Practical Synthesis of Aromatic Nitriles via Gallium-catalysed Electrophilic Cyanation of Aromatic C–H Bonds

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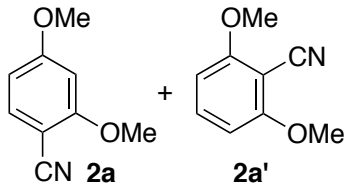
Supporting Information

1. General. NMR spectra were recorded on JEOL EX-400 spectrometer (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR). Chemical shifts are reported in δ ppm referenced to $CDCl_3$ (δ 7.26 for ^{1}H NMR and δ 77.00 for ^{13}C NMR). Melting points (mp) are uncorrected. IR spectra were recorded with an FT-IR spectrometer (JASCO FT/IR-460 Plus). Elemental analyses were performed at the Center for Organic Elemental Microanalysis (Kyoto University). High-resolution mass spectra (HRMS) were measured with JEOL JMX-SX 102A spectrometer. Dry 1,2-dichloroethane was purchased from Aldrich. 1,3-Dimethoxy-2,4,6-trideuteriobenzene (**1a**-d_3) [1] and 2,2'-dimethoxydiphenylacetylene (**7**) [2] were prepared according to the reported procedures. All other materials were purchased and used without further purification.

2. Gallium-catalysed cyanation reactions (Tables 1–3, eq. 2).

2,4-Dimethoxybenzonitrile (**2a**) [CAS 4107-65-7]
2,6-Dimethoxybenzonitrile (**2a'**) [CAS 16932-49-3]



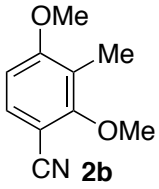
A typical procedure: in a 20-mL Schlenk flask equipped with a J. Young valve, $GaCl_3$ (7.0 mg, 40 μmol) was dissolved in 1,2-dichloroethane (1.6 mL) under N_2. Then BrCN[3] (50.8 mg, 0.48 mmol) and arene **1a** (0.40 mmol) was subsequently added to the solution. After stirring at 120 °C for 1 h, the reaction mixture was quenched

(1) S. T. Chadwick, R. A. Rennels, J. L. Rutherford, D. B. Collum, *J. Am. Chem. Soc.* 2000, **122**, 8640.
(2) M. J. Mio, L. C. Kopel, J. B. Braun, T. L. Gadzikwa, K. L. Hull, R. G. Brisbois, C. J. Markworth, P. A. Grieco, *Org. Lett.* 2002, **4**, 3199.
(3) Cyanogen bromide (mp 52 °C) is a commercially available reagent and stable to the air and the moisture. However, the handling of cyanogen bromide requires special care to avoid inhalation of hydrogen cyanide, and all operations should be carried out in a well-fumed hood.

with sat. $NaHCO_3$ aq and extracted with EtOAc three times. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated under vacuum. The residue was subjected to column chromatography on silica gel (hexane/EtOAc = 20/1) to afford 53.2 mg of aromatic nitriles **2a** and **2a'** (0.33 mmol, 83% yield, **2a**/**2a'** = 95:5) as a white solid. Mp 93–94 °C.

(Major isomer **2a**) ^{1}H NMR ($CDCl_3$): δ 3.86 (s, 3H), 3.91 (s, 3H), 6.46 (d, *J* = 2.0 Hz, 1H), 6.51 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 55.6, 55.9, 93.8, 98.4, 105.8, 116.8, 134.8, 162.8, 164.6.

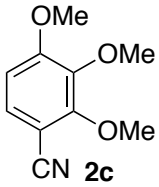
2,4-Dimethoxy-3-methylbenzonitrile (**2b**) [CAS 81574-51-8]



2b

A pale yellow solid (58.0 mg, 0.33 mmol, 83% yield). Mp 48–49 °C. ^{1}H NMR ($CDCl_3$): δ 2.13 (s, 3H), 3.88 (s, 3H), 3.95 (s, 3H), 6.66 (d, *J* = 8.8 Hz, 1H), 7.41 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 8.7, 55.9, 61.6, 98.1, 106.2, 117.3, 120.7, 131.9, 161.2, 162.4.

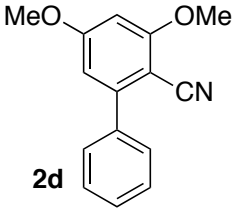
2,3,4-Trimethoxybenzonitrile (**2c**) [CAS 43020-38-8]



2c

A pale yellow solid (52.0 mg, 0.27 mmol, 68% yield). Mp 49–50 °C. ^{1}H NMR ($CDCl_3$): δ 3.87 (s, 3H), 3.92 (s, 3H), 4.06 (s, 3H), 6.68 (d, *J* = 8.8 Hz, 1H), 7.28 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 56.3, 61.1, 61.8, 99.2, 107.5, 116.5, 128.8, 141.9, 155.9, 158.0.

3,5-Dimethoxy-1,1'-biphenyl-2-carbonitrile (**2d**)

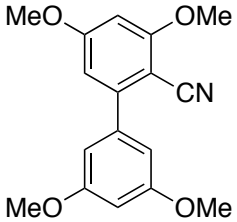


2d

A pale yellow solid (74.2 mg, 0.31mmol, 78% yield). Mp 74–75 °C. IR (KBr): 2216, 1601, 1573, 1349, 1206, 778 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 3.88 (s, 3H), 3.94 (s, 3H), 6.46 (d, *J* = 2.0 Hz, 1H), 6.55 (d, *J* = 2.0 Hz, 1H), 7.38–7.50 (m, 3H), 7.54 (d, *J* =

8.3 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 55.7, 56.1, 93.3, 96.9, 106.9, 116.4, 128.4, 128.5, 128.7, 138.2, 148.5, 163.6, 163.8. Elemental analysis calcd for $C_{15}H_{13}O_2N$, C: 75.30; H: 5.48; N: 5.85, found C: 75.18; H: 5.85; N: 5.62.

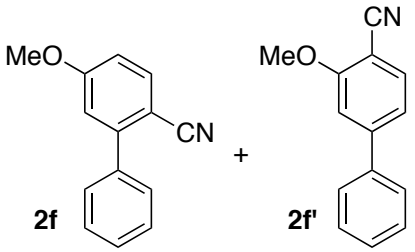
3,3',5,5'-Tetramethoxy-1,1'-biphenyl-2-carbonitrile (**2e**)



2e

A pale yellow solid (35.8 mg, 0.12 mmol, 30% yield). Mp 116–117 °C. IR (KBr): 2213, 1540, 1456, 1283, 1206, 823 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 3.84 (s, 6H), 3.88 (s, 3H), 3.94 (s, 3H), 6.46 (d, *J* = 2.4 Hz, 1H), 6.52 (t, *J* = 2.4 Hz, 1H), 6.56 (d, *J* = 2.0 Hz, 1H), 6.67 (d, *J* = 2.4 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 55.4, 55.7, 56.2, 93.4, 97.1, 100.9, 106.7, 106.8, 116.3, 140.1, 148.5, 160.7, 163.6, 163.7. HRMS (FAB) calcd for $C_{17}H_{18}NO_4$ $(M+H)^+$ 300.1236, found 300.1242. Elemental analysis calcd for $C_{17}H_{17}NO_4$, C: 68.21; H: 5.72; N: 4.68, found C: 67.93; H: 5.72; N: 4.39.

5-Methoxy-1,1'-biphenyl-2-carbonitrile (**2f**) [CAS 500309-63-7]
3-Methoxy-1,1'-biphenyl-4-carbonitrile (**2f'**)



2f **2f'**

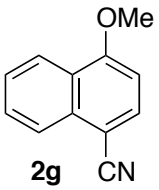
A pale yellow solid (50.2 mg, 0.24 mmol, 60% yield). Mp (a regioisomeric mixture) 74–75 °C. IR (KBr): 2224, 1498, 1294, 1024, 734 cm^{-1}. Two regioisomers were assigned by NMR spectra (**2f**/**2f'** = 6:1).[4]

2f (Major isomer): ^{1}H NMR ($CDCl_3$): δ 3.88 (s, 3H), 6.93 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.98 (d, *J* = 2.4 Hz, 1H), 7.40–7.52 (m, 3H), 7.55 (d, *J* = 7.8 Hz, 2H), 7.67 (d, *J* = 8.3 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 55.6, 103.0, 113.4, 115.4, 119.0, 128.5, 128.6, 128.7, 135.3, 138.1, 147.5, 162.6.

2f' (Minor isomer): ^{1}H NMR ($CDCl_3$): δ 3.99 (s, 3H), 7.14 (s, 1H), 7.21 (d, *J* = 8.3 Hz, 1H), 7.40–7.52 (m, 5H), 7.60 (d, *J* = 8.3 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 56.0, 100.4, 110.0, 116.5, 119.7, 128.5, 128.7 129.0, 133.8, 139.5, 147.7, 161.4.

(4) W. Li, Z. Xu, P. Sun, X. Jiang, M. Fang, *Org. Lett.* 2011, **13**, 1286.

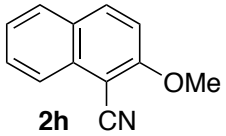
4-Methoxy-1-naphthonitrile (**2g**) [CAS 52449-79-3]



2g

A white solid (45.0 mg, 0.25 mmol, 63% yield). Mp 102–103 °C. ^{1}H NMR ($CDCl_3$): δ 6.83 (d, *J* = 8.3 Hz, 1H), 7.58 (t, *J* = 7.6 Hz, 1H), 7.69 (t, *J* = 8.3 Hz, 1H), 7.85 (d, *J* = 7.8 Hz, 1H), 8.16 (d, *J* = 8.3 Hz, 1H), 8.31 (d, *J* = 8.3 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 55.9, 101.9, 103.3, 118.4, 122.7, 124.9, 125.2, 126.7, 128.9, 133.4, 134.0, 159.4.

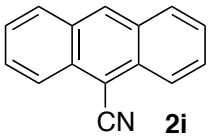
2-Methoxy-1-naphthonitrile (**2h**) [CAS 16000-39-8]



2h

A pale yellow solid (58.8 mg, 0.32 mmol, 80% yield). Mp 94–95 °C. ^{1}H NMR ($CDCl_3$): δ 7.27 (d, *J* = 8.8 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.64 (t, *J* = 7.8 Hz, 1H), 7.82 (d, *J* = 8.3 Hz, 1H), 8.03 (d, *J* = 9.2 Hz, 1H), 8.09 (d, *J* = 8.3 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 56.6, 95.2, 112.0, 115.6, 124.0, 125.0, 128.0, 128.4, 129.1, 133.5, 135.0, 161.2.

9-Anthracenecarbonitrile (**2i**) [CAS 1210-12-4]

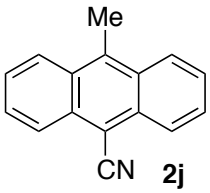


2i

A yellow solid (66.3 mg, 0.33 mmol, 83% yield). Mp 178–179 °C. ^{1}H NMR ($CDCl_3$): δ 7.59 (t, *J* = 7.6 Hz, 2H), 7.72 (t, *J* = 7.1 Hz, 2H), 8.08 (d, *J* = 7.8 Hz, 2H), 8.42 (d, *J* = 8.3 Hz, 2H), 8.68 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 105.3, 117.2, 125.2, 126.3, 128.86, 128.88, 130.5, 132.6, 133.2.

In the large-scale cyanation reaction, the crude product was purified by recrystallisation from hexane/$CHCl_3$ to give 1.59 g of nitrile **2i** (7.8 mmol, 78% yield).

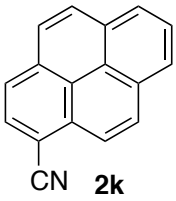
10-Methyl-9-anthracenecarbonitrile (**2j**) [CAS 1467-01-2]



2j

A yellow solid (74.1 mg, 0.34 mmol, 85% yield). Mp 203–204 °C. ^{1}H NMR ($CDCl_3$): δ 3.17 (s, 3H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.71 (t, *J* = 7.6 Hz, 1H), 8.35 (d, *J* = 8.8 Hz, 1H), 8.44 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 14.6, 104.0, 117.6, 125.2, 125.8, 126.0, 128.2, 129.1, 132.6, 137.9.

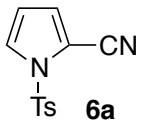
1-Pyrenecarbonitrile (**2k**) [CAS 4107-64-6]



2k

A pale yellow solid (80.0 mg, 0.35 mmol, 88% yield). Mp 154–155 °C. ^{1}H NMR ($CDCl_3$): δ 8.05–8.35 (m, 8H), 8.45 (d, *J* = 9.3 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 105.2, 118.7, 123.0, 123.4, 123.5, 124.0, 126.5, 126.70, 126.73, 126.8, 129.1, 130.0, 130.1, 130.5, 134.5, 133.7.

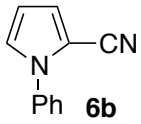
1-Tosyl-1*H*-pyrrole-2-carbonitrile (**6a**) [CAS 220105-73-7]



6a

A white solid (78.3 mg, 0.32 mmol, 80% yield). Mp 116–117 °C. ^{1}H NMR ($CDCl_3$): δ 2.44 (s, 3H), 6.32 (t, *J* = 3.4 Hz, 1H), 6.96 (dd, *J* = 3.9, 2.0 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.48 (dd, *J* = 2.9, 1.5 Hz, 1H), 7.93 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 21.7, 103.7, 111.6, 112.3, 126.5, 126.6, 127.8, 130.3, 134.1, 149.5.

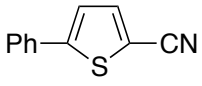
1-Phenyl-1*H*-pyrrole-2-carbonitrile (**6b**) [CAS 35524-46-0]



6b

A pale yellow oil (48.0 mg, 0.29 mmol, 73% yield). Mp 59–61 °C. ^{1}H NMR ($CDCl_3$): δ 6.34 (br s, 1H), 6.99 (br s, 1H), 7.08 (br s, 1H), 7.35–7.55 (m, 5H). ^{13}C NMR ($CDCl_3$): δ 104.0, 110.6, 113.8, 122.2, 124.1, 126.9, 128.3, 129.6, 138.2.

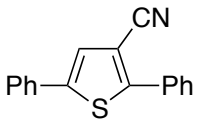
5-Phenyl-2-thiophenecarbonitrile (**6c**) [CAS 215361-51-6]



6c

A pale yellow solid (61.8 mg, 0.33 mmol, 83% yield). Mp 86–87 °C. ^{1}H NMR ($CDCl_3$): δ 7.27 (d, J = 3.9 Hz, 1H), 7.33–7.46 (m, 3H), 7.55–7.68 (m, 3H). ^{13}C NMR ($CDCl_3$): δ 108.3, 114.4, 123.2, 126.4, 129.3, 129.4, 132.3, 138.3, 151.8.

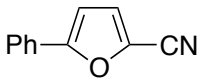
2,5-Dihenyl-3-thiophenecarbonitrile (**6d**) [CAS 437606-44-5]



6d

An orange solid (75.9 mg, 0.29 mmol, 73% yield). Mp 59–60 °C. ^{1}H NMR ($CDCl_3$): δ 7.34–7.52 (m, 7H), 7.58 (d, J = 7.8 Hz, 2H), 7.80 (d, J = 7.3 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 106.5, 115.8, 125.5, 125.8, 127.5, 128.8, 129.18, 129.20, 129.7, 131.3, 132.2, 144.1, 152.4.

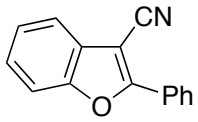
5-Phenyl-2-furancarbonitrile (**6e**) [CAS 57666-50-9]



6e

A brown solid (33.1 mg, 0.20 mmol, 50% yield). Mp 70–71 °C. ^{1}H NMR ($CDCl_3$): δ 6.73 (d, J = 3.9 Hz, 1H), 7.17 (d, J = 3.9 Hz, 1H), 7.34–7.48 (m, 3H), 7.72 (d, J = 7.3 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 106.0, 111.9, 124.0, 124.8, 125.1, 128.7, 129.0, 129.5, 158.6.

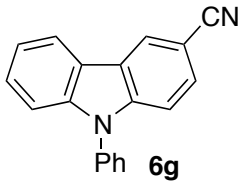
2-Phenylbenzofuran-3-carbonitrile (**6f**) [CAS 37883-72-0]



6f

A pale yellow solid (78.7 mg, 0.36 mmol, 90% yield). Mp 73–74 °C. ^{1}H NMR ($CDCl_3$): δ 7.35–7.47 (m, 2H), 7.50–7.62 (m, 4H), 7.72 (d, J = 8.3 Hz, 1H), 8.20 (d, J = 7.8 Hz, 2H). ^{13}C NMR ($CDCl_3$): δ 88.0, 111.6, 114.2, 119.8, 124.6, 126.3, 126.4, 127.1, 127.7, 129.1, 131.1, 153.2, 161.5.

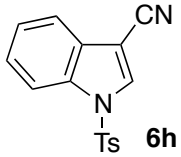
9-Phenyl-9*H*-carbazole-2-carbonitrile (**6g**) [CAS 540473-55-0]



6g

A pale yellow solid (85.8 mg, 0.32 mmol, 80% yield). Mp 172–173 °C. ^{1}H NMR ($CDCl_3$): δ 7.32–7.43 (m, 3H), 7.48 (d, *J* = 8.3 Hz, 1H), 7.50–7.58 (m, 3H), 7.60–7.70 (m, 3H), 8.15 (d, *J* = 7.3 Hz, 1H), 8.45 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 102.5, 110.3, 110.4, 120.3, 120.5, 121.1, 122.1, 123.4, 125.1, 127.0, 127.3, 128.3, 129.0, 130.1, 136.3, 141.5, 142.4.

1-Tosyl-1*H*-indole-3-carbonitrile (**6h**) [CAS 859205-33-7]

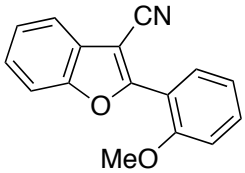


6h

A white solid (46.5 mg, 0.16 mmol, 40% yield). Mp 157–158 °C. ^{1}H NMR ($CDCl_3$): δ 2.38 (s, 1H), 7.30 (d, *J* = 8.3 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.3 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.83 (d, *J* = 8.3 Hz, 2H), 8.00 (d, *J* = 8.3 Hz, 1H), 8.10 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 21.7, 93.7, 113.4, 113.8, 120.3, 124.8, 126.5, 127.2, 128.3, 130.4, 133.1, 133.6, 134.1, 146.3.

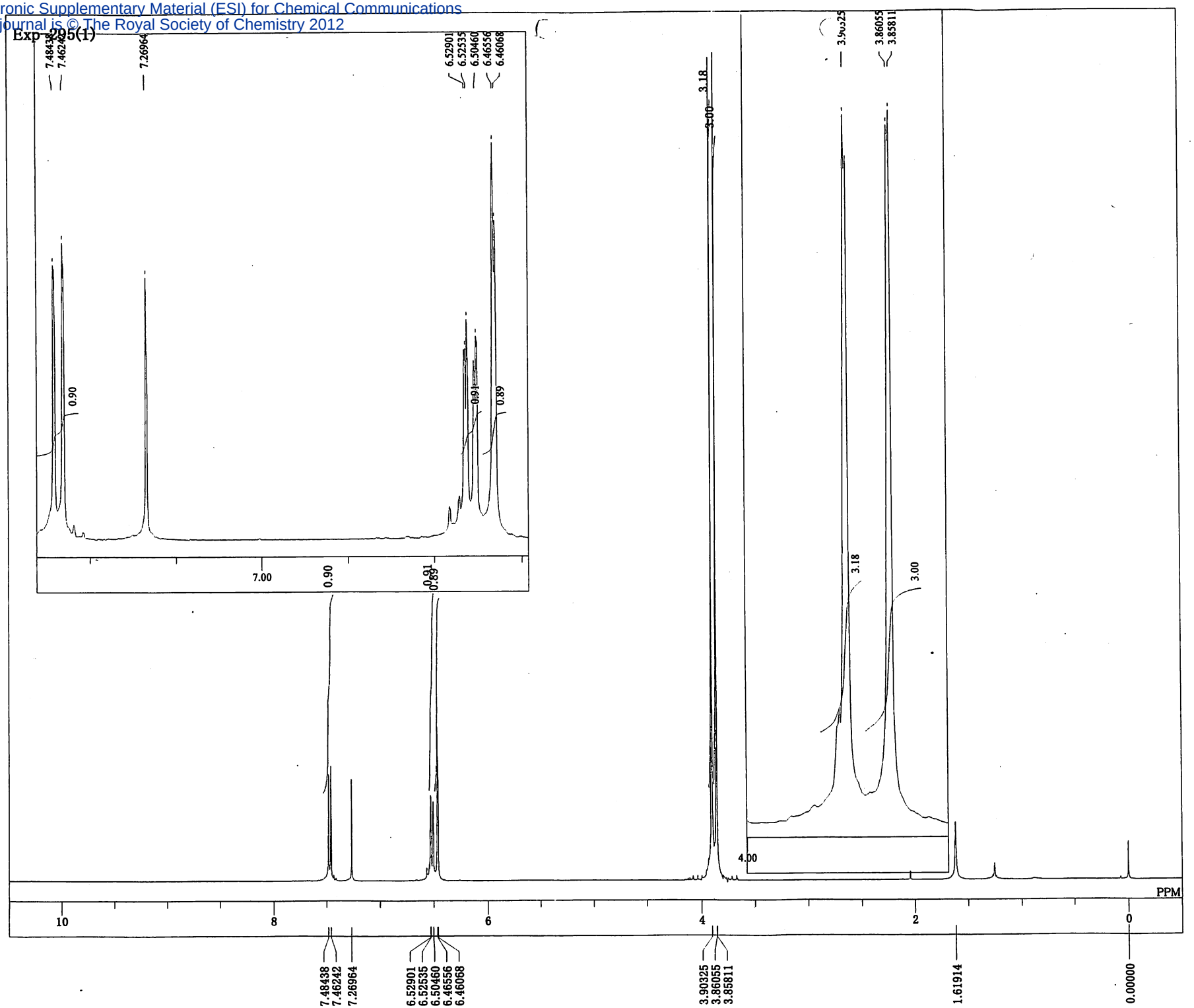
3. Tandem cyanation-demethylation reaction (eq. 1)

2-(2-Methoxyphenyl)benzofuran-3-carbonitrile (**8**)

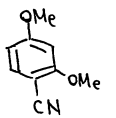


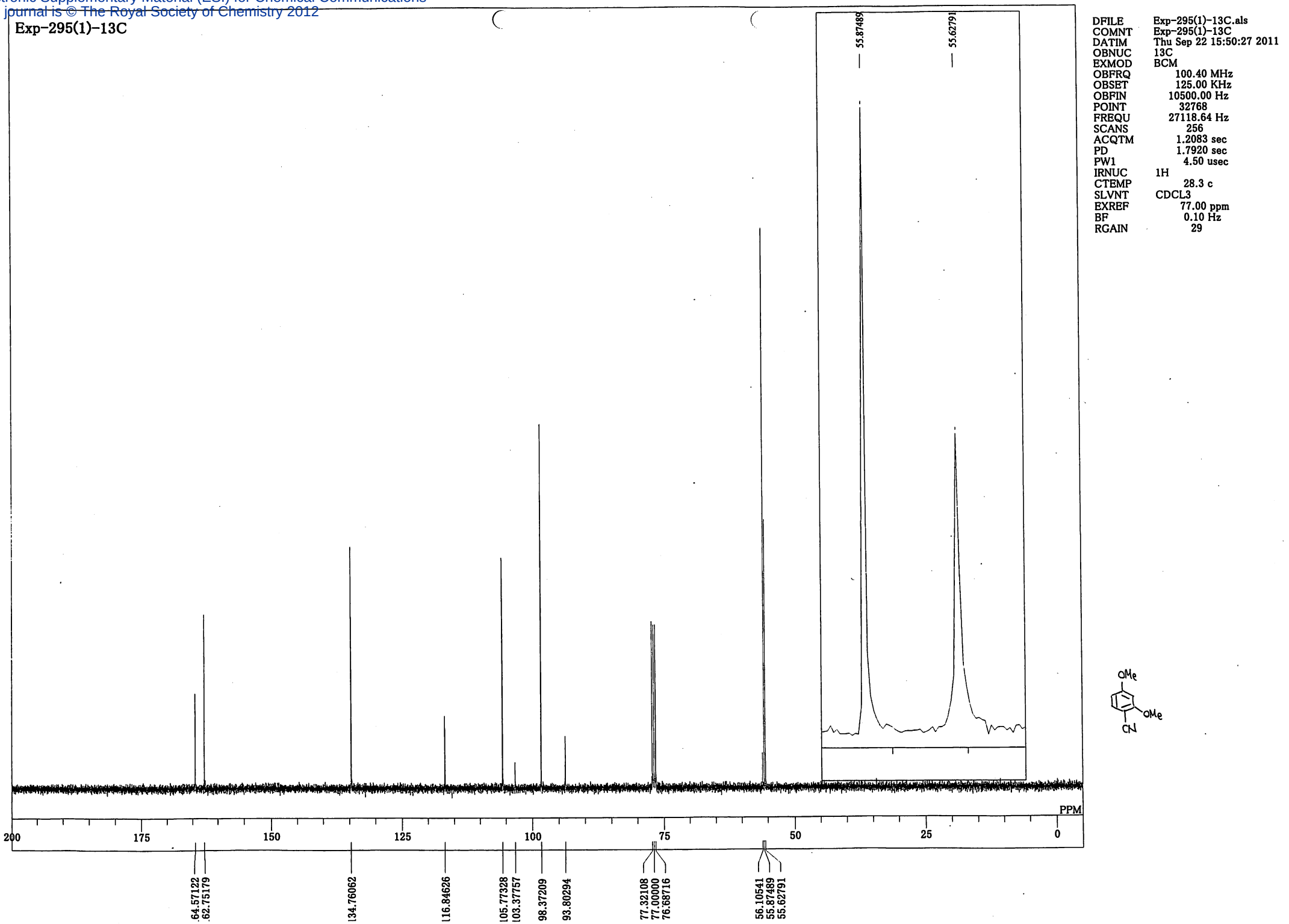
8

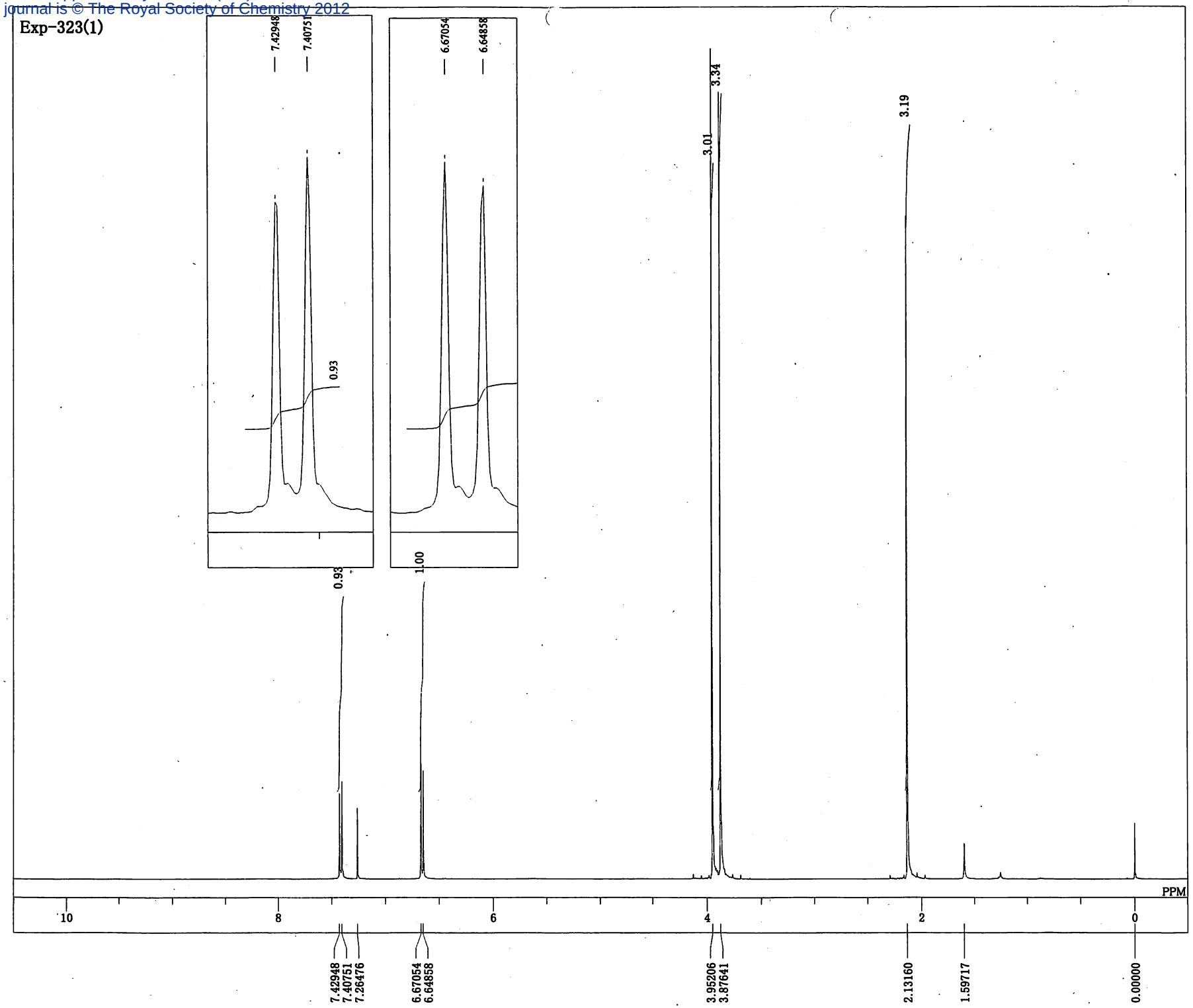
A white solid (54.7 mg, 0.22 mmol, 55% yield). Mp 104–105 °C. IR (KBr): 2224, 1498, 1294, 1024, 734 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 4.03 (s, 3H), 7.07 (d, *J* = 8.8 Hz, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 7.35–7.43 (m, 2H), 7.50 (t, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 6.8 Hz, 1H), 7.75 (d, *J* = 7.3 Hz, 1H), 7.86 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 54.7, 92.0, 111.45, 111.47, 113.8, 117.2, 119.9, 120.7, 124.3, 126.0, 128.0, 129.6, 132.5, 153.1, 157.4, 158.5. HRMS (FAB) calcd for $C_{16}H_{11}NO_2$ (M^+) 249.0790, found 249.0791.



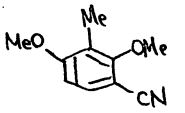
DFILE	_DEFAULT.ALS
COMNT	Exp-295(1)
DATIM	Mon Aug 08 15:53:24 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	17

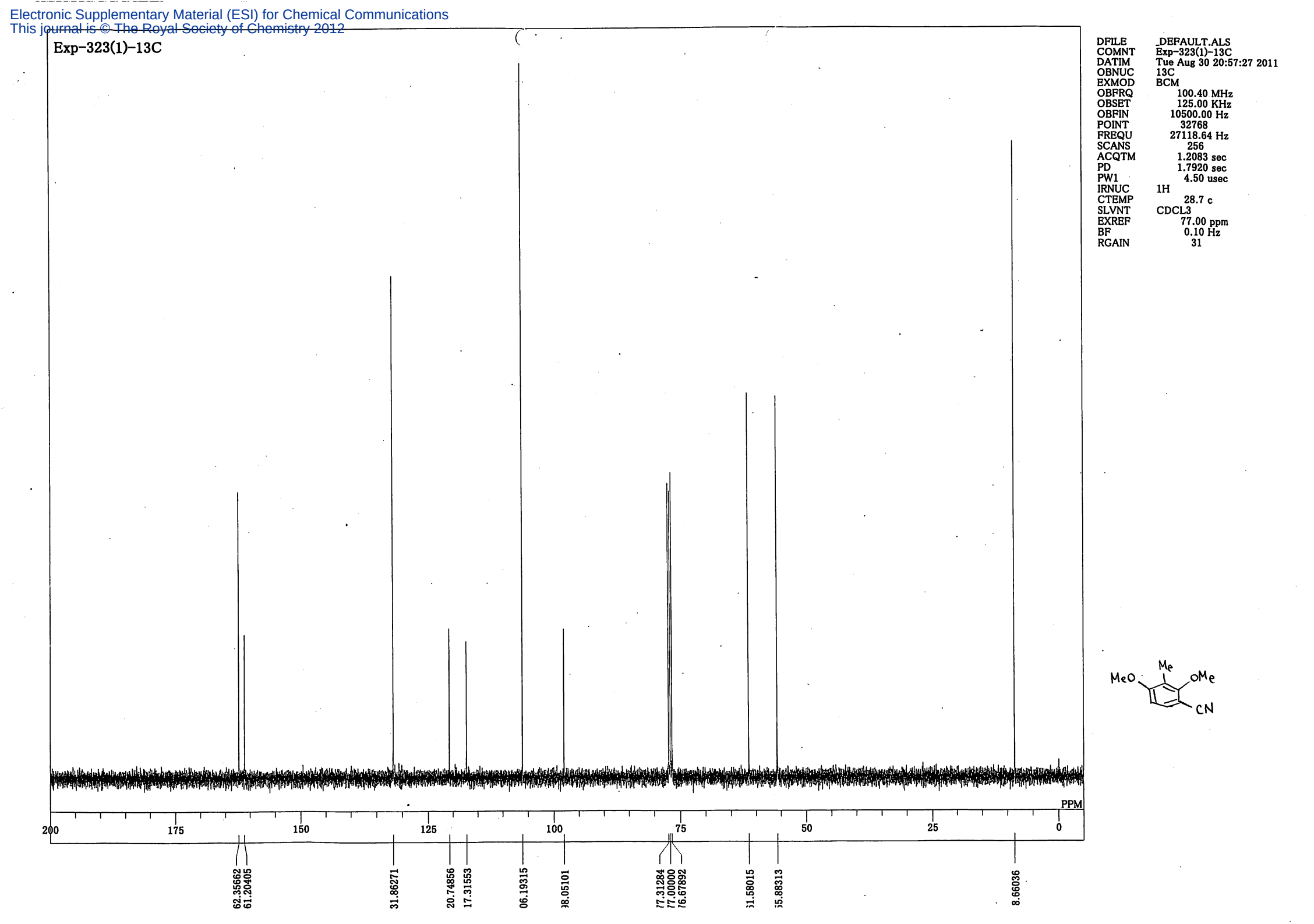


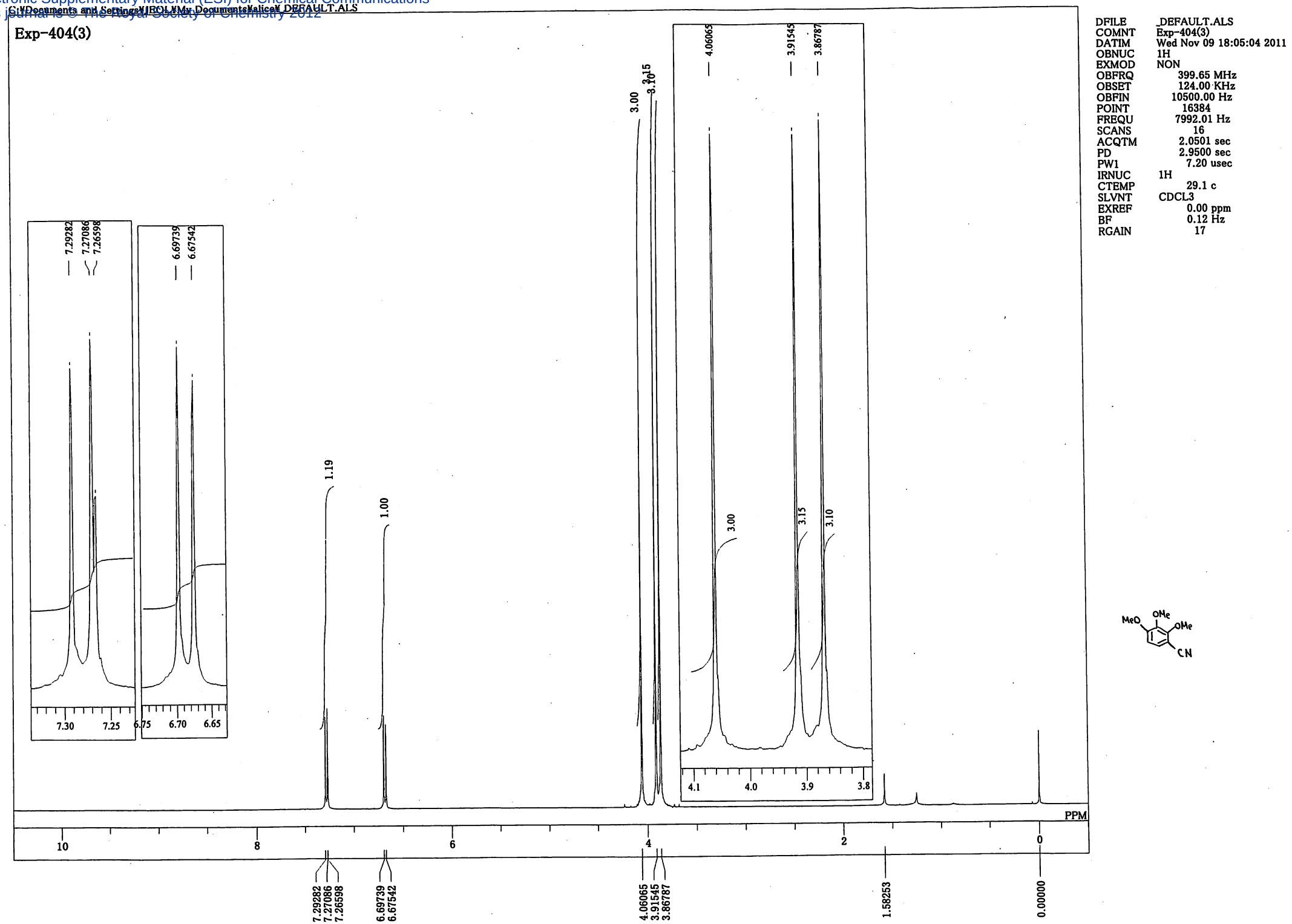


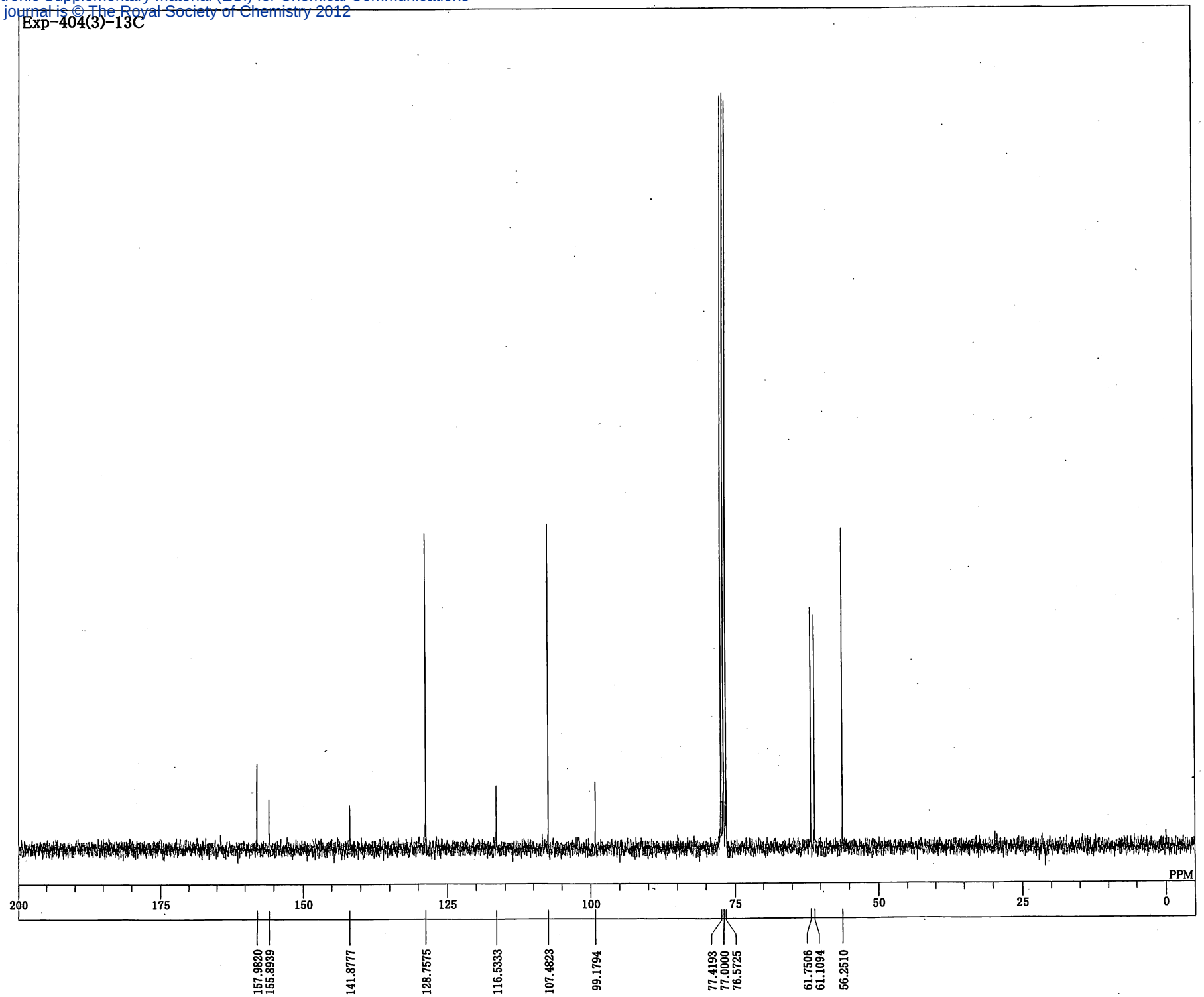


DFILE	_DEFAULT.ALS
COMNT	Exp-323(1)
DATIM	Tue Aug 30 20:39:52 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	29.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	16

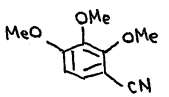


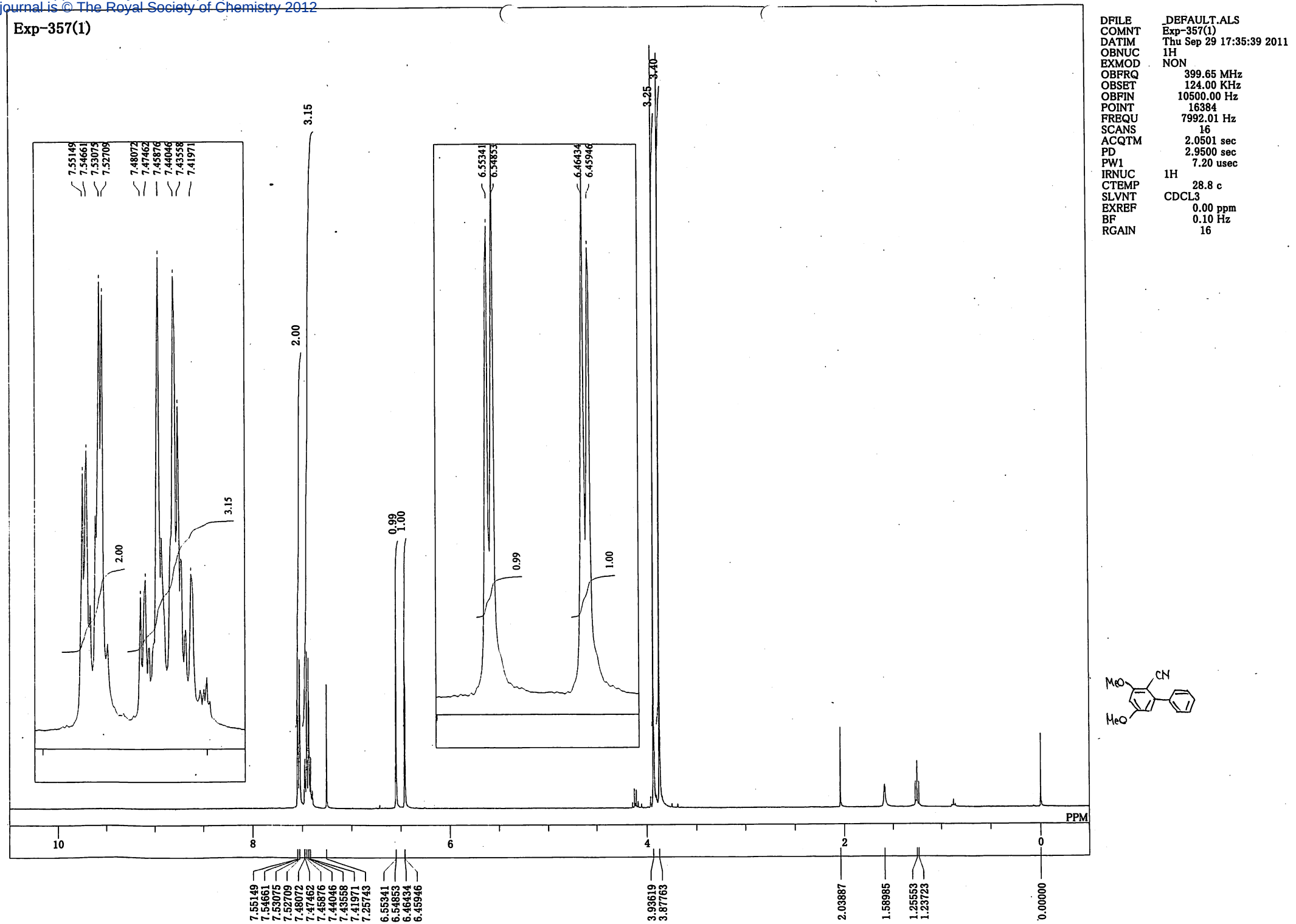


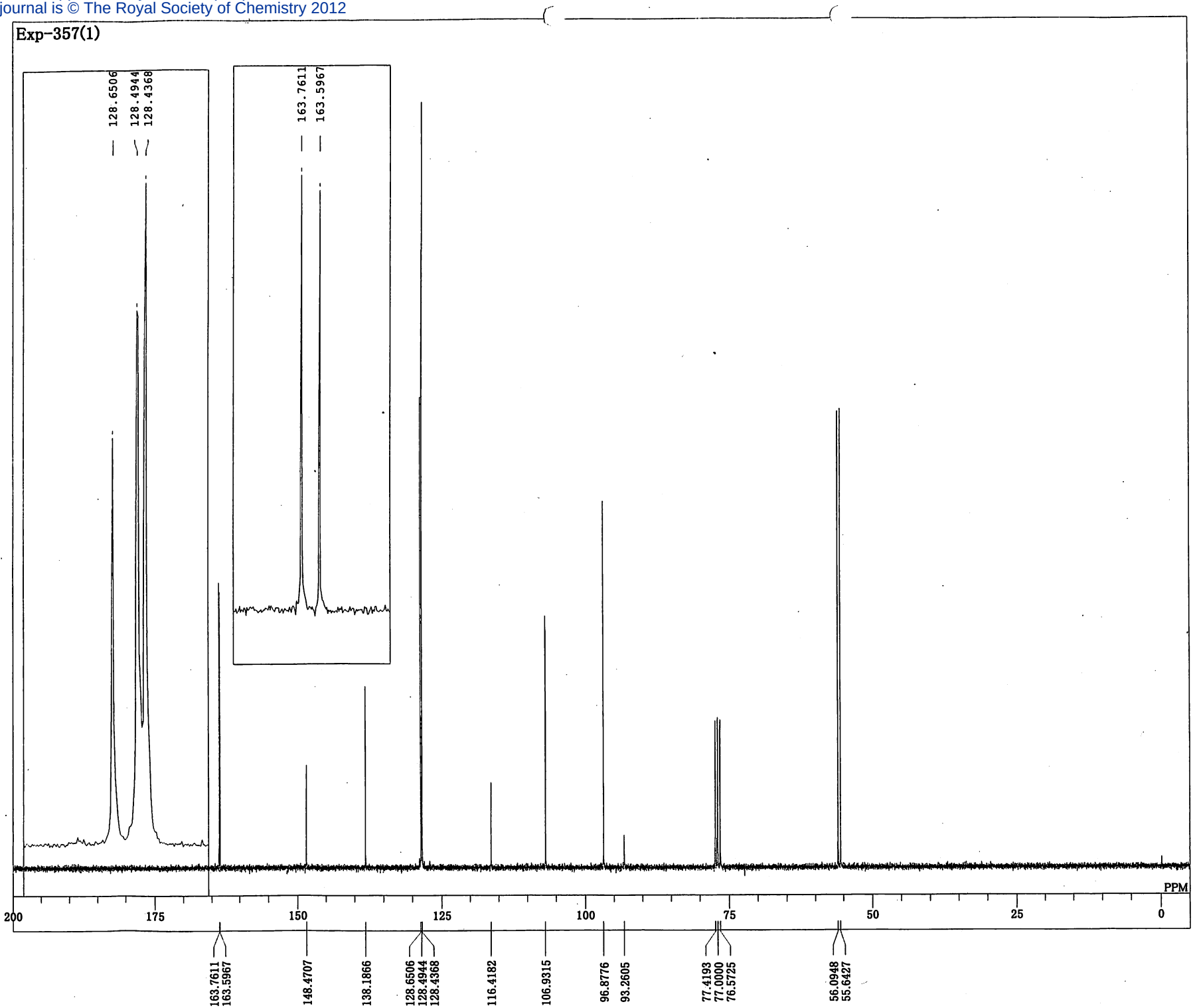




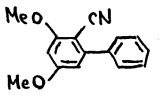
DFILE	_DEFAULT.ALS
COMNT	Exp-404(3)-13C
DATIM	Wed Nov 09 20:56:05 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1024
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	28

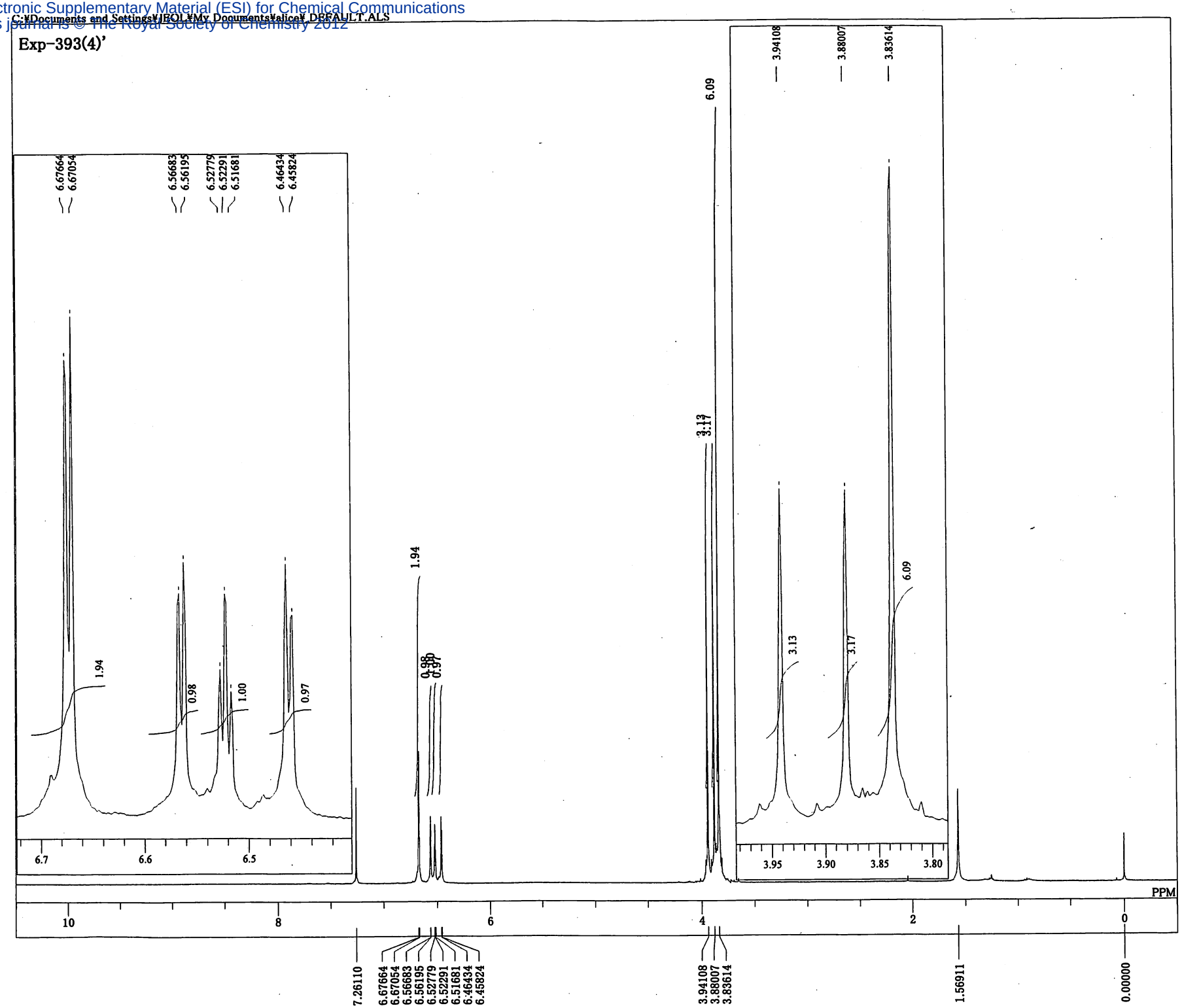




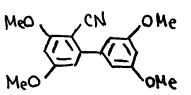


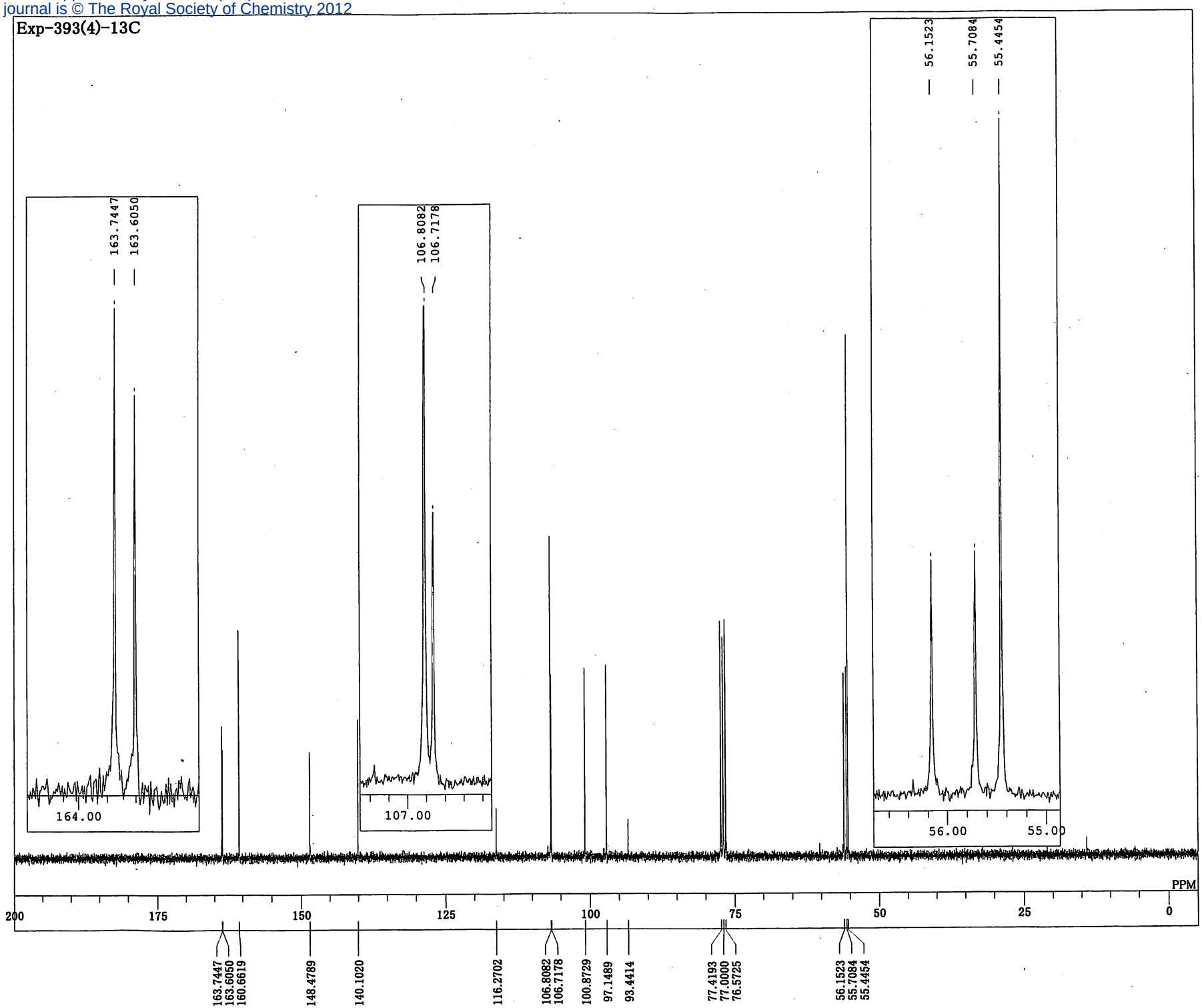
DFILE	Exp-357(1)-13C.als
COMNT	Exp-357(1)
DATIM	Wed Oct 12 12:18:54 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1024
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	29



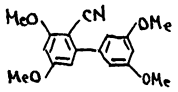


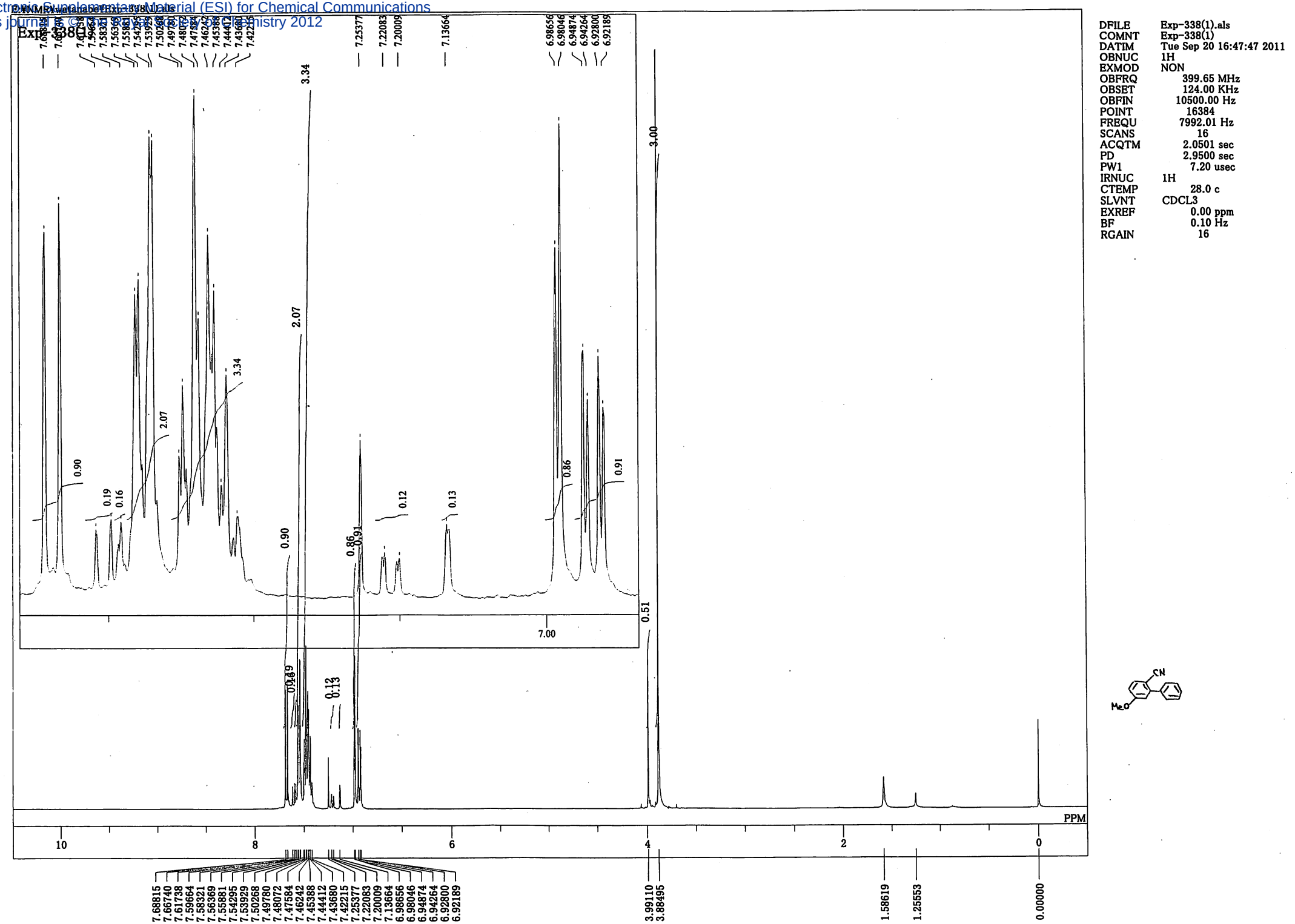
DFILE	_DEFAULT.ALS
COMNT	Exp-393(4)'
DATIM	Wed Nov 02 16:56:06 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	19

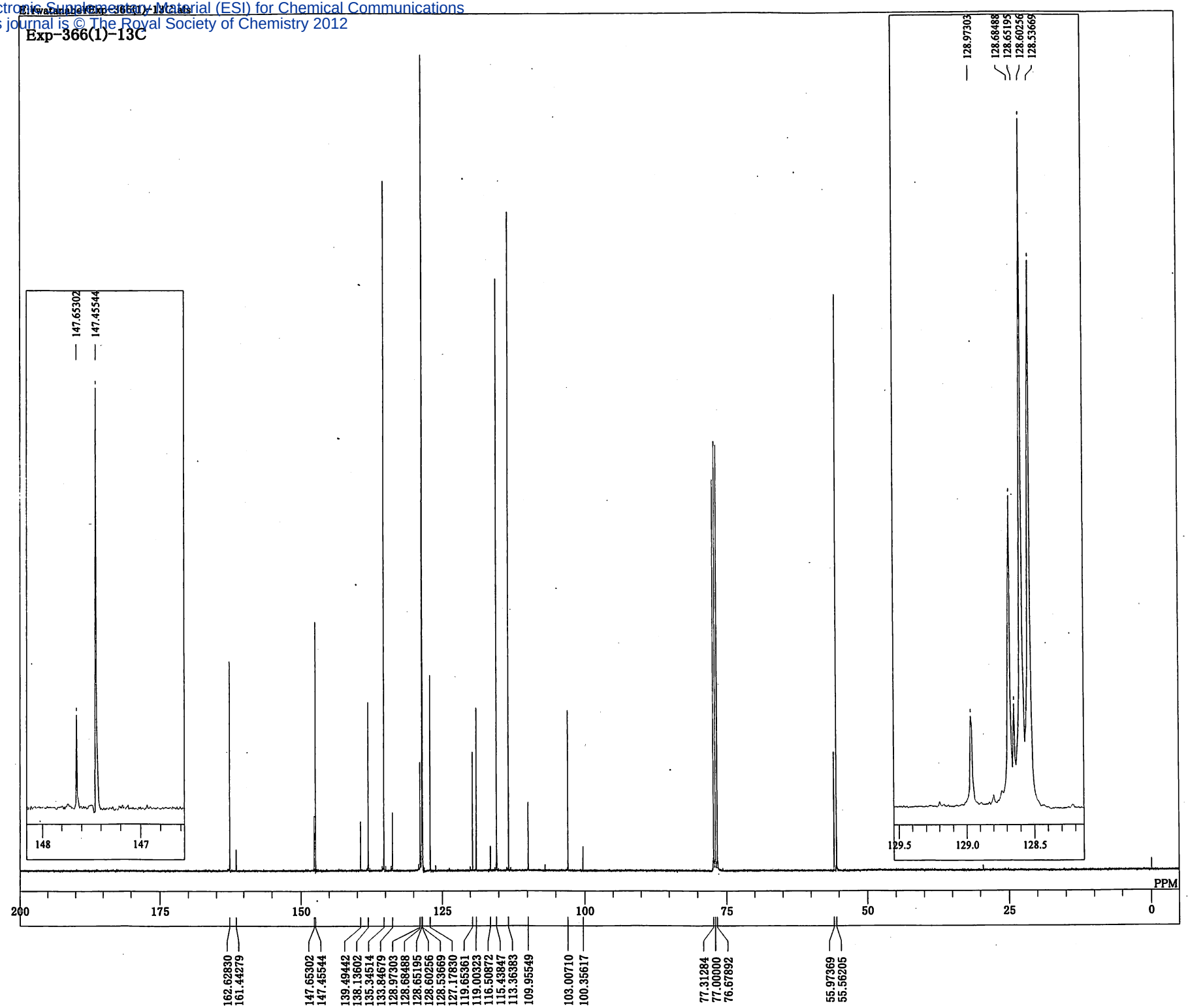




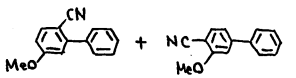
DFILE	_DEFAULT.ALS
COMNT	Exp-393(4)-13C
DATIM	Wed Nov 02 13:45:15 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1024
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28

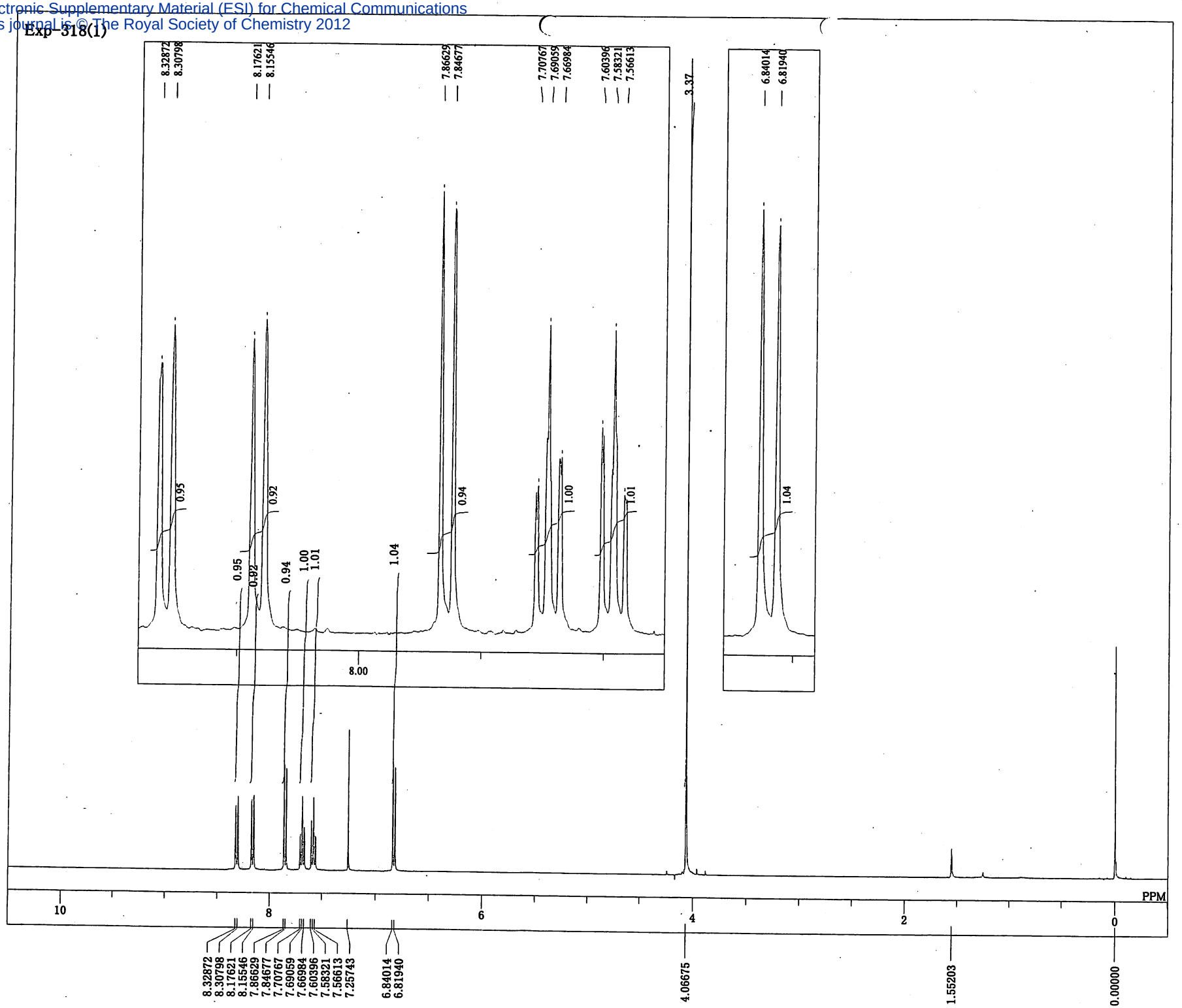




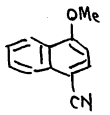


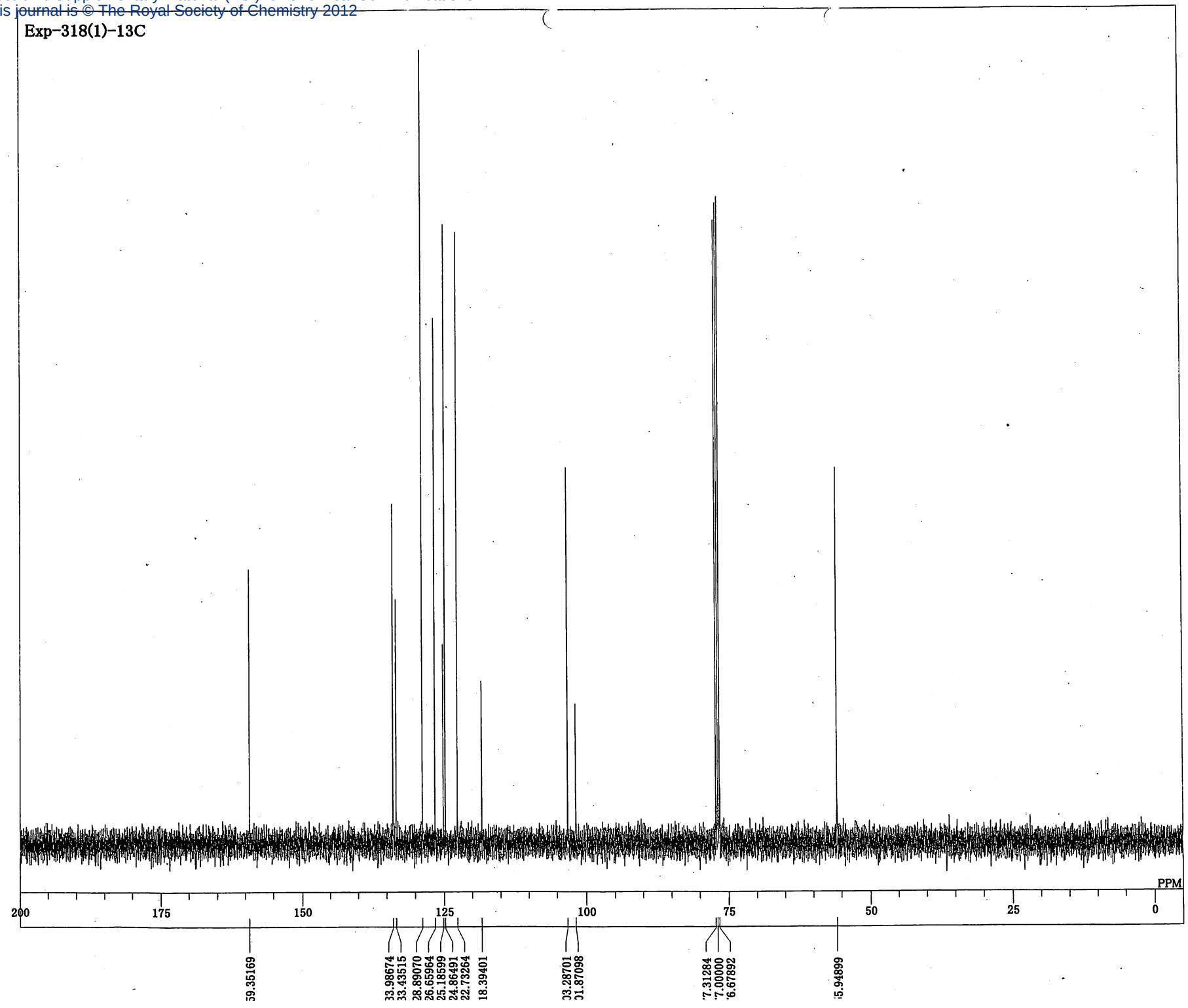
DFILE	Exp-366(1)-13C.als
COMNT	Exp-366(1)-13C
DATIM	Tue Nov 22 09:41:29 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	12000
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	28.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	31



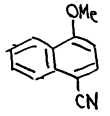


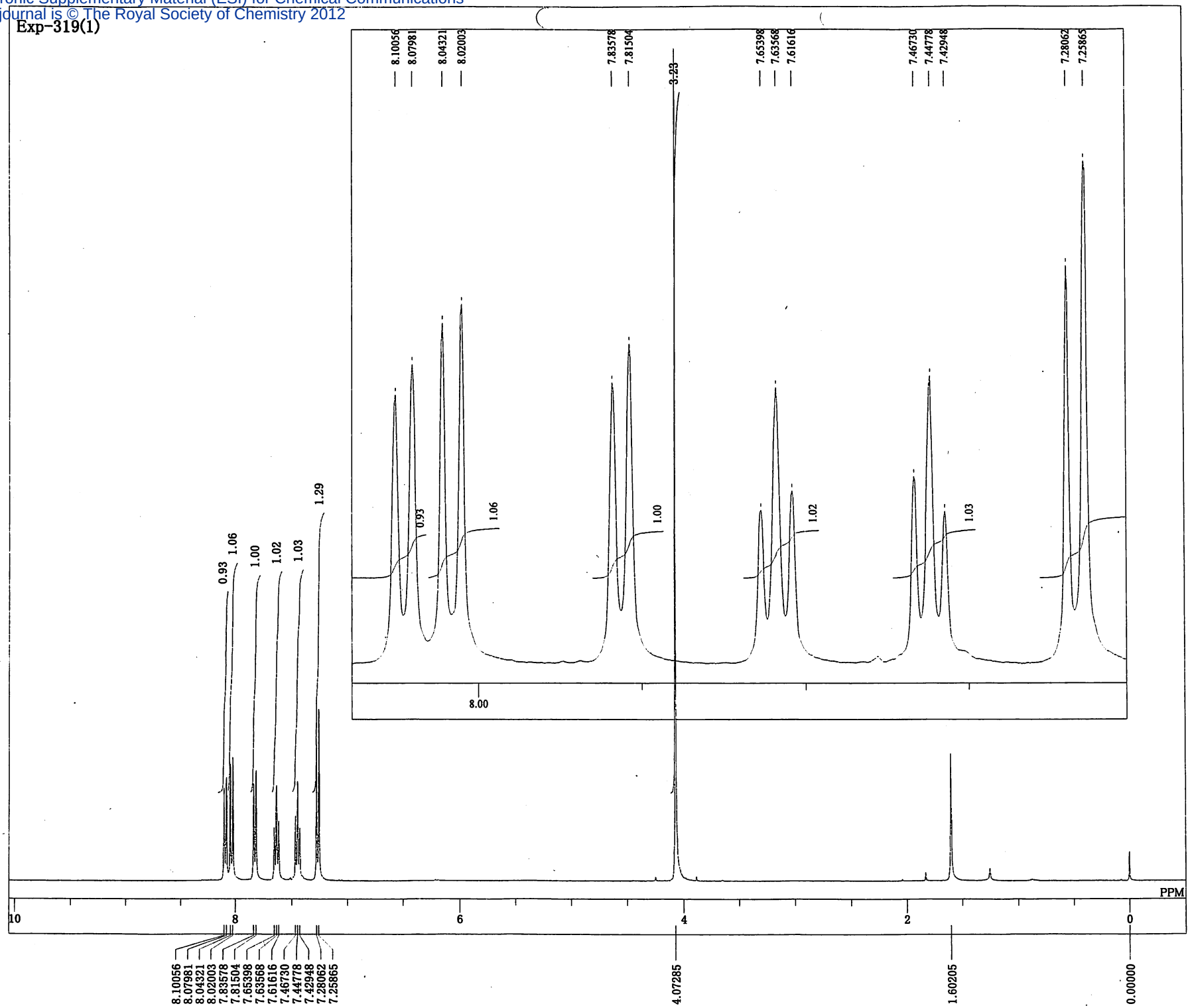
DFILE	_DEFAULT.ALS
COMNT	Exp-318(1)
DATIM	Wed Aug 31 16:12:17 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	29.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	20



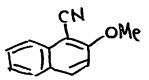


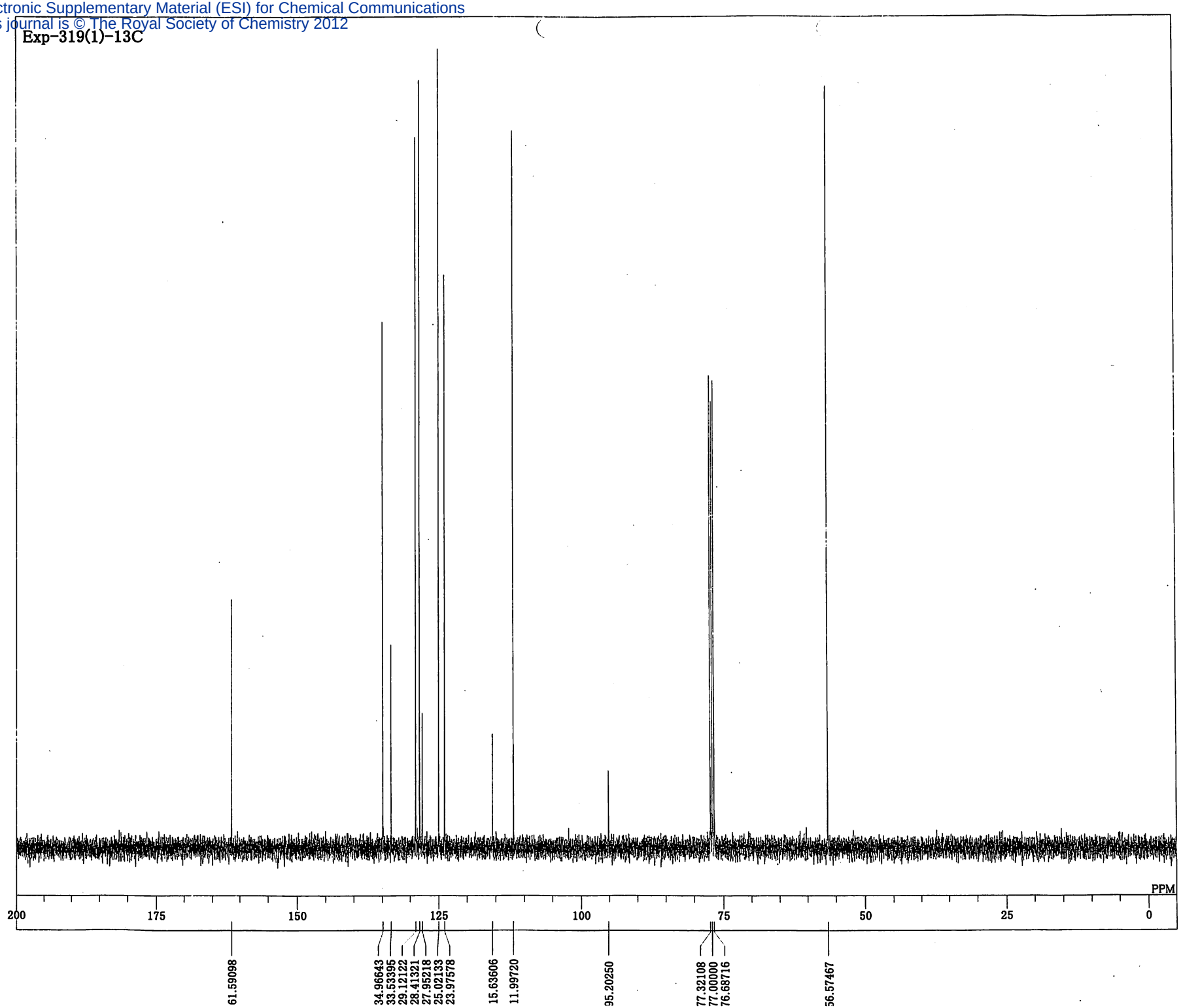
DFILE	_DEFAULT.ALS
COMNT	Exp-318(1)-13C
DATIM	Wed Aug 31 21:07:33 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	256
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	28.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	31

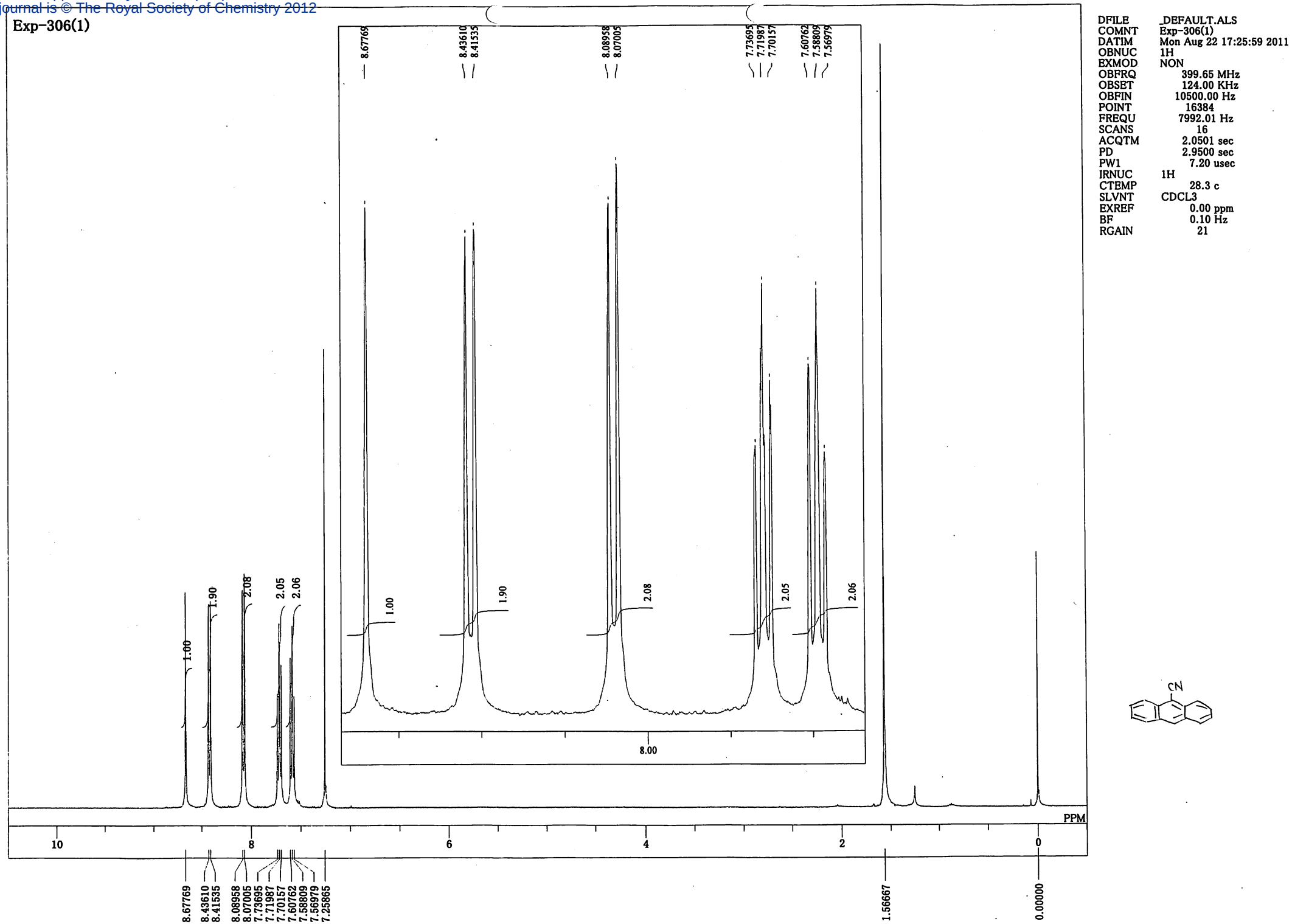




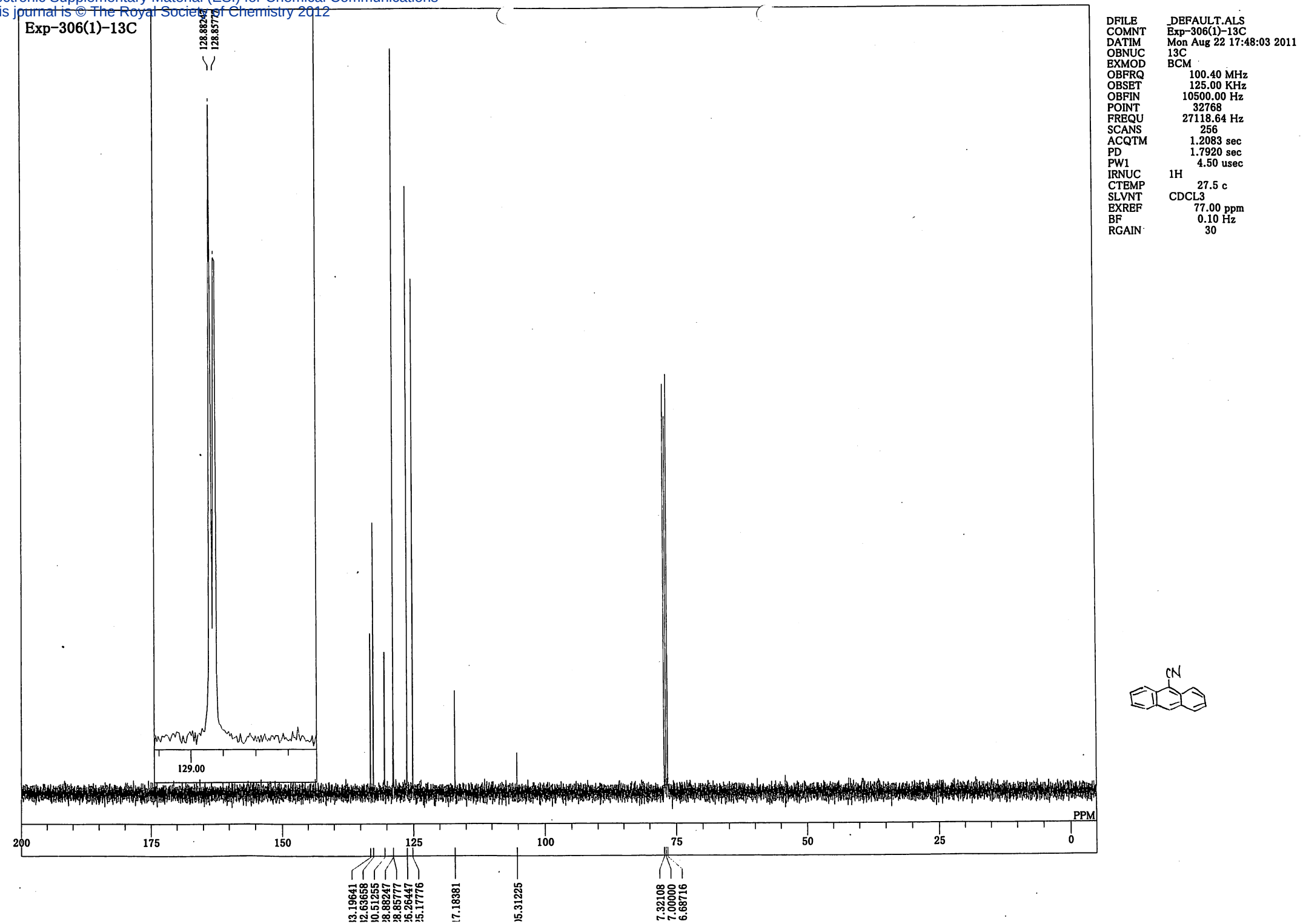
DFILE _DEFAULT.ALS
COMNT Exp-319(1)
DATIM Mon Aug 29 13:20:35 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0501 sec
PD 2.9500 sec
PW1 7.20 usec
IRNUC 1H
CTEMP 28.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 17

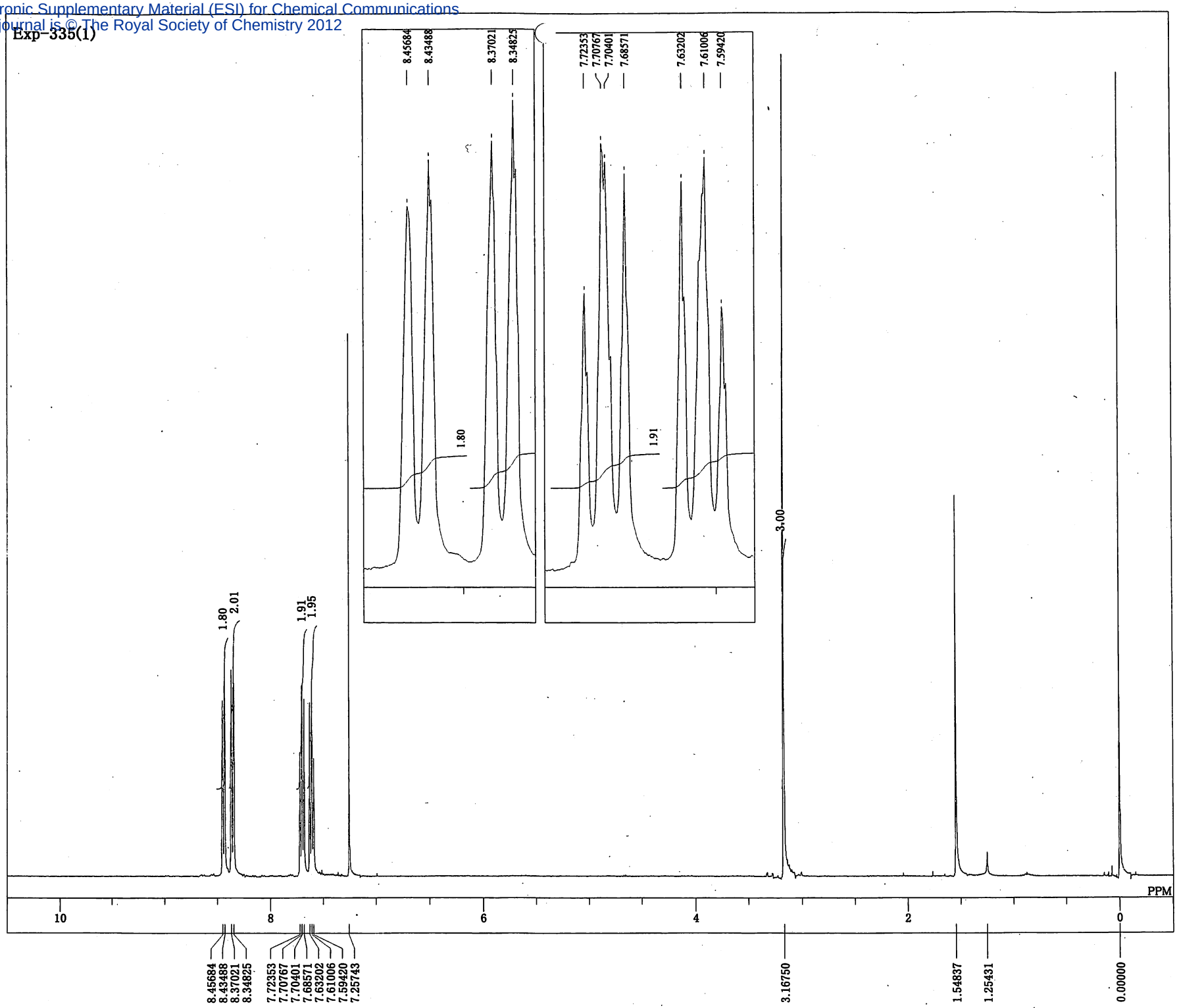




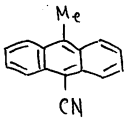


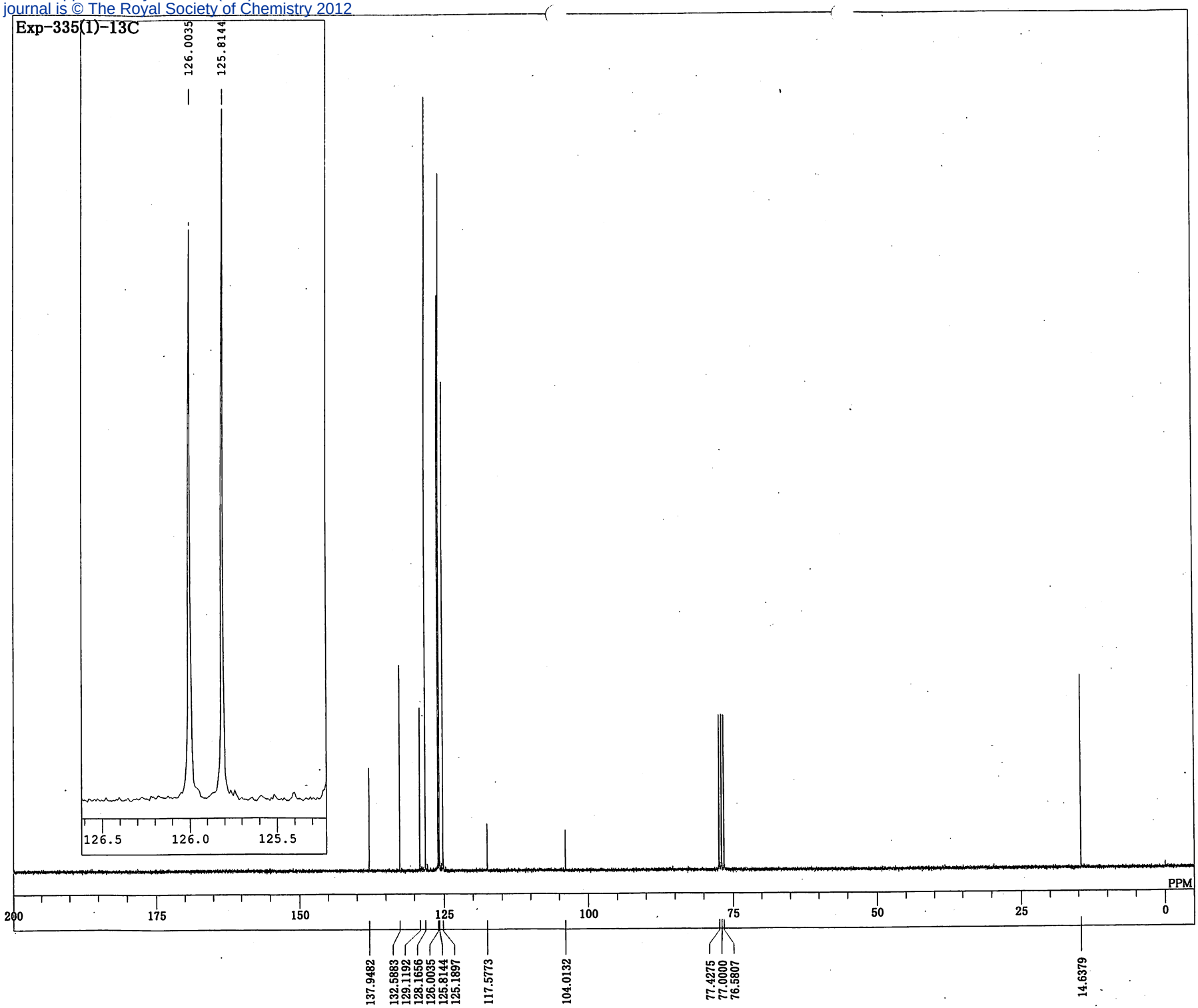
DFILE	_DEFAULT.ALS
COMNT	Exp-306(1)
DATIM	Mon Aug 22 17:25:59 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	21



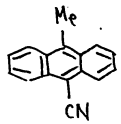


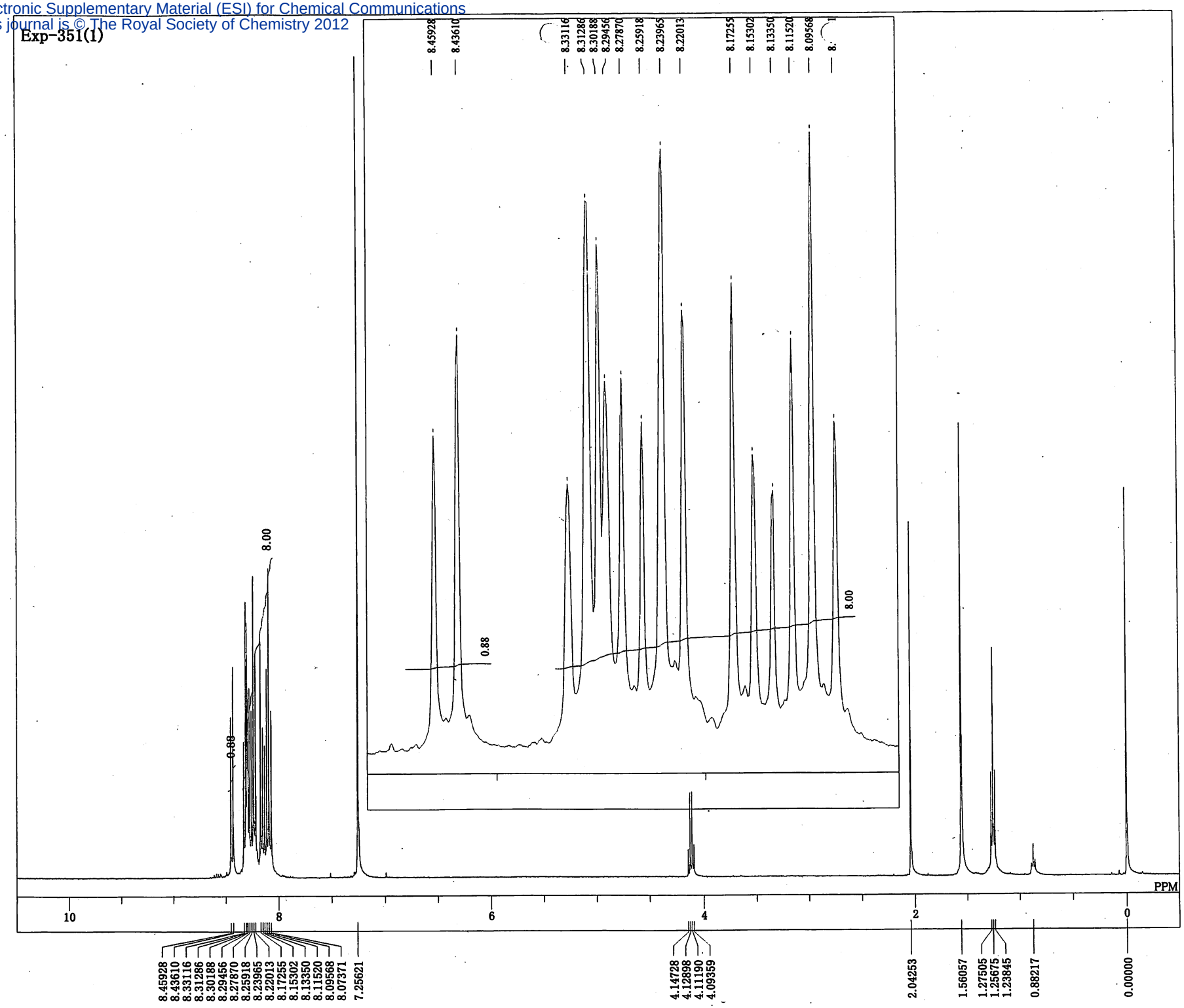
DFILE .DEFAULT.ALS
COMNT Exp-335(1)
DATIM Thu Sep 15 13:36:27 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0501 sec
PD 2.9500 sec
PW1 7.20 usec
IRNUC 1H
CTEMP 29.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 21



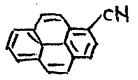


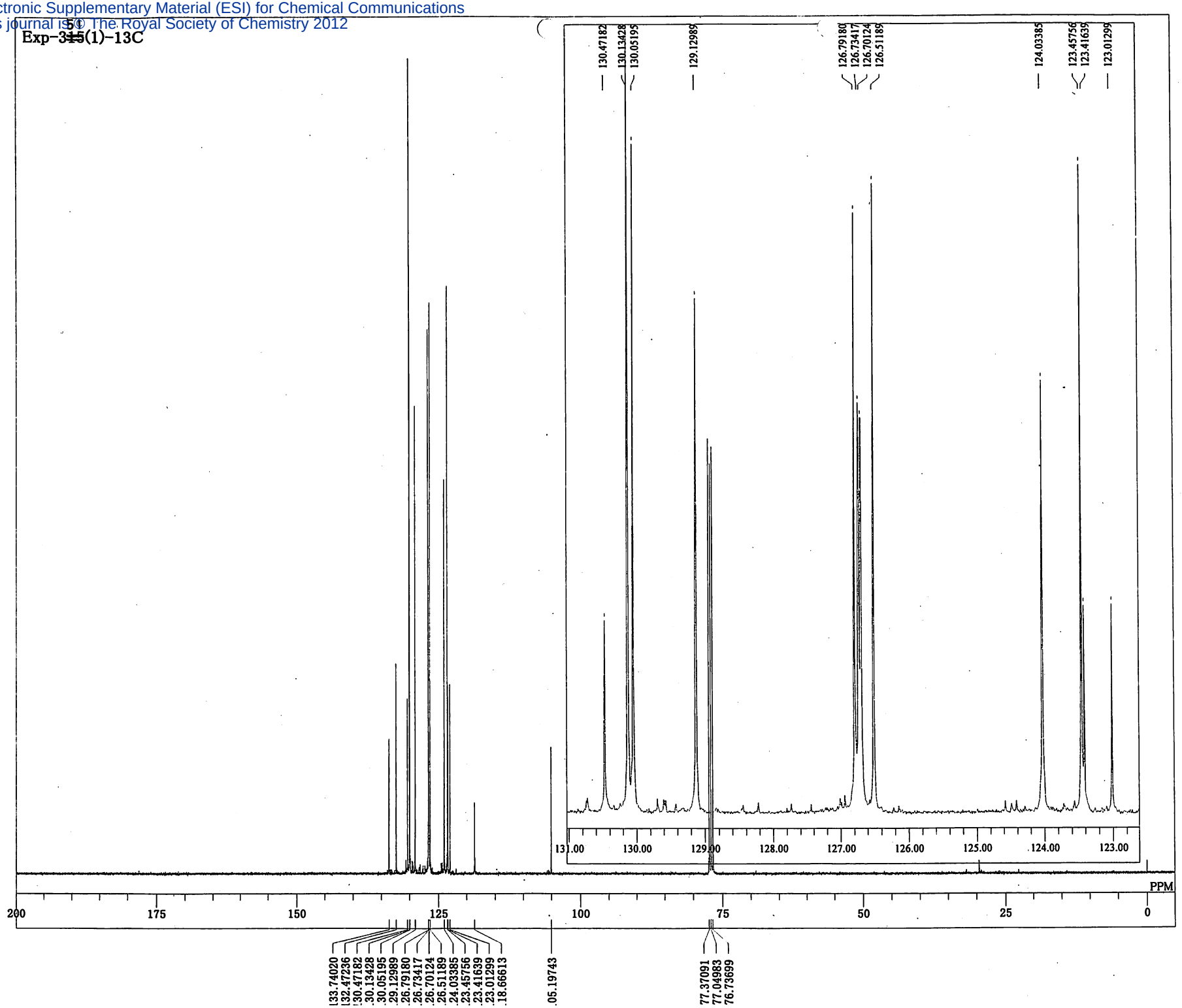
DFILE	_DEFAULT.ALS
COMNT	Exp-335(1)-13C
DATIM	Thu Sep 15 19:02:54 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	2048
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28

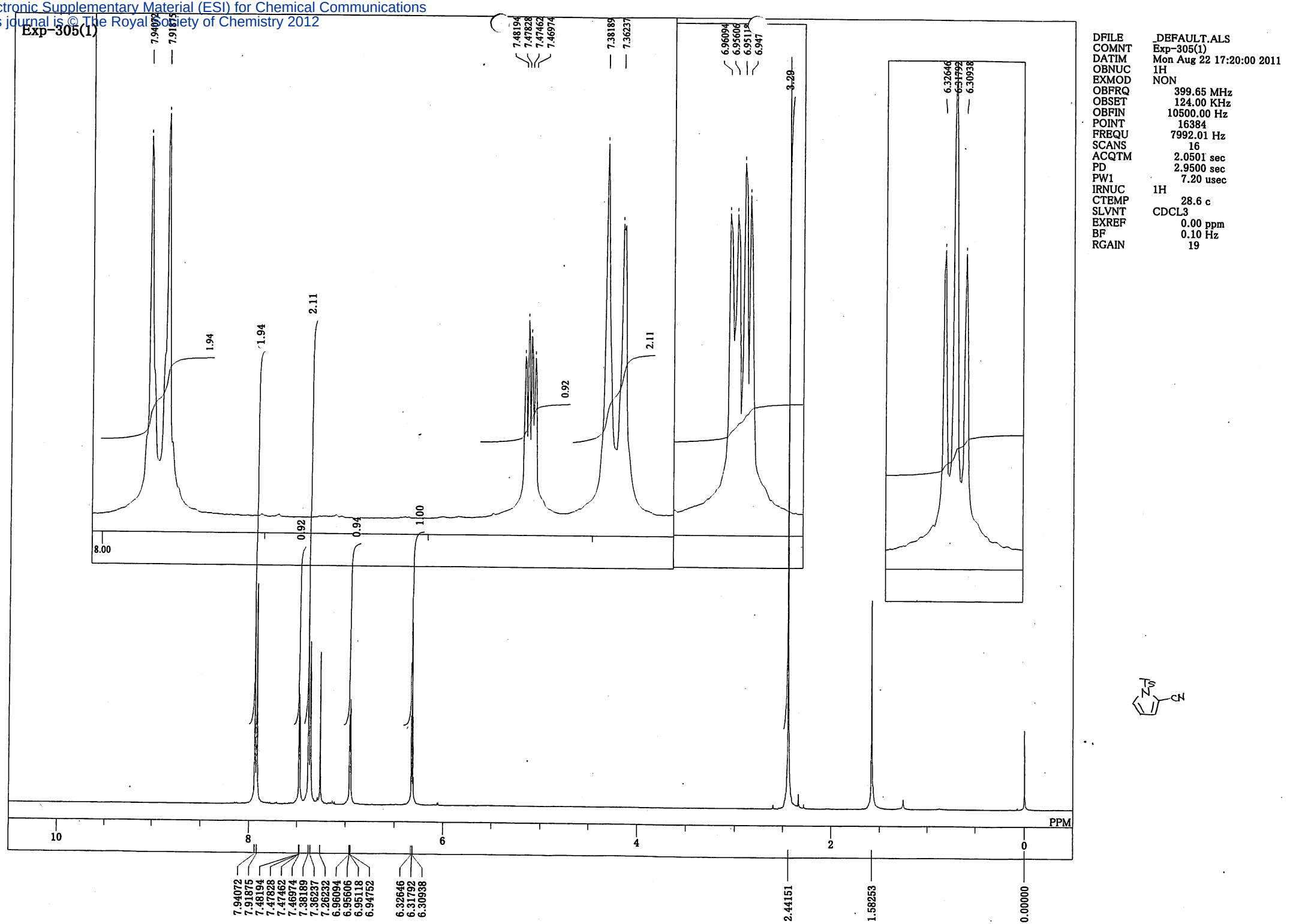




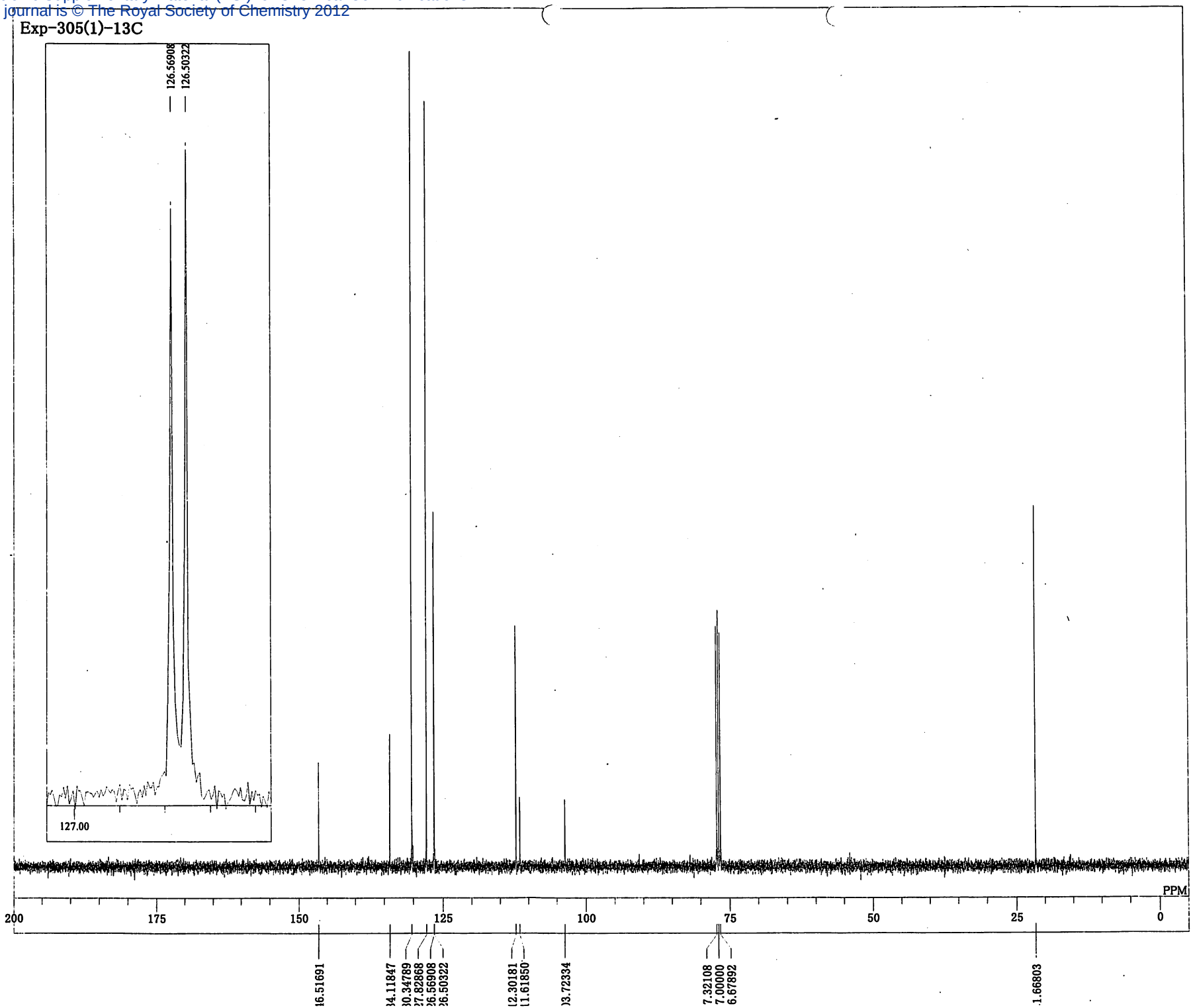
DFILE	Exp-351(1).als
COMNT	Exp-351(1)
DATIM	Tue Sep 27 19:51:01 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	21



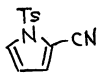


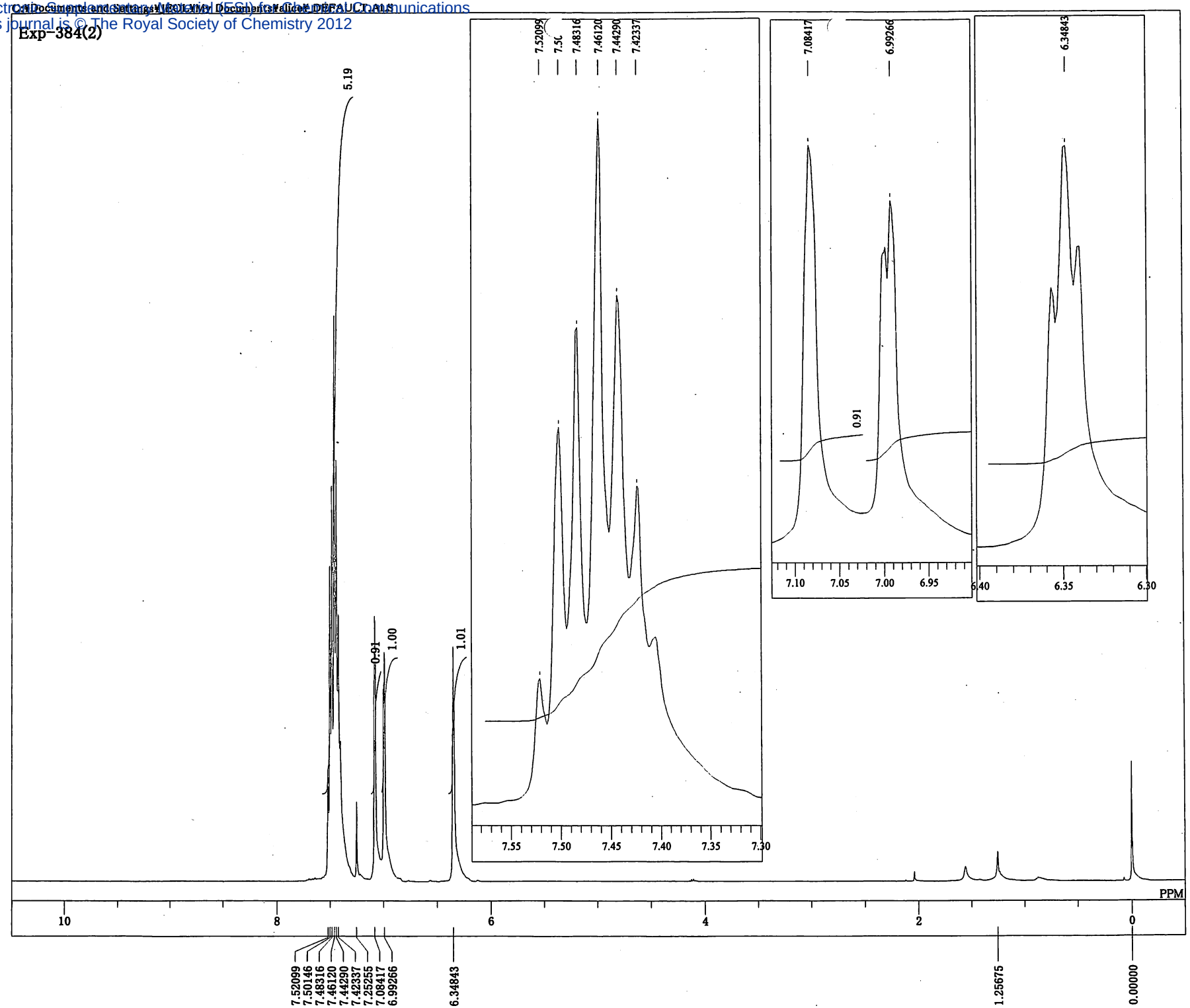


DFILE	_DEFAULT.ALS
COMNT	Exp-305(1)
DATIM	Mon Aug 22 17:20:00 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	19

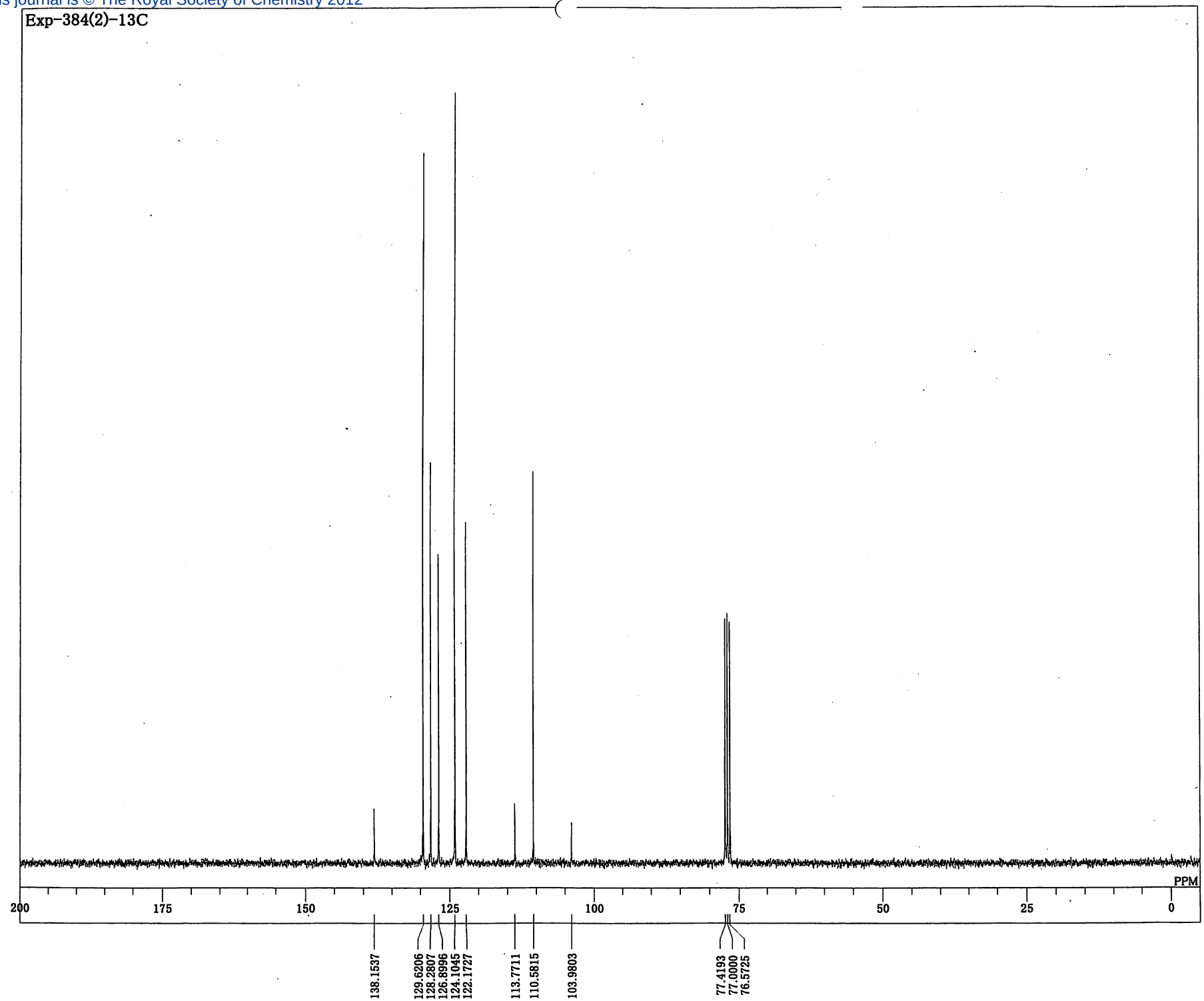


DFILE	_DEFAULT.ALS
COMNT	Exp-305(1)-13C
DATIM	Mon Aug 22 13:41:05 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
POINT	32768
FREQU	27118.64 Hz
SCANS	256
ACQTM	1.2083 sec
PD	1.7920 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	28.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	30

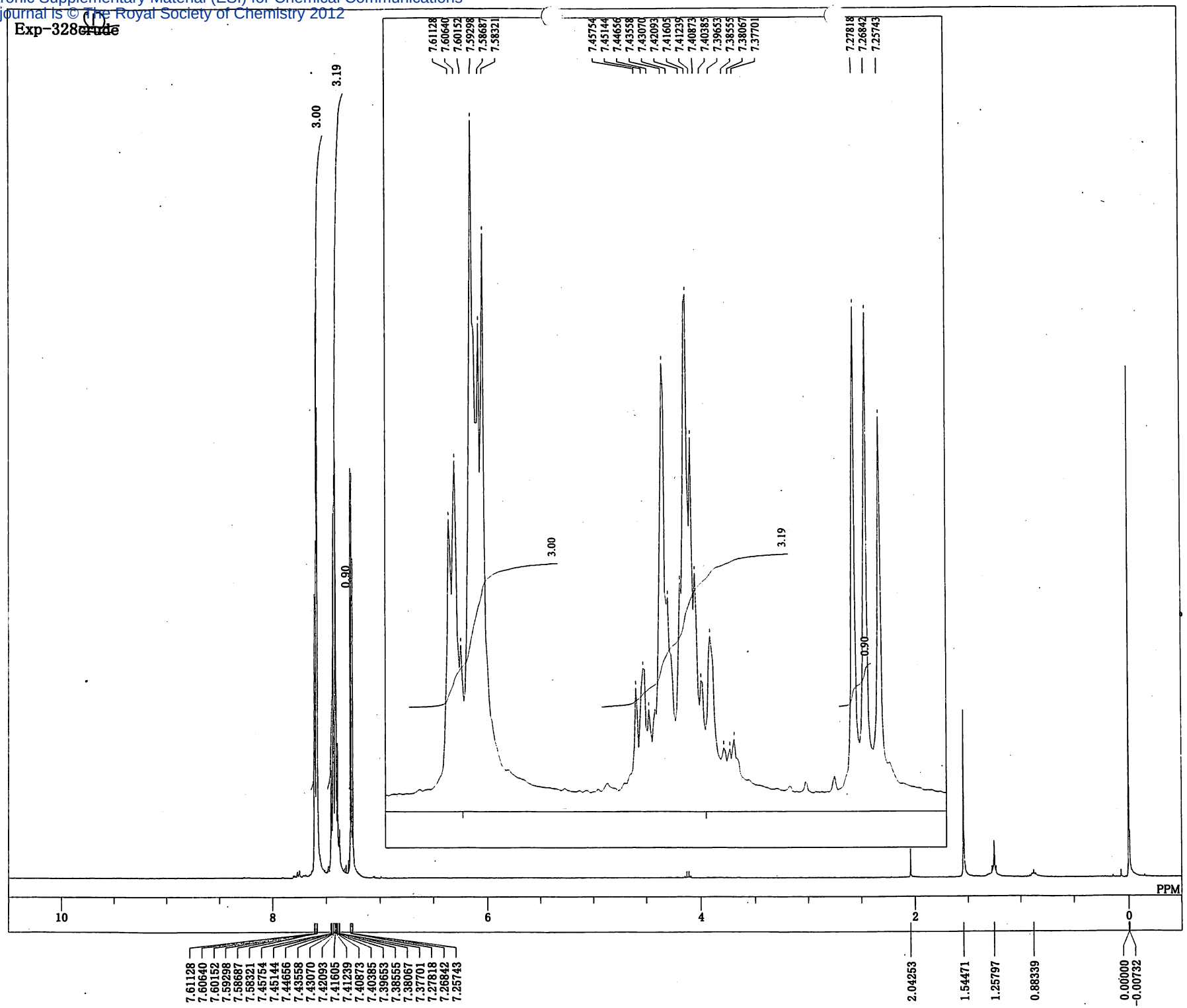




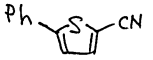
DFILE _DEFAULT.ALS
COMNT Exp-384(2)
DATIM Tue Nov 15 16:29:54 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0501 sec
PD 2.9500 sec
PW1 7.20 usec
IRNUC 1H
CTEMP 28.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 16

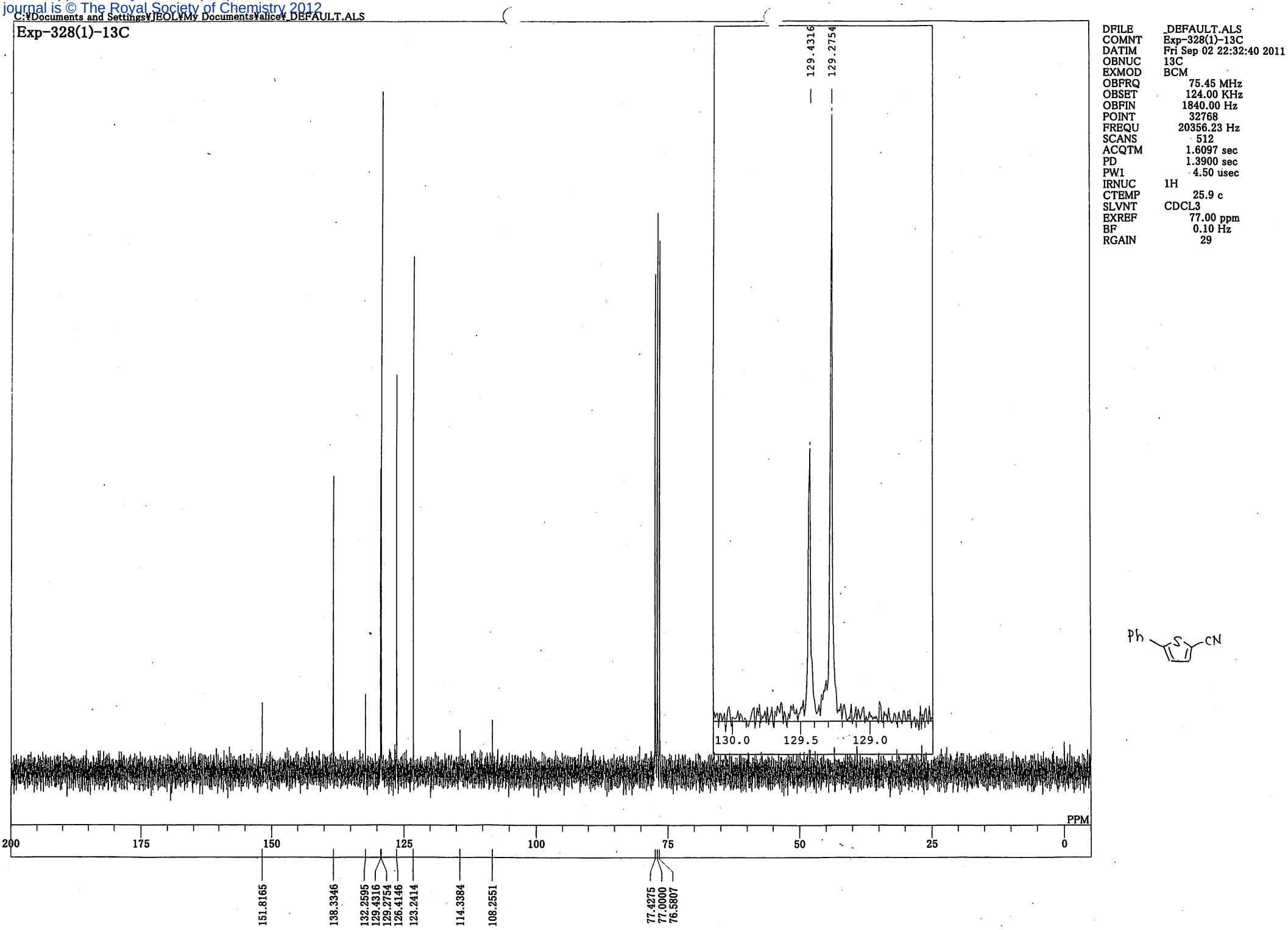


DFILE	_DEFAULT.ALS
COMNT	Exp-384(2)-13C
DATIM	Tue Nov 15 17:51:08 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	700
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	28

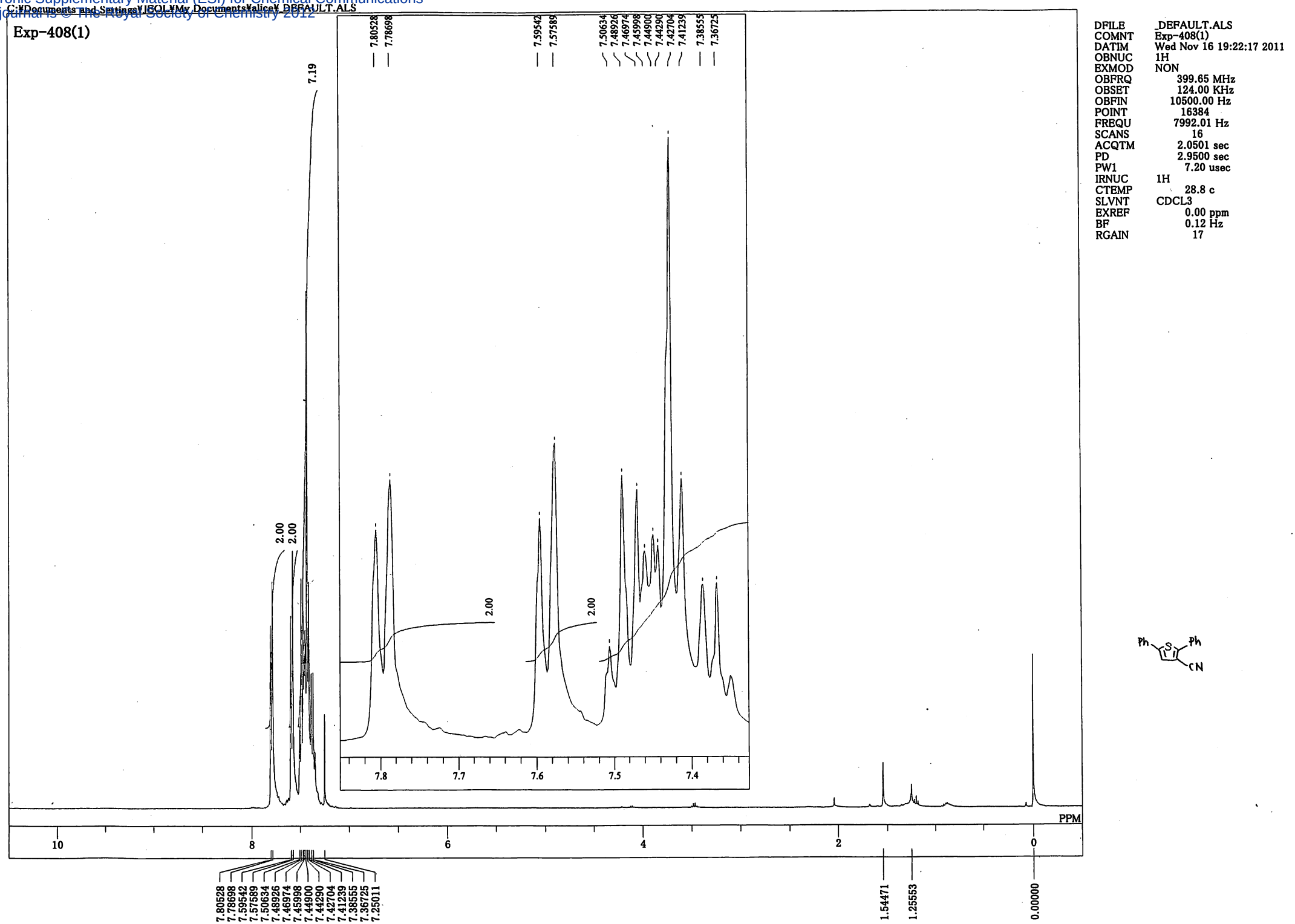


DFILE _DEFAULT.ALS
COMNT Exp-328crude(1)
DATIM Fri Sep 02 21:22:16 2011
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0501 sec
PD 2.9500 sec
PW1 7.20 usec
IRNUC 1H
CTEMP 29.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.20 Hz
RGAIN 19

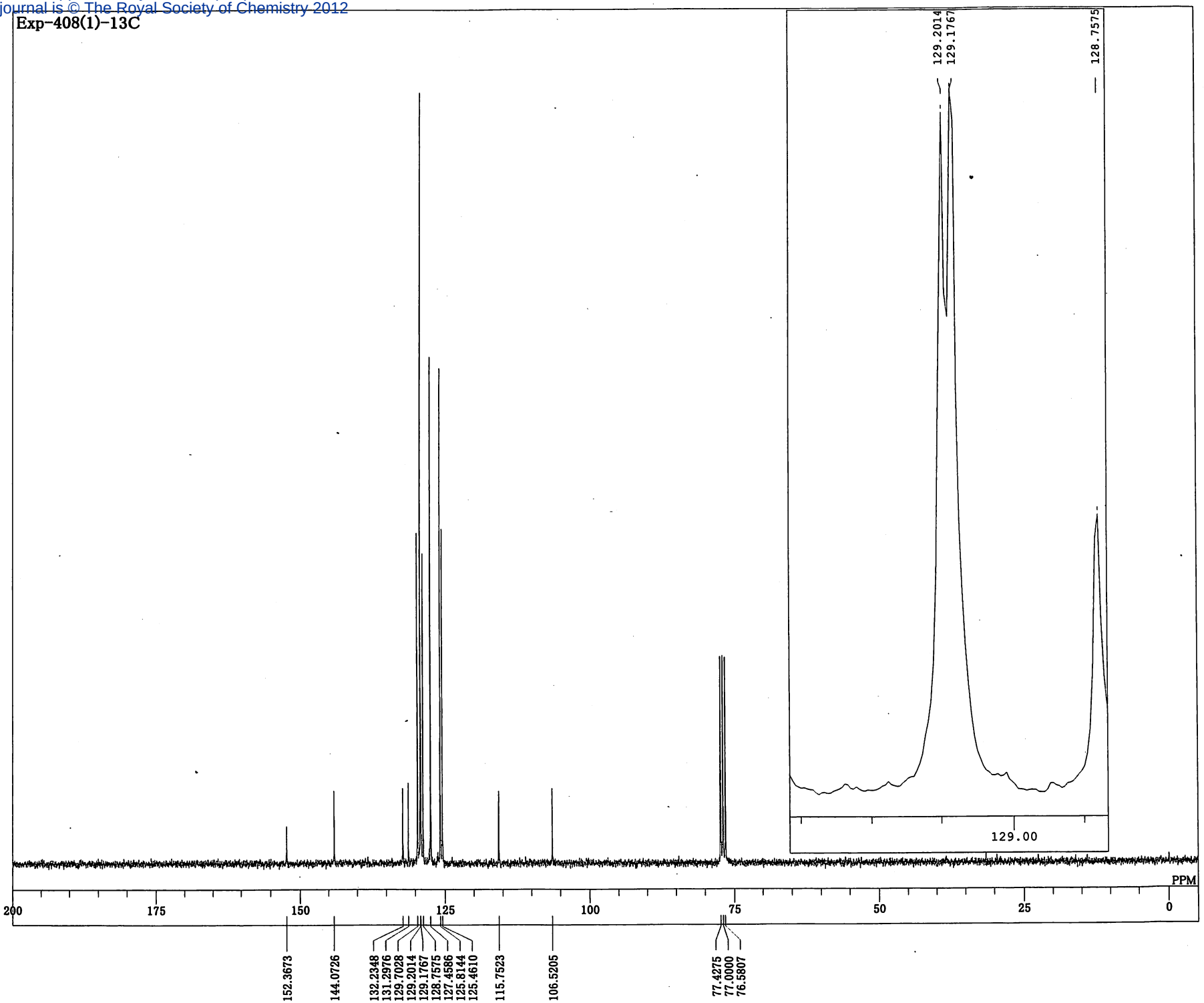




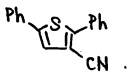
DFILE	_DEFAULT.ALS
COMNT	Exp-328(1)-13C
DATIM	Fri Sep 02 22:32:40 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	512
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	29

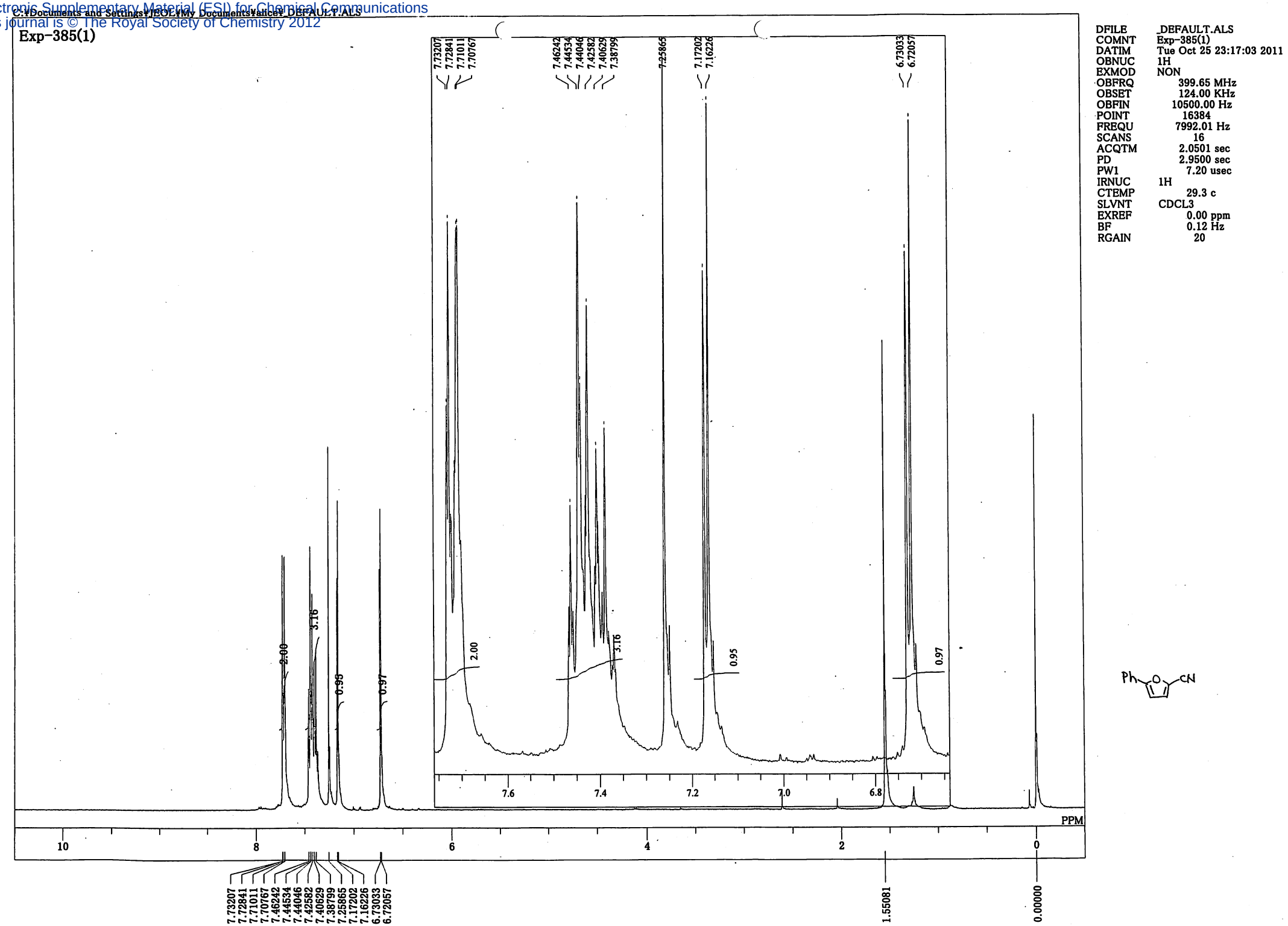


DFILE	_DEFAULT.ALS
COMNT	Exp-408(1)
DATIM	Wed Nov 16 19:22:17 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	28.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	17

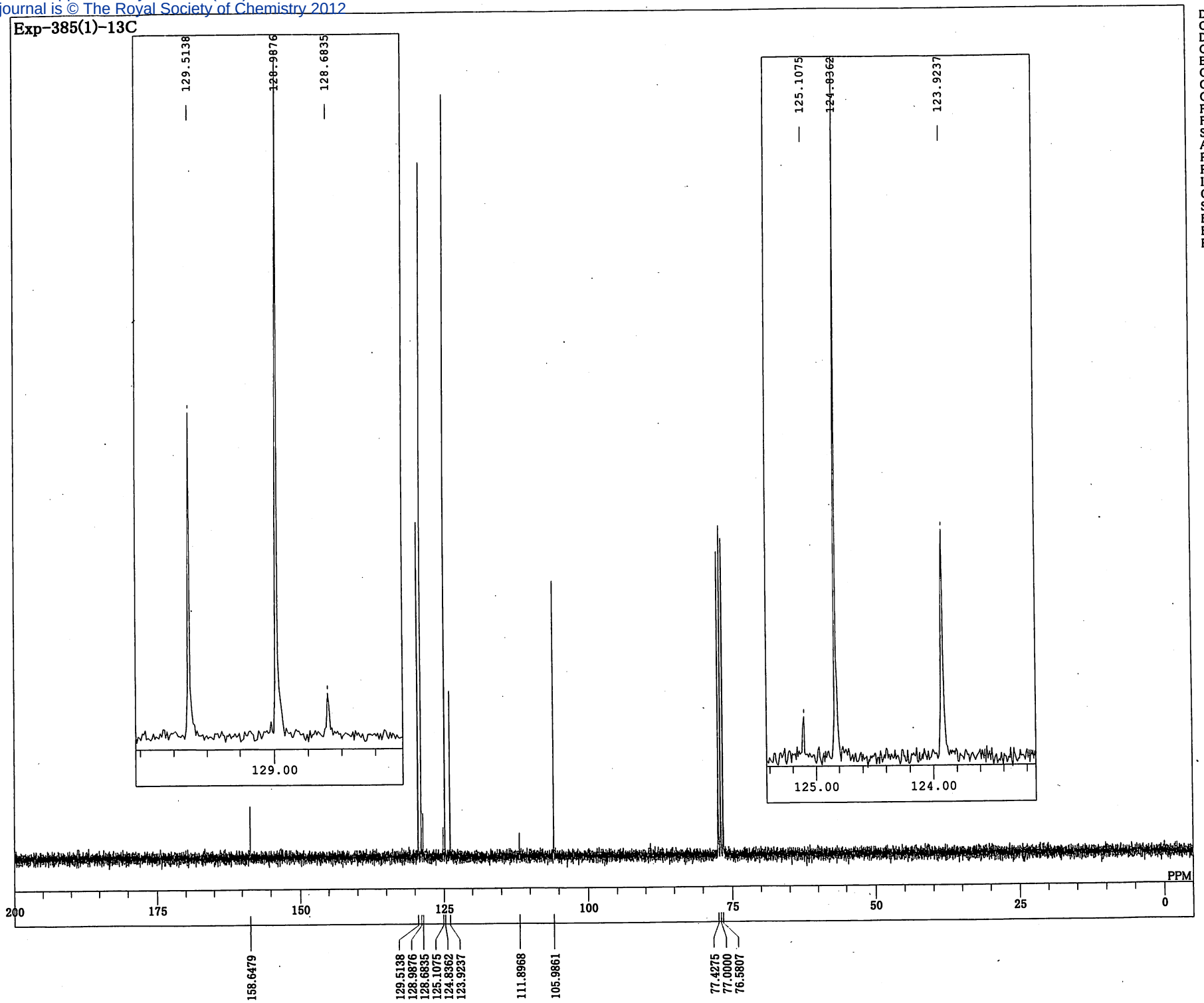


DFILE	_DEFAULT.ALS
COMNT	Exp-408(1)-13C
DATIM	Wed Nov 16 20:55:26 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1000
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	28

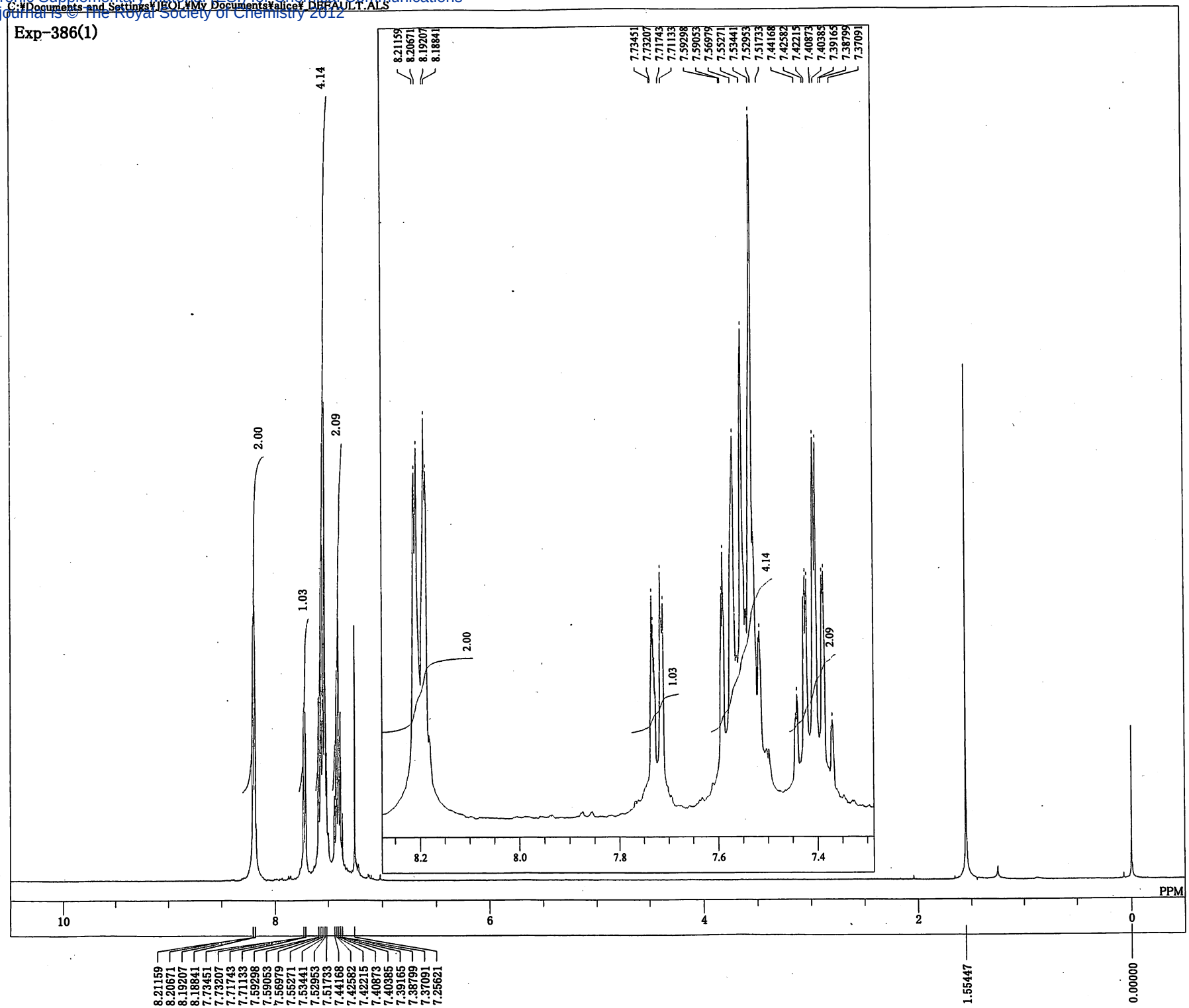




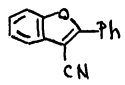
DFILE	_DEFAULT.ALS
COMNT	Exp-385(1)
DATIM	Tue Oct 25 23:17:03 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	29.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20

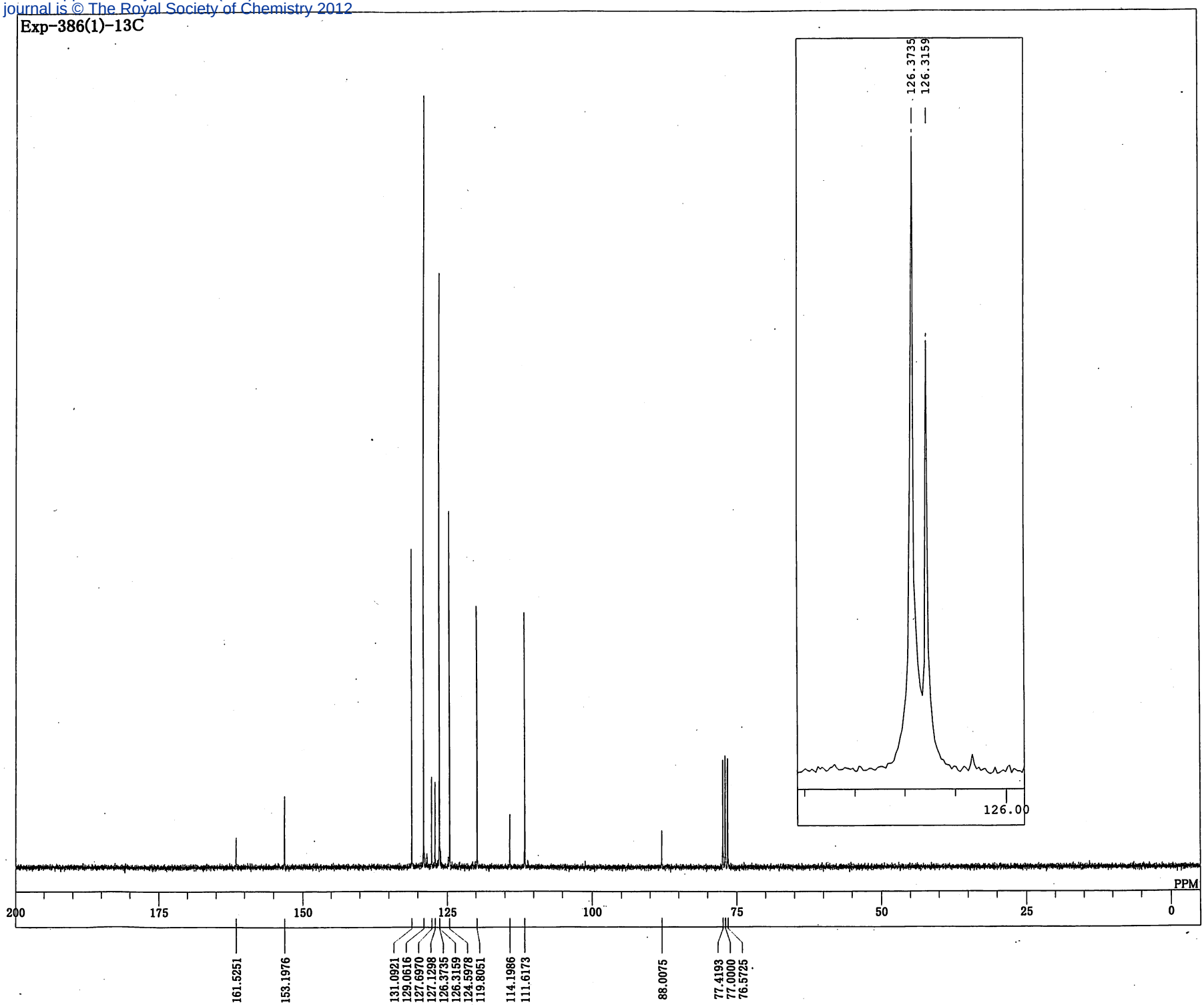


DFILE	_DEFAULT.ALS
COMNT	Exp-385(1)-13C
DATIM	Wed Oct 26 11:40:21 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	1024
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.10 Hz
RGAIN	28

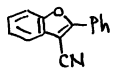


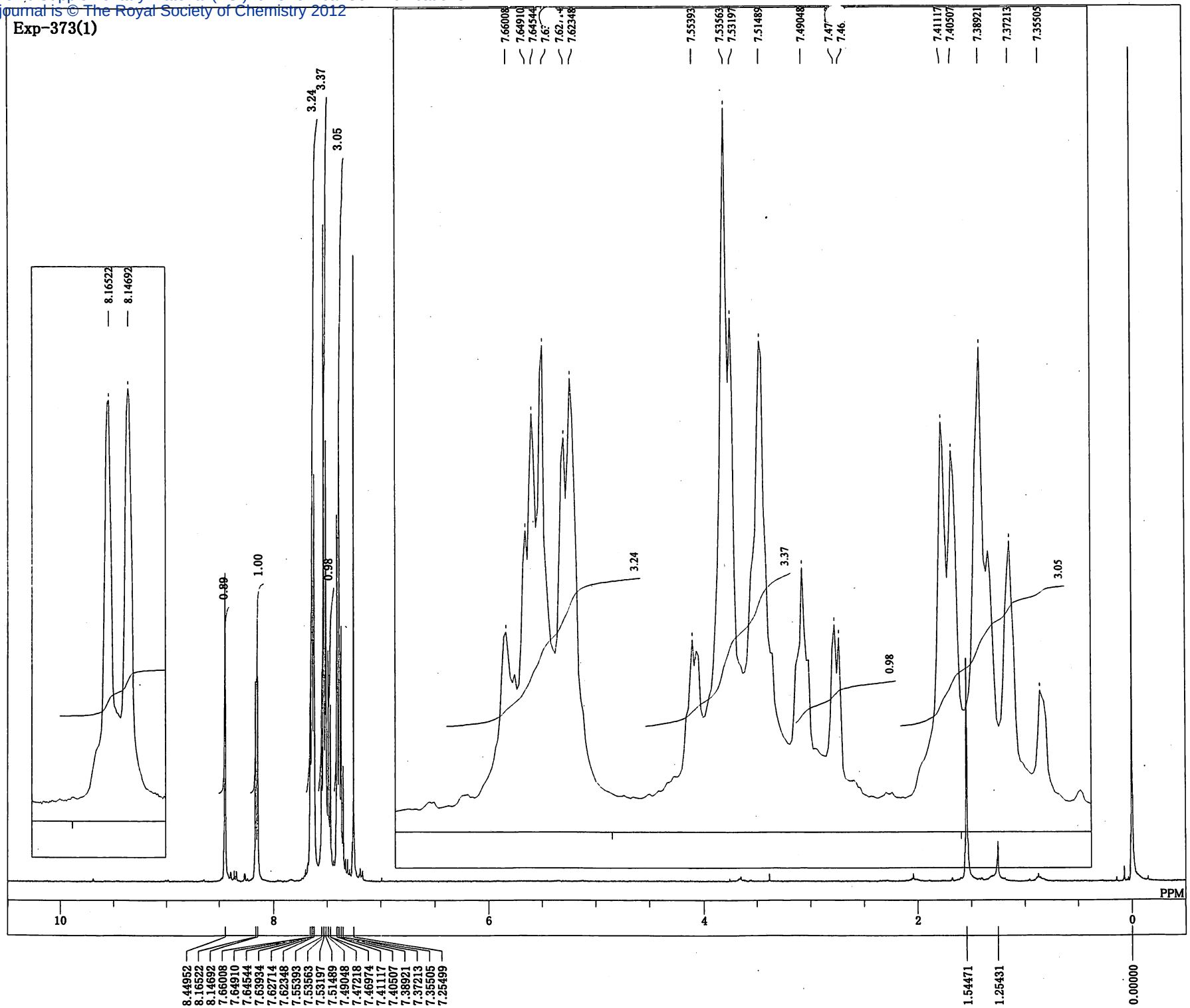
DFILE	DEFAULT.ALS
COMNT	Exp-386(1)
DATIM	Wed Nov 02 10:54:18 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	32
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	29.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	19



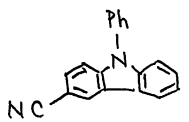


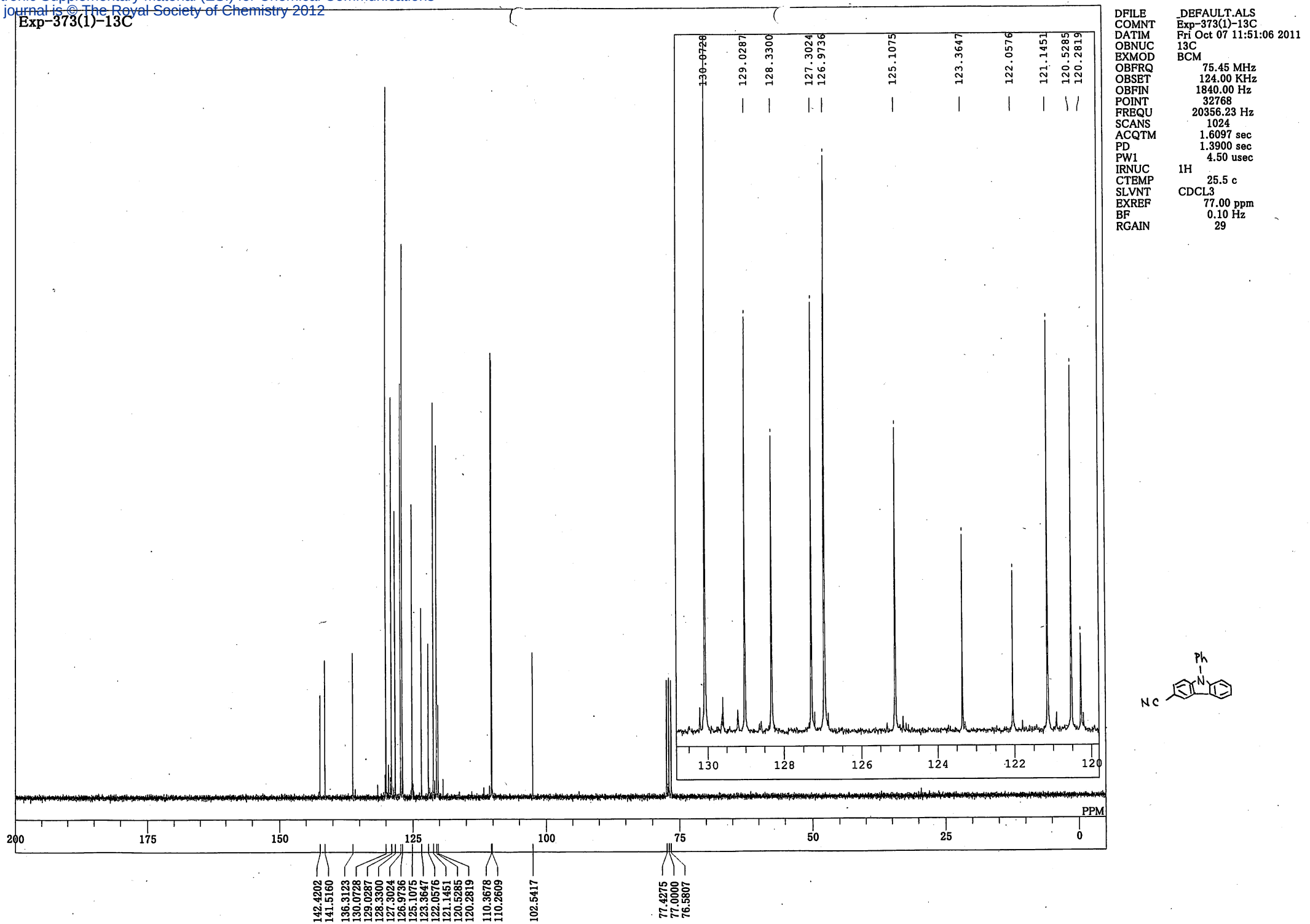
DFILE	Exp-386(1)-13C.als
COMNT	Exp-386(1)-13C
DATIM	Wed Nov 02 12:46:45 2011
OBNUC	13C
EXMOD	BCM
OBFRQ	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
POINT	32768
FREQU	20356.23 Hz
SCANS	800
ACQTM	1.6097 sec
PD	1.3900 sec
PW1	4.50 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	28





DFILE	_DEFAULT.ALS
COMNT	Exp-373(1)
DATIM	Thu Oct 06 20:00:19 2011
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	29.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20

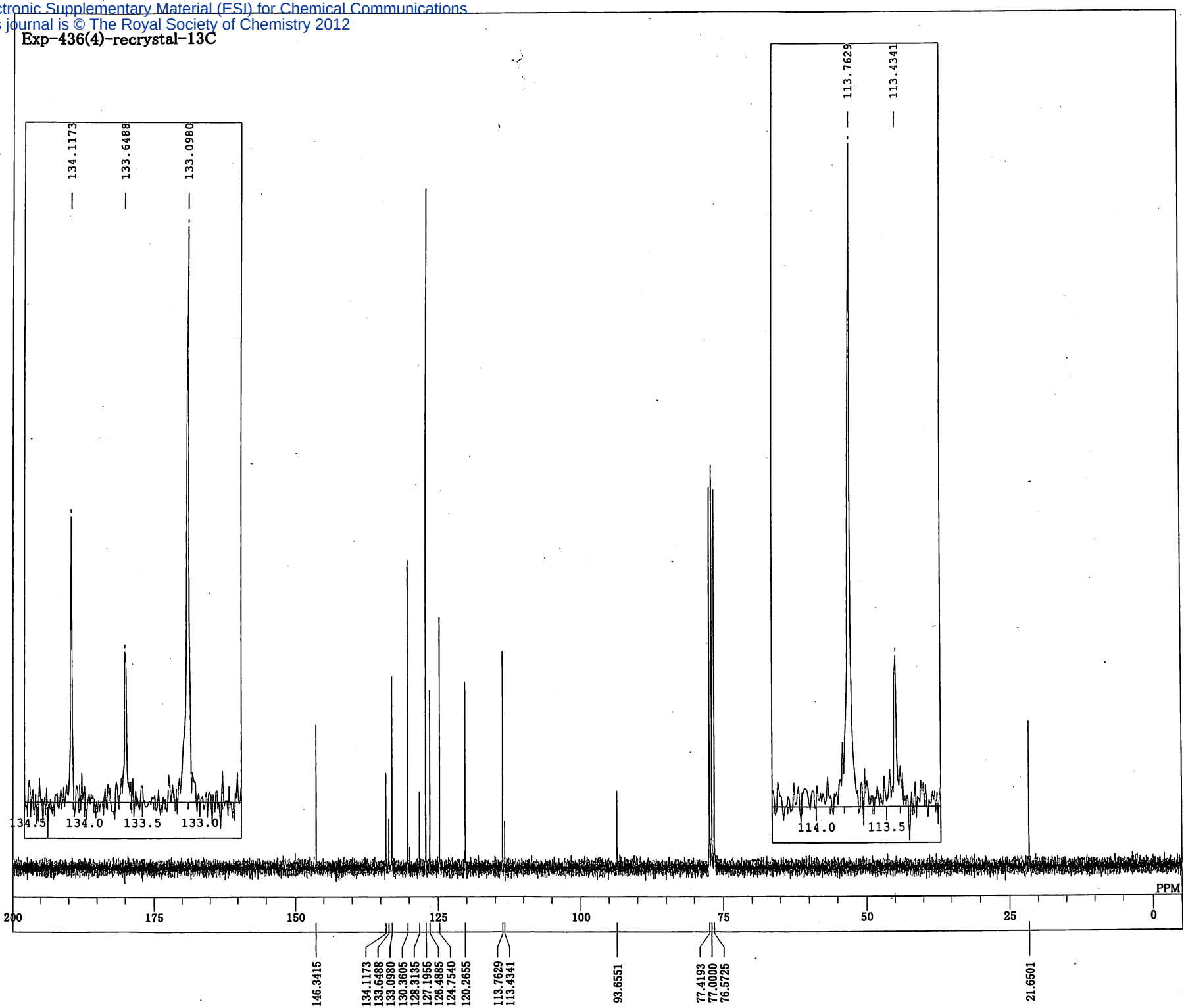




C:\Documents and Settings\JEOL\My Documents\alice_DEFAULT.ALS

Exp-436(4)-recrystal

DFILE	_DEFAULT.ALS
COMNT	Exp-436(4)-recrystal
DATIM	Wed Jan 25 15:42:02 2012
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0501 sec
PD	2.9500 sec
PW1	7.20 usec
IRNUC	1H
CTEMP	27.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	19



MENUF	BCM
OBNUC	13C
OFR	75.45 MHz
OBSET	124.00 KHz
OBFIN	1840.00 Hz
PW1	4.50 usec
DEADT	27.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000
DUMMY	1
FREQU	20356.23 Hz
FLT	10200 Hz
DELAY	19.60 usec
ACQTM	1.6097 sec
PD	1.3900 sec
ADBIT	16
RGAIN	28
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Se
IRNUC	1H
IFR	300.40 MHz
IRSET	130.00 KHz
IRFIN	1150.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5
LKSET	25.60 KHz
LKFIN	52.0 Hz
LKLEV	200
LGAIN	26
LKPHS	201
LKSIG	414
CSPED	12 Hz
FILDC	
FILDF