A Straightforward Access to Guaiazulene Derivatives using Palladium-Catalysed sp^2 or sp^3 C-H Bond Functionalisation

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General. All reactions were perfomed in Schlenck tubes under argon. DMAc or ethylbenzene analytical grade were not distilled before use. Potassium acetate 99+, cesium acetate and potassium carbonate 99% were used. Commercial guaiazulene and aryl bromides were used without purification. ^{1}H (400 or 500 MHz), ^{13}C (100 or 125 MHz) spectra were recorded in $CDCl_3$ solutions. Chemical shifts are reported in ppm relative to $CDCl_3$ (^{1}H: 7.29 and ^{13}C: 77.0). Flash chromatography was performed on silica gel (230-400 mesh) using pentane\ether.

General procedure for the synthesis of 1a-17a

As a typical experiment, reaction of the aryl bromide (1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) at 150 ^{o}C during 16 h in DMAc (5 mL) in the presence of $PdCl(C_3H_5)(dppb)$ (12.2 mg, 0.02 mmol) under argon afforded the corresponding arylation products after extraction with dichloromethane, evaporation and filtration on silica gel.

4-(7-Isopropyl-1-methylazulen-4-ylmethyl)-benzonitrile (1a)

From 4-bromobenzonitrile (0.182 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)(dppb)$ (12.2 mg, 0.02 mmol) **1a** was obtained in 61% (0.182 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (s, 1H), 7.58 (d, *J* = 3.2 Hz, 1H), 7.45 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 9.7 Hz, 1H), 7.29 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 3.2 Hz, 1H), 6.86 (d, *J* = 9.7 Hz, 1H), 4.46 (s, 2H), 3.02 (sept.,

J = 6.8 Hz, 1H), 2.60 (s, 3H), 1.29 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.2, 144.2, 140.7, 137.3, 137.2, 136.8, 135.1, 133.7, 132.3, 129.2, 126.0, 125.0, 119.0, 113.1, 110.1, 43.3, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{22}H_{21}N$ (299.41): C 88.25, H 7.07; found: C 88.10, H 7.14.

4-(7-Isopropyl-1-methylazulen-4-ylmethyl)-benzaldehyde (2a)

From 4-bromobenzaldehyde (0.185 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **2a** was obtained in 45% (0.136 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 9.87 (s, 1H), 8.15 (s, 1H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.59 (d, *J* = 3.2 Hz, 1H), 7.40-7.33 (m, 3H), 7.23 (d, *J* = 3.2 Hz, 1H), 6.89 (d, *J* = 10.7 Hz, 1H), 4.49 (s, 2H), 3.02 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.29 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.9, 147.9, 144.8, 140.5, 137.4, 137.1, 136.8, 135.1, 134.8, 133.7, 130.0, 129.2, 125.9, 125.1, 113.1, 43.5, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{22}H_{22}O$ (302.41): C 87.38, H 7.33; found: C 87.57, H 7.50.

1-[4-(7-Isopropyl-1-methylazulen-4-ylmethyl)-phenyl]-propan-1-one (3a)

From 4-bromopropiophenone (0.213 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **3a** was obtained in 46% (0.152 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.77 (d, *J* = 8.0 Hz, 2H), 7.58 (s, 1H), 7.35 (d, *J* = 10.7 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 1H), 7.17 (s, 1H), 6.89 (d, *J* = 10.7 Hz, 1H), 4.46 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.86 (q, *J* = 7.6 Hz, 2H), 2.60 (s, 3H), 1.29 (d, *J* = 6.8 Hz, 6H), 1.12 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 200.4, 146.0, 145.2, 140.4, 137.5, 137.0, 136.7, 135.1, 135.1, 133.6, 128.8, 128.3, 125.8, 125.1, 113.1, 43.2, 38.3, 24.7, 12.9, 8.3. Elemental analysis: calcd (%) for $C_{24}H_{26}O$ (330.46): C 87.23, H 7.93; found: C 87.14, H 7.99.

Ethyl 4-(7-isopropyl-1-methylazulen-4-ylmethyl)-benzoate (4a)

From ethyl 4-bromobenzoate (0.229 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **4a** was obtained in 67% (0.232 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.84 (d, *J* = 8.1 Hz, 2H), 7.57 (d, *J* = 3.5 Hz, 1H), 7.33 (d, *J* = 10.7 Hz, 1H), 7.28-7.22 (m, 3H), 6.87 (d, *J* = 10.7 Hz, 1H), 4.46 (s, 2H), 4.25 (q, *J* = 7.6 Hz, 2H), 3.00 (sept.,

J = 6.8 Hz, 1H), 2.60 (s, 3H), 1.30-1.25 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 145.9, 145.3, 140.3, 137.5, 137.0, 136.7, 135.1, 133.6, 129.8, 128.7, 128.6, 125.8, 125.1, 113.1, 60.8, 43.2, 38.3, 24.7, 14.3, 12.9. Elemental analysis: calcd (%) for $C_{24}H_{26}O_2$ (346.46): C 83.20, H 7.56; found: C 83.34, H 7.40.

7-Isopropyl-1-methyl-4-(4-trifluoromethylbenzyl)-azulene (5a)

From 4-(trifluoromethyl)bromobenzene (0.225 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **5a** was obtained in 68% (0.233 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.58 (d, *J* = 3.6 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.34 (d, *J* = 10.7 Hz, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.23 (d, *J* = 3.6 Hz, 1H), 6.87 (d, *J* = 10.7 Hz, 1H), 4.45 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.0, 144.7, 140.5, 137.5, 137.1, 136.8, 135.1, 133.7, 128.9, 128.6 (q, *J* = 32.2 Hz), 125.9, 125.3 (q, *J* = 3.7 Hz), 125.1, 124.2 (q, *J* = 271.9 Hz), 113.1, 43.0, 38.3, 24.7, 13.0. Elemental analysis: calcd (%) for $C_{22}H_{21}F_3$ (342.40): C 77.17, H 6.18; found: C 77.25, H 6.07.

4-(4-Chlorobenzyl)-7-isopropyl-1-methyl-azulene (6a)

From 4-bromochlorobenzene (0.191 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **6a** was obtained in 66% (0.203 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.58 (d, *J* = 3.6 Hz, 1H), 7.34 (d, *J* = 9.7 Hz, 1H), 7.24 (d, *J* = 3.6 Hz, 1H), 7.10 (s, 4H), 6.87 (d, *J* = 9.7 Hz, 1H), 4.38 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 140.3, 139.1, 137.4, 136.9, 136.7, 135.1, 133.5, 132.0, 130.0, 128.6, 125.7, 125.0, 113.0, 42.5, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{21}H_{21}Cl$ (308.84): C 81.67, H 6.85; found: C 81.40, H 6.99.

4-(4-Fluorobenzyl)-7-isopropyl-1-methyl-azulene (7a)

From 4-bromofluorobenzene (0.175 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **7a** was obtained in 52% (0.152 g) yield as a blue oil. Eluent pentane:diethylether 20:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 7.58 (d, *J* = 3.7 Hz, 1H), 7.34 (d, *J* = 9.3 Hz, 1H), 7.27 (d, *J* = 3.7 Hz, 1H), 7.17-7.12 (m, 2H), 6.90-6.80 (m, 3H), 4.39 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.7 (d, *J* = 246.5 Hz), 146.1, 140.2, 137.4, 136.9,

136.6, 136.2, 135.1, 133.5, 130.0 (d, *J* = 7.9 Hz), 125.7, 125.0, 115.2 (d, *J* = 21.3 Hz), 112.9, 42.4, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{21}H_{21}F$ (292.39): C 86.26, H 7.24; found: C 86.15, H 7.16.

7-Isopropyl-1-methyl-4-(4-methylbenzyl)-azulene (8a)

From 4-bromotoluene (0.171 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **8a** was obtained in 42% (0.121 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 7.57 (s, 1H), 7.32 (d, *J* = 10.7 Hz, 1H), 7.18 (s, 1H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 10.7 Hz, 1H), 4.39 (s, 2H), 2.99 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 2.21 (s, 3H), 1.27 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 139.9, 137.6, 137.5, 136.7, 136.5, 135.7, 135.1, 133.3, 129.1, 128.6, 125.5, 125.1, 112.9, 42.7, 38.2, 24.7, 21.0, 12.9. Elemental analysis: calcd (%) for $C_{22}H_{24}$ (288.43): C 91.61, H 8.39; found: C 91.75, H 8.30.

4-(4-*tert*-Butylbenzyl)-7-isopropyl-1-methyl-azulene (9a)

From 4-*tert*-butylbromobenzene (0.213 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **9a** was obtained in 48% (0.158 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 7.57 (d, *J* = 3.2 Hz, 1H), 7.35-7.25 (m, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 10.7 Hz, 1H), 4.39 (s, 2H), 2.98 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 1.27 (d, *J* = 6.8 Hz, 6H), 1.20 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.0, 146.8, 139.9, 137.7, 137.5, 136.7, 136.5, 135.2, 133.4, 128.3, 125.5, 125.4, 125.3, 113.0, 42.7, 38.3, 34.4, 31.4, 24.8, 13.0. Elemental analysis: calcd (%) for $C_{25}H_{30}$ (330.51): C 90.85, H 9.15; found: C 90.74, H 9.20.

3-(7-Isopropyl-1-methylazulen-4-ylmethyl)-benzonitrile (10a)

From 3-bromobenzonitrile (0.182 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **10a** was obtained in 54% (0.161 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (s, 1H), 7.59 (d, *J* = 3.6 Hz, 1H), 7.50-7.30 (m, 4H), 7.24 (t, *J* = 7.7 Hz, 1H), 7.20-7.15 (m, 1H), 6.87 (d, *J* = 10.7 Hz, 1H), 4.43 (s, 2H), 3.02 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.3, 142.1, 140.7, 137.3, 137.2, 136.9, 135.1, 133.8, 133.0, 132.0, 130.1, 129.2, 126.1, 124.9, 118.9, 113.0, 112.5, 42.8, 38.2, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{22}H_{21}N$ (299.41): C 88.25, H 7.07; found: C 88.01, H 7.17.

1-[3-(7-Isopropyl-1-methylazulen-4-ylmethyl)-phenyl]-ethanone (11a)

From 3-bromoacetophenone (0.199 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **11a** was obtained in 51% (0.161 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.86 (s, 1H), 7.67 (d, *J* = 7.7 Hz, 1H), 7.59 (d, *J* = 3.6 Hz, 1H), 7.30-7.20 (m, 4H), 6.9 (d, *J* = 10.7 Hz, 1H), 4.47 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 2.48 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.3, 145.5, 141.2, 140.3, 137.4, 137.3, 136.9, 136.8, 135.1, 133.6, 133.3, 128.7, 128.4, 126.5, 125.8, 125.0, 113.0, 43.1, 38.3, 26.7, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{23}H_{24}O$ (316.44): C 87.30, H 7.64; found: C 87.48, H 7.51.

7-Isopropyl-1-methyl-4-(3-trifluoromethyl-benzyl)-azulene (12a)

From 3-(trifluoromethyl)bromobenzene (0.225 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **12a** was obtained in 71% (0.243 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.58 (d, *J* = 1.6 Hz, 1H), 7.50 (s, 1H), 7.40-7.30 (m, 3H), 7.30-7.20 (m, 2H), 6.87 (d, *J* = 10.7 Hz, 1H), 4.46 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.0, 141.5, 140.4, 137.5, 137.1, 136.8, 135.1, 133.6, 132.0, 130.8 (q, *J* = 32.0 Hz), 128.9, 125.9, 125.3 (s, *J* = 3.8 Hz), 124.9, 124.1 (q, *J* = 272.3 Hz), 123.2 (q, *J* = 3.7 Hz), 113.0, 42.9, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{22}H_{21}F_3$ (342.40): C 77.17, H 6.18; found: C 77.45, H 6.24.

4-(3-Chlorobenzyl)-7-isopropyl-1-methyl-azulene (13a)

From 3-bromochlorobenzene (0.191 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **13a** was obtained in 69% (0.212 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.22 (s, 1H), 7.66 (d, *J* = 3.6 Hz, 1H), 7.42 (d, *J* = 10.7 Hz, 1H), 7.33 (d, *J* = 3.6 Hz, 1H), 7.27 (s, 1H), 7.20-7.15 (m, 3H), 6.96 (d, *J* = 10.7 Hz, 1H), 4.46 (s, 2H), 3.09 (sept., *J* = 6.8 Hz, 1H), 2.68 (s, 3H), 1.36 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.3, 142.6, 140.3, 137.5, 137.0, 136.7, 135.1, 134.2, 133.6, 129.7, 128.7, 126.9, 126.5, 125.8, 125.0, 113.0, 42.9, 38.3, 24.7, 13.0. Elemental analysis: calcd (%) for $C_{21}H_{21}Cl$ (308.84): C 81.67, H 6.85; found: C 81.79, H 6.70.

7-Isopropyl-1-methyl-4-(3-methylbenzyl)-azulene (14a)

From 3-bromotoluene (0.171 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **14a** was obtained in 54% (0.155 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 7.57 (s, 1H), 7.35-7.25 (m, 2H), 7.10-6.95 (m, 3H), 6.95-6.88 (m, 2H), 4.38 (s, 2H), 2.98 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 2.20 (s, 3H), 1.27 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.7, 140.5, 139.9, 138.1, 137.7, 136.8, 136.5, 135.1, 133.4, 129.5, 128.4, 127.0, 125.8, 125.5, 125.2, 113.0, 43.1, 38.3, 24.8, 21.4, 13.0. Elemental analysis: calcd (%) for $C_{22}H_{24}$ (288.43): C 91.61, H 8.39; found: C 91.77, H 8.57.

4-(2-Fluorobenzyl)-7-isopropyl-1-methyl-azulene (15a)

From 2-bromofluorobenzene (0.175 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **15a** was obtained in 63% (0.184 g) yield as a blue oil. Eluent pentane:diethylether 10:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, *J* = 2.0 Hz, 1H), 7.57 (s, 1H), 7.34 (dd, *J* = 10.4, 2.0 Hz, 1H), 7.15-6.80 (m, 6H), 4.44 (s, 2H), 2.99 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.8 (d, *J* = 246.0 Hz), 145.1, 140.3, 137.5, 136.9, 136.7, 135.0, 133.5, 130.6 (d, *J* = 4.2 Hz), 127.9 (d, *J* = 8.1 Hz), 127.2 (d, *J* = 15.7 Hz), 125.6, 124.7, 124.0 (d, *J* = 3.5 Hz), 115.2 (d, *J* = 22.3 Hz), 112.9, 38.3, 35.4, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{21}H_{21}F$ (292.39): C 86.26, H 7.24; found: C 86.44, H 7.51.

7-Isopropyl-1-methyl-4-naphthalen-2-ylmethylazulene (16a)

From 2-bromonaphthalene (0.207 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **16a** was obtained in 45% (0.146 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (s, 1H), 7.70-7.60 (m, 4H), 7.59 (d, *J* = 3.6 Hz, 1H), 7.40-7.28 (m, 5H), 6.94 (d, *J* = 10.7 Hz, 1H), 4.58 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.61 (s, 3H), 1.27 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.3, 140.1, 138.2, 137.7, 136.9, 136.6, 135.1, 133.6, 133.4, 132.2, 128.1, 127.7, 127.6, 127.3, 127.0, 125.9, 125.6, 125.4, 125.2, 113.0, 43.3, 38.3, 24.7, 13.0. Elemental analysis: calcd (%) for $C_{25}H_{24}$ (324.46): C 92.54, H 7.46; found: C 92.67, H 7.66.

3-(7-Isopropyl-1-methylazulen-4-ylmethyl)-pyridine (17a)

From 3-bromopyridine (0.158 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), CsOAc (0.767 g, 4 mmol) and K_2CO_3 (0.552 g, 4 mmol) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **17a** was obtained in 61% (0.168 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 8.35 (d, *J* = 3.8 Hz, 1H), 8.14 (s, 1H), 7.59 (d, *J* = 3.6 Hz, 1H), 7.44 (d, *J* = 7.7 Hz, 1H), 7.35 (d, *J* = 10.7 Hz, 1H), 7.25 (d, *J* = 3.6 Hz, 1H), 7.04 (dd, *J* = 7.7, 4.0 Hz, 1H), 6.88 (d, *J* = 10.7 Hz, 1H), 4.40 (s, 2H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.9, 147.8, 144.8, 140.5, 137.3, 137.1, 136.8, 136.1, 135.8, 135.1, 133.6, 125.9, 124.9, 123.4, 113.0, 40.4, 38.3, 24.7, 12.9. Elemental analysis: calcd (%) for $C_{20}H_{21}N$ (275.39): C 87.23, H 7.69; found: C 87.45, H 7.87.

General procedure for the synthesis of 1b, 6b-8b, 14b and 18b

As a typical experiment, reaction of the aryl bromide (1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), and KOAc (0.784 g, 8 mmol) at 150 °C during 16 h in ethylbenzene (5 mL) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) under argon afforded the corresponding arylation product after extraction with dichloromethane, evaporation and filtration on silica gel.

4-(7-Isopropyl-1,4-dimethylazulen-2-yl)-benzonitrile (1b)

From 4-bromobenzonitrile (0.182 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **1b** was obtained in 38% (0.114 g) yield (containing around 10% of **1c**) as a blue oil. Eluent pentane:diethylether 100:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (s, 1H), 7.67 (s, 4H), 7.37 (d, *J* = 10.6 Hz, 1H), 7.25 (bs, 1H), 7.01 (d, *J* = 10.6 Hz, 1H), 3.04 (sept., *J* = 6.8 Hz, 1H), 2.81 (s, 3H), 2.63 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.6, 145.2, 143.3, 141.5, 137.9, 136.8, 135.6, 134.4, 132.1, 130.2, 126.3, 121.6, 119.2, 113.2, 110.3, 38.4, 24.8, 24.2, 11.6. Elemental analysis: calcd (%) for $C_{22}H_{21}N$ (299.41): C 88.25, H 7.07; found: C 88.20, H 7.21.

2-(4-Chlorophenyl)-7-isopropyl-1,4-dimethylazulene (6b)

From 4-bromochlorobenzene (0.191 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **6b** was obtained in 53% (0.163 g) yield as a blue oil. Eluent pentane:diethylether 100:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18 (s, 1H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 10.6 Hz, 1H), 7.24 (s, 1H), 6.99 (d, *J* = 10.6 Hz, 1H), 3.02 (sept., *J* = 6.8 Hz, 1H), 2.77 (s, 3H), 2.62 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 144.2, 141.1, 137.8, 136.9, 136.7, 134.7, 133.6, 133.0, 130.9, 128.6, 126.0, 121.3, 113.2, 38.4, 24.8, 24.2, 11.6. Elemental analysis: calcd (%) for $C_{21}H_{21}Cl$ (308.84): C 81.67, H 6.85; found: C 81.68, H 6.97.

2-(4-Fluorophenyl)-7-isopropyl-1,4-dimethylazulene (7b)

From 4-bromofluorobenzene (0.175 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **7b** was obtained in 51% (0.149 g) yield as a blue oil. Eluent pentane:diethylether 100:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18 (s, 1H), 7.55 (dd, *J* = 7.9, 5.6 Hz, 2H), 7.33 (d, *J* = 10.6 Hz, 1H), 7.24 (s, 1H), 7.10 (t, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 10.6 Hz, 1H), 3.02 (sept., *J* = 6.8 Hz, 1H), 2.78 (s, 3H), 2.62 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.2 (d, *J* = 246.5 Hz), 147.2, 143.9, 141.0, 137.8, 136.7, 134.5, 134.4, 133.4, 131.1 (d, *J* = 7.9 Hz), 125.9, 121.2, 115.3 (d, *J* = 21.3 Hz), 113.3, 38.4, 24.8, 24.2, 11.6. Elemental analysis: calcd (%) for $C_{21}H_{21}F$ (292.39): C 86.26, H 7.24; found: C 86.48, H 7.34.

7-Isopropyl-1,4-dimethyl-2-*p*-tolylazulene (8b)

From 4-bromotoluene (0.171 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **8b** was obtained in 56% (0.161 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (s, 1H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 10.6 Hz, 1H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.20 (s, 1H), 6.97 (d, *J* = 10.6 Hz, 1H), 3.03 (sept., *J* = 6.8 Hz, 1H), 2.78 (s, 3H), 2.65 (s, 3H), 2.36 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 143.5, 140.8, 137.8, 136.8, 136.6, 135.5, 134.2, 133.1, 129.6, 129.1, 125.7, 121.3, 113.5, 38.4, 24.8, 24.2, 21.3, 11.6. Elemental analysis: calcd (%) for $C_{22}H_{24}$ (288.43): C 91.61, H 8.39; found: C 91.78, H 8.24.

7-Isopropyl-1,4-dimethyl-2-*m*-tolylazulene (14b)

From 3-bromotoluene (0.171 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **14b** was obtained in 54% (0.156 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 1.7 Hz, 1H), 7.45-7.25 (m, 5H), 7.31 (d, *J* = 10.6 Hz, 1H), 7.10 (d, *J* = 7.1 Hz, 1H), 6.97 (d, *J* = 10.6 Hz, 1H), 3.03 (sept., *J* = 6.8 Hz, 1H), 2.78 (s, 3H), 2.64 (s, 3H), 2.38 (s,

3H), 1.30 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.5, 143.7, 140.8, 138.4, 137.9, 137.8, 136.6, 134.3, 133.3, 130.4, 128.2, 127.8, 126.8, 125.7, 121.4, 113.7, 38.4, 24.8, 24.2, 21.6, 11.6. Elemental analysis: calcd (%) for $C_{22}H_{24}$ (288.43): C 91.61, H 8.39; found: C 91.79, H 8.49.

2-(4-Phenyl)-7-isopropyl-1,4-dimethylazulene (18b)

From bromobenzene (0.157 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in ethylbenzene in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **18b** was obtained in 56% (0.153 g) yield as a blue oil. Eluent pentane

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18 (s, 1H), 7.59 (d, *J* = 8.5 Hz, 2H), 7.41 (t, *J* = 8.5 Hz, 2H), 7.33-7.25 (m, 3H), 6.98 (d, *J* = 10.6 Hz, 1H), 3.03 (sept., *J* = 6.8 Hz, 1H), 2.78 (s, 3H), 2.65 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 143.8, 140.9, 138.5, 137.8, 136.7, 134.4, 133.4, 129.7, 128.4, 127.0, 125.8, 121.4, 113.6, 38.4, 24.8, 24.2, 11.6. Elemental analysis: calcd (%) for $C_{21}H_{22}$ (274.40): C 91.92, H 8.08; found: C 92.02, H 7.99.

General procedure for the synthesis of 1c and 14c

As a typical experiment, reaction of the aryl bromide (1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), and KOAc (0.784 g, 8 mmol) at 150 ^{o}C during 16 h in DMAc (5 mL) in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) under argon afforded the corresponding arylation product after extraction with dichloromethane, evaporation and filtration on silica gel.

4-(5-Isopropyl-3,8-dimethylazulen-1-yl)-benzonitrile (1c)

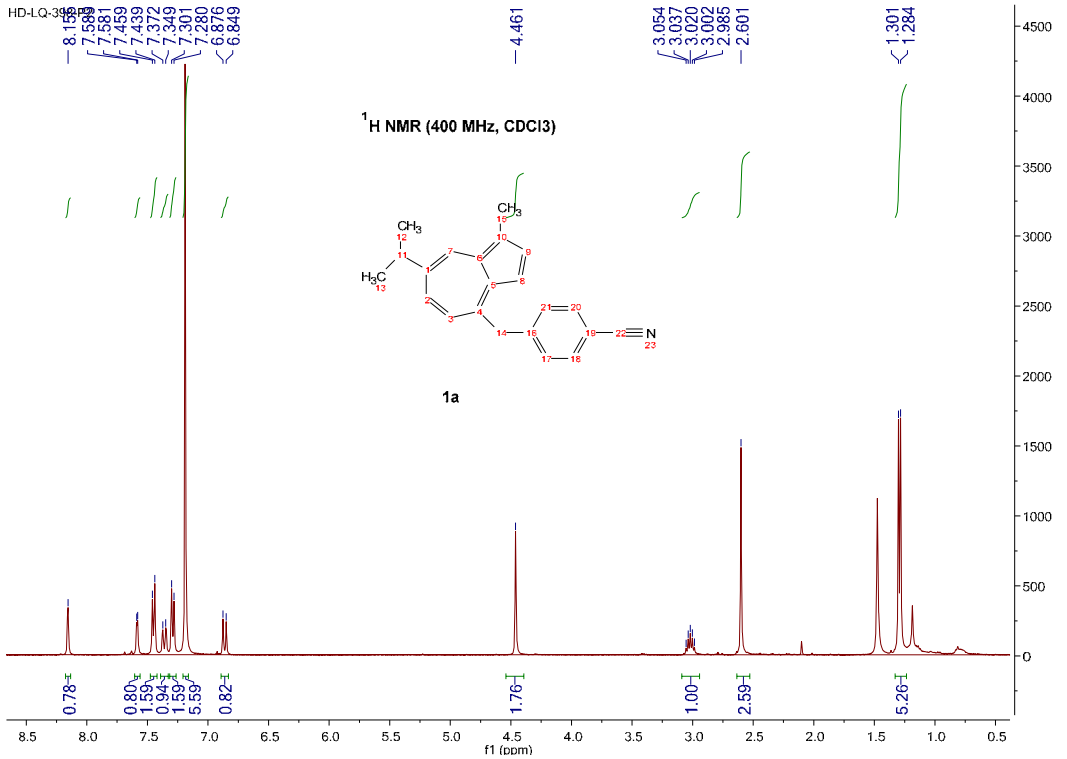
From 4-bromobenzonitrile (0.182 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in DMAc in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **1c** was obtained in 51% (0.153 g) yield as a green oil. Eluent pentane:diethylether 100:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (d, *J* = 2.1 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.46 (s, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.36 (dd, *J* = 10.7, 2.1 Hz, 1H), 6.92 (d, *J* = 10.7 Hz, 1H), 3.02 (sept., *J* = 6.8 Hz, 1H), 2.59 (s, 3H), 2.33 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.4, 145.7, 141.0, 139.7, 138.5, 135.3, 134.2, 132.2, 131.1, 131.0, 127.7, 127.0, 124.7, 119.4, 109.2, 37.9, 27.9, 24.6, 12.8. Elemental analysis: calcd (%) for $C_{22}H_{21}N$ (299.41): C 88.25, H 7.07; found: C 88.24, H 7.25.

7-Isopropyl-1,4-dimethyl-3-*m*-tolylazulene (14c)

From 3-bromotoluene (0.171 g, 1 mmol), 7-isopropyl-1,4-dimethylazulene (0.296 g, 1.5 mmol), KOAc (0.784 g, 8 mmol) in DMAc in the presence of $PdCl(C_3H_5)$(dppb) (12.2 mg, 0.02 mmol) **14c** was obtained in 38% (0.110 g) yield as a green oil. Eluent pentane:diethylether 100:1

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26 (d, *J* = 2.1 Hz, 1H), 7.47 (s, 1H), 7.28 (dd, *J* = 10.7, 2.1 Hz, 1H), 7.22-7.03 (m, 4H), 6.82 (d, *J* = 10.7 Hz, 1H), 3.00 (sept., *J* = 6.8 Hz, 1H), 2.60 (s, 3H), 2.34 (s, 3H), 2.32 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 141.4, 140.1, 139.6, 137.7, 136.6, 134.8, 133.7, 131.6, 131.5, 129.5, 127.8, 127.1, 126.7, 126.6, 124.0, 37.9, 27.6, 24.7, 21.5, 12.8. Elemental analysis: calcd (%) for $C_{22}H_{24}$ (288.43): C 91.61, H 8.39; found: C 91.87, H 8.17.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3.054
3.037
3.020
3.002
2.985
2.601
1.301
1.284
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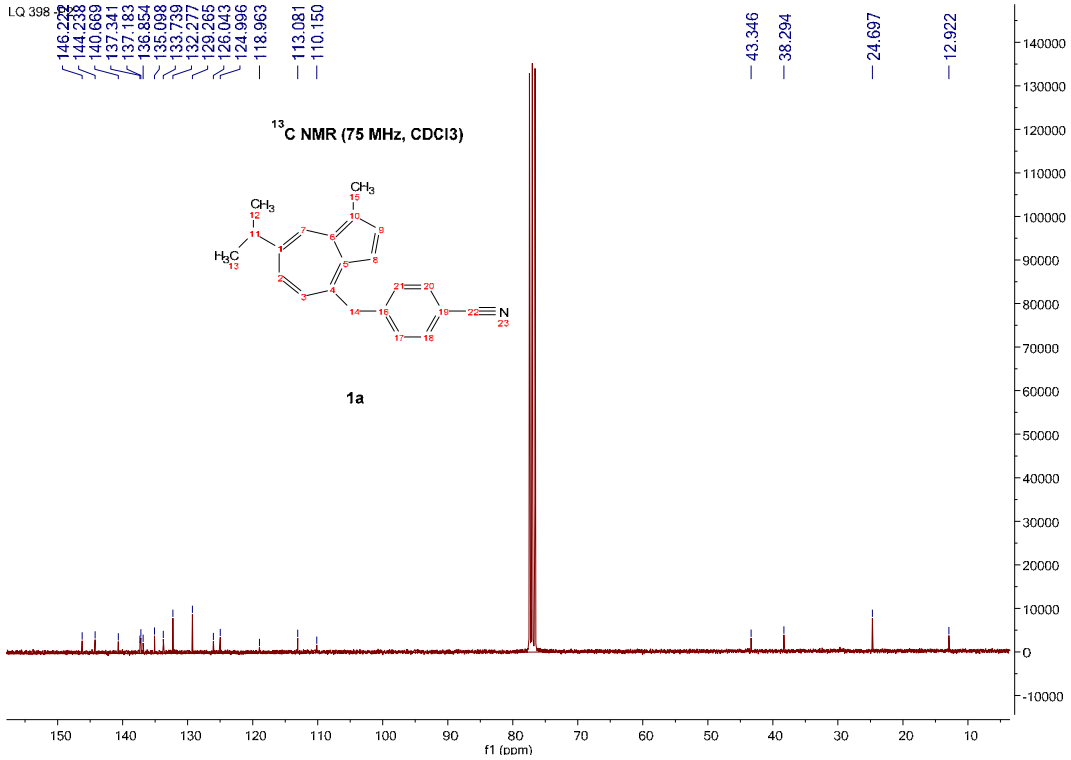
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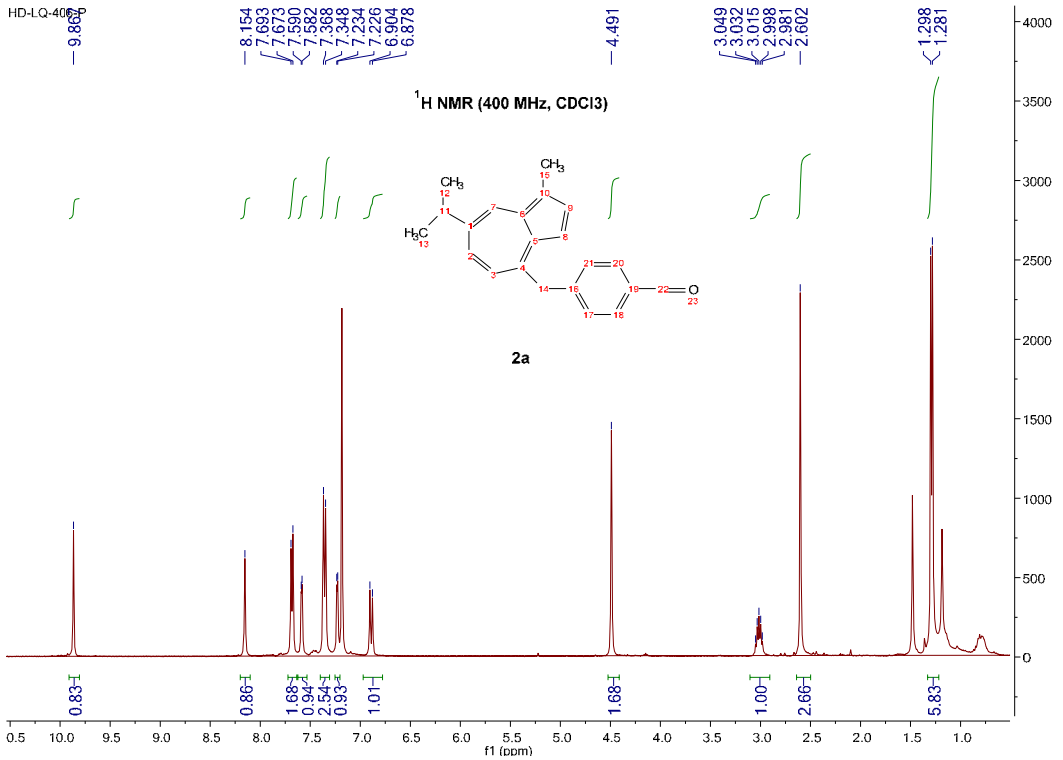
1a
f1 (ppm)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4.491
3.049
3.032
3.015
2.998
2.981
2.602
1.298
1.281
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2a
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1.00
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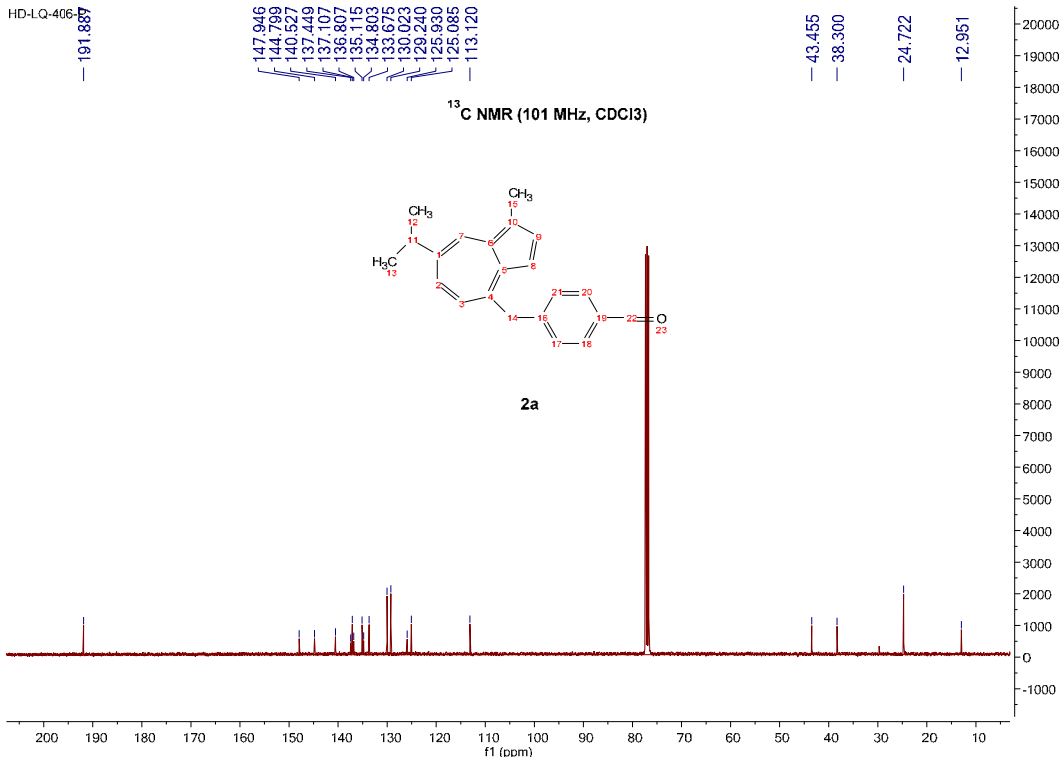
f1 (ppm)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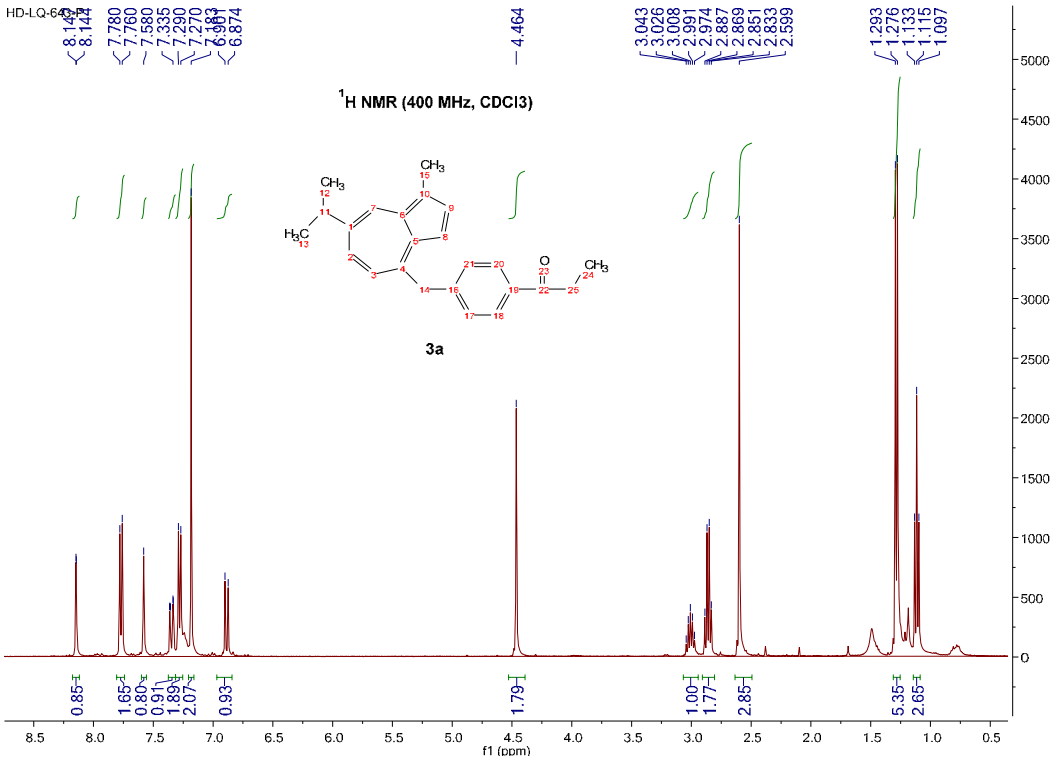
2a
f1 (ppm)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1.293
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1.133
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^{1}H NMR (400 MHz, CDCl3)
3a
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1.65
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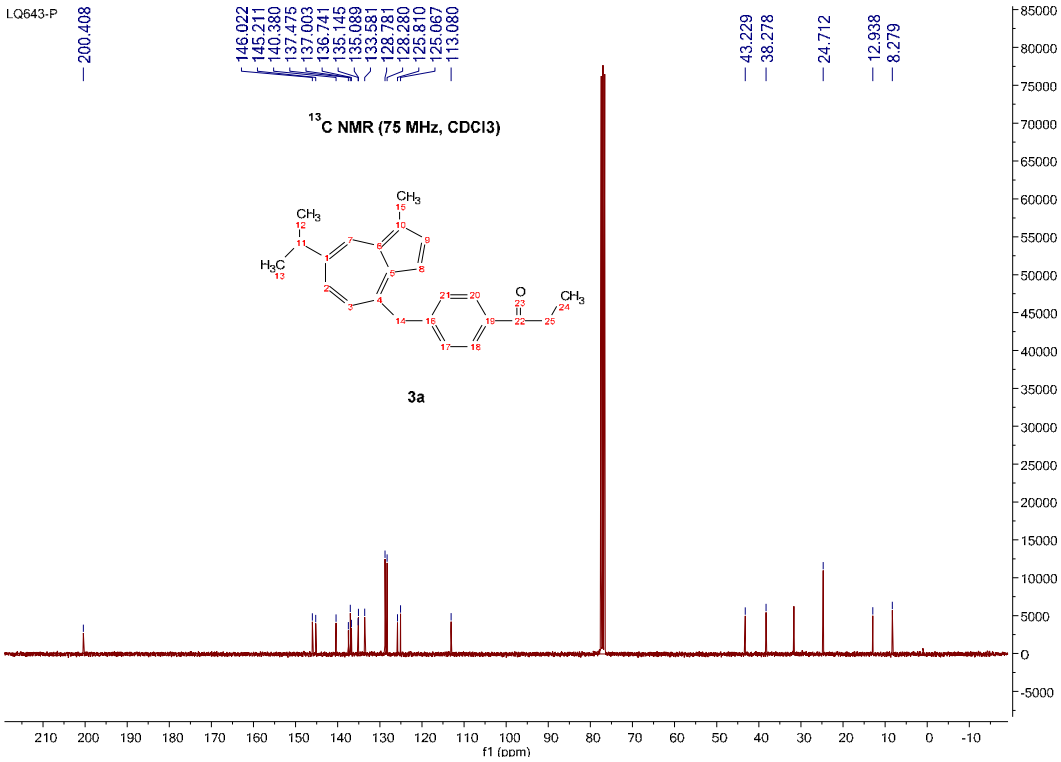
f1 (ppm)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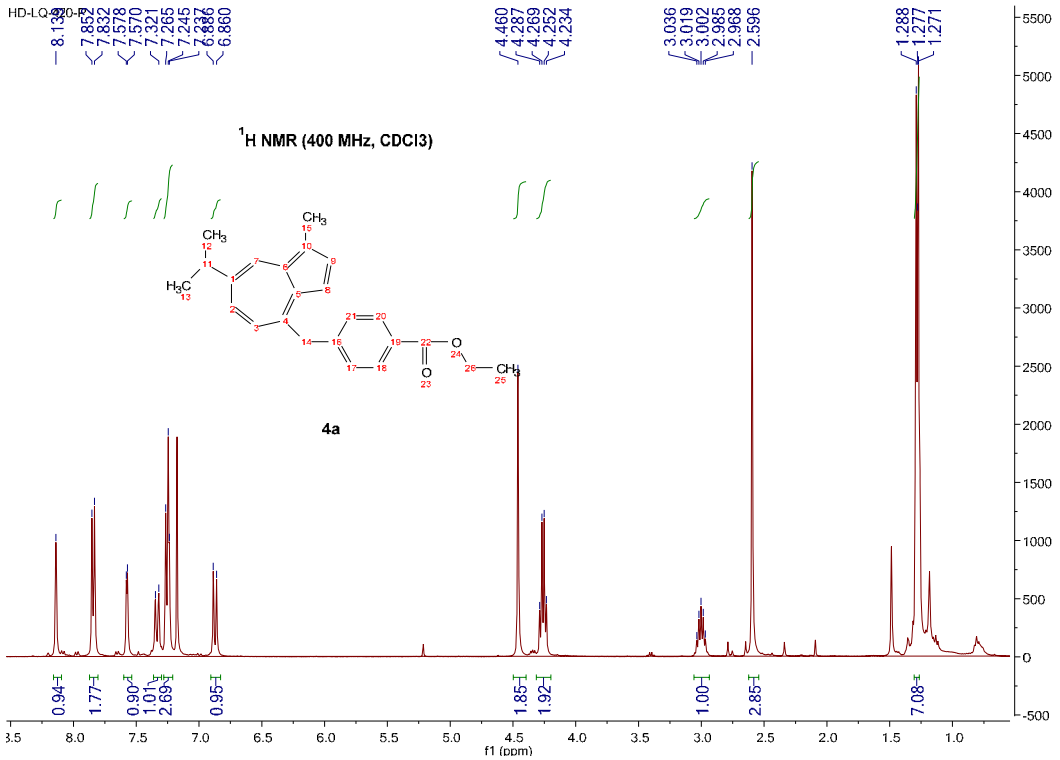
^{13}C NMR (75 MHz, CDCl3)
3a
f1 (ppm)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4.252
4.234
3.036
3.019
3.002
2.985
2.968
2.596
1.288
1.277
1.271
^{1}H NMR (400 MHz, CDCl3)
4a
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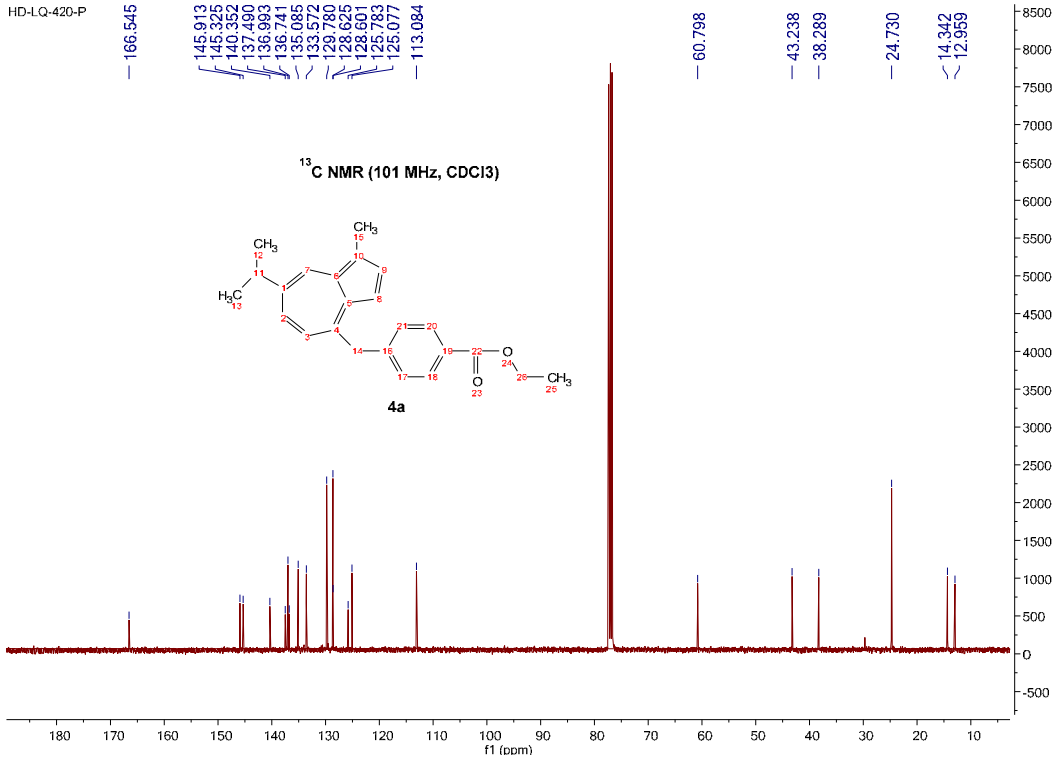
1.00
2.85
7.08
f1 (ppm)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128.601
125.783
125.077
113.084
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14.342
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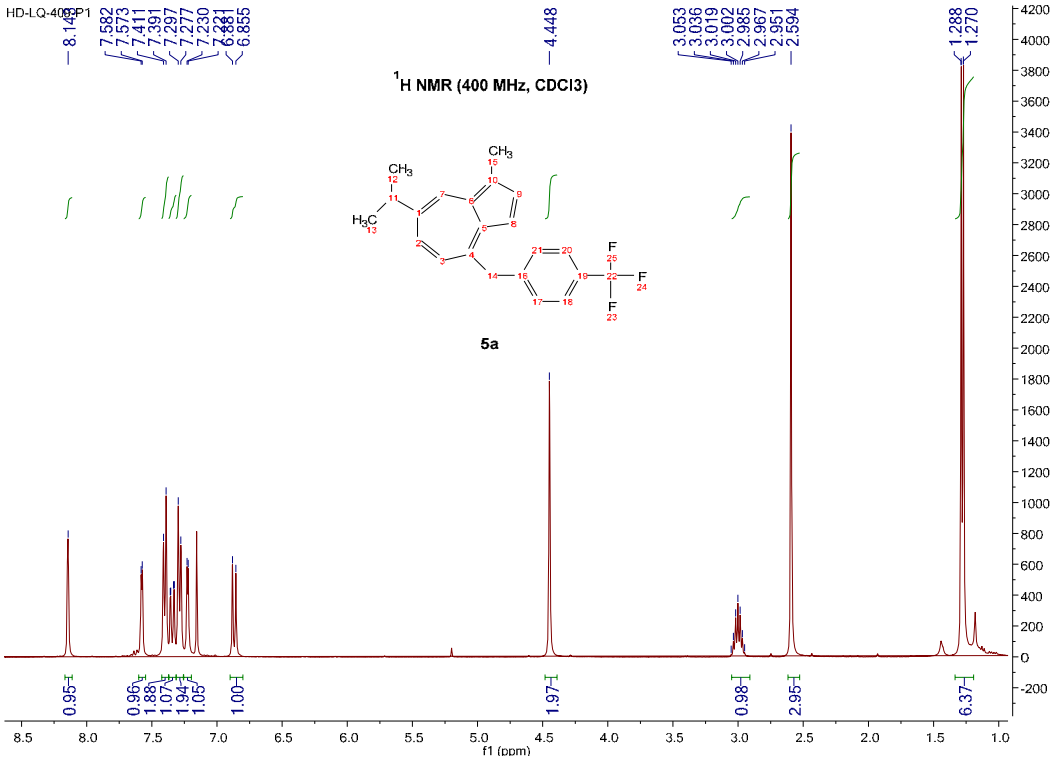
^{13}C NMR (101 MHz, CDCl3)
4a
f1 (ppm)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3.053
3.036
3.019
3.002
2.985
2.967
2.951
2.594
1.288
1.270
^{1}H NMR (400 MHz, CDCl3)
5a
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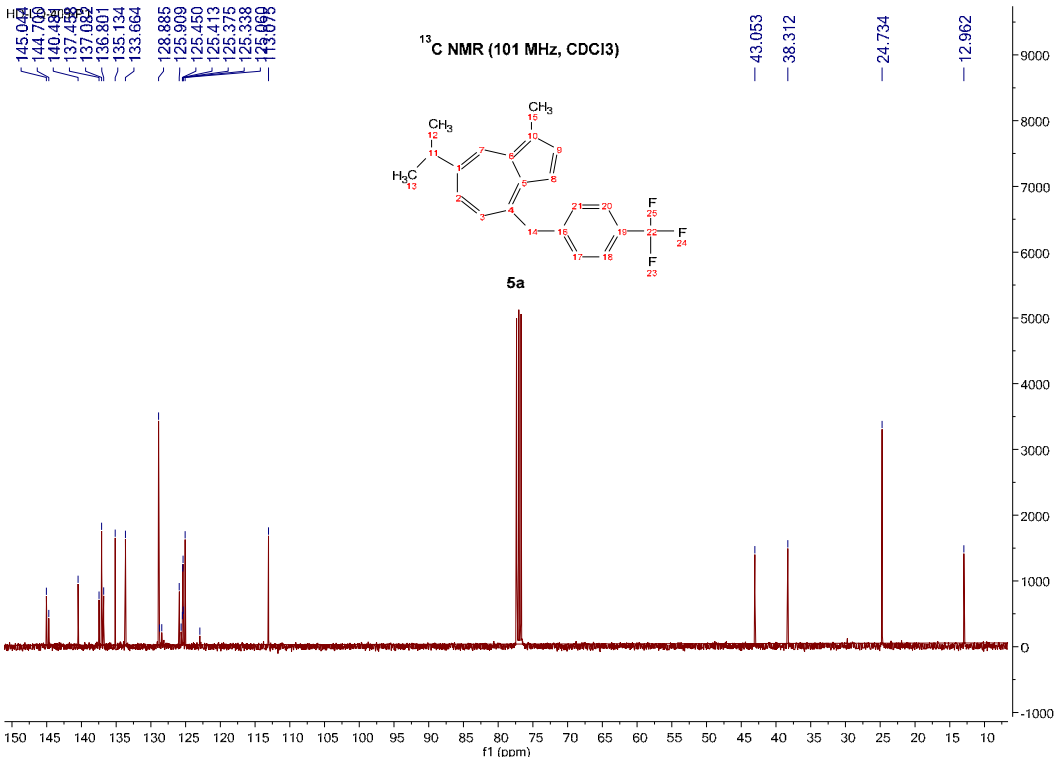
6.37
f1 (ppm)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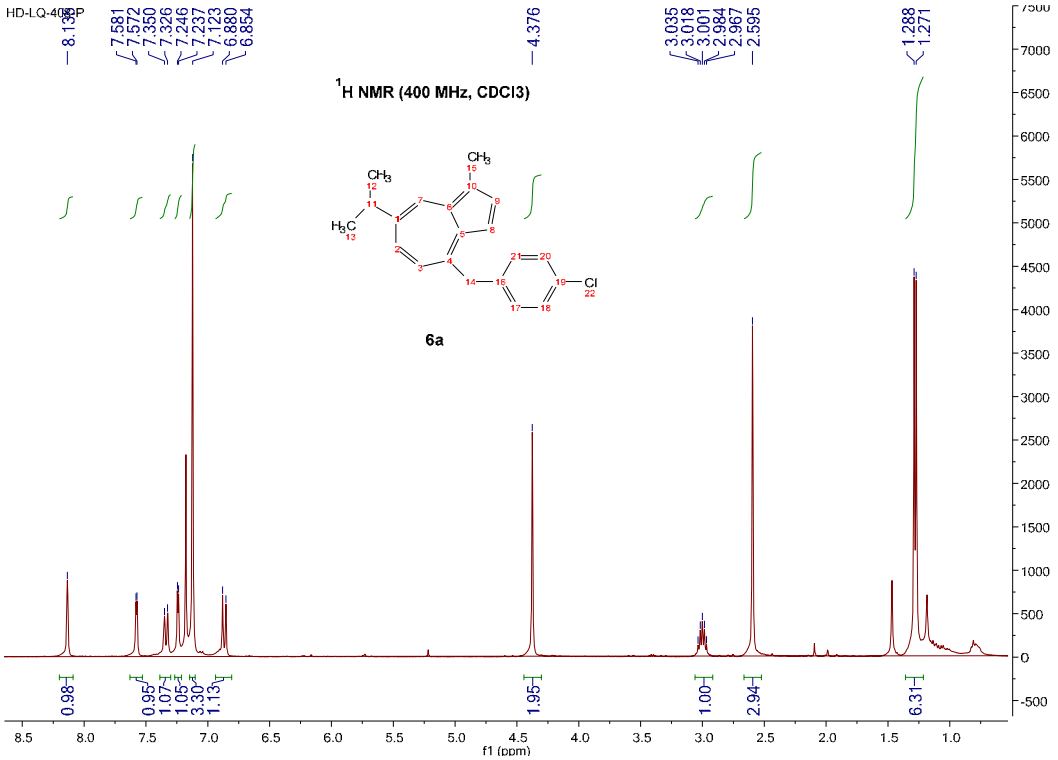
^{13}C NMR (101 MHz, CDCl3)
5a
f1 (ppm)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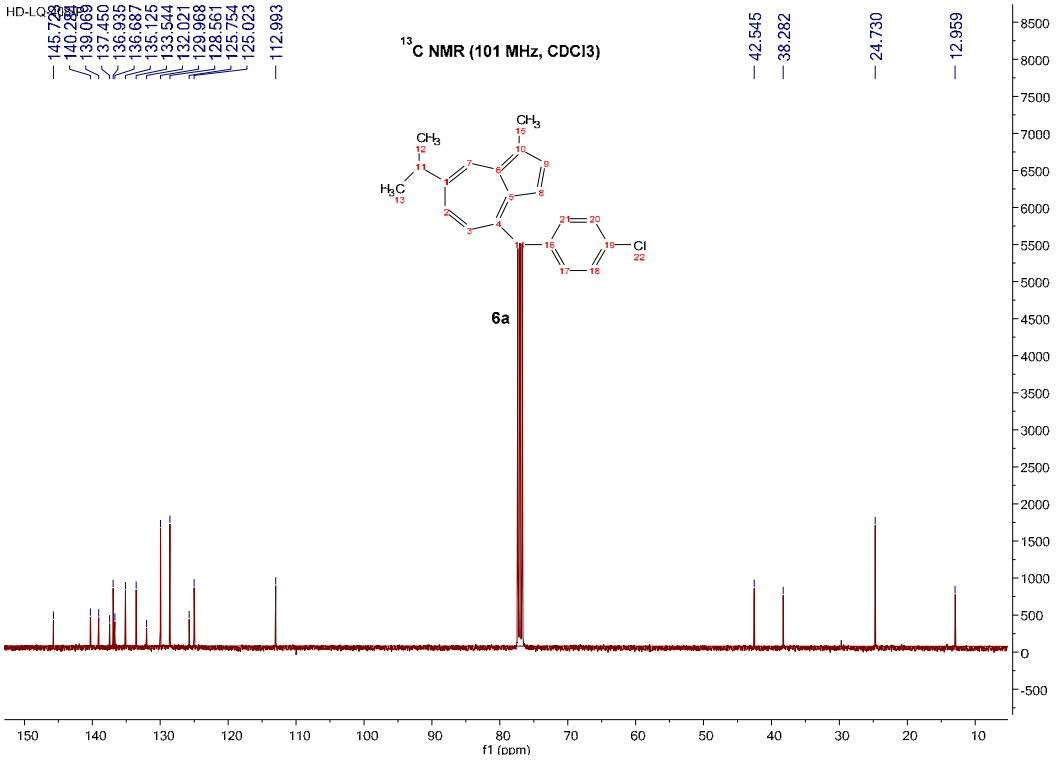
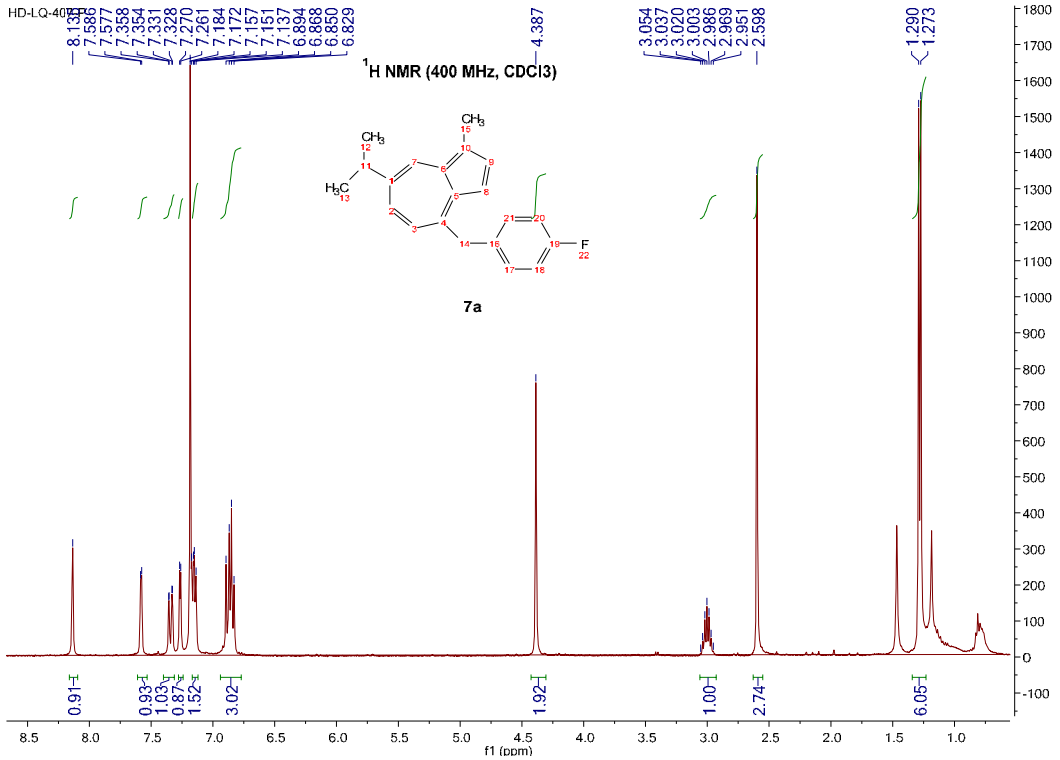
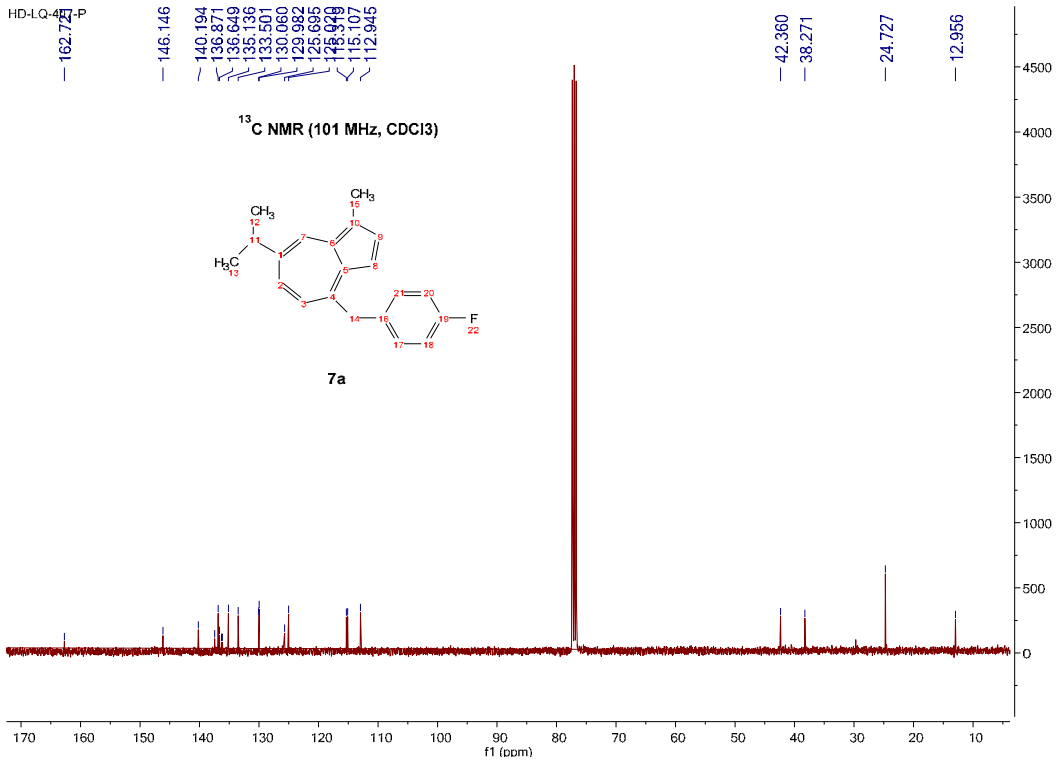
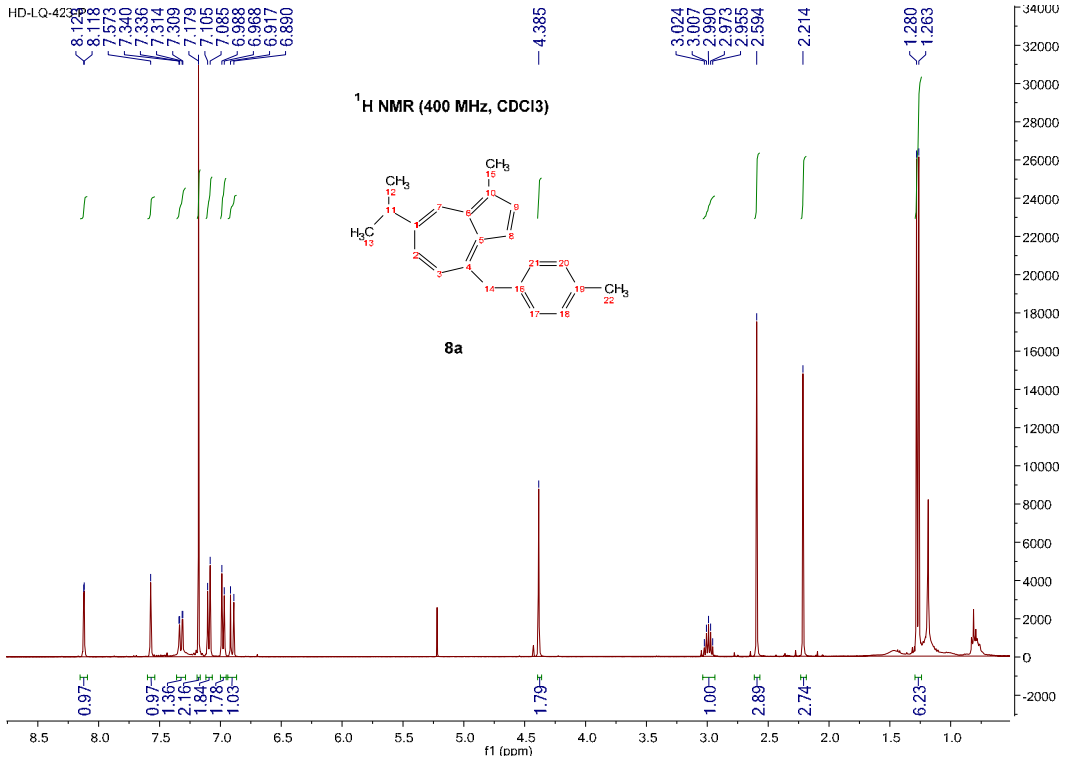
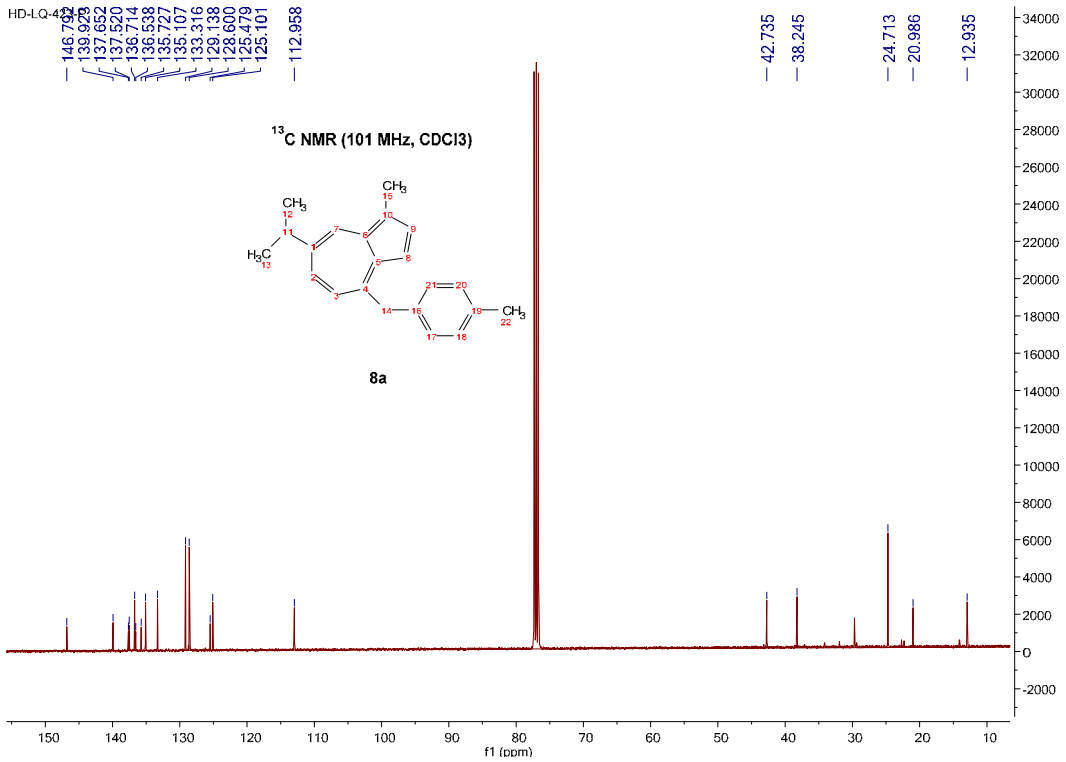
3.018
3.001
2.984
2.967
2.595
1.288
1.271
^{1}H NMR (400 MHz, CDCl3)
6a
0.98
0.95
1.07
1.05
3.30
1.13
1.95
1.00
2.94
6.31
f1 (ppm)

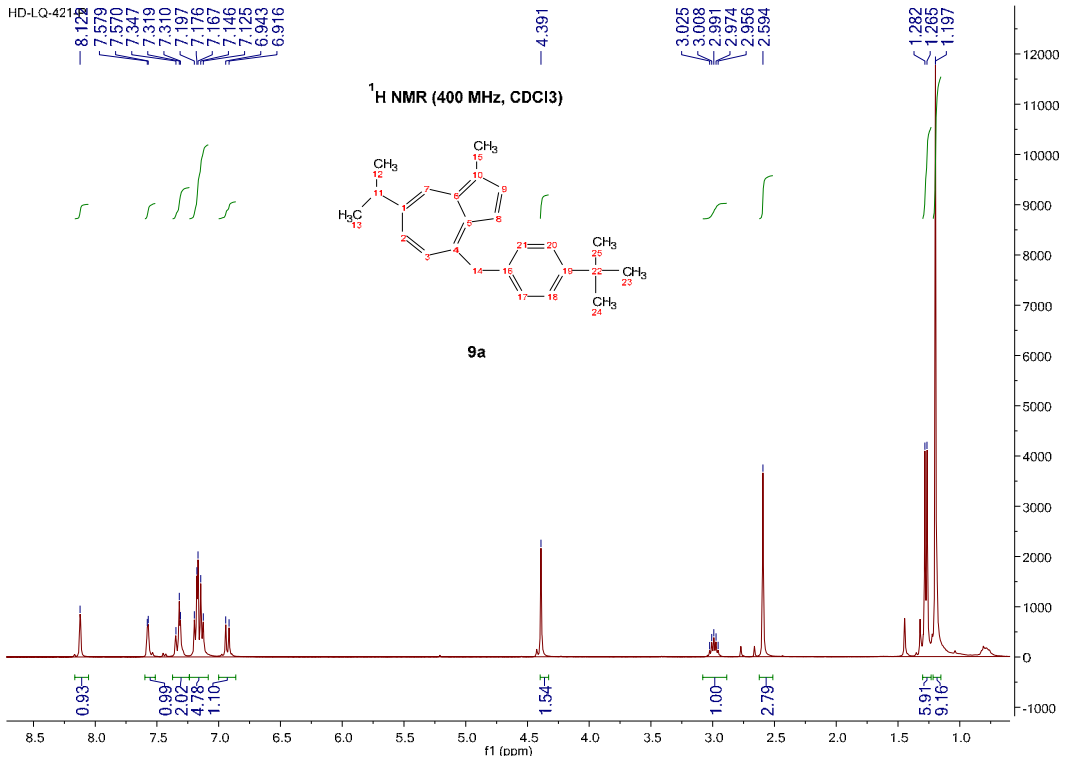

HD-LQ-101P
145.720
140.288
139.069
137.450
136.935
136.687
135.125
133.544
132.021
129.968
128.561
125.754
125.023
112.993
42.545
38.282
24.730
12.959
^{13}C NMR (101 MHz, CDCl3)
6a
f1 (ppm)

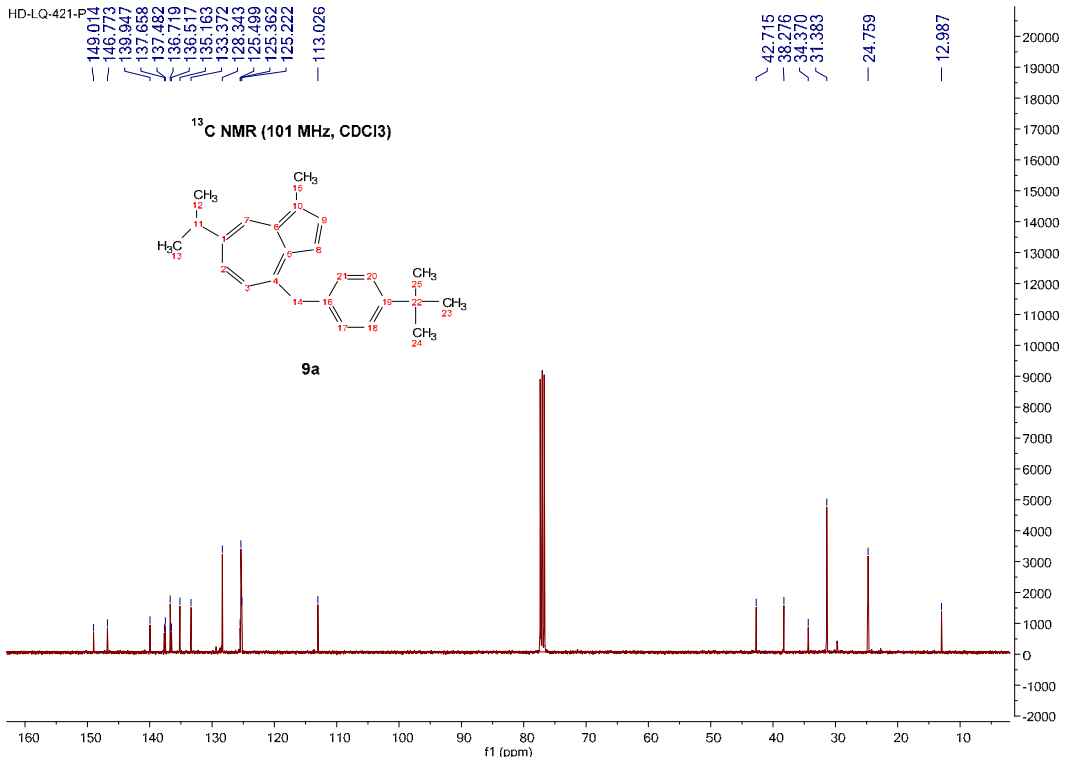

HD-LQ-405
8.134
7.586
7.577
7.358
7.354
7.331
7.328
7.270
7.261
7.184
7.172
7.157
7.151
7.137
6.894
6.868
6.850
6.829
4.387
3.054
3.037
3.020
3.003
2.986
2.969
2.951
2.598
1.290
1.273
^{1}H NMR (400 MHz, CDCl3)
7a
0.91
0.93
1.03
0.87
1.52
3.02
1.92
1.00
2.74
6.05
f1 (ppm)

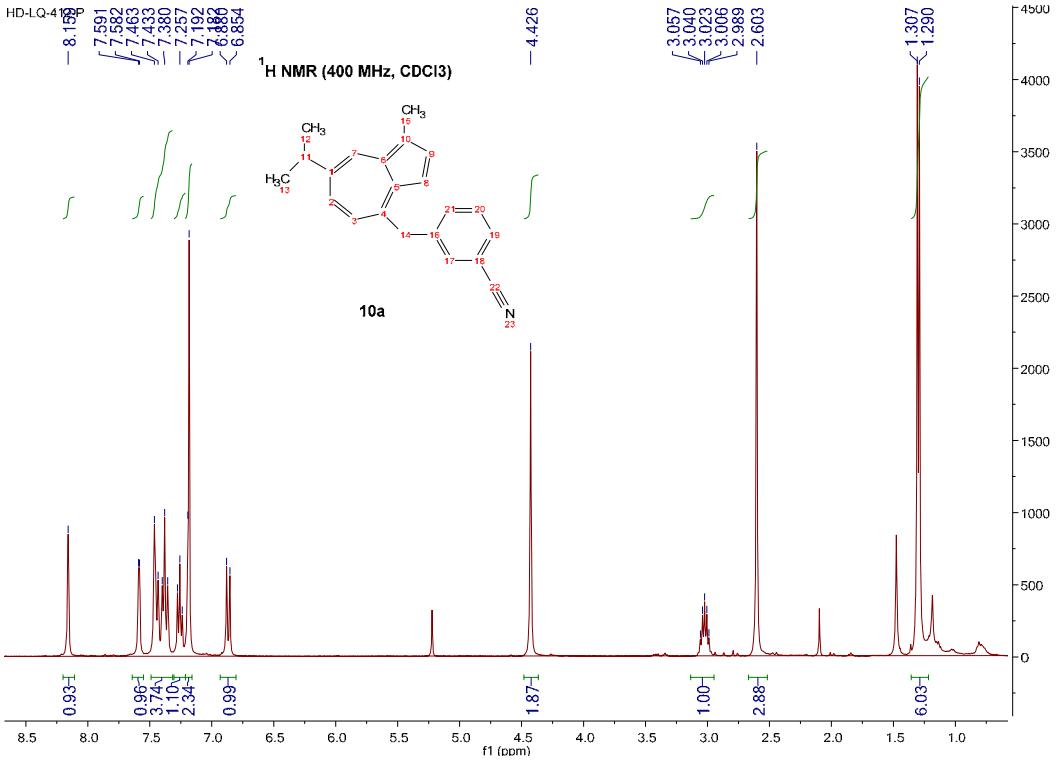

HD-LQ-417-P
162.727
146.146
140.194
136.871
136.649
135.136
133.501
130.060
129.982
125.695
125.620
115.319
115.107
112.945
42.360
38.271
24.727
12.956
^{13}C NMR (101 MHz, CDCl3)
7a
f1 (ppm)

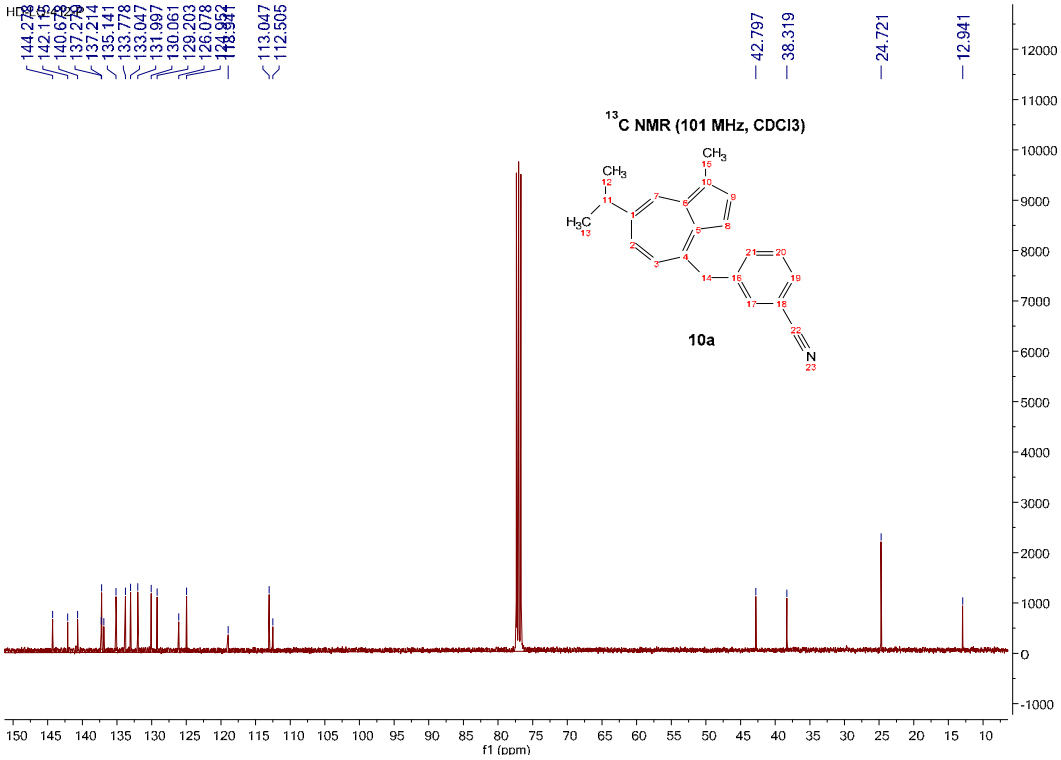

HD-LQ-423
8.128
8.118
7.573
7.340
7.336
7.314
7.309
7.179
7.105
7.085
6.988
6.968
6.917
6.890
4.385
3.024
3.007
2.990
2.973
2.955
2.594
2.214
1.280
1.263
^{1}H NMR (400 MHz, CDCl3)
8a
0.97
0.97
1.36
2.16
1.84
1.78
1.03
1.79
1.00
2.89
2.74
6.23
f1 (ppm)

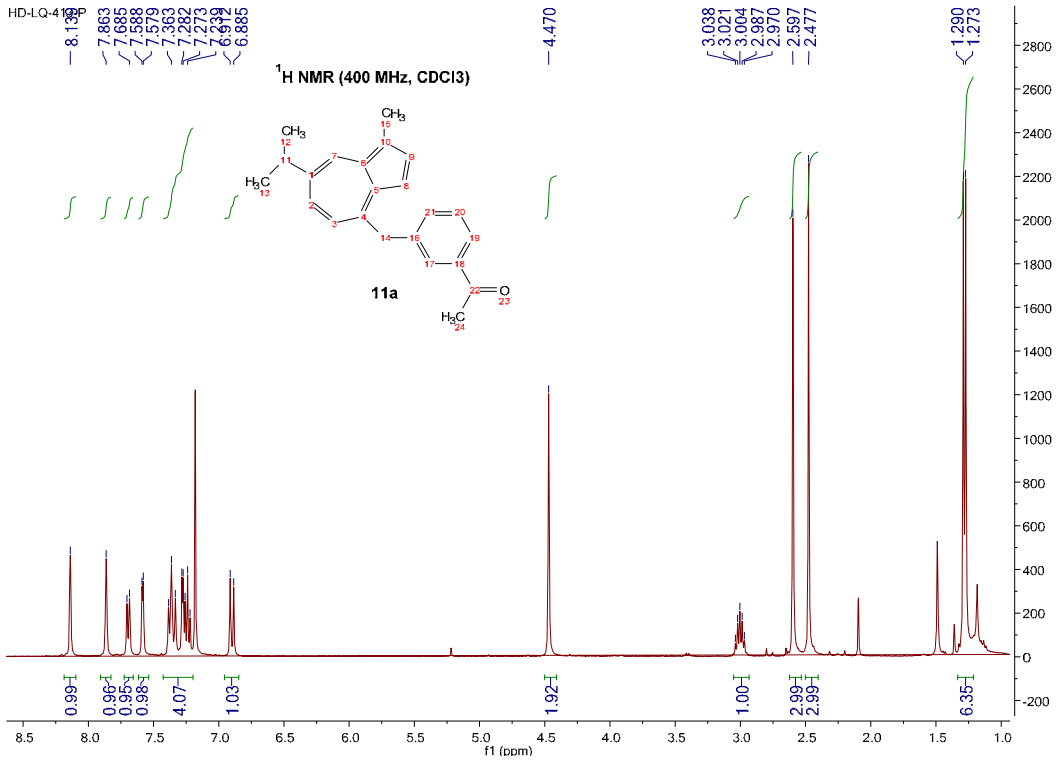

HD-LQ-423
146.792
139.929
137.652
137.520
136.714
136.538
135.727
135.107
133.316
129.138
128.600
125.479
125.101
112.958
42.735
38.245
24.713
20.986
12.935
^{13}C NMR (101 MHz, CDCl3)
8a
f1 (ppm)

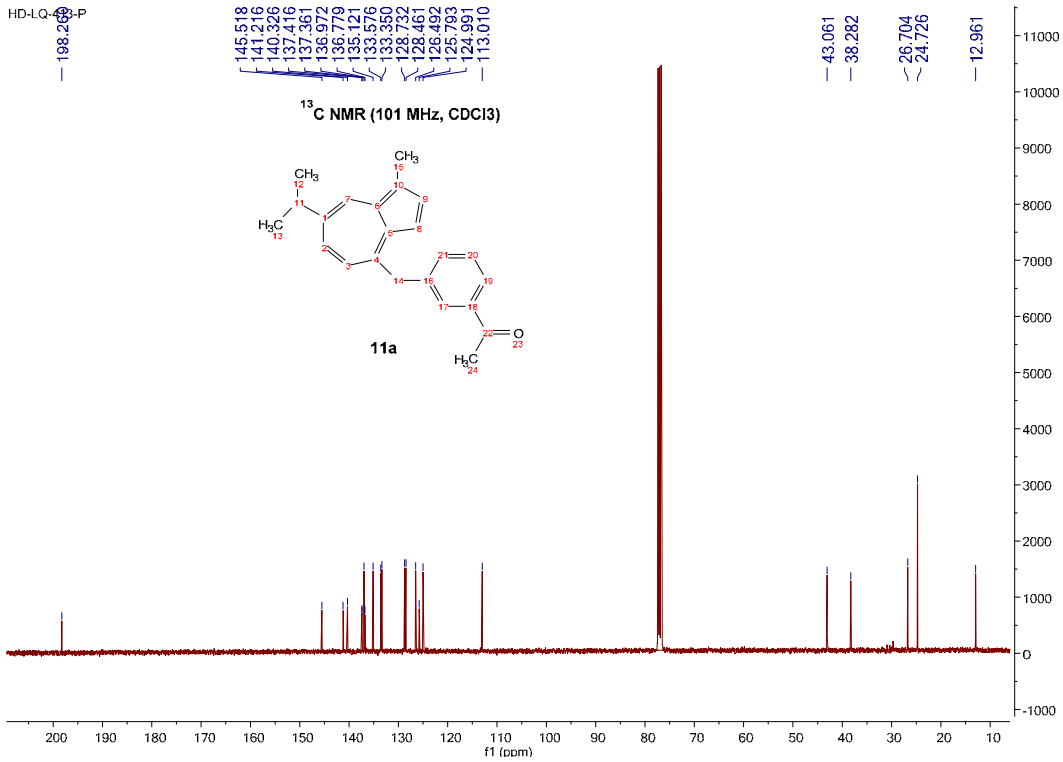

HD-LQ-421a
8.124
7.579
7.570
7.347
7.319
7.310
7.197
7.176
7.167
7.146
7.125
6.943
6.916
4.391
3.025
3.008
2.991
2.974
2.956
2.594
1.282
1.265
1.197
^{1}H NMR (400 MHz, CDCl3)
9a
0.93
0.99
2.02
4.78
1.10
1.54
1.00
2.79
5.91
9.16
f1 (ppm)

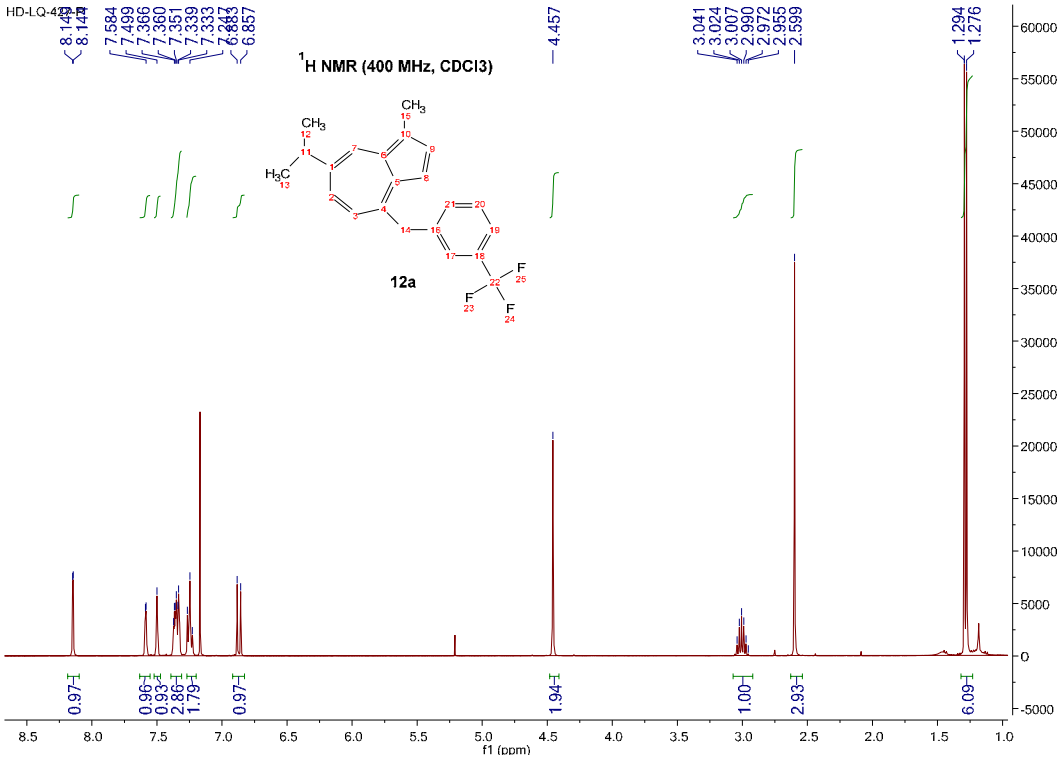

HD-LQ-421-P
149.014
146.773
139.947
137.658
137.482
136.719
136.517
135.163
133.372
128.343
125.499
125.362
125.222
113.026
42.715
38.276
34.370
31.383
24.759
12.987
^{13}C NMR (101 MHz, CDCl3)
9a
f1 (ppm)

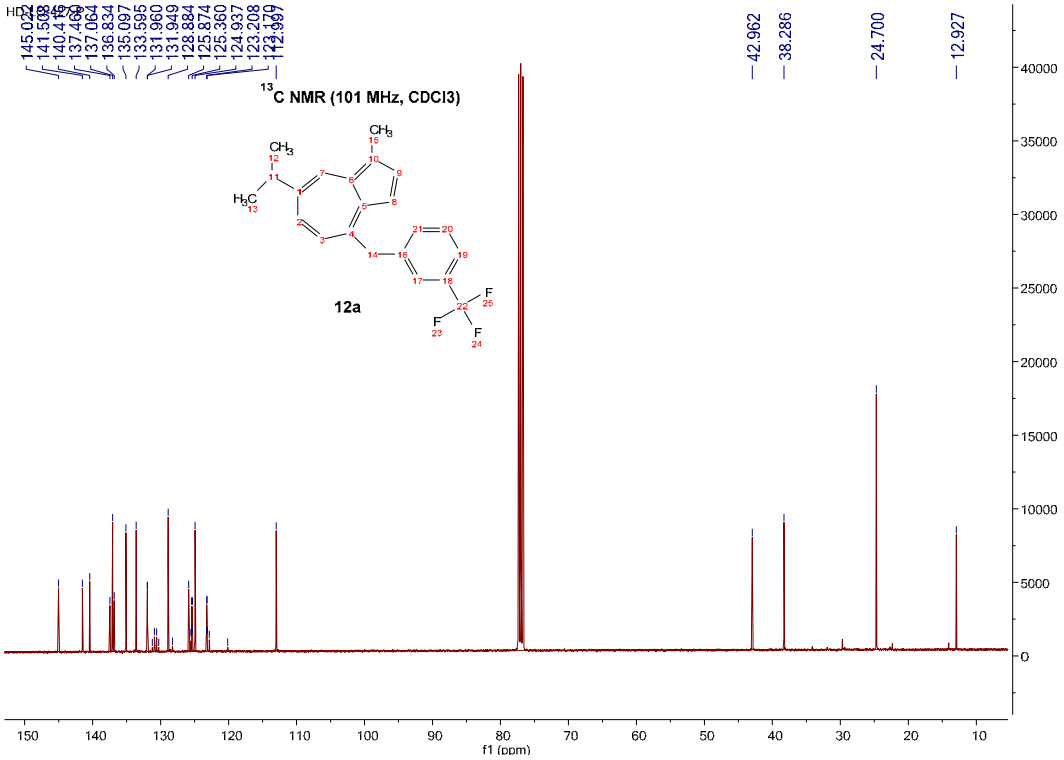

HD-LQ-4152P
8.159
7.591
7.582
7.463
7.433
7.380
7.257
7.192
7.182
6.880
6.854
4.426
3.057
3.040
3.023
3.006
2.989
2.603
1.307
1.290
^{1}H NMR (400 MHz, CDCl3)
10a
0.93
0.96
3.74
1.10
2.34
0.99
1.87
1.00
2.88
6.03
f1 (ppm)

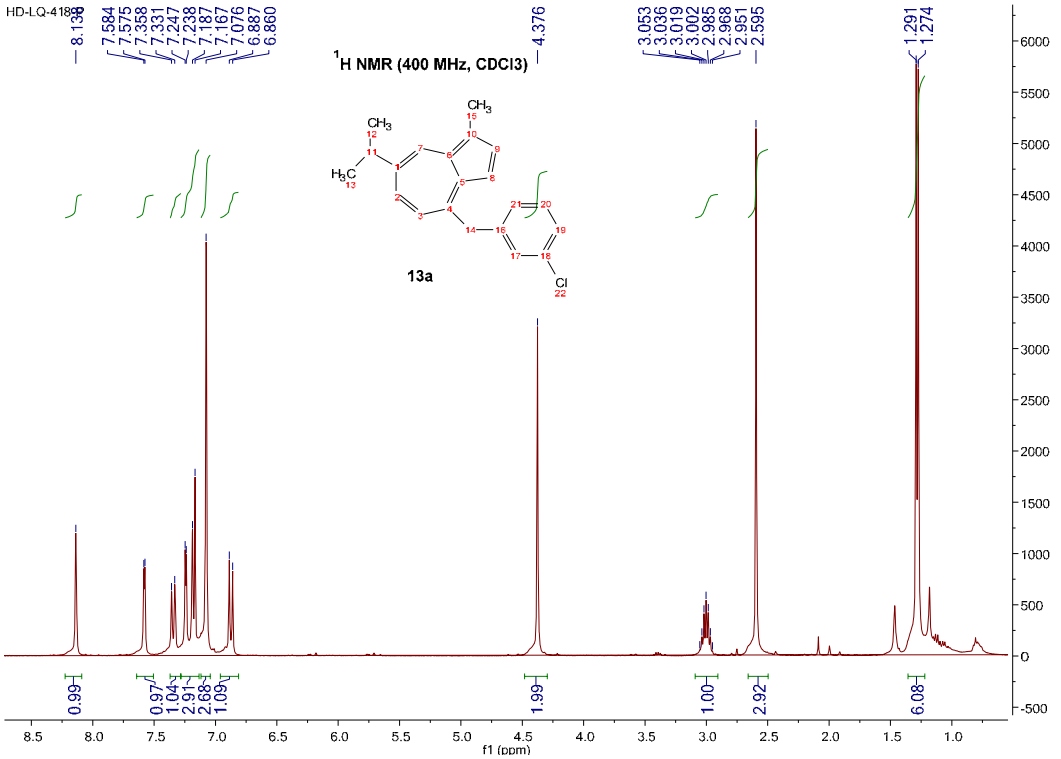

144.274
142.117
140.672
137.276
137.214
135.141
133.778
133.047
131.997
130.061
129.203
126.078
124.952
118.941
113.047
112.505
42.797
38.319
24.721
12.941
^{13}C NMR (101 MHz, CDCl3)
10a
f1 (ppm)

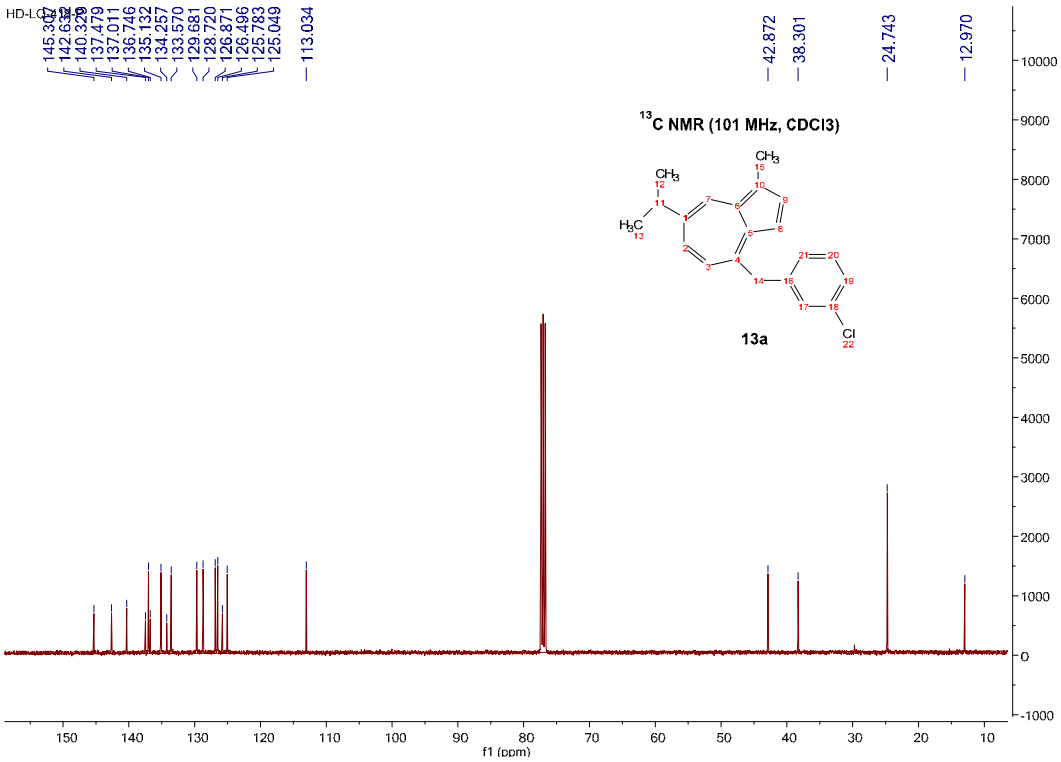

HD-LQ-419-P
8.139
7.863
7.685
7.588
7.579
7.363
7.282
7.273
7.239
6.912
6.885
4.470
3.038
3.021
3.004
2.987
2.970
2.597
2.477
1.290
1.273
^{1}H NMR (400 MHz, CDCl3)
11a
0.99
0.96
0.95
0.98
4.07
1.03
1.92
1.00
2.99
2.99
6.35
f1 (ppm)

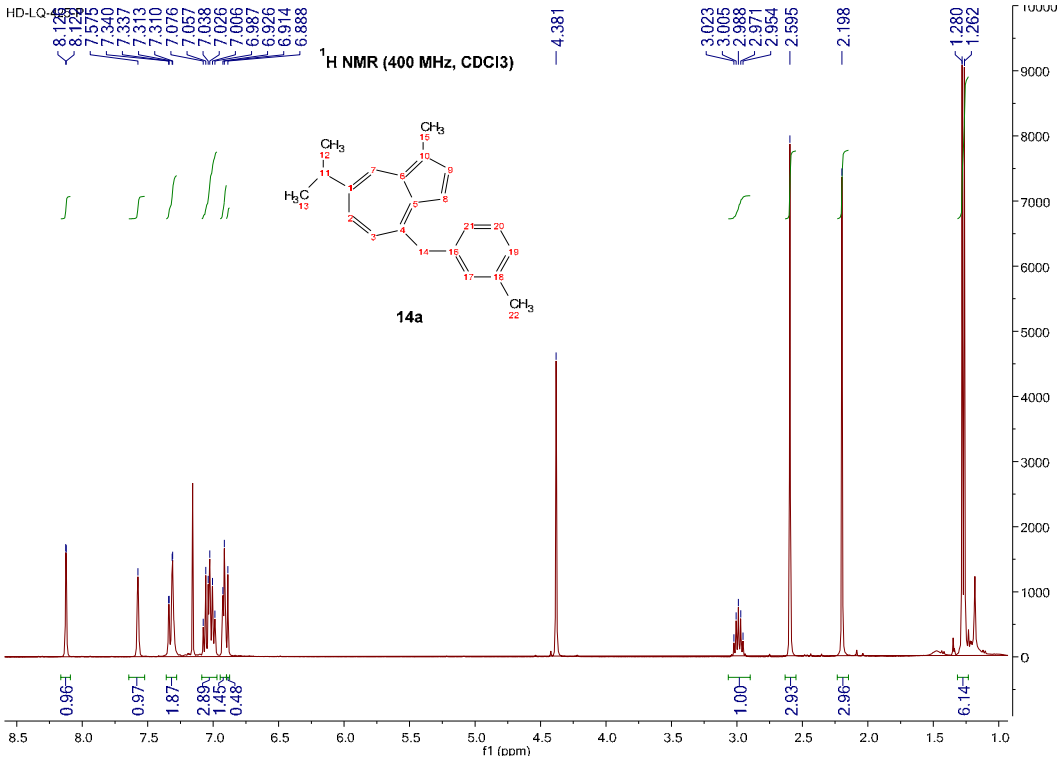

HD-LQ-419-P
198.263
145.518
141.216
140.326
137.416
137.361
136.972
136.779
135.121
133.576
133.350
128.732
128.461
126.492
125.793
124.991
113.010
43.061
38.282
26.704
24.726
12.961
^{13}C NMR (101 MHz, CDCl3)
11a
f1 (ppm)

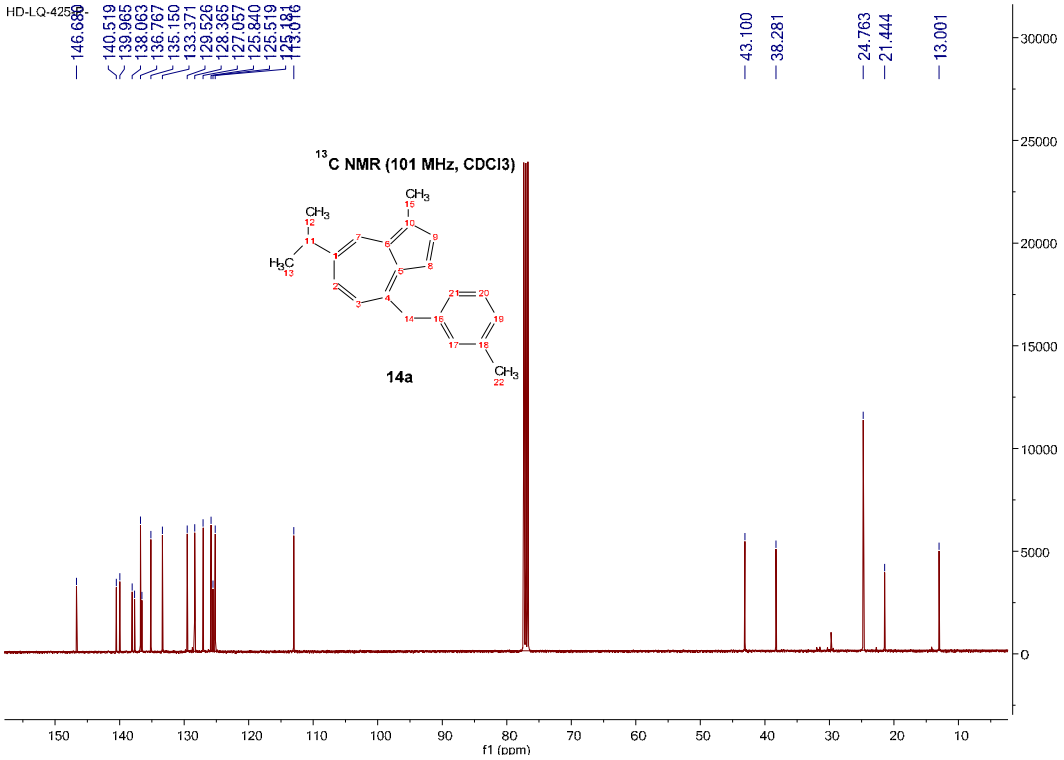

HD-LQ-422-1
^{1}H NMR (400 MHz, CDCl3)
12a
f1 (ppm)

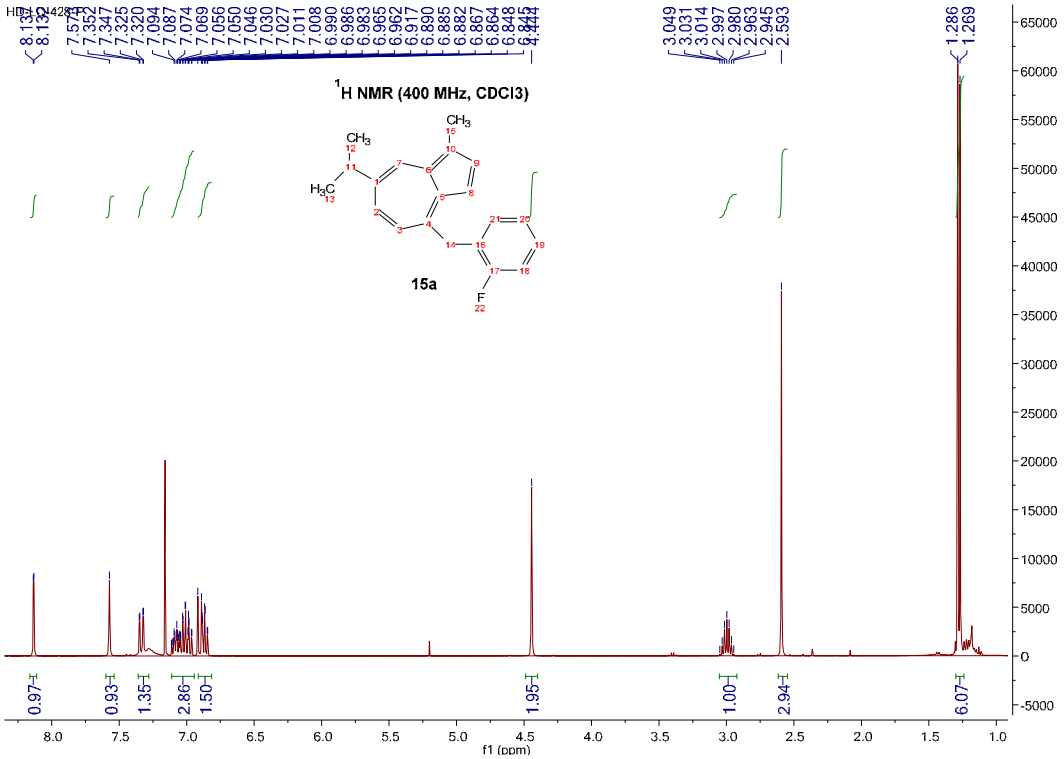

HD-LQ-422-2
^{13}C NMR (101 MHz, CDCl3)
12a
f1 (ppm)

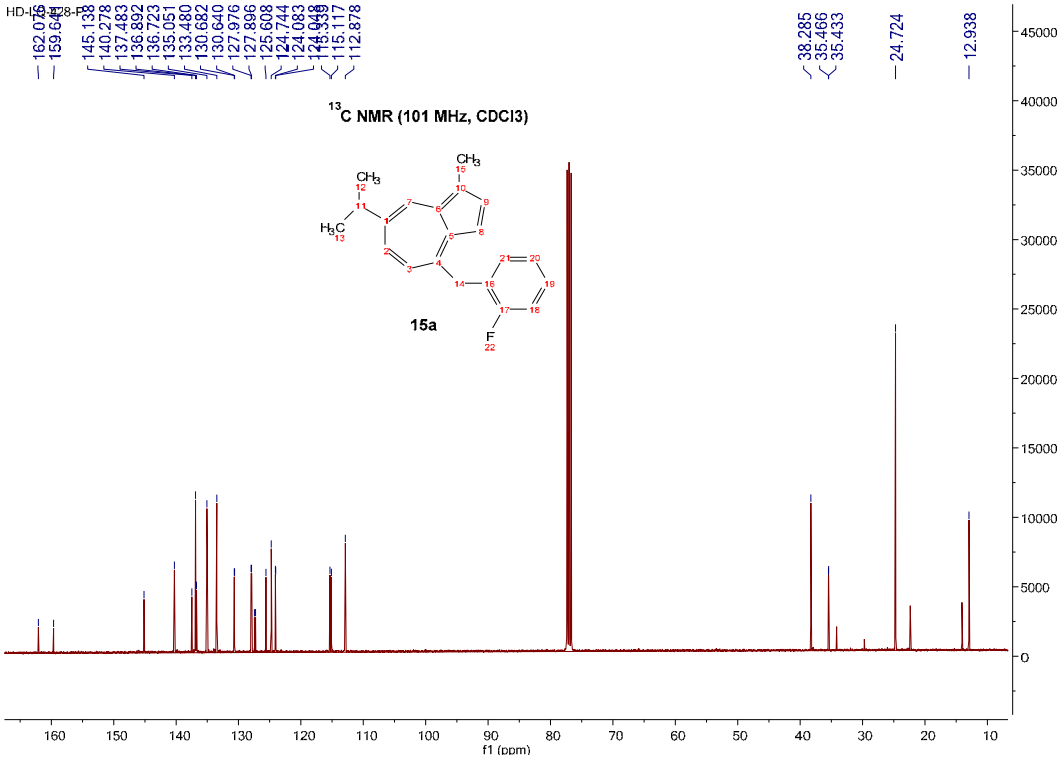

HD-LQ-4188
8.139
7.584
7.575
7.358
7.331
7.247
7.238
7.187
7.167
7.076
6.887
6.860
4.376
3.053
3.036
3.019
3.002
2.985
2.968
2.951
2.595
1.291
1.274
^{1}H NMR (400 MHz, CDCl3)
13a
0.99
0.97
1.04
2.91
2.68
1.09
1.99
1.00
2.92
6.08
f1 (ppm)

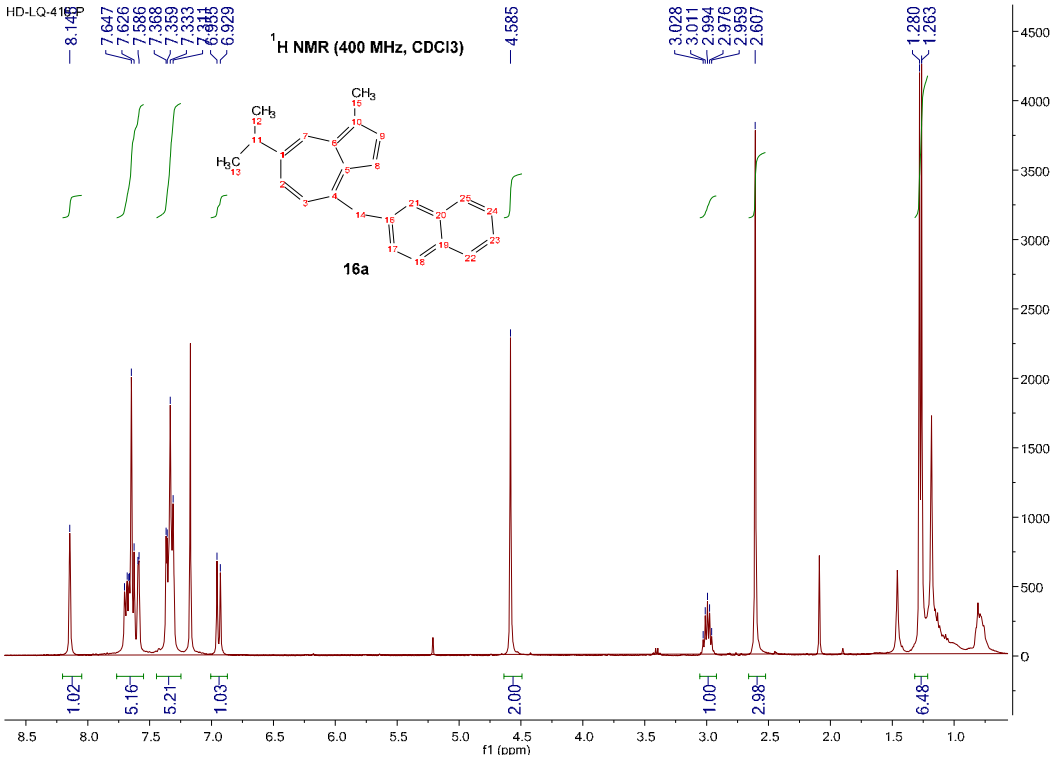

HD-LQ-4188-C
145.306
142.634
140.328
137.479
137.011
136.746
135.132
134.257
133.570
129.681
128.720
126.871
126.496
125.783
125.049
113.034
42.872
38.301
24.743
12.970
^{13}C NMR (101 MHz, CDCl3)
13a
f1 (ppm)

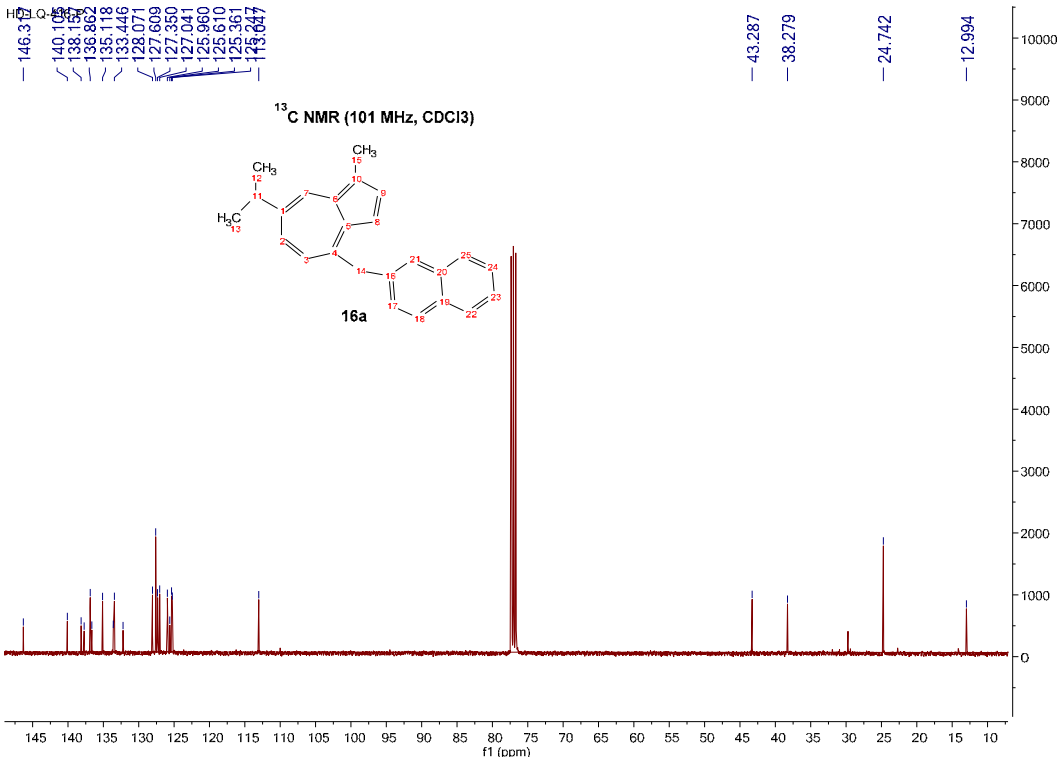

HD-LQ-425
8.128
8.125
7.575
7.340
7.337
7.313
7.310
7.076
7.057
7.038
7.026
7.006
6.987
6.926
6.914
6.888
4.381
3.023
3.005
2.988
2.971
2.954
2.595
2.198
1.280
1.262
^{1}H NMR (400 MHz, CDCl3)
14a
0.96
0.97
1.87
2.89
1.45
0.48
1.00
2.93
2.96
6.14
f1 (ppm)

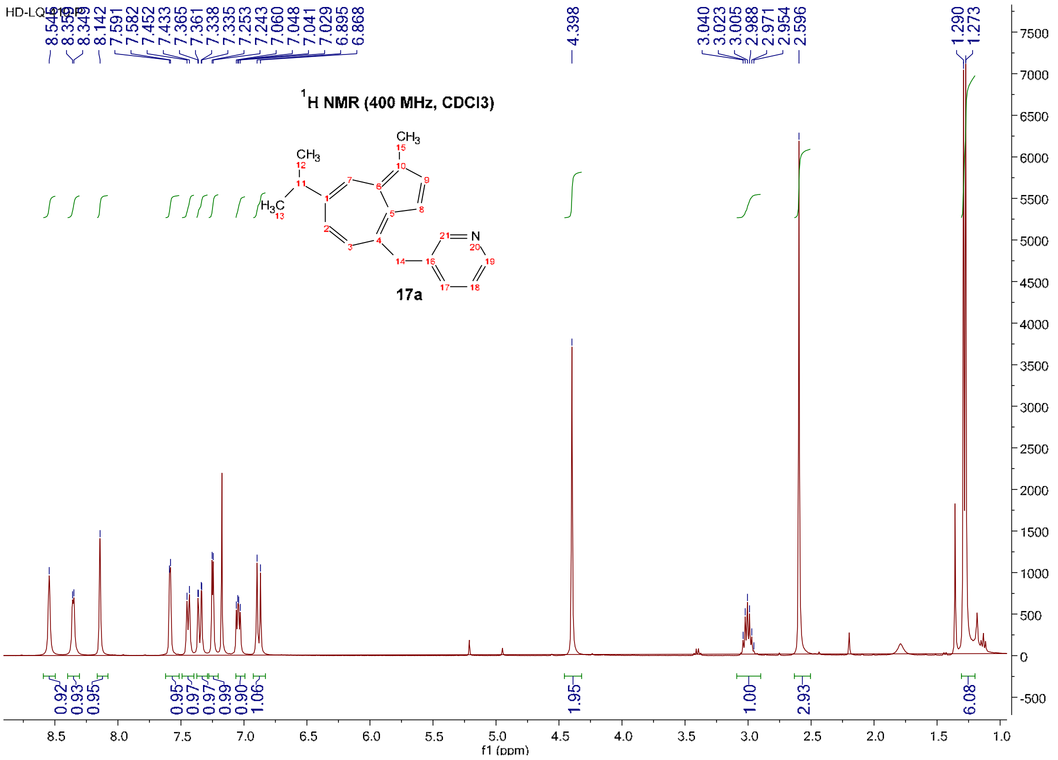

HD-LQ-425
146.680
140.519
139.965
138.063
136.767
135.150
133.371
129.526
128.365
127.057
125.840
125.519
43.100
38.281
24.763
21.444
13.001
^{13}C NMR (101 MHz, CDCl3)
14a
f1 (ppm)

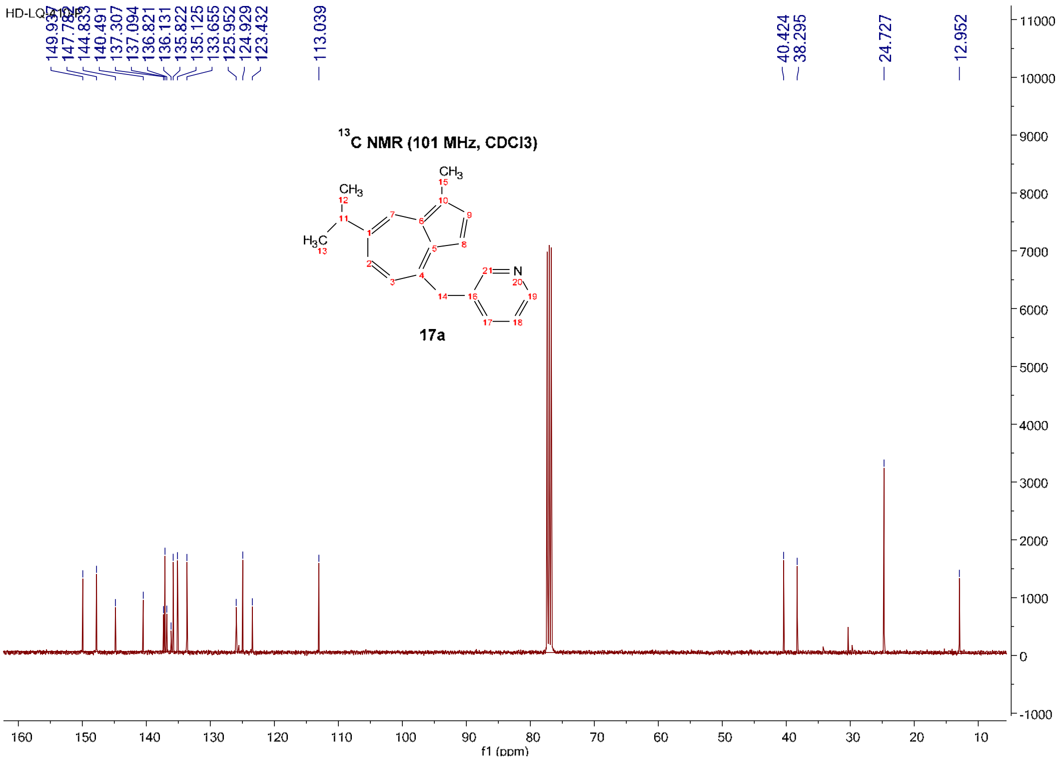

^{1}H NMR (400 MHz, CDCl3)
15a
f1 (ppm)

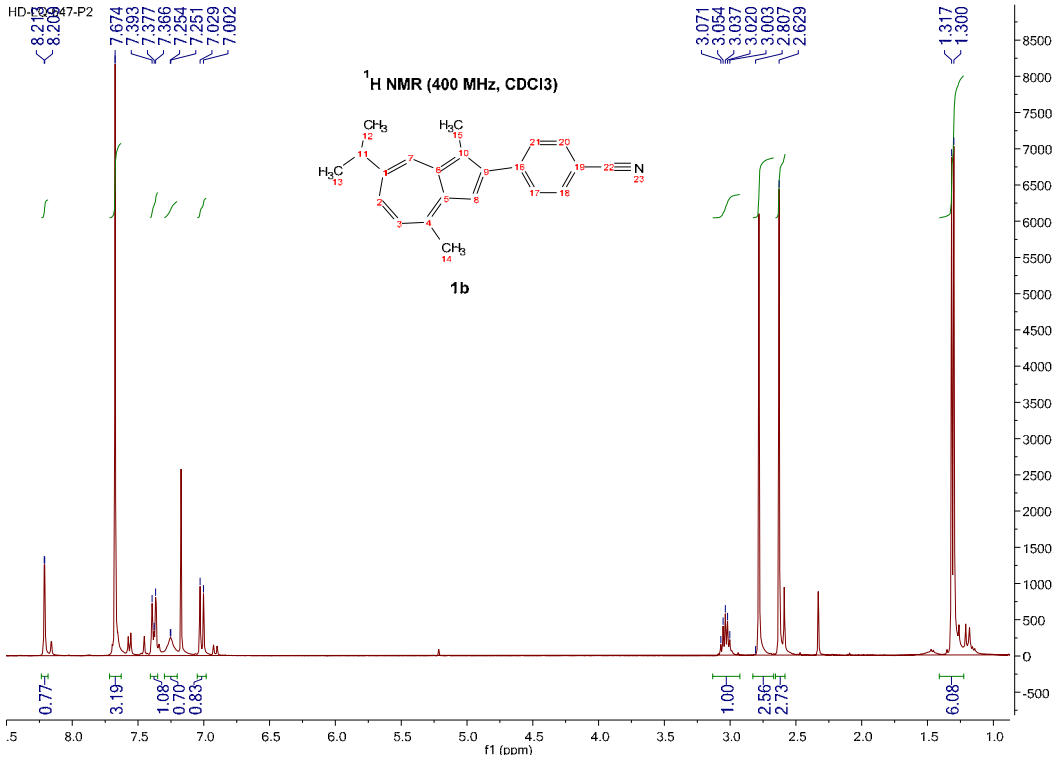

^{13}C NMR (101 MHz, CDCl3)
15a
f1 (ppm)

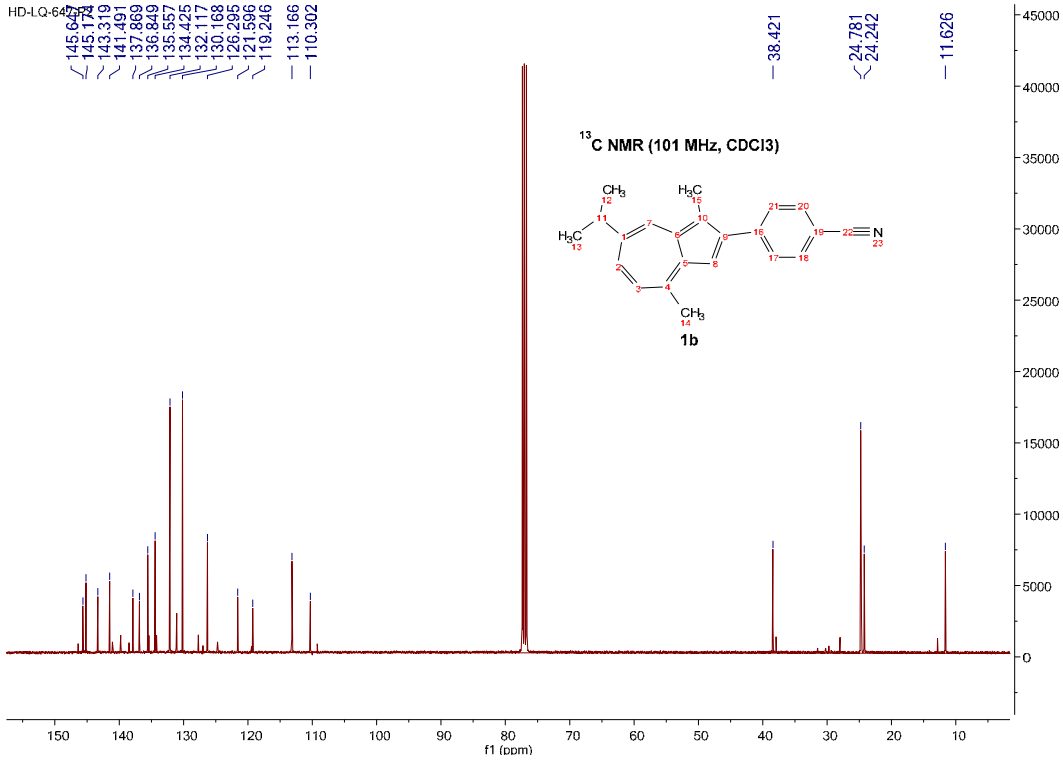

HD-LQ-416-P
8.148
7.647
7.626
7.586
7.368
7.359
7.333
7.311
6.935
6.929
4.585
3.028
3.011
2.994
2.976
2.959
2.607
1.280
1.263
^{1}H NMR (400 MHz, CDCl3)
16a
1.02
5.16
5.21
1.03
2.00
1.00
2.98
6.48
f1 (ppm)

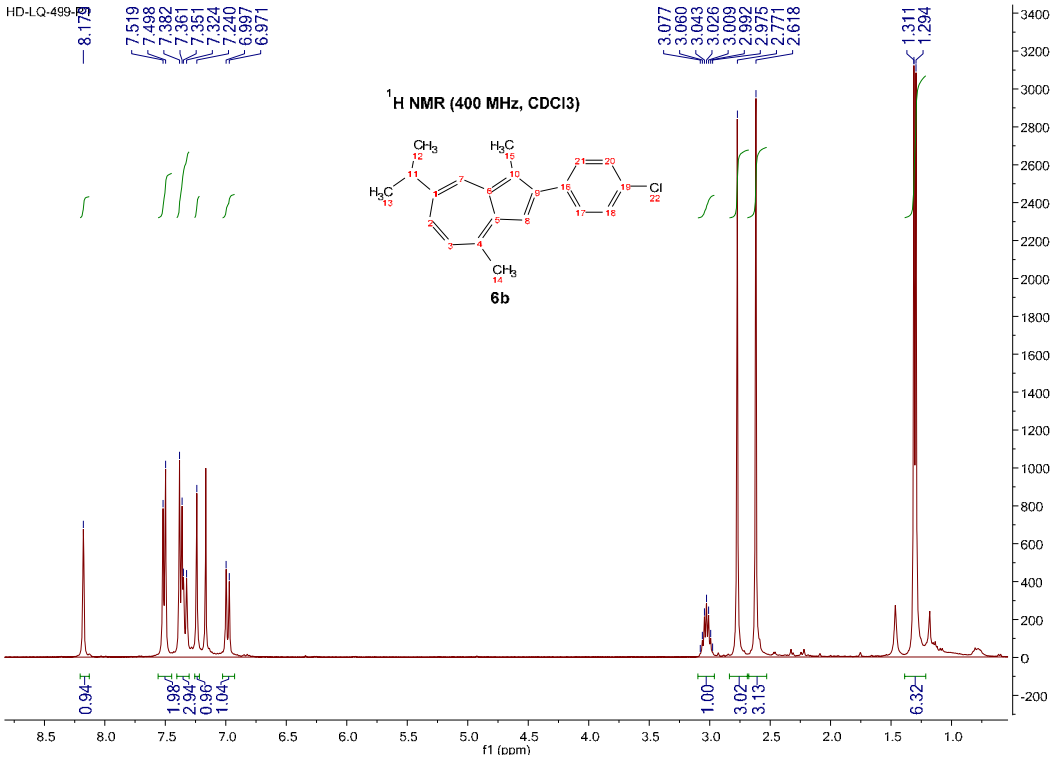

146.312
140.105
138.155
136.862
135.118
133.446
128.071
127.609
127.350
127.041
125.960
125.610
125.361
43.287
38.279
24.742
12.994
^{13}C NMR (101 MHz, CDCl3)
16a
f1 (ppm)

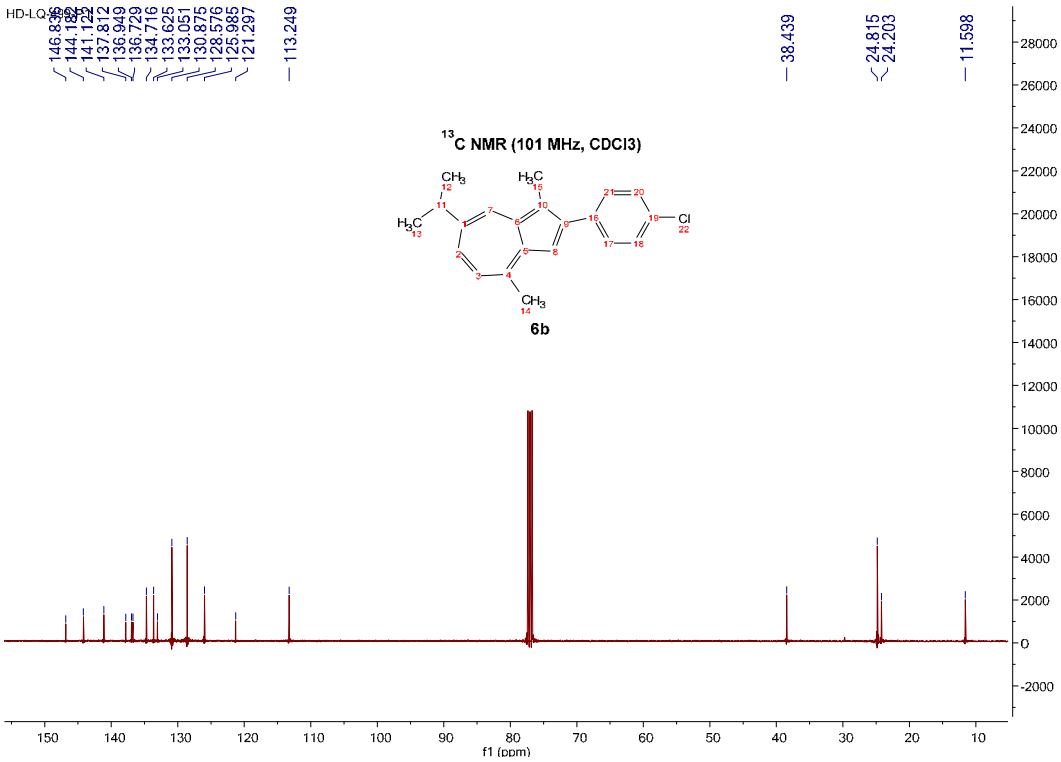

HD-LQ
1H NMR (400 MHz, CDCl3)
17a
f1 (ppm)

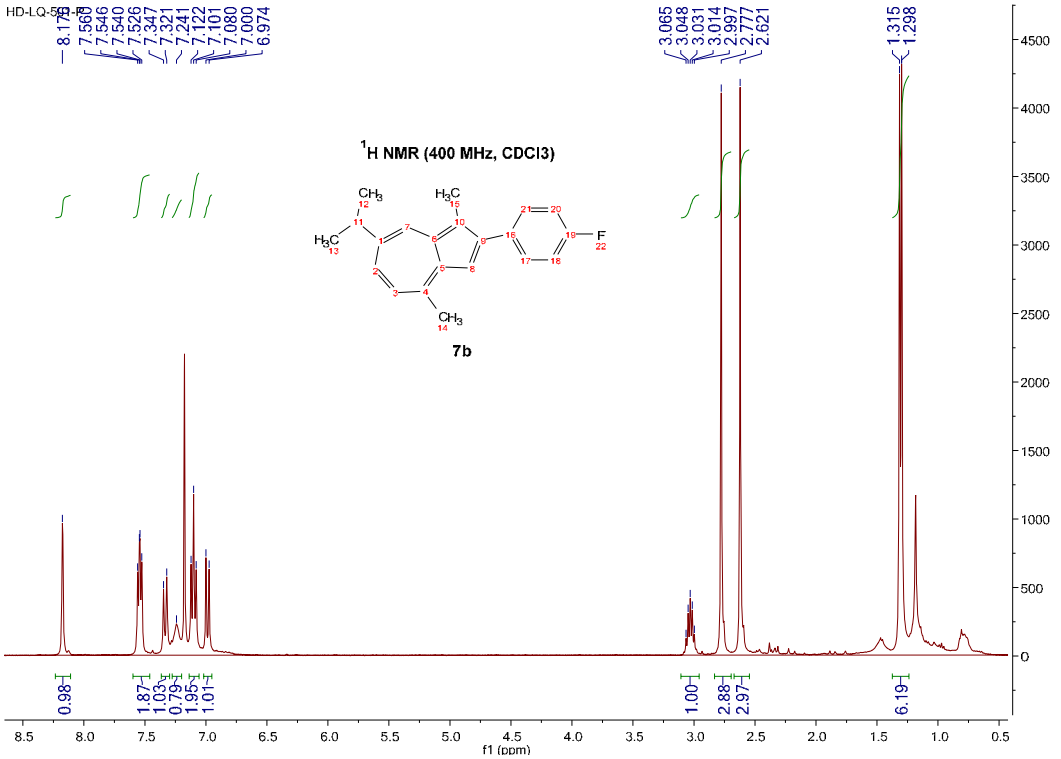

HD-LQ
13C NMR (101 MHz, CDCl3)
17a
f1 (ppm)

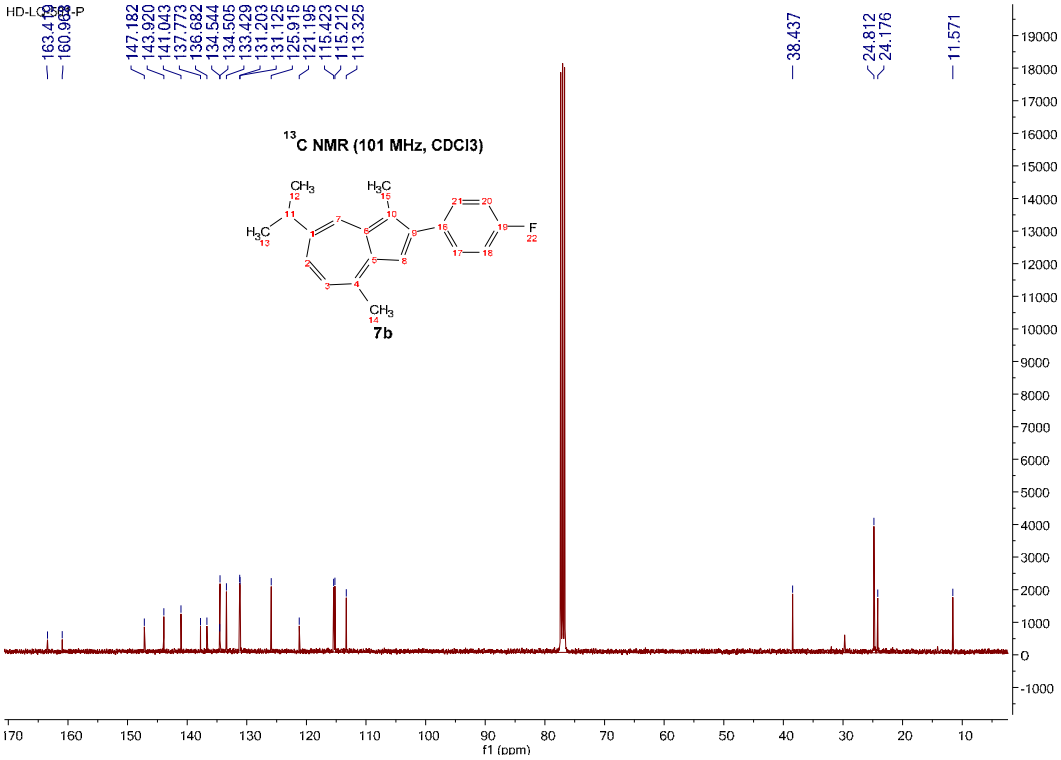

HD-LQ-647-P2
8.218
8.208
7.674
7.393
7.377
7.366
7.254
7.251
7.029
7.002
3.071
3.054
3.037
3.020
3.003
2.807
2.629
1.317
1.300
^{1}H NMR (400 MHz, CDCl3)
1b
0.77
3.19
1.08
0.70
0.83
1.00
2.56
2.73
6.08
f1 (ppm)

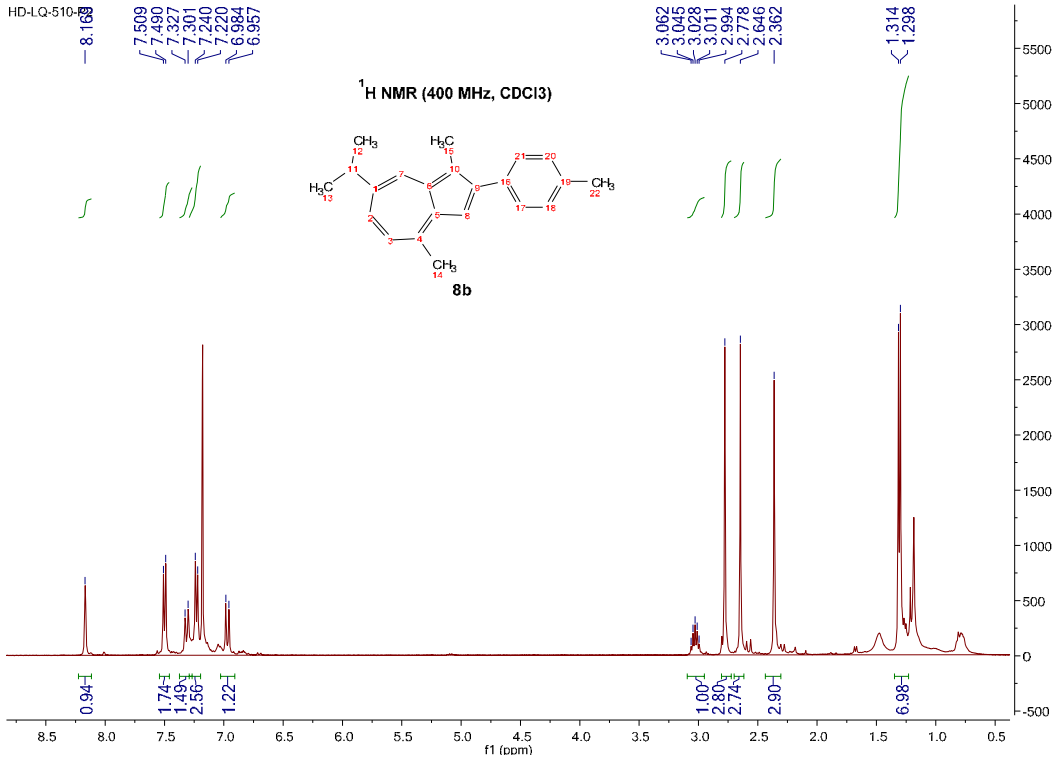

HD-LQ-647-P4
145.647
145.174
143.319
141.491
137.869
136.849
135.557
134.425
132.117
130.168
126.295
121.596
119.246
113.166
110.302
38.421
24.781
24.242
11.626
^{13}C NMR (101 MHz, CDCl3)
1b
f1 (ppm)

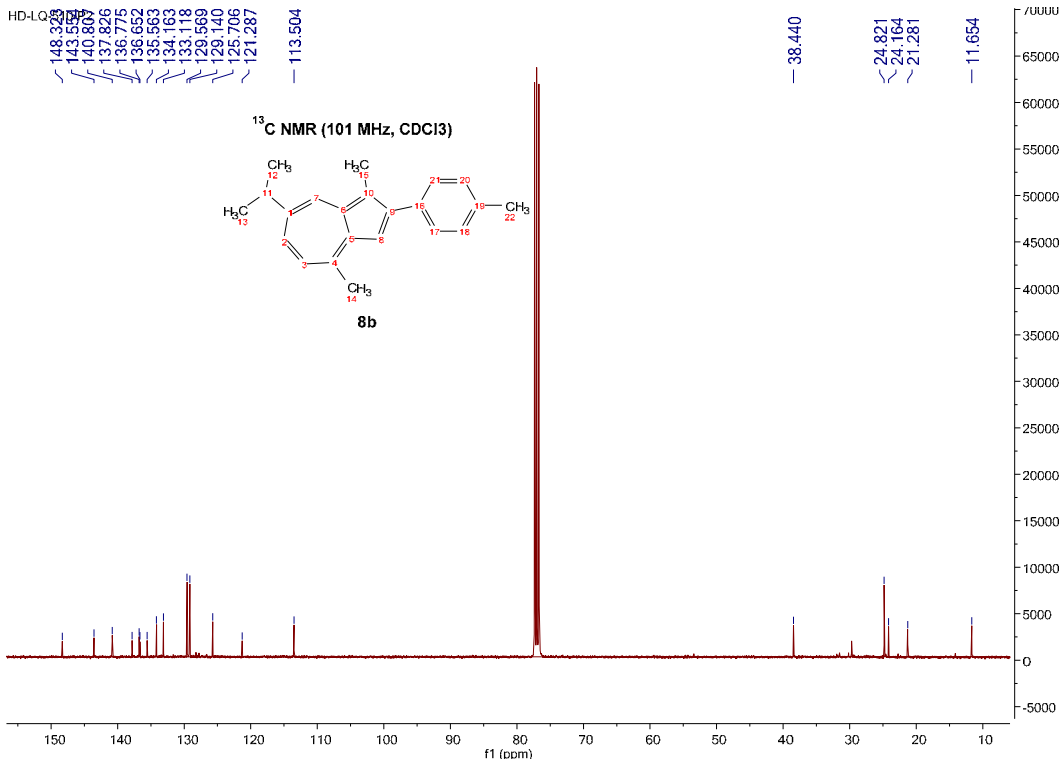

HD-LQ-499-H
8.179
7.519
7.498
7.382
7.361
7.351
7.324
7.240
6.997
6.971
3.077
3.060
3.043
3.026
3.009
2.992
2.975
2.771
2.618
1.311
1.294
^{1}H NMR (400 MHz, CDCl3)
6b
0.94
1.98
2.94
0.96
1.04
1.00
3.02
3.13
6.32
f1 (ppm)

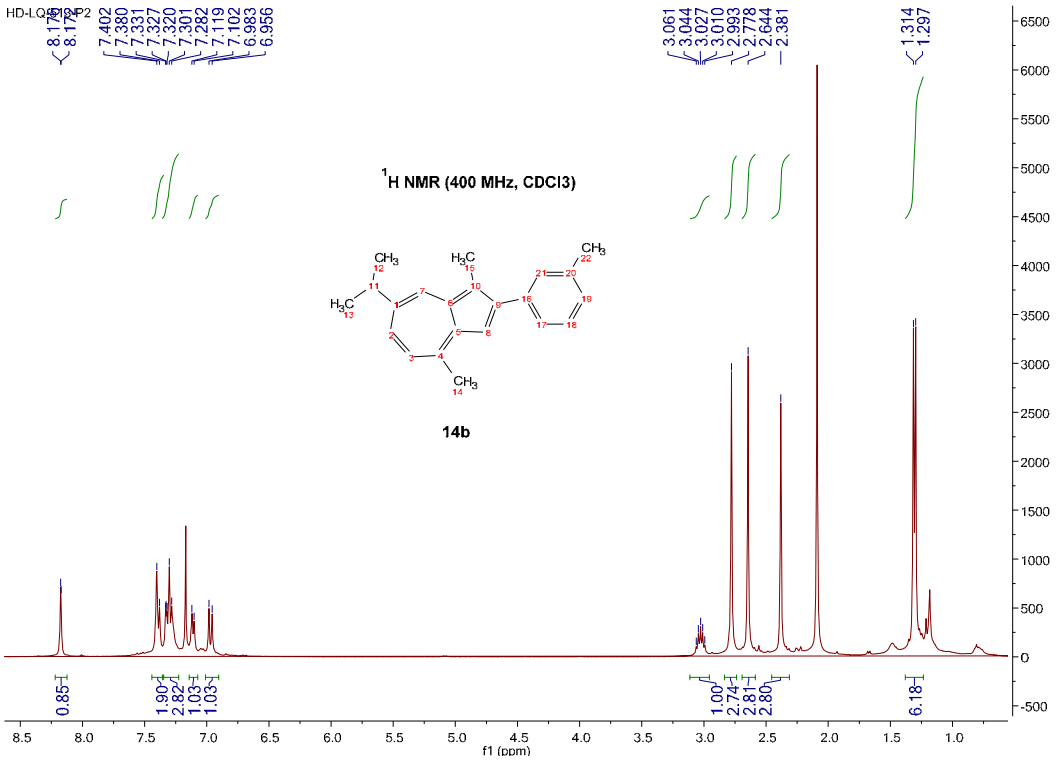

146.837
144.182
141.122
137.812
136.949
136.729
134.716
133.625
133.051
130.875
128.576
125.985
121.297
113.249
38.439
24.815
24.203
11.598
^{13}C NMR (101 MHz, CDCl3)
6b
f1 (ppm)

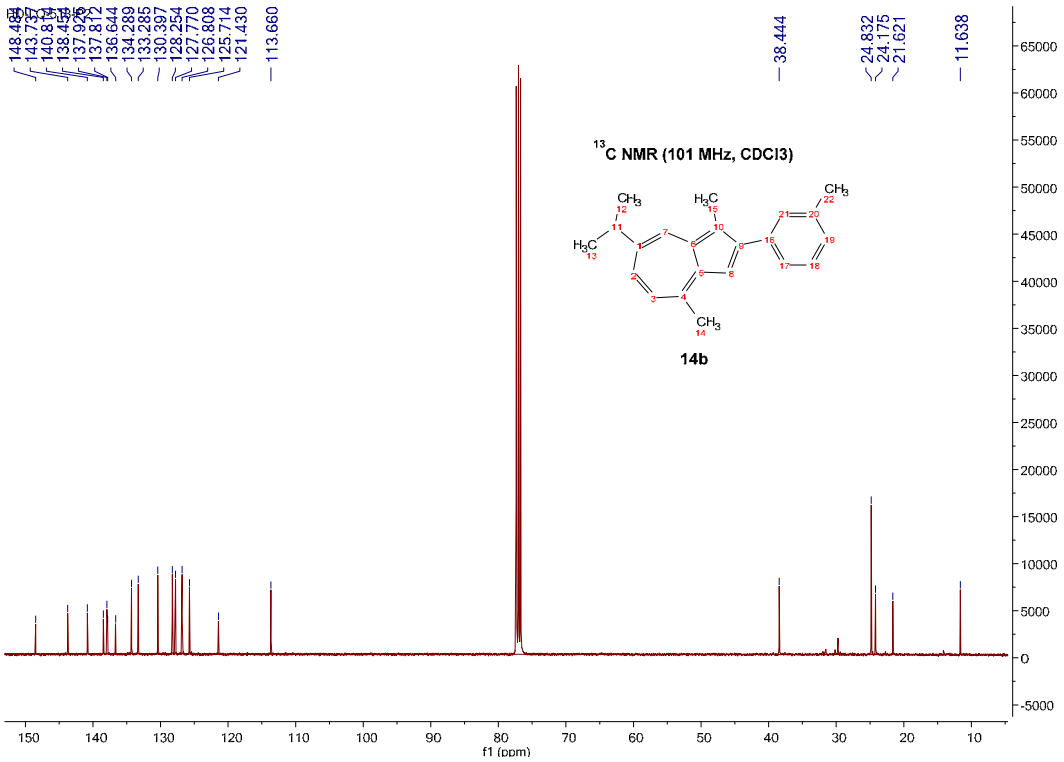

HD-LQ-5
8.179
7.560
7.546
7.540
7.526
7.347
7.321
7.241
7.122
7.101
7.080
7.000
6.974
3.065
3.048
3.031
3.014
2.997
2.777
2.621
1.315
1.298
^{1}H NMR (400 MHz, CDCl3)
7b
0.98
1.87
1.03
0.79
1.95
1.01
1.00
2.88
2.97
6.19
f1 (ppm)

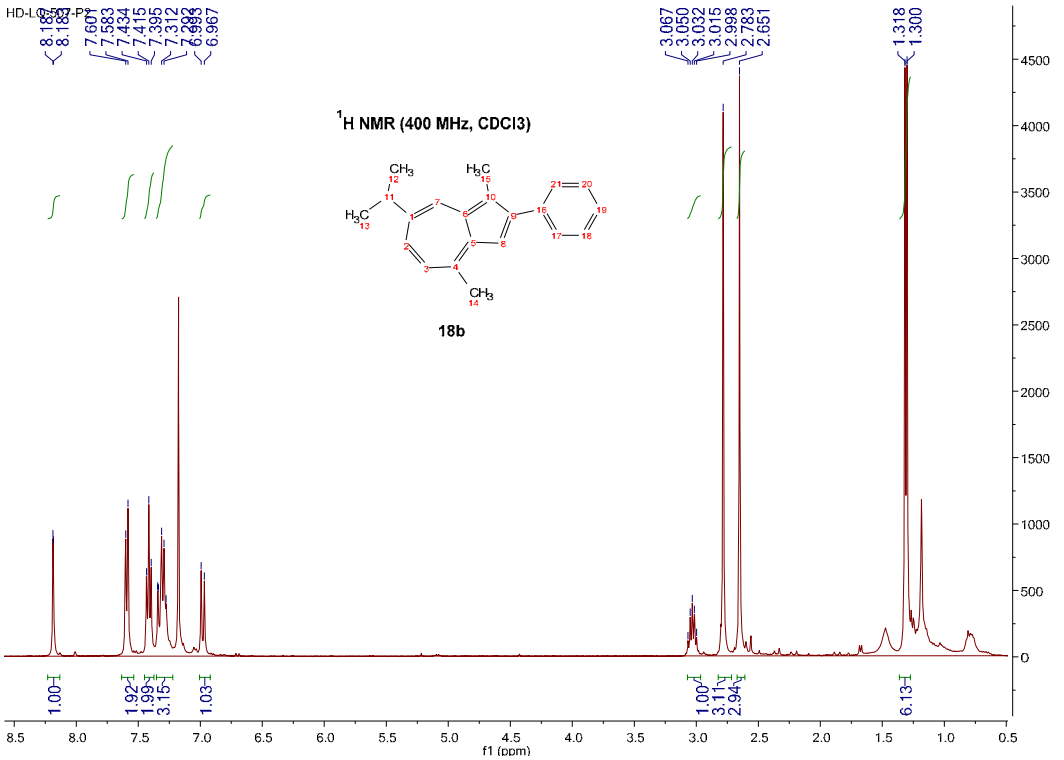

HD-LQ-5
163.418
160.983
147.182
143.920
141.043
137.773
136.682
134.544
134.505
133.429
131.203
131.125
125.915
121.195
115.423
115.212
113.325
38.437
24.812
24.176
11.571
^{13}C NMR (101 MHz, CDCl3)
7b
f1 (ppm)

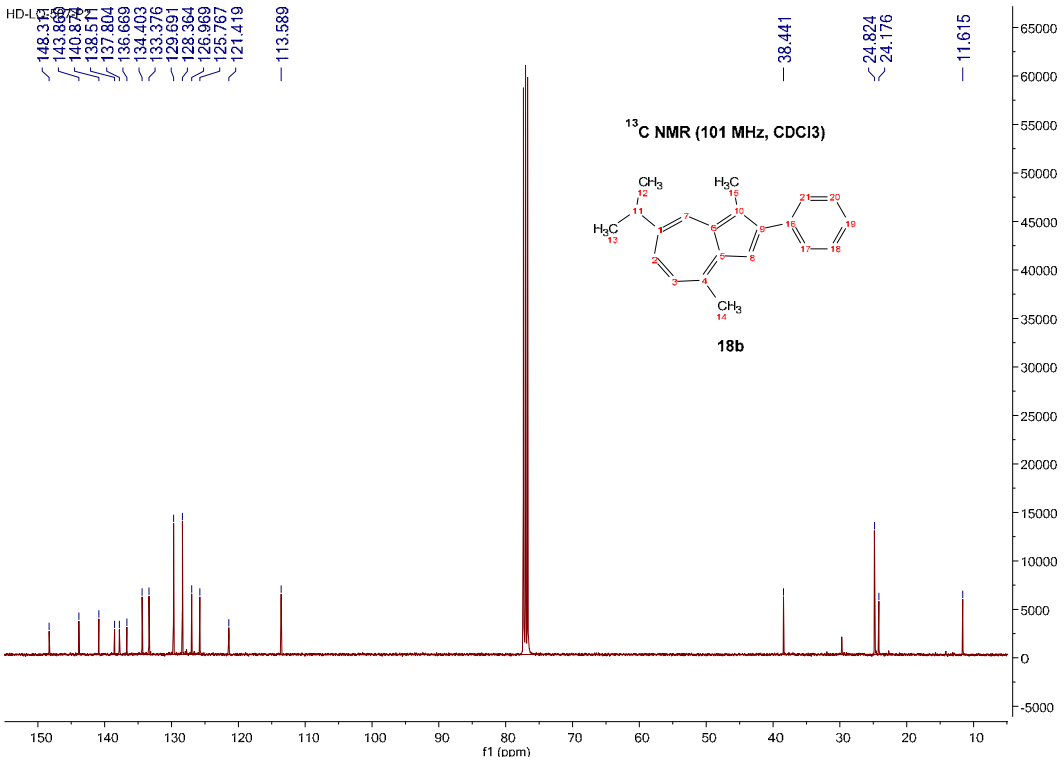

HD-LQ-510-13
8.169
7.509
7.490
7.327
7.301
7.240
7.220
6.984
6.957
3.062
3.045
3.028
3.011
2.994
2.778
2.646
2.362
1.314
1.298
^{1}H NMR (400 MHz, CDCl3)
8b
0.94
1.74
1.49
2.56
1.22
1.00
2.80
2.74
2.90
6.98
f1 (ppm)

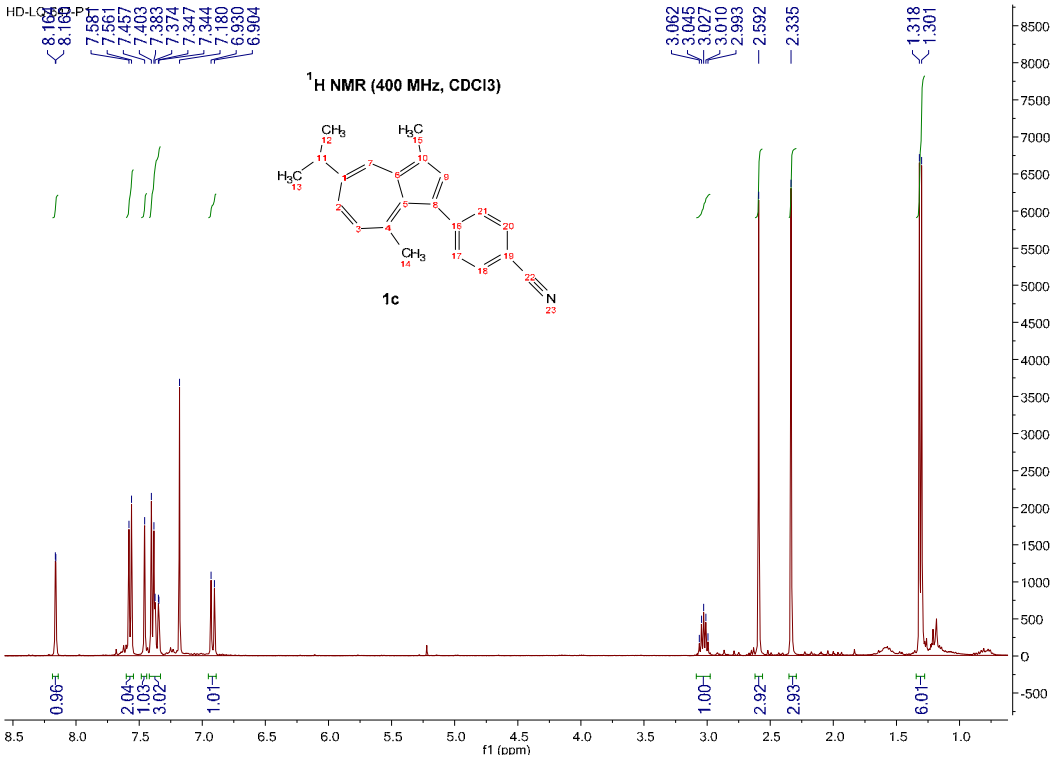

148.323
143.557
140.807
137.826
136.775
136.652
135.563
134.163
133.118
129.569
129.140
125.706
121.287
113.504
38.440
24.821
24.164
21.281
11.654
^{13}C NMR (101 MHz, CDCl3)
8b
f1 (ppm)

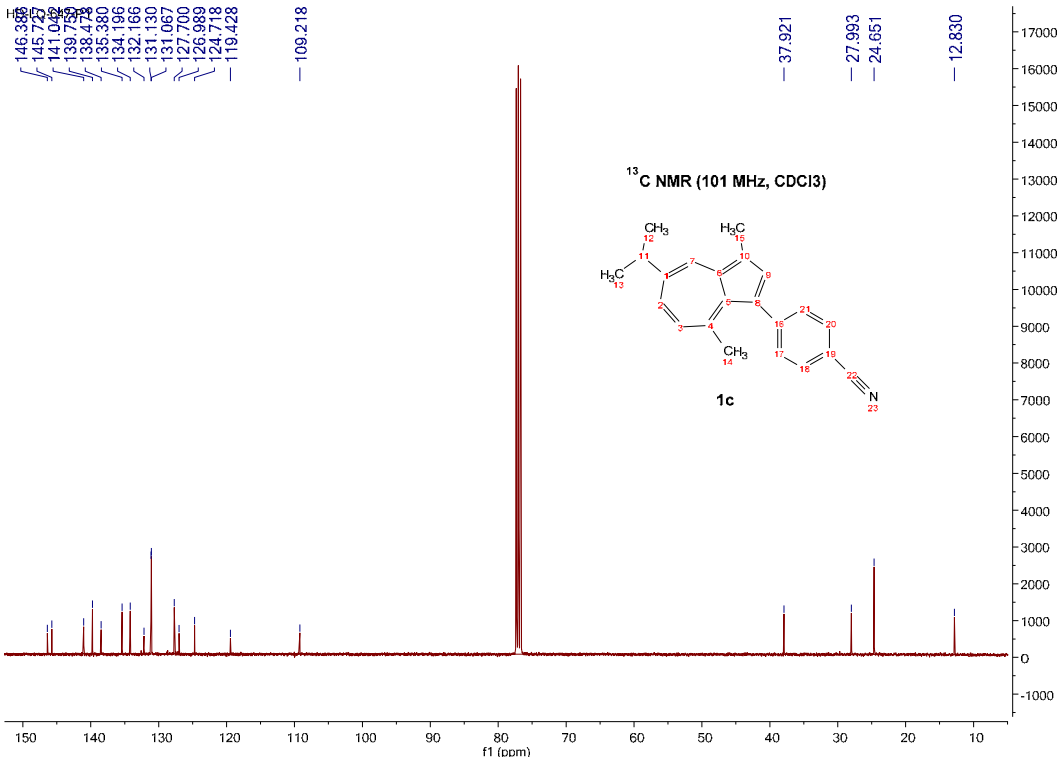

HD-LQ-512-P2
8.172
8.172
7.402
7.380
7.331
7.327
7.320
7.301
7.282
7.119
7.102
6.983
6.956
3.061
3.044
3.027
3.010
2.993
2.778
2.644
2.381
1.314
1.297
^{1}H NMR (400 MHz, CDCl3)
14b
0.85
1.90
2.82
1.03
1.03
1.00
2.74
2.81
2.80
6.18
f1 (ppm)

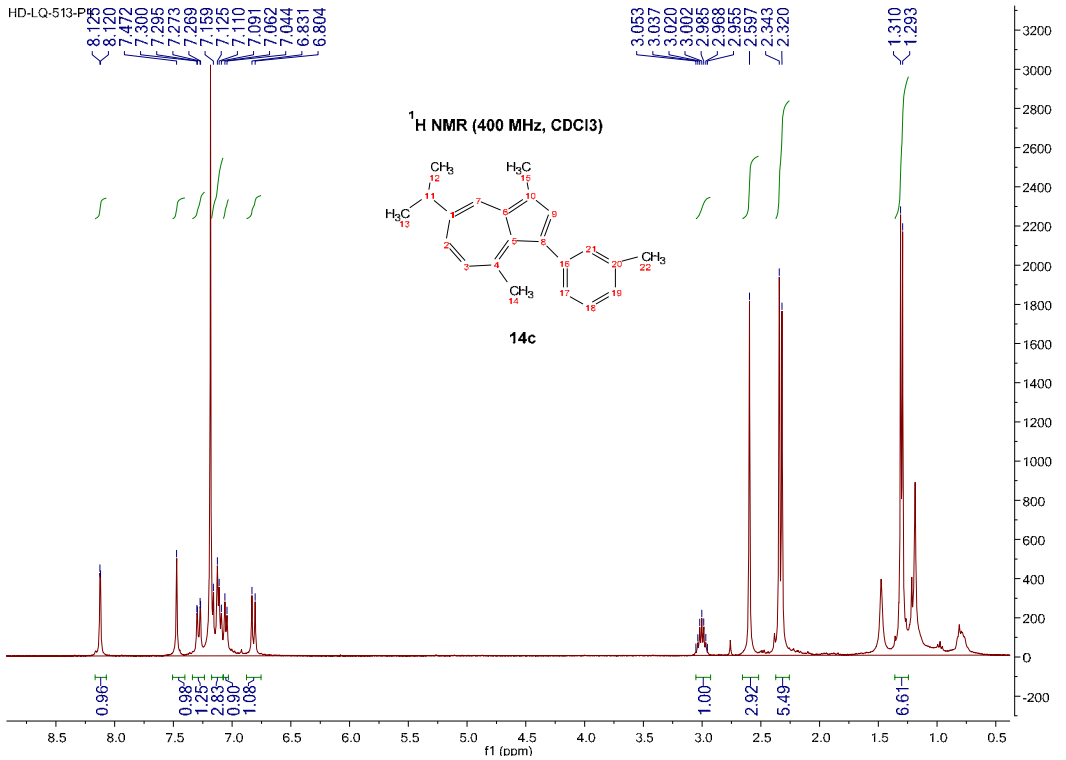

148.486
143.737
140.815
138.455
137.943
137.812
136.644
134.289
133.285
130.397
128.254
127.770
126.808
125.714
121.430
113.660
38.444
24.832
24.175
21.621
11.638
^{13}C NMR (101 MHz, CDCl3)
14b
f1 (ppm)

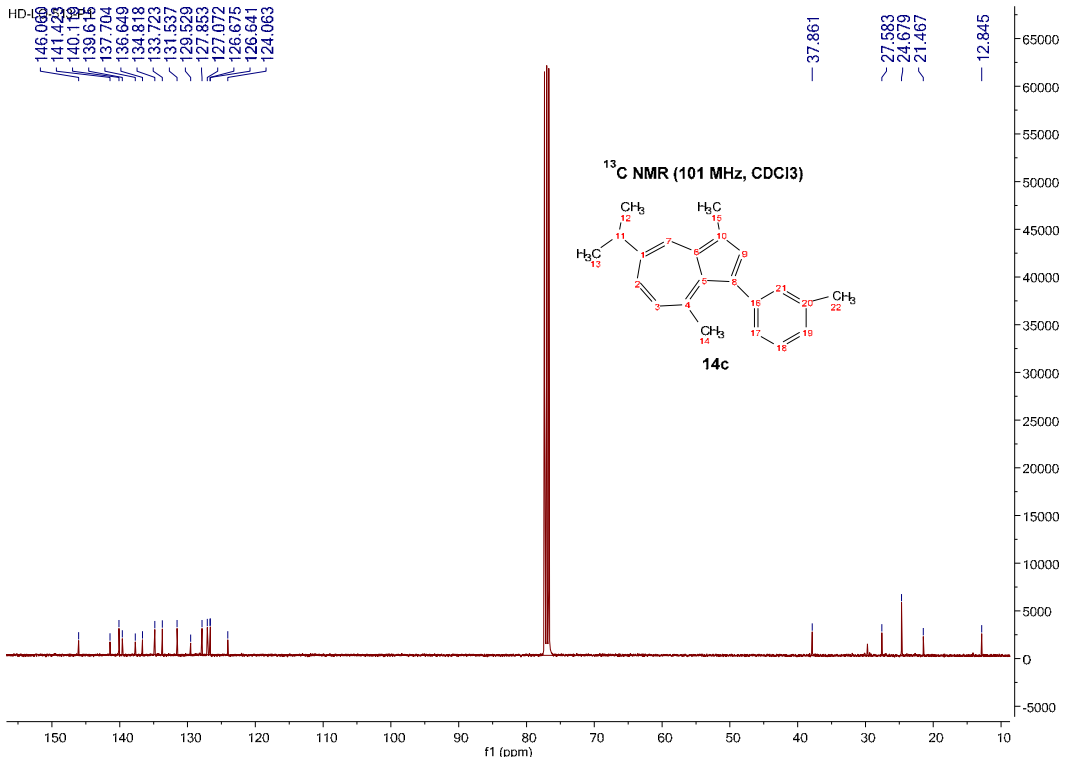

HD-LG-507-P?
8.189
8.182
7.607
7.583
7.434
7.415
7.395
7.312
7.283
6.993
6.967
3.067
3.050
3.032
3.015
2.998
2.783
2.651
1.318
1.300
^{1}H NMR (400 MHz, CDCl3)
18b
1.00
1.92
1.99
3.15
1.03
1.00
3.11
2.94
6.13
f1 (ppm)


HD-LG-507-C?
148.316
143.869
140.877
138.571
137.804
136.689
134.403
133.376
129.691
128.364
126.969
125.767
121.419
113.589
38.441
24.824
24.176
11.615
^{13}C NMR (101 MHz, CDCl3)
18b
f1 (ppm)


HD-LQ-552-P1
8.168
8.163
7.581
7.561
7.457
7.403
7.383
7.374
7.347
7.344
7.180
6.930
6.904
3.062
3.045
3.027
3.010
2.993
2.592
2.335
1.318
1.301
^{1}H NMR (400 MHz, CDCl3)
1c
0.96
2.04
1.03
3.02
1.01
1.00
2.92
2.93
6.01
f1 (ppm)

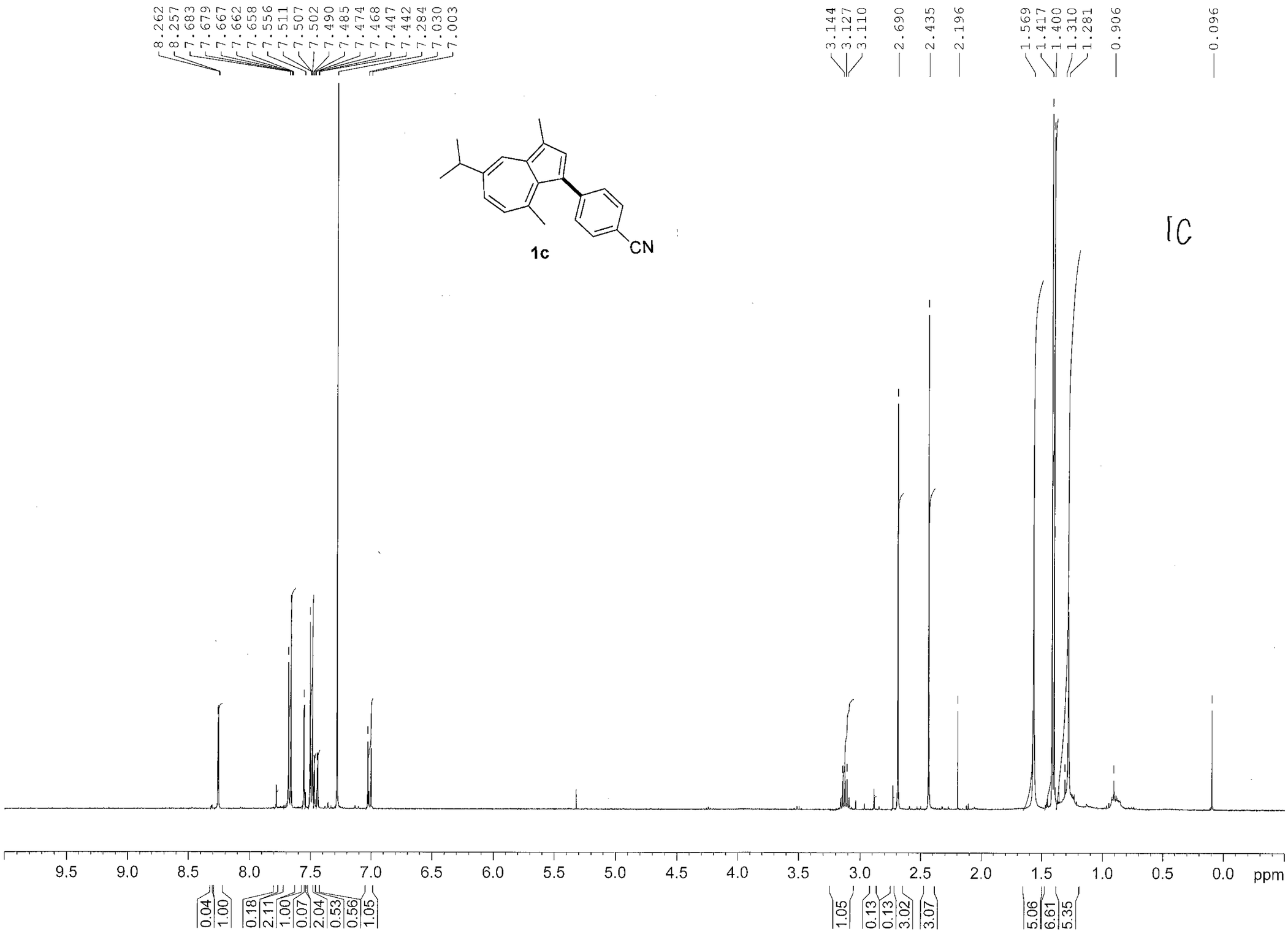

146.386
145.726
141.045
139.759
138.477
135.380
134.196
132.166
131.130
131.067
127.700
126.989
124.718
119.428
109.218
37.921
27.993
24.651
12.830
^{13}C NMR (101 MHz, CDCl3)
1c
f1 (ppm)


HD-LQ-513-PH
8.123
8.120
7.472
7.300
7.295
7.273
7.269
7.159
7.125
7.110
7.091
7.062
7.044
6.831
6.804
3.053
3.037
3.020
3.002
2.985
2.968
2.955
2.597
2.343
2.320
1.310
1.293
^{1}H NMR (400 MHz, CDCl3)
14c
0.96
0.98
1.25
2.83
0.90
1.08
1.00
2.92
5.49
6.61
f1 (ppm)


146.069
141.423
140.114
139.614
137.704
136.649
134.818
133.723
131.537
129.529
127.853
127.072
126.675
126.641
124.063
37.861
27.583
24.679
21.467
12.845
^{13}C NMR (101 MHz, CDCl3)
14c
f1 (ppm)

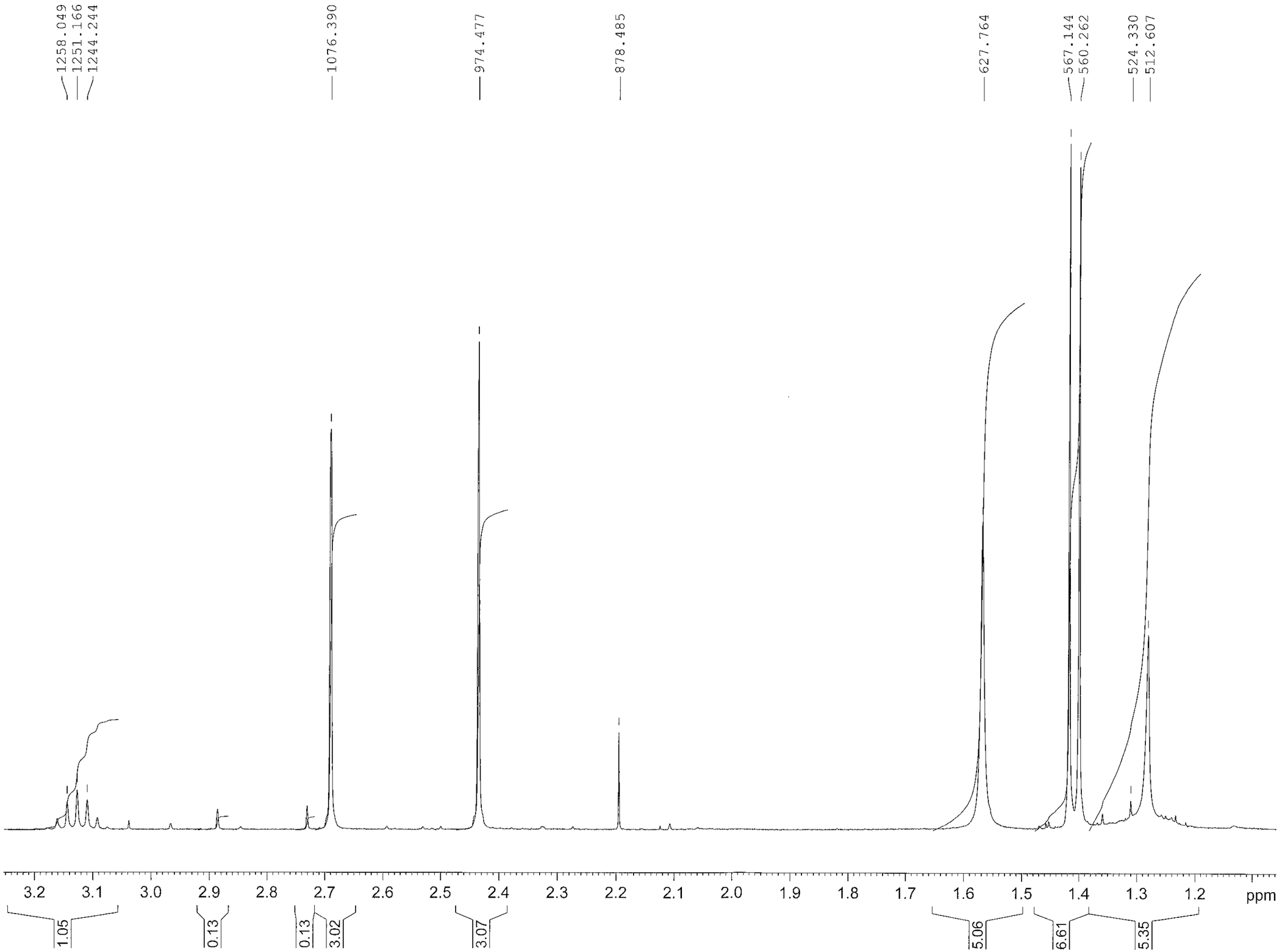
NMR experiments for the determination of the C2:C3 regioselectivity

lq-398-p 1H S.SINBANDHIT



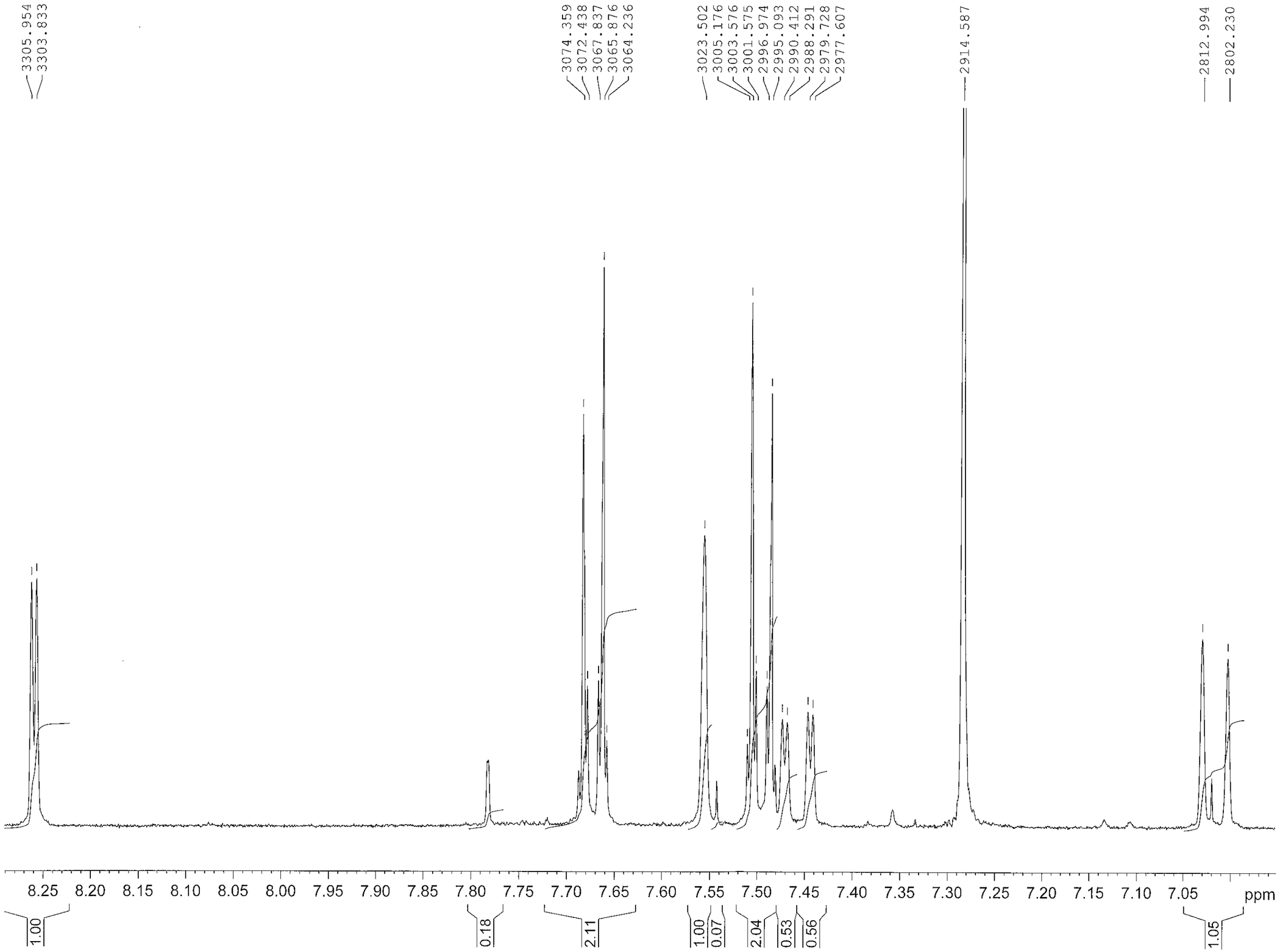
NAME	lq-398-p
EXPNO	1
PROCNO	1
Date_	20121030
Time	10.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4526453 sec
RG	10
DW	83.200 use
DE	6.50 use
TE	298.1 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	10.00 use
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1326809 MHz
SI	32768
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50

lq-398-p 1H S.SINBANDHIT



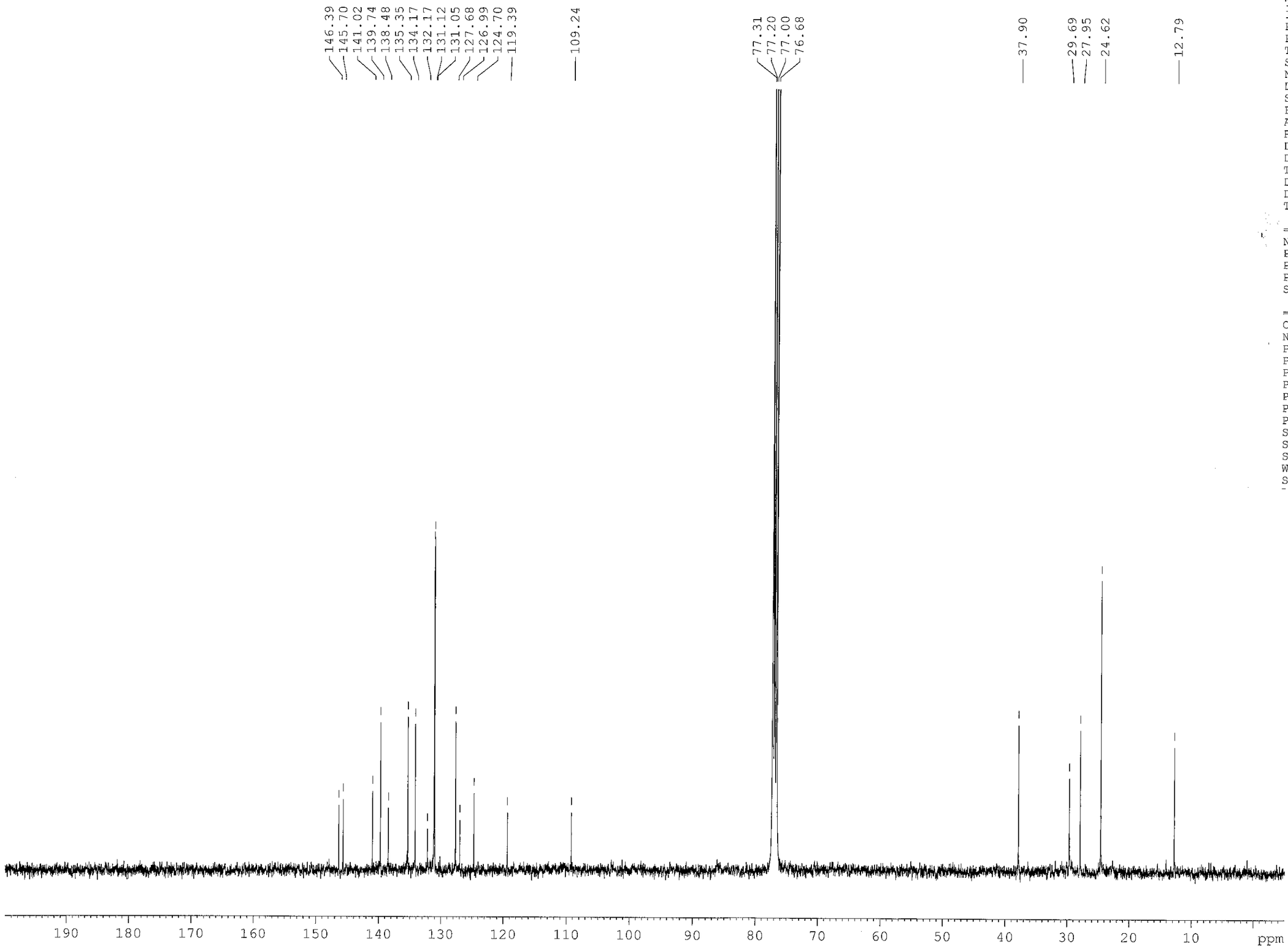
NAME	lq-398-p
EXPNO	1
PROCNO	1
Date_	20121030
Time	10.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4526453 sec
RG	10
DW	83.200 use
DE	6.50 use
TE	298.1 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	10.00 use
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1326809 MHz
SI	32768
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50

lq-398-p 1H S.SINBANDHIT



NAME	lq-398-p
EXPNO	1
PROCNO	1
Date_	20121030
Time	10.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	64
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4526453 sec
RG	10
DW	83.200 use
DE	6.50 use
TE	298.1 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	10.00 use
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1326809 MHz
SI	32768
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.50

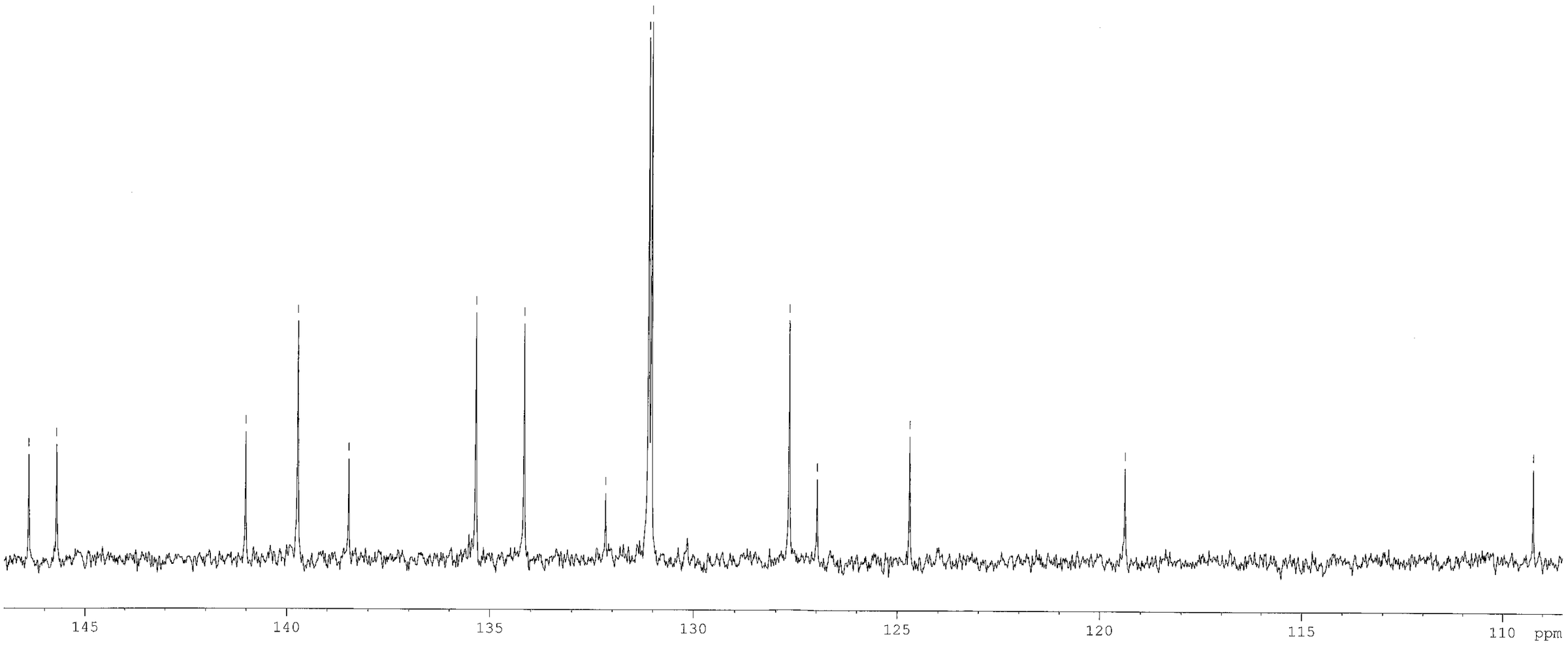
lq-398-p 13C S.SINBANDHIT



NAME	lq-398-p
EXPNO	4
PROCNO	1
Date_	20121031
Time	9.12
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	4570
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 use
DE	10.00 use
TE	299.5 K
D1	5.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	6.80 use
PL1	-3.10 dB
PL1W	66.67198181 W
SFO1	100.6233333 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 use
PL2	-4.80 dB
PL12	15.20 dB
PL13	15.20 dB
PL2W	24.27448273 W
PL12W	0.24274483 W
PL13W	0.24274483 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0

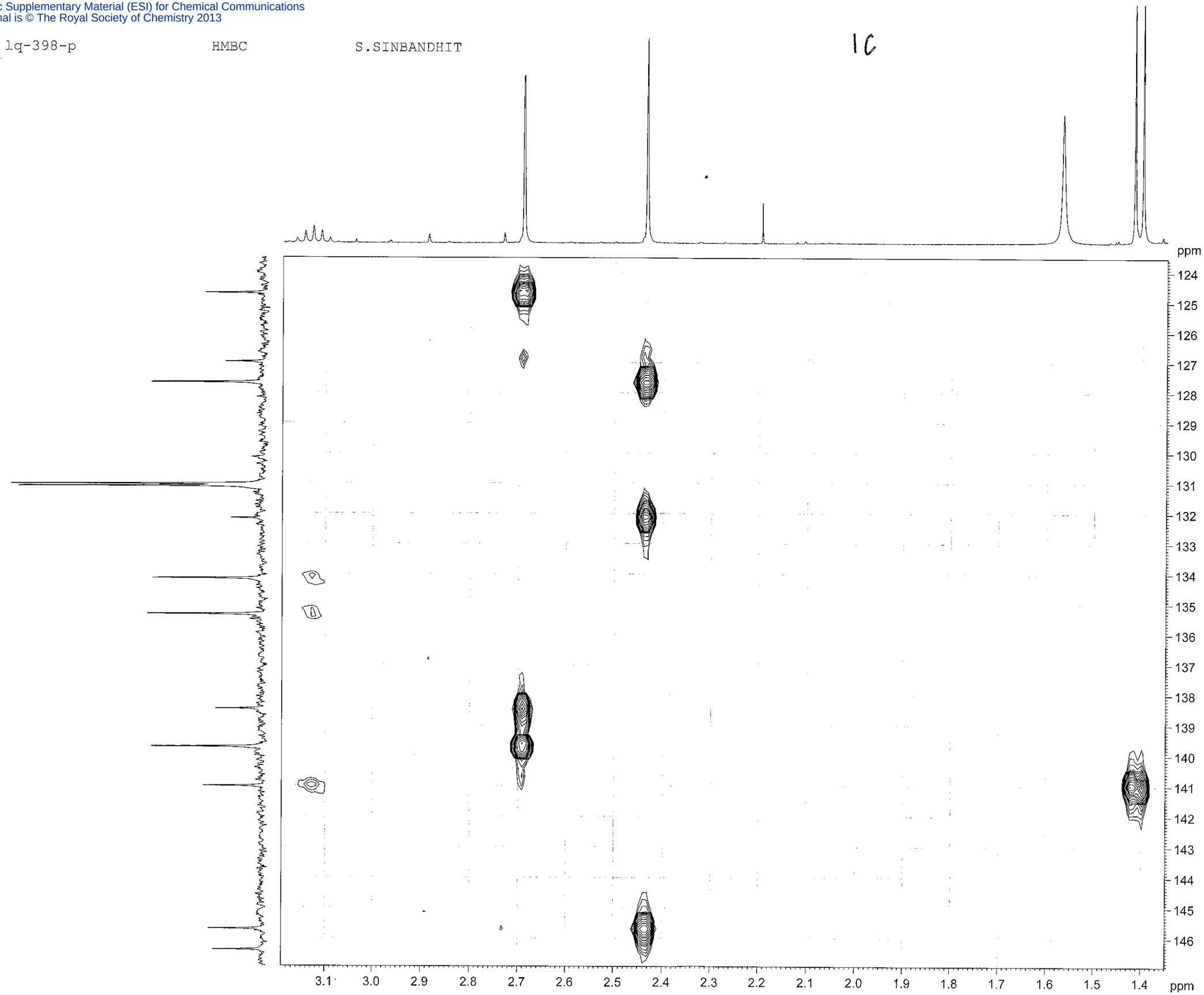
lq-398-p 13C S.SINBANDHIT

146.39
145.70
141.02
139.74
138.48
135.35
134.17
132.17
131.12
131.05
127.68
126.99
124.70
119.39
109.24

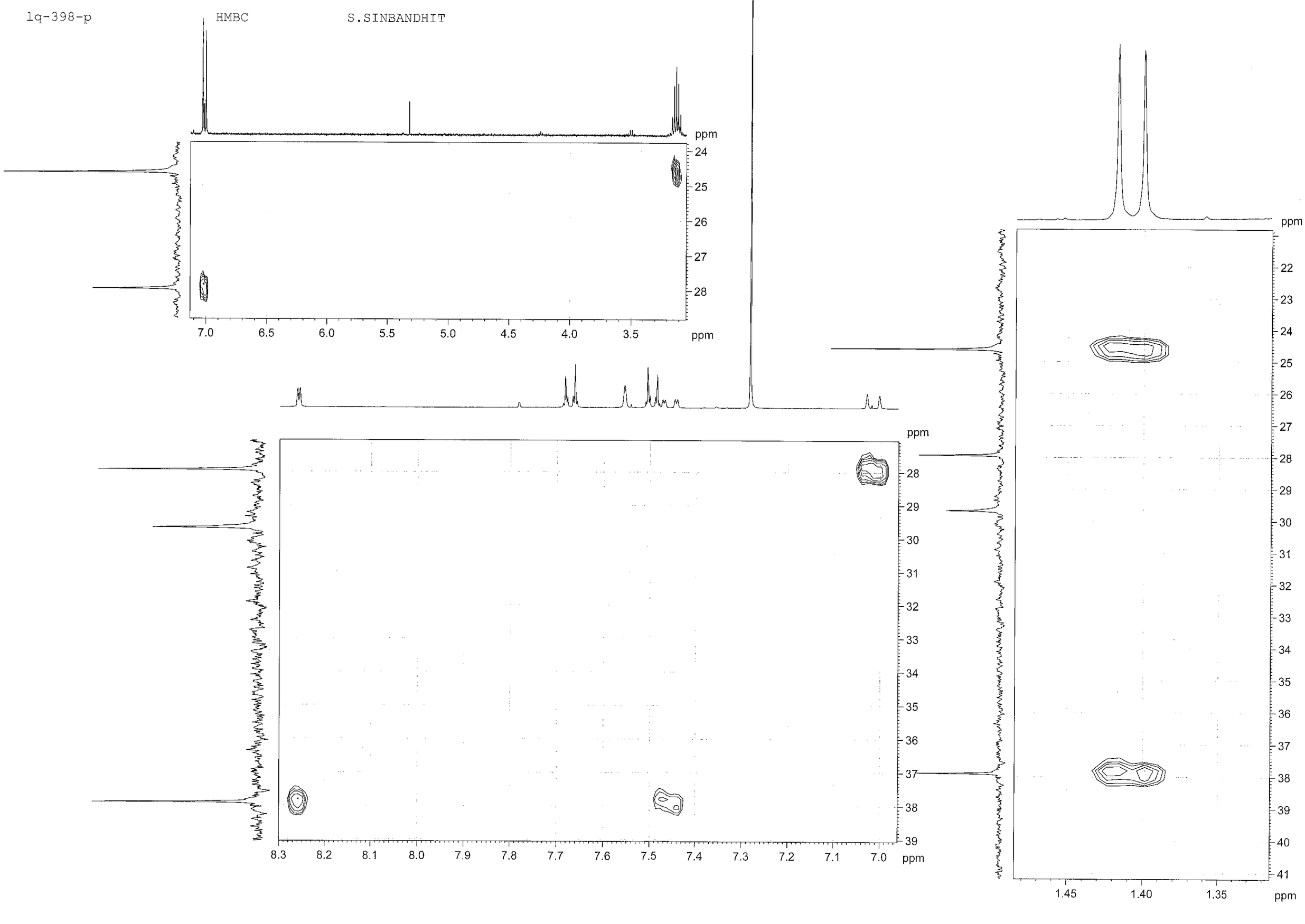


lq-398-p HMBC S.SINBANDHIT

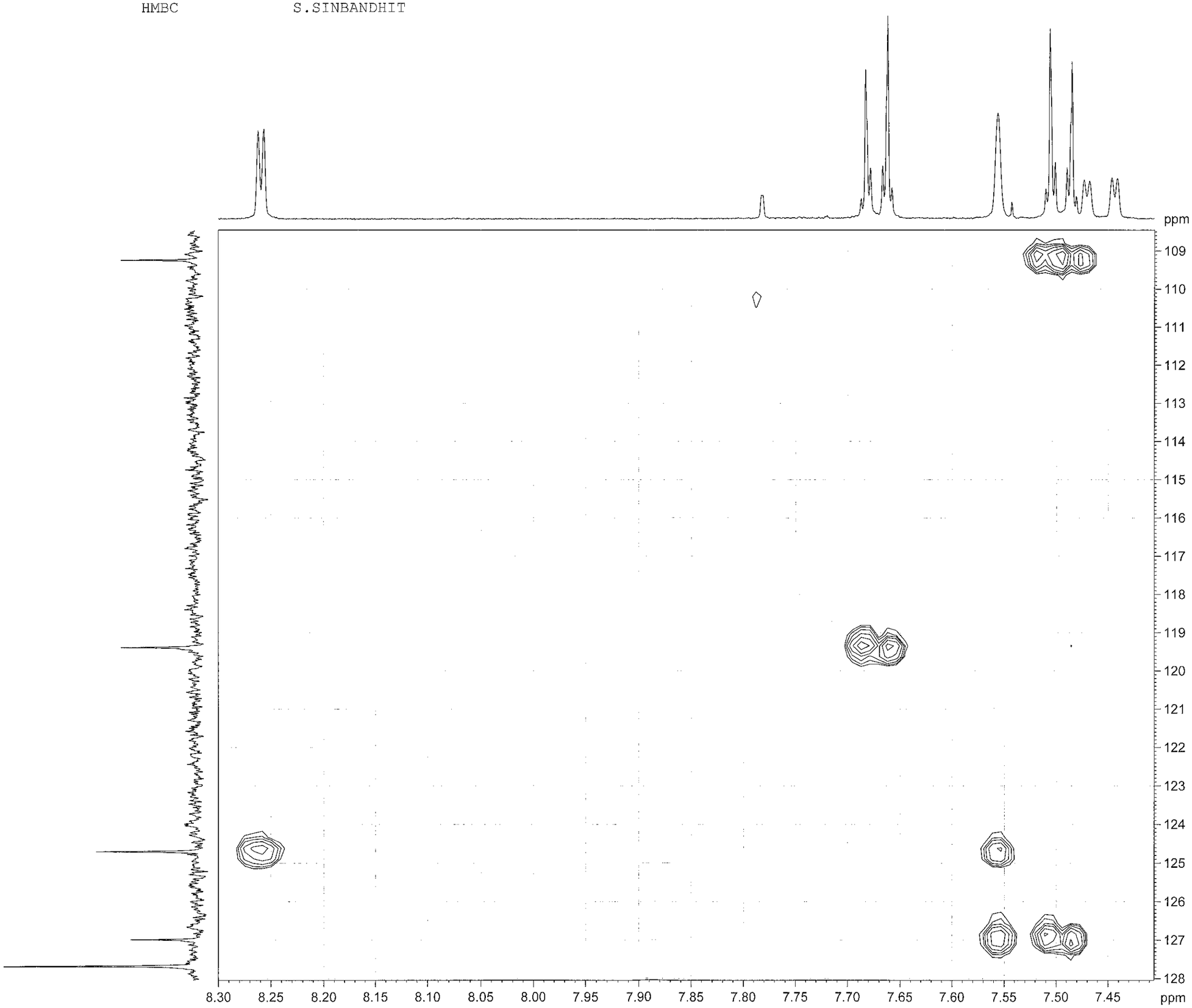
16



NAME	lq-398-p
EXPNO	3
PROCNO	1
Date_	20121030
Time	17.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	40
DS	16
SWH	3440.367 Hz
FIDRES	1.679867 Hz
AQ	0.2976927 sec
RG	2050
DW	145.333 usec
DE	6.50 usec
TE	299.7 K
CNST2	164.0000000
CNST13	6.3000002
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00304878 sec
D6	0.07936507 sec
D16	0.00010000 sec
IN0	0.00003500 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1320007 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	9.00 usec
PL2	-3.10 dB
PL2W	66.67198181 W
SFO2	100.6208180 MHz
======= GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6208 MHz
FIDRES	44.650490 Hz
SW	142.000 ppm
FnMODE	QF
SI	1024
SF	400.1299982 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127590 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

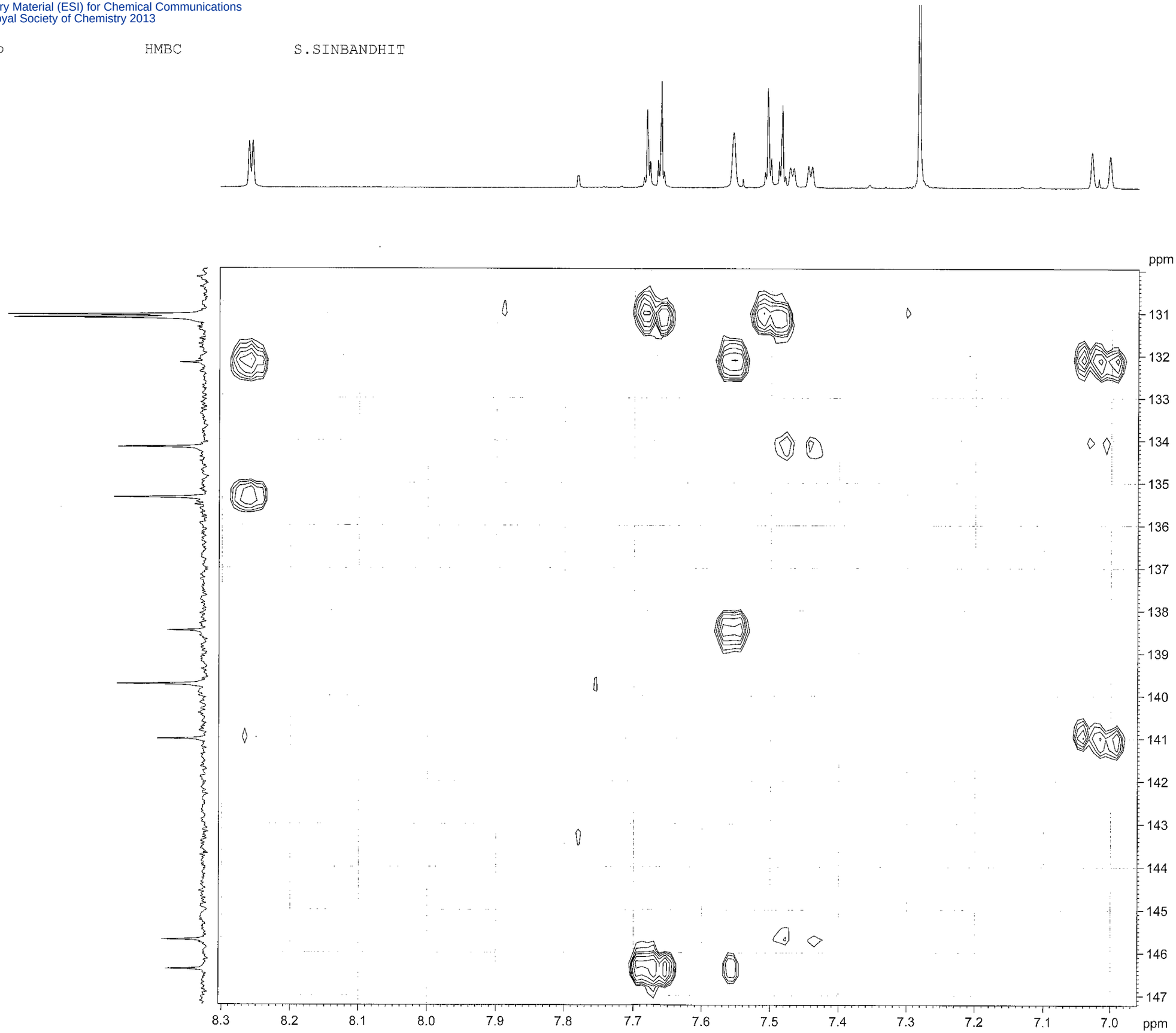


lq-398-p HMBC S.SINBANDHIT



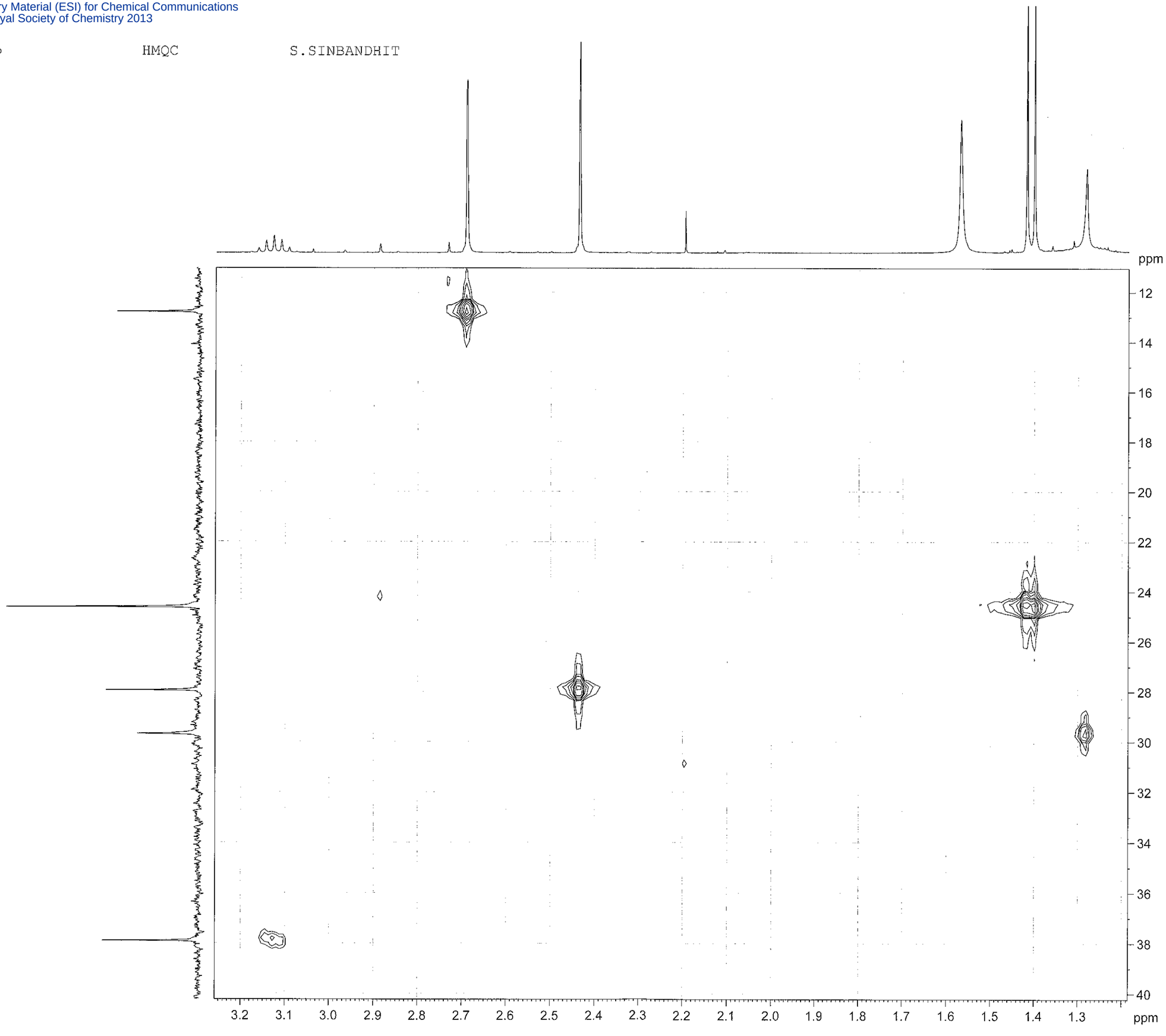
NAME	lq-398-p
EXPNO	3
PROCNO	1
Date_	20121030
Time	17.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	40
DS	16
SWH	3440.367 Hz
FIDRES	1.679867 Hz
AQ	0.2976927 sec
RG	2050
DW	145.333 usec
DE	6.50 usec
TE	299.7 K
CNST2	164.0000000
CNST13	6.3000002
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00304878 sec
D6	0.07936507 sec
D16	0.00010000 sec
IN0	0.00003500 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1320007 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	9.00 usec
PL2	-3.10 dB
PL2W	66.67198181 W
SFO2	100.6208180 MHz
======= GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6208 MHz
FIDRES	44.650490 Hz
SW	142.000 ppm
FnMODE	QF
SI	1024
SF	400.1299982 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127590 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

lq-398-p HMBC S.SINBANDHIT



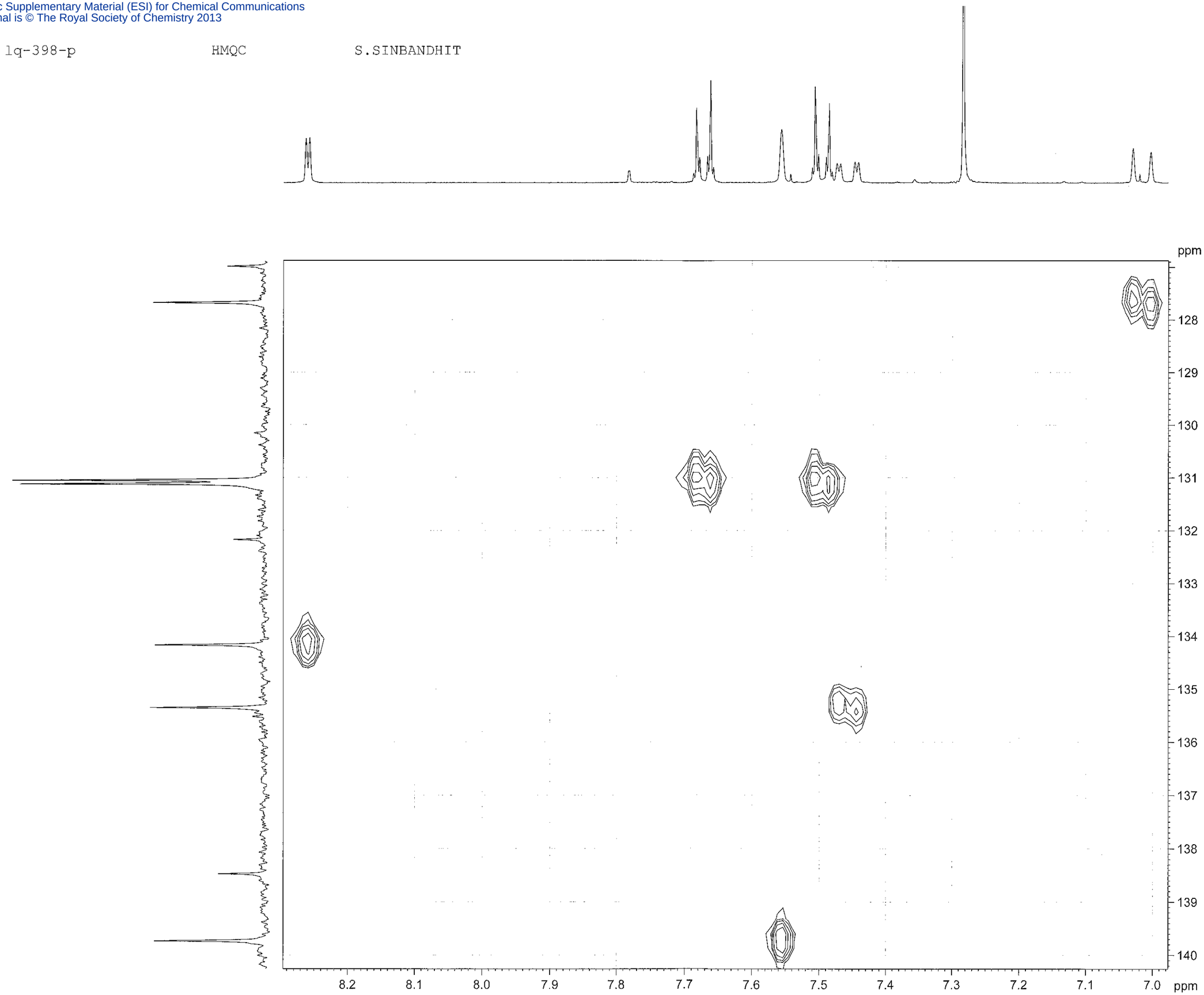
NAME	lq-398-p
EXPNO	3
PROCNO	1
Date_	20121030
Time	17.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	40
DS	16
SWH	3440.367 Hz
FIDRES	1.679867 Hz
AQ	0.2976927 sec
RG	2050
DW	145.333 usec
DE	6.50 usec
TE	299.7 K
CNST2	164.0000000
CNST13	6.3000002
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00304878 sec
D6	0.07936507 sec
D16	0.00010000 sec
IN0	0.00003500 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1320007 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	9.00 usec
PL2	-3.10 dB
PL2W	66.67198181 W
SFO2	100.6208180 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6208 MHz
FIDRES	44.650490 Hz
SW	142.000 ppm
FnMODE	QF
SI	1024
SF	400.1299982 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127590 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

lq-398-p HMQC S.SINBANDHIT



NAME	lq-398-p
EXPNO	2
PROCNO	1
Date_	20121030
Time	11.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmqcgpqf
TD	2048
SOLVENT	CDCl3
NS	32
DS	16
SWH	3440.367 Hz
FIDRES	1.679867 Hz
AQ	0.2976927 sec
RG	2050
DW	145.333 usec
DE	6.50 usec
TE	298.7 K
CNST2	144.0000000
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00347222 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00010000 sec
IN0	0.00003500 sec
========	CHANNEL f1 ========
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1320007 MHz
========	CHANNEL f2 ========
CPDPRG2	garp
NUC2	13C
P3	9.00 usec
PCPD2	80.00 usec
PL2	-3.10 dB
PL12	15.50 dB
PL2W	66.67198181 W
PL12W	0.92032951 W
SFO2	100.6208180 MHz
======	GRADIENT CHANNEL =====
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6208 MHz
FIDRES	44.650490 Hz
SW	142.000 ppm
FnMODE	QF
SI	1024
SF	400.1299980 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127590 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

lq-398-p HMQC S.SINBANDHIT



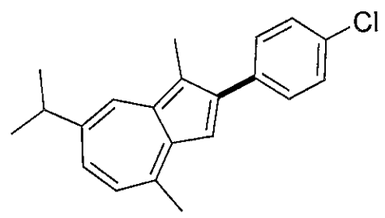
NAME	lq-398-p
EXPNO	2
PROCNO	1
Date_	20121030
Time	11.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmqcgpqf
TD	2048
SOLVENT	CDCl3
NS	32
DS	16
SWH	3440.367 Hz
FIDRES	1.679867 Hz
AQ	0.2976927 sec
RG	2050
DW	145.333 usec
DE	6.50 usec
TE	298.7 K
CNST2	144.0000000
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00347222 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00010000 sec
IN0	0.00003500 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1320007 MHz
======== CHANNEL f2 ========	
CPDPRG2	garp
NUC2	13C
P3	9.00 usec
PCPD2	80.00 usec
PL2	-3.10 dB
PL12	15.50 dB
PL2W	66.67198181 W
PL12W	0.92032951 W
SFO2	100.6208180 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6208 MHz
FIDRES	44.650490 Hz
SW	142.000 ppm
FnMODE	QF
SI	1024
SF	400.1299980 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127590 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

66

8.297 8.292 7.637 7.636 7.632 7.621 7.616 7.610 7.506 7.500 7.495 7.484 7.480 7.479 7.468 7.463 7.442 7.437 7.357 7.284 7.115 7.088

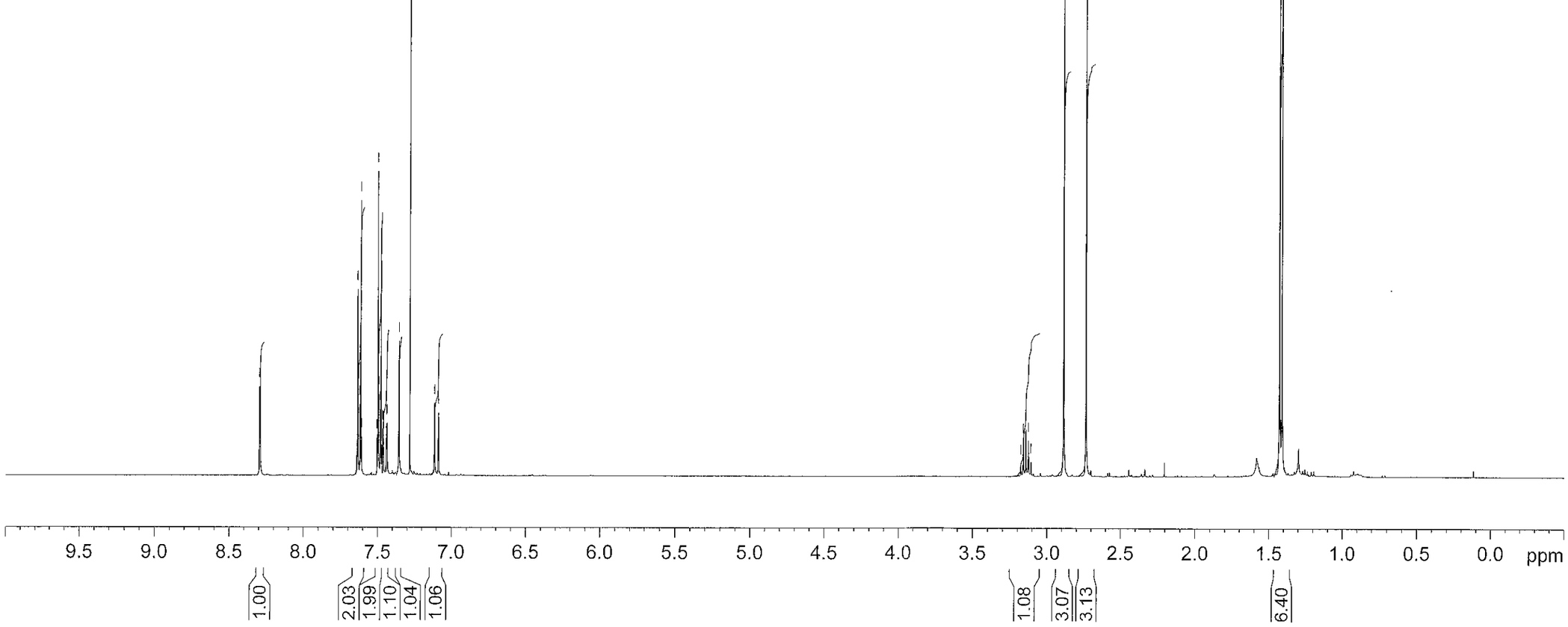
3.179 3.161 3.144 3.127 3.110 2.889 2.736

1.429 1.422 1.419 1.412 1.405

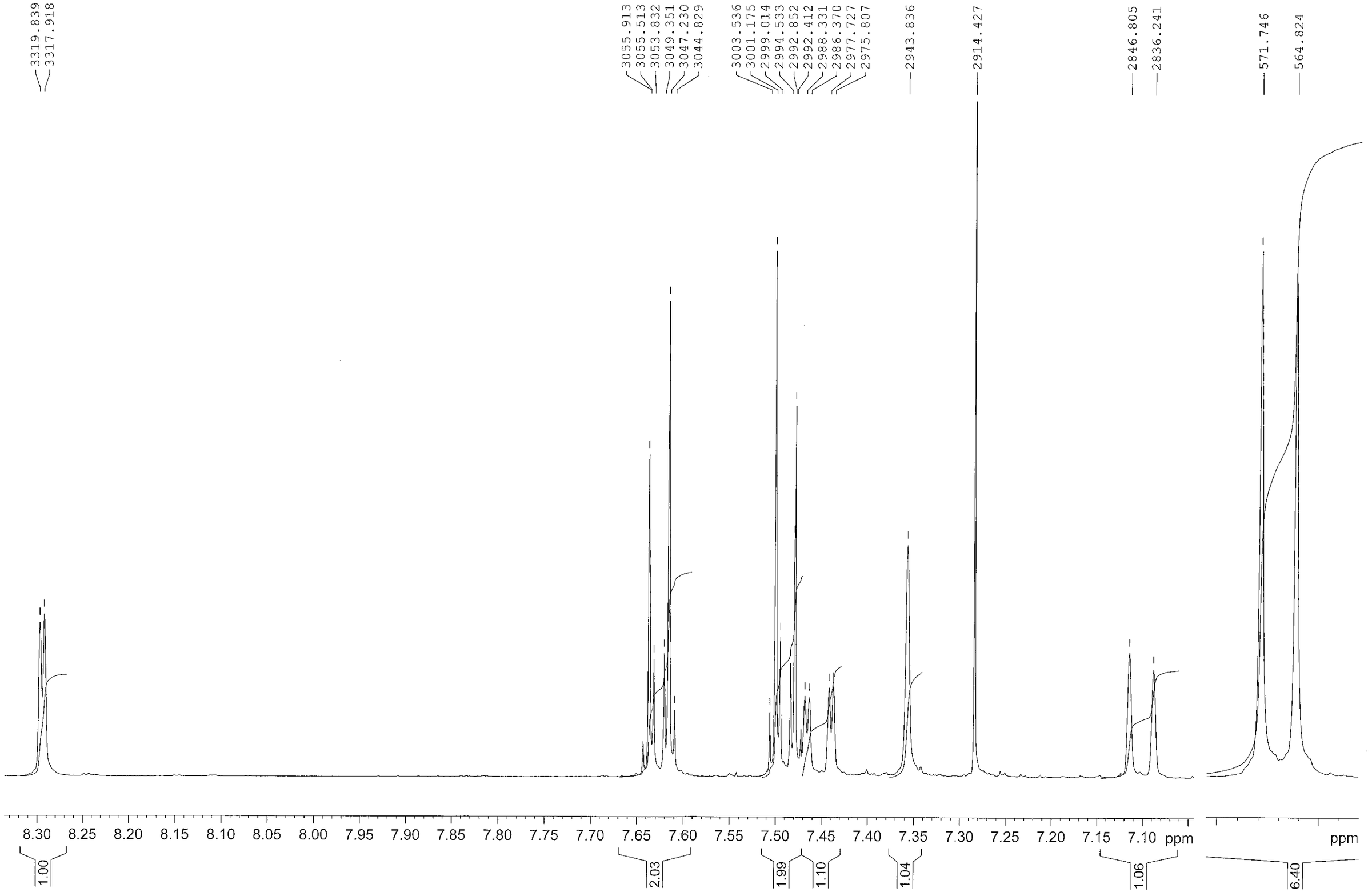


6b

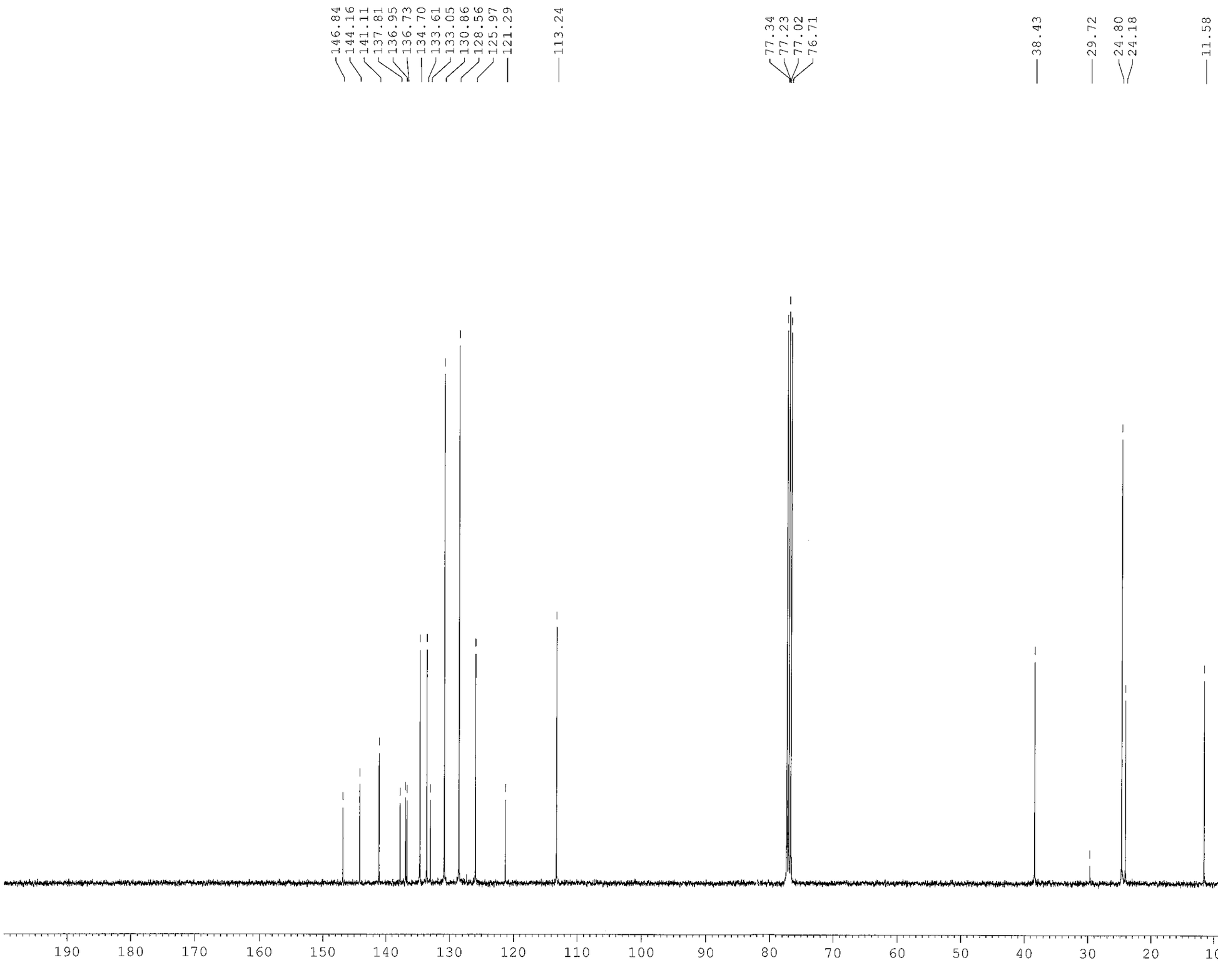
NAME	lq499-p1
EXPNO	1
PROCNO	1
Date_	20121217
Time	16.59
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	74
DS	2
SWH	6009.615 Hz
FIDRES	0.091699 Hz
AQ	5.4526453 sec
RG	101
DW	83.200 use
DE	6.50 use
TE	297.1 K
D1	0.50000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	1H
P1	10.00 use
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1326809 MHz
SI	32768
SF	400.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.60



lq499-p1 1H S.SINBANDHIT

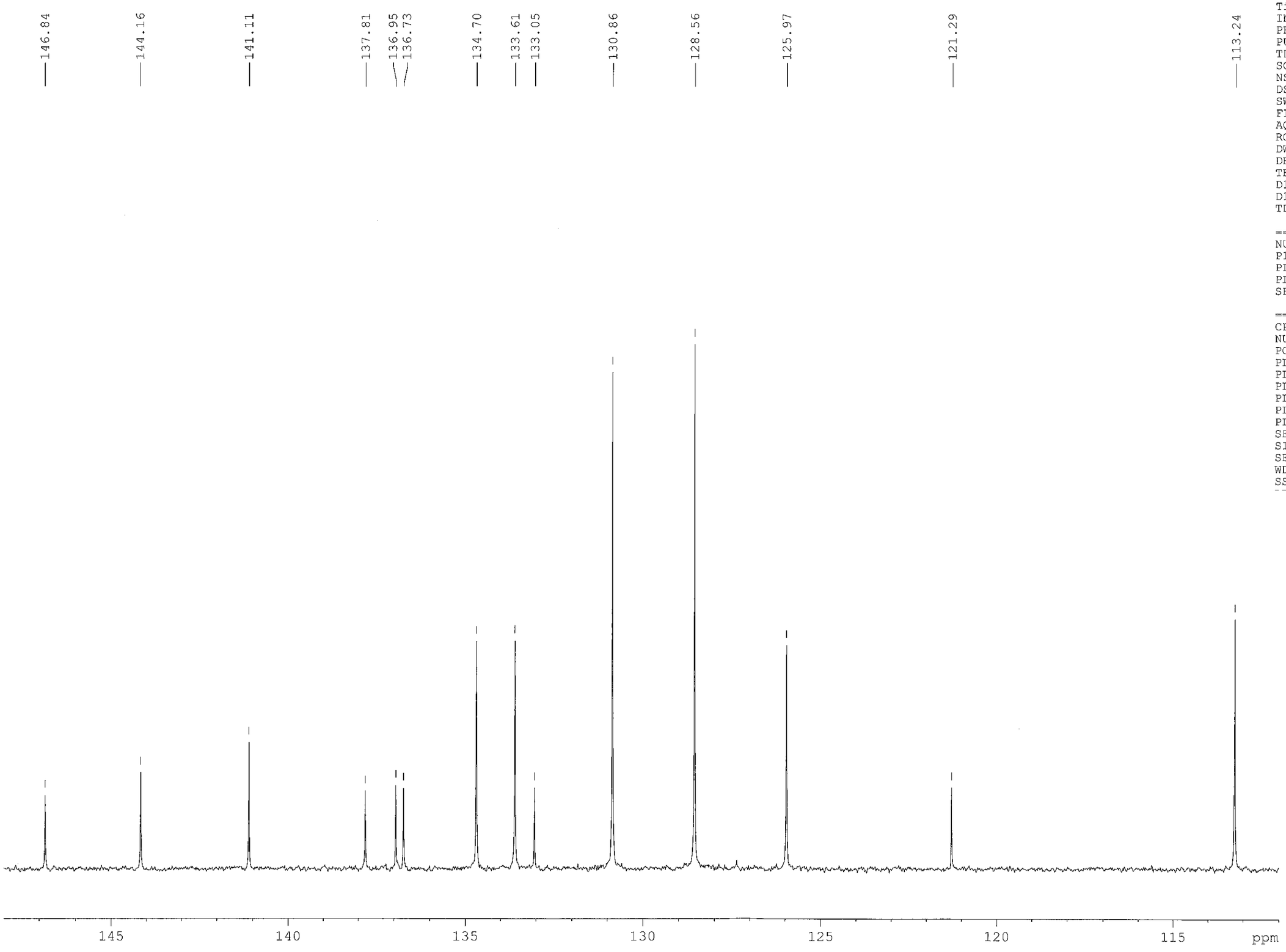


lq499-p1 13C S.SINBANDHIT



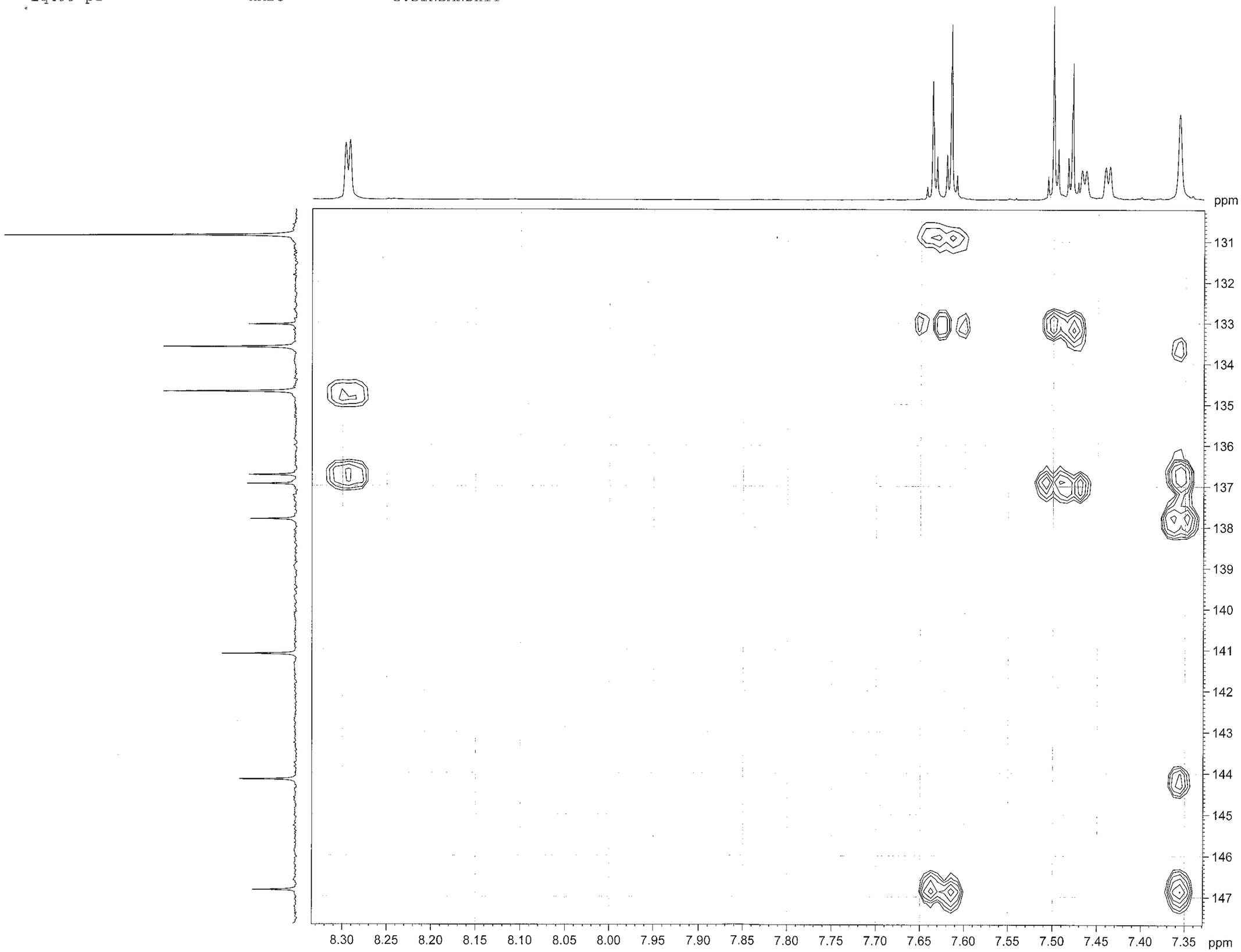
NAME	lq499-p1
EXPNO	4
PROCNO	1
Date_	20121217
Time	17.09
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	853
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 use
DE	10.00 use
TE	298.0 K
D1	5.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	6.70 use
PL1	-3.10 dB
PL1W	66.67198181 W
SFO1	100.6233333 MH:
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 use
PL2	-4.80 dB
PL12	15.20 dB
PL13	15.20 dB
PL2W	24.27448273 W
PL12W	0.24274483 W
PL13W	0.24274483 W
SFO2	400.1316005 MH:
SI	32768
SF	100.6127690 MH:
WDW	EM
SSB	0

lq499-p1 13C S.SINBANDHIT

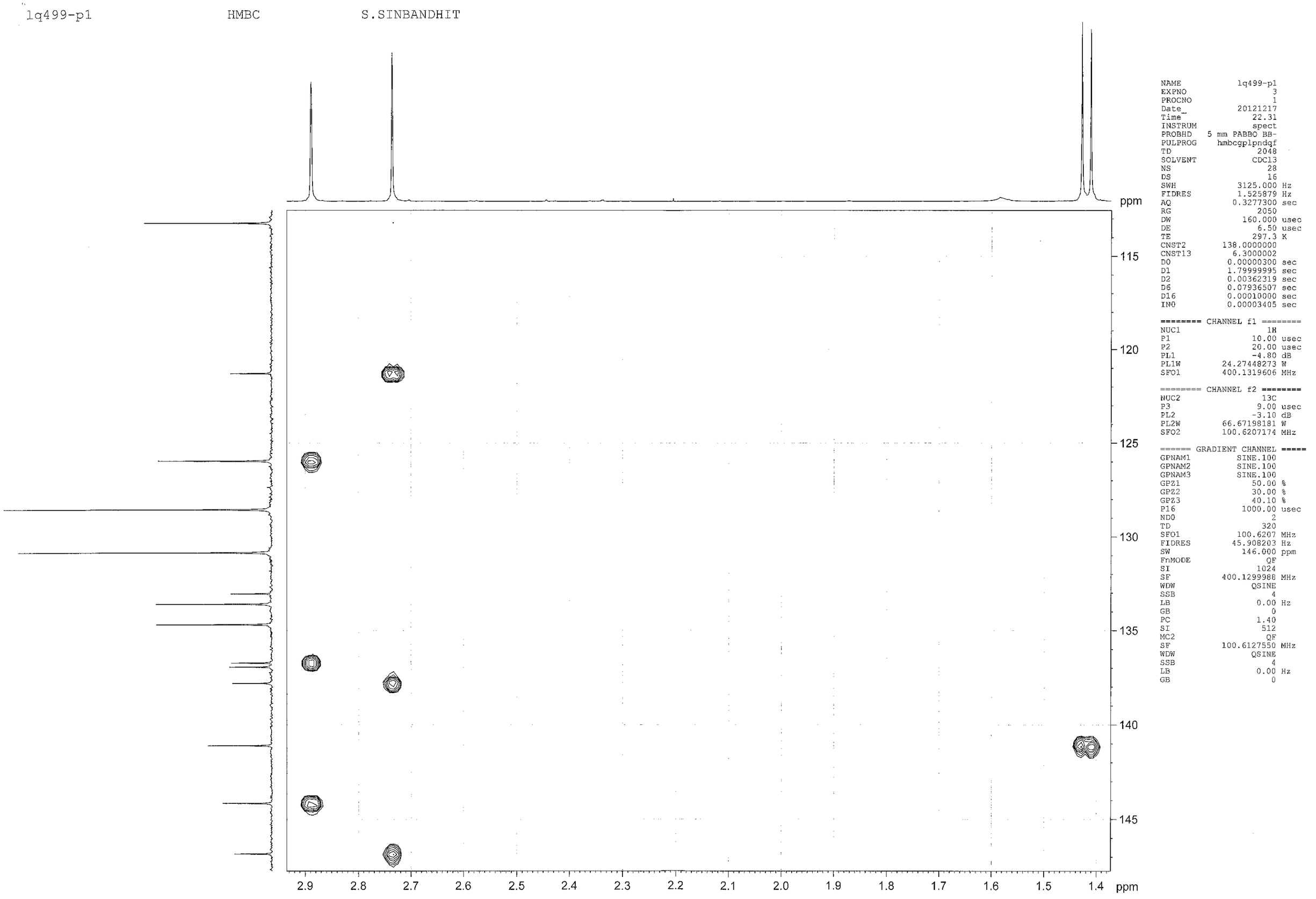


NAME	lq499-p1
EXPNO	4
PROCNO	1
Date_	20121217
Time	17.09
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	853
DS	2
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	10.00 usec
TE	298.0 K
D1	5.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	6.70 usec
PL1	-3.10 dB
PL1W	66.67198181 W
SFO1	100.6233333 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-4.80 dB
PL12	15.20 dB
PL13	15.20 dB
PL2W	24.27448273 W
PL12W	0.24274483 W
PL13W	0.24274483 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0

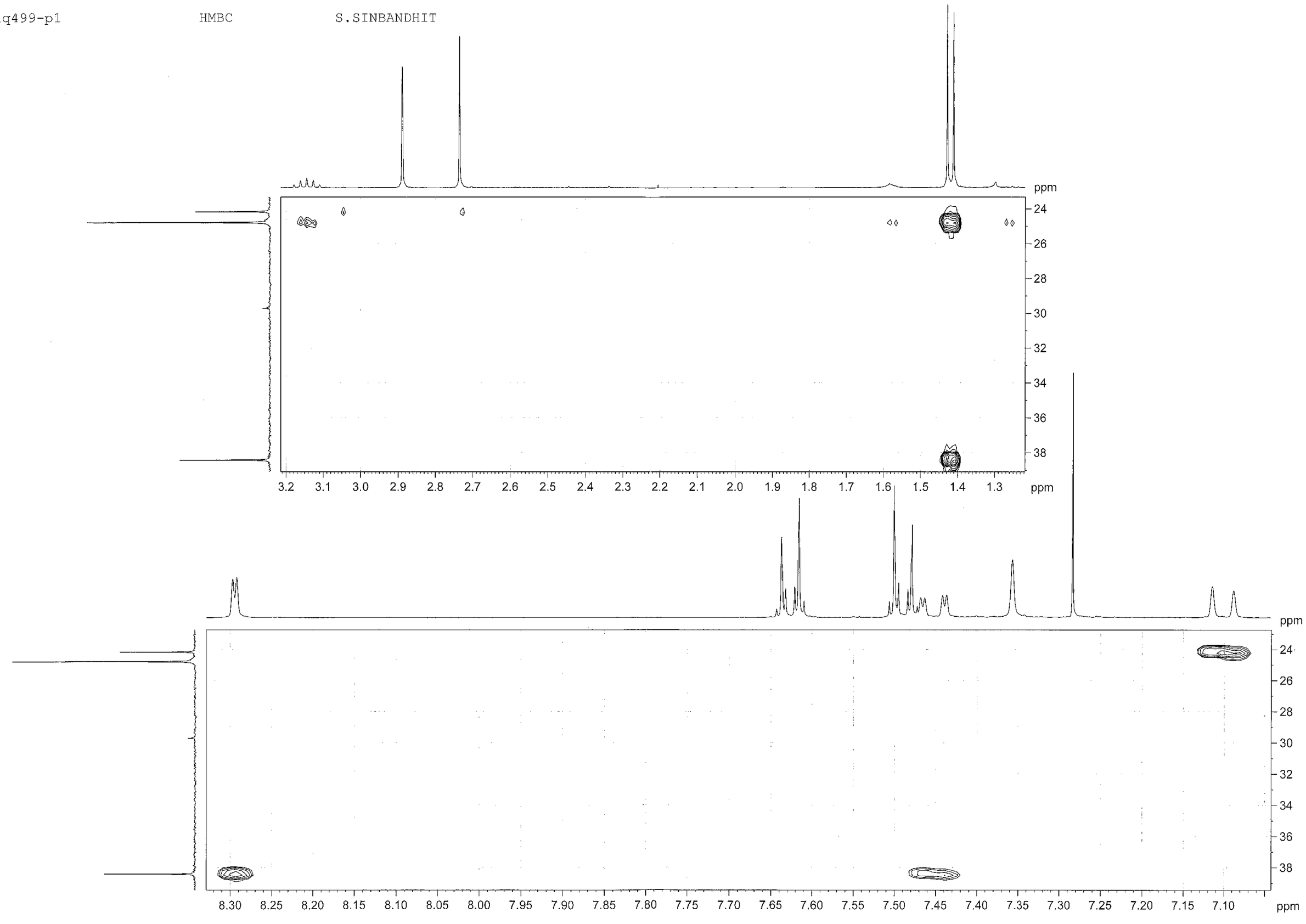
lq499-p1 HMBC S.SINBANDHIT

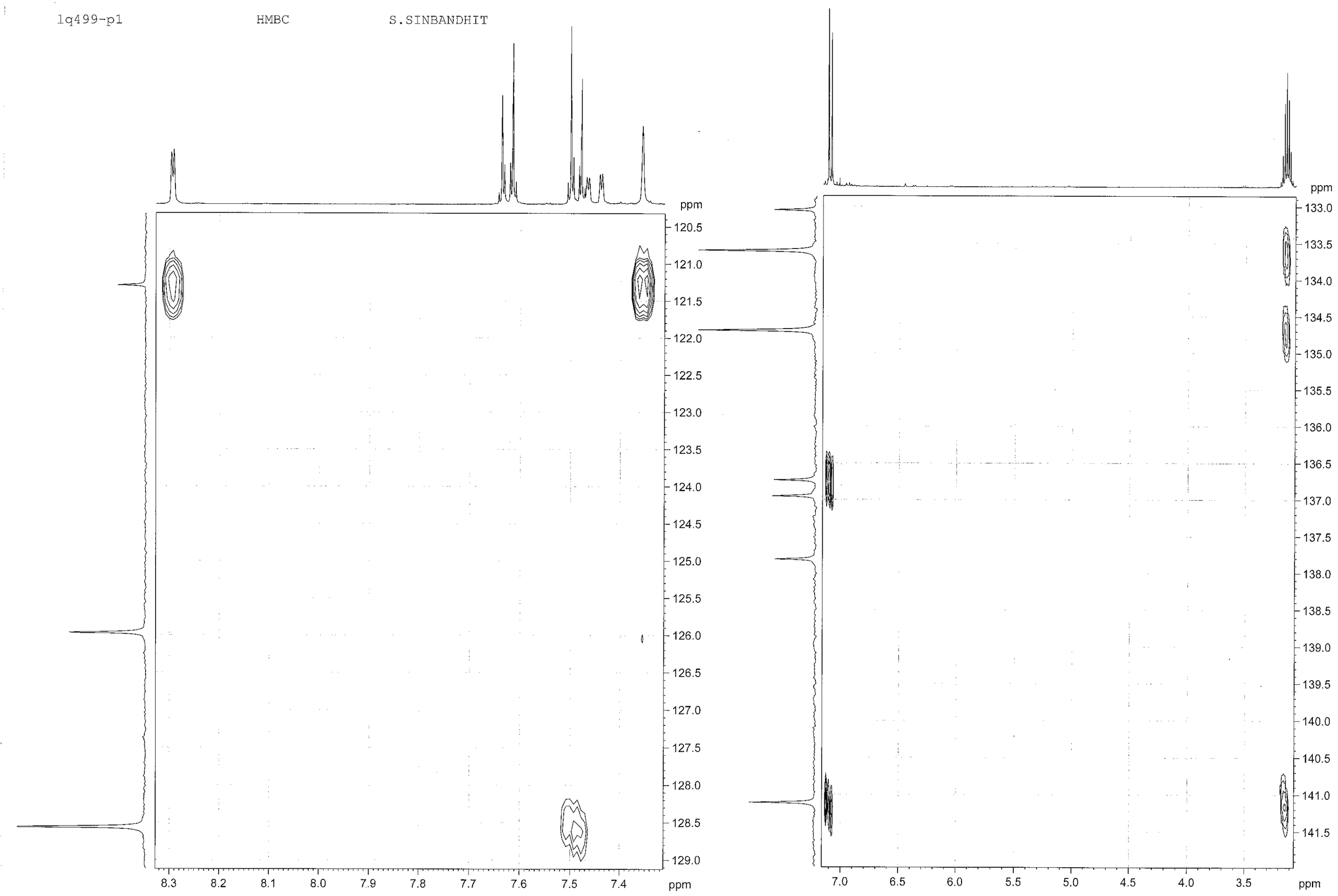


NAME	lq499-p1
EXPNO	3
PROCNO	1
Date_	20121217
Time	22.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	28
DS	16
SWH	3125.000 Hz
FIDRES	1.525879 Hz
AQ	0.3277300 sec
RG	2050
DW	160.000 usec
DE	6.50 usec
TE	297.3 K
CNST2	138.0000000
CNST13	6.3000002
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00362319 sec
D6	0.07936507 sec
D16	0.00010000 sec
IN0	0.00003405 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1319606 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	9.00 usec
PL2	-3.10 dB
PL2W	66.67198181 W
SFO2	100.6207174 MHz
======= GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
NDO	2
TD	320
SFO1	100.6207 MHz
FIDRES	45.908203 Hz
SW	146.000 ppm
FnMODE	QF
SI	1024
SF	400.1299988 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127550 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0



NAME	lq499-p1
EXPNO	3
PROCNO	1
Date_	20121217
Time	22.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	28
DS	16
SWH	3125.000 Hz
FIDRES	1.525879 Hz
AQ	0.3277300 sec
RG	2050
DW	160.000 usec
DE	6.50 usec
TE	297.3 K
CNST2	138.0000000
CNST13	6.3000002
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00362319 sec
D6	0.07936507 sec
D16	0.00010000 sec
IN0	0.00003405 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1319606 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	9.00 usec
PL2	-3.10 dB
PL2W	66.67198181 W
SFO2	100.6207174 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6207 MHz
FIDRES	45.908203 Hz
SW	146.000 ppm
FnMODE	QF
SI	1024
SF	400.1299988 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127550 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0





lq499-p1 HMQC S.SINBANDHIT

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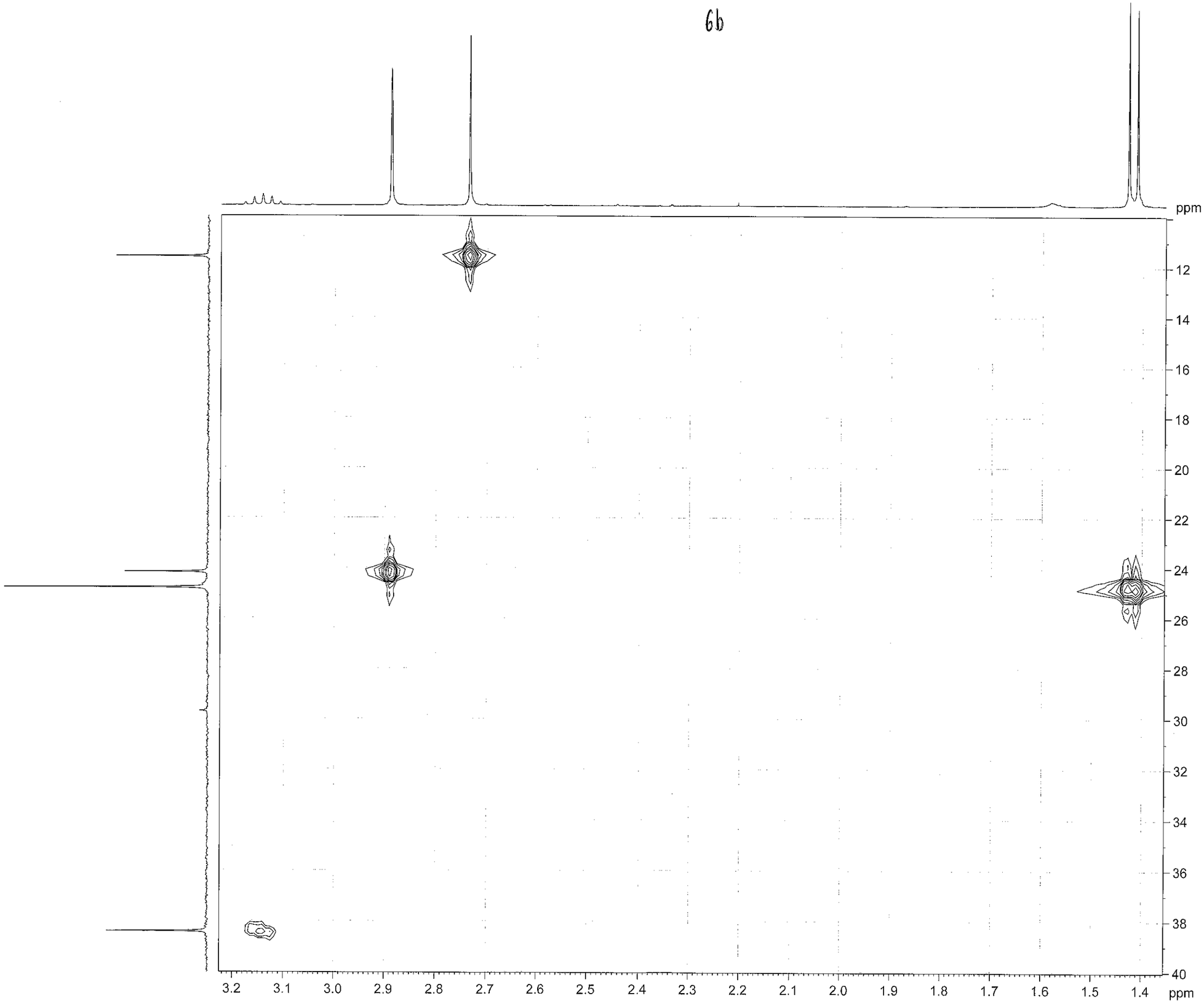
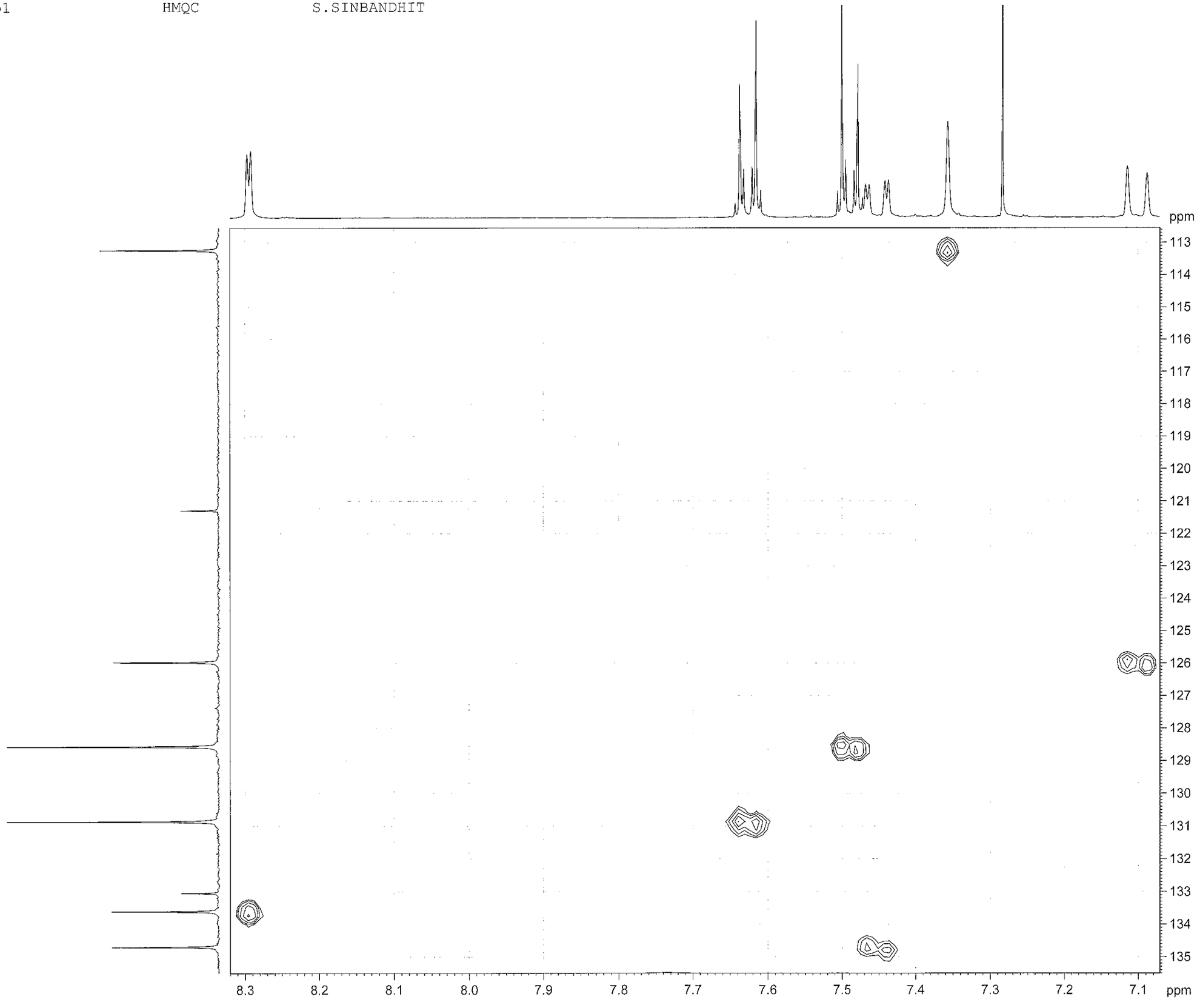


NAME	lq499-p1
EXPNO	2
PROCNO	1
Date_	20121217
Time	18.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmqcgpqf
TD	2048
SOLVENT	CDCl3
NS	20
DS	16
SWH	3205.128 Hz
FIDRES	1.565004 Hz
AQ	0.3195380 sec
RG	2050
DW	156.000 usec
DE	6.50 usec
TE	297.9 K
CNST2	150.0000000
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00333333 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00010000 sec
IN0	0.00003710 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1319606 MHz
======== CHANNEL f2 ========	
CPDPRG2	garp
NUC2	13C
P3	9.00 usec
PCPD2	80.00 usec
PL2	-3.10 dB
PL12	15.50 dB
PL2W	66.67198181 W
PL12W	0.92032951 W
SFO2	100.6201137 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6201 MHz
FIDRES	42.134674 Hz
SW	134.000 ppm
FnMODE	QF
SI	1024
SF	400.1299980 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127540 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0

lq499-p1 HMQC S.SINBANDHIT



NAME	lq499-p1
EXPNO	2
PROCNO	1
Date_	20121217
Time	18.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	hmqcgpqf
TD	2048
SOLVENT	CDCl3
NS	20
DS	16
SWH	3205.128 Hz
FIDRES	1.565004 Hz
AQ	0.3195380 sec
RG	2050
DW	156.000 usec
DE	6.50 usec
TE	297.9 K
CNST2	150.0000000
D0	0.00000300 sec
D1	1.79999995 sec
D2	0.00333333 sec
D12	0.00002000 sec
D13	0.00000400 sec
D16	0.00010000 sec
IN0	0.00003710 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	10.00 usec
P2	20.00 usec
PL1	-4.80 dB
PL1W	24.27448273 W
SFO1	400.1319606 MHz
======== CHANNEL f2 ========	
CPDPRG2	garp
NUC2	13C
P3	9.00 usec
PCPD2	80.00 usec
PL2	-3.10 dB
PL12	15.50 dB
PL2W	66.67198181 W
PL12W	0.92032951 W
SFO2	100.6201137 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	320
SFO1	100.6201 MHz
FIDRES	42.134674 Hz
SW	134.000 ppm
FnMODE	QF
SI	1024
SF	400.1299980 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0
PC	1.40
SI	512
MC2	QF
SF	100.6127540 MHz
WDW	QSINE
SSB	4
LB	0.00 Hz
GB	0