912
39.2690
50.0833
76.5312
77.0000
77.4688
119.6571
122.4532
124.9204
125.5783
127.0175
127.2889
128.3004
138.4157
141.0638
142.4783
147.6676
149.2959
209.8481

1-34-S.

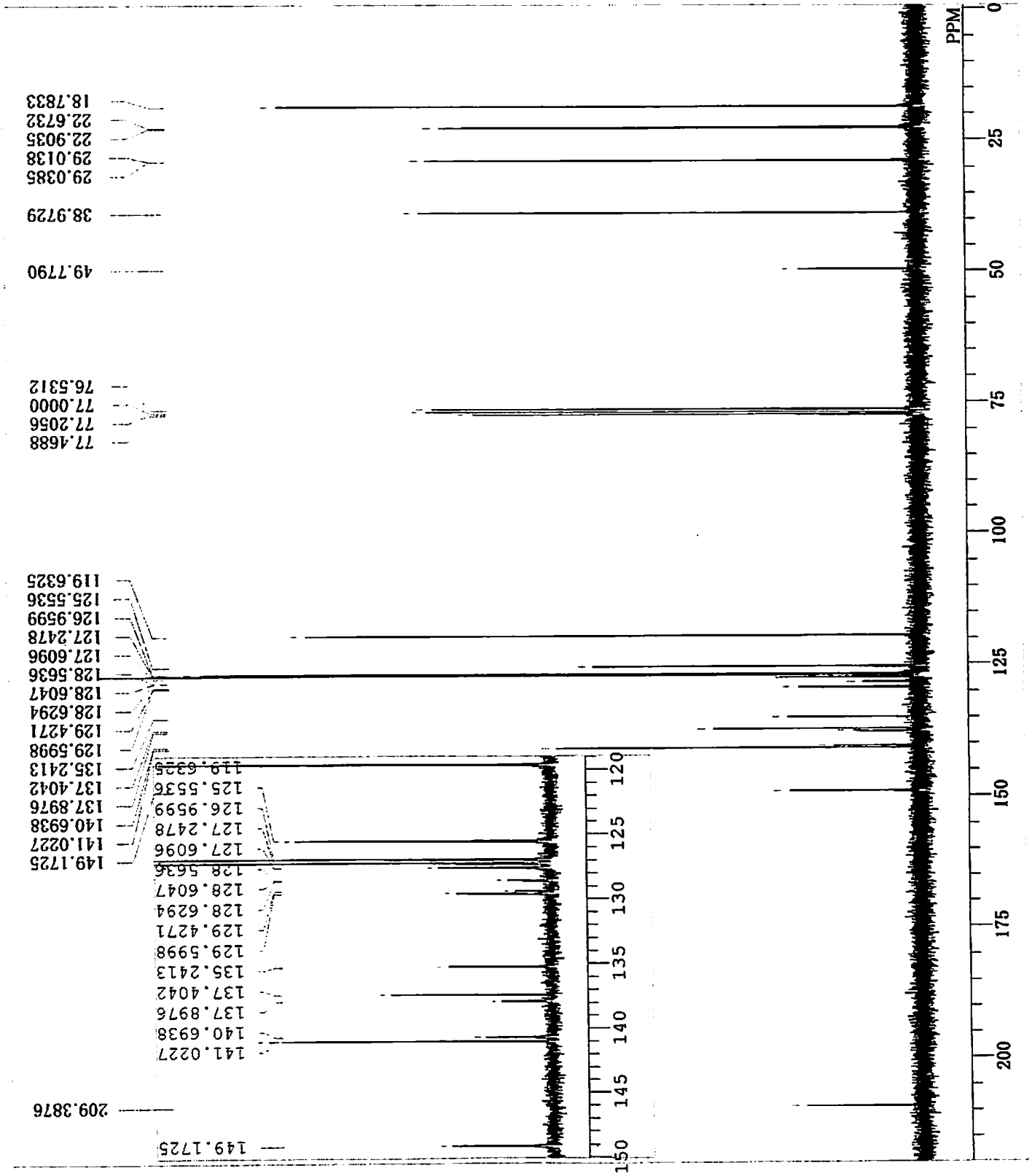

DFILE DEFAULT.ALS
COMNT ike
DATIM Fri Jul 13 17:04:33 2007
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 16
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 17.1 c
SLVNT CDCL3
EXREF 7.25 ppm
BF 0.12 Hz
RGAIN 21
3da
PPM
ike

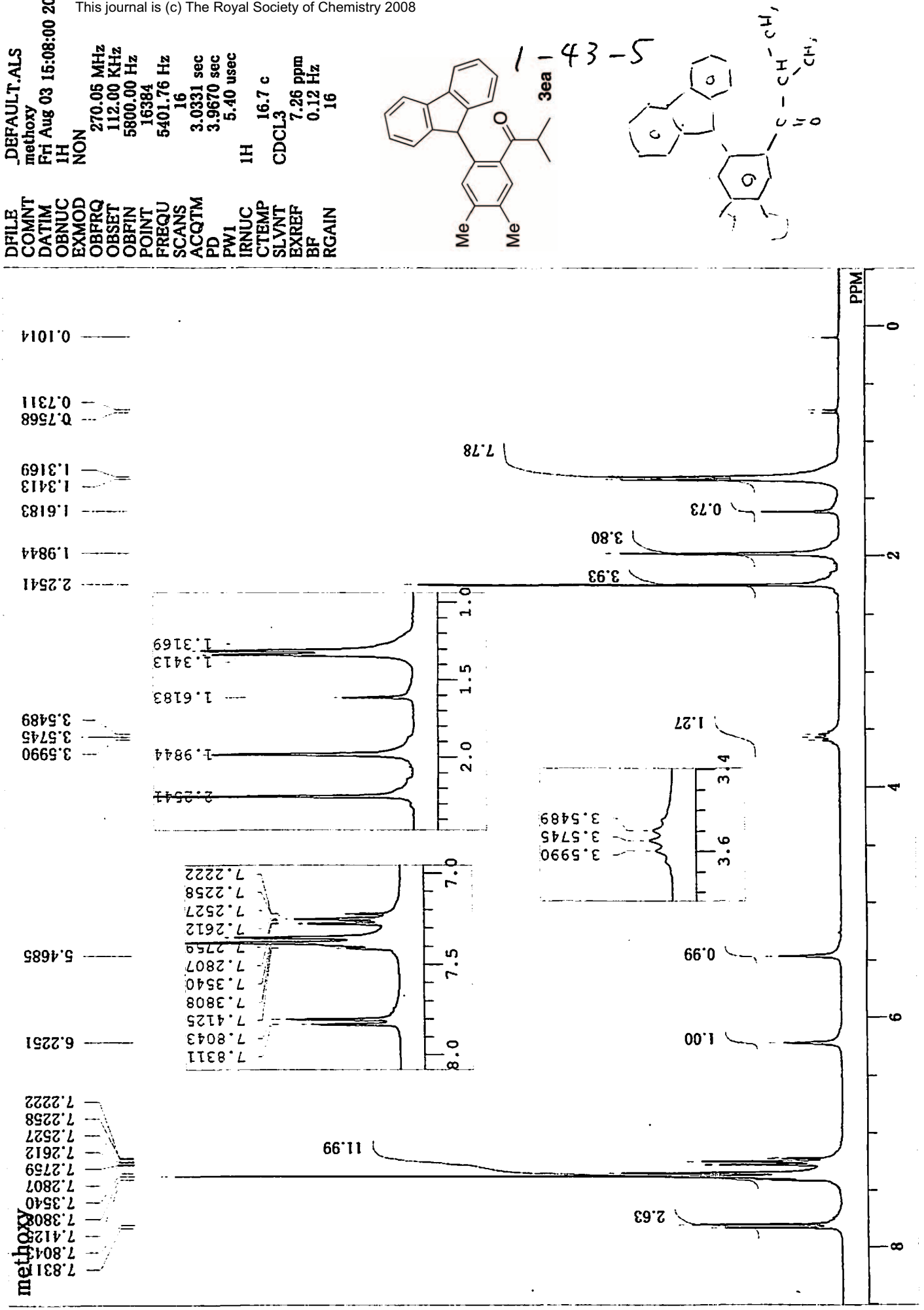


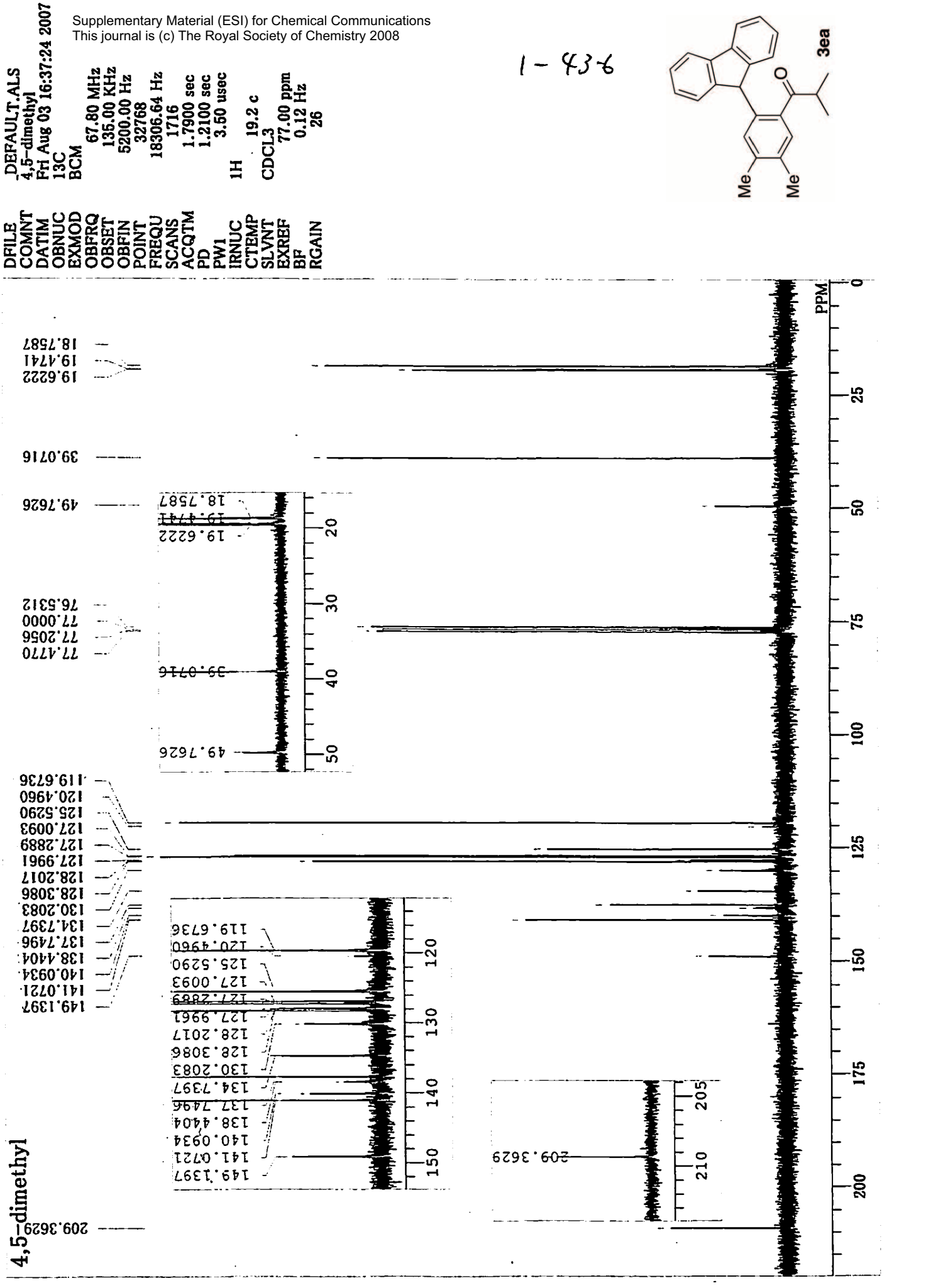
_DEFAULT.ALS
Fri Jan 25 17:52:33 2008
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
774
1.7900 sec
1.2100 sec
3.50 usec
1H
13.6 c
CDCL3
77.00 ppm
0.12 Hz
26

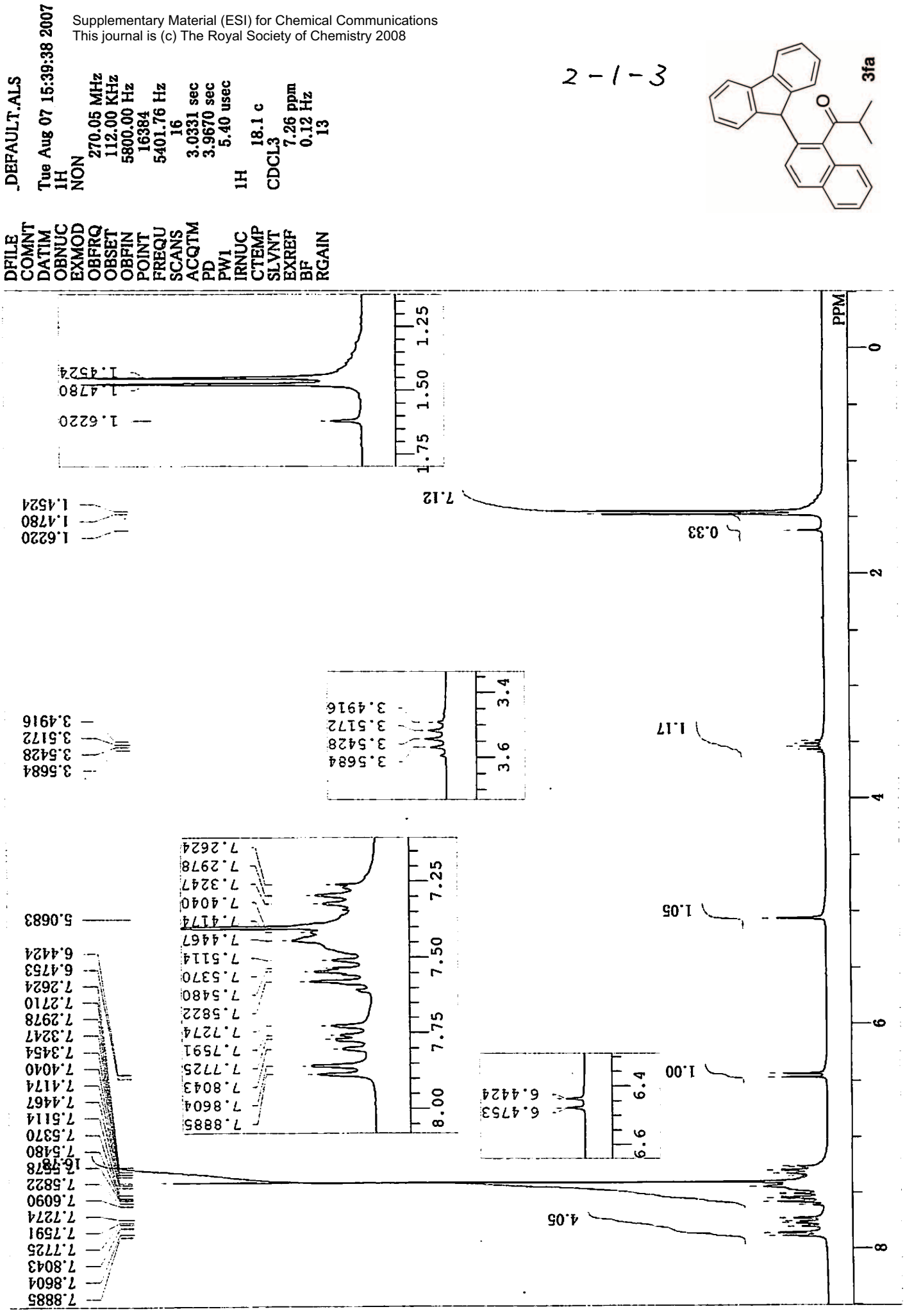
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

137-4.



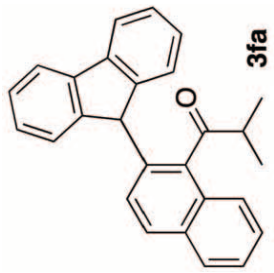

DFILE DEFAULT.ALS
COMNT methoxy
DATIM Fri Aug 03 16:08:00 2007
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 16
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.40 usec
IRNUC 1H
CTEMP 16.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 16
1-43-5
3ea
Me
Me
O
PPM
methoxy


DEFAULT.ALS
4,5-dimethyl
Fri Aug 03 16:37:24 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
1716
1.7900 sec
1.2100 sec
3.50 usec
1H
19.2 c
CDCL3
77.00 ppm
0.12 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
1-436
3ea
Me
Me
O
4,5-dimethyl
PPM

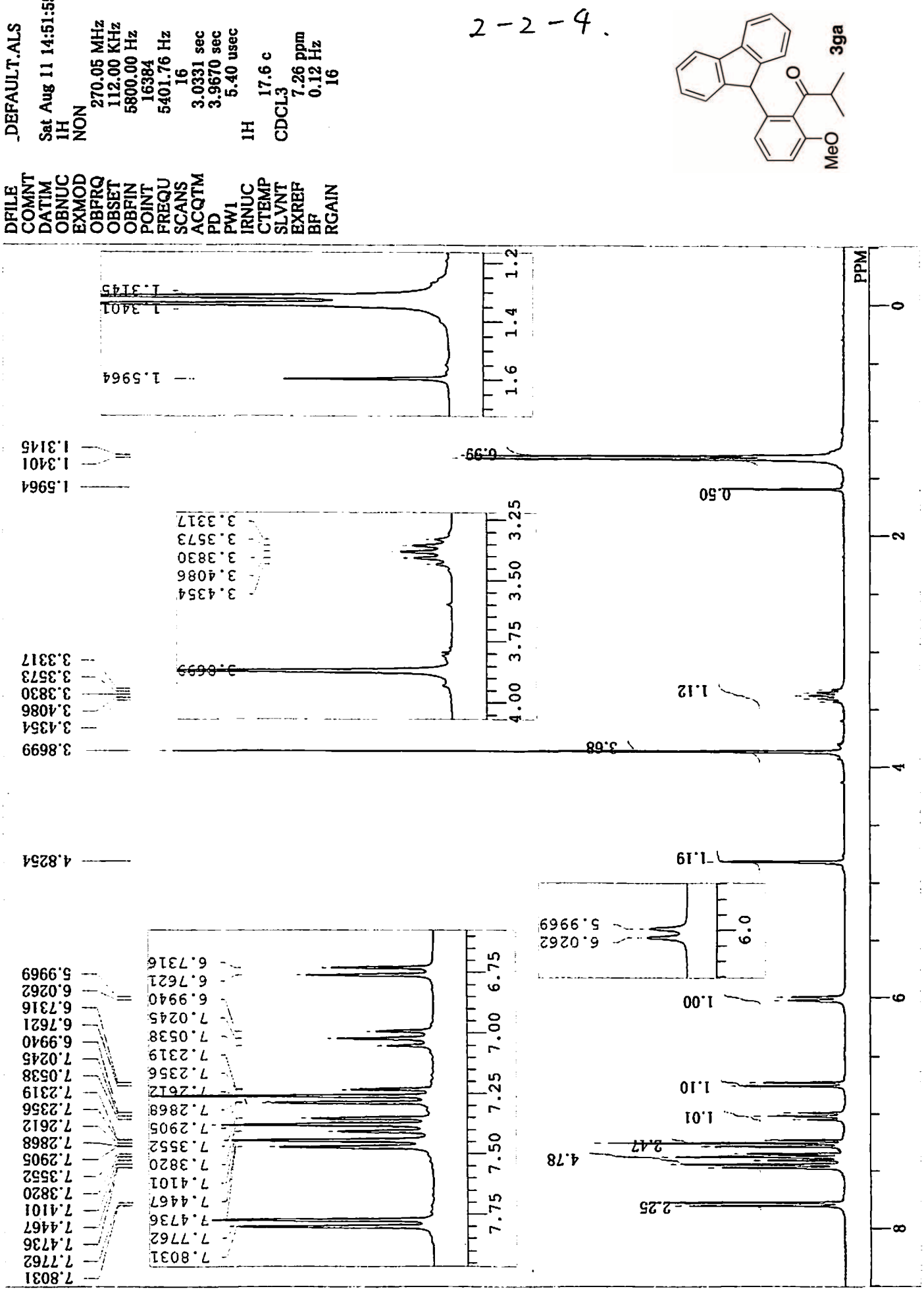

_DEFAULT.ALS
Tue Aug 07 15:39:38 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
18.1 c
CDCL3
7.26 ppm
0.12 Hz
13
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
2-1-3
3fa
O
PPM
0
2
4
6
8
7.12
0.33
1.17
1.05
1.00
4.05
5.0683
1.6220
1.4780
1.4524
3.4916
3.5172
3.5428
3.5684
7.2624
7.2978
7.3247
7.4040
7.4174
7.4467
7.5114
7.5370
7.5480
7.5822
7.6978
7.6090
7.7274
7.7591
7.7725
7.8043
7.8604
7.8885
6.4424
6.4753
7.2624
7.2710
7.2978
7.3247
7.3454
7.4040
7.4174
7.4467
7.5114
7.5370
7.5480
7.5822
7.6978
7.6090
7.7274
7.7591
7.7725
7.8043
7.8604
7.8885

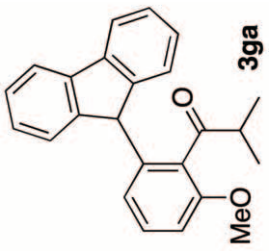
^{13}C NMR

2-1-4

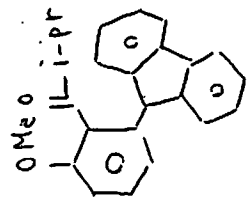

3fa

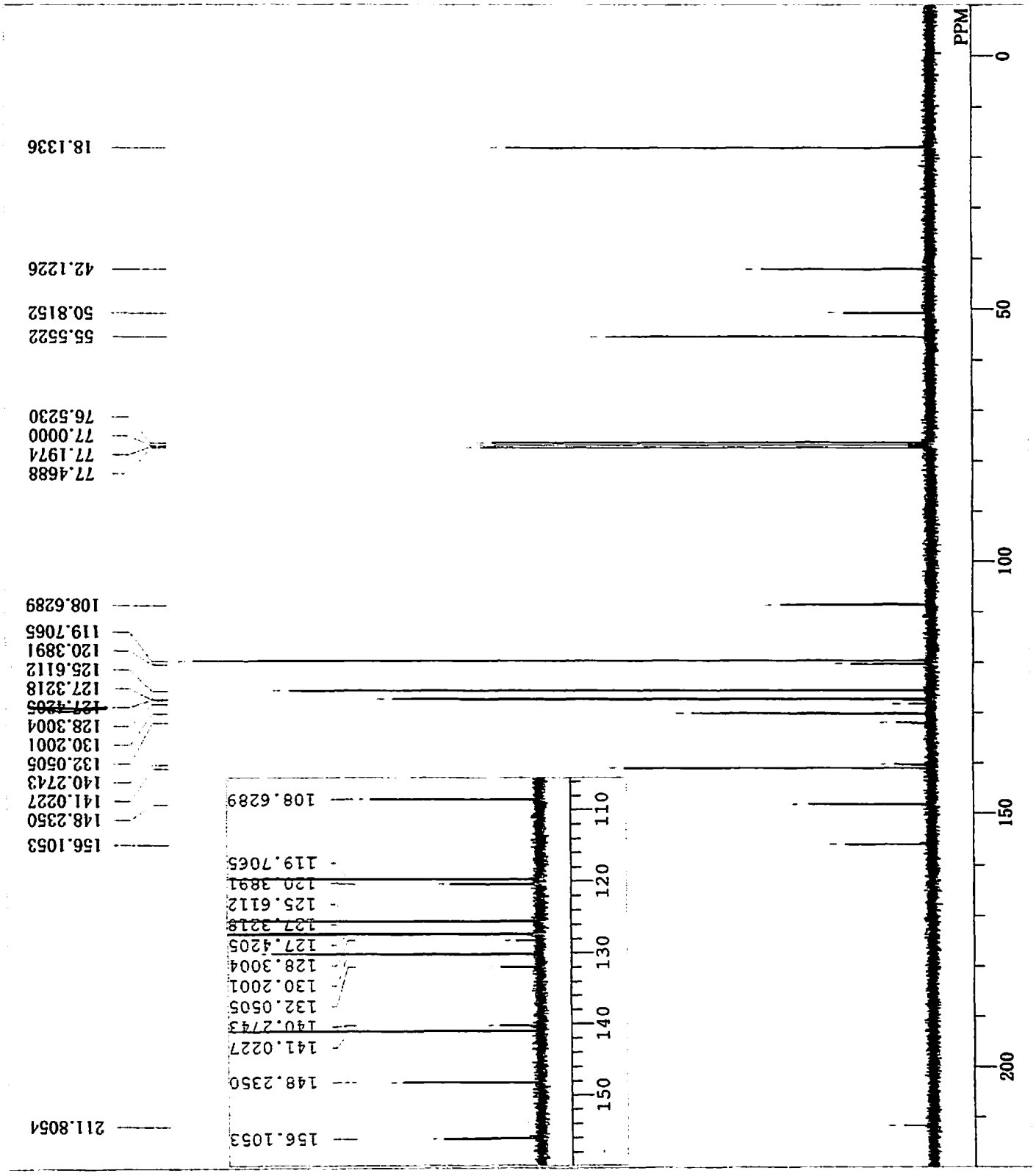
_DEFAULT.ALS
Tue Aug 07 17:44:03 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
2404
1.7900 sec
1.2100 sec
3.50 usec
1H
20.1 c
CDCL3
77.00 ppm
0.12 Hz
28
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
18.3475
43.7592
51.2429
51.3416
76.5312
77.0000
77.2056
77.4688
119.7887
119.9121
124.7806
125.3069
125.4221
125.4878
125.6194
125.8004
125.8744
125.9977
126.8283
127.5520
127.6260
128.3004
129.5340
129.6985
129.7314
132.2643
135.4716
139.2135
147.6758
213.6064

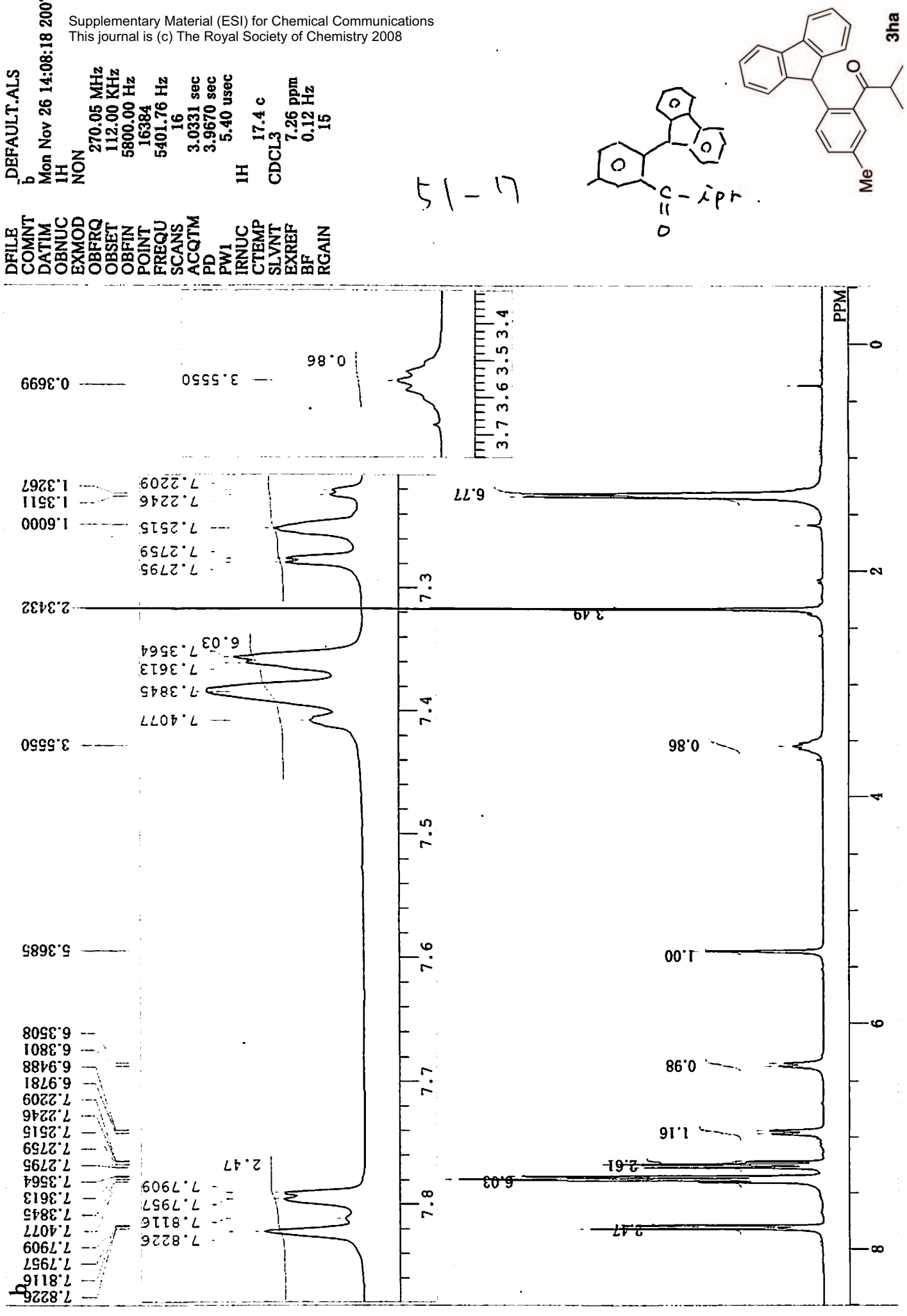

DEFAULT.ALS
Sat Aug 11 14:51:55 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
17.6 c
CDCL3
7.26 ppm
0.12 Hz
16
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
2-2-4.
3ga
O
MeO
PPM
0
2
4
6
8
1.2
1.4
1.6
3.25
3.50
3.75
4.00
6.75
7.00
7.25
7.50
7.75
6.0
1.5964
1.3401
1.3145
3.3317
3.3573
3.3830
3.4086
3.4354
3.8699
4.8254
5.9969
6.0262
6.7316
6.7621
6.9940
7.0245
7.0538
7.2319
7.2356
7.2612
7.2868
7.2905
7.3552
7.3820
7.4101
7.4467
7.4736
7.7762
7.8031
0.99
0.50
1.12
3.68
1.19
1.00
1.10
1.01
2.41
4.78
2.25

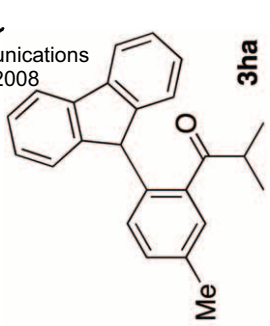

3ga
O
MeO

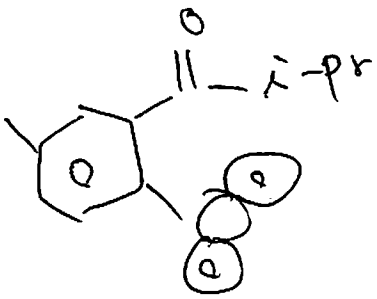
2-2-5


OMe
i-Pr

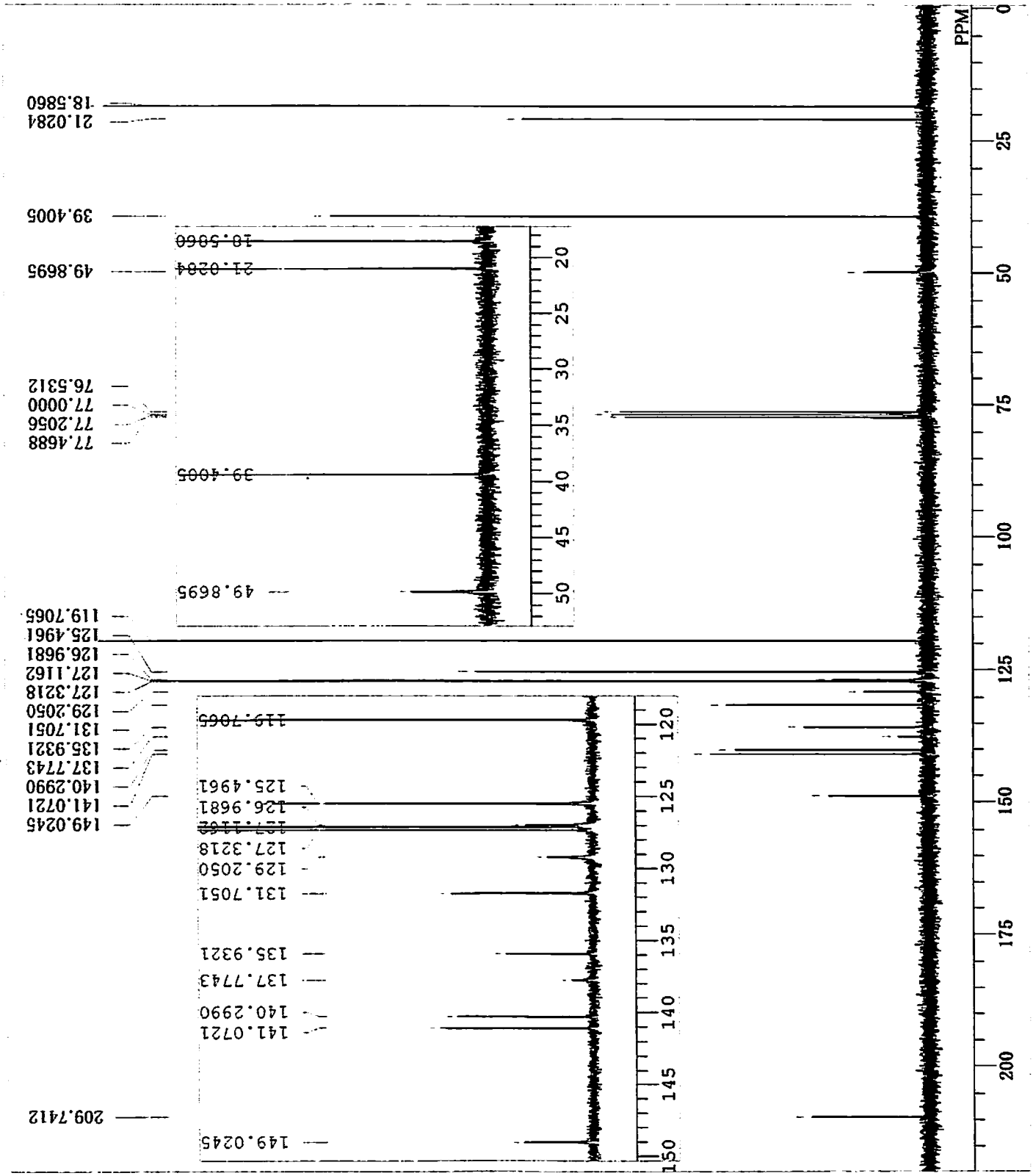

DEFAULT.ALS
Sat Aug 11 16:33:13 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
1919
1.7900 sec
1.2100 sec
3.50 usec
1H
19.7 c
CDCL3
77.00 ppm
0.12 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
18.1336
42.1226
50.8152
55.5522
76.5230
77.0000
77.1974
77.4688
108.6289
119.7065
120.3891
125.6112
127.3218
127.4205
128.3004
130.2001
132.0505
140.2743
141.0227
148.2350
156.1053
211.8054

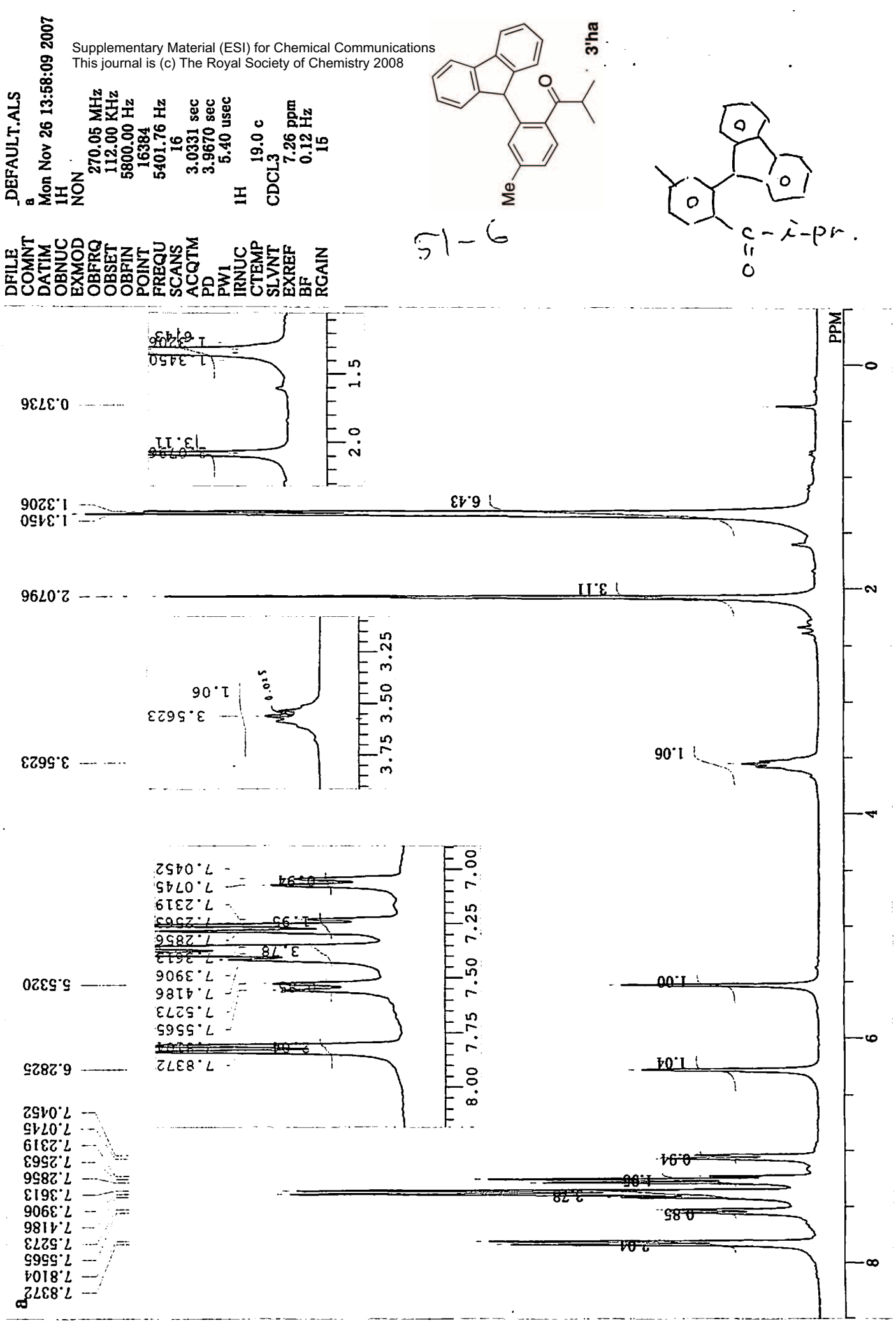

DEFAULT.ALS
b
Mon Nov 26 14:08:18 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
17.4 c
CDCL3
7.26 ppm
0.12 Hz
15
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
3ha
Me
O
PPM

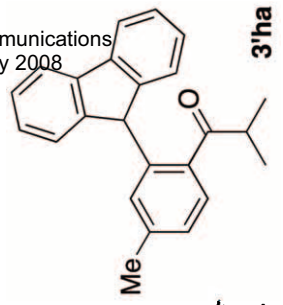

3ha
O
Me


O
i-Pr

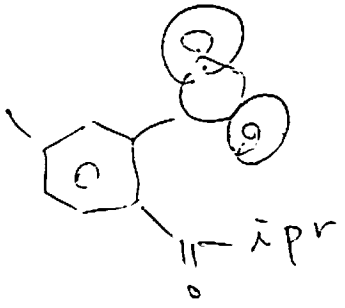
51-9


_DEFAULT.ALS
Tue Nov 27 14:57:00 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
638
1.7900 sec
1.2100 sec
3.50 usec
1H
21.4 c
CDCL3
77.00 ppm
0.12 Hz
27
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
18.5860
21.0284
39.4005
49.8695
76.5312
77.0000
77.2056
77.4688
119.7065
125.4961
126.9681
127.1162
127.3218
129.2050
131.7051
135.9321
137.7743
140.2990
141.0721
149.0245
209.7412


3'ha
Me
DEFAULT.ALS
a
Mon Nov 26 13:58:09 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
19.0 c
CDCL3
7.26 ppm
0.12 Hz
15
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
6.43
3.11
1.06
1.00
1.04
0.94
1.05
3.28
0.85
2.01
1.3450
1.3206
2.0796
3.5623
5.5320
6.2825
7.0452
7.0745
7.2319
7.2563
7.2856
7.3613
7.3906
7.4186
7.5273
7.5565
7.8104
7.8372



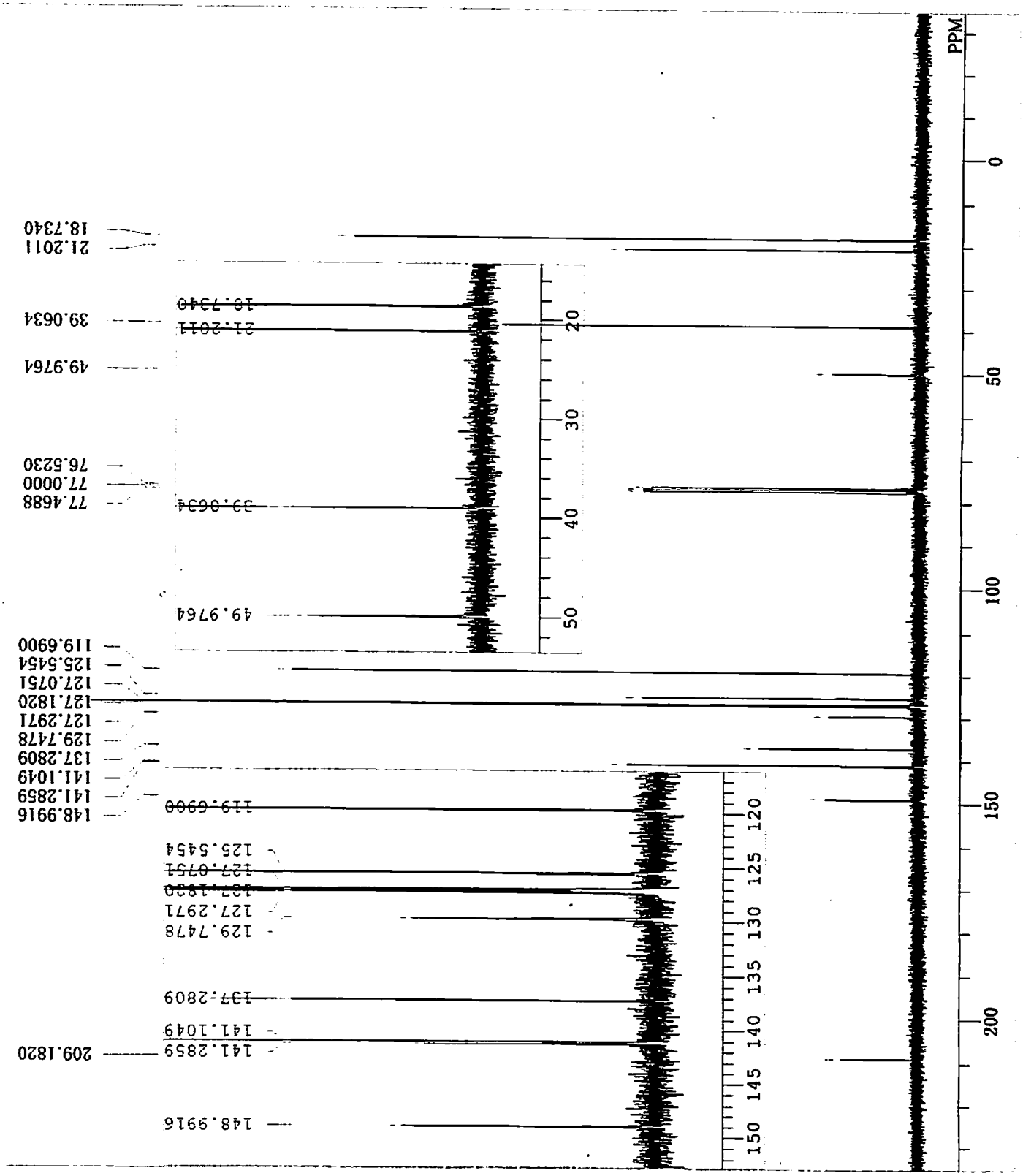
3'ha

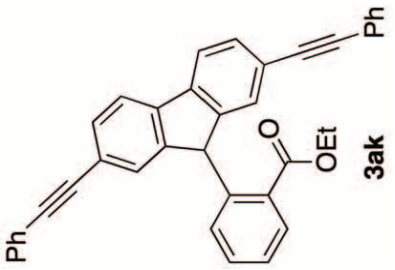


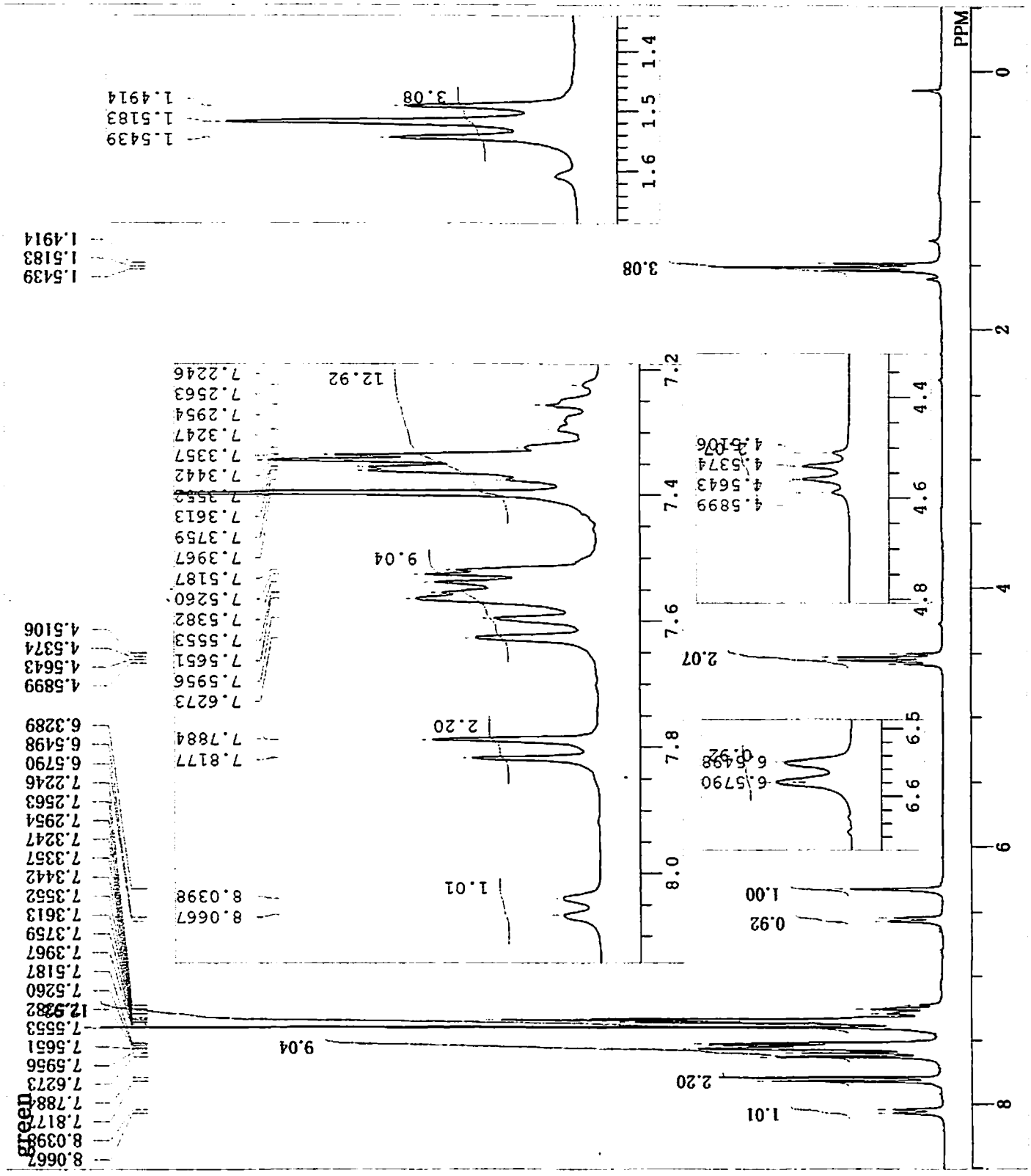
51-8

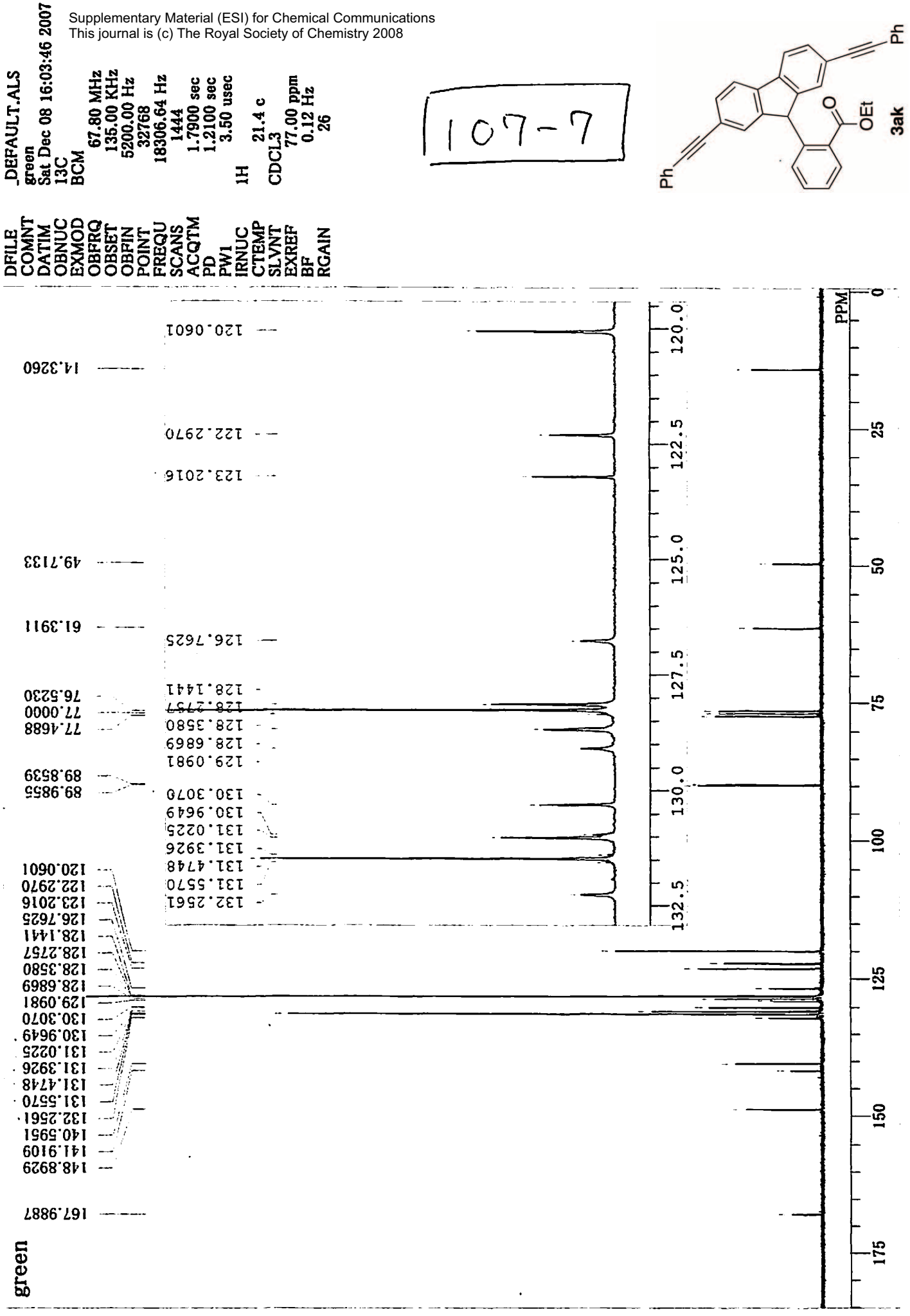
_DEFAULT.ALS
Mon Nov 26 18:49:46 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
846
1.7900 sec
1.2100 sec
3.50 usec
1H
20.9 c
CDCL3
77.00 ppm
0.12 Hz
28

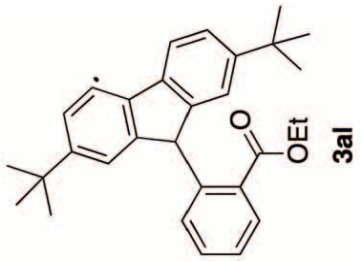
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN



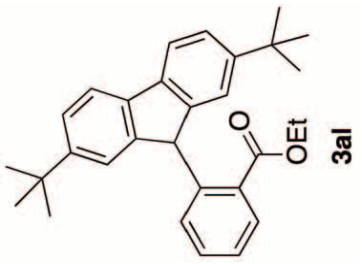

Ph
O
OEt
Ph
3ak


DEFAULT.ALS
green
Fri Dec 07 15:51:37 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
17.6 c
CDCL3
7.26 ppm
0.12 Hz
13
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM
green


DEFAULT.ALS
green
Sat Dec 08 16:03:46 2007
13C
BCM
67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
1444
1.7900 sec
1.2100 sec
3.50 usec
1H
21.4 c
CDCL3
77.00 ppm
0.12 Hz
26
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
107-7
Ph
O
OEt
Ph
3ak
PPM
green



3al

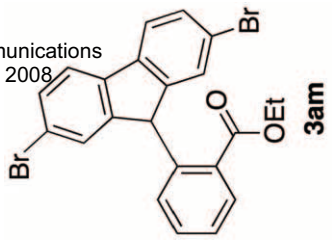


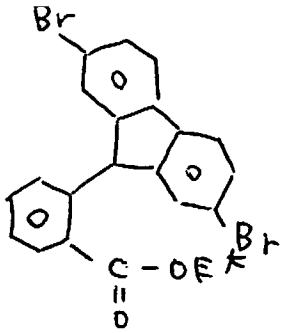
3al

2-3-5

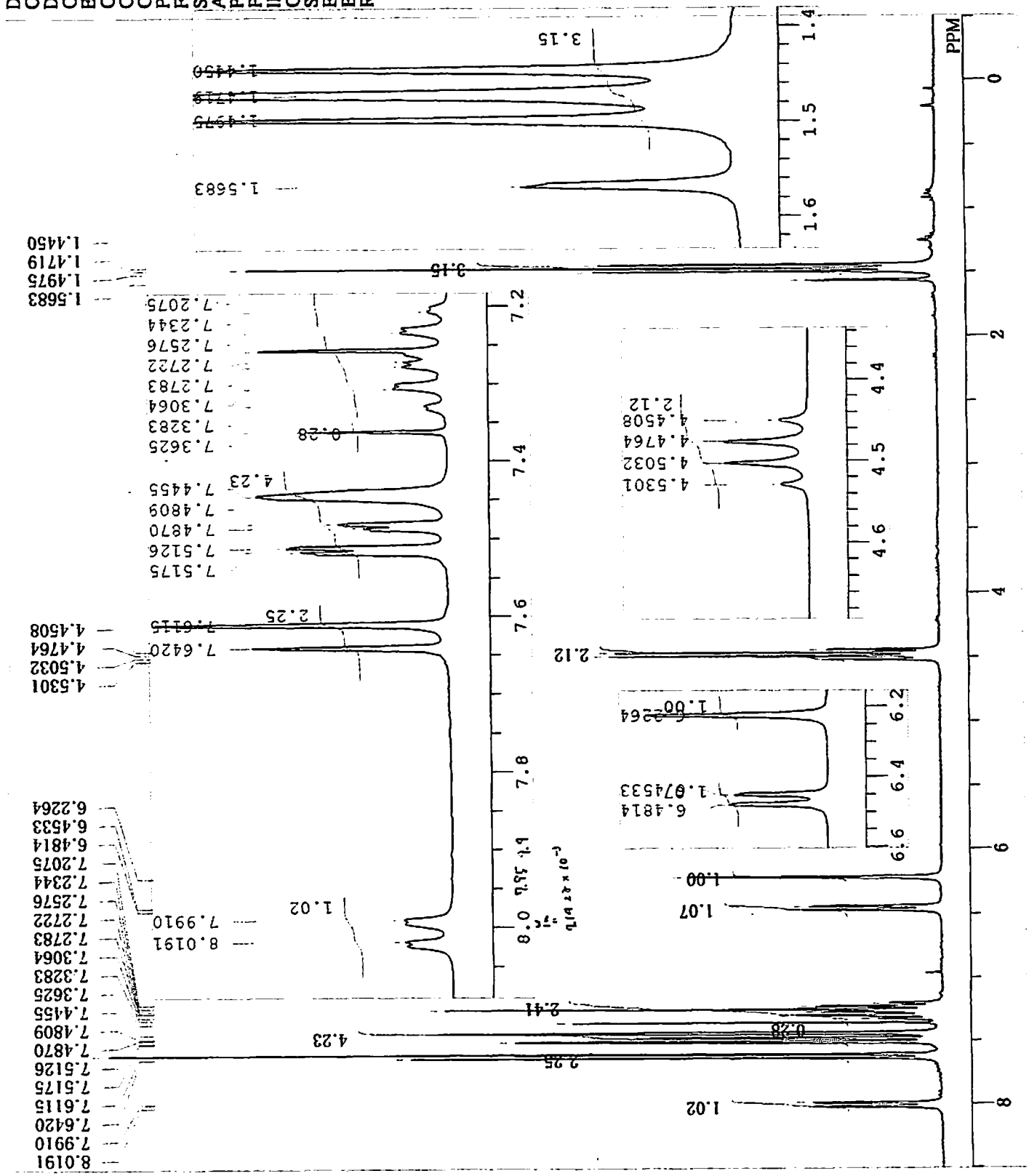
_DEFAULT.ALS
Thu Aug 23 19:21:10 2007
13C
BCM 67.80 MHz
135.00 KHz
5200.00 Hz
32768
18306.64 Hz
1280
1.7900 sec
1.2100 sec
3.50 usec
1H 19.5 c
CDCL3 77.00 ppm
0.12 Hz
26

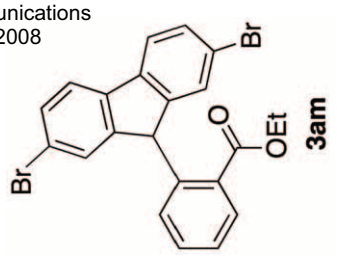
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

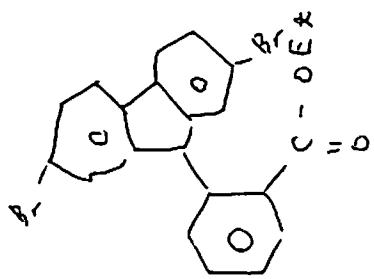

Br
Br
O
OEt
3am


Br
Br
O
O
O

1-31-10.


_DEFAULT.ALS
Thu Nov 29 11:00:52 2007
1H
NON
270.05 MHz
112.00 KHz
5800.00 Hz
16384
5401.76 Hz
16
3.0331 sec
3.9670 sec
5.40 usec
1H
18.7 c
CDCL3
7.26 ppm
0.12 Hz
19
DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN
PPM





1-31-11

DFILE _DEFAULT.ALS
COMNT
DATIM Thu Nov 29 11:55:47 2007
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQU 18306.64 Hz
SCANS 989
ACQTM 1.7900 sec
PD 1.2100 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26