Supporting Materials

Iron-catalyzed cross-aldol reactions of ortho-diketones and methyl ketones

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Contents

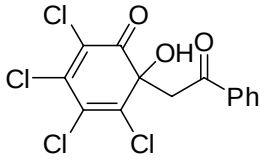
1) Experimental details and characterization data for all compounds

General information: ^{1}H NMR spectra were recorded on AVANCE 400 MHz, JEOL 400 MHz and JEOL 600 MHz spectrometers and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. The peak patterns are indicated as follows: s, singlet; d, doublet; bs, broad singlet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, *J*, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained on AVANCE 100.6 MHz, JEOL 99.5 and JEOL 150.9 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in $CDCl_3$). Mass spectra were determined with AEI-MS 50 for EI-MS; APEX Ⅱ (Bruker Inc.) for HR-MS and ESI-MS. IR spectra were recorded by a Nicolet 5MX-S infrared spectrometer. Flash column chromatography was performed over silica gel 200-300. X-ray data collections were performed at 20 °C on a Rigaku RAXIS RAPID IP diffractometer, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the Rapid-AUTO (Rigaku 2000) program package. The raw frame data were processed using Crystal Structure (Rigaku/MSC 2000) to yield the reflection data file. The structure was solved by use of SHELXTL program. Refinement was performed on F^2 anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. (G. M. Sheldrick, *SHELXTL 5.10 for Windows NT*: Structure Determination Software Programs; Bruker Analytical X-ray Systems, Inc.: Madison, WI, 1997.) All reagents were weighed and handled in air at room temperature. Unless otherwise noted, all reactions were performed under a nitrogen atmosphere. All chemicals were purchased from Alfa, Acros, Aldrich, TCI, and Strem and used without further purification.

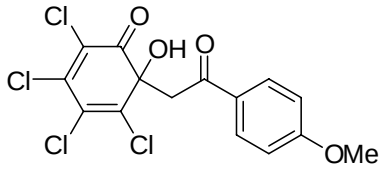
General procedure for products 3: To a 2.0 mL solution of **1** (0.5 mmol) in petroleum ether (PE) under N_2 at room temperature was added **2** (0.75 mmol, 1.5 eq). The resulting mixture was stirred for 24h at room temperature. The resulting reaction solution was mixed with few silica gel and evaporated in vacuo. The residue was purified by flash column chromatography using silica gel (ethyl acetate : PE = 1 : 5) to afford the desired products.

General procedure for products 4: An oven-dried Schlenk tube was charged with **3** (0.2 mmol) under N_2 at room temperature. The Schlenk tube was put into a pre-heated oil

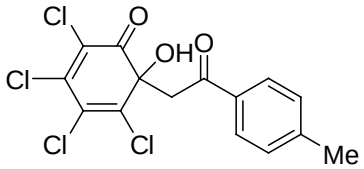
bath at 130 ^{o}C for 40 minutes. The reaction mixture was quenched with saturated $NaHCO_3$ and washed with 10 mL ether. The resulting aqua phase was acidified by 2 mL 3N HCl and extracted with 15 mL ether. The extract was washed with water and dried over Na_2SO_4. The solvent was evaporated in vacuo to afford the desired products.



2,3,4,5-Tetrachloro-6-hydroxy-6-(2-oxo-2-phenylethyl)cyclohexa-2,4-dienone (3a). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.4). ^{1}H NMR (ppm) δ 7.86(dd, J = 8.4, 1.2Hz, 2H), 7.62-7.59(m, 1H), 7.48-7.45(m, 2H), 4.17(d, J = 16.8Hz, 1H), 3.96(d, J = 16.8Hz, 1H), 3.60(bs, 1H); ^{13}C NMR (ppm) δ 196.1, 189.6, 145.2, 138.3, 135.0, 134.3, 129.8, 128.9, 128.3, 127.6, 76.3, 51.3; MS(EI) *m/z*(%): 366(M^+), 330, 293, 248, 219,147, 120, 105(100), 91, 77, 51, 36; HRMS(ESI) calcd for $C_{14}H_8Cl_4O_3$(M^++Na): 386.9120; found: 386.9128.

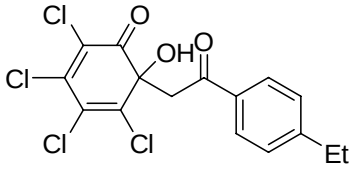


2,3,4,5-Tetrachloro-6-hydroxy-6-(2-(4-methoxyphenyl)-2-oxoethyl)cyclohexa-2,4-dienone (3b). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 7.79(d, J = 8.8Hz, 2H), 6.87(d, J = 8.8Hz, 2H), 4.06(d, J = 16.8Hz, 1H), 3.85(d, J = 16.8Hz, 1H), 3.82(s, 3H), 3.59(bs, 1H); ^{13}C NMR (ppm) δ 194.4, 189.6, 164.4, 144.9, 138.5, 130.7, 129.8, 128.2, 127.4, 114.0, 76.5, 55.6, 51.0; MS(EI) *m/z*(%): 396(M^+), 256, 248, 223, 177, 169, 149, 135(100), 113, 107, 85, 71, 57, 43, 30; HRMS(ESI) calcd for $C_{15}H_{10}Cl_4O_4$ (M^++Na): 416.9225; found: 416.9229.

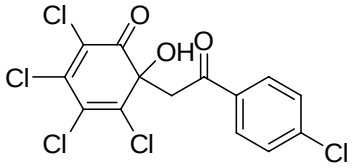


2,3,4,5-Tetrachloro-6-hydroxy-6-(2-oxo-2-p-tolylethyl)cyclohexa-2,4-dienone (3c). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.4). ^{1}H NMR (ppm) δ 7.78(d, J = 8.0Hz, 2H), 7.26(d, J = 8.0Hz, 2H), 4.32(bs, 1H), 4.18(d, J

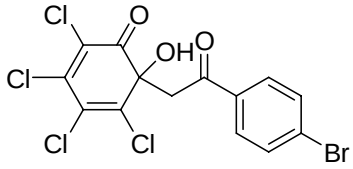
= 17.2Hz, 1H), 3.98(d, *J* = 17.2Hz, 1H), 2.41(s,3H); ^{13}C NMR (ppm) δ 195.9, 189.5, 145.3, 144.8, 138.7, 132.6, 129.7, 129.5, 128.4, 127.2, 76.2, 51.1, 21.7; MS(EI) *m/z*(%): 380(M^+), 342, 329, 327, 299, 281, 248, 222, 185, 183, 161, 149, 134, 119(100), 105, 91, 73, 65, 55, 40; HRMS(ESI) calcd for $C_{15}H_{10}Cl_4O_3$ (M^++Na): 400.9276; found: 402.9257.



2,3,4,5-Tetrachloro-6-(2-(4-ethylphenyl)-2-oxoethyl)-6-hydroxycyclohexa-2,4-dienone (3d). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.4). ^{1}H NMR (ppm) δ 7.79(d, *J* = 8.4Hz, 2H), 7.28(d, *J* = 8.4Hz, 2H), 4.15(d, *J* = 17.4Hz, 1H), 3.94(d, *J* = 17.4Hz, 1H), 3.71(bs, 1H), 2.70(q, *J* = 7.8Hz, 2H), 1.24(t, *J* = 7.8Hz, 3H); ^{13}C NMR (ppm) δ 195.7, 189.6, 151.5, 145.0, 138.4, 132.7, 129.7, 128.5, 128.3, 127.5, 76.3, 51.2, 29.0, 15.0; MS(EI) *m/z*(%):394(M^+), 358, 327, 299, 248, 224, 222, 175, 148, 133(100), 119, 105, 91, 77, 51, 37; HRMS(ESI) calcd for $C_{16}H_{12}Cl_4O_3$ (M^++Na): 414.9433; found: 416.9406.

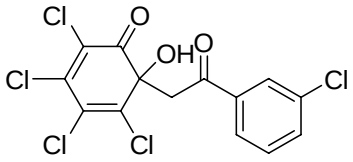


2,3,4,5-Tetrachloro-6-(2-(4-chlorophenyl)-2-oxoethyl)-6-hydroxycyclohexa-2,4-dienone (3e). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.4). ^{1}H NMR (ppm) δ 7.76(d, *J* = 8.8Hz, 2H), 7.39(d, *J* = 8.8Hz, 2H), 4.07(d, *J* = 17.2Hz, 1H), 3.88(d, *J* = 17.2Hz, 1H), 3.68(bs,1H); ^{13}C NMR (ppm) δ 195.0, 189.4, 145.1, 140.9, 138.3, 133.3, 129.7, 129.2, 127.5, 76.2, 51.1; MS(EI) *m/z*(%): 400(M^+), 384, 248, 212, 181, 154, 139(100), 113, 111, 87, 75, 50, 37; HRMS(ESI) calcd for $C_{14}H_7Cl_5O_3$ (M^++Na): 420.8730; found: 420.8726.

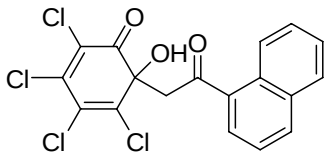


6-(2-(4-Bromophenyl)-2-oxoethyl)-2,3,4,5-tetrachloro-6-hydroxycyclohexa-2,4-dienone (3f). Isolated by flash column chromatography (ethyl acetate/petroleum ether =

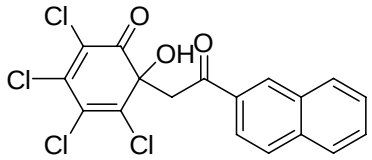
1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 7.73(d, J = 8.8Hz, 2H), 7.62(d, J = 8.8Hz, 2H), 4.10(d, J = 16.8Hz, 1H), 3.91(d, J = 16.8Hz, 1H), 3.50(bs,1H); ^{13}C NMR (ppm) δ 195.1, 189.5, 145.2, 138.0, 133.7, 132.2, 129.8, 129.7, 129.6, 127.7, 76.2, 51.1; MS(EI) *m/z*(%): 442(M^+), 410, 408, 380, 373, 345, 327, 303, 299, 250, 248, 222, 198, 185(100), 169, 155, 131, 119, 104, 76, 63, 50; HRMS(ESI) calcd for $C_{14}H_7BrCl_4O_3$ (M^++Na): 464.8225; found: 464.8236.



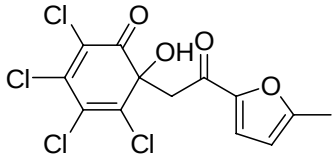
2,3,4,5-Tetrachloro-6-(2-(3-chlorophenyl)-2-oxoethyl)-6-hydroxycyclohexa-2,4-dienone(3g). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 7.79(s,1H), 7.70(d, J = 7.6Hz, 1H), 7.51(d, J = 7.6Hz, 1H), 7.36(t, J = 7.6Hz, 1H), 4.08(d, J = 17.2Hz, 1H), 3.92(d, J = 17.2Hz, 1H); ^{13}C NMR (ppm) δ 195.0, 189.4, 145.2, 138.2, 136.4, 135.2, 134.1, 130.2, 129.7, 128.3, 127.6, 126.4, 76.1, 51.1; MS(EI) *m/z*(%): 400(M^+), 384, 250, 248, 223, 181, 154, 139(100), 125, 111, 97, 79, 75, 52; HRMS(ESI) calcd for $C_{14}H_7Cl_5O_3$ (M^++Na): 420.8730; found: 420.8732.



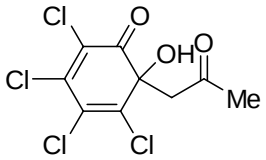
2,3,4,5-Tetrachloro-6-hydroxy-6-(2-(naphthalen-1-yl)-2-oxoethyl)cyclohexa-2,4-dienone (3h). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 8.49(d, J = 8.0Hz, 1H), 7.95(d, J = 8.0Hz, 1H), 7.88(d, J = 7.2Hz, 1H), 7.79(d, J = 8.0Hz, 1H), 7.54-7.40(m, 3H), 4.18(d, J = 17.2Hz, 1H), 4.09(d, J = 17.2Hz, 1H); ^{13}C NMR (ppm) δ 199.5, 189.6, 145.0, 138.6, 134.3, 133.8, 132.6, 129.8, 129.7, 129.4, 128.6, 128.4, 127.4, 126.7, 125.4, 124.2, 77.2, 53.9; MS(EI) *m/z*(%): 416(M^+), 380, 377, 351, 349, 315, 250, 248, 223, 212, 195, 182, 170, 155(100), 147, 127, 111, 101, 81, 77, 59; HRMS(ESI) calcd for $C_{18}H_{10}Cl_4O_3$ (M^++Na):436.9276; found: 438.9244.



2,3,4,5-Tetrachloro-6-hydroxy-6-(2-(naphthalen-2-yl)-2-oxoethyl)cyclohexa-2,4-dienone (3i). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 8.43(s,1H), 7.96(d, J = 8.0Hz, 1H), 7.87(s,1H), 7.86(d, J = 8.0Hz, 2H), 7.64(t, J = 8.0Hz, 1H), 7.57(t, J = 8.0Hz, 1H), 4.35(d, J = 17.2Hz, 1H), 4.14(d, J = 17.2Hz, 1H), 3.68(bs,1H); ^{13}C NMR (ppm) δ 196.1, 189.7, 145.1, 138.4, 135.9, 132.2, 132.1, 130.6, 129.8, 129.7, 129.1, 128.7, 127.8, 127.5, 127.1, 123.2, 76.3, 51.4; MS(EI) *m/z*(%): 416(M^+), 250, 248, 210, 197, 181, 165, 155, 149, 123(100), 111, 95, 87, 75, 57; HRMS(ESI) calcd for $C_{18}H_{10}Cl_4O_3$ (M^+): 414.9457; found: 416.9430.

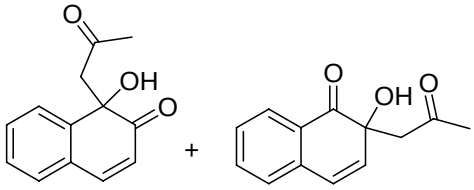


2,3,4,5-Tetrachloro-6-hydroxy-6-(2-(5-methylfuran-2-yl)-2-oxoethyl)cyclohexa-2,4-dienone (3j). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.4). ^{1}H NMR (ppm) δ 7.11(d, J = 3.2Hz, 1H), 6.12(d, J = 3.2Hz, 1H), 4.35(bs, 1H), 3.83(d, J = 16.8Hz, 1H), 3.63(d, J =16.8Hz, 1H), 2.32(s, 3H); ^{13}C NMR (ppm) δ 189.6, 183.0, 159.4, 149.9, 144.8, 138.6, 129.7, 127.0, 121.3, 109.7, 76.6, 50.0, 14.0; MS(EI) *m/z*(%): 360, 337, 326, 309, 293, 284, 260, 248, 239, 214, 212, 197, 183, 169, 149, 141, 127, 109, 97, 71, 57(100), 43, 41, 27; HRMS(ESI) calcd for $C_{13}H_8Cl_4O_4$ (M^++H): 368.9250; found: 370.3785.

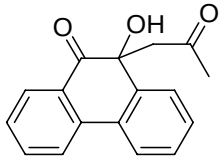


2,3,4,5-Tetrachloro-6-hydroxy-6-(2-oxopropyl)cyclohexa-2,4-dienone (3k).[1] Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.3). ^{1}H NMR (ppm) δ 3.79(bs,1H), 3.51(d, J = 17.4Hz, 1H), 3.44(d, J =17.4Hz, 1H), 2.15(s, 3H); ^{13}C NMR (ppm) δ 205.3, 189.6, 145.2, 138.1, 129.5, 127.4, 75.9, 54.8, 29.9; MS(EI) *m/z*(%):

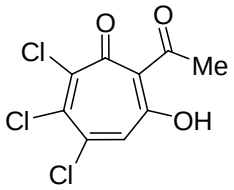
304(M^+), 143, 105, 85, 58, 43(100), 36; HRMS(ESI) calcd for $C_9H_6Cl_4O_3$ (M^++H): 301.9071; found: 301.9031.



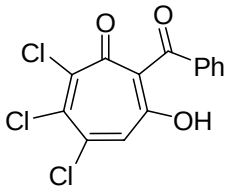
1-Hydroxy-1-(2-oxopropyl)naphthalen-2(1H)-one and 2-hydroxy-2-(2-oxopropyl) naphthalene-1(2H)-one (3l+3l'). A ratio of two isomers is 4:3. Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, R_f = 0.5). **overlap:** ^{1}H NMR (ppm) δ 7.96(d, J =7.6Hz, 1H)， 7.65(d, J =7.6Hz, 1H), 7.59(t, J =7.6Hz, 1H),7.45-7.28(m, 5H), 7.21(d, J =7.6Hz, 1H), 6.55(d, J =10.0Hz, 1H), 6.30(d, J =10.0Hz, 1H), 6.23(d, J =10.0Hz, 1H); **major isomer:** ^{1}H NMR (ppm) δ 4.31(s,1H), 2.93(dd, J = 14.0Hz, 8.4 Hz ,2H), 2.10(s, 3H), **minor isomer:** ^{1}H NMR (ppm) δ 3.98(s,1H), 2.87(dd, J =15.2Hz, 6.4Hz ,2H), 2.18(s, 3H); ^{13}C NMR (ppm) δ 205.8, 205.5, 202.7, 201.2, 145.1, 142.5, 137.1, 135.0, 134.3, 130.3, 129.5, 128.7, 128.4, 128.3, 128.2, 127.5, 127.1, 126.0, 125.9, 122.8, 77.5, 74.7, 55.8, 52.6, 31.9, 31.7; MS(EI) *m/z*(%): 216(M^+), 198, 188, 173, 160, 158, 145, 131(100), 117, 115, 102, 91, 85, 77, 63, 58, 51, 43, 27, 25; HRMS(ESI) calcd for $C_{13}H_{12}O_3$(M^++Na): 239.0679; found: 239.0676.



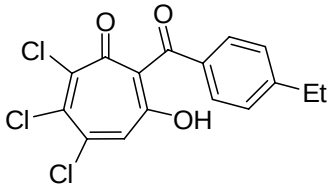
10-Hydroxy-10-(2-oxo-propyl)-10H-phenanthren-9-one (3m).[2] Isolated by flash column chromatography (ethyl acetate/hexane = 1:5, R_f = 0.2). ^{1}H NMR (ppm) δ 7.91(d, J = 7.8Hz, 1H), 7.87(d, J = 7.8Hz, 1H), 7.79-7.78(m, 1H), 7.74-7.72(m, 1H), 7.66(t, J =7.8Hz, 1H), 7.42-7.37(m, 3H), 4.51(bs, 1H), 2.97(d, J = 15.0Hz, 1H), 2.79(d, J = 15.0Hz, 1H), 2.06(s, 3H); ^{13}C NMR (ppm) δ 205.5, 202.2, 139.2, 136.6, 134.7, 129.2, 129.1, 128.8, 128.6, 128.5, 127.5, 125.8, 124.3, 123.0, 78.2, 55.4, 31.9.



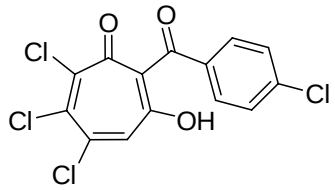
(2*Z*,4*E*,6*E*)-7-Acetyl-2,3,4-trichloro-6-hydroxycyclohepta-2,4,6-trienone (4a).[1] ^{1}H NMR (ppm) δ 7.03(s, 1H), 2.41(s, 3H); ^{13}C NMR (ppm) δ 204.6, 181.5, 171.3, 140.6, 140.5, 133.9, 128.2, 116.6, 28.7; MS(EI) *m/z*(%): 266(M^+), 251, 238, 225, 223, 210, 196, 189, 171, 167, 159, 147, 133, 119, 107, 97, 87, 84, 69, 61, 43(100), 37, 27; HRMS(ESI) calcd for $C_9D_6Cl_4O_3$ (M^+): 266.9377; found: 266.9378.



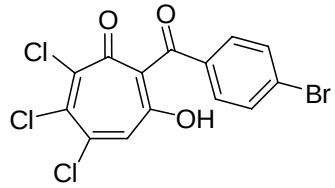
(2*Z*,4*E*,6*E*)-7-Benzoyl-2,3,4-trichloro-6-hydroxycyclohepta-2,4,6-trienone (4b). Crystals for X-ray analyses of **4b** were obtained on slow solvent evaporation of ether and petroleum ether. ^{1}H NMR (ppm) δ 7.56-7.53(m, 3H), 7.46-7.42(m, 2H), 7.22(s, 1H); ^{13}C NMR (ppm) δ 200.2, 181.4, 169.6, 141.2, 140.8, 136.5, 134.8, 132.8, 128.5, 128.3, 128.1, 115.3; MS(EI) *m/z*(%): 328(M^+), 301, 299, 225, 223, 173, 119, 105, 97, 77(100), 63, 51, 40, 37; HRMS(ESI) calcd for $C_{14}H_7Cl_3O_3$ (M^+): 328.9534; found: 328.9542.



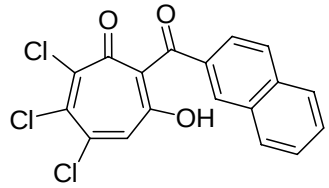
(2*Z*,4*E*,6*E*)-2,3,4-Trichloro-7-(4-ethylbenzoyl)-6-hydroxycyclohepta-2,4,6-trienone (4c). ^{1}H NMR (ppm) δ 7.48(dd, J = 8.4, 1.8Hz, 2H), 7.26-7.25(m, 2H), 7.21(s, 1H), 2.70(q, J = 7.8Hz, 2H), 1.26(t, J = 7.8Hz, 3H); ^{13}C NMR (ppm) δ 199.7, 181.6, 168.4, 150.0, 140.9, 140.7, 134.7, 133.8, 128.5, 128.4, 128.0, 115.2, 28.9, 14.9; MS(EI) *m/z*(%): 356(M^+), 355, 329, 327, 299, 285, 265, 249, 236, 222, 207, 186, 173, 167, 149, 133(100), 119, 105, 91, 77, 63, 51, 37, 27; HRMS(ESI) calcd for $C_{16}H_{11}Cl_3O_3$ (M^++H): 356.9847; found: 356.9843.



(2*Z*,4*E*,6*E*)-2,3,4-Trichloro-7-(4-chlorobenzoyl)-6-hydroxycyclohepta-2,4,6-trienone (4d). ^{1}H NMR (ppm) δ 7.49(d, J = 8.4Hz, 2H), 7.41(d, J = 8.4Hz, 2H), 7.23(s, 1H); ^{13}C NMR (ppm) δ 198.9, 181.3, 168.8, 141.5, 140.9, 139.2, 135.1, 134.9, 129.5, 128.9, 128.2, 115.2; MS(EI) *m/z*(%): 382(M^+), 364, 336, 299, 248, 222, 167, 149, 139, 119, 111, 91, 71, 57, 43(100), 41, 28; HRMS(ESI) calcd for $C_{14}H_6Cl_4O_3$ (M^++Na): 384.8963; found: 384.8970.



(2*Z*,4*E*,6*E*)-7-(4-Bromobenzoyl)-2,3,4-trichloro-6-hydroxycyclohepta-2,4,6-trienone (4e). ^{1}H NMR (ppm) δ 7.59-7.57(m, 2H), 7.42-7.40(m, 2H), 7.23(s, 1H); ^{13}C NMR (ppm) δ 199.1, 181.2, 168.9, 141.6, 141.0, 135.3, 135.1, 131.8, 129.5, 128.2, 127.8, 115.2; MS(EI) *m/z*(%): 408(M^+), 406, 380, 377, 363, 345, 327, 309, 299, 282, 264, 250, 236, 224, 207, 196, 183(100), 167, 155, 139, 131, 111, 97, 84, 76, 50; HRMS(ESI) calcd for $C_{14}H_6BrCl_3O_3$ (M^++H): 406.9639; found: 406.8638.



(2*Z*,4*E*,6*E*)-7-(2-naphthoyl)-2,3,4-trichloro-6-hydroxycyclohepta-2,4,6-trienone (4f). ^{1}H NMR (ppm) δ 8.05(s, 1H), 7.87-7.83(m, 3H), 7.60-7.51(m, 3H), 7.21(s, 1H); ^{13}C NMR (ppm) δ 199.9, 181.6, 168.4, 141.1, 140.7, 135.3, 134.8, 133.5, 132.2, 129.8, 129.2, 128.6, 128.5, 128.3, 127.8, 127.0, 124.0, 115.5; MS(EI) *m/z*(%): 378(M^+), 351, 279, 250, 248, 223, 205, 189, 184, 170, 155, 149, 127(100), 115, 101, 93, 77, 63, 57; HRMS(ESI) calcd for $C_{18}H_9Cl_3O_3$ (M^++Na): 400.9510; found: 400.9509.

References:

(1) H. Kogler, H.-W. Fehlhaber, K. Leube, W. Durckheimer, *Chem. Ber.* 1989, **122**, 2205-2206.

(2) R. V. Linko, V. K. Belsky, A. V. Varlamov, B. E. Zaitsev, A. I. Chernyshev, *Russ. Chem. Bull. Int. Ed.* 2001, **50**, 1625-1629.

2) X-ray crystallography data for 4b

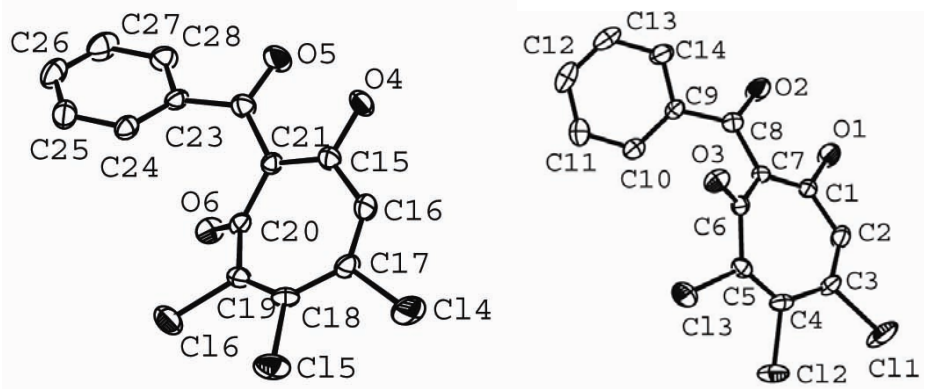


Fig. 1 ORTEP drawing of **4b** with 30% thermal ellipsoids.
Hydrogen atoms omitted for clarity.

(**Note:** Intramolecular hydrogen bonding is responsible for the two configurations of tropone **4b** as shown in Fig. 1. The two configurations break the symmetry and change the crystal system to triclinic.)

Table 1. Crystal data and structure refinement for **4b**.

Identification code	lzpa	
Empirical formula	C14 H7 Cl3 O3	
Formula weight	329.55	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 4.1248(8) Å	α= 62.21(3) °
	b = 18.813(4) Å	β= 89.89(3) °
	c = 19.845(4) Å	γ = 89.83(3) °

Volume	1362.3(5) $Å^3$
Z	4
Density (calculated)	1.607 Mg/m^3
Absorption coefficient	0.674 mm^{-1}
F(000)	664
Crystal size	0.50 x 0.30 x 0.20 mm^3
Theta range for data collection	1.16 to 27.48°.
Index ranges	-5<=h<=5, -24<=k<=24, -25<=l<=22
Reflections collected	12452
Independent reflections	6165 [R(int) = 0.0332]
Completeness to theta = 27.48°	98.1 %
Absorption correction	Empirical
Max. and min. transmission	0.8769 and 0.7291
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6165 / 1 / 369
Goodness-of-fit on F^2	1.058
Final R indices [I>2sigma(I)]	R1 = 0.0417, wR2 = 0.0724
R indices (all data)	R1 = 0.1080, wR2 = 0.0780
Largest diff. peak and hole	0.241 and -0.354 e. $Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **4b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3334(6)	674(2)	570(2)	41(1)
C(2)	2784(6)	-175(2)	955(2)	49(1)
C(3)	2903(6)	-701(1)	1693(2)	44(1)
C(4)	3688(6)	-564(1)	2328(1)	41(1)
C(5)	5182(6)	102(2)	2278(1)	38(1)
C(6)	6445(6)	816(1)	1585(1)	34(1)
C(7)	4801(6)	1143(1)	851(1)	35(1)
C(8)	4949(6)	2026(2)	349(2)	45(1)
C(9)	5512(6)	2623(1)	621(2)	38(1)

C(10)	4384(6)	2524(2)	1315(2)	44(1)
C(11)	4736(6)	3154(2)	1494(2)	56(1)
C(12)	6202(7)	3863(2)	987(2)	67(1)
C(13)	7324(7)	3954(2)	307(2)	65(1)
C(14)	6967(6)	3338(2)	115(2)	52(1)
C(15)	1652(6)	5675(2)	5570(2)	43(1)
C(16)	2238(6)	4823(2)	5956(2)	49(1)
C(17)	2097(6)	4302(1)	6697(2)	44(1)
C(18)	1306(6)	4441(1)	7330(1)	39(1)
C(19)	-165(6)	5105(2)	7272(1)	37(1)
C(20)	-1454(6)	5817(1)	6583(1)	35(1)
C(21)	217(6)	6143(1)	5848(1)	35(1)
C(22)	43(6)	7023(2)	5350(2)	45(1)
C(23)	-502(6)	7619(1)	5623(2)	39(1)
C(24)	618(6)	7529(2)	6314(2)	42(1)
C(25)	282(7)	8145(2)	6499(2)	55(1)
C(26)	-1203(7)	8860(2)	5989(2)	64(1)
C(27)	-2309(7)	8954(2)	5307(2)	67(1)
C(28)	-1977(6)	8342(2)	5113(2)	52(1)
Cl(1)	1931(2)	-1681(1)	1908(1)	91(1)
Cl(2)	2643(2)	-1314(1)	3220(1)	70(1)
Cl(3)	6007(2)	178(1)	3088(1)	67(1)
Cl(4)	3074(2)	3321(1)	6907(1)	91(1)
Cl(5)	2355(2)	3686(1)	8220(1)	70(1)
Cl(6)	-1009(2)	5178(1)	8087(1)	68(1)
O(1)	2309(5)	990(1)	-148(1)	69(1)
O(2)	4390(6)	2284(1)	-342(1)	82(1)
O(3)	8827(4)	1158(1)	1662(1)	49(1)
O(4)	2701(5)	5990(1)	4851(1)	68(1)
O(5)	622(6)	7285(1)	4659(1)	82(1)
O(6)	-3831(4)	6158(1)	6664(1)	49(1)
H(2)	2256	-396	637	59
H(10)	3406	2045	1657	53
H(11)	3977	3096	1958	67
H(12)	6423	4280	1112	80
H(13)	8333	4430	-30	78

H(14)	7704	3405	-354	62
H(16)	2803	4601	5641	59
H(24)	1600	7050	6656	51
H(25)	1046	8084	6963	66
H(26)	-1439	9276	6115	77
H(27)	-3296	9434	4970	80
H(28)	-2731	8410	4645	62
H(1)	3020(90)	1580(30)	-340(20)	138(15)
H(4)	1940(80)	6539(13)	4640(20)	129(14)

Table 3. Bond lengths [Å] and angles [°] for **4b**.

C(1)-O(1)	1.331(3)	C(12)-H(12)	0.9300
C(1)-C(7)	1.385(3)	C(13)-C(14)	1.387(4)
C(1)-C(2)	1.433(3)	C(13)-H(13)	0.9300
C(2)-C(3)	1.332(4)	C(14)-H(14)	0.9300
C(2)-H(2)	0.9300	C(15)-O(4)	1.336(3)
C(3)-C(4)	1.438(4)	C(15)-C(21)	1.372(3)
C(3)-Cl(1)	1.736(3)	C(15)-C(16)	1.438(3)
C(4)-C(5)	1.358(3)	C(16)-C(17)	1.335(4)
C(4)-Cl(2)	1.726(3)	C(16)-H(16)	0.9300
C(5)-C(6)	1.499(3)	C(17)-C(18)	1.435(4)
C(5)-Cl(3)	1.713(3)	C(17)-Cl(4)	1.738(2)
C(6)-O(3)	1.224(3)	C(18)-C(19)	1.343(3)
C(6)-C(7)	1.458(3)	C(18)-Cl(5)	1.728(2)
C(7)-C(8)	1.488(3)	C(19)-C(20)	1.496(3)
C(8)-O(2)	1.244(3)	C(19)-Cl(6)	1.720(3)
C(8)-C(9)	1.476(3)	C(20)-O(6)	1.222(3)
C(9)-C(10)	1.380(3)	C(20)-C(21)	1.463(3)
C(9)-C(14)	1.385(3)	C(21)-C(22)	1.482(3)
C(10)-C(11)	1.395(3)	C(22)-O(5)	1.244(3)
C(10)-H(10)	0.9300	C(22)-C(23)	1.472(3)
C(11)-C(12)	1.381(4)	C(23)-C(24)	1.382(3)
C(11)-H(11)	0.9300	C(23)-C(28)	1.399(3)
C(12)-C(13)	1.360(4)	C(24)-C(25)	1.375(3)

C(24)-H(24)	0.9300	C(10)-C(9)-C(14)	120.3(2)
C(25)-C(26)	1.392(4)	C(10)-C(9)-C(8)	122.6(2)
C(25)-H(25)	0.9300	C(14)-C(9)-C(8)	116.8(2)
C(26)-C(27)	1.359(4)	C(9)-C(10)-C(11)	118.8(3)
C(26)-H(26)	0.9300	C(9)-C(10)-H(10)	120.6
C(27)-C(28)	1.381(4)	C(11)-C(10)-H(10)	120.6
C(27)-H(27)	0.9300	C(12)-C(11)-C(10)	120.6(3)
C(28)-H(28)	0.9300	C(12)-C(11)-H(11)	119.7
O(1)-H(1)	1.03(5)	C(10)-C(11)-H(11)	119.7
O(2)-H(1)	1.44(5)	C(13)-C(12)-C(11)	120.2(3)
O(4)-H(4)	0.968(19)	C(13)-C(12)-H(12)	119.9
		C(11)-C(12)-H(12)	119.9
O(1)-C(1)-C(7)	121.4(2)	C(12)-C(13)-C(14)	120.1(3)
O(1)-C(1)-C(2)	110.4(2)	C(12)-C(13)-H(13)	119.9
C(7)-C(1)-C(2)	128.1(2)	C(14)-C(13)-H(13)	119.9
C(3)-C(2)-C(1)	130.7(3)	C(9)-C(14)-C(13)	120.0(3)
C(3)-C(2)-H(2)	114.7	C(9)-C(14)-H(14)	120.0
C(1)-C(2)-H(2)	114.7	C(13)-C(14)-H(14)	120.0
C(2)-C(3)-C(4)	128.9(2)	O(4)-C(15)-C(21)	121.4(2)
C(2)-C(3)-Cl(1)	114.8(2)	O(4)-C(15)-C(16)	110.2(2)
C(4)-C(3)-Cl(1)	116.2(2)	C(21)-C(15)-C(16)	128.5(2)
C(5)-C(4)-C(3)	125.1(2)	C(17)-C(16)-C(15)	130.1(3)
C(5)-C(4)-Cl(2)	118.3(2)	C(17)-C(16)-H(16)	114.9
C(3)-C(4)-Cl(2)	116.54(19)	C(15)-C(16)-H(16)	114.9
C(4)-C(5)-C(6)	128.9(2)	C(16)-C(17)-C(18)	129.3(2)
C(4)-C(5)-Cl(3)	120.0(2)	C(16)-C(17)-Cl(4)	114.0(2)
C(6)-C(5)-Cl(3)	111.07(19)	C(18)-C(17)-Cl(4)	116.6(2)
O(3)-C(6)-C(7)	119.8(2)	C(19)-C(18)-C(17)	124.7(2)
O(3)-C(6)-C(5)	117.7(2)	C(19)-C(18)-Cl(5)	119.2(2)
C(7)-C(6)-C(5)	122.3(2)	C(17)-C(18)-Cl(5)	116.14(19)
C(1)-C(7)-C(6)	123.7(2)	C(18)-C(19)-C(20)	129.6(2)
C(1)-C(7)-C(8)	118.1(2)	C(18)-C(19)-Cl(6)	119.2(2)
C(6)-C(7)-C(8)	118.0(2)	C(20)-C(19)-Cl(6)	111.05(19)
O(2)-C(8)-C(9)	117.4(2)	O(6)-C(20)-C(21)	120.4(2)
O(2)-C(8)-C(7)	118.1(2)	O(6)-C(20)-C(19)	117.7(2)
C(9)-C(8)-C(7)	124.4(2)	C(21)-C(20)-C(19)	121.7(2)

C(15)-C(21)-C(20)	123.6(2)	C(26)-C(25)-H(25)	120.1
C(15)-C(21)-C(22)	118.6(2)	C(27)-C(26)-C(25)	120.3(3)
C(20)-C(21)-C(22)	117.6(2)	C(27)-C(26)-H(26)	119.8
O(5)-C(22)-C(23)	117.1(2)	C(25)-C(26)-H(26)	119.8
O(5)-C(22)-C(21)	118.1(2)	C(26)-C(27)-C(28)	120.5(3)
C(23)-C(22)-C(21)	124.5(2)	C(26)-C(27)-H(27)	119.7
C(24)-C(23)-C(28)	119.5(2)	C(28)-C(27)-H(27)	119.7
C(24)-C(23)-C(22)	123.6(2)	C(27)-C(28)-C(23)	119.7(3)
C(28)-C(23)-C(22)	116.7(2)	C(27)-C(28)-H(28)	120.2
C(25)-C(24)-C(23)	120.2(3)	C(23)-C(28)-H(28)	120.2
C(25)-C(24)-H(24)	119.9	C(1)-O(1)-H(1)	100(2)
C(23)-C(24)-H(24)	119.9	C(8)-O(2)-H(1)	100.4(16)
C(24)-C(25)-C(26)	119.9(3)	C(15)-O(4)-H(4)	102(2)
C(24)-C(25)-H(25)	120.1		

Symmetry transformations used to generate equivalent atoms:

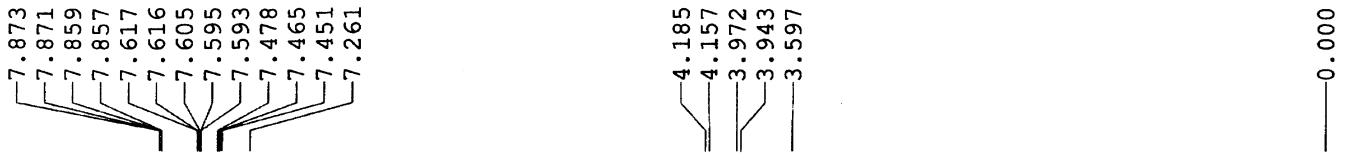
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **4b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	62(2)	33(2)	29(2)	-14(1)	2(1)	-3(1)
C(2)	67(2)	38(2)	47(2)	-25(2)	-5(1)	-2(1)
C(3)	53(2)	24(1)	50(2)	-13(1)	-6(1)	-3(1)
C(4)	39(2)	31(2)	38(2)	-3(1)	3(1)	1(1)
C(5)	36(2)	42(2)	32(1)	-15(1)	-1(1)	6(1)
C(6)	33(2)	31(1)	40(2)	-17(1)	4(1)	5(1)
C(7)	41(2)	29(1)	30(1)	-11(1)	5(1)	1(1)
C(8)	56(2)	37(2)	39(2)	-14(1)	2(1)	-1(1)
C(9)	41(2)	29(1)	41(2)	-15(1)	-3(1)	0(1)
C(10)	42(2)	37(2)	52(2)	-19(1)	1(1)	-1(1)
C(11)	53(2)	62(2)	68(2)	-43(2)	-4(2)	9(2)
C(12)	67(2)	49(2)	99(3)	-46(2)	-17(2)	2(2)
C(13)	67(2)	34(2)	84(3)	-19(2)	2(2)	-13(1)
C(14)	59(2)	37(2)	52(2)	-14(2)	3(1)	-8(1)
C(15)	61(2)	36(2)	32(2)	-14(1)	-2(1)	4(1)
C(16)	66(2)	38(2)	49(2)	-24(2)	3(1)	7(1)
C(17)	53(2)	26(2)	49(2)	-13(1)	3(1)	7(1)
C(18)	38(2)	34(2)	32(2)	-3(1)	-2(1)	2(1)
C(19)	35(2)	40(2)	32(1)	-14(1)	3(1)	-5(1)
C(20)	36(2)	30(1)	39(2)	-16(1)	-3(1)	-4(1)
C(21)	41(2)	27(1)	33(1)	-13(1)	-5(1)	4(1)
C(22)	56(2)	39(2)	36(2)	-14(1)	0(1)	6(1)
C(23)	44(2)	25(1)	44(2)	-12(1)	6(1)	0(1)
C(24)	42(2)	34(2)	51(2)	-20(1)	3(1)	2(1)
C(25)	55(2)	60(2)	65(2)	-40(2)	7(2)	-7(2)
C(26)	64(2)	47(2)	95(3)	-44(2)	18(2)	-4(2)
C(27)	67(2)	38(2)	87(3)	-23(2)	6(2)	13(1)
C(28)	58(2)	40(2)	48(2)	-14(2)	-1(1)	8(1)
Cl(1)	141(1)	30(1)	85(1)	-13(1)	-26(1)	-17(1)
Cl(2)	84(1)	52(1)	45(1)	3(1)	7(1)	-14(1)
Cl(3)	90(1)	72(1)	40(1)	-27(1)	-2(1)	-5(1)
Cl(4)	141(1)	32(1)	83(1)	-13(1)	28(1)	20(1)
Cl(5)	85(1)	52(1)	45(1)	2(1)	-5(1)	15(1)
Cl(6)	92(1)	73(1)	39(1)	-27(1)	4(1)	7(1)
O(1)	127(2)	43(1)	36(1)	-18(1)	-15(1)	-7(1)

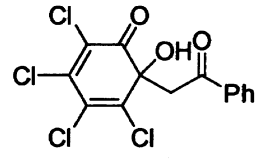
O(2)	165(2)	38(1)	32(1)	-8(1)	-14(1)	-14(1)
O(3)	35(1)	44(1)	64(1)	-21(1)	-4(1)	-3(1)
O(4)	126(2)	43(1)	33(1)	-16(1)	15(1)	11(1)
O(5)	161(2)	37(1)	35(1)	-7(1)	13(1)	17(1)
O(6)	35(1)	47(1)	63(1)	-23(1)	6(1)	5(1)

3) Copies of ^{1}H NMR and ^{13}C NMR spectra for all compounds

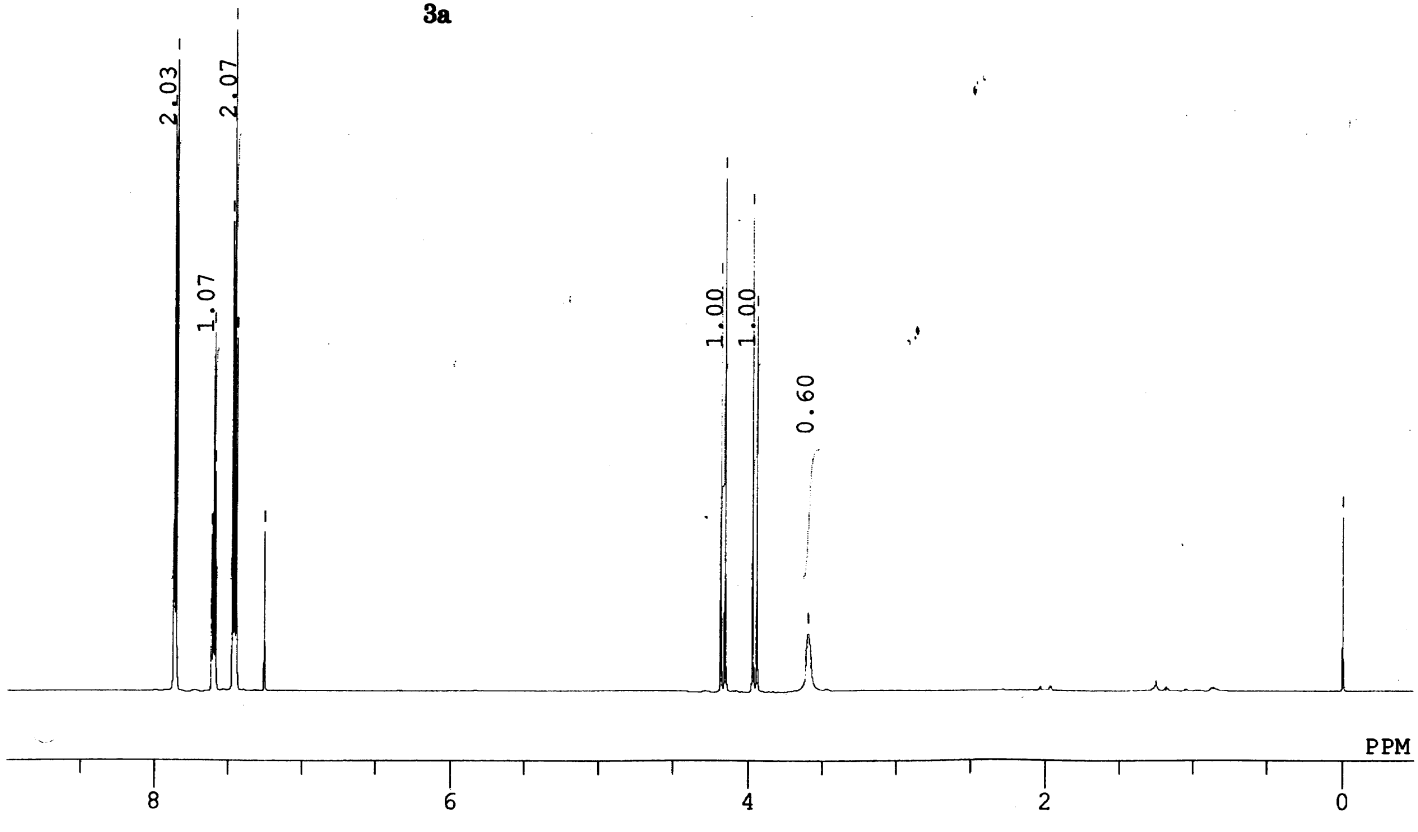
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R867-0H-1.jdf
lee-R867-0H



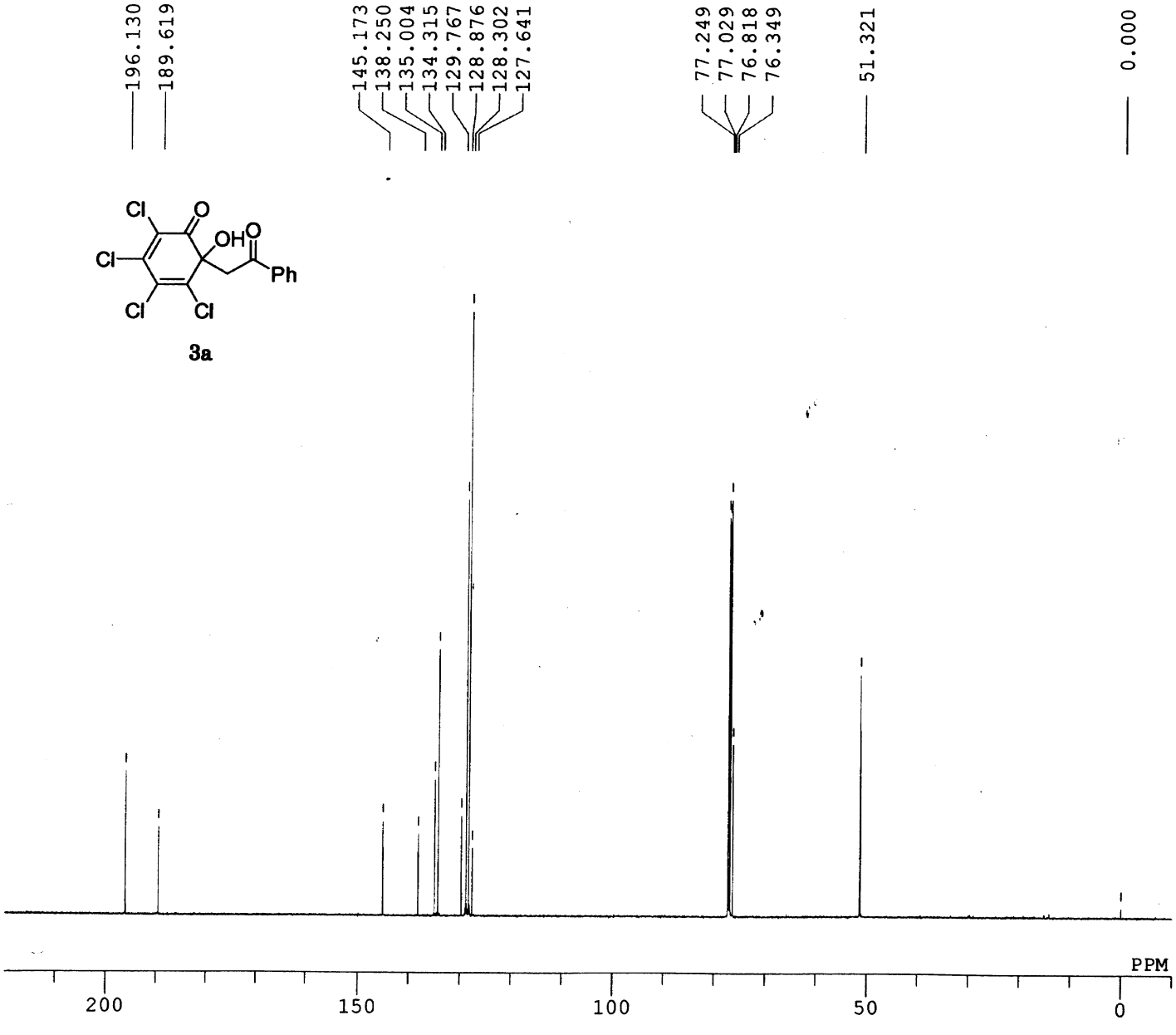
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R867-0H
DATIM	11-02-2009 21:14:54
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	32768
FREQU	11261.26 Hz
SCANS	8
ACQTM	2.9098 sec
PD	5.0000 sec
PW1	6.90 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



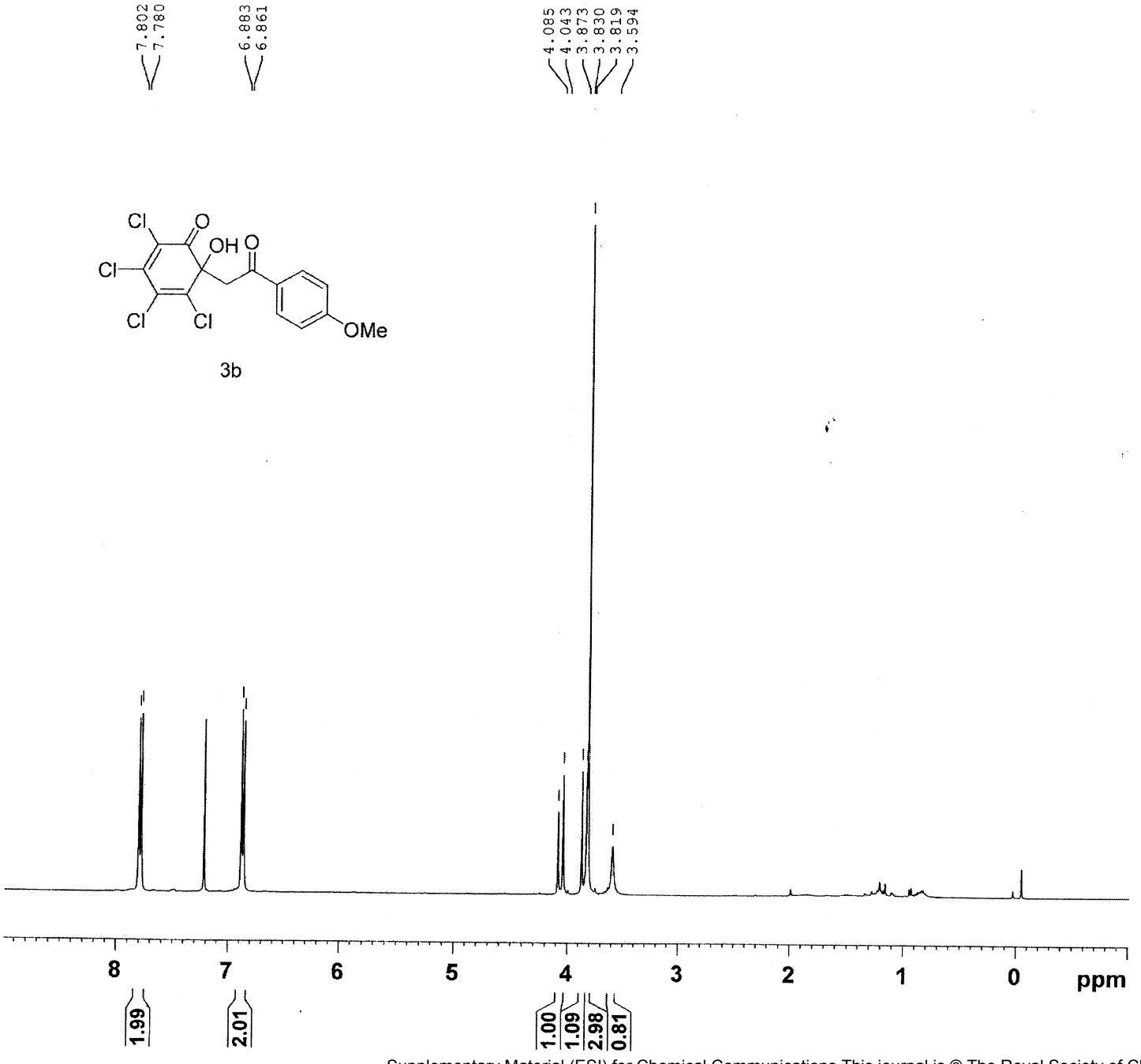
3a



C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R867-0C-1.jdf
single pulse decoupled gated NOE

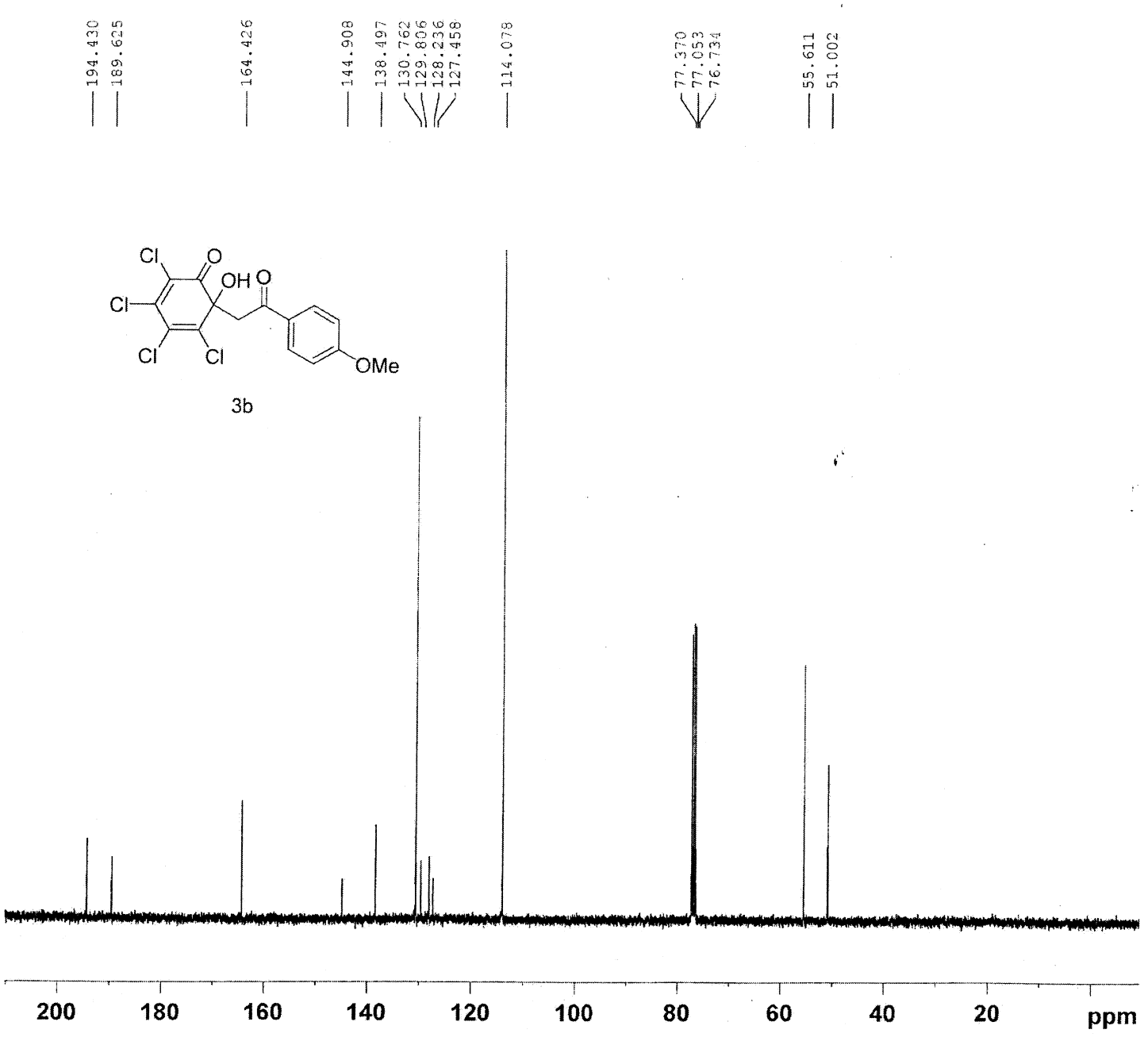


DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	single pulse decoupled gate
DATIM	11-02-2009 22:46:55
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	2000
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	50



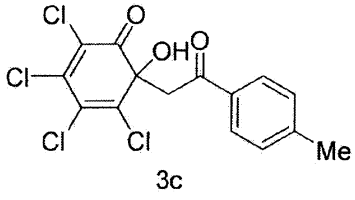
BRUKER

NAME	guoxw-lihj-788+-20081211
EXPNO	1
PROCNO	1
Date_	20081211
Time	14.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	101
DW	60.800 usec
DE	6.50 usec
TE	291.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300259 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

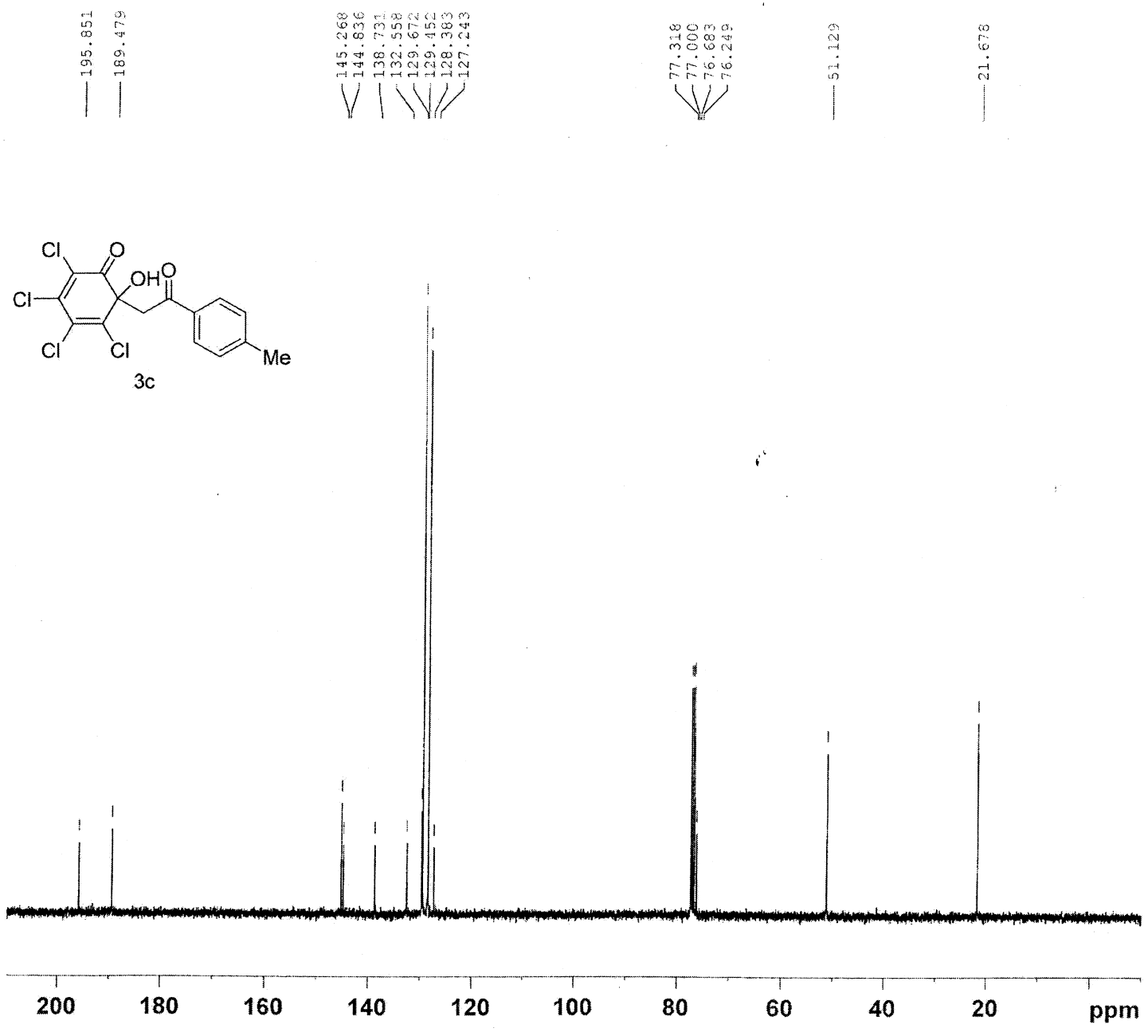
NAME	guoxw-lihj-788-20081212
EXPNO	2
PROCNO	1
Date_	20081212
Time	10.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	161
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	292.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127744 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



3c

BRUKER

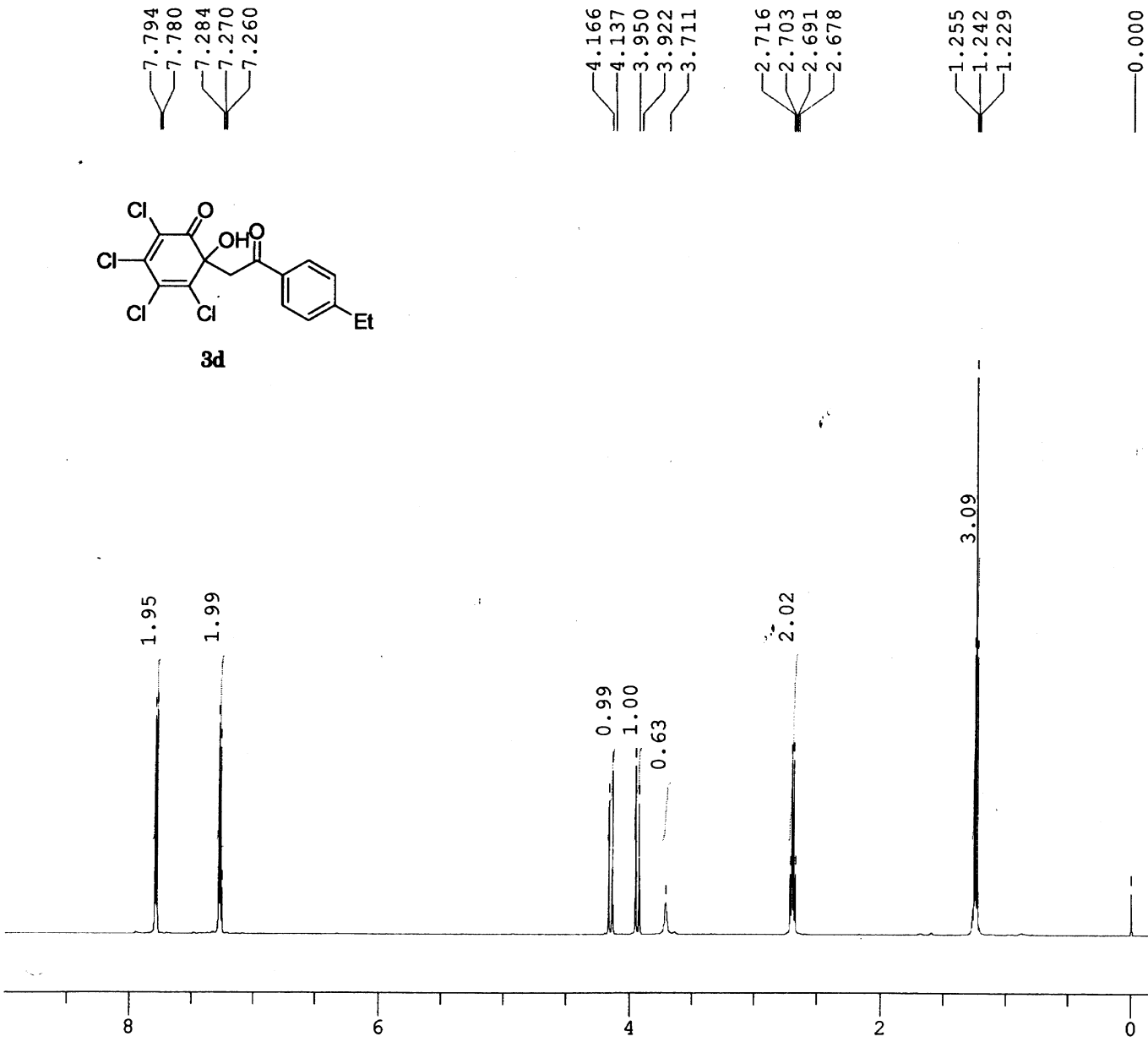
NAME	guoxw-lihj-798-20081215
EXPNO	1
PROCNO	1
Date_	20081215
Time	16.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	292.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	guoxw-lihj-798-20081215
EXPNO	2
PROCNO	1
Date_	20081215
Time	16.37
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	88
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	292.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127798 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-4c-H-1.jdf
lee-4c-H

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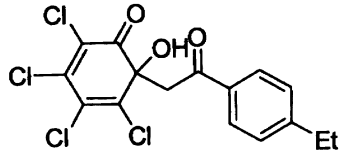
COMNT   lee-4c-H
DATIM   18-02-2009 22:41:43
OBNUC   1H
EXMOD   single_pulse.ex2
OBFRQ         600.17 MHz
OBSET           5.30 KHz
OBFIN           5.47 Hz
POINT          32768
FREQU       11261.26 Hz
SCANS              8
ACQTM         2.9098 sec
PD            5.0000 sec
PW1             6.90 usec
IRNUC   1H
CTEMP           19.5 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             38
```

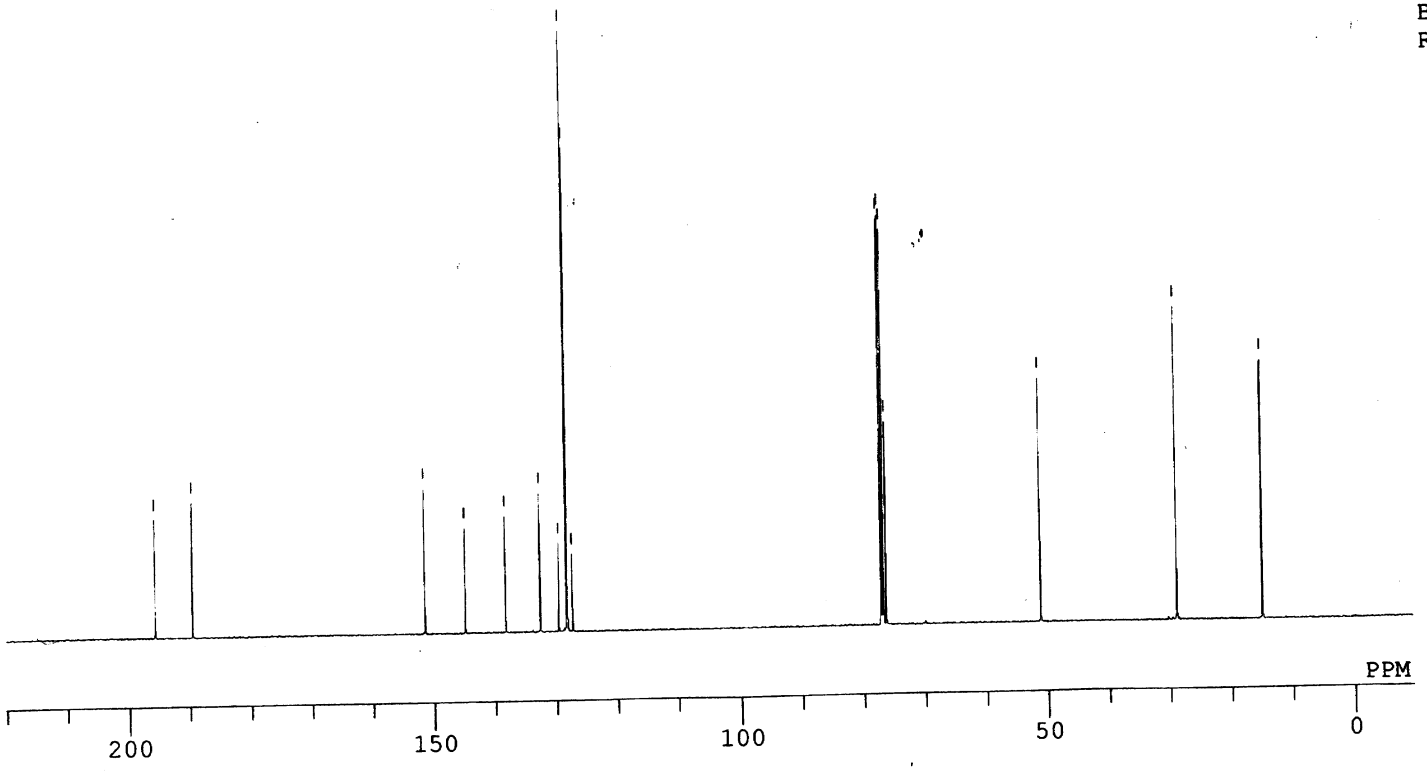
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-4c-C-1.jdf
lee-4c-C

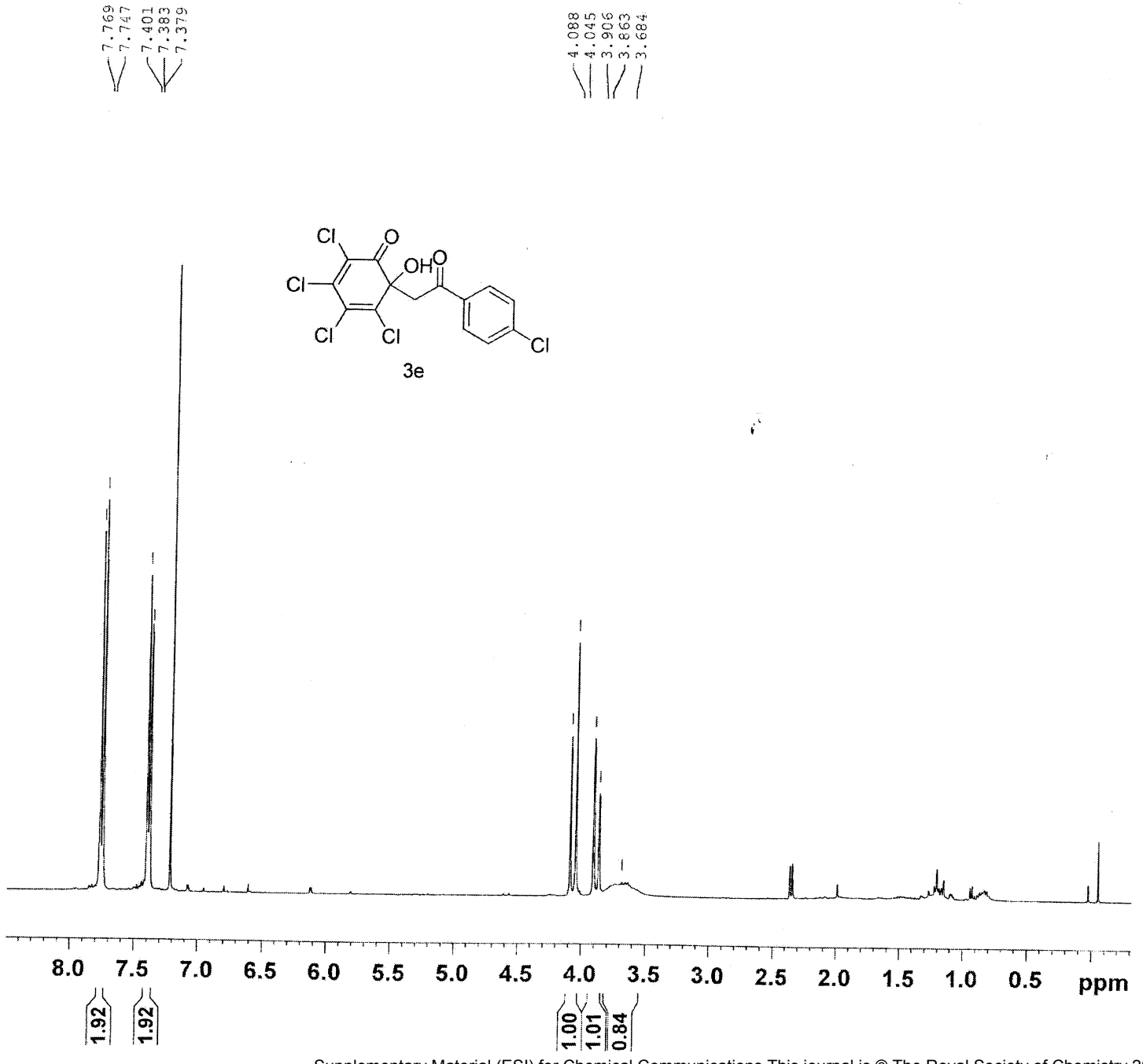
195.737
189.571
151.501
144.990
138.365
132.744
129.728
128.512
128.330
127.478
77.211
77.000
76.789
76.349
51.225
28.963
15.032



3d

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-4c-C
DATIM	18-02-2009 23:31:17
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	19.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



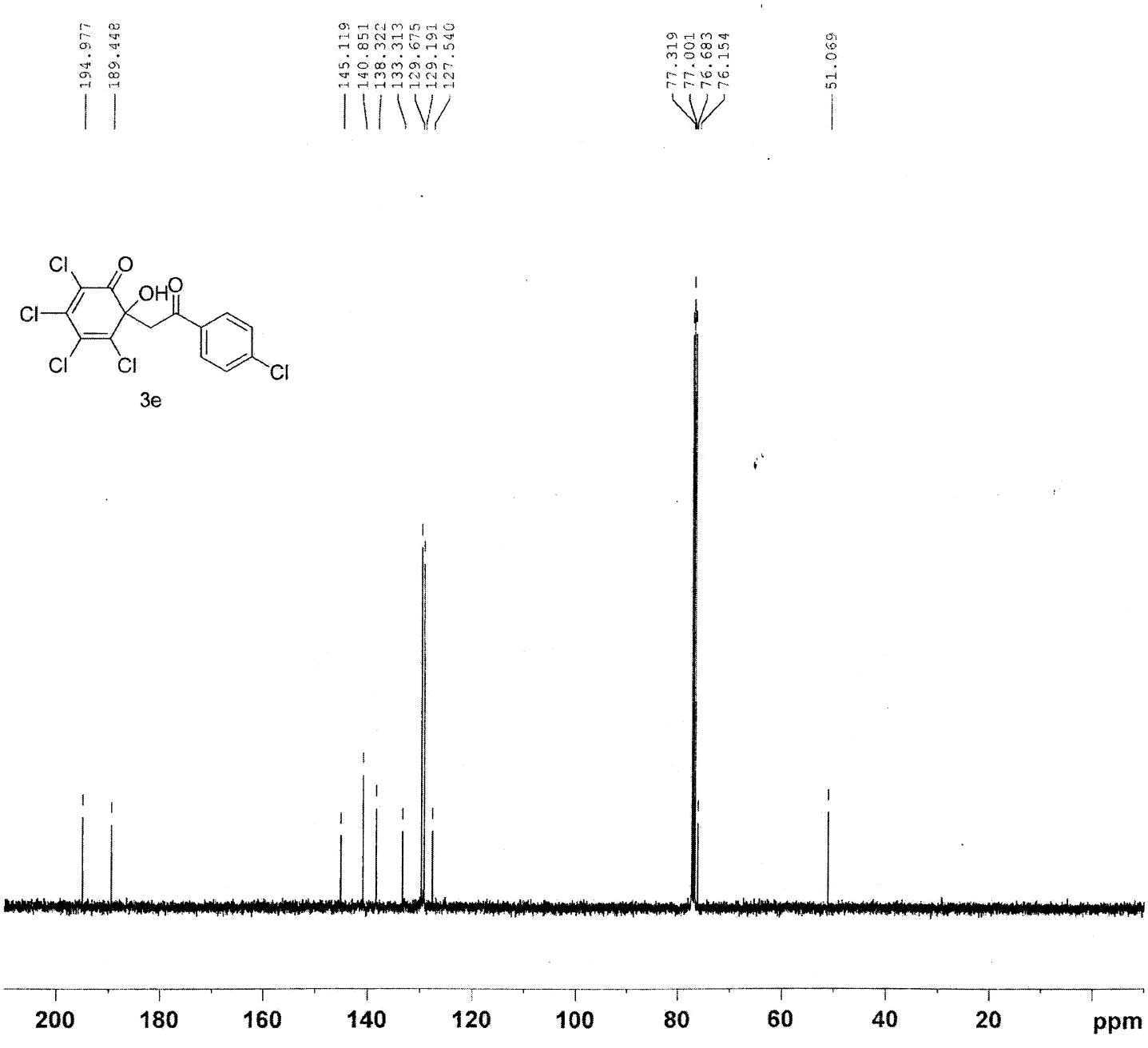


BRUKER

NAME	guozw-lihj-785+-20081211
EXPNO	1
PROCNO	1
Date_	20081211
Time	13.49
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	101
DW	60.800 usec
DE	6.50 usec
TE	291.4 K
D1	1.00000000 sec
TD0	1

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

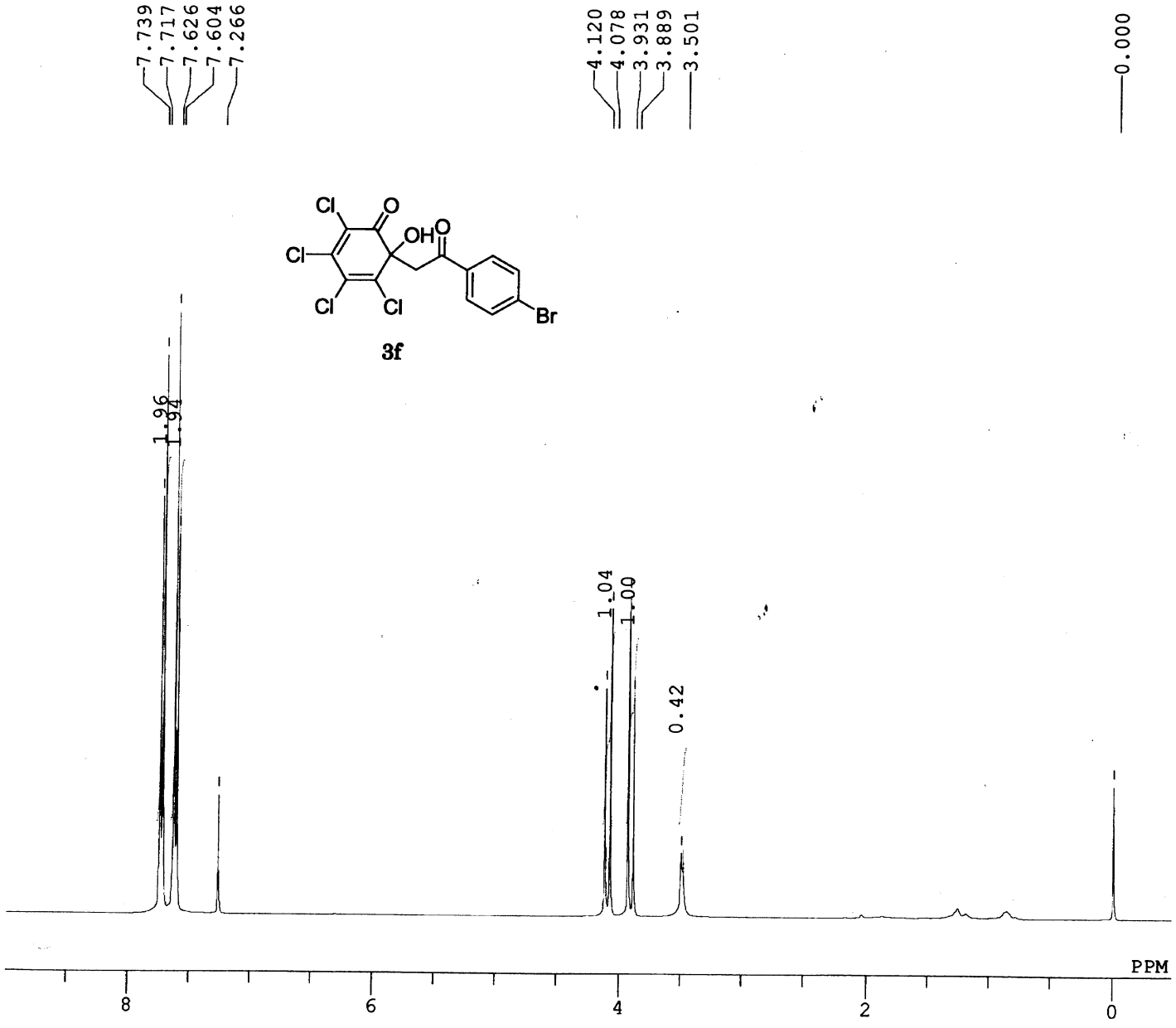
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300259 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	lihr-lihj785-20081218-C
EXPNO	1
PROCNO	1
Date_	20081218
Time	11.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	103
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	101
DW	20.800 usec
DE	6.50 usec
TE	291.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ==========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
========= CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	120.00 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127757 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

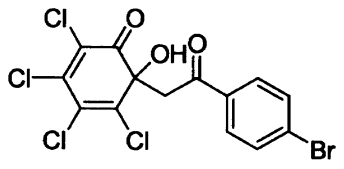
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-4e-H-1.jdf
lee-4e-H



DFILE C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT lee-4e-H
DATIM 18-02-2009 22:31:39
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

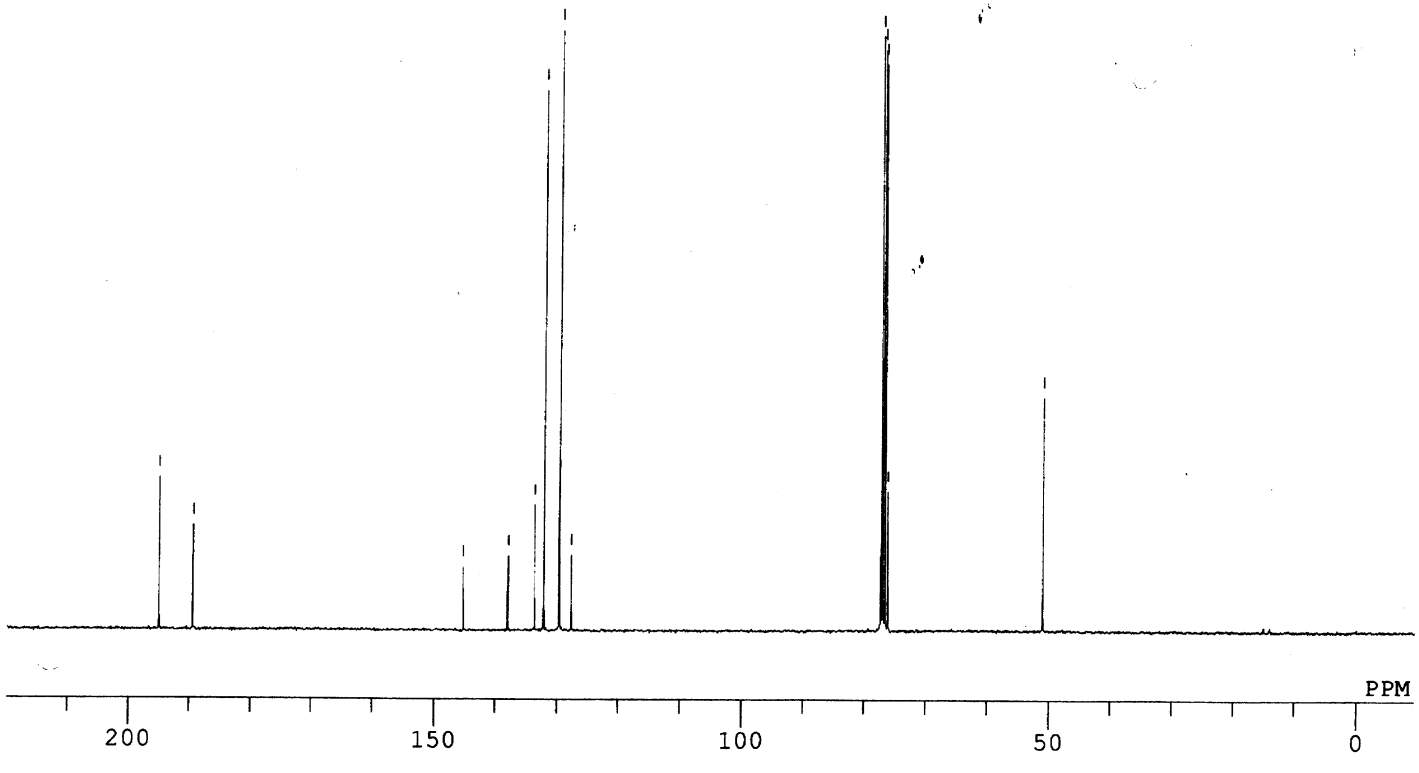
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-4e-C-1.jdf
lee-4e-C

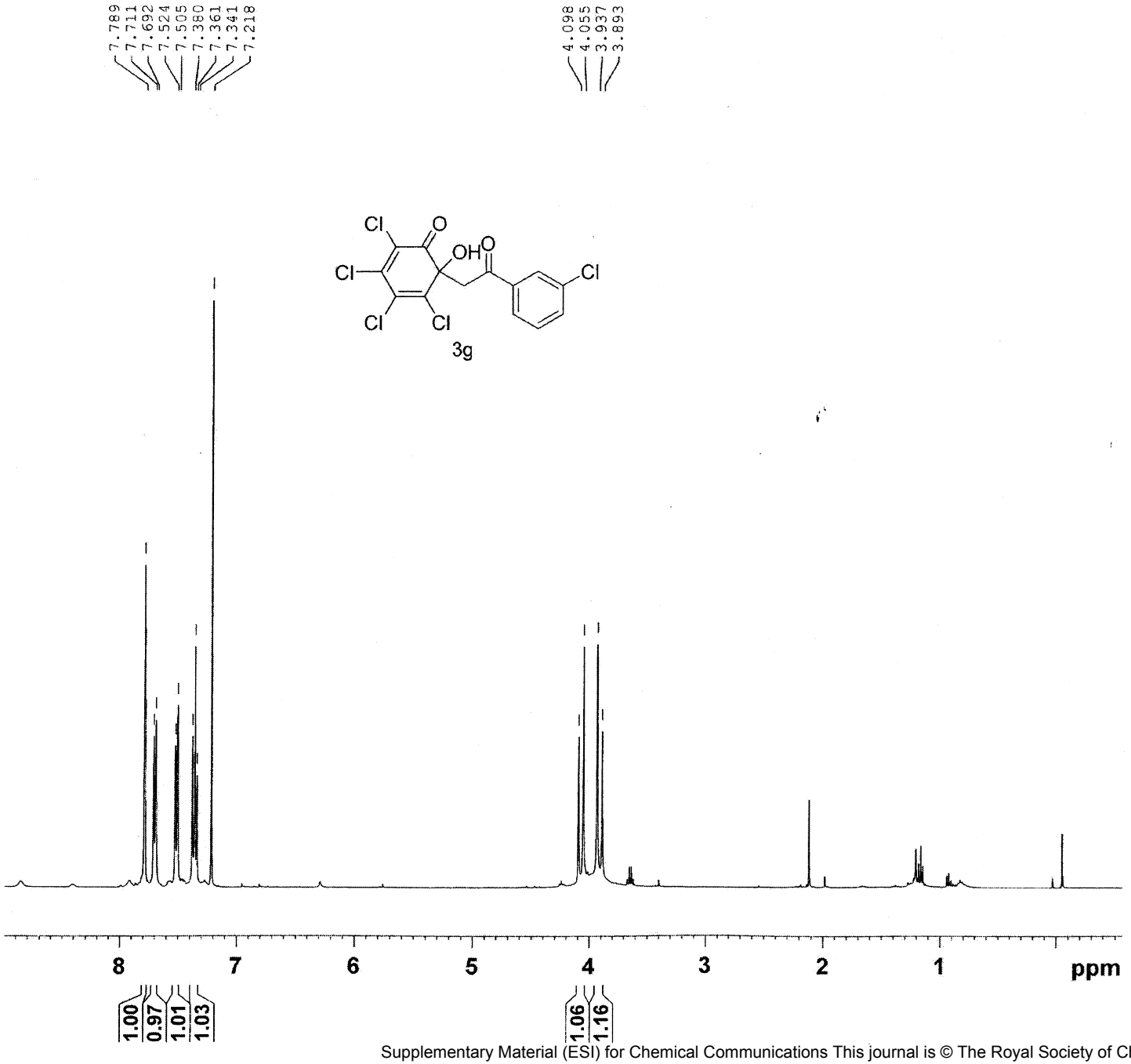
195.057
189.489
145.249
138.002
133.693
132.224
129.755
129.698
129.650
127.714
77.315
77.000
76.676
76.218
51.075



3f

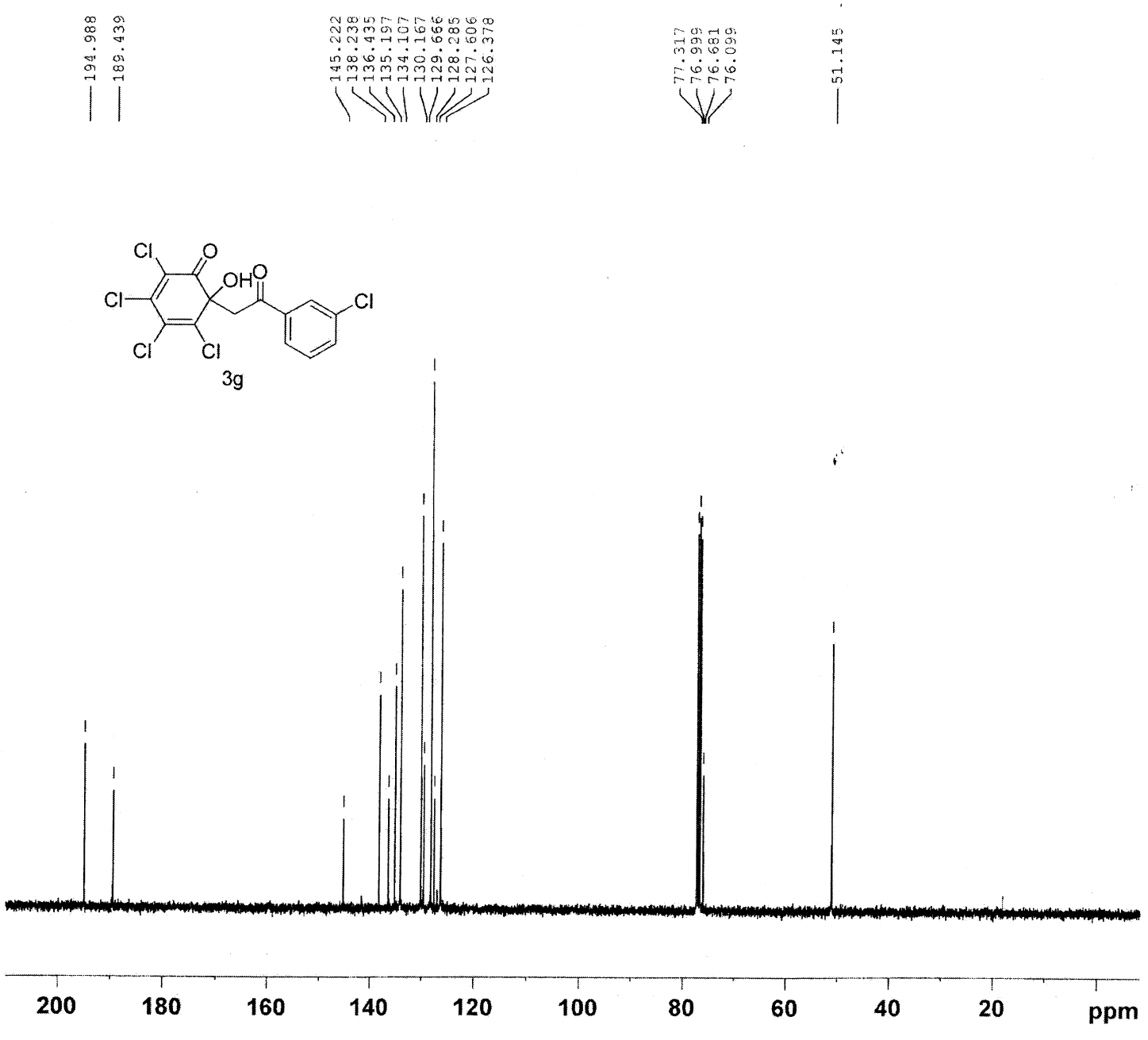
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-4e-C
DATIM	18-02-2009 23:28:48
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32768
FREQU	31407.03 Hz
SCANS	721
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.00 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	56





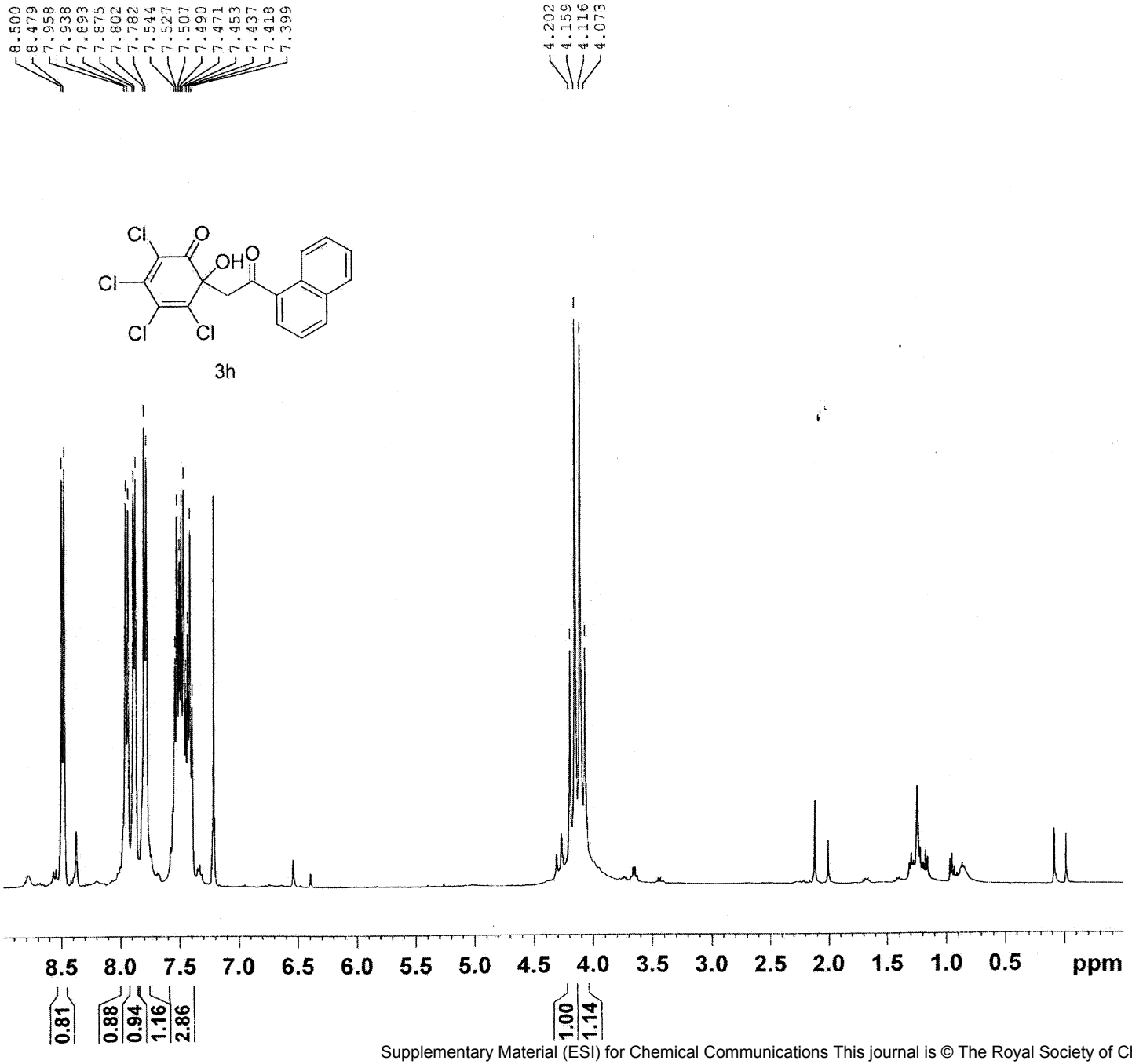
BRUKER

NAME	guoxw-lihj-792-20081216
EXPNO	1
PROCNO	1
Date_	20081216
Time	10.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	292.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300259 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



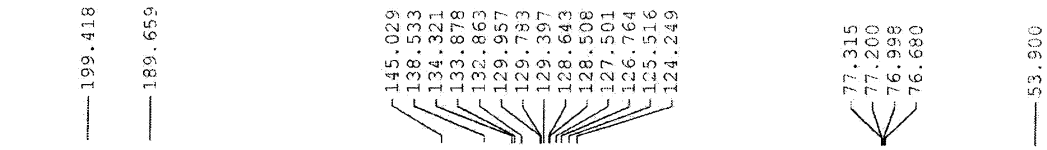
BRUKER

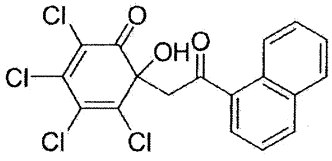
NAME	guoxw-lihj-792-20081216
EXPNO	2
PROCNO	1
Date_	20081216
Time	10.15
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	160
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	293.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127774 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	guoxw-liwj-40-20081215
EXPNO	1
PROCNO	1
Date_	20081215
Time	16.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	292.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300259 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

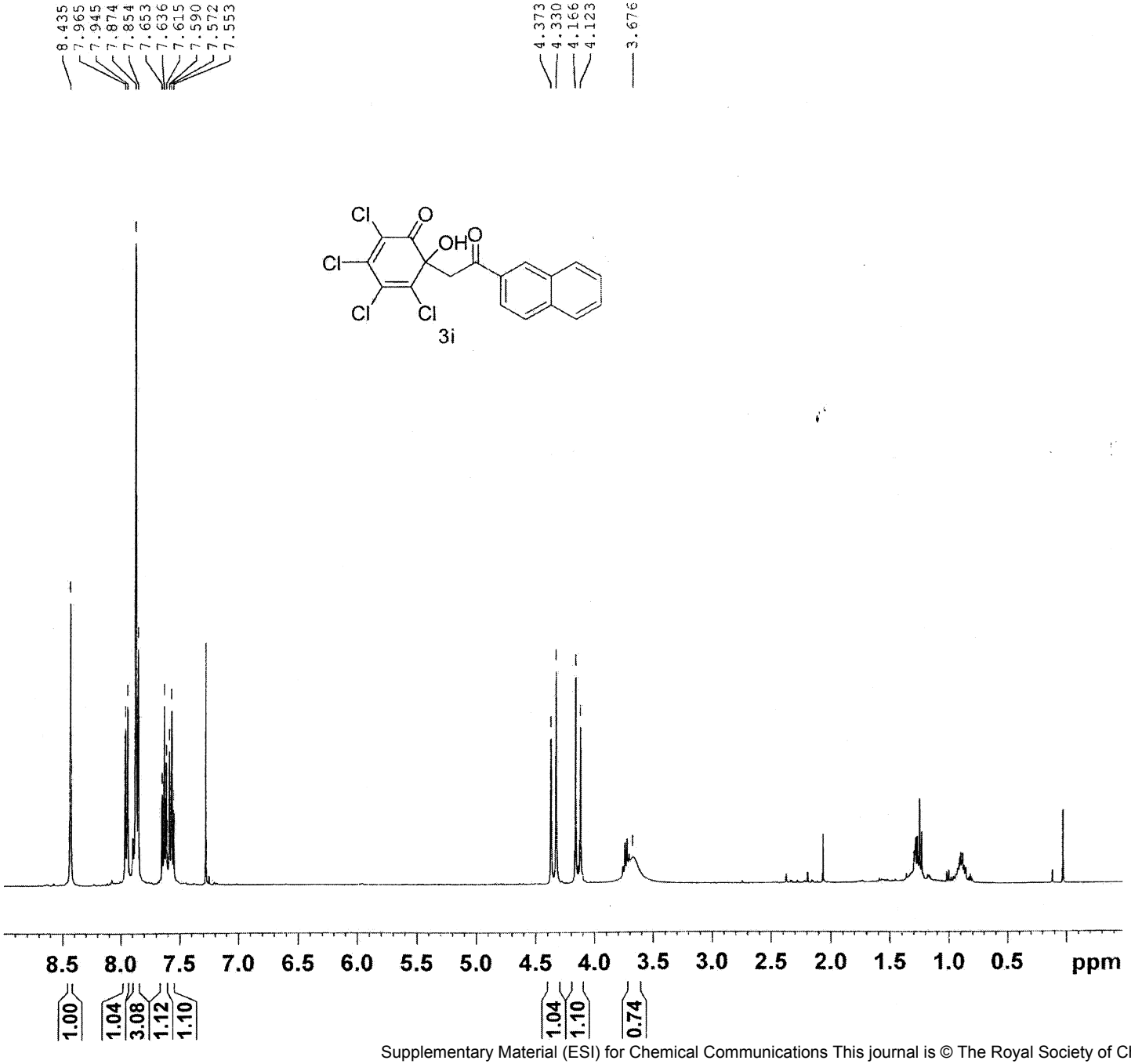




3h

BRUKER

NAME	guoxw-lihj-8-20081223
EXPNO	2
PROCNO	1
Date_	20081223
Time	16.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	204
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	292.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127751 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

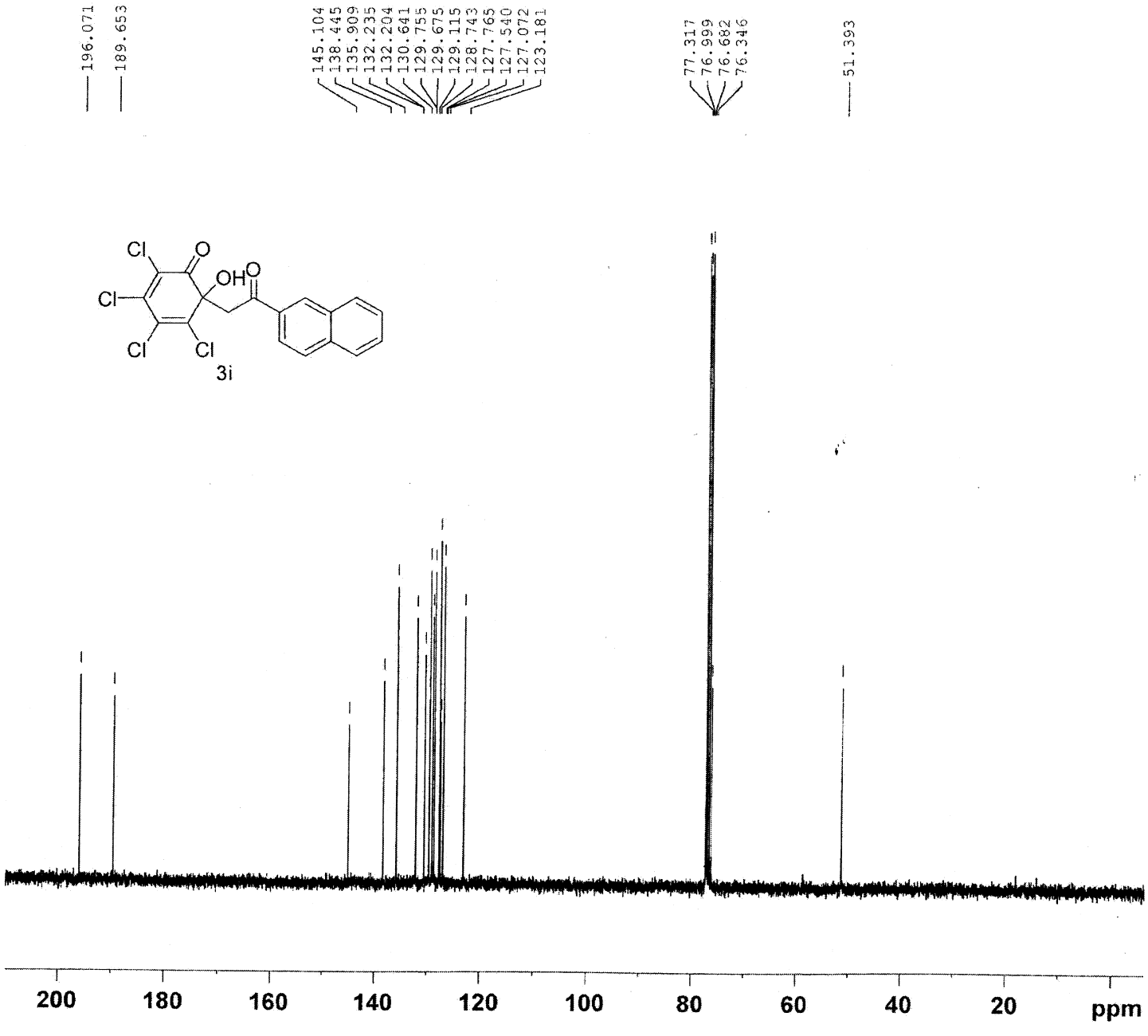


BRUKER

NAME	lihr-lihj791-H	
EXPNO	1	
PROCNO	1	
Date_	20081215	
Time	11.48	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	11.3	
DW	60.800	usec
DE	6.50	usec
TE	291.7	K
D1	1.00000000	sec
TD0	1	

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

NUC1	1H	
P1	12.30	usec
PL1	-1.00	dB
PL1W	17.01305389	W
SFO1	400.1324710	MHz
SI	32768	
SF	400.1300001	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

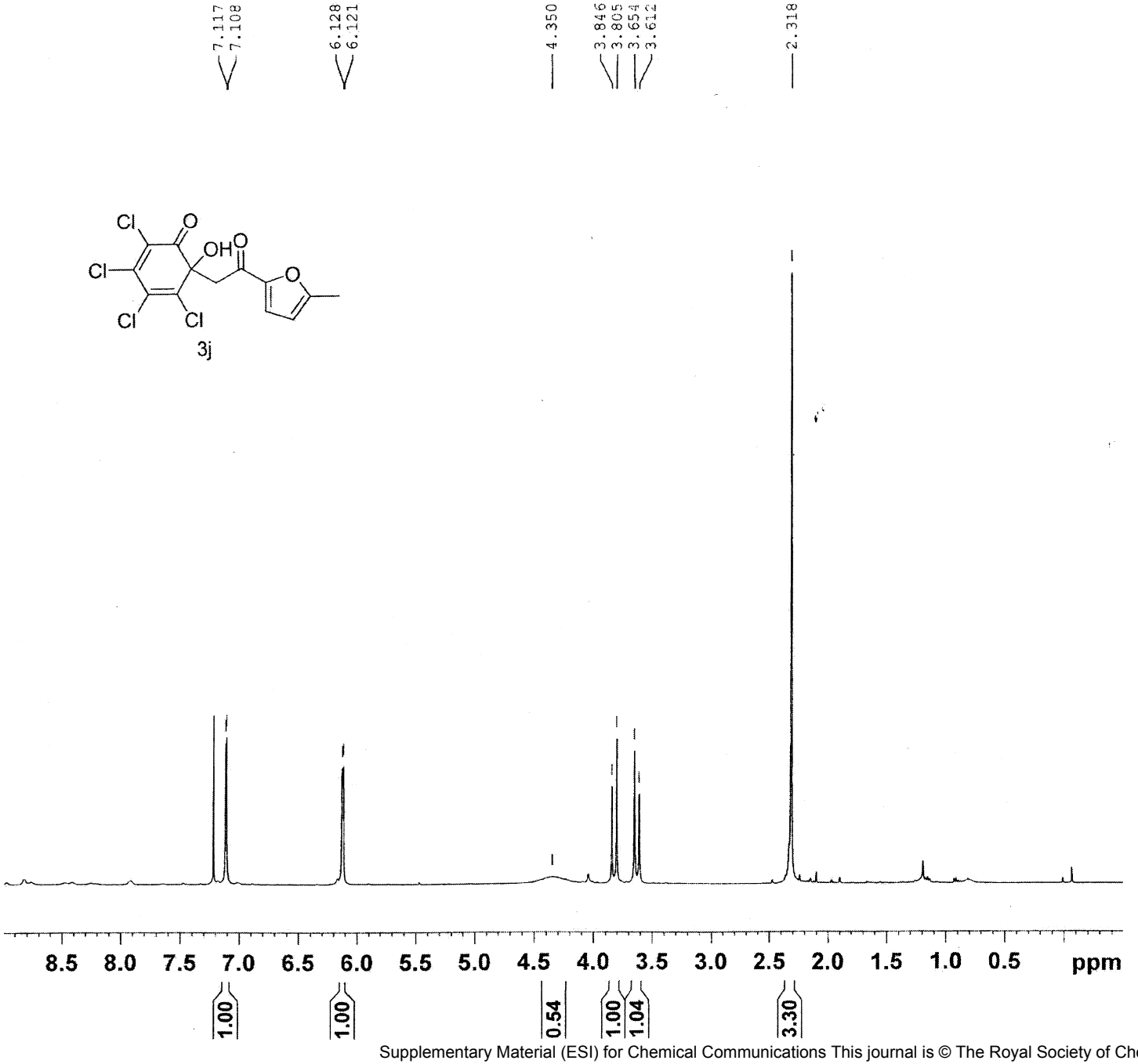


BRUKER

NAME	lihr-lihj791-C
EXPNO	1
PROCNO	1
Date_	20081215
Time	11.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	114
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	144
DW	20.800 usec
DE	6.50 usec
TE	291.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

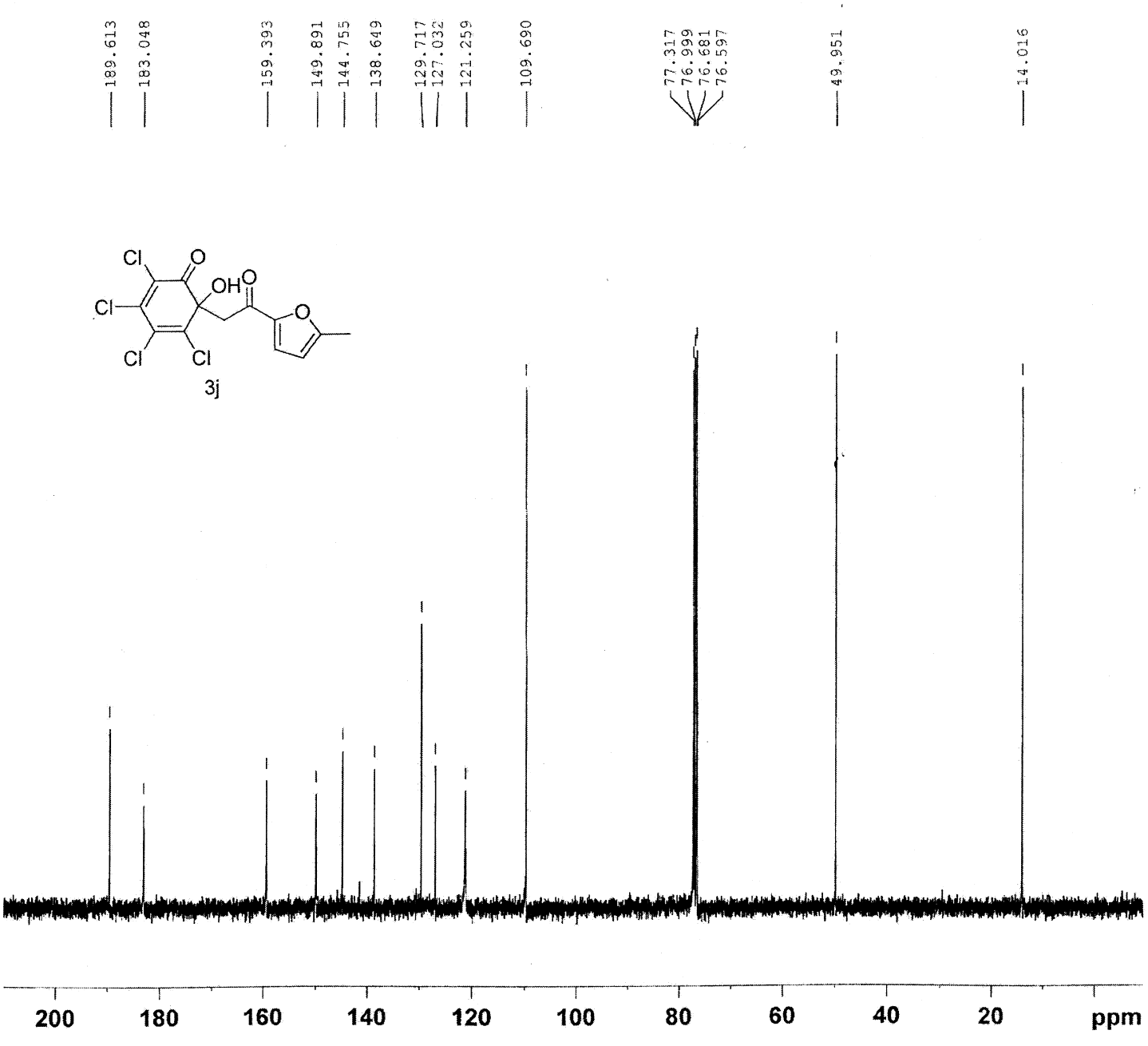
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	120.00 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127792 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

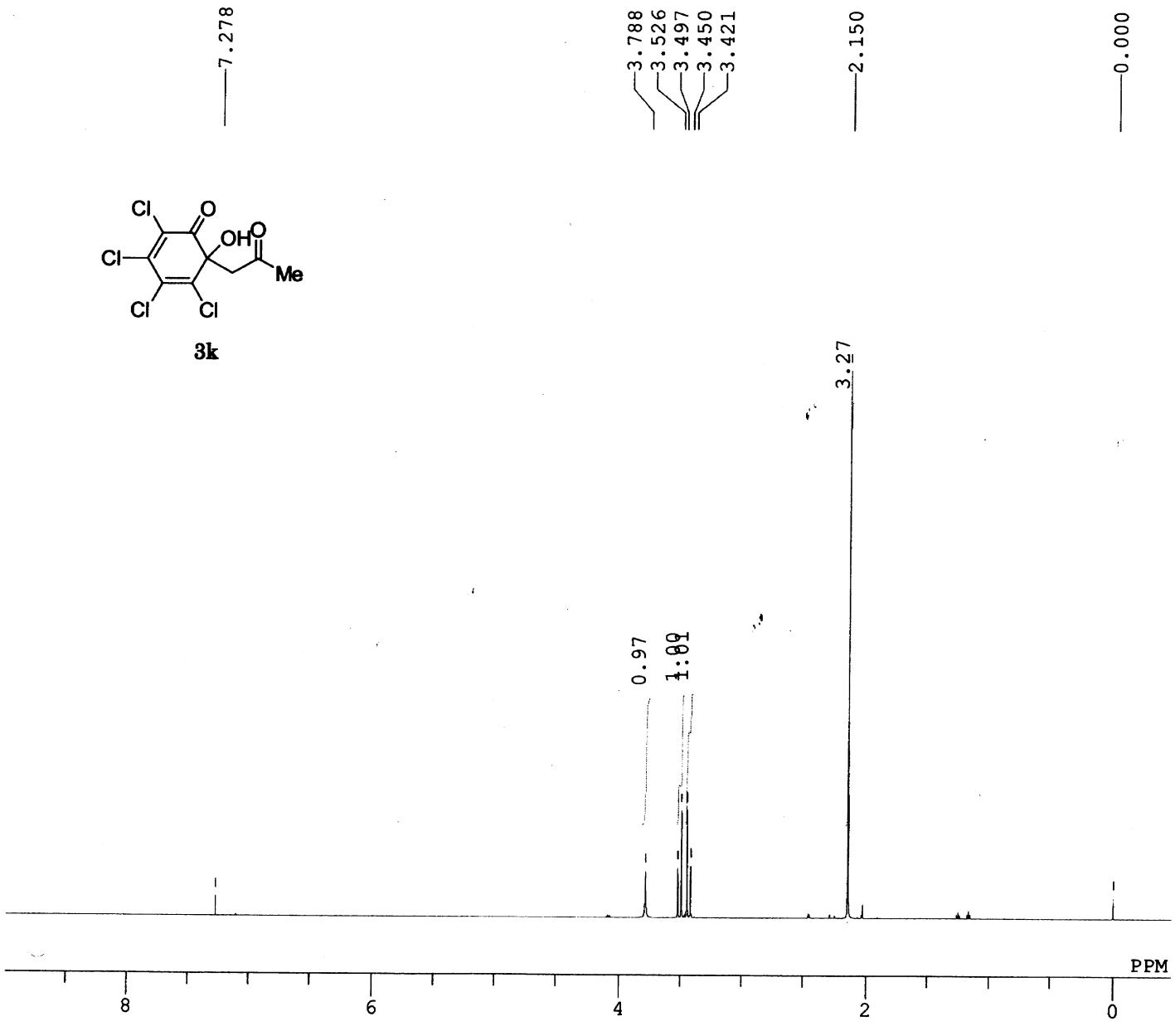
NAME	guoxw-lihj-799-20081216
EXPNO	1
PROCNO	1
Date_	20081216
Time	10.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	293.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300259 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

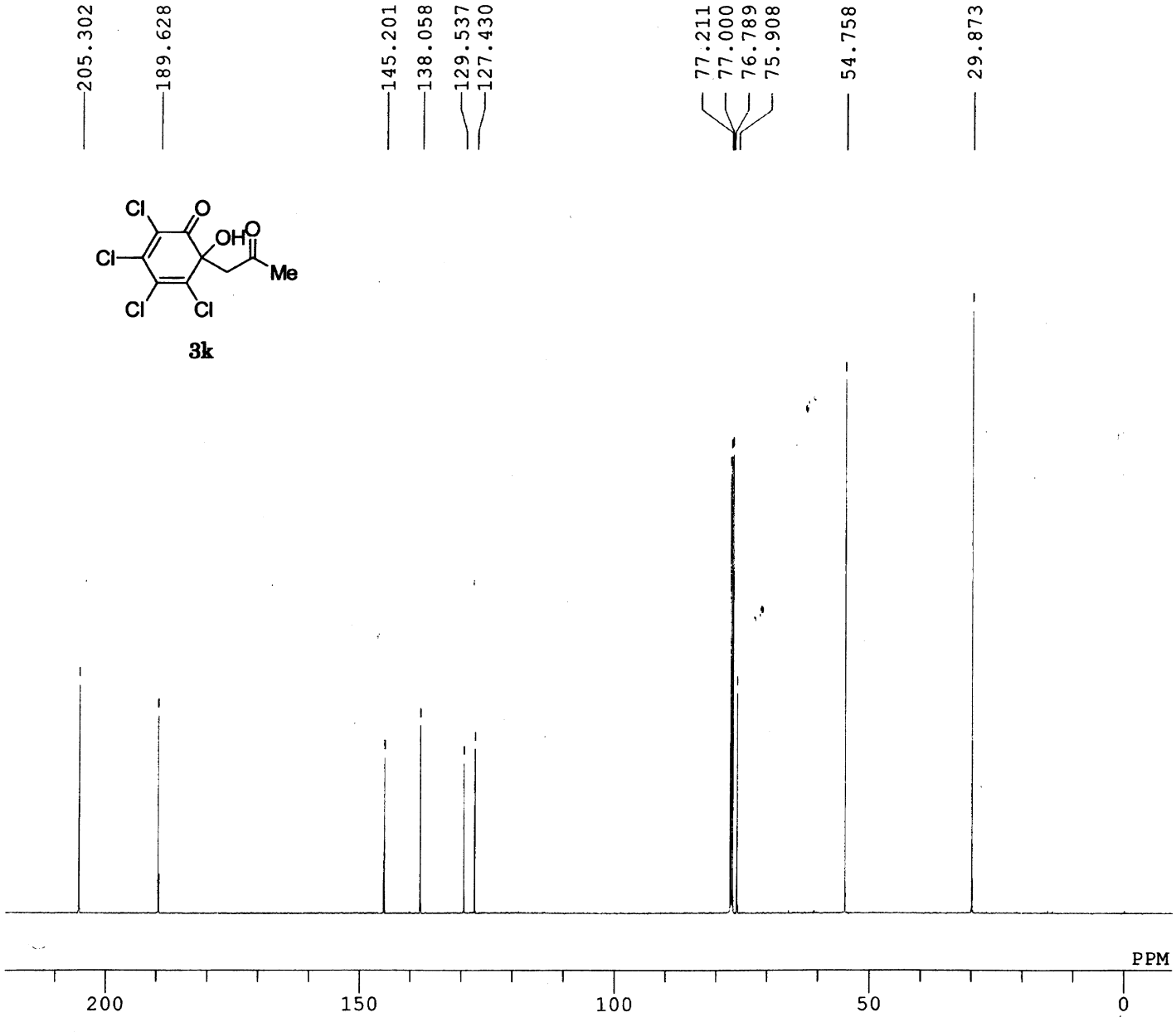
NAME	guoxw-lihj-799-20081216
EXPNO	2
PROCNO	1
Date_	20081216
Time	10.42
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	160
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	80.6
DW	20.800 usec
DE	6.50 usec
TE	293.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.38087484 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127787 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R876-0H-2.jdf
lee-R876-0H



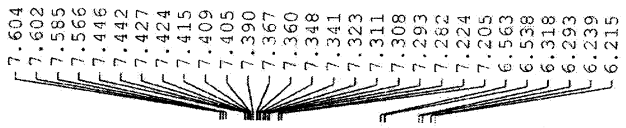
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R876-0H
DATIM	17-02-2009 12:35:22
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	32768
FREQU	11261.26 Hz
SCANS	8
ACQTM	2.9098 sec
PD	5.0000 sec
PW1	6.90 usec
IRNUC	1H
CTEMP	18.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42

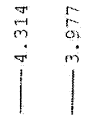
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R876-0C-1.jdf
lee-R876-0C

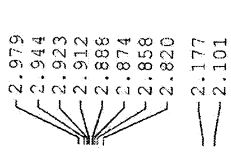


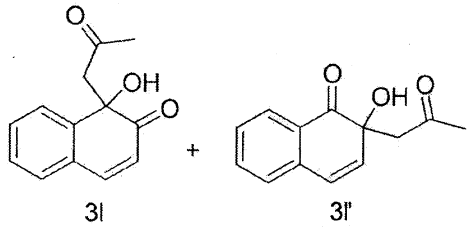
3k

```
DFILE   C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT   lee-R876-0C
DATIM   17-02-2009 14:06:40
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         150.92 MHz
OBSET           8.52 KHz
OBFIN           1.74 Hz
POINT          32768
FREQU       47348.49 Hz
SCANS           2002
ACQTM         0.6921 sec
PD            2.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP           20.2 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              1.20 Hz
RGAIN             60
```









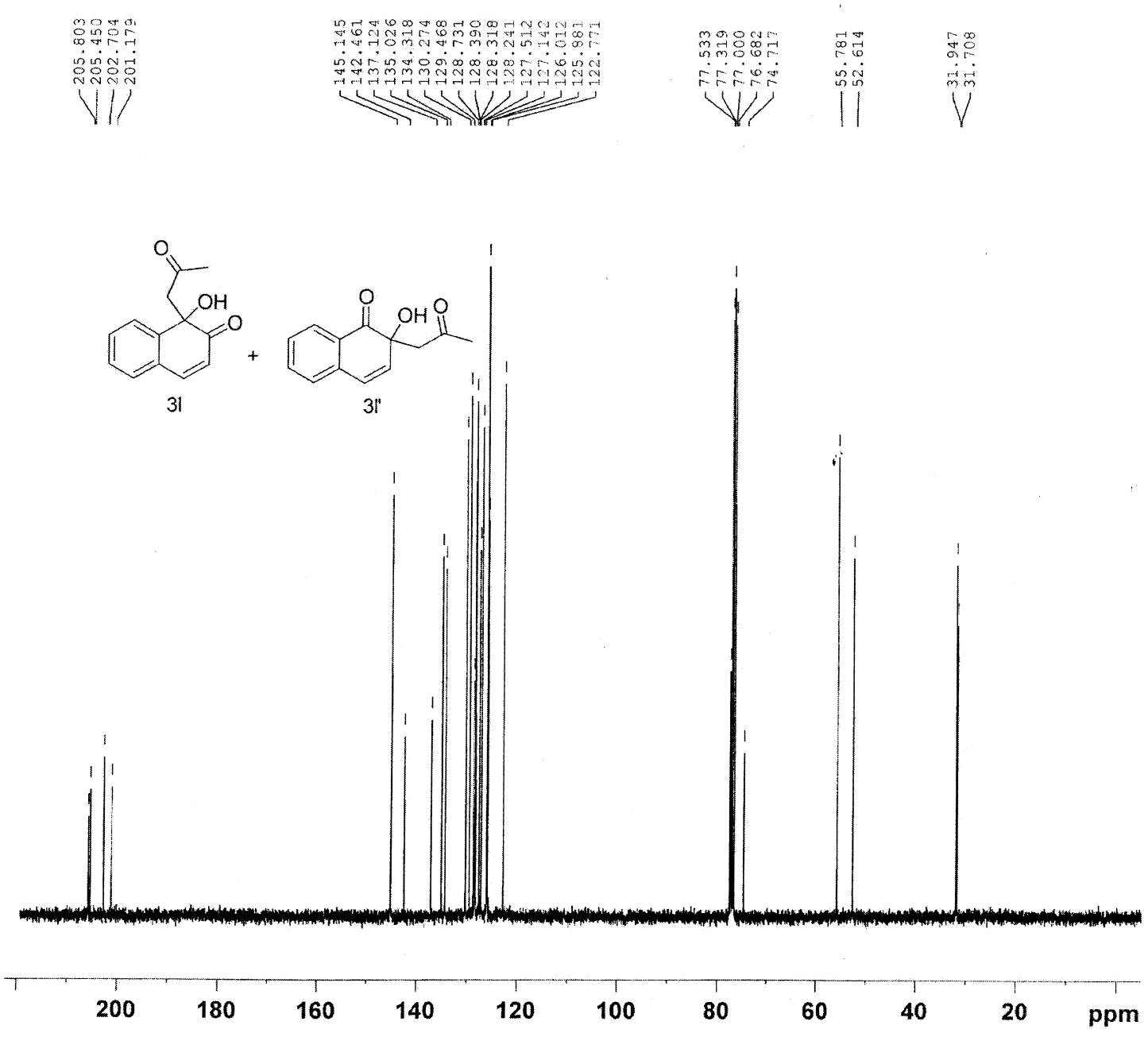
3l 3l'

BRUKER

NAME	guoxw-lwj-20-20081124
EXPNO	1
PROCNO	1
Date_	20081124
Time_	16.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	20.2
DW	60.800 usec
DE	6.50 usec
TE	292.2 K
D1	1.00000000 sec
TD0	1

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

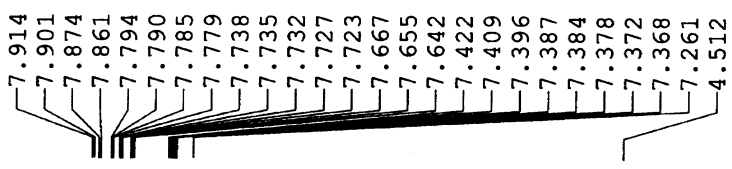
NUC1	1H
P1	12.30 usec
PL1	-1.00 dB
PL1W	17.01305389 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

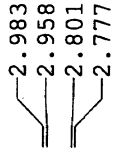


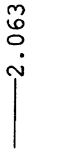
BRUKER

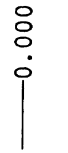
NAME	guoxw-lihj-201-20081208
EXPNO	2
PROCNO	1
Date_	20080708
Time	15.05
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	200
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	912
DW	20.800 usec
DE	6.50 usec
TE	292.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.80 usec
PL1	2.00 dB
PL1W	55.31277084 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	15.50 dB
PL13	120.00 dB
PL2W	17.01305389 W
PL12W	0.38087484 W
PL13W	0.00000000 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127839 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R869-2H-1.jdf
lee-R869-2H

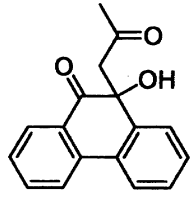




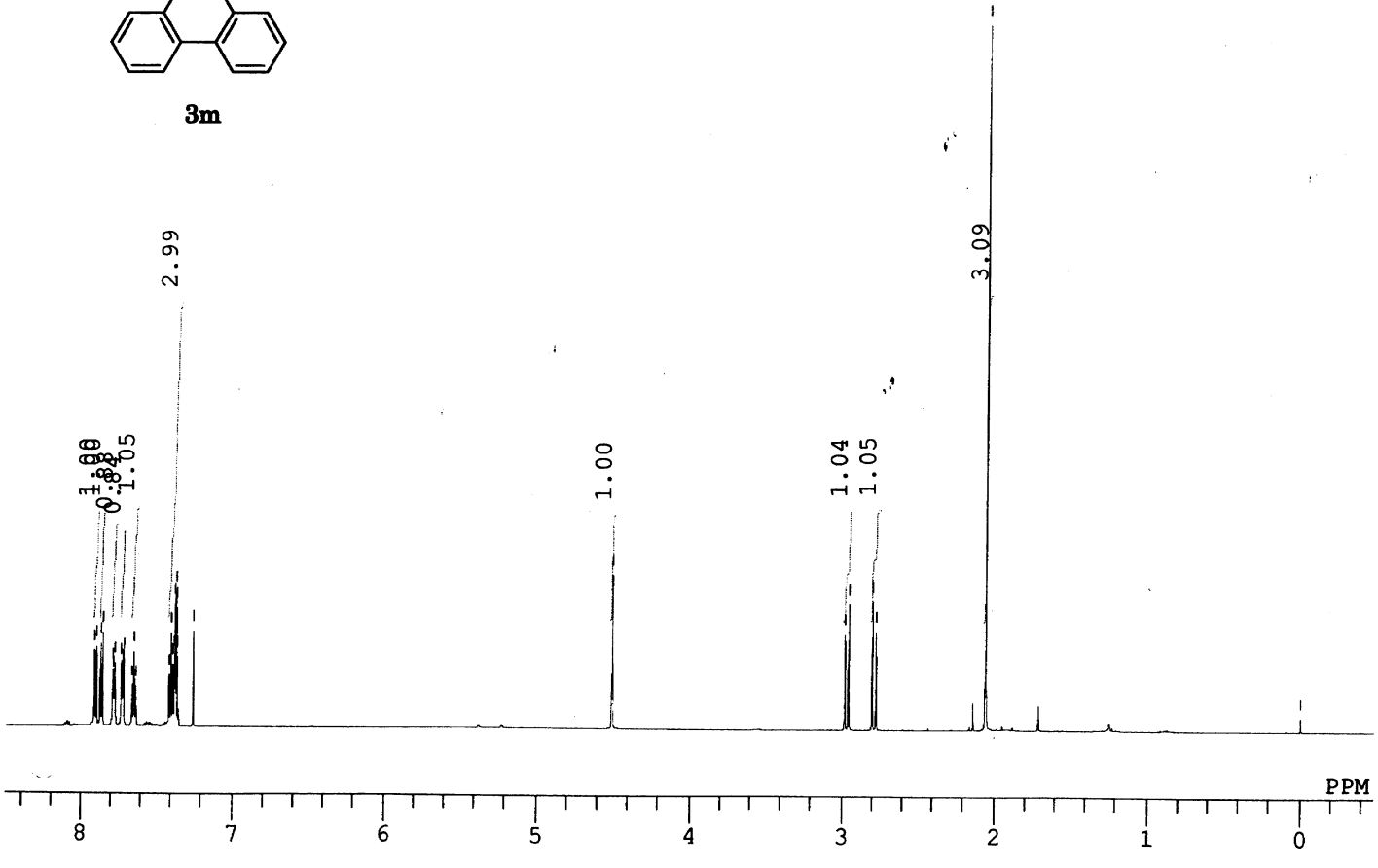




DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R869-2H
DATIM	13-02-2009 22:27:56
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	32768
FREQU	11261.26 Hz
SCANS	8
ACQTM	2.9098 sec
PD	5.0000 sec
PW1	6.90 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	34



3m



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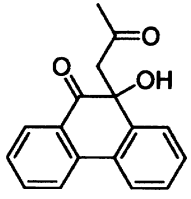
lee-R869-2C

205.455
202.219
139.246
136.612
134.736
129.182
129.154
128.771
128.618
128.531
127.488
125.812
124.280
123.016
78.159
77.211
77.000
76.789
55.399
31.932

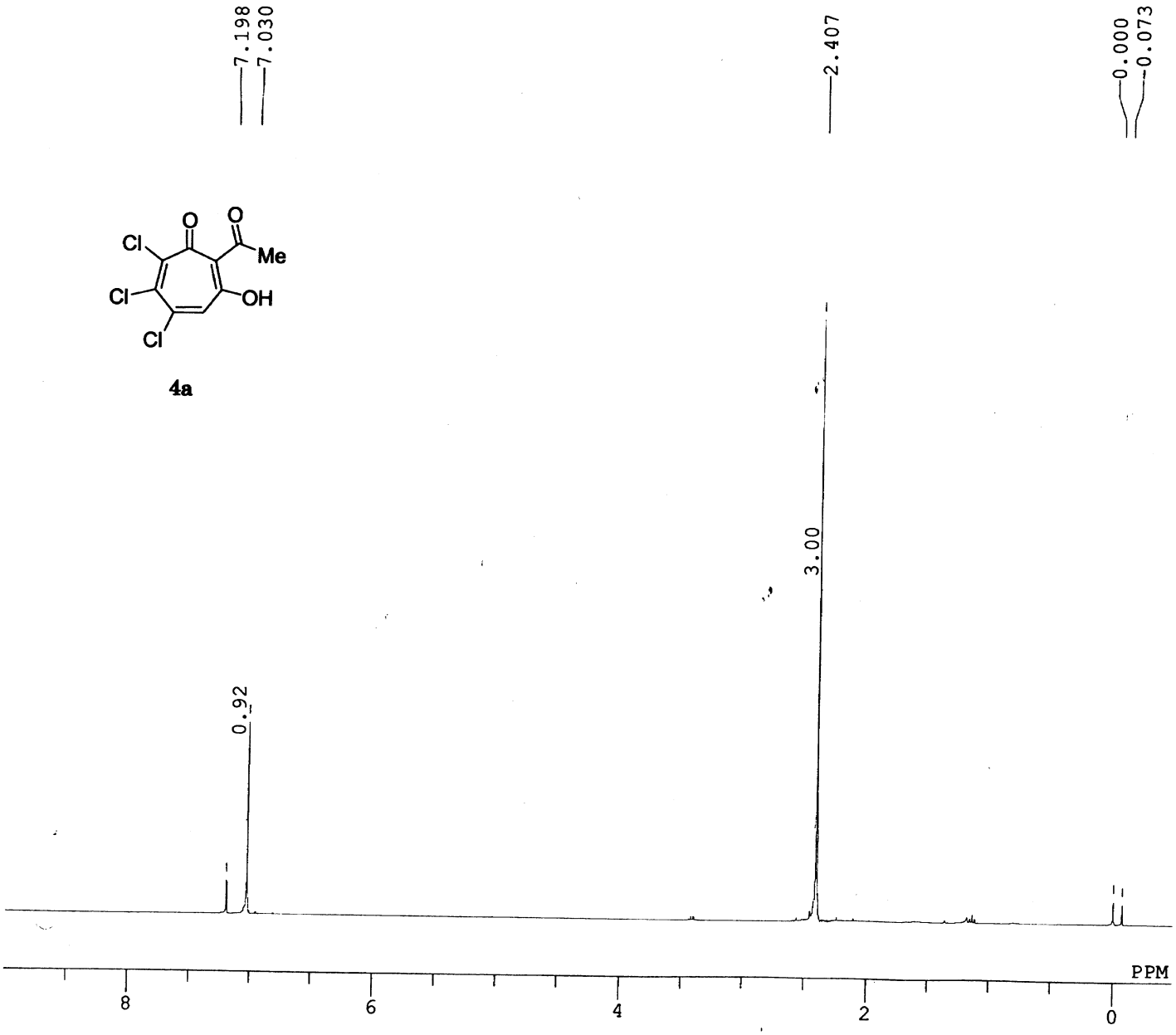


3m

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R869-2C
DATIM	13-02-2009 23:05:22
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	800
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

PPM
200
150
100
50
0

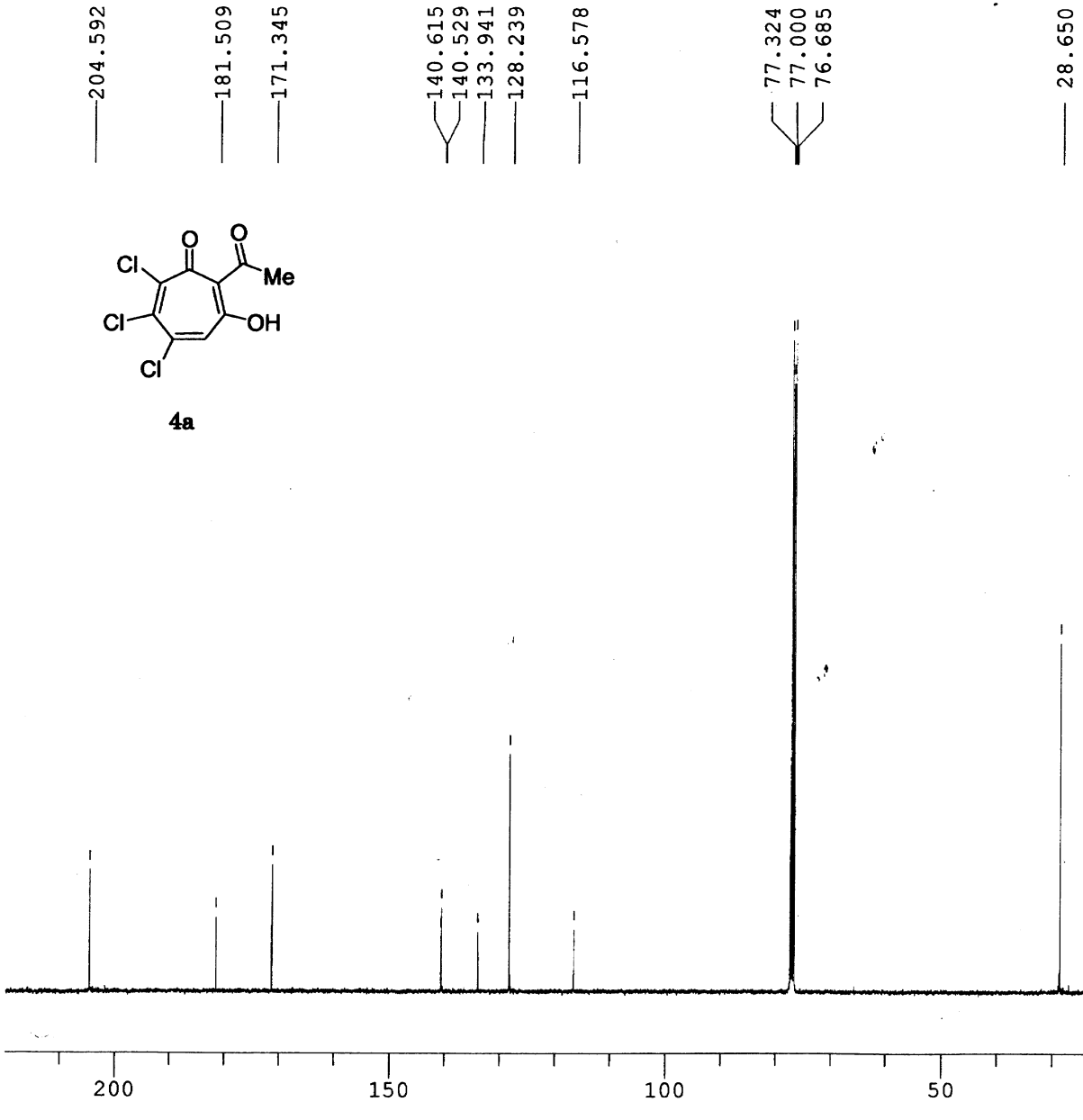
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R876-3H-1.jdf
lee-R876-3H



4a

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R876-3H
DATIM	17-02-2009 15:01:52
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	32768
FREQU	7503.00 Hz
SCANS	8
ACQTM	4.3673 sec
PD	5.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R876-3C-1.jdf
lee-R876-3C

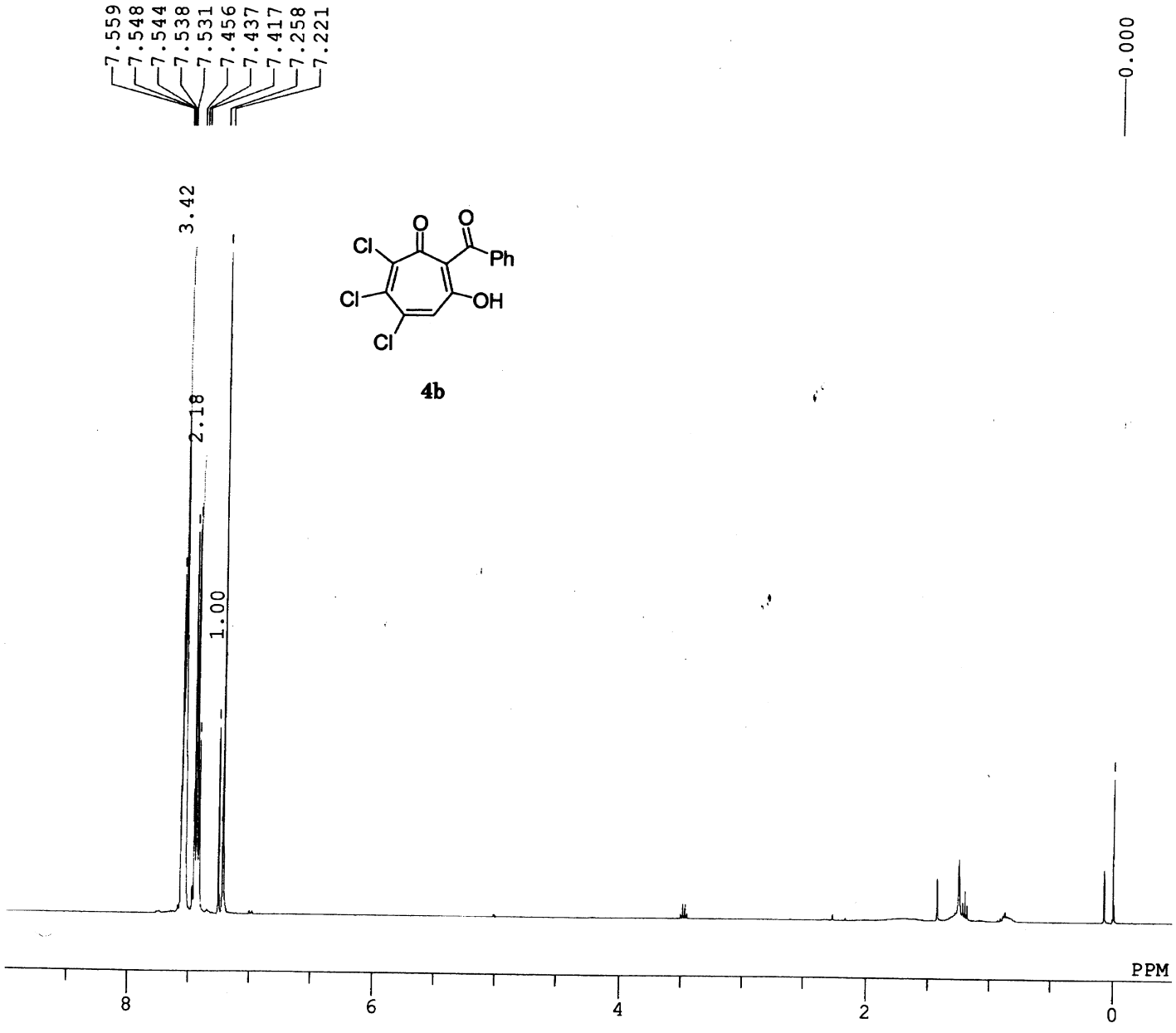


4a

```
DFILE   C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT   lee-R876-3C
DATIM   17-02-2009 16:47:04
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         100.53 MHz
OBSET           5.35 KHz
OBFIN           5.86 Hz
POINT          32768
FREQU       31407.03 Hz
SCANS           2001
ACQTM         1.0433 sec
PD            2.0000 sec
PW1             3.00 usec
IRNUC   1H
CTEMP           19.6 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              1.20 Hz
RGAIN             60
```

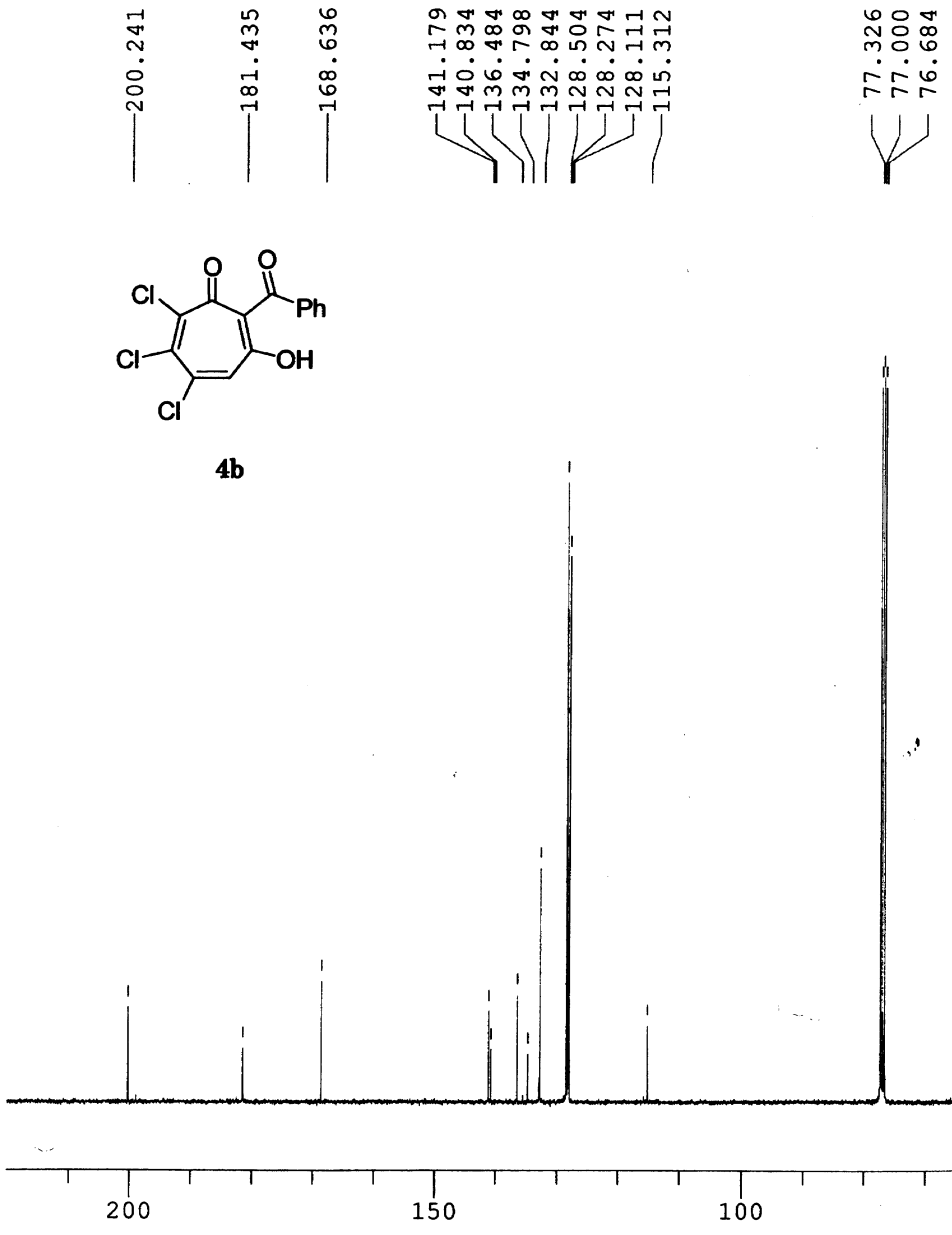
45

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R867-3H-1.jdf
lee-R867-3H



DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R867-3H
DATIM	11-02-2009 21:07:48
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	32768
FREQU	7422.80 Hz
SCANS	8
ACQTM	4.4145 sec
PD	5.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	20.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

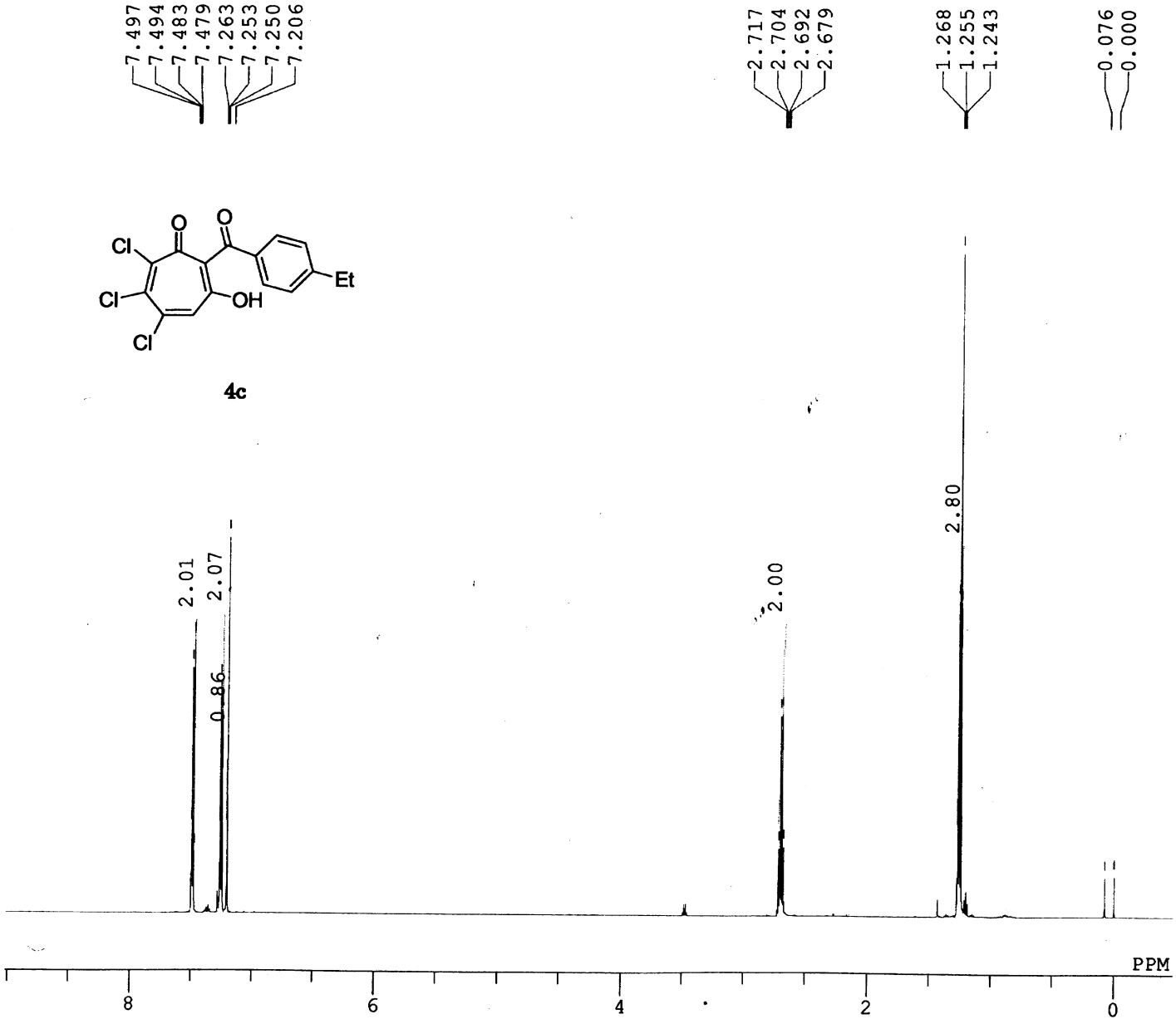
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R867-3C-1.jdf
lee-R867-3C



4b

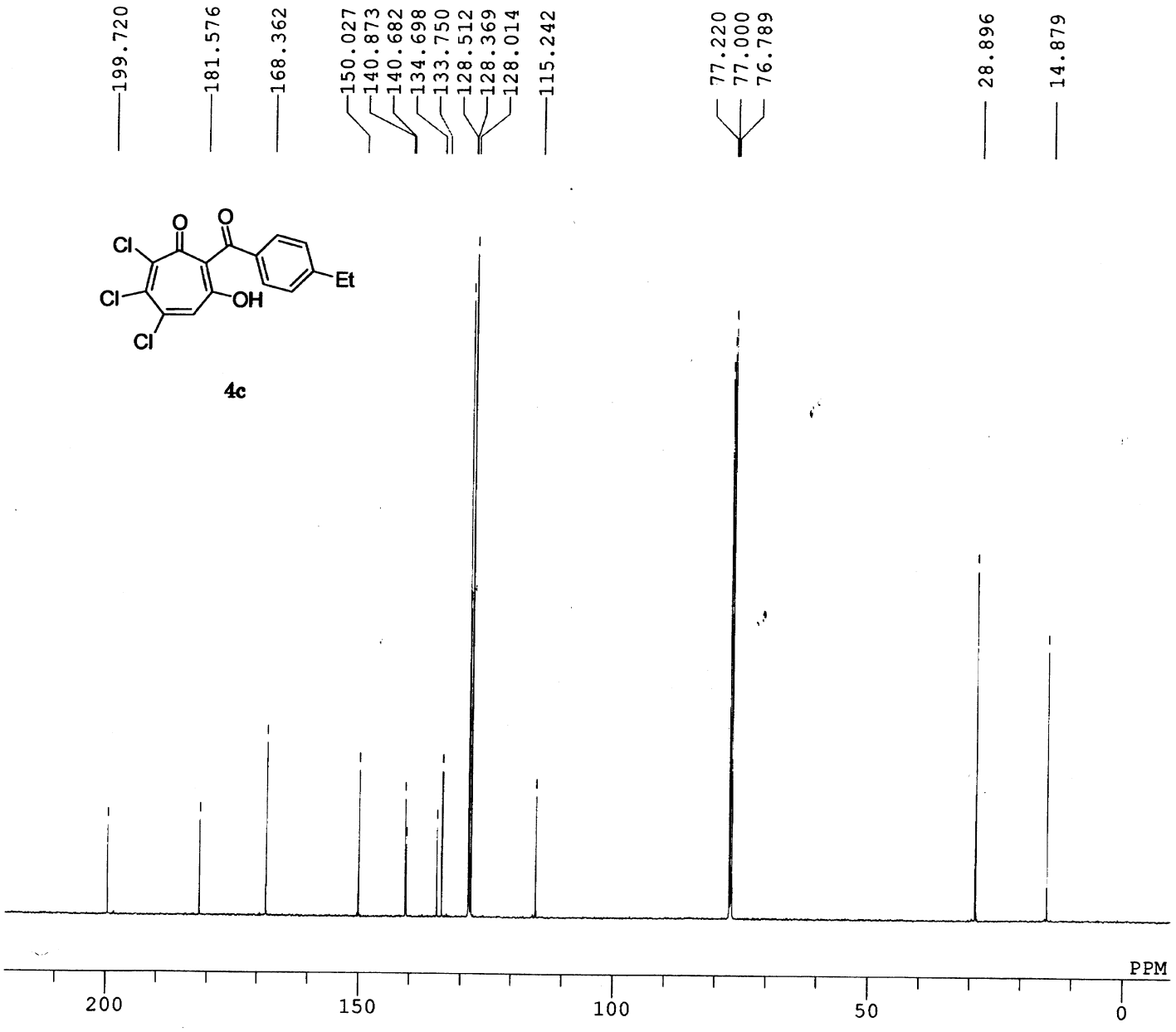
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R867-3C
DATIM	11-02-2009 22:52:51
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	32768
FREQU	31250.00 Hz
SCANS	2000
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.20 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R868-3H-1.jdf
lee-R868-3H



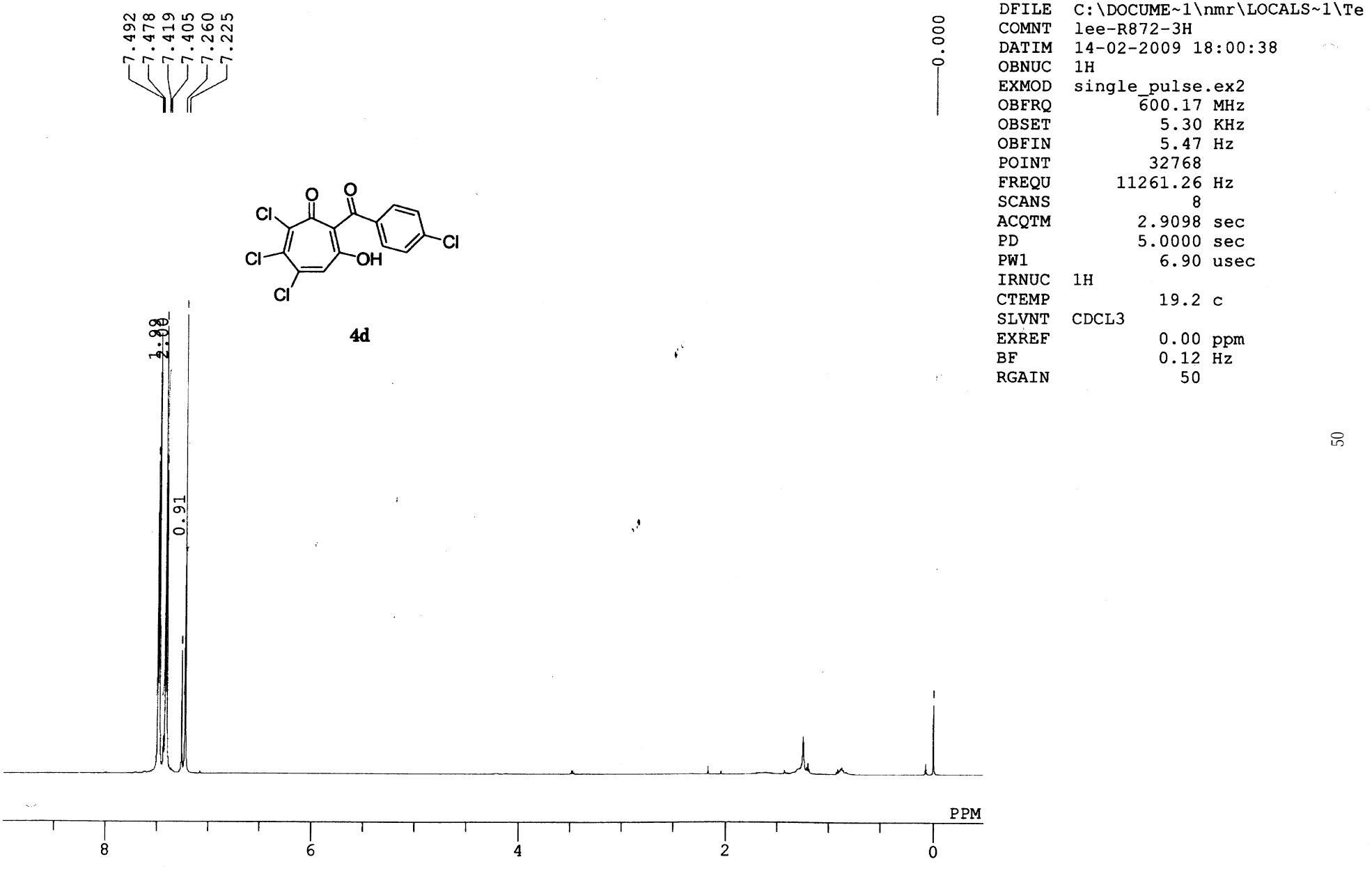

```
DFILE   C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT   lee-R868-3H
DATIM   12-02-2009 16:56:32
OBNUC   1H
EXMOD   single_pulse.ex2
OBFRQ         600.17 MHz
OBSET           5.30 KHz
OBFIN           5.47 Hz
POINT          32768
FREQU       11261.26 Hz
SCANS              8
ACQTM          2.9098 sec
PD             5.0000 sec
PW1              6.90 usec
IRNUC   1H
CTEMP           19.1 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.12 Hz
RGAIN             42
```

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R868-3C-1.jdf
lee-R868-3C



DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R868-3C
DATIM	12-02-2009 17:24:45
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	602
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

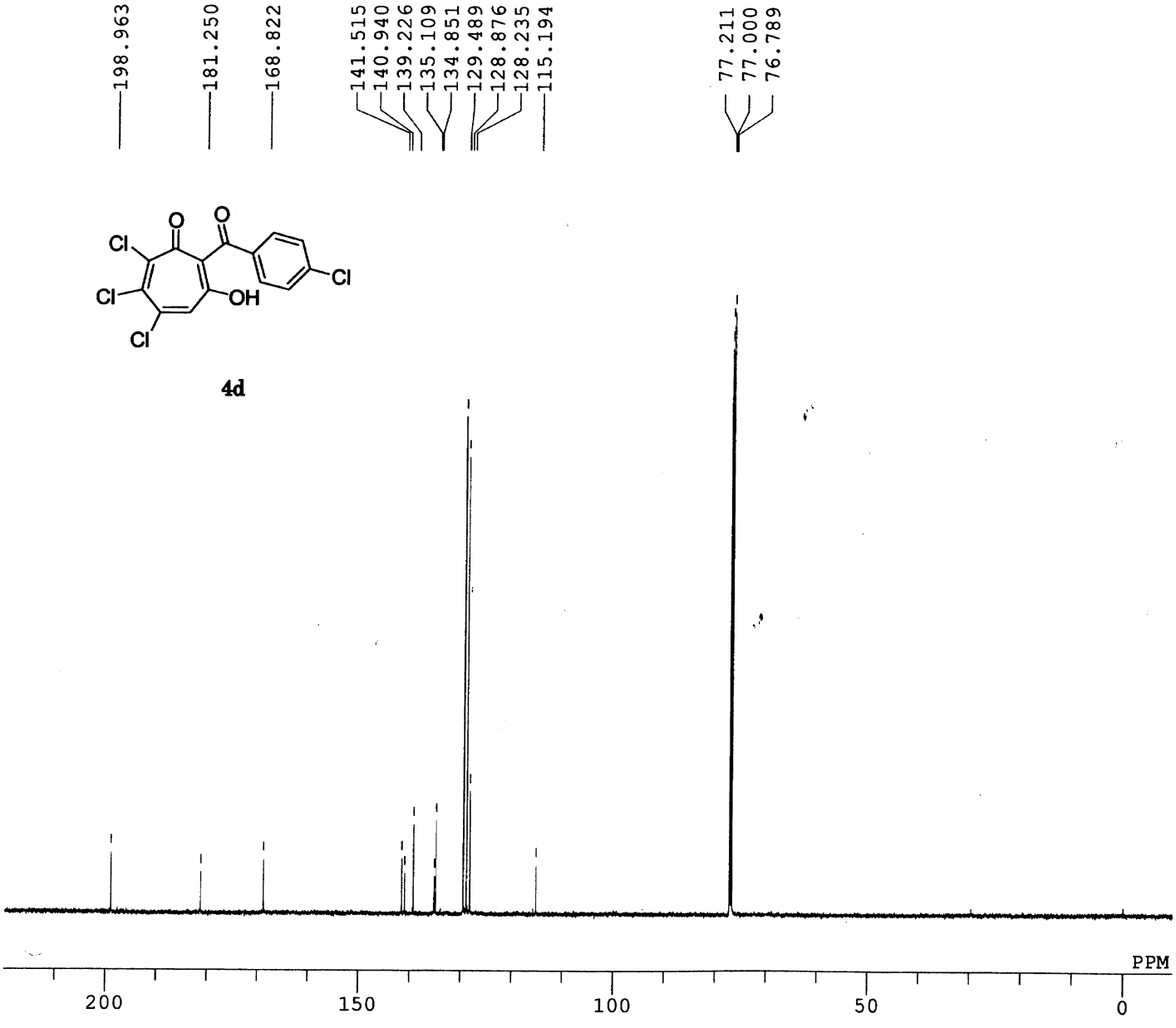
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R872-3H-1.jdf
lee-R872-3H



DFILE C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT lee-R872-3H
DATIM 14-02-2009 18:00:38
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 32768
FREQU 11261.26 Hz
SCANS 8
ACQTM 2.9098 sec
PD 5.0000 sec
PW1 6.90 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

50

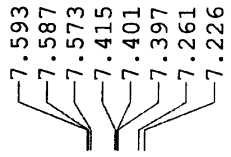
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R872-3C-1.jdf
lee-R872-3C



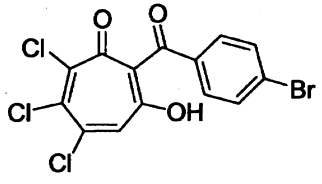
4d

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R872-3C
DATIM	14-02-2009 18:47:43
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	50

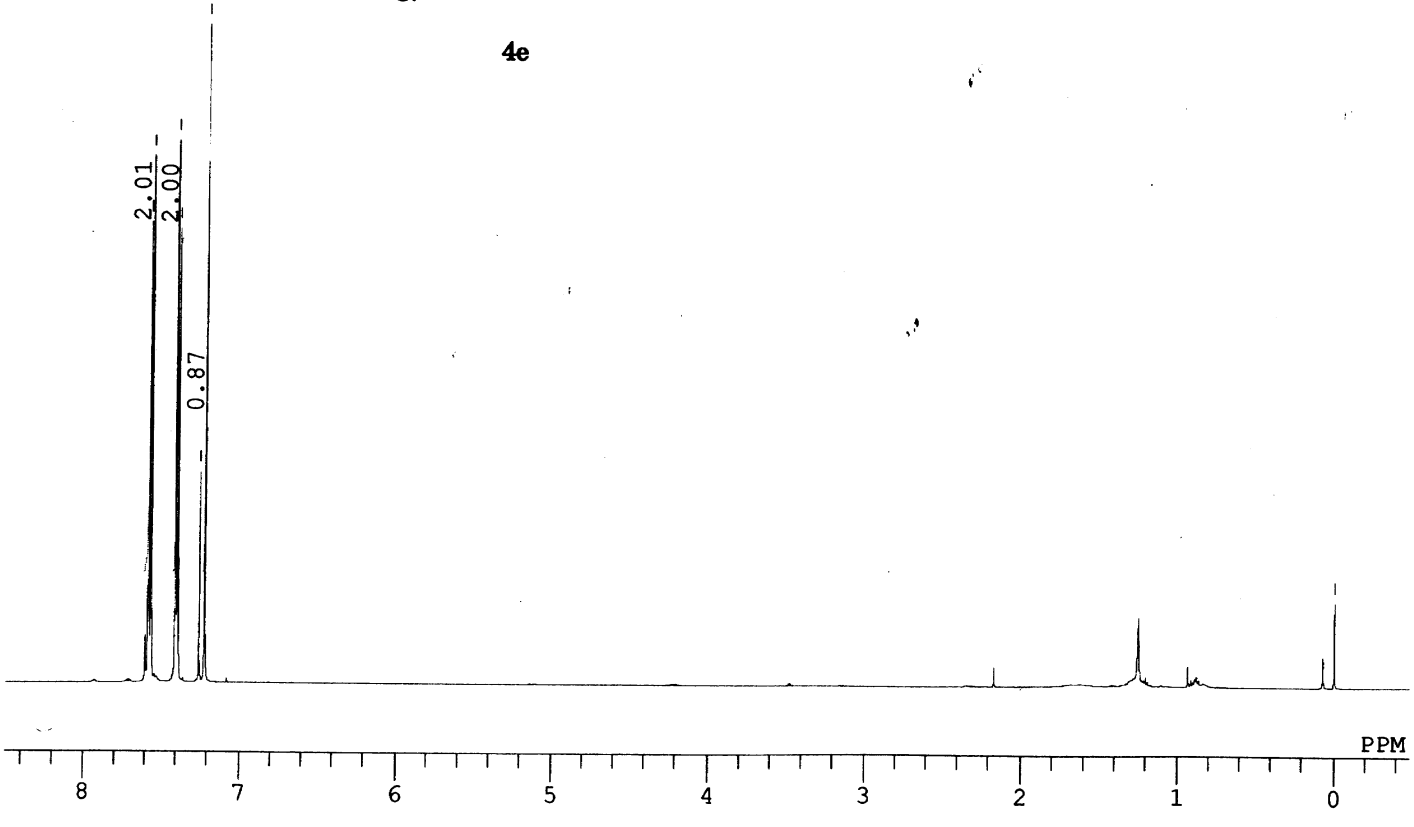
C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R870-3H-3.jdf
lee-R870-3H



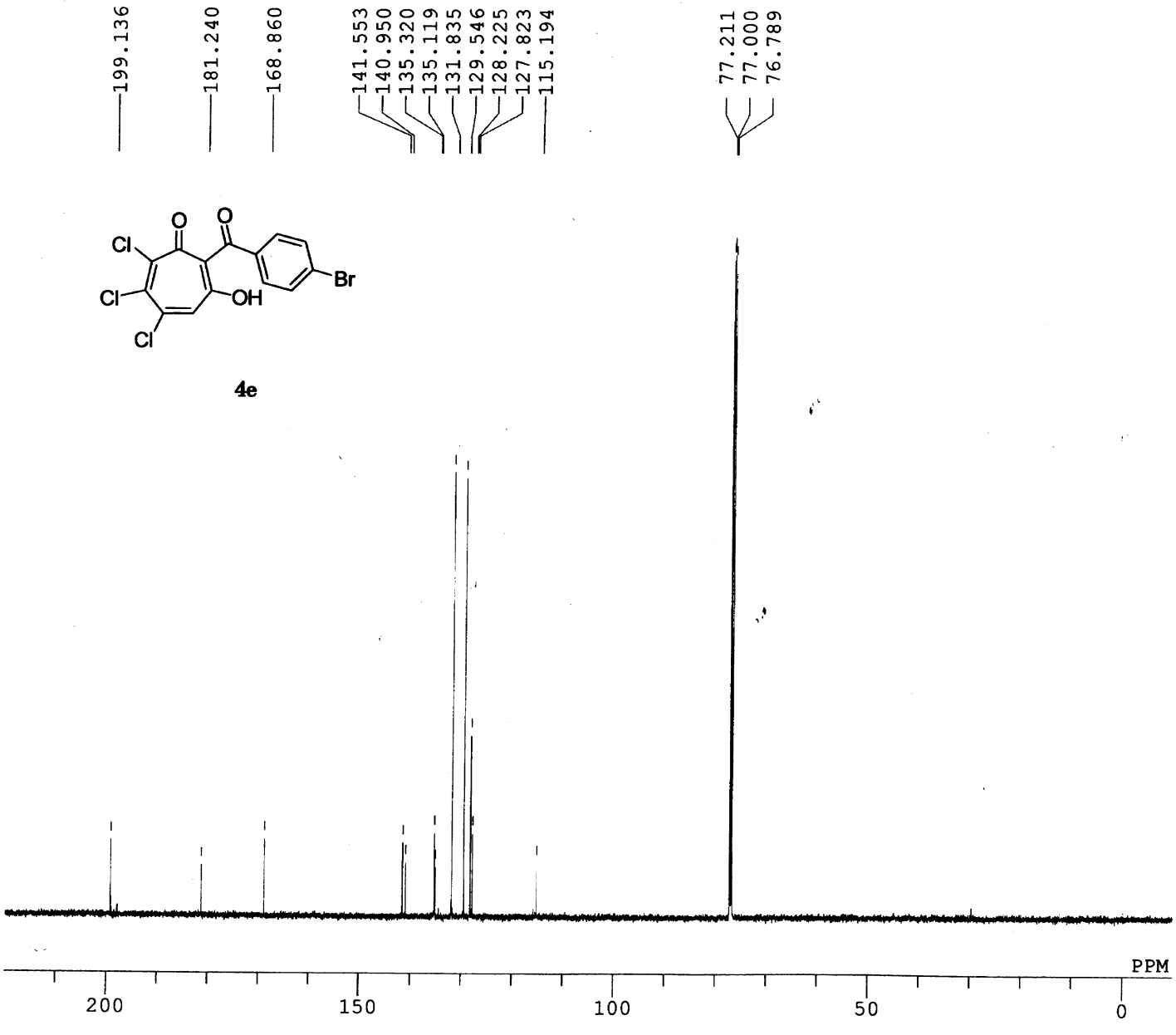
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R870-3H
DATIM	14-02-2009 14:06:41
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	32768
FREQU	11261.26 Hz
SCANS	8
ACQTM	2.9098 sec
PD	5.0000 sec
PW1	6.90 usec
IRNUC	1H
CTEMP	18.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



4e



C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R870-3C-2.jdf
lee-R870-3C



4e

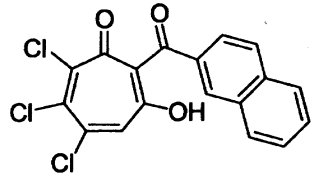
DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R870-3C
DATIM	14-02-2009 14:35:12
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	610
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	20.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R874-3H-1.jdf
lee-R874-2H

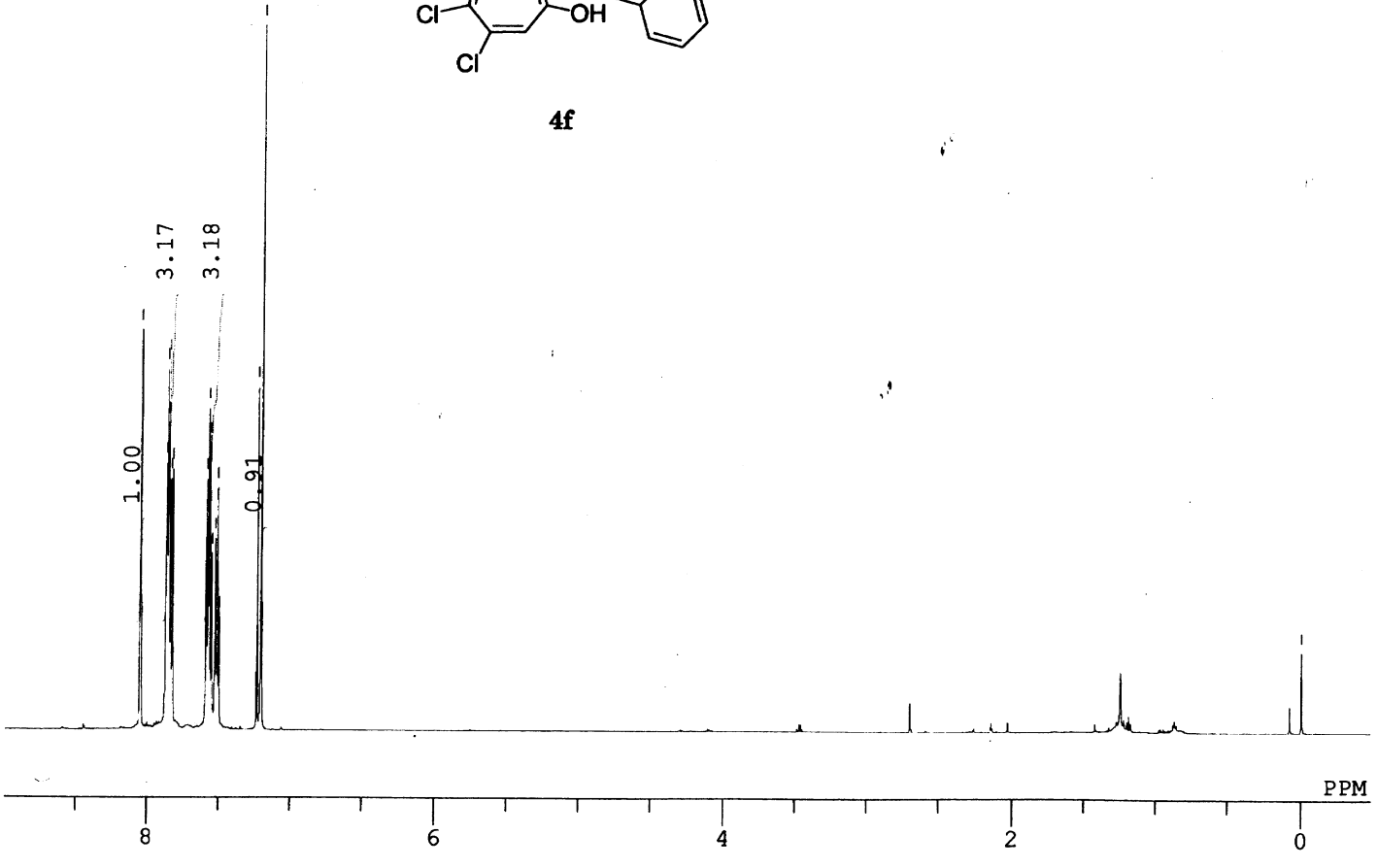
8.052 7.874 7.869 7.861 7.855 7.843 7.829 7.596 7.593 7.583 7.579 7.573 7.571 7.560 7.557 7.533 7.531 7.520 7.518 7.508 7.506 7.240 7.214

0.000

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R874-2H
DATIM	16-02-2009 16:03:12
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	600.17 MHz
OBSET	5.30 KHz
OBFIN	5.47 Hz
POINT	32768
FREQU	11261.26 Hz
SCANS	8
ACQTM	2.9098 sec
PD	5.0000 sec
PW1	6.90 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	C6D6
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



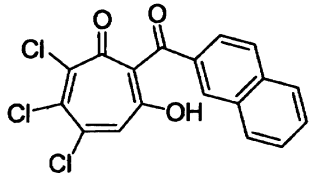
4f



54

C:\DOCUME~1\nmr\LOCALS~1\Temp\lee-R874-3C-1.jdf
lee-R874-3C

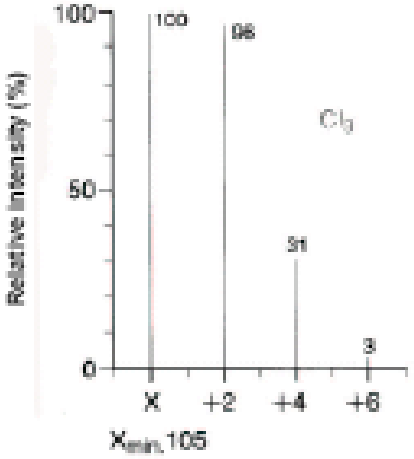
199.911
181.566
168.372
141.074
140.682
135.291
134.832
133.501
132.151
129.834
129.230
128.560
128.464
128.302
127.804
126.961
123.964
115.500
77.211
77.000
76.789



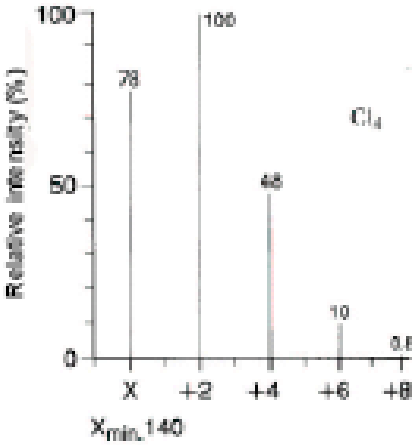
4f

DFILE	C:\DOCUME~1\nmr\LOCALS~1\Te
COMNT	lee-R874-3C
DATIM	16-02-2009 16:49:30
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.92 MHz
OBSET	8.52 KHz
OBFIN	1.74 Hz
POINT	32768
FREQU	47348.49 Hz
SCANS	1000
ACQTM	0.6921 sec
PD	2.0000 sec
PW1	4.17 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	C6D6
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

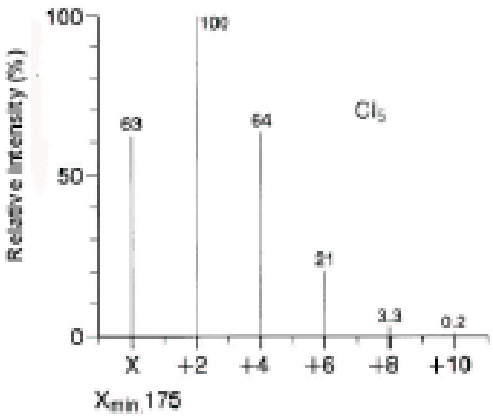
4) Standard isotopic distribution for compounds containing chlorine and bromine



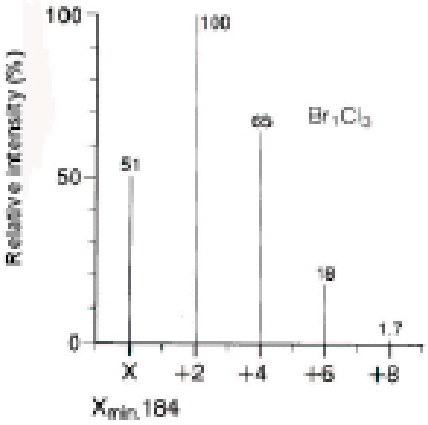
(a) Compound with Cl_3



(b) Compound with Cl_4



(c) Compound with Cl_5



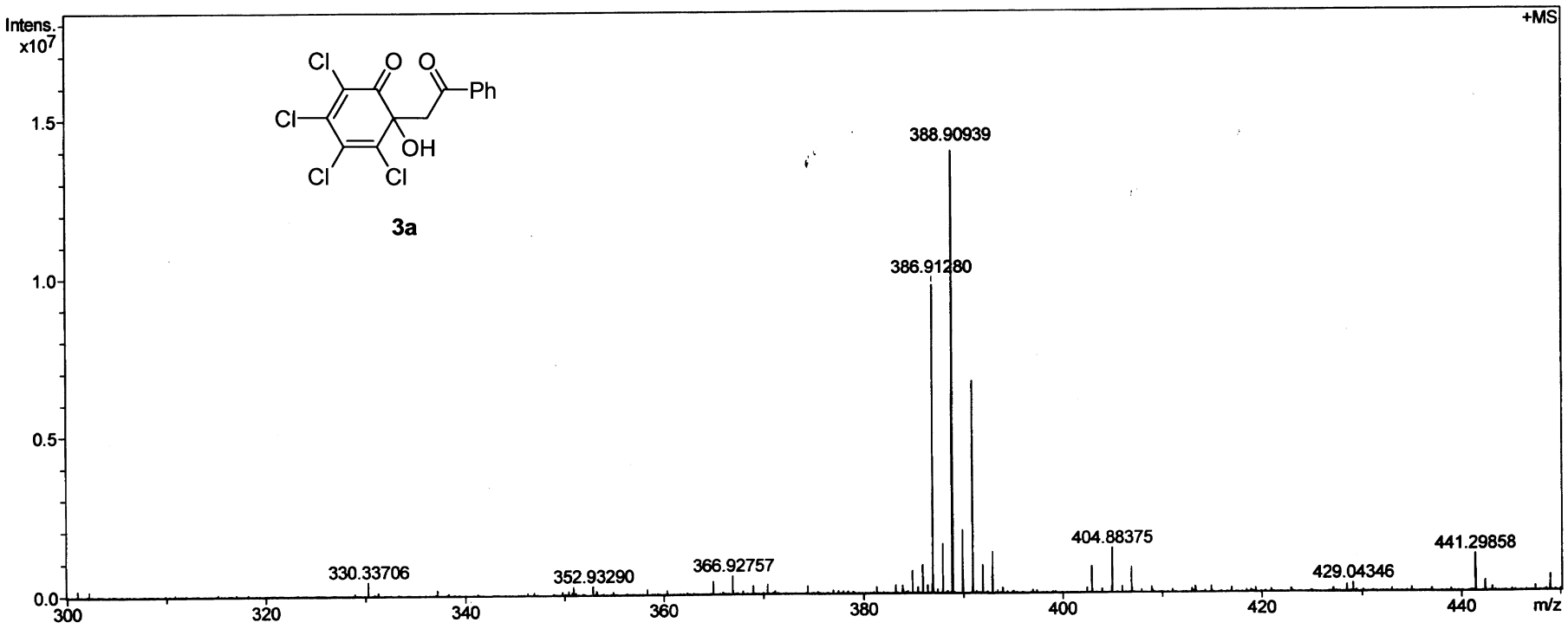
(d) Compound with Br_1Cl_3

5) Copies of the representative mass spectra

Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

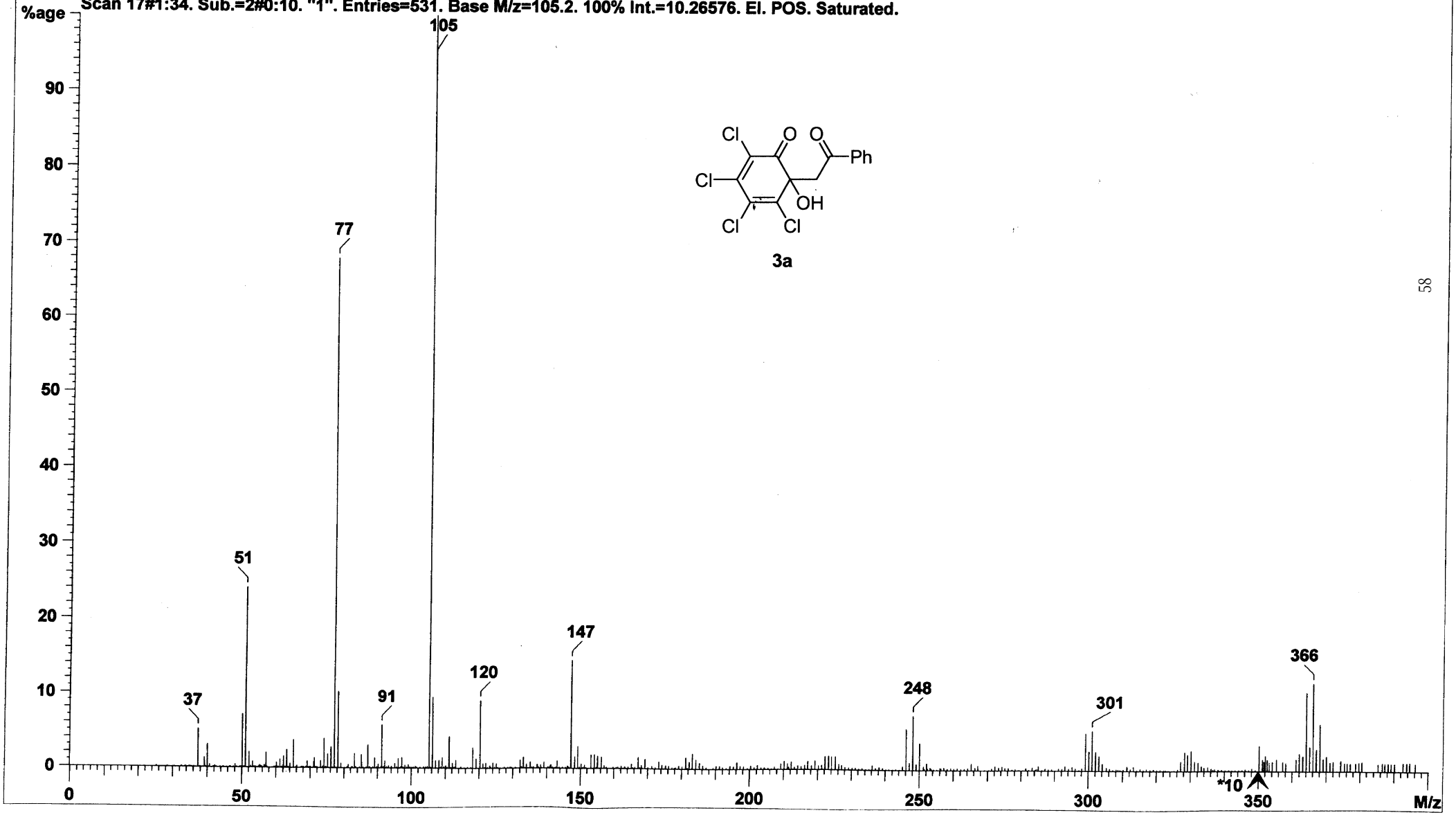
Analysis Name	81345_20081209_000001.d	Acquisition Date	12/9/2008 3:46:49 PM
Sample	1	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 8 Cl 4 Na 1 O 3	0.029	386.91198	-0.63	-0.92	-0.24	8.50	ok	even

File Name	:	j:\maspec2\data\81444.ms2
Creation Date/Time	:	08-12-10 at 17:50:00
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碘, Min.Hgt:1000碘, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 17#1:34. Sub.=2#0:10. "1". Entries=531. Base M/z=105.2. 100% Int.=10.26576. EI. POS. Saturated.



3a

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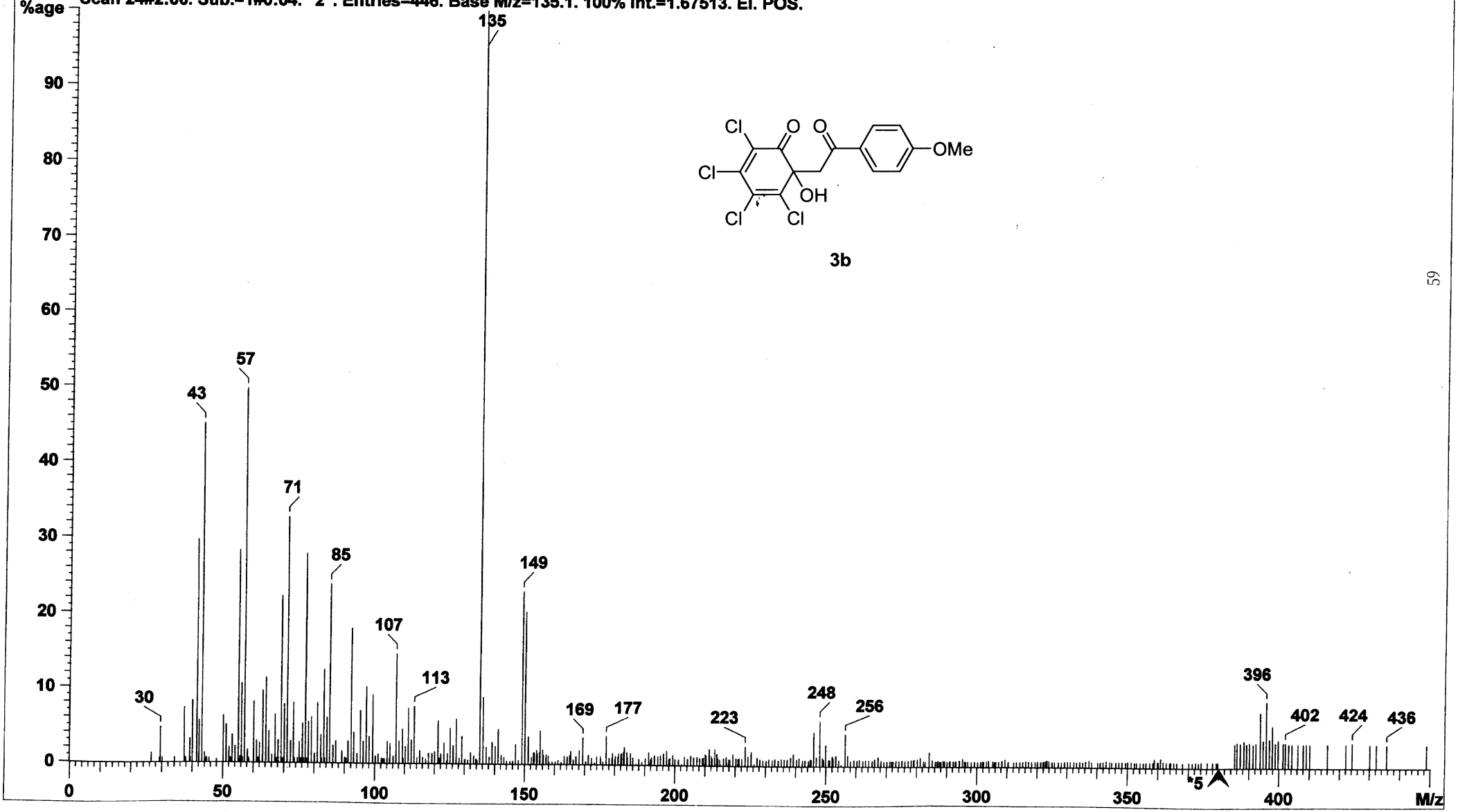
File Name	:	j:\maspec2\data\81466.ms2
Creation Date/Time	:	08-12-15 at 8:38:03
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000磺, Min.Hgt:1000磺, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 24#2:06. Sub.=1#0:04. "2". Entries=446. Base M/z=135.1. 100% Int.=1.67513. EI. POS.



3b

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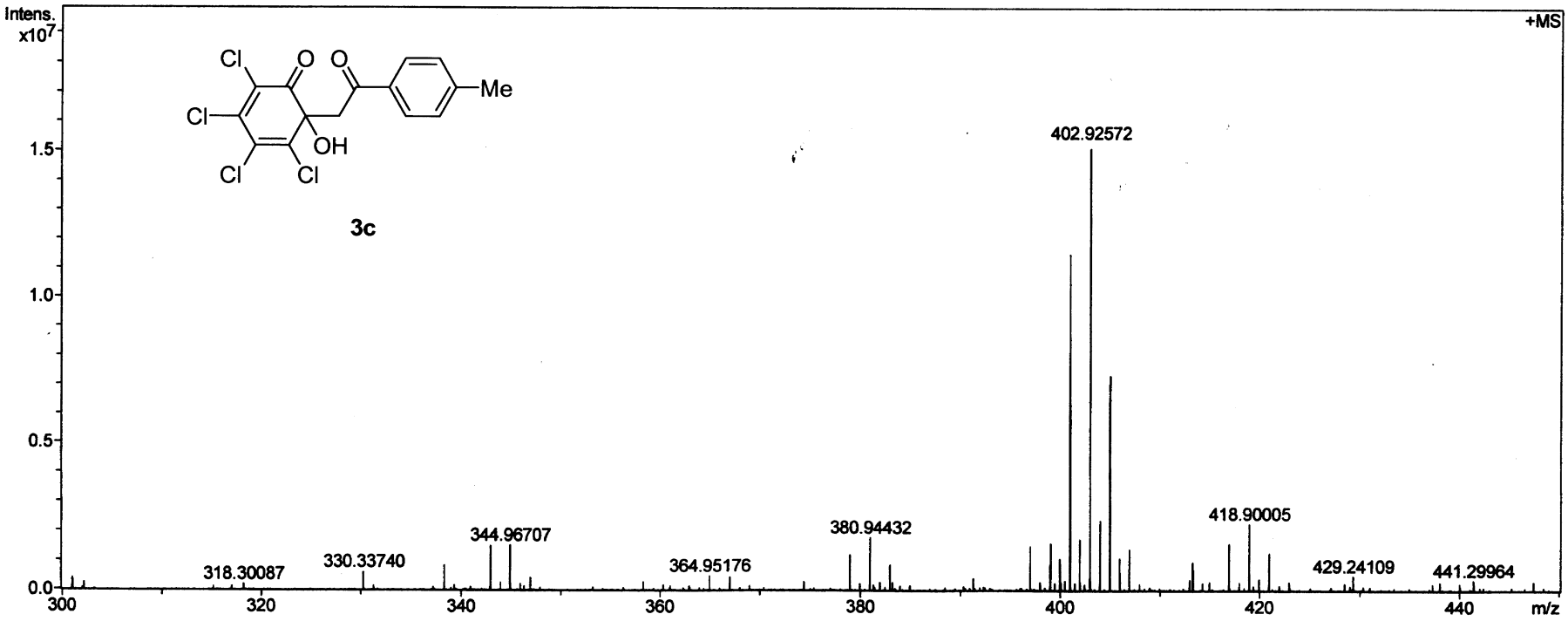
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81347_20081209_000001.d	Acquisition Date	12/9/2008 4:04:45 PM
Sample	5	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

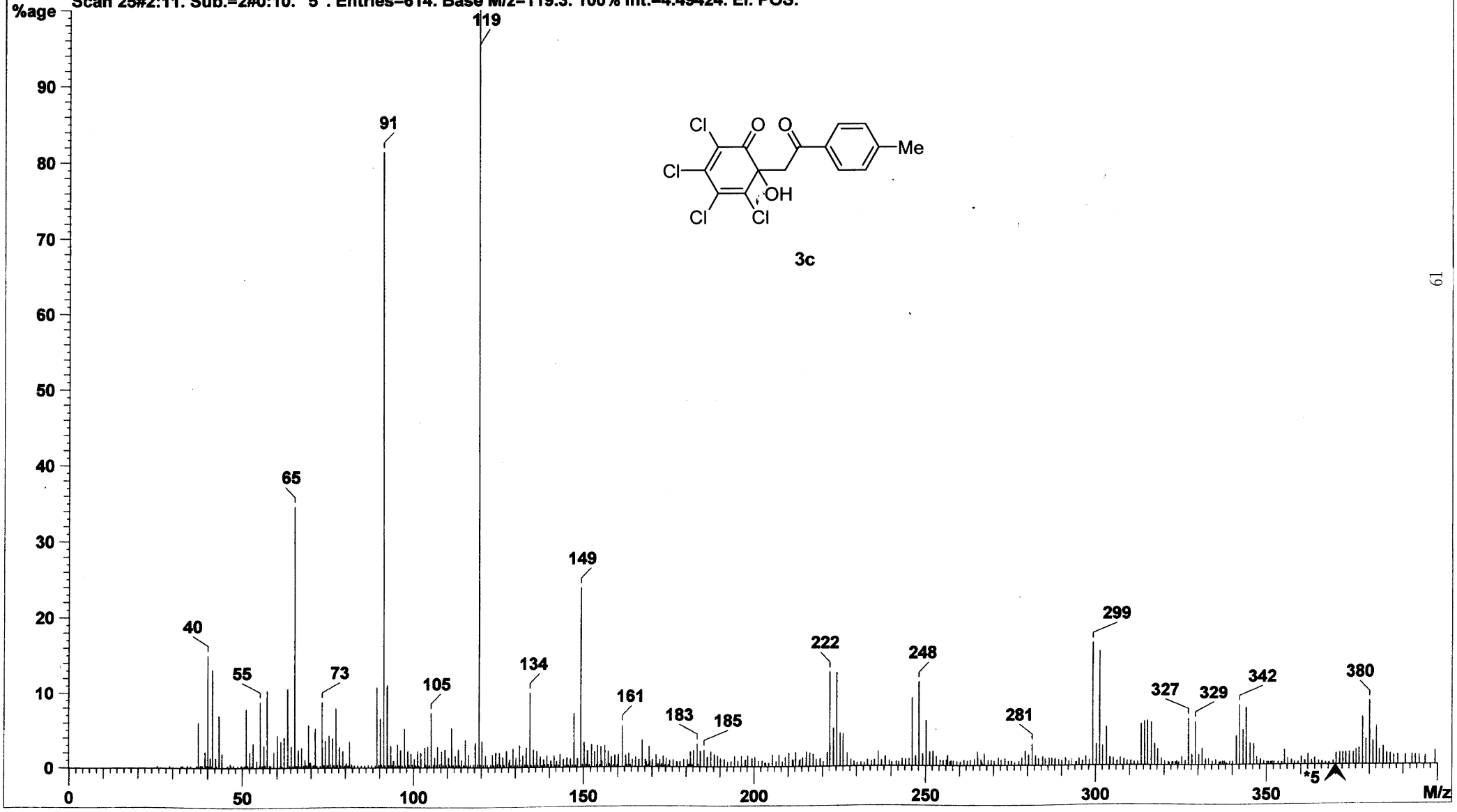


3c

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 15 H 10 Cl 4 Na 1 O 3	0.008	400.92763	-2.27	-2.18	-0.91	8.50	ok	even

File Name	:	j:\maspec2\data\81446.ms2
Creation Date/Time	:	08-12-10 at 18:06:37
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 25#2:11. Sub.=2#0:10. "5". Entries=614. Base M/z=119.3. 100% Int.=4.49424. El. POS.

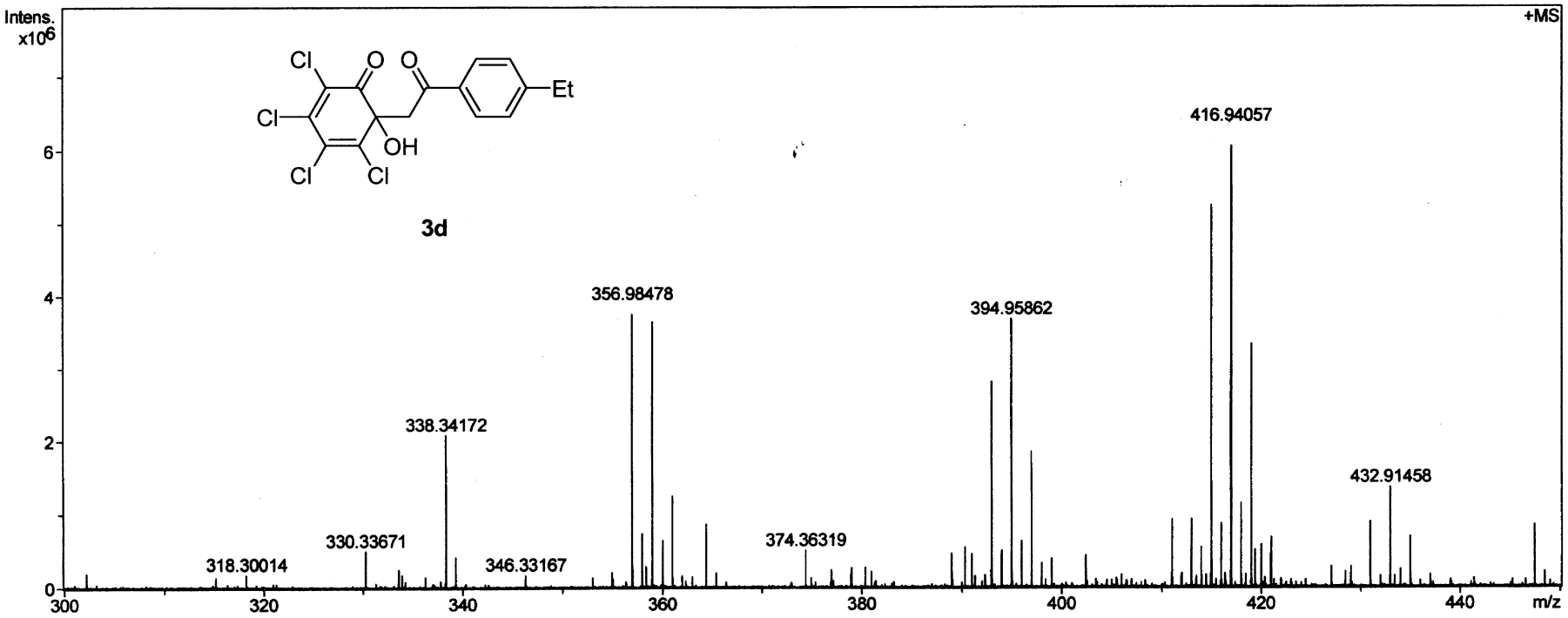


3c

Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

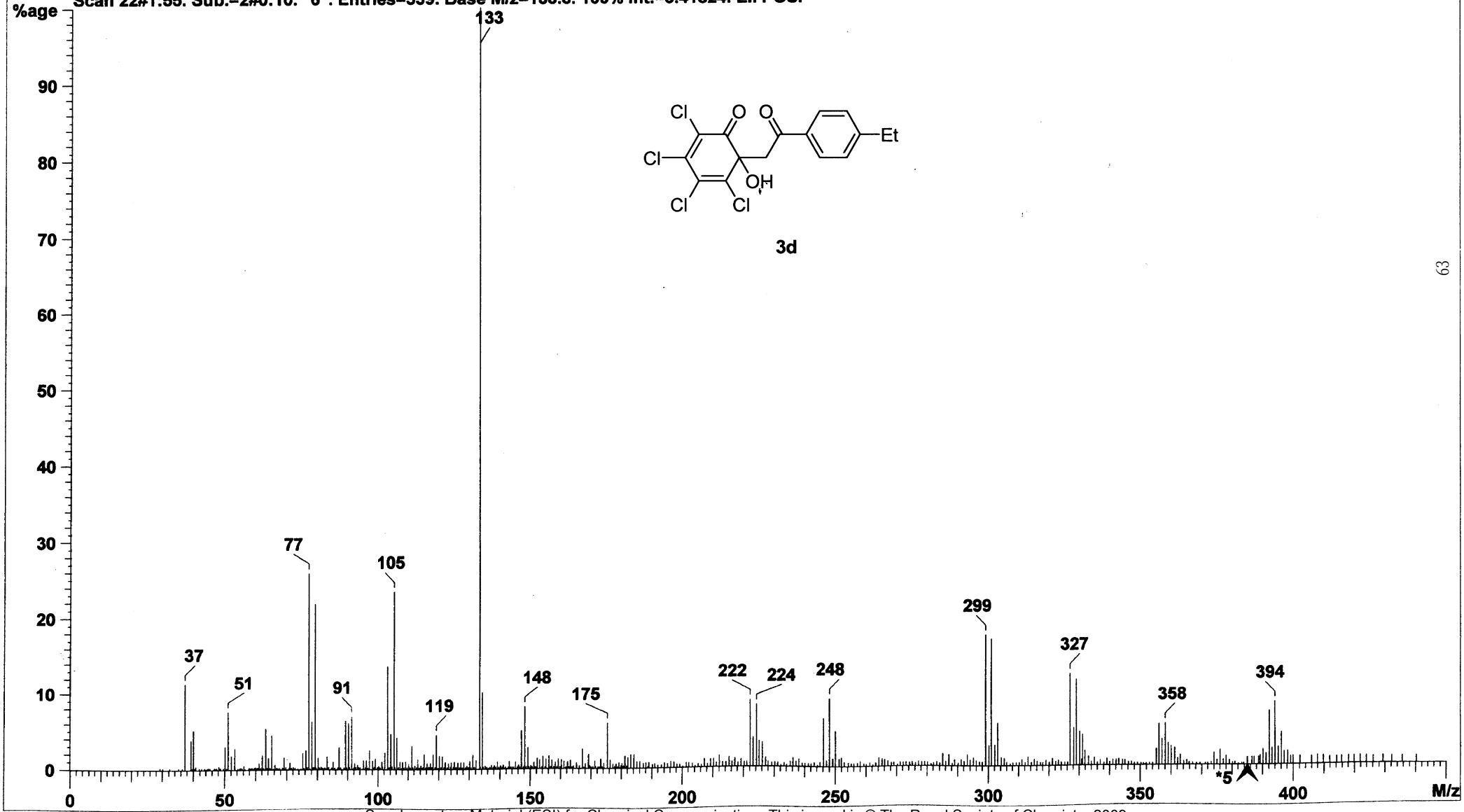
Analysis Name	81348_20081209_000001.d	Acquisition Date	12/9/2008 4:07:01 PM
Sample	6	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 16 H 12 Cl 4 Na 1 O 3	0.029	414.94328	-0.24	-0.18	-0.10	8.50	ok	even

File Name	:	j:\maspec2\data\81447.ms2
Creation Date/Time	:	08-12-10 at 18:14:43
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 22#1:55. Sub.=2#0:10. "6". Entries=539. Base M/z=133.3. 100% Int.=5.41824. EI. POS.



3d

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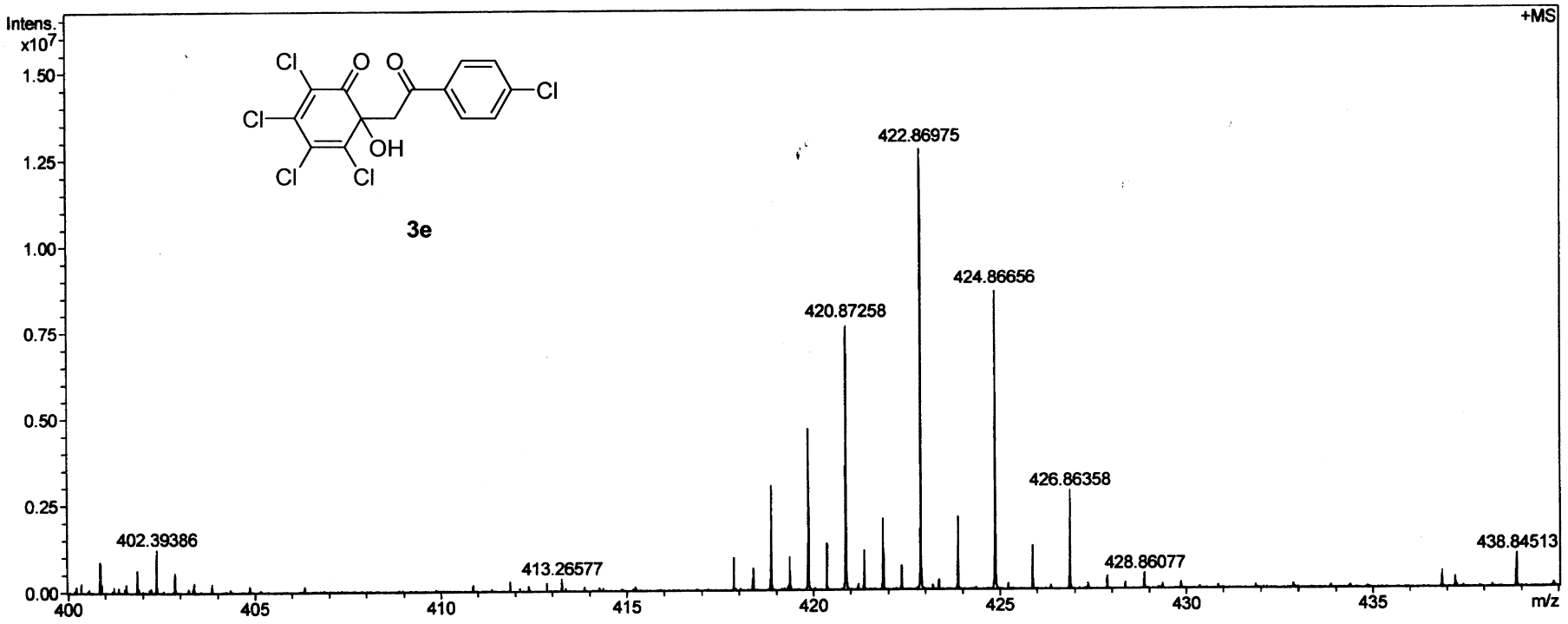
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81367_20081210_000001.d	Acquisition Date	12/10/2008 3:33:10 PM
Sample	3	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

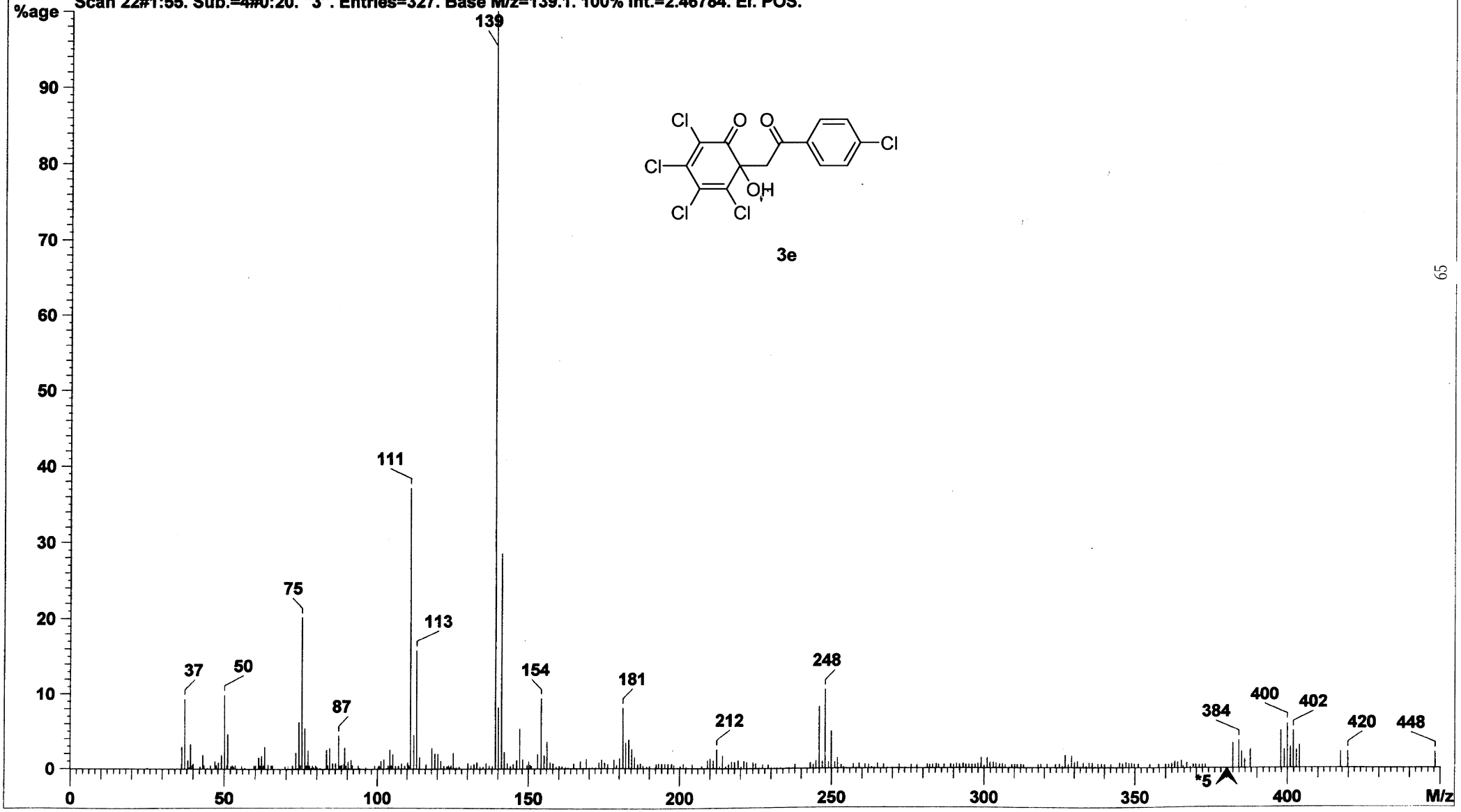


Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 7 Cl 5 Na 1 O 3	0.012	420.87300	1.00	1.32	0.42	8.50	ok	even

Bruker Daltonics DataAnalysis 3.4 printed: 12/10/2008 3:35:24 PM Page 1 of 1

File Name	:	j:\maspec2\data\81453.ms2
Creation Date/Time	:	08-12-12 at 8:35:17
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 22#1:55. Sub.=4#0:20. "3". Entries=327. Base M/z=139.1. 100% Int.=2.46784. EI. POS.



3e

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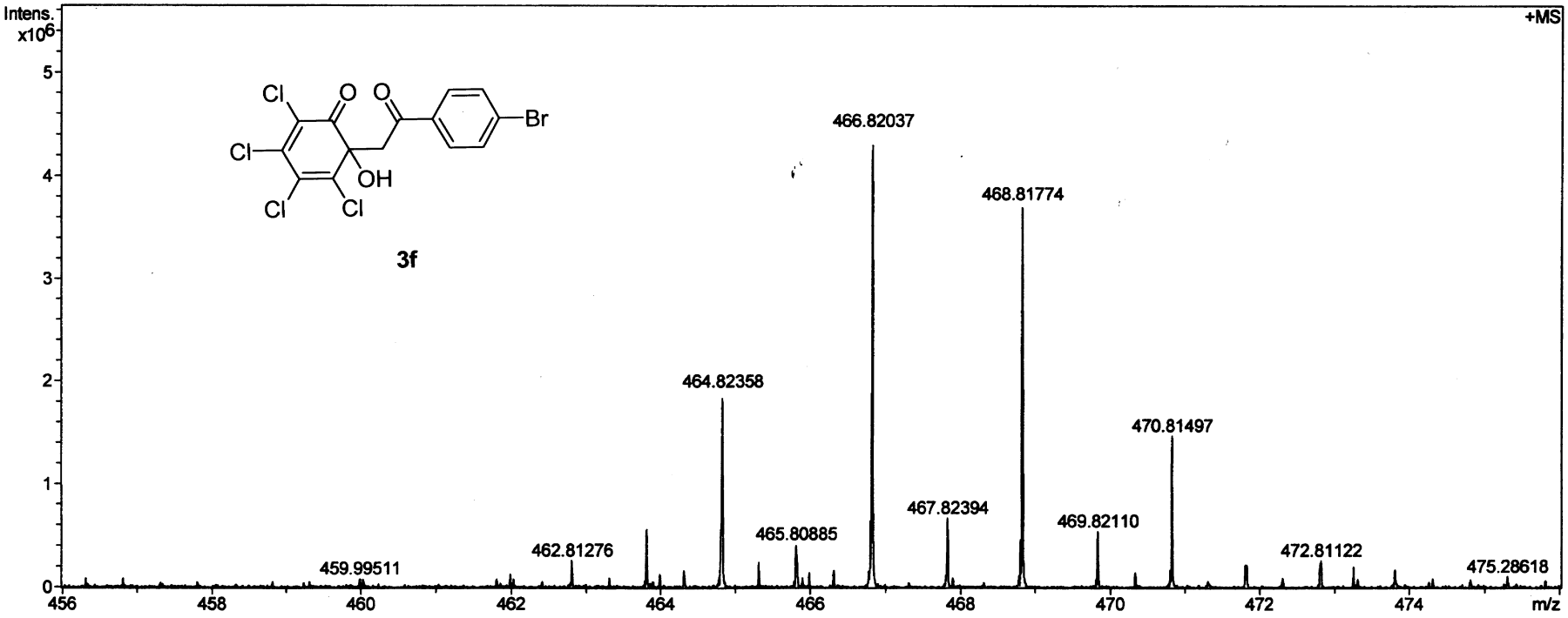
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81426_20081216_000001.d	Acquisition Date	12/16/2008 4:44:23 PM
Sample	4	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

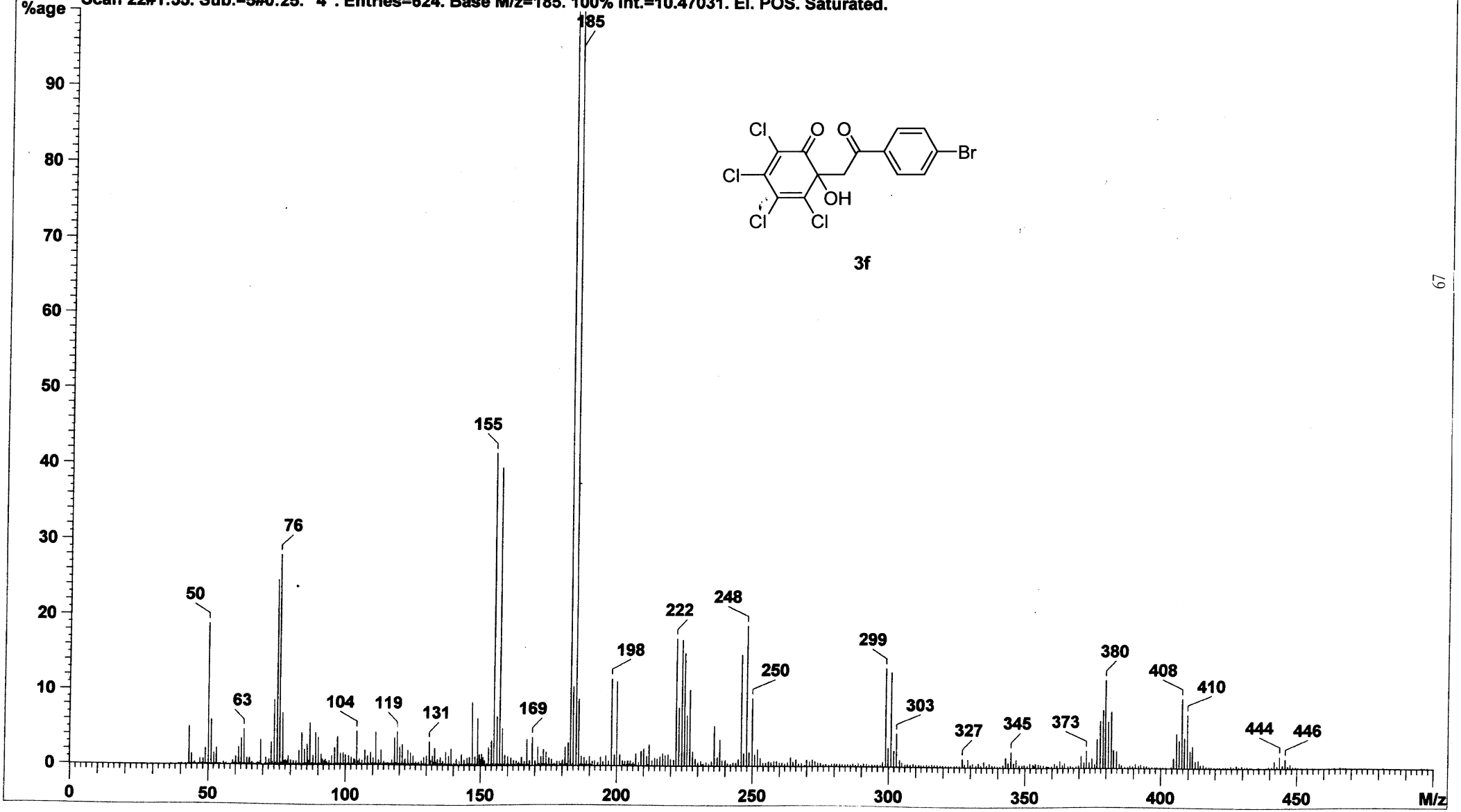


Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 7 Br 1 Cl 4 Na 1 O 3	0.011	464.82249	-2.36	0.18	-1.09	8.50	ok	even
C 16 H 6 Br 1 Cl 4 O 3	0.027	464.82489	2.82	4.22	1.31	11.50	ok	even

Bruker Daltonics DataAnalysis 3.4 printed: 12/16/2008 4:46:59 PM Page 1 of 1

File Name	:	j:\maspec2\data\81478.ms2
Creation Date/Time	:	08-12-17 at 9:44:24
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000磧, Min.Hgt:1000磧, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 22#1:55. Sub.=5#0:25. "4". Entries=624. Base M/z=185. 100% Int.=10.47031. EI. POS. Saturated.



3f

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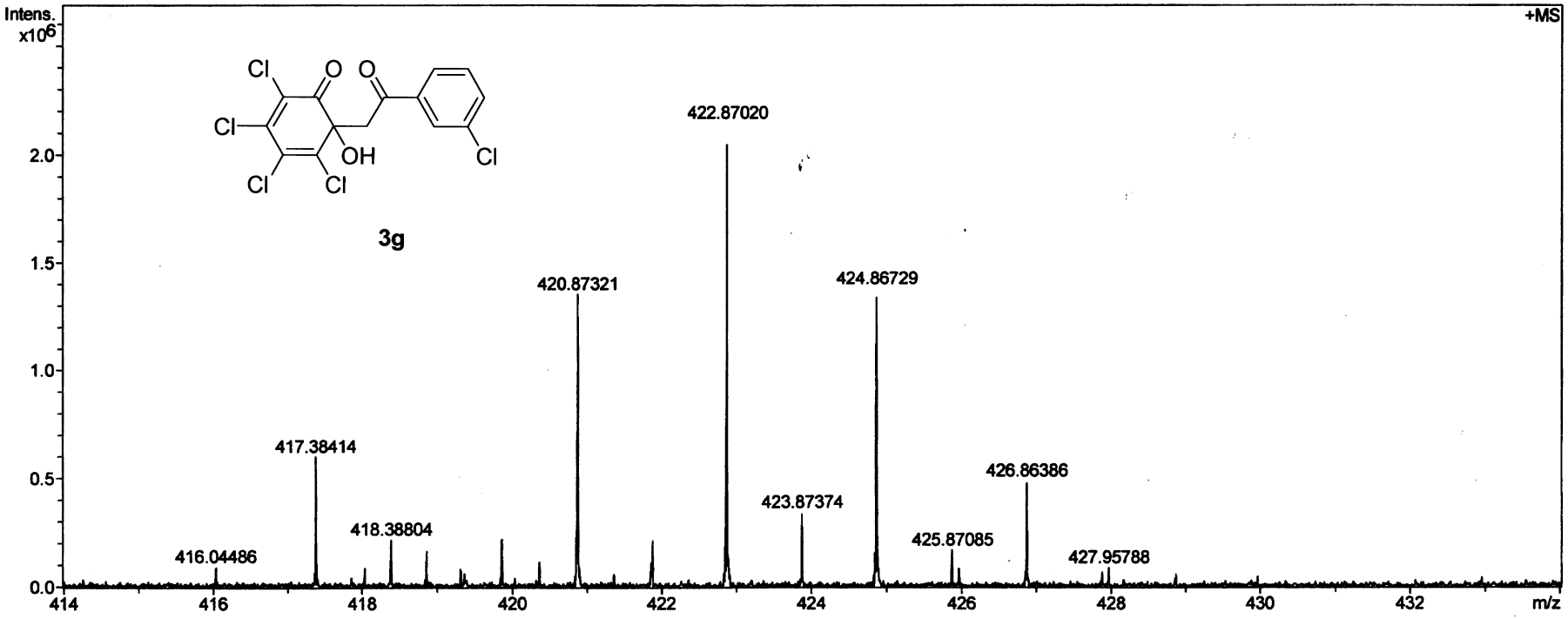
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81428_20081216_000001.d	Acquisition Date	12/16/2008 4:54:41 PM
Sample	18	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

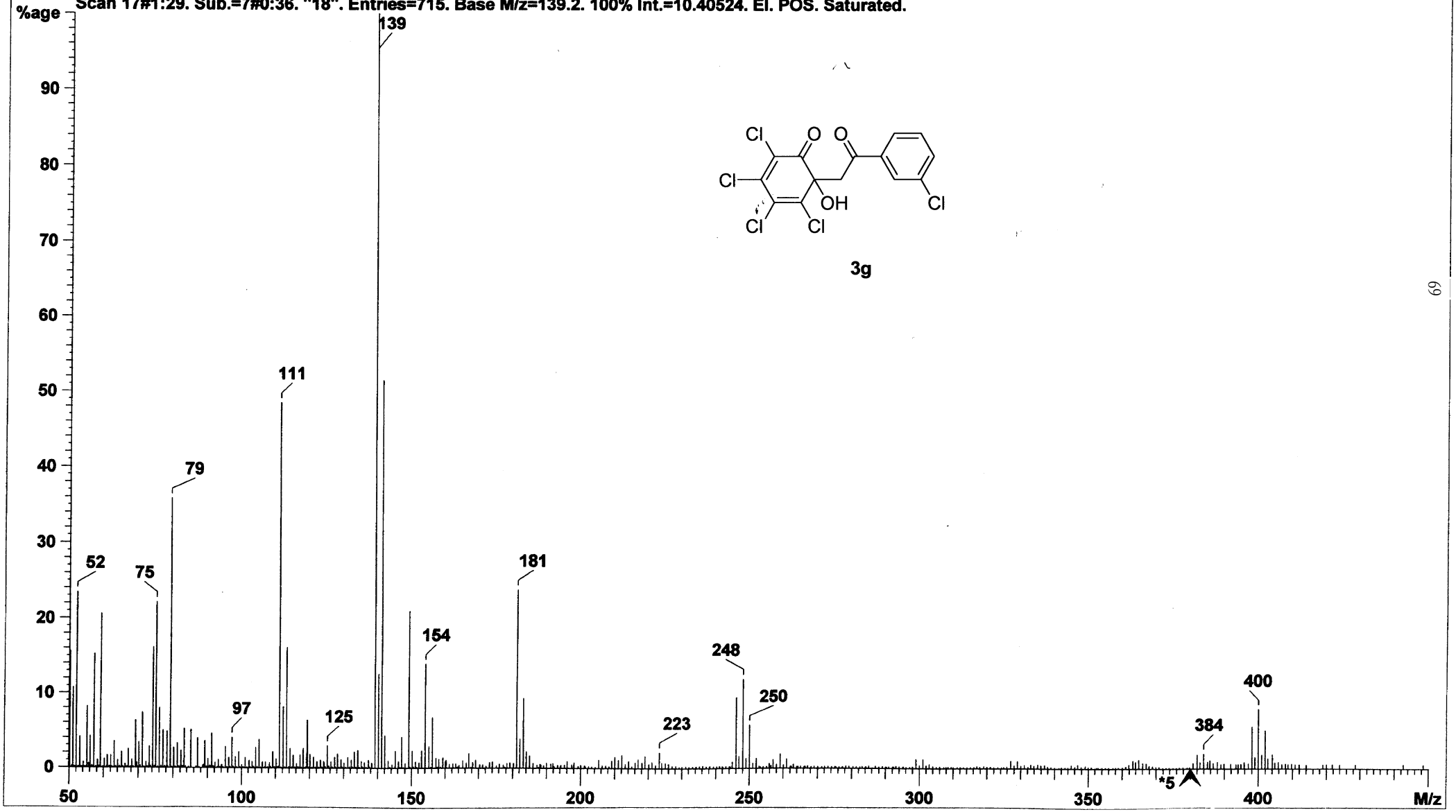


Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 7 Cl 5 Na 1 O 3	0.010	420.87300	-0.49	-0.04	-0.21	8.50	ok	even

Bruker Daltonics DataAnalysis 3.4 printed: 12/16/2008 4:56:07 PM Page 1 of 1

File Name	:	j:\maspec2\data\81480.ms2
Creation Date/Time	:	08-12-17 at 12:07:30
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碘, Min.Hgt:1000碘, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 17#1:29. Sub.=7#0:36. "18". Entries=715. Base M/z=139.2. 100% Int.=10.40524. El. POS. Saturated.



3g

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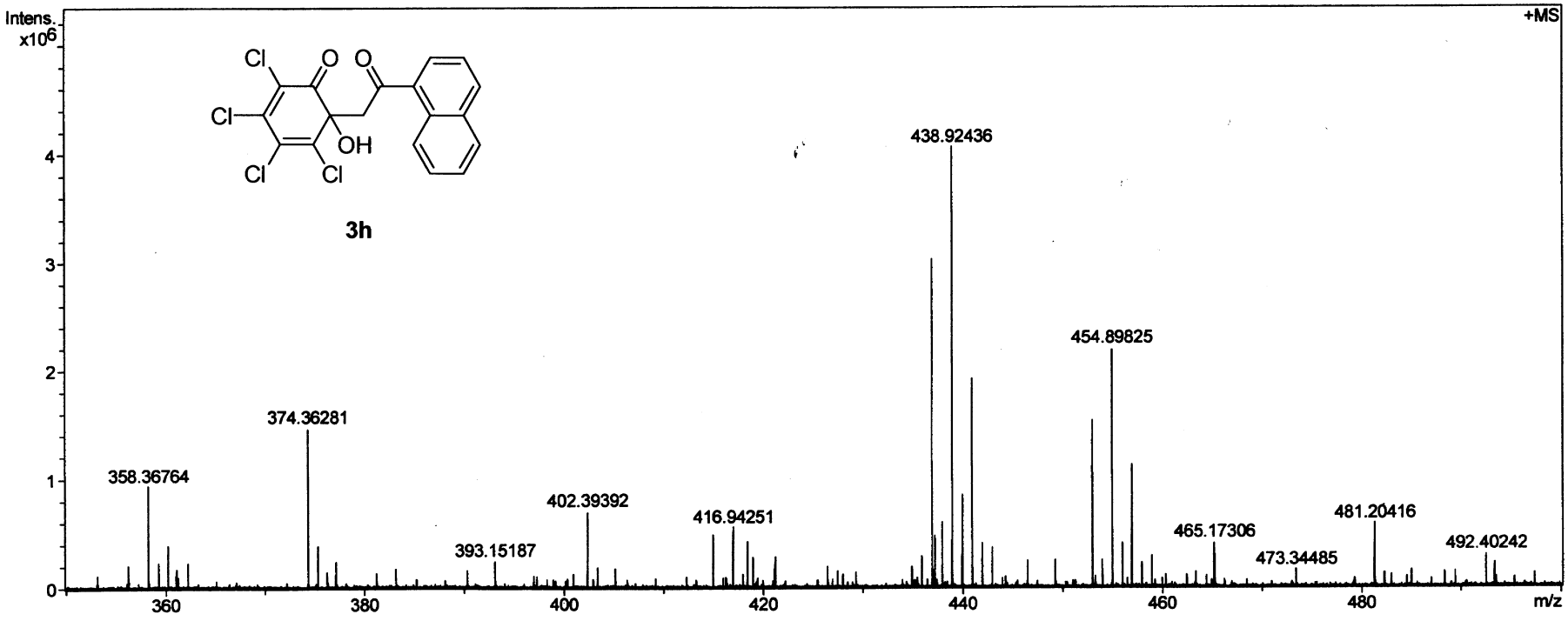
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

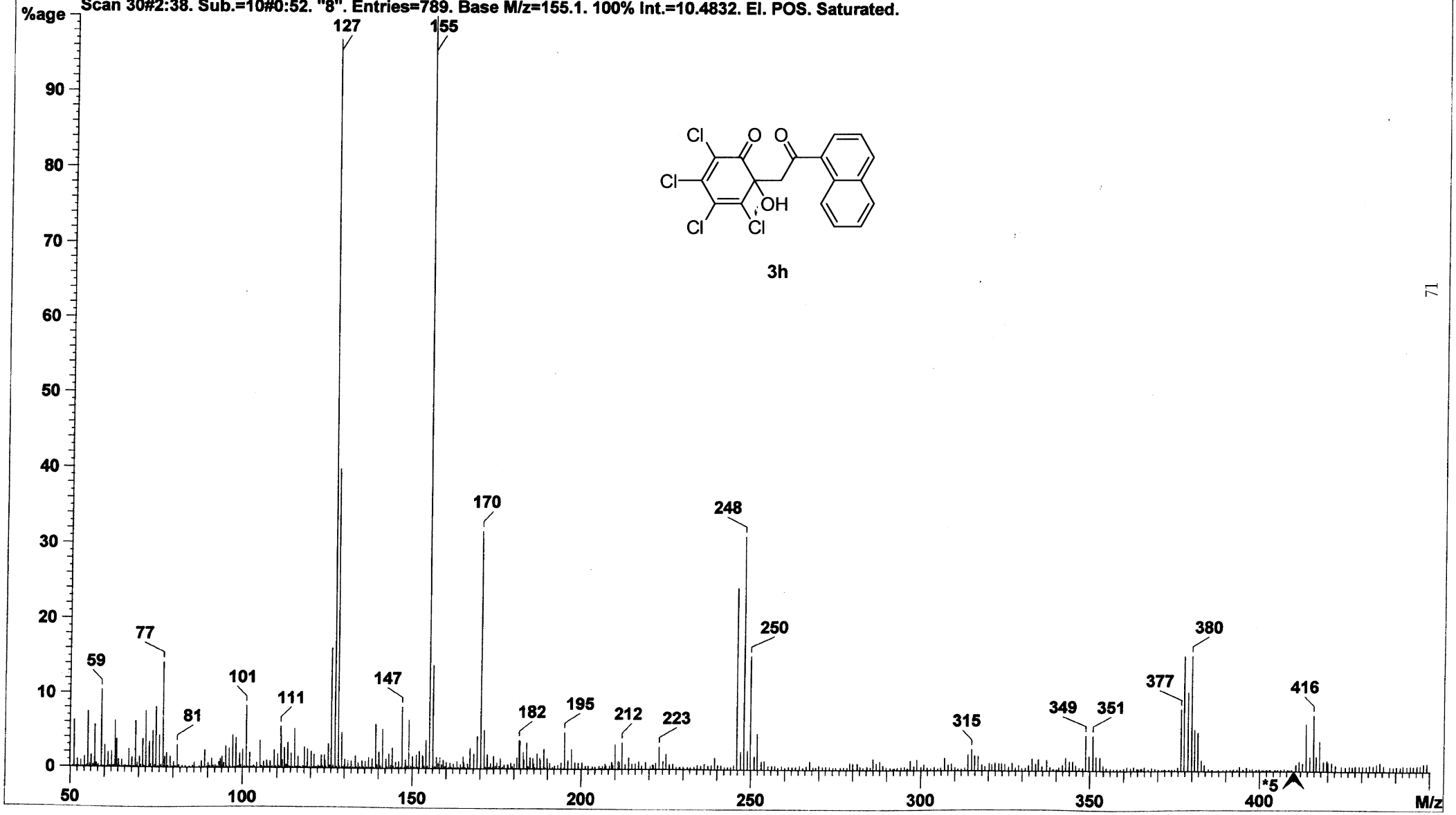
Analysis Name	81350_20081209_000001.d	Acquisition Date	12/9/2008 4:13:51 PM
Sample	8	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 18 H 10 Cl 4 Na 1 O 3	0.011	436.92763	1.10	0.81	0.48	11.50	ok	even

File Name	:	j:\maspec2\data\81449.ms2
Creation Date/Time	:	08-12-10 at 18:29:37
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 30#2:38. Sub.=10#0:52. "8". Entries=789. Base M/z=155.1. 100% Int.=10.4832. El. POS. Saturated.



3h

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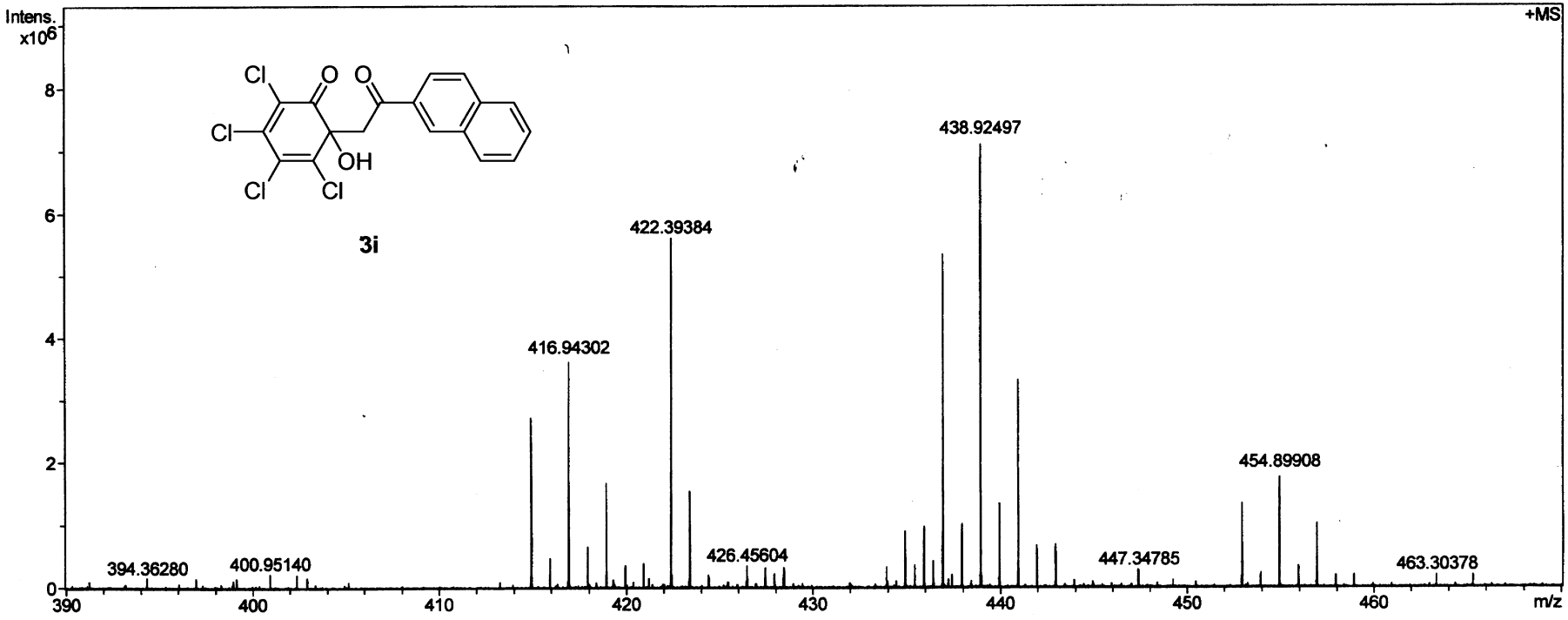
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

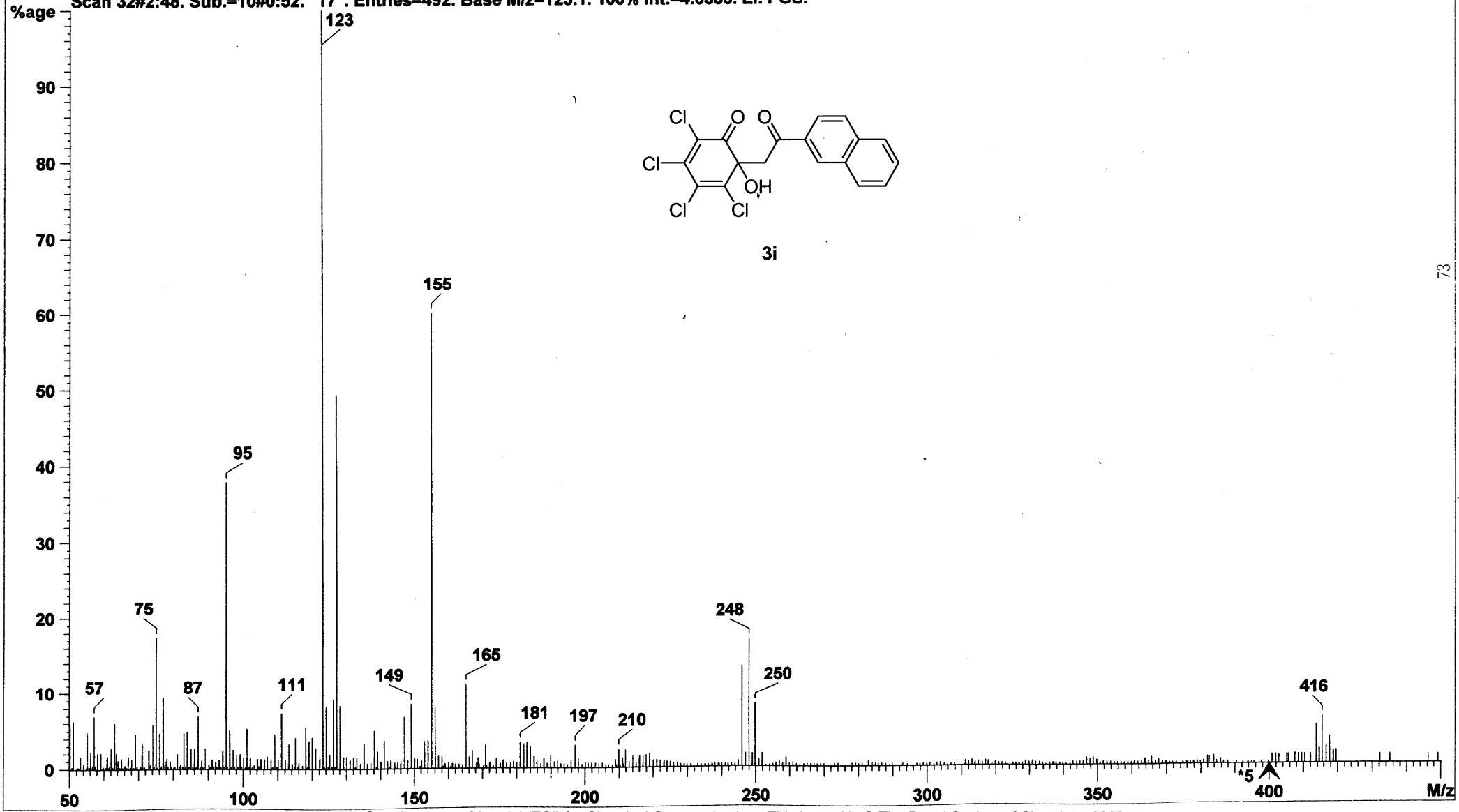
Analysis Name	81396_20081212_000001.d	Acquisition Date	12/12/2008 3:22:26 PM
Sample	17	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 18 H 11 Cl 4 O 3	0.012	414.94568	-0.29	-0.29	-0.12	11.50	ok	even

File Name	:	j:\maspec2\data\81468.ms2
Creation Date/Time	:	08-12-15 at 8:53:24
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 32#2:48. Sub.=10#0:52. "17". Entries=492. Base M/z=123.1. 100% Int.=4.6336. EI. POS.

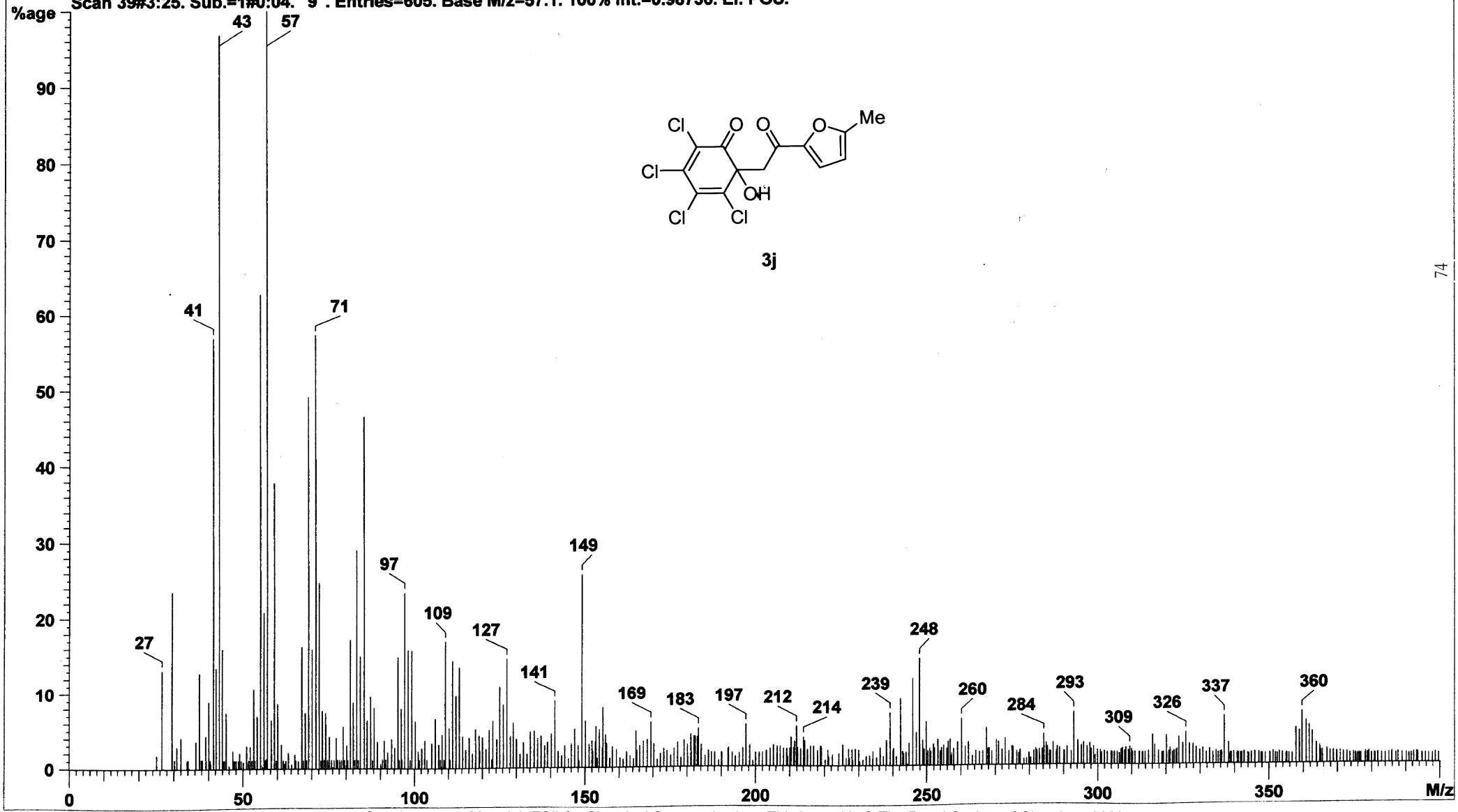


3i

73

File Name	:	j:\maspec2\data\81454.ms2
Creation Date/Time	:	08-12-12 at 8:49:33
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 39#3:25. Sub.=1#0:04. "9". Entries=605. Base M/z=57.1. 100% Int.=0.98736. El. POS.

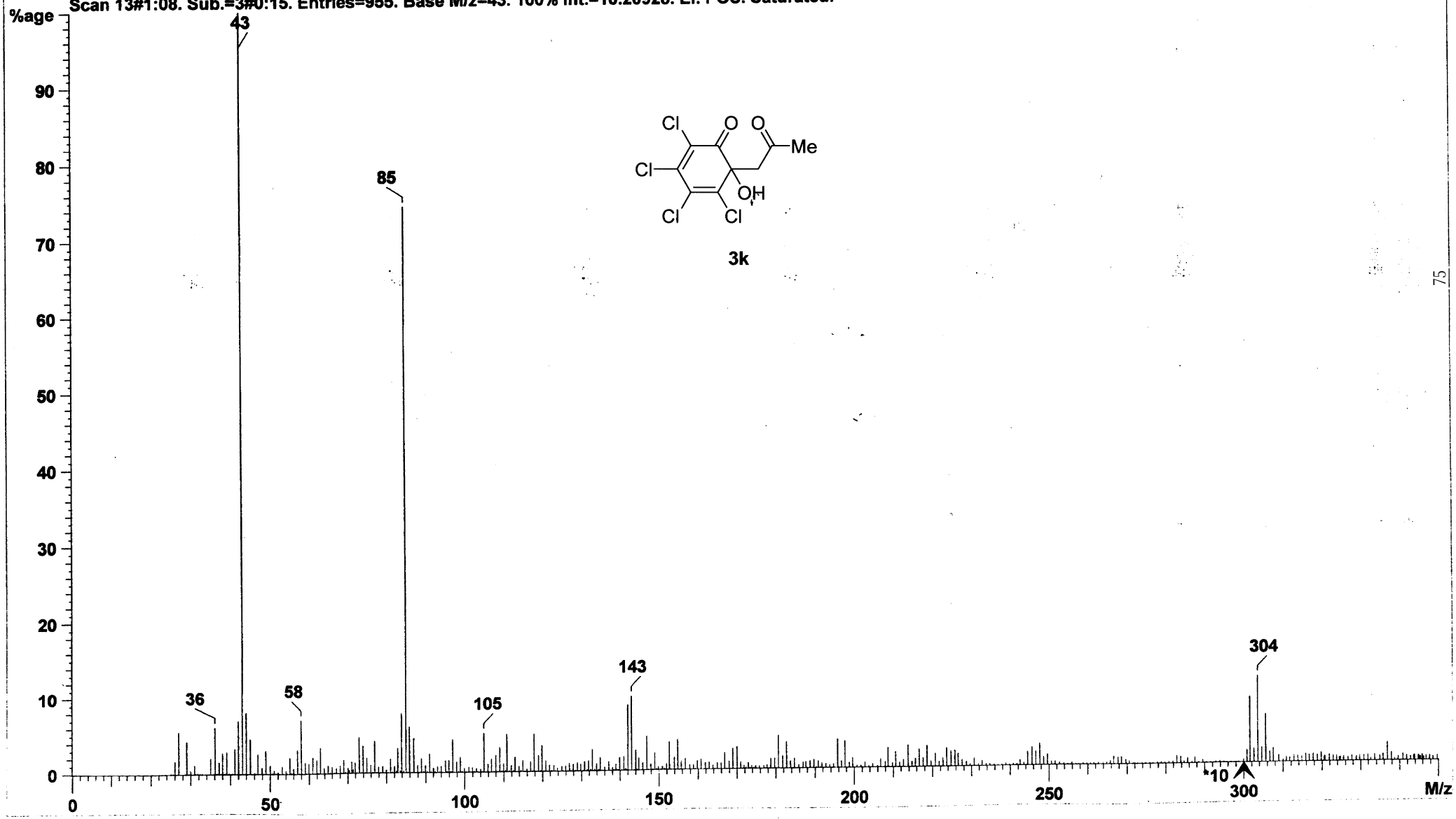


3j

74

File Name	:	e:\maspec2\data\73467.ms2
Creation Date/Time	:	08-1-4 at 14:58:56
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

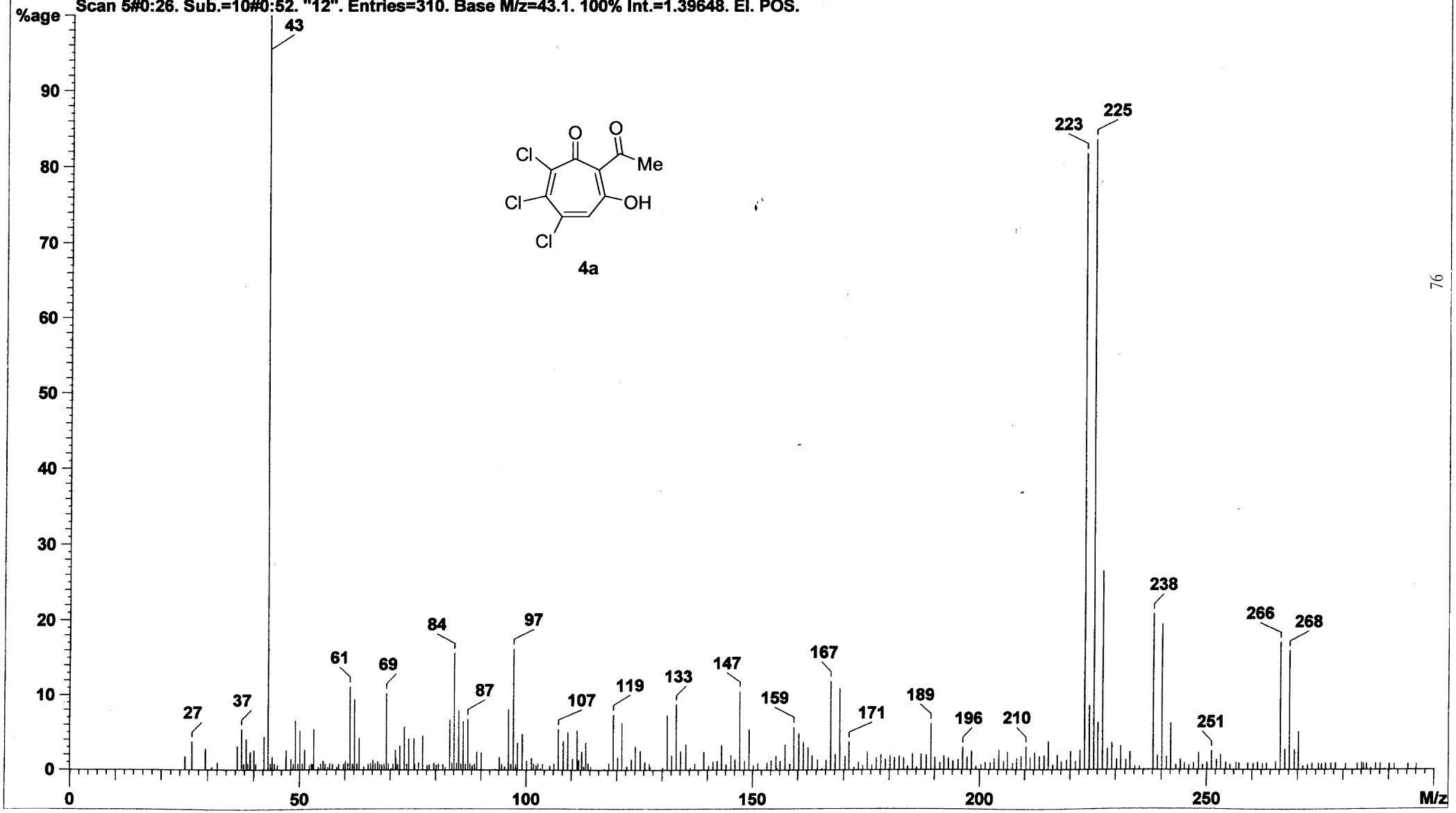
SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 13#1:08. Sub.=3#0:15. Entries=955. Base M/z=43. 100% Int.=10.20928. EI. POS. Saturated.



3k

File Name	:	j:\maspec2\data\81455.ms2
Creation Date/Time	:	08-12-12 at 9:14:41
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碳, Min.Hgt:1000碳, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 5#0:26. Sub.=10#0:52. "12". Entries=310. Base M/z=43.1. 100% Int.=1.39648. EI. POS.



4a

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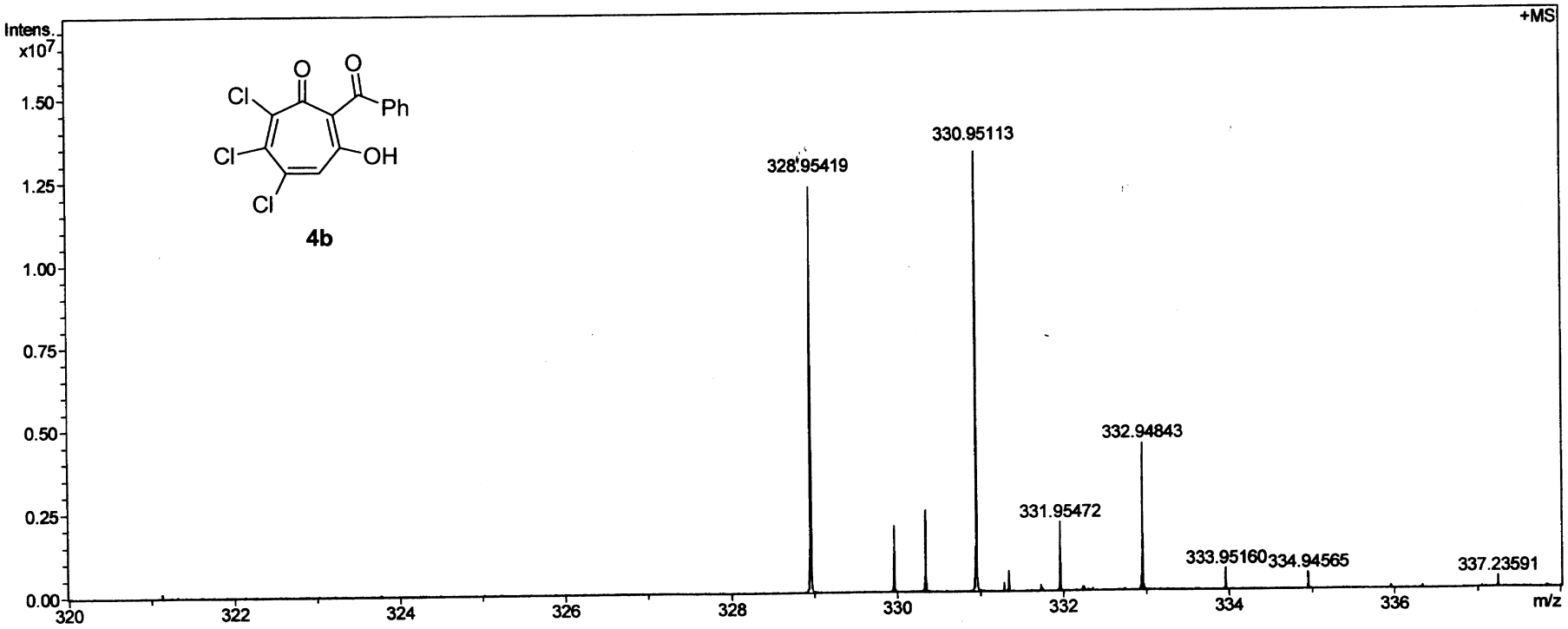
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81370_20081210_000001.d	Acquisition Date	12/10/2008 4:34:01 PM
Sample	13	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

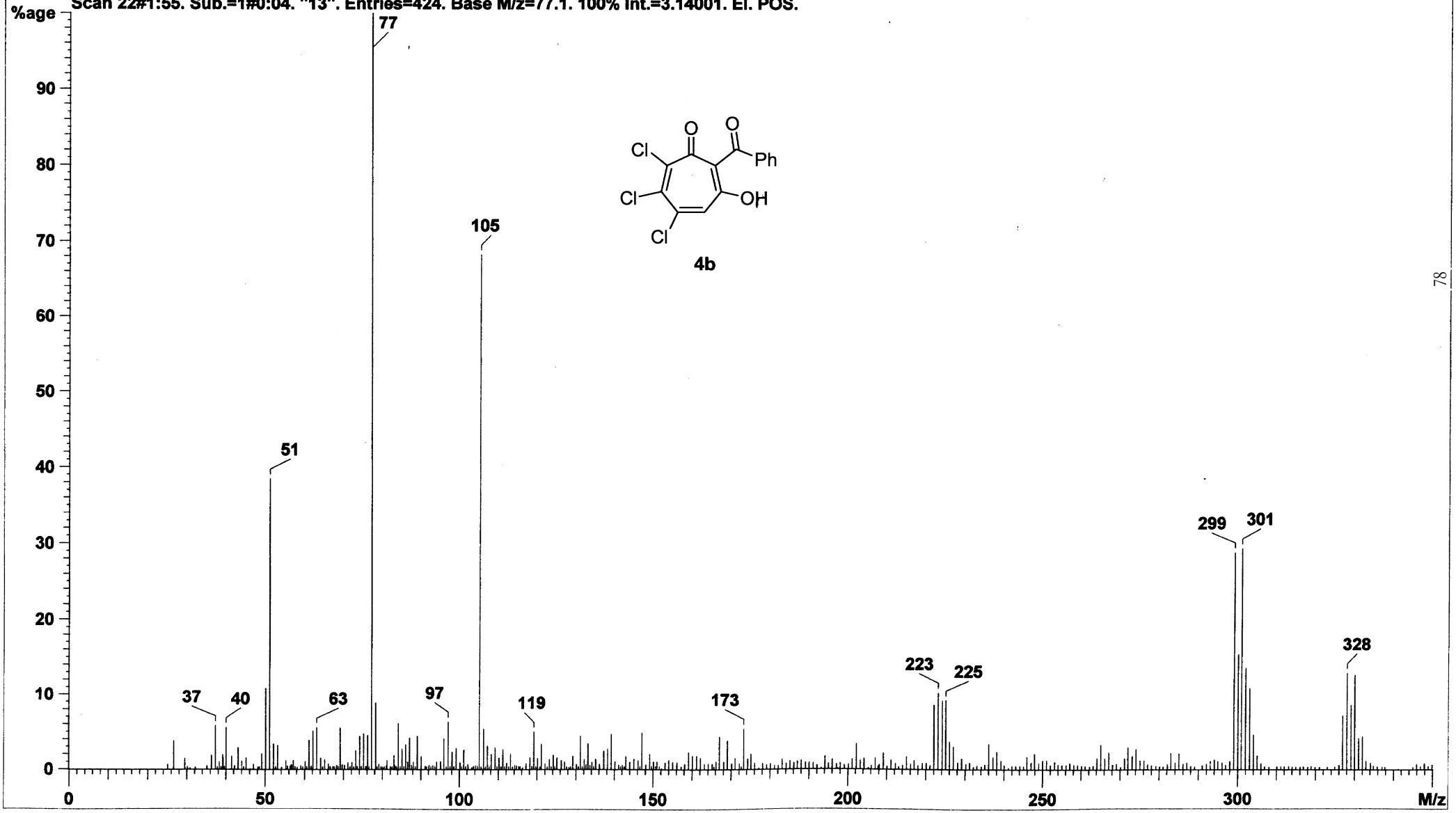


4b

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 8 Cl 3 O 3	0.027	328.95335	-2.53	-2.05	-0.83	9.50	ok	even
C 20 H 3 Cl 2 O 1	0.167	328.95555	4.13	5.10	1.36	18.50	ok	even

File Name	:	j:\maspec2\data\81456.ms2
Creation Date/Time	:	08-12-12 at 9:17:51
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

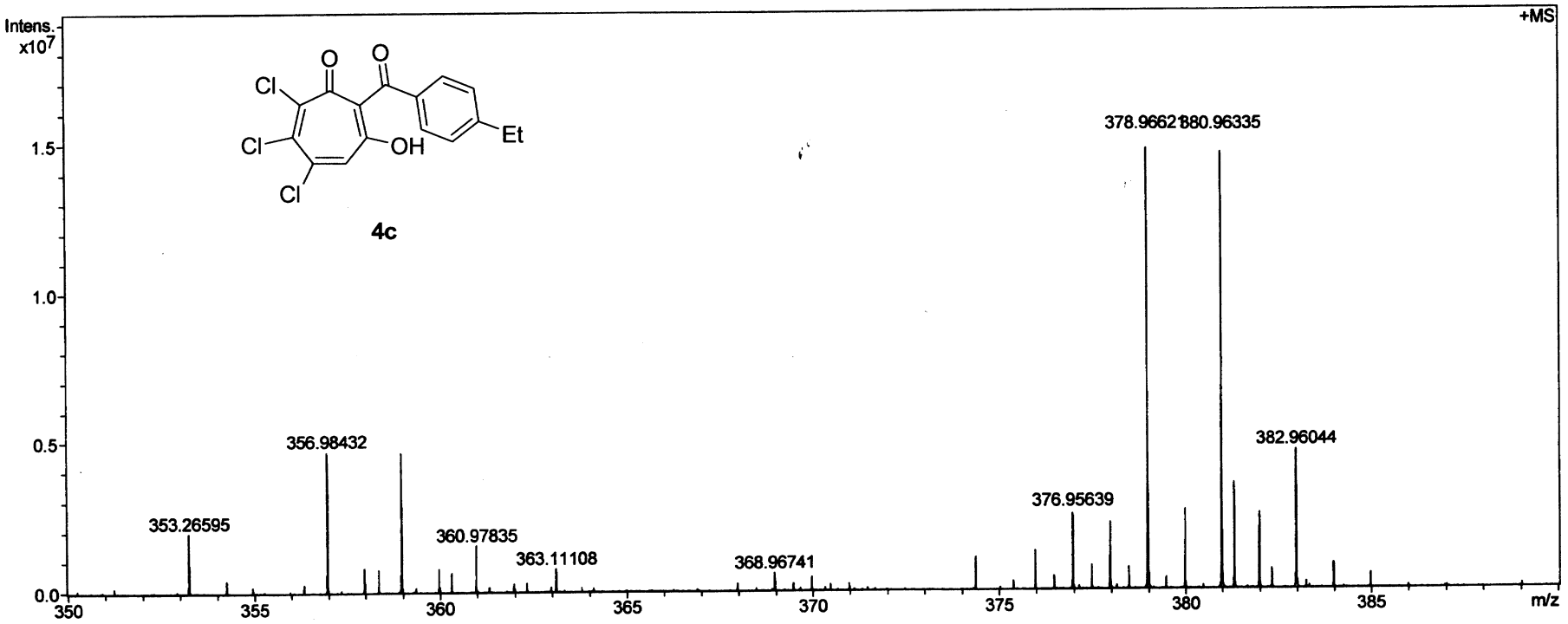
SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 22#1:55. Sub.=1#0:04. "13". Entries=424. Base M/z=77.1. 100% Int.=3.14001. El. POS.



Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

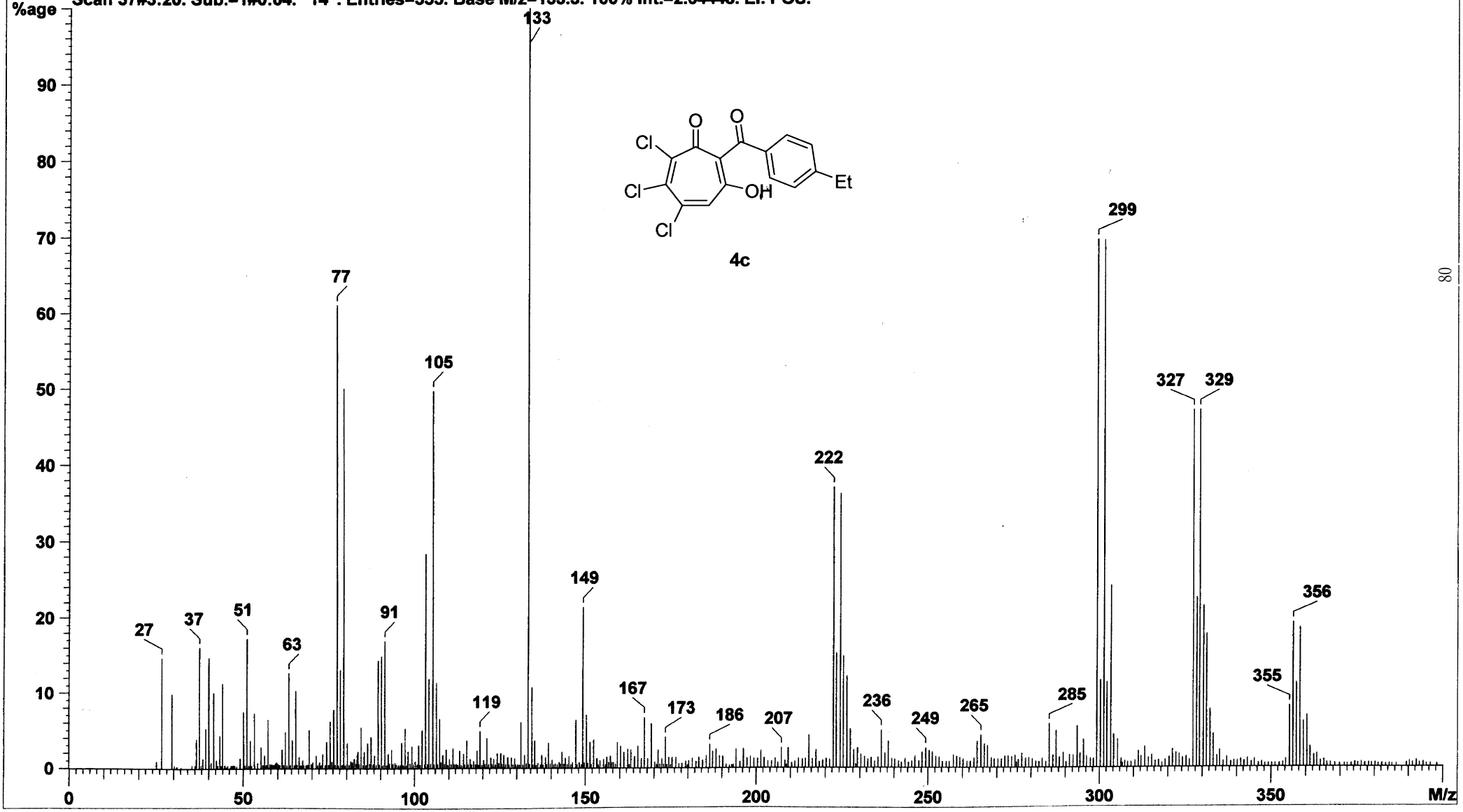
Analysis Name	81371_20081210_000004.d	Acquisition Date	12/10/2008 4:58:49 PM
Sample	14	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 16 H 12 Cl 3 O 3	0.004	356.98465	0.93	1.45	0.33	9.50	ok	even
C 14 H 10 Cl 3 N 3 O 2	0.010	356.98331	-2.83	-2.58	-1.01	10.00	ok	odd
C 17 H 7 Cl 2 N 2 O 3	0.161	356.98282	-4.19	-3.46	-1.50	14.50	ok	even

File Name	:	j:\maspec2\data\81457.ms2
Creation Date/Time	:	08-12-12 at 9:31:52
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

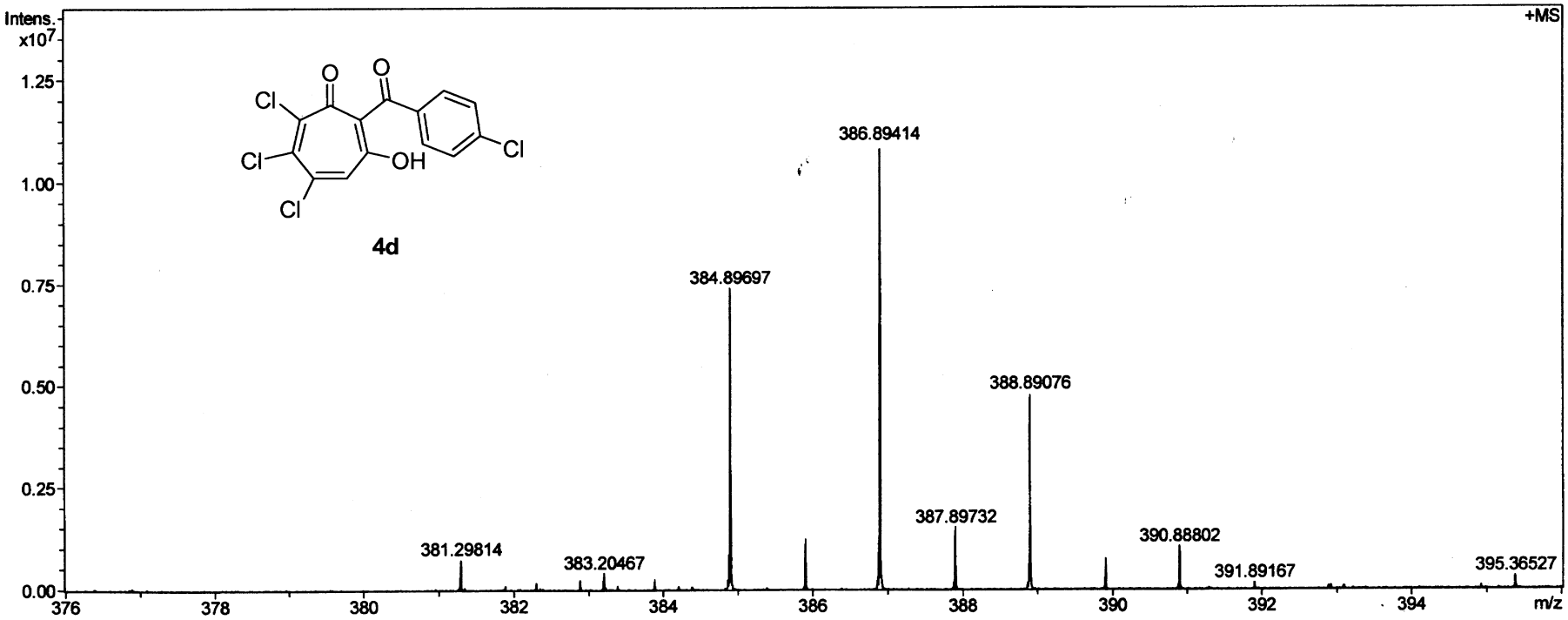
SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 37#3:20. Sub.=1#0:04. "14". Entries=555. Base M/z=133.3. 100% Int.=2.64448. EI. POS.



Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

Analysis Name	81427_20081216_000001.d	Acquisition Date	12/16/2008 4:50:06 PM
Sample	15	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University

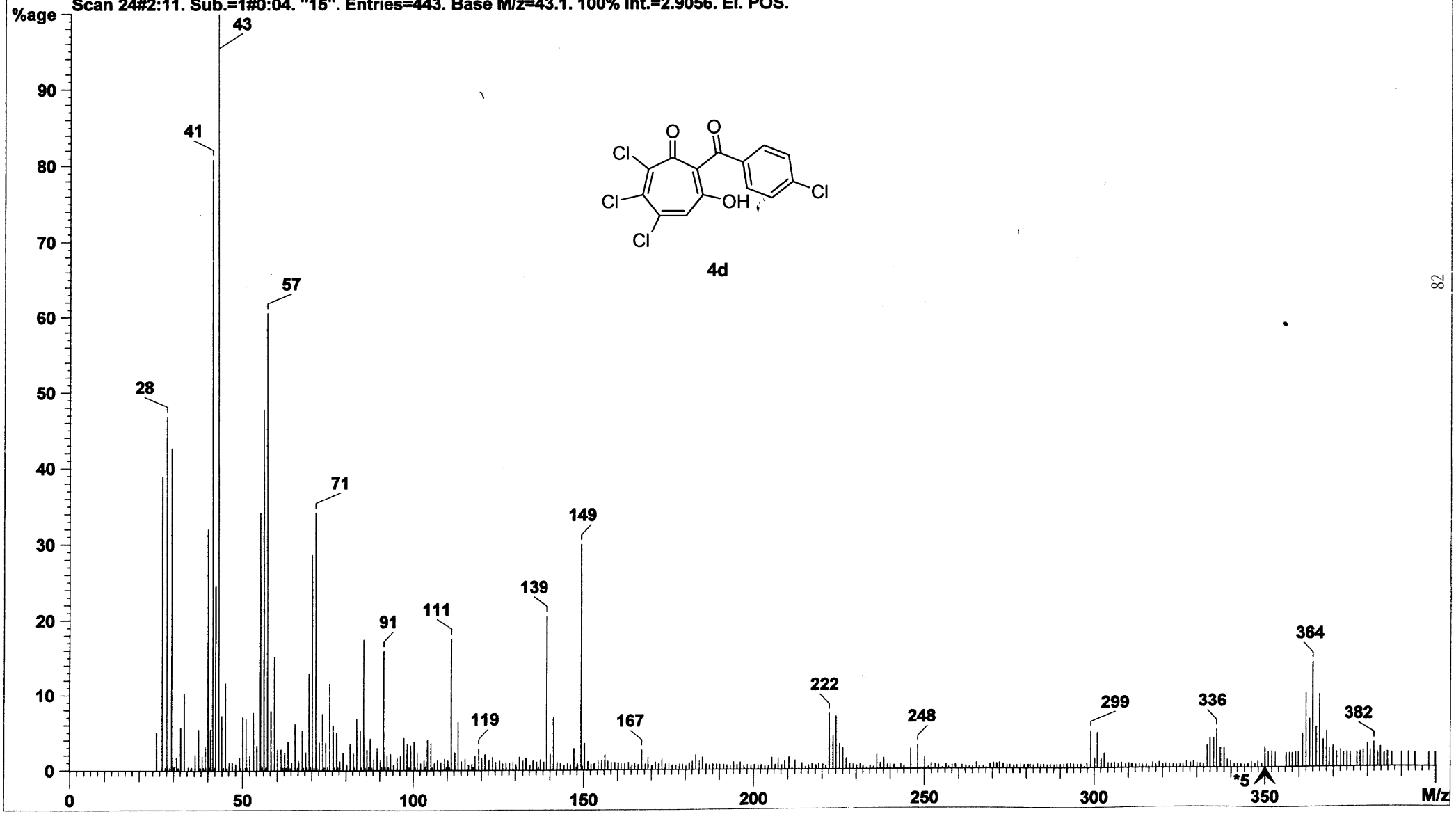


Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 6 Cl 4 Na 1 O 3	0.036	384.89633	-1.67	-1.24	-0.64	9.50	ok	even
C 16 H 5 Cl 4 O 3	0.038	384.89873	4.58	5.07	1.76	12.50	ok	even

Bruker Daltonics DataAnalysis 3.4 printed: 12/16/2008 4:52:09 PM Page 1 of 1

File Name	:	j:\maspec2\data\81479.ms2
Creation Date/Time	:	08-12-17 at 10:23:06
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000磺, Min.Hgt:1000磺, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 24#2:11. Sub.=1#0:04. "15". Entries=443. Base M/z=43.1. 100% Int.=2.9056. EI. POS.



4d

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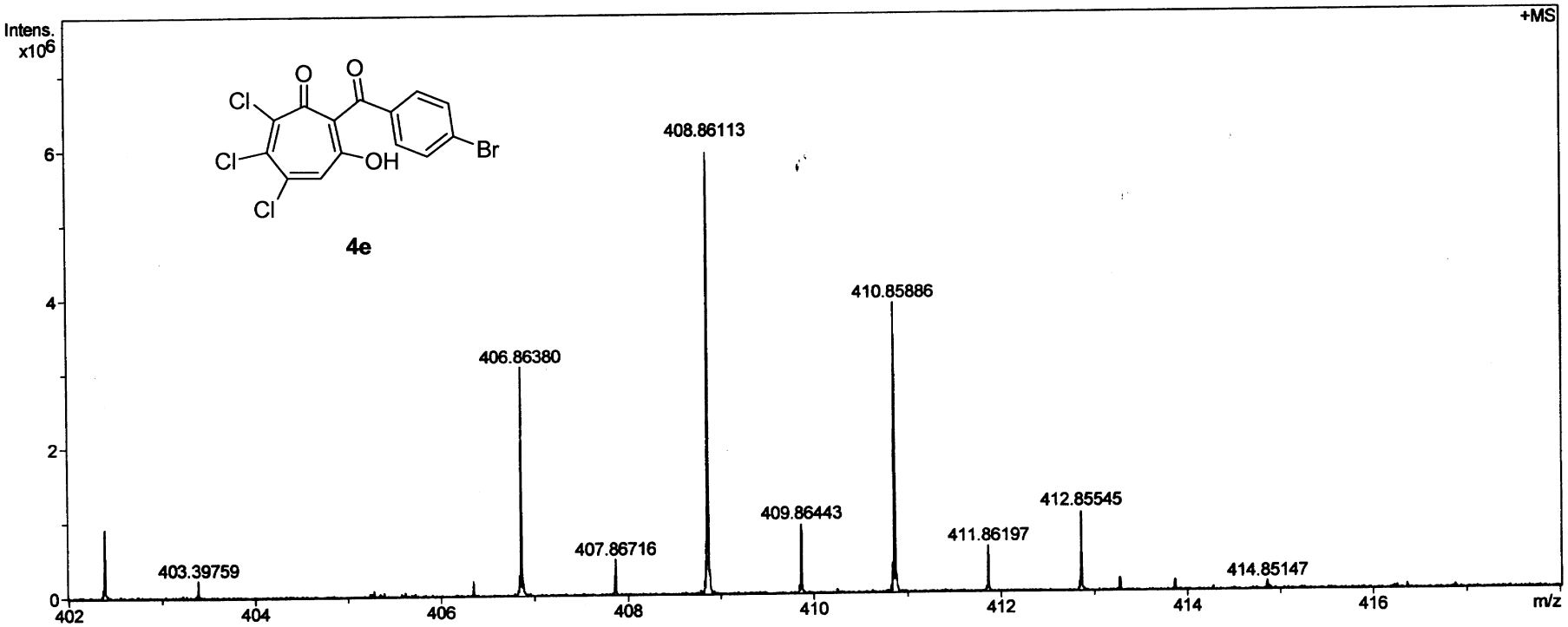
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

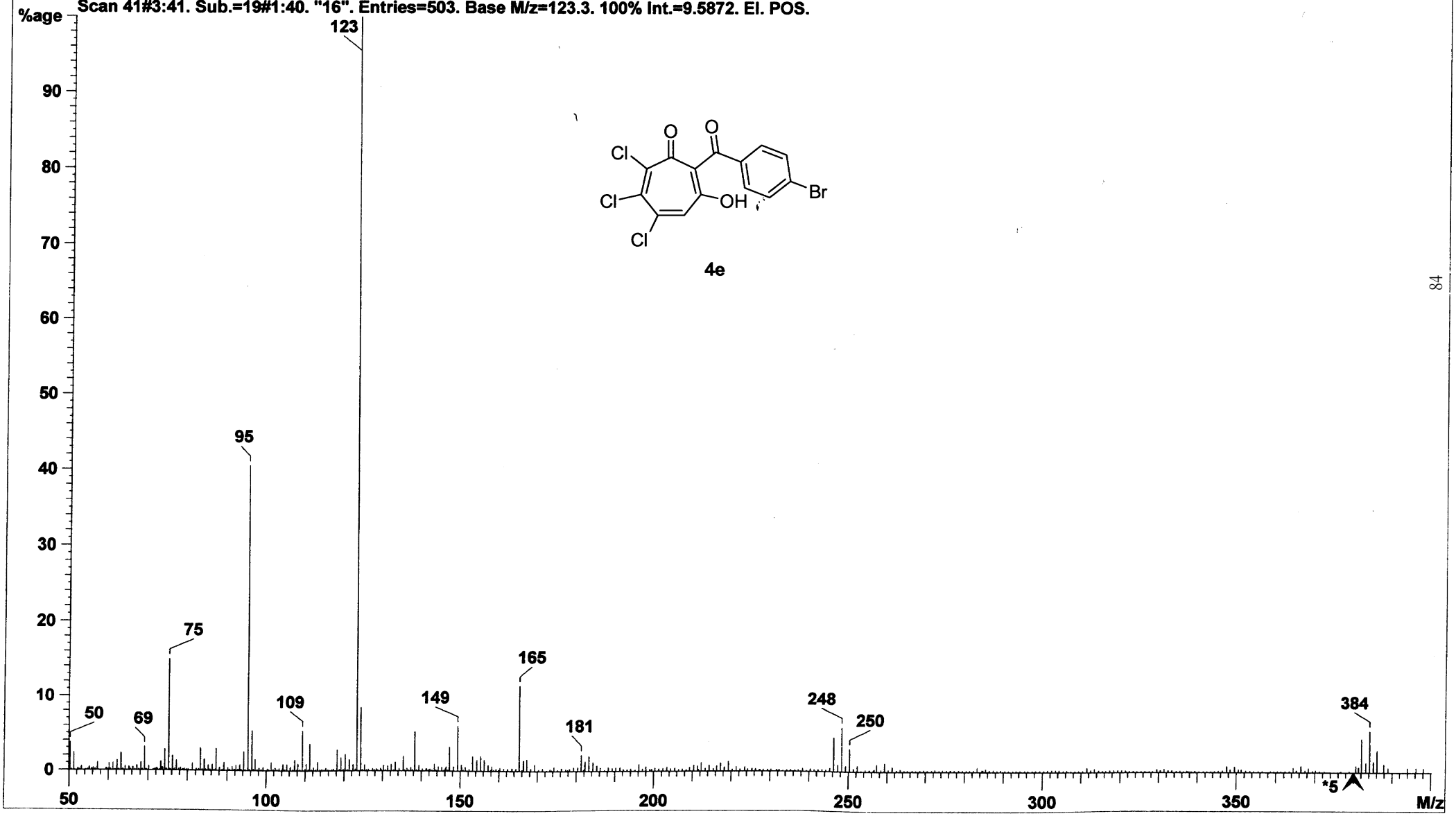
Analysis Name	81430_20081216_000001.d	Acquisition Date	12/16/2008 5:16:48 PM
Sample	20	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 14 H 7 Br 1 Cl 3 O 3	0.006	406.86387	0.15	0.60	0.06	9.50	ok	even

File Name	:	j:\maspec2\data\81467.ms2
Creation Date/Time	:	08-12-15 at 8:45:30
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 41#3:41. Sub.=19#1:40. "16". Entries=503. Base M/z=123.3. 100% Int.=9.5872. EI. POS.



4e

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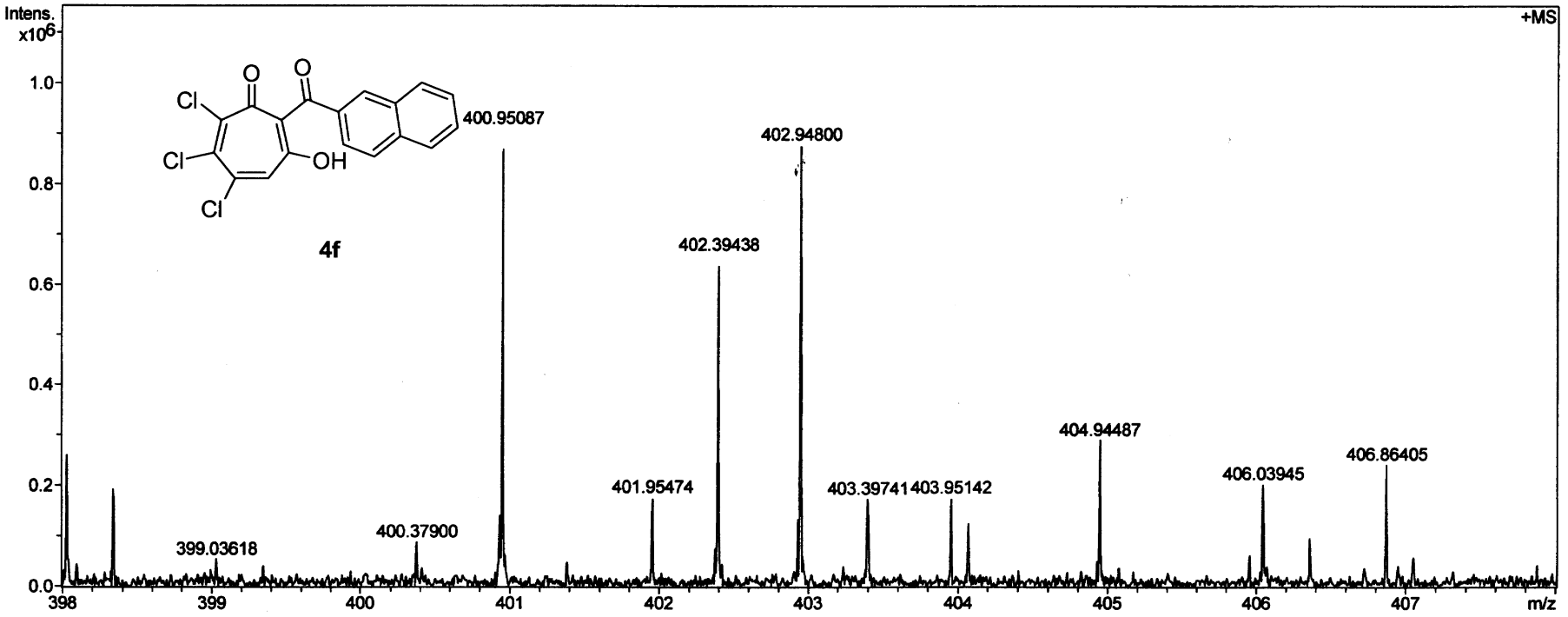
Peking University Mass Spectrometry Sample Analysis Report

Analysis Info

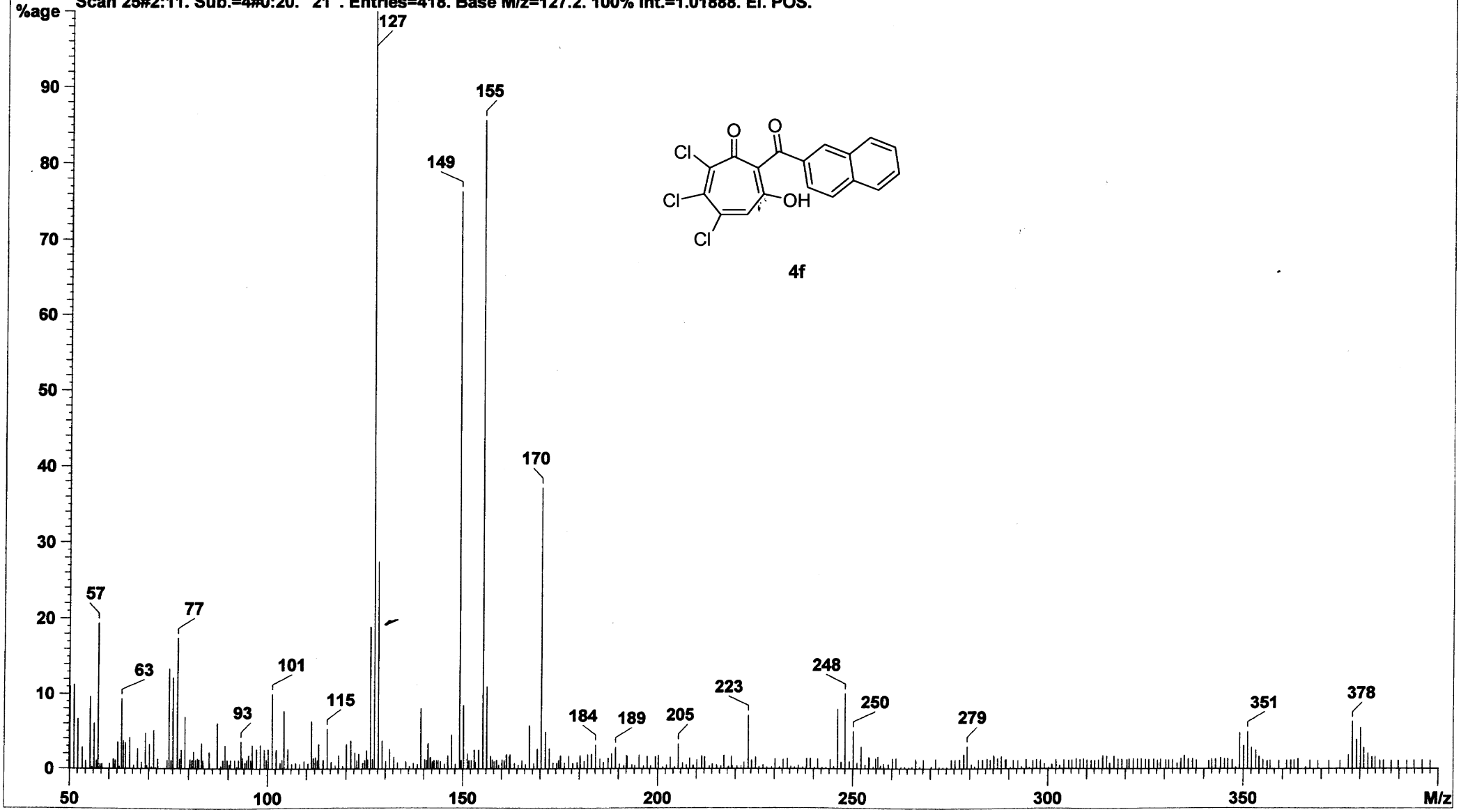
Analysis Name	81431_20081216_000001.d	Acquisition Date	12/16/2008 5:21:07 PM
Sample	21	Instrument	Bruker Apex IV FTMS
Comment	ESI Positive	Operator	Peking University



Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 18 H 9 Cl 3 Na 1 O 3	0.017	400.95095	0.19	0.47	0.07	12.50	ok	even

File Name	:	j:\maspec2\data\81483.ms2
Creation Date/Time	:	08-12-17 at 12:24:57
File Type	:	Lo-Res Data - Raw (Magnet)
File Source	:	Acquired on MASPEC II system [msw/9888]
Operator	:	Peking University
Instrument	:	ZAB-HS

SCAN GRAPH. Flagging=Nom.M/z. Ctd=[Thr:1000碛, Min.Hgt:1000碛, Min.Wid(Mlt):10(7), Inc:10%, Res:10%].
Scan 25#2:11. Sub.=4#0:20. "21". Entries=418. Base M/z=127.2. 100% Int.=1.01888. El. POS.



4f