The photochemistry of allenylsalicylaldehydes

Supporting Information

Frederic Birbaum, Antonia Neels, Christian G. Bochet.*

Department of Chemistry, University of Fribourg, Chemin du Musee 9, 1700 Fribourg, Switzerland

christian.bochet@unifr.ch

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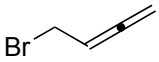
General. Unless otherwise indicated, all starting materials were obtained from commercial suppliers (Fluka, Sigma) and were used without further purification. Deuterated solvents such as dichloromethane, acetonitrile, chloroform, were obtained from Cambridge Isotope Laboratories. Solvents were dried on Alox and degassed with argon. Analytical thin layer chromatography was performed on Kieselgel F-254 pre-coated aluminium sheets TLC plates from Merck. Visualization was performed with, either a 254 nm ultraviolet lamp, or a $KMnO_4$ solution. Silica gel column chromatography was carried out with silica gel (32-63, 60 Å) from Brunschwig and aluminium oxide neutral, Brockmann I activity, from Fluka for all the separations containing acids sensitive functions. The ^1H and ^{13}C NMR spectra were recorded on a Brucker 360 or 500 MHz in $CDCl_3$, CD_3CN, CD_2Cl_2. Chemical shifts are expressed in parts per million (δ) using residual solvent protons as reference: chloroform (δ 7.27 for ^1H, δ 77.0 for ^{13}C), acetonitrile (δ 1.94 for ^1H), dichloromethane (δ 5.30 for ^1H). Coupling constant (J) are reported in Hz. Splitting patterns are designated as s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), bs (broad signal).

Combination gas chromatography and mass spectroscopy were obtained on a Thermoquest Trace GC 2000 set with a Zebron ZB-1, 30 m x 0.25 mm, 100% methylpolysiloxane column, mass detection was obtained on a Thermoquest Voyager with EI capability. Gas chromatography was done on a Fisons HRGC Mega 2 set with a 5% diphenyl 95% dimethylpolysiloxane column (60°C 1min - 10°C/min - 150°C 15 min).

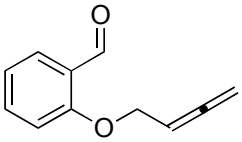
ESI-HRMS mass spectra were determined by Brucker FTMS 4.7T BioAPEX II. IR spectroscopy was done on Mattson Galaxy Series 5000 FT-IR in chloroform or neat on a NaCl cell.



4-Bromo-buta-1,2-diene -5-. Prepared according to a literature procedure[1] then purified by distillation to afforded 8.04 g (60%, 60 mmol).

^{1}H NMR (360 MHz, CDCl3) δ ppm 5.42-5.48 (dddd, *J* = 8.3, 8.2, 6.4, 6.6 Hz, 1H), 4.93-4.95 (dt, *J*= 2.1, 6.6 Hz, 2H), 3.95-3.98 (dt, *J* = 1.9, 8 Hz).

^{13}C NMR (90 MHz, CDCl3) δ ppm 209.6, 89.3, 77.1, 29.9.



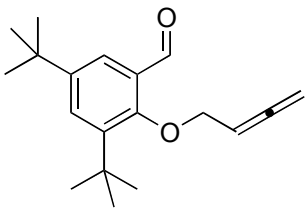
2-Buta-2,3-dienyloxy-benzaldehyde -6a-. DMF (25 mL) was added to K_2CO_3 (1.2 g, 8.7 mmol, 1.3 eq) in a round bottom flask containing salicylaldehyde (1 g, 8.2 mmol, 1.2 eq) at room temperature. 4-Bromo-buta-1,2-diene **-2-** (0.9 g, 6.8 mmol, 1.0 eq) was added over a period of 1 hour by syringe and stirred over a period of 16h. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (50 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by flash column chromatography (dichloromethane/pentane 1:2) on neutral alox afforded 0.83 g (70%, 4.8 mmol) of the title compound.

^{1}H NMR (500 MHz, $CDCl_3$) δ ppm 4.69 (dt, *J*=6.6, 2.7 Hz, 2H), 4.90 (dt, *J*=6.6, 2.7 Hz, 2H), 5.41 (quint, *J*=6.6 Hz, 1H), 6.98-7.05 (2H), 7.53 (ddd, *J*=9.2, 7.3, 1.9 Hz, 1H), 7.84 (dd, *J*=7.3 1.9 Hz, 1H), 10.50 (s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ ppm 66.1, 86.5, 113.0, 120.9, 125.2, 128.3, 135.7, 160.7, 189.8, 209.4.

EI-MS [m/z] (%) 174.4 (7.5), 145.4 (13), 122.4 (48), 121.3 (100), 120.4 (17), 92.4 (14), 53.3 (11).

IR (film): ν [cm^{-1}] = 3077, 2865, 2760, 1957, 1862, 1692, 1599, 1484, 1458, 1399, 1377, 1285, 1237, 1161, 1102, 1041, 1002, 851, 758, 654.



2-Buta-2,3-dienyloxy-3,5-di-tert-butyl-benzaldehyde -6b-. DMF (20 mL) was added to K_2CO_3 (550 mg, 4 mmol, 1.2 eq) in a round bottom flask containing 3,5-di-*tert*-butyl-2-hydroxy-benzaldehyde (1.0 g, 4.2 mmol, 1.2 eq) at room temperature. 4-Bromo-buta-1,2-

[1] (a) Molander, G.A.; Cormier, E.P. *J. Org. Chem*, **2005**, *70*, 2622-2626. (b) Meguro, M.; Yamamoto, Y. *J.Org.Chem.* **1999**, *64*, 694-695.

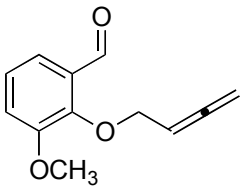
diene (450 mg, 3.4 mmol, 1.0 eq) was added over a period of 1 hour by syringe and stirred overnight. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (50 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by flash column chromatography (ether/pentane 1:4) on silica gel afforded 655 mg (67%, 2.3 mmol) of the title compound.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.33 (s, 9H), 1.44 (s, 9H), 4.48 (dt, *J*=6.4, 2.7 Hz, 2H), 4.89 (dt, *J*=6.8, 3.2 Hz, 2H), 5.50 (quint, *J*=6.4 Hz, 1H), 7.63 (d, *J*=2.3 Hz, 1H), 7.71 (d, *J*=2.3 Hz, 1H), 10.33 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 30.8, 31.3, 34.7, 35.3, 76.0, 76.7, 87.0, 124.0, 129.4, 130.8, 143.0, 146.5, 159.4, 190.7, 209.5.

ESI-HRMS [m/z] calcd for $C_{19}H_{26}NaO_2$ $[M+Na]^+$ 309.1830, found 309.1826.

IR (film): ν [cm^{-1}] = 2962, 2871, 2746, 1959, 1689, 1596, 1478, 1395, 1364, 1237, 1202, 1163, 980, 849.



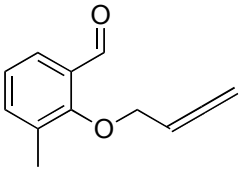
2-Buta-2,3-dienyloxy-3-methoxy-benzaldehyde -6c-. DMF (20 mL) was added to K_2CO_3 (545 mg, 3.9 mmol, 1.2 eq) in a round bottom flask containing 2-hydroxy-3-methoxy-benzaldehyde (500 mg, 3.3 mmol, 1.27 eq) at room temperature. 4-Bromo-buta-1,2-diene (350 mg, 2.6 mmol, 1.0 eq) was added over a period of 1 hour by syringe and stirred overnight. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (50 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by flash column chromatography (ether/pentane 1:2) on silica gel afforded 530 mg (quantitative yield) of the title compound.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 3.89 (s, 3H), 4.69 (dt, *J*=7.3, 1.8 Hz, 2H), 4.76 (dt, *J*=6.4, 1.8 Hz, 2H), 5.38 (quint, *J*=6.4 Hz, 1H), 7.13 (d, *J*=5.0 Hz, 2H), 7.40 (quint, *J*=4.1 Hz, 1H), 10.44 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 56.4, 72.3, 76.4, 87.1, 118.3, 119.4, 126.7, 130.7, 151.1, 153.4, 191.0, 210.5.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_3$ $[M+Na]^+$ 227.0684, found 227.0677.

IR (film): ν [cm^{-1}] = 3078, 2957, 2840, 1955, 1691, 1584, 1481, 1441, 1390, 1368, 1310, 1251, 1205, 1067, 972, 910, 851.



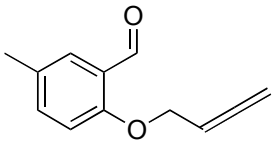
2-Buta-2,3-dienyloxy-3-methyl-benzaldehyde -6d-. DMF (35 mL) was added to K_2CO_3 (3.0 g, 22 mmol, 1.8 eq) in a round bottom flask containing 2-hydroxy-3-methyl-benzaldehyde (1.66 g, 12.2 mmol, 1.0 eq) at room temperature. 4-Bromo-buta-1,2-diene (2.2 g, 16.5 mmol, 1.35 eq) was added over a period of 1 hour by syringe and stirred overnight. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (50 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by a plug of silica gel which afforded 1.6 g (70%, 8.5 mmol) of the title compound.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.31 (s, 3H), 4.45 (dt, *J*=7.3, 2.1 Hz, 2H), 4.77 (dt, *J*=6.6, 2.1 Hz, 2H), 5.40 (quint, *J*=6.6 Hz, 1H), 7.10 (t, *J*=7.7 Hz, 1H), 7.40 (d, *J*=7.5 Hz, 1H), 7.65 (d, *J*=7.5 Hz, 1H), 10.35 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 15.8, 73.0, 76.2, 86.3, 124.3, 126.1, 129.4, 130.3, 137.5, 159.8, 190.3, 209.8.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.0732.

IR (film): ν [cm^{-1}] = 3352, 3069, 2928, 2863, 2749, 1956, 1694, 1588, 1470, 1368, 1247, 1198, 1086, 975, 850, 767.



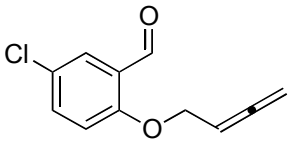
2-Buta-2,3-dienyloxy-5-methyl-benzaldehyde -6e-. DMF (20 mL) was added to K_2CO_3 (1.5 g, 11 mmol, 1.8 eq) in a round bottom flask containing 2-hydroxy-3-methyl-benzaldehyde (0.83 g, 6.1 mmol, 1.0 eq) at room temperature. 4-Bromo-buta-1,2-diene (1.1 g, 7.7 mmol, 1.3 eq) was added over a period of 1 hour by syringe and stirred over a period of 36 hours. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by a plug of silica gel which afforded 630 mg (55%, 3.35 mmol) of the title compound.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.31 (s, 3H), 4.66 (dt, *J*=6.4, 2.7 Hz, 2H), 4.90 (dt, *J*=6.6, 2.7 Hz, 2H), 5.41 (quint, *J*=6.6 Hz, 1H), 6.90 (d, *J*=8.4 Hz, 1H), 7.33 (dd, *J*=8.4, 2.0 Hz, 1H), 7.64 (s, 1H), 10.48 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 20.3, 66.3, 86.7, 113.1, 124.9, 128.4, 130.4, 136.4, 158.8, 190.1, 209.5.

EI-MS [m/z] (%) 188.3 ($M^{+\cdot}$, 7.1), 145.4 (20), 136.4 (100), 135.5 (87), 118.4 (11), 107.3 (25), 77.4 (11).

IR (film): ν [cm^{-1}] = 3352, 3066, 2925, 2864, 2760, 1957, 1863, 1686, 1609, 1582, 1494, 1396, 1284, 1243, 1161, 1114, 1003, 941, 851, 811, 727, 649.



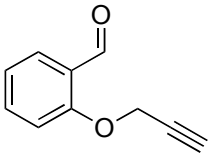
2-Buta-2,3-dienyloxy-5-chloro-benzaldehyde -6f-. DMF (20 mL) was added to K_2CO_3 (545 mg, 3.9 mmol, 1.2 eq) in a round bottom flask containing 5-chloro-2-hydroxy-benzaldehyde (650 mg, 4.2 mmol, 1.2 eq) at room temperature. 4-Bromo-buta-1,2-diene (450 mg, 3.4 mmol, 1.0 eq) was added over a period of 1 hour by syringe and stirred overnight. Ether was added (50 mL) and the organic layer was washed with an aqueous solution of K_2CO_3 (1 M, 3 x 50 mL) and with HCl 1N (3 x 50 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (50 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo, no purification was needed.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 4.69 (dt, *J*=6.6, 2.5 Hz, 2H), 4.91 (dt, *J*=6.6, 2.5 Hz, 2H), 5.40 (quint, *J*=6.6 Hz, 1H), 6.96 (d, *J*=9.1 Hz, 2H), 7.48 (dd, *J*=8.6, 2.3 Hz, 1H), 7.79 (d, *J*=2.7 Hz, 1H), 10.43 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 66.6, 86.3, 114.7, 126.1, 126.6, 128.0, 135.2, 159.1, 188.5, 209.6.

ESI-HRMS [m/z] calcd for $C_{11}H_9ClNaO_2$ $[M+Na]^+$ 213.0189, found 231.0185.

IR (film): ν [cm^{-1}] = 3019, 2882, 1956, 1682, 1596, 1477, 1394, 1269, 1215, 1128, 1001, 903, 853, 756, 669.



2-Prop-2-ynyloxy-benzaldehyde -7-. DMF (150 mL) was added to K_2CO_3 (12.7 g, 90.2 mmol, 1.1 eq) in a round bottom flask containing salicylaldehyde (10 g, 82.0 mmol, 1 eq) at room temperature. Propargyl bromide (10.9 g, 90.2 mmol, 1.1 eq, 80% in toluene) was added over a period 1 hour by syringe and stirred overnight. Ether was added (100 mL) and the organic layer was washed

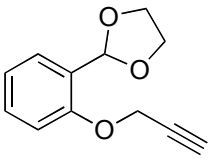
with an aqueous solution of K_2CO_3 (1 M, 3 x 100 mL) and with HCl 1N (3 x 100 mL) to get rid of the DMF. Finally the organic layer was washed with $NaCl_{sat}$ (100 mL) dried over $MgSO_4$ and filtered. The solvent was removed in vacuo and purification of the residue by flash column chromatography (ether/pentane 1:1) on silica gel to afforded 10.6 g (81%, 67 mmol) of the title compound. Recrystallization can be done in pentane/2-propanol 10:1.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.58 (s, 1H), 4.84 (d, *J*=1.5 Hz, 2H), 7.11 (m, *J*=8.4, 7.7, 7.1 Hz, 2H), 7.57 (2H), 7.87 (d, *J*=7.1 Hz, 2H), 10.50 (s, 1H).

^{13}C NMR (360 MHz, $CDCl_3$) δ ppm 189.9, 161.1, 136.06, 129.0, 122.1, 121.7, 113.6, 77.2, 76.8, 56.7.

ESI-HRMS [m/z] calcd for $C_{10}H_8NaO_2$ $[M+Na]^+$ 183.0422, found 183.0415.

IR (film): ν [cm^{-1}] = 3019, 1690, 1601, 1215, 750, 669.



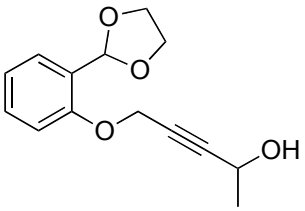
2-(2-Prop-2-ynyloxy-phenyl)-[1,3]dioxolane -8-. In a 25 mL round bottom flask fitted with a Dean Stark was added 2-prop-2-ynyloxy-benzaldehyde **-5-** (8 g, 50 mmol), ethylene glycol (4.8 g, 75 mmol), *p*-toluenesulfonic acid monohydrate (50 mg, 0.25 mmol) and dry toluene (150 mL). The reaction mixture was refluxed over a period of 24 hours. After cooling the reaction mixture was concentrated in vacuo and purification of the residue was done by flash column chromatography (ether/pentane 1:9) on neutral alox and afforded the title compound 5.5 g (54%, 27 mmol).

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.51 (t, *J*=2.2 Hz, 1H), 4.10-4.60 (4H), 4.77 (d, *J*=2.5 Hz, 2H) 6.19 (s, 1H), 7.04 (m, *J*=8.0, 6.6 Hz, 2H), 7.04 (m, *J*=8.0, 6.6 Hz, 2H) 7.35 (td, *J*=7.7, 1.6 Hz, 1H), 7.57 (dd, *J*=8.0, 1.6 Hz, 1H).

^{13}C NMR (360 MHz, $CDCl_3$) δ ppm 56.3, 65.3, 75.7, 78.5, 99.2, 122.6, 121.5, 126.6, 127.0, 130.1, 155.7.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_3$ $[M+Na]^+$ 227.0684, found 227.0678.

IR (film): ν [cm^{-1}] = 3285, 3044, 2955, 2888, 2120, 1690, 1605, 1492, 1459, 1399, 1288.



5-(2-[1,3]Dioxolan-2-yl-phenoxy)-pent-3-yn-2-ol -9-. To solution of 2-(2-prop-2-ynyloxy-phenyl)-[1,3]-dioxolane **-8-** (700 mg, 3.4 mmol) in THF (20 mL) was added 1.1 equiv of *n*-BuLi (2.4 mL, hexane solution, 1.6 M) at -70 °C over a period of 30 min. After being stirred for a few minutes, the dark yellow solution was allowed to warm to -30 °C.

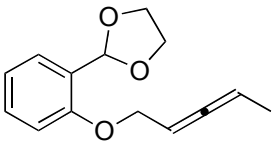
After 15 min, 1.5 equiv of acetaldehyde (230 mg, 3.8 mmol) was slowly added by syringe at -30 °C. After being stirred for 1 h at room temperature, the yellow solution was quenched with a saturated NH_4Cl solution (20 mL). Ether was added (50 mL) and the organic layer was washed with a saturated $NaHCO_3$ solution (50 mL) and brine (2 x 50 mL). The water layer was extracted twice with diethyl ether (2 x 20 mL) and the combined organic fractions were then dried on $MgSO_4$. After filtration, the solvent was evaporated. The raw product was purified by *Kugelrohr* distillation at low pressure (105 °C, 0.5 mmHg): yield 600 mg (70%, 2.4 mmol) of a yellow oil.

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.40 (d, *J*=6.6 Hz, 3H), 4.10-4.60 (4H), 4.51 (bd, *J*=6.4 Hz 1H), 4.77 (s, 2H), 6.18 (s, 1 H), 7.03 (2H), 7.33(td, *J*=8, 1.4 Hz, 1H), 7.54 (d, *J*=7.5 Hz, 3H).

^{13}C NMR (360 MHz, $CDCl_3$) δ ppm 23.9, 57.0, 58.1, 65.3, 75.7, 89.5, 99.1, 113.2, 121.6, 126.8, 127.0, 130.2, 155.8.

ESI-HRMS [m/z] calcd for $C_{14}H_{16}NaO_4$ $[M+Na]^+$ 271.0946, found 271.0930.

IR (film): ν [cm^{-1}] = 3433, 3058, 2983, 2882, 1737, 1605, 1492, 1548, 1376, 1288, 1227, 1150, 1077, 1018, 940, 891, 842, 758, 737.



2-(2-Penta-2,3-dienyloxy-phenyl)-[1,3]dioxolane -10-[2]. Diethyl azodicarboxylate (1.32 g, 3.04 mmol, 1.3 equiv, 40% in toluene) was added to a solution of triphenylphosphine (813 mg, 3.04 mmol, 1.3 equiv) in THF (10 mL) at -15 °C. After 10 min, a solution of 5-(2-[1,3]-dioxolan-2-yl-phenoxy)-pent-3-yn-2-ol **-7-**, (580 mg, 2.3 mmol, 1 equiv) in THF (7 mL) was added to the yellow reaction mixture, followed 10 min later by a solution of *o*-nitrobenzenesulfonylhydrazide (670 mg, 3.04 mmol, 1.3 equiv) in THF (10 mL). The resulting suspension was held at -15 °C for 1 h. The reaction mixture was warmed to room temperature and allowed to stand overnight (8 h). Concentration of the reaction mixture and purification of the residue by chromatography on silica gel afforded the title allene as a colourless oil 180 mg (34%, 0.77 mmol).

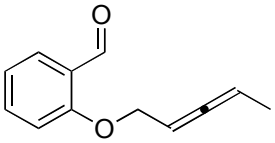
^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.66 (dd, *J*=7.0, 3.2 Hz, 3H), 3.99-4.63 (4H), 4.62 (dd, *J*=6.6, 2.3 Hz, 2H), 5.20-5.26 (1H), 5.32 (m, *J*=3.2 Hz, 1H), 6.19 (s, 1H), 6.95 (m, 2H), 7.30 (td, *J*=8.2, 1.6 Hz, 1H), 7.54 (dd, *J*=7.5, 1.4 Hz, 1H).

[2] Myers, A. G.; Zheng, B. *J. Am. Chem Soc.* **1996**, *118*, 4492-4493.

^{13}C NMR (360 MHz, $CDCl_3$) δ ppm 13.8, 65.3, 66.7, 87.1, 87.5, 99.3, 112.5, 120.6, 126.3, 126.9, 130.0, 156.6, 205.9.

ESI-HRMS [m/z] calcd for $C_{14}H_{16}NaO_3$ $[M+Na]^+$ 255.0997, found 255.0995.

IR (film): ν [cm^{-1}] = 3066, 2950, 2893, 1969, 1869, 1688, 1599, 1492, 1455, 1396, 1286, 1242, 1120, 1074, 1006, 860, 755.



2-Penta-2,3-dienyloxy-benzaldehyde -11-. 2-(2-Penta-2,3-dienyloxy-phenyl)-[1,3]dioxolane **-10-** (180 mg, 0.78 mmol, 1 eq) and *p*-toluenesulfonic acid monohydrate (8 mg, 39.5 μmol, 5% mol) was added in an acetone/water mixture (12 mL, 1:1.2 water/acetone) under argon atmosphere over 60 h at room temperature. Silica gel was then added and the solvent was removed under vacuum. The solid residue was further purified by silica gel flash column chromatography (ether/pentane 1:3) and afforded the title compound 80 mg (55%, 0.43 mmol).

^{1}H NMR (360 MHz, CD_2Cl_2) δ ppm 1.66 (dd, *J*=7.0, 3.2 Hz, 3H), 4.7 (dd, *J*=6.1, 2.3 Hz, 2H), 5.24-5.32 (1H), 5.35 (m, *J*=3.2 Hz, 1H), 7.03 (m, *J*=8.2 Hz, 2H), 7.55 (td, *J*=8.0, 1.8 Hz, 1H), 7.79 (d, *J*=8.0, 1.8 Hz, 1H), 10.50 (s, 1H).

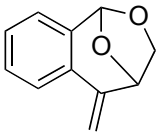
^{13}C NMR (90 MHz, CD_2Cl_2, ppm) δ 206.8, 190.3, 161.7, 136.5, 128.8, 126.0, 121.5, 114.2, 88.8. 87.3, 67.6, 14.4.

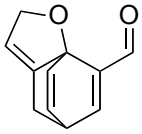
EI-MS [m/z] (%), 188.1 ($M^{+\cdot}$, 5); 186.9 (10); 173.0 (15); 158.9 (30); 144.7 (20); 122.0 (90); 120.9 (100); 103.9 (15), 91.6 (15); 76.9 (20), 66.9 (40); 64.8 (75); 50.9 (20).

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.07310.

IR (film): ν [cm^{-1}] = 2860, 1970, 1689, 1599, 1481, 1457, 1396, 1286, 1162, 1000, 758, 651.

UV-VIS λ_{max} = 320.0 nm.





8-Methylene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]dodeca-2,4,6-triene -12a- and **2-Oxa-tricyclo[5.2.2.0^{1,5}]undeca-4,8,10-triene-9-carbaldehyde -13a-.**. 2-Buta-2,3-dienyloxy-benzaldehyde **-6a-** (40 mg, 230 μmol) was added to dry dichloromethane (3 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 254 nm over 5h at room temperature. A flash column chromatography

(dichloromethane/pentane, 3:5) afforded 5 mg (13 %, 29 μmol) of **12a** and 40 mg (40 %, 230 μmol) of **13a**.

When a solution of buta-2,3-dienyloxy-benzaldehyde **6a** (5.8 mg, 10 mmol) was irradiated at 350 nm over a period of 1h30, 28% of **12a** and 48% of **13a** (yields determined by GC) were obtained.

12a:

^{1}H **NMR** (500 MHz, $CDCl_3$) δ ppm 3.73 (dd, *J*=7.2, 1.4 Hz, 1H), 4.10 (dd, *J*=7.2, 5.9 Hz, 1H), 5.10 (dd, *J*=5.9, 1.4 Hz, 1H), 5.15 (s, 1H), 5.67 (s, 1H), 6.09 (s, 1H), 7.19 (dd, *J*=7.4, 1.2 Hz, 1H), 7.32 (dtd, *J*=20.0, 7.4, 1.4 Hz, 2H), 7.70 (d, *J*=7.4, Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ ppm 68.9, 77.9, 101.3, 107.1, 123.4, 125.0, 128.6, 128.9.

EI-MS [m/z] (%) 174.4 ($M^{+\cdot}$,19), 145.4 (17), 144.5 (100), 116.4 (42.4), 155.5 (58.8).

IR (film): ν [cm^{-1}] = 3021, 1527, 1425, 1215, 669.

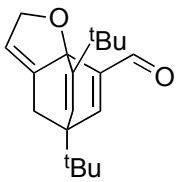
13a:

^{1}H NMR (500 MHz, $CDCl_3$) δ ppm 2.11 (dquint, *J*=11.2, 1.7 Hz, 1H), 2.19 (dquint, *J*=11.2, 1.7 Hz, 1H), 3.97 (m, 1H), 5.07 (m, *J*=1.4 Hz, 1H), 5.41 (quint, *J*=1.7 Hz, 1H), 6.18 (ddd, *J*=7.7, 5.9, 0.8 Hz, 1H), 6.58 (dd, *J*=7.6, 1.5 Hz, 1H), 7.07 (d, *J*=6.2 Hz, 1H), 9.70 (s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ ppm 26.8, 39.3, 79.9, 96.4, 112.5, 129.2, 138.9, 138.9, 146.4, 148.6, 187.3.

EI-MS [m/z] (%) 175.4 ($M^{+\cdot}$,10), 174.4 (39), 173.4 (25), 145.4 (63), 131.4 (44), 120.4 (80), 115.4 (40), 90.4 (100), 65.4 (15), 53.3 (17).

IR (film): ν [cm^{-1}] =.3021, 2864, 2286, 1682, 1570, 1215, 1167, 1012 669.



7,10-Di-tert-butyl-2-oxa-tricyclo[5.2.2.0^{1,5}]undeca-4,8,10-triene-9-carbaldehyde -13b-. 2-Buta-2,3-dienyloxy-3,5-di-tert-butyl-benzaldehyde **-3b-** (100 mg, 350 μmol) was added to dry dichloromethane (15 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 350 nm over 1h at room temperature. A flash column chromatography (ether/pentane, 1:7) afforded 94 mg (94%, 329 μmol) of the title compound.

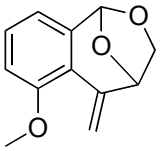
When a solution of 2-buta-2,3-dienyloxy-3,5-di-tert-butyl-benzaldehyde **-6b-** (6 mg, 7.0 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 5 min, a complete conversion into the title compound was obtained (yield determined by GC).

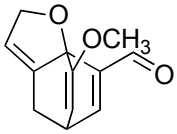
^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.13 (d, *J*=3.0 Hz, 18H), 2.11 (qd, *J*=15.4, 2.3 Hz, 2H), 5.05 (m, 2H), 5.26 (s, 1H), 5.70 (s, 1H), 7.10 (s, 1H), 9.72 (s, 1H).

^{13}C NMR (125 MHz, $CDCl_3$) δ ppm 26.5, 27.8, 28.5, 32.4, 34.5, 54.8, 79.2, 98.3, 110.6, 122.4, 142.5, 148.5, 149.7, 158.4, 187.8.

ESI-HRMS [m/z] calcd for $C_{19}H_{26}NaO_2$ $[M+Na]^+$ 309.1830, found 309.1821.

IR (film): ν [cm^{-1}] =.3019, 2965, 2856, 2402, 1679, 1621, 1576, 1475, 1372, 1216, 1148, 1013, 669.





6-Methoxy-8-methylene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]-dodeca-2,4,6-triene -12c- and **10-Methoxy-2-oxa-tricyclo[5.2.2.0^{1,5}]undeca-4,8,10-triene-9-carbaldehyde -13c-.** 2-Buta-2,3-dienyloxy-3-methoxy-benzaldehyde **6c** (162 mg, 79.0 mmol, 28 mM) was added to dry dichloromethane (28 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 350 nm over a period of 1h20 at room temperature. A flash column chromatography (ether/pentane, 1:2) afforded 51 mg (31 %, 24.0 mmol) of an inseparable mixture of two compounds (76% of **12c** and 24% B) and 108 mg (67%, 529 μmol) of **13c.**

When a solution of 2-Buta-2,3-dienyloxy-3-methoxy-benzaldehyde **6c** (8.7 mg, 14 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 30 min, 23% of **12c** and 57% of **13c** (yields determined by GC) were obtained.

12c:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm A: 3.74 (d, *J*=9.3 Hz, 1H), 3.92 (s, 3H), 4.07 (t, *J*=6.8, Hz, 1H), 4.99 (d, *J*=5.9 Hz, 1H), 5.31 (s, 1H), 6.06 (s, 1H), 6.28 (s, 1H), 6.83 (d, *J*=10.1 Hz, 1H), 6.93 (d, *J*=11.6 Hz, 1H), 7.23 (m, 1H), B: 3.83 (d, *J*=18 Hz, 1H), 3.91 (s, 3H), 4.01 (t, *J*=6.0, Hz, 1H), 5.25 (d, *J*=4.5 Hz, 1H), 5.33 (s, 1H), 5.81 (s, 1H), 6.32 (s, 1H), 6.77 (d, *J*=10.4 Hz, 1H), 6.90 (d, *J*=11.6 Hz, 1H), 7.23 (m, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm A: 55.3, 69.3, 79.8, 101.5, 111.7, 114.1, 117.4, 128.9, 138.1, 140.1, 159.2, B: 55.3, 72.4, 76.1, 79.8, 106.1, 110.9, 115.4, 116.5, 117.1, 128.5, 138.6, 139.1, 159.9.

EI-MS [m/z] (%) 204.25 ($M^{+\cdot}$,50), 175.4 (16), 174.4 (91), 173.3 (30), 159.4 (100), 146.4 (15), 145.4 (58), 144.4 (22), 131.4 (20), 118.35 (33), 116.35 (24), 115.35 (52), 103.35 (11.13).

IR (film): ν [cm^{-1}] = 3018, 2966, 2989, 2840, 2405, 1623, 1598, 1479, 1341, 1266, 1216, 1084, 1216, 1084, 973, 957, 903, 752, 666.

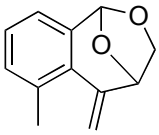
13c:

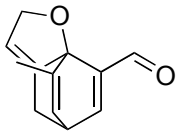
^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.13 (d, *J*=15.9 Hz, 1H), 2.28 (d, *J*=15.4 Hz, 1H), 3.56 (s, 3H), 3.82 (quint, *J*=3.6 Hz, 1H), 4.81 (d, *J*=6.8 Hz, 1H), 5.11 (m, 2H), 5.42 (s, 1H), 7.19 (d, *J*=5.9 Hz, 1H), 9.71 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 28.0, 36.5, 56.3, 80.8, 92.4, 94.2, 112.9, 139.3, 147.0, 147.3, 164.5, 187.3.

EI-MS [m/z] (%), 204.25 ($M^{+\cdot}$,100), 176.35 (31), 175.35 (45), 161.35 (33), 147.35 (33), 120.35 (52), 119.35 (65), 115.4 (42), 91.35 (66), 77.35 (41).

IR (film): ν [cm^{-1}] =.2964, 2893, 2804, 2250, 1689, 1597, 1479, 1345, 1264, 1084, 956, 906, 797, 733.





6-Methyl-8-methylene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]-dodeca-2,4,6-triene -12d- and **10-Methyl-2-oxa-tricyclo-[5.2.2.01,5]undeca-4,8,10-triene-9-carbaldehyde-13d-.** 2-Buta-2,3-dienyloxy-3-methyl-benzaldehyde **6d** (340 mg, 1.8 mmol, 0.12 M) was added to dry dichloromethane (15 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 350 nm over a period of 8h at room temperature. Two flash column chromatographies (ether/pentane, 1:4, and dichloromethane) afforded 43 mg (13 %, 0.28 mmol) of **12d** and 34 mg (10 %, 0.18 mmol) of **13d**.

When a solution of 2-buta-2,3-dienyloxy-3-methyl-benzaldehyde **6d** (7.0 mg, 13 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 160 min, 36% of **12d** and 46% of **13d** (yields determined by GC) were obtained.

12d:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.54 (s, 3H), 3.73 (d, *J*=7.3 Hz, 1H), 4.09 (t, *J*=6.1 Hz, 1H), 5.04 (d, *J*=5.7 Hz, 1H), 5.36 (s, 1H), 5.64 (s, 1H), 6.07 (s, 1H), 7.07 (m, 1H), 7.18 (m, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 24.2, 68.9, 80.3, 101.9, 113.7, 113.2, 123.2, 127.3, 127.7, 132.7, 137.1, 143.1.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.0728.

IR (film): ν [cm^{-1}] = 3068, 2969, 2893, 2251, 1715, 1680, 1624, 1469, 1448, 1347, 1248, 1225, 1137, 1084, 1051, 973, 910, 775, 732, 662.

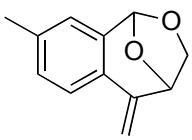
13d:

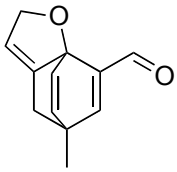
^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.85 (d, *J*=1.4 Hz, 3H), 2.07 (dm, *J*=15.4, 2.3 Hz, 1H), 2.20 (dm, *J*=15.4, 2.3 Hz, 1H), 3.83 (m, *J*=3.2 Hz, 1H), 5.05 (m, 2H), 5.34 (s, 1H), 5.75 (d, *J*=5 Hz, 1H), 7.11 (d, *J*=6.4 Hz, 1H), 9.67 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 14.5, 27.4, 38.6, 80.2, 97.6, 112.2, 122.1, 139.2, 147.5, 147.8, 148.3, 187.5.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.0727.

IR (film): ν [cm^{-1}] =.3019, 2920, 2859, 2400, 2252, 1679, 1581, 1442, 1348, 1216, 1156, 1027, 909, 757, 668.





4-Methyl-8-methylene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]-dodeca-2,4,6-triene -12e- and **7-methyl-2-oxa-tricyclo[5.2.2.01,5]undeca-4,8,10-triene-9-carbaldehyde -13e-.** 2-Buta-2,3-dienyloxy-5-methyl-benzaldehyde **6e** (104 mg, 55 μmol, 3.7 mM) was added to dry dichloromethane (15 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 350 nm over a period of 16h at room temperature. A flash column chromatography (ether/pentane, 1:2) afforded 11 mg (11 %, 5.8 μmol) of **12e** and 35 mg (34 %, 0.19 mmol) **of 13e**.

When a solution of 2-buta-2,3-dienyloxy-5-methyl-benzaldehyde **6e** (6.3 mg, 11 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 90 min, 22% of **12e** and 65% of **13e** (yields determined by GC) were obtained.

12e:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.35 (s, 3H), 3.71 (dd, *J*=7.3, 1.1 Hz, 1H), 4.08 (t, *J*=6.1 Hz, 1H), 5.08 (m, 2H), 5.59 (s, 1H), 6.04 (s, 1H), 7.0 (s, 1H), 7.13 (d, *J*=8.0 Hz, 1H), 7.58 (d, *J*=8.0 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 21.2, 68.9, 78.0, 101.4, 106.1, 123.4, 125.5, 126.2, 129.6, 136.0, 138.7, 142.0.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.0730.

IR (film): ν [cm^{-1}] = 3020, 2970, 2896, 2403, 1679, 1498, 1422, 1338, 1215, 1102, 1085, 964, 903, 878, 827, 755, 669.

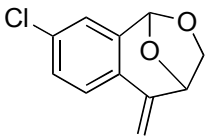
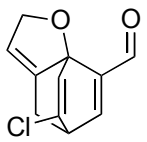
13e:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.65 (s, 3H), 1.99 (dm, *J*=15.4 Hz, 1H), 2.06 (dm, *J*=13.0 Hz, 1H), 5.06 (d, *J*=1.6 Hz, 2H), 5.34 (m, 1H), 5.90 (d, *J*=7.5 Hz, 1H), 6.54 (d, *J*=7.5 Hz, 1H), 6.80 (s 1H), 9.66 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 21.5, 34.6, 45.2, 19.9, 96.7, 111.8, 134.8, 138.4, 140.7, 148.1, 151.5, 187.3.

ESI-HRMS [m/z] calcd for $C_{12}H_{12}NaO_2$ $[M+Na]^+$ 211.0735, found 211.0729.

IR (film): ν [cm^{-1}] =.3020, 2965, 2956, 1680, 1614, 1564, 1455, 1351, 1215, 1169, 1009, 909, 760, 669.

4-Chloro-8-methylene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]-dodeca-2,4,6-triene -12f- and **7-Chloro-2-oxa-tricyclo-[5.2.2.0^{1,5}]undeca-4,8,10-triene-9-carbaldehyde-13f-.** 2-Buta-2,3-dienyloxy-5-chloro-benzaldehyde **6f** (165 mg, 790 μmol, 40 mM) was added to dry dichloromethane (20 mL) in a quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 350 nm over 2h30 at room temperature. A flash column chromatography (ether/pentane, 1:9) followed by a second column (dichloromethane/pentane; 1:1) afforded 66 mg, (40 %, 320 μmol) of **12f** and 46 mg, (28 %, 221 μmol) of **13f**.

When a solution of 2-buta-2,3-dienyloxy-5-chloro-benzaldehyde **6f** (7.5 mg, 12 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 2h00, 44% of **12f** and 31% of **13f** (yields determined by GC) were obtained.

12f:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 3.72 (d, *J*=7.3 Hz, 1H), 4.09 (t, *J*=6.8 Hz, 1H), 5.09 (d, *J*=5.9 Hz, 1H), 5.17 (s, 1H), 5.64 (s, 1H), 6.03 (s, 1H), 7.18 (d, *J*=1.8 Hz, 1H), 7.26-7.30 (1H), 7.62(d, *J*=9 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 69.0, 77.8, 100.6, 107.7, 125.0, 125.1 127.5, 129.0, 134.2, 137.6, 141.1.

EI-MS [m/z] (%) 193 (10), 178 (22), 151 (45), 136 (58), 115 (100), 87 (82), 75 (100), 50 (55).

IR (film): ν [cm^{-1}] = 3020, 2972, 2896, 1898, 1807, 1685, 1639, 1596, 1417, 1334, 1216, 1193, 1113, 1086, 1012, 966, 904, 875, 815, 758.

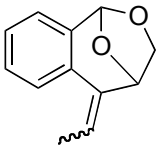
13f:

^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 2.58 (dd, *J*=23.2, 15.4 Hz, 2H), 5.06 (m, 2H), 5.43 (s, 1H), 6.19 (d, *J*=8.2 Hz, 1H), 6.56 (d, *J*=7.7 Hz, 1H), 6.97 (s, 1H), 9.69 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ ppm 38.1, 65.6, 80.2, 95.4, 112.7, 114.0, 137.49, 137.51, 146.3, 147.6, 186.3.

EI-MS [m/z] (%), 172 (100), 155 (5), 144 (50), 115 (75), 89 (30), 63 (12), 50 (5).

IR (film): ν [cm^{-1}] = 3020, 2868, 2360, 1690, 1607, 1564, 1350, 1216, 1169, 1043, 1013, 926, 757, 682.



8-Ethylidene-11,12-dioxa-tricyclo[7.2.1.0^{2,7}]dodeca-2,4,6-triene -14-. 2-Penta-2,3-dienyloxy-benzaldehyde **-11-** (50 mg, 266 μmol) was added to dry dichloromethane (1.5 mL) in a NMR quartz tube sealed with a rubber septum. The reaction mixture was purged with argon over 15 min and irradiated at 254 nm over 3h at room temperature. A flash column chromatography (pentane/ether, 9:1) afforded 21.4 mg (43 %, 114 μmol) of the title compound.

When a solution of 2-penta-2,3-dienyloxy-benzaldehyde **-11-** (6.8 mg, 5.5 mmol) was irradiated in dry dichloromethane (3 mL) at 350 nm over a period of 1h00, 68% of the title compound (GC yield) was obtained.

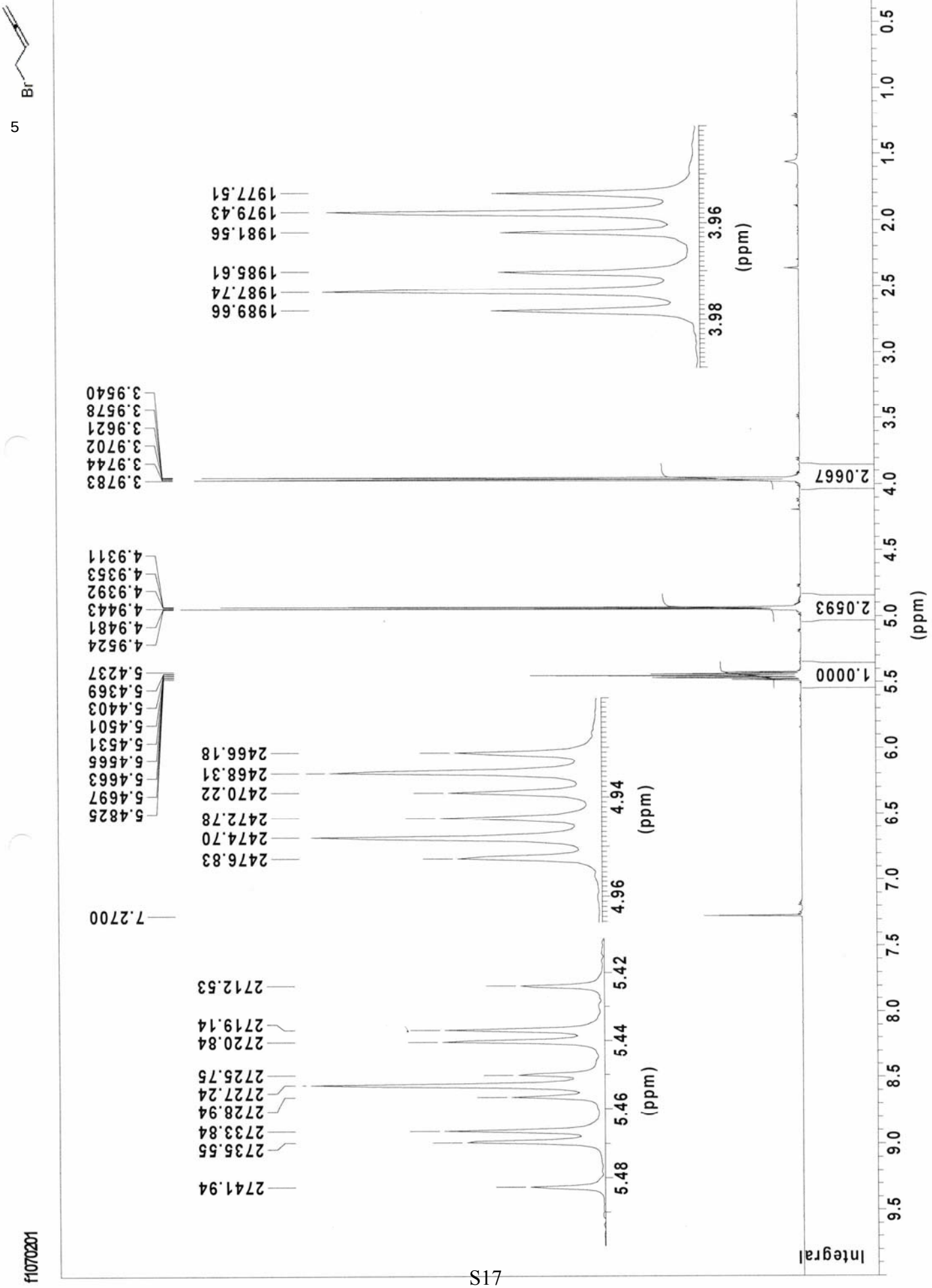
^{1}H NMR (360 MHz, $CDCl_3$) δ ppm 1.90 (d, *J*=7.3 Hz, 3H), 2.09 (d, *J*=7.5 Hz, 3H), 3.72 (td, *J*=6.2, 0.7 Hz, 2H), 4.08 (quint, *J*=6.4 Hz, 2H), 4.93 (d, *J*=5.2 Hz, 1H), 5.53 (d, *J*=5.7 Hz, 1H), 5.81 (dd, *J*=15.0, 7.5 Hz, 1H), 6.06 (d, *J*=5.8 Hz, 2H), 6.31 (dd, *J*=14.3, 7.2 Hz, 1H), 7.10-7.40 (6H), 7.62 (d, *J*=8.0 Hz, 1H), 7.75 (d, *J*=7.7 Hz, 1H).

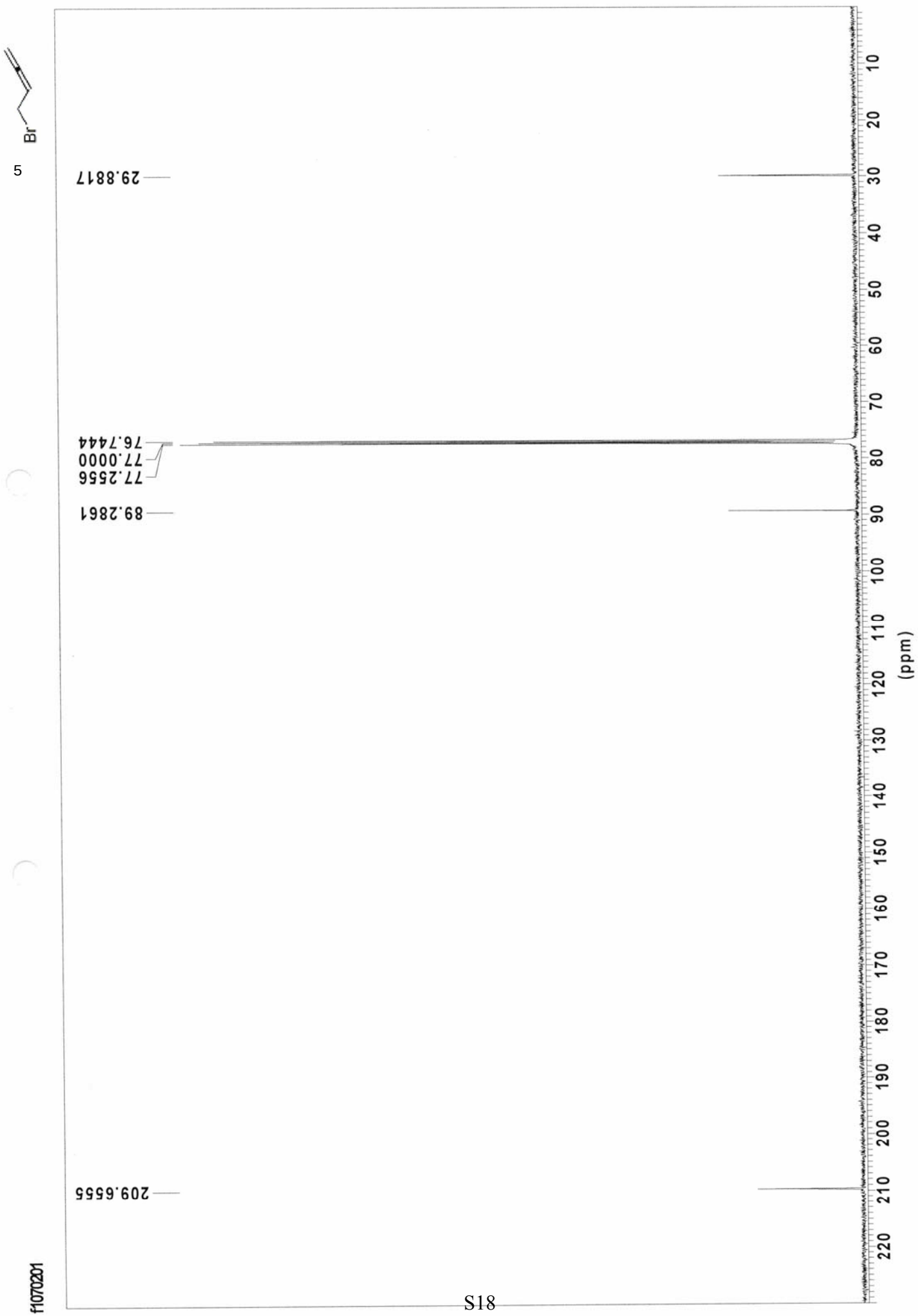
^{13}C NMR (90 MHz, $CDCl_3$, ppm) δ 136.5, 135.4, 134.7, 134.2, 130.2, 128.8, 128.5, 128.0, 127.4, 127.1, 125.1, 124.9, 122.8, 122.1, 118.2, 101.5, 101.3, 80.6, 72.1, 69.1, 68.9, 15.6, 13.1.

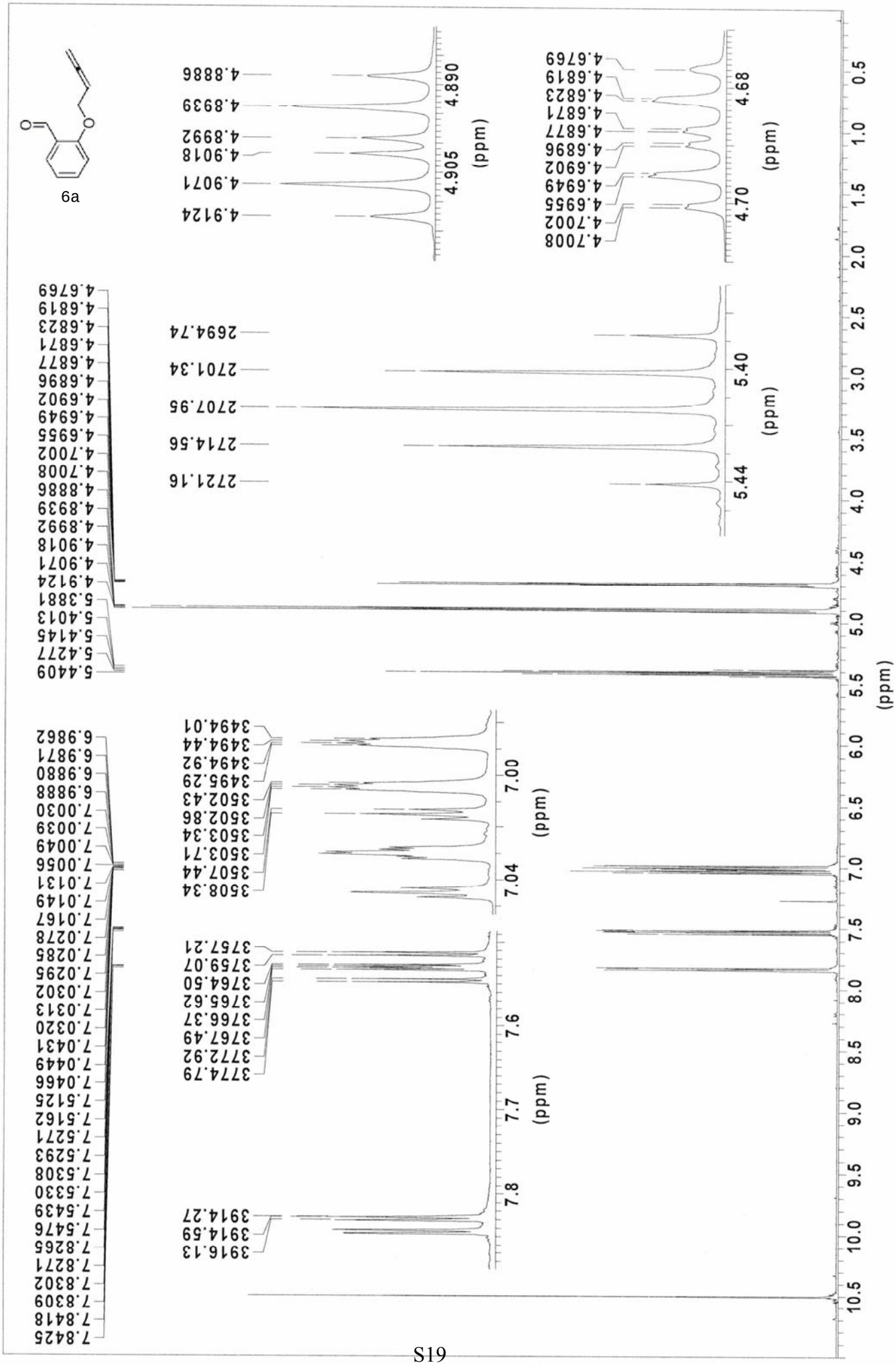
EI-MS [m/z] (%), 188.0 ($M^{+\cdot}$,10); 159.1 (15); 158.0 (100); 130.0 (10); 129.0 (20); 127.9 (35), 114.9 (30); 101.9 (10), 77.9 (10); 76.9 (15); 62.9 (10), 50.9 (10).

ESI-HRMS [m/z] calcd for $C_{19}H_{26}NaO_2$ $[M+Na]^+$ 211.0735, found 211.07317.

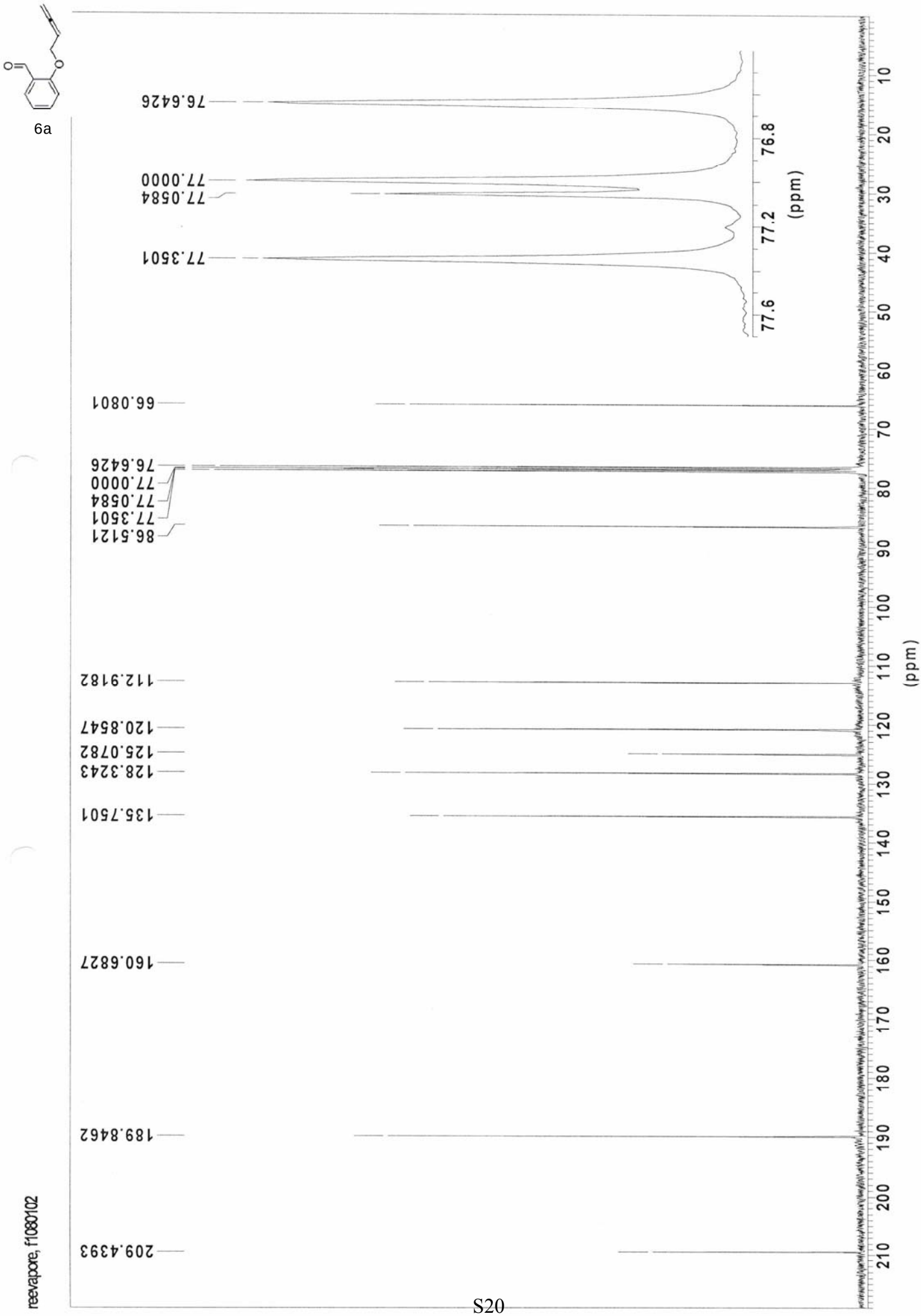
IR (film): ν [cm^{-1}] = 3156, 3009, 2254, 1794, 1472, 1382, 1167, 1084.


Br
5
f10702201
Integral
(ppm)
1.0000
2.0593
2.0667
7.2700
5.4825
5.4697
5.4663
5.4666
5.4531
5.4501
5.4403
5.4369
5.4237
4.9524
4.9481
4.9443
4.9392
4.9353
4.9311
3.9783
3.9744
3.9702
3.9621
3.9578
3.9540
2741.94
2735.55
2733.84
2728.94
2727.24
2725.75
2720.84
2719.14
2712.53
2476.83
2474.70
2472.78
2470.22
2468.31
2466.18
1989.66
1987.74
1985.61
1981.56
1979.43
1977.51


Br
5
29.8817
76.7444
77.0000
77.2556
89.2861
209.6555
f1070201
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
(ppm)


6a
(ppm)
7.8425
7.8418
7.8309
7.8302
7.8271
7.8265
7.5476
7.5439
7.5330
7.5308
7.5293
7.5271
7.5162
7.5125
7.0466
7.0449
7.0431
7.0320
7.0313
7.0302
7.0295
7.0285
7.0278
7.0167
7.0149
7.0131
7.0056
7.0049
7.0039
7.0030
6.9888
6.9880
6.9871
6.9862
5.4409
5.4277
5.4145
5.4013
5.3881
4.9124
4.9071
4.9018
4.8992
4.8939
4.8886
4.7008
4.7002
4.6955
4.6949
4.6902
4.6896
4.6877
4.6871
4.6823
4.6819
4.6769
3916.13
3914.59
3914.27
3774.79
3772.92
3767.49
3766.37
3765.62
3764.50
3759.07
3757.21
3508.34
3507.44
3503.71
3503.34
3502.86
3502.43
3495.29
3494.92
3494.44
3494.01
2721.16
2714.56
2707.95
2701.34
2694.74
4.9124
4.9071
4.9018
4.8992
4.8939
4.8886
4.7008
4.7002
4.6955
4.6949
4.6902
4.6896
4.6877
4.6871
4.6823
4.6819
4.6769

f1080102


6a
reevapore, f1080102
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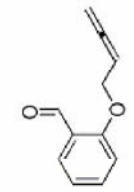
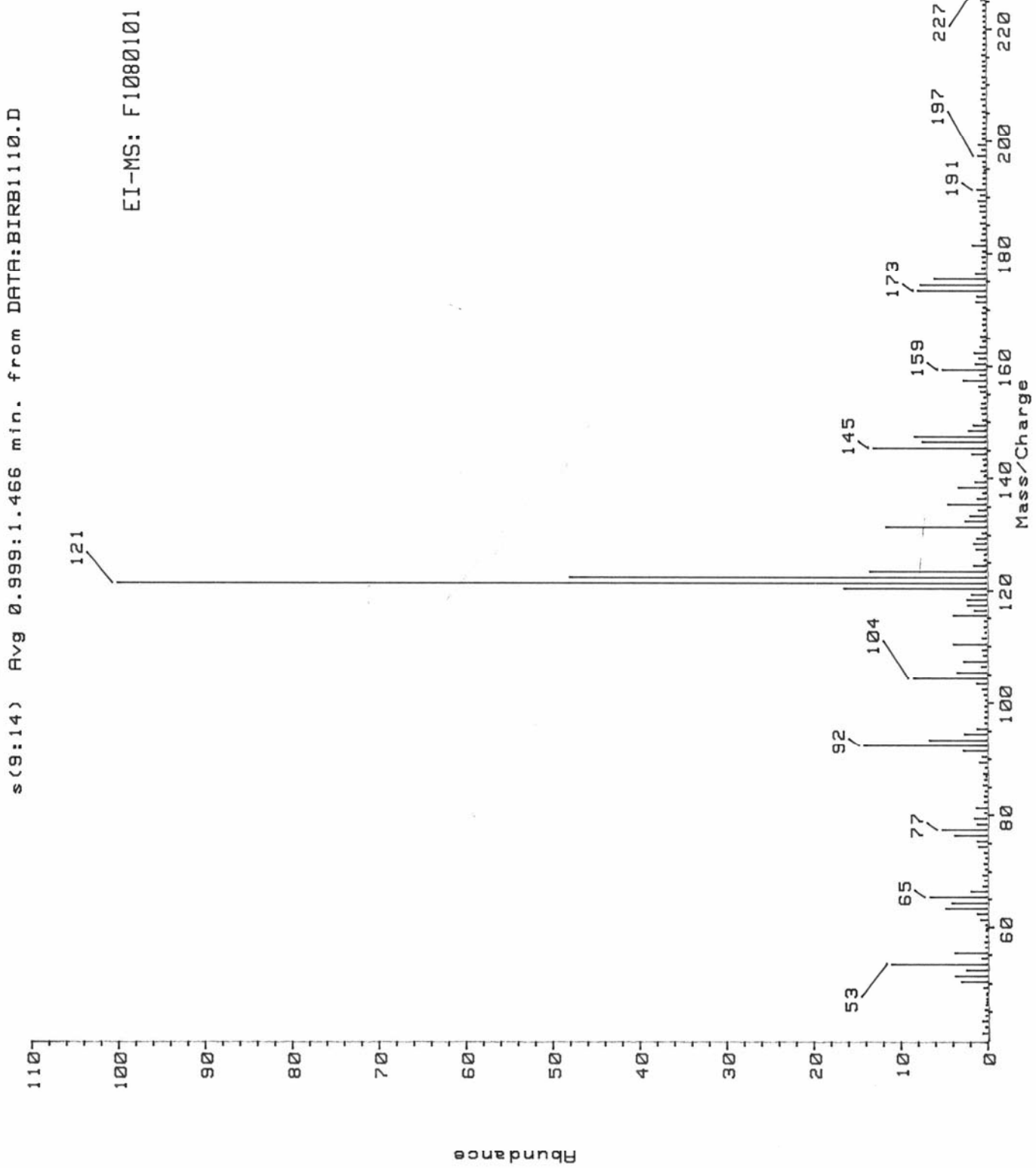
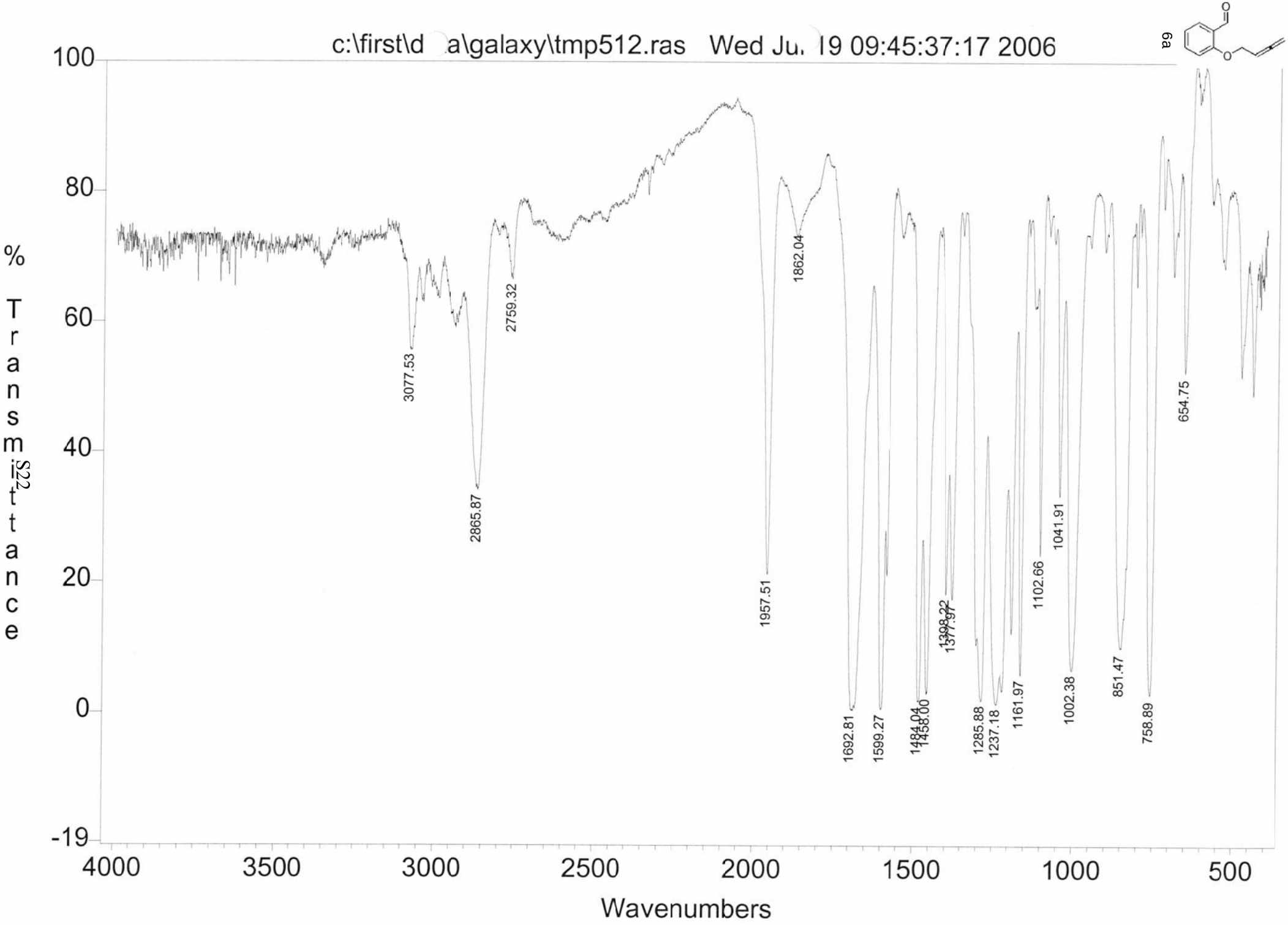
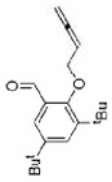
189.8462
160.6827
135.7501
128.3243
125.0782
120.8547
112.9182
86.5121
77.3501
77.0584
77.0000
76.6426
66.0801
77.3501
77.0584
77.0000
76.6426
77.6
77.2
76.8
(ppm)
(ppm)



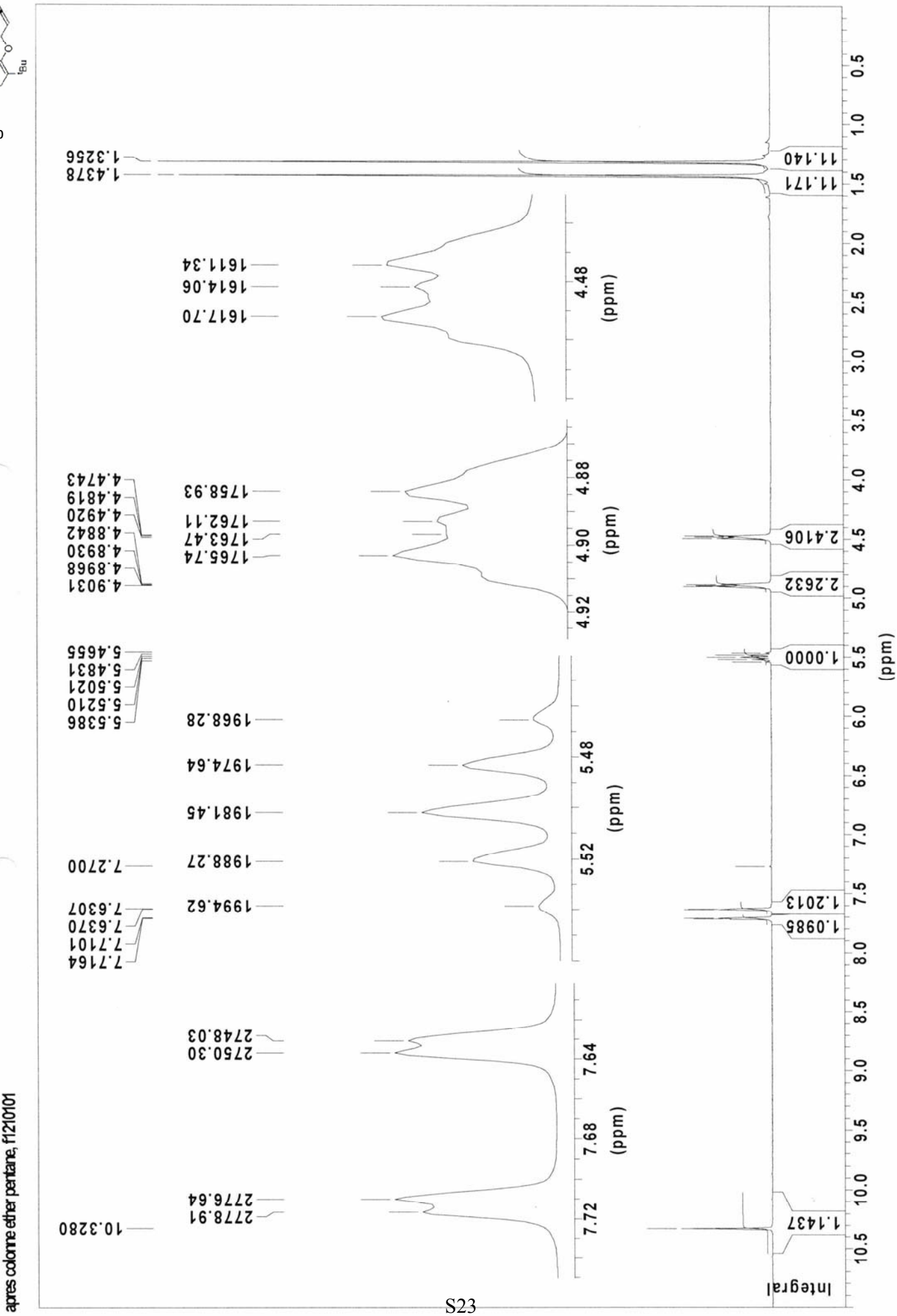
6a


s(9:14) Avg 0.999:1.466 min. from DATA:BIRB1110.D
EI-MS: F1080101
Mass/Charge
Abundance
121
145
159
173
191
197
227
243
249
104
92
77
65
53


c:\first\d a\galaxy\tmp512.ras Wed Ju. 19 09:45:37:17 2006
6a
% Transmittance
Wavenumbers
100
80
60
40
20
0
-19
4000
3500
3000
2500
2000
1500
1000
500
3077.53
2865.87
2759.32
1957.51
1862.04
1692.81
1599.27
1484.04
1458.00
1398.22
1377.97
1285.88
1237.18
1161.97
1102.66
1041.91
1002.38
851.47
758.89
654.75



6b


apres colonne ether pentane, f1210101
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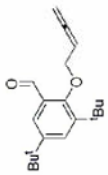
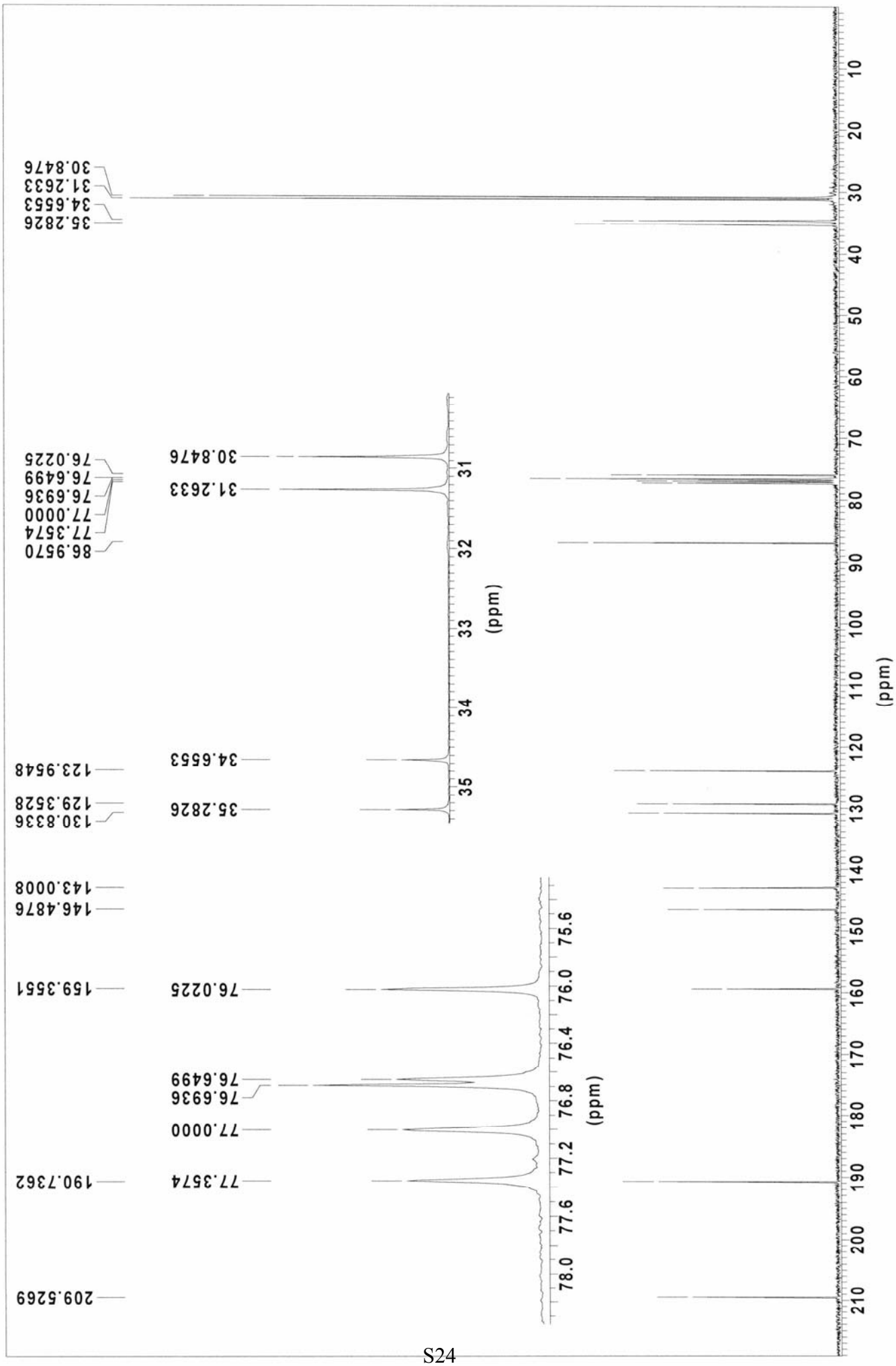
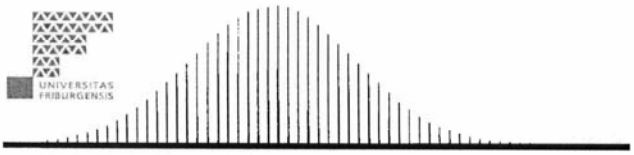
2776.64
7.72
7.68
7.64
(ppm)
2750.30
2748.03
7.7164
7.7101
7.6370
7.6307
1994.62
1988.27
7.2700
1981.45
1974.64
1968.28
5.52
5.48
(ppm)
5.5386
5.5210
5.5021
5.4831
5.4655
4.9031
4.8968
4.8930
4.8842
4.8920
4.8819
4.8743
1765.74
1763.47
1762.11
1758.93
4.92
4.90
4.88
(ppm)
1617.70
1614.06
1611.34
4.48
(ppm)
1.3256
1.4378
11.140
11.171
2.4106
2.2632
1.0000
1.2013
1.0985
1.1437
Integral
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
10.0
10.5
(ppm)

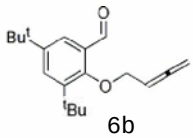


6b



colonne ether pentane, f1210101





6b

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F1210101

XMASS Mass Analysis for /Data/UNI_FR/BIRB1433_ESI/3/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

Ion mass = 309.1825880

Charge = +1

#	C	H	O	Na	mass	DBE	error

*** Mass Analysis for mass 309.1825880

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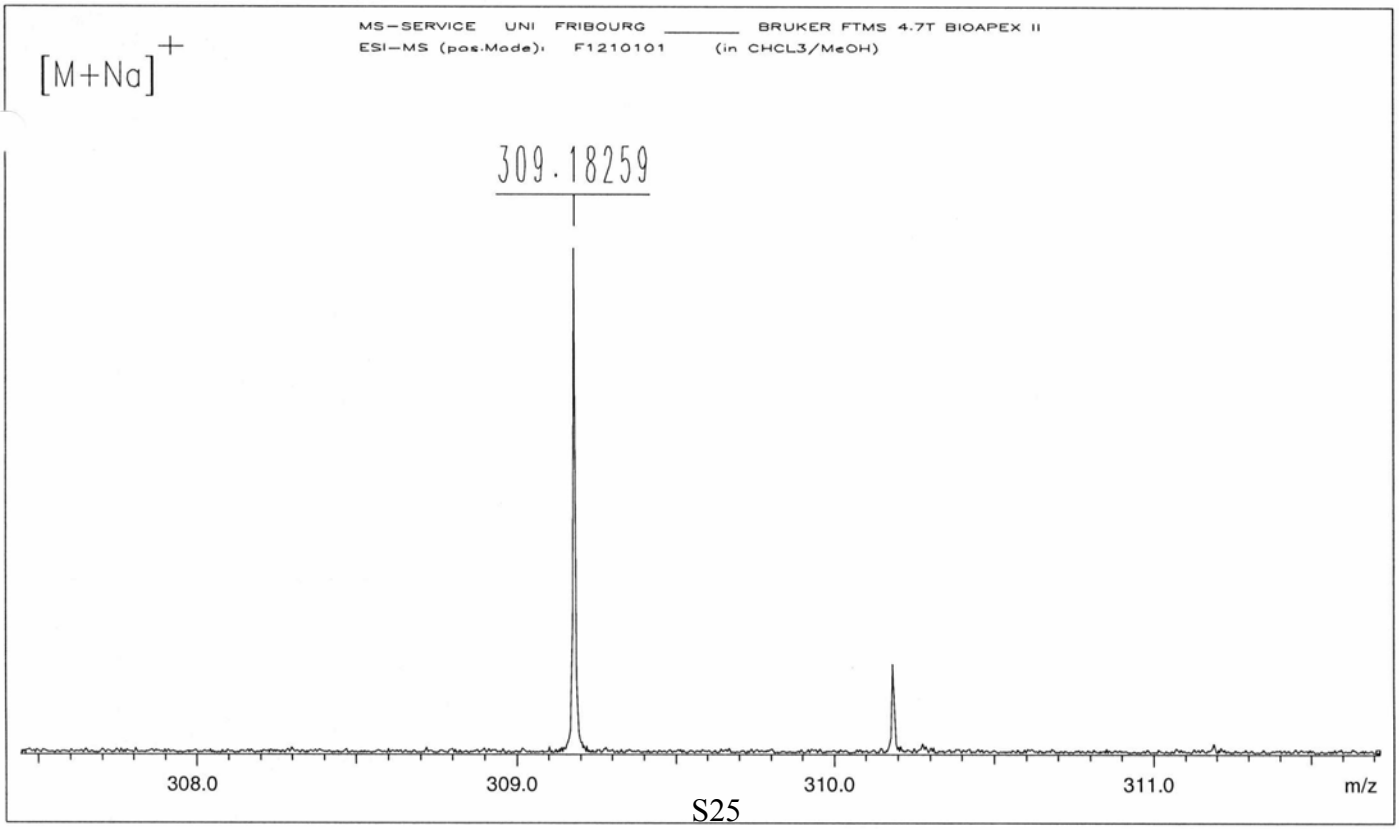
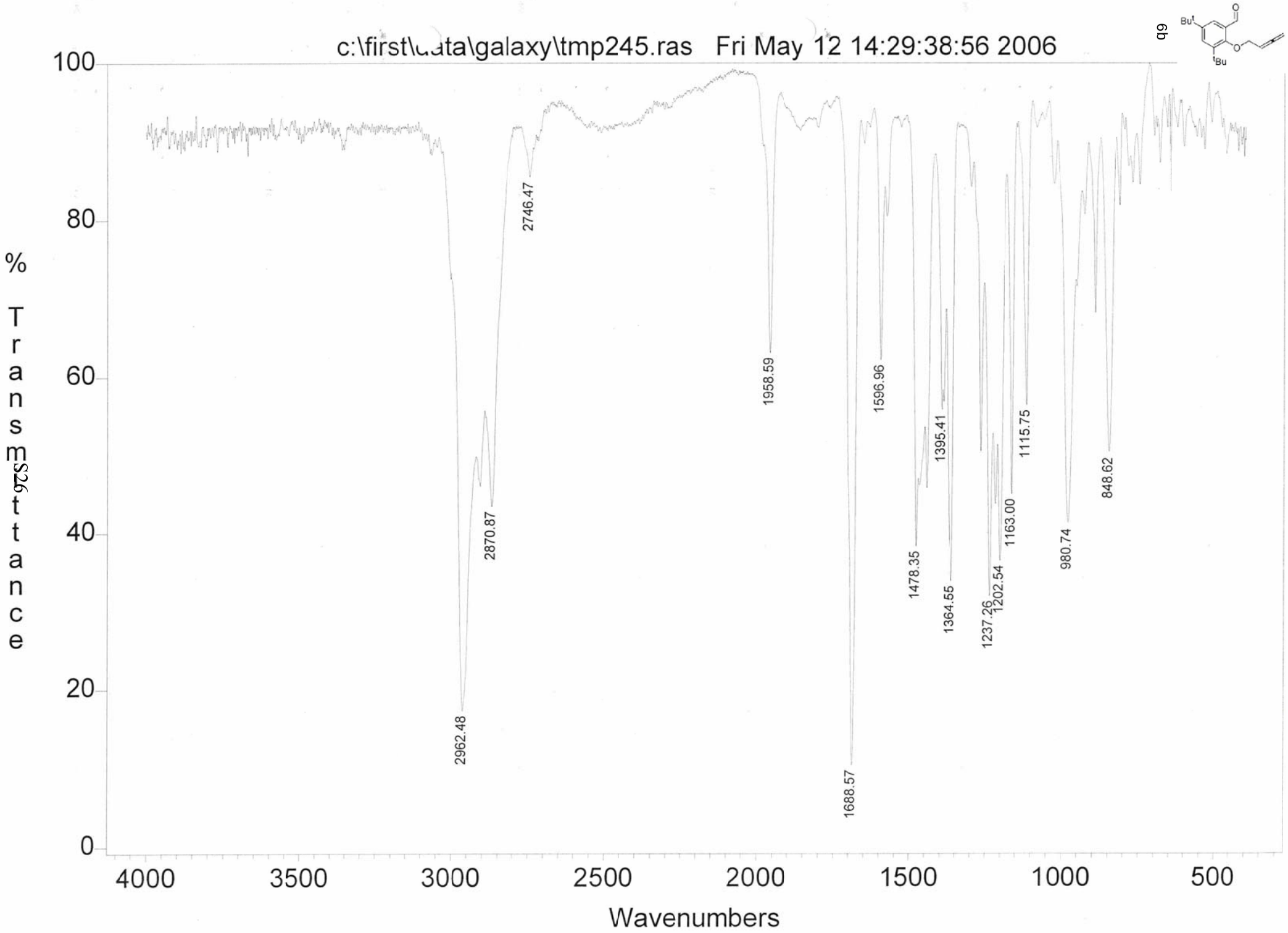
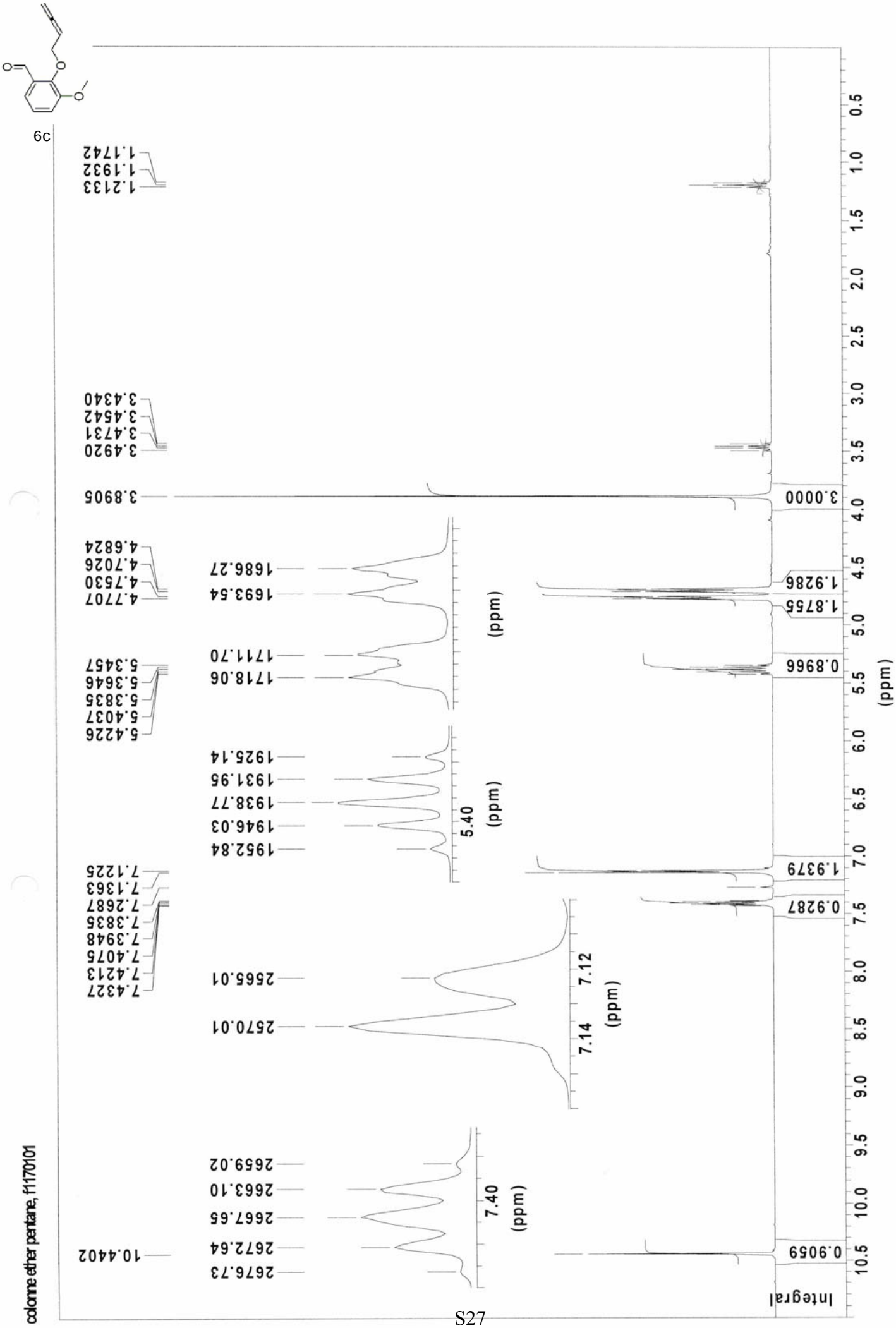
2	21	25	2	0	309.1849065	9.5	2.318e-03
3	12	30	7	1	309.1883744	-2.5	5.786e-03
4	14	29	7	0	309.1907797	0.5	8.192e-03
5	17	25	5	0	309.1696503	5.5	1.294e-02
6	15	26	5	1	309.1672450	2.5	1.534e-02
7	16	30	4	1	309.2036305	1.5	2.104e-02
8	18	29	4	0	309.2060358	4.5	2.345e-02
9	13	25	8	0	309.1543942	1.5	2.819e-02
10	11	26	8	1	309.1519889	-1.5	3.060e-02

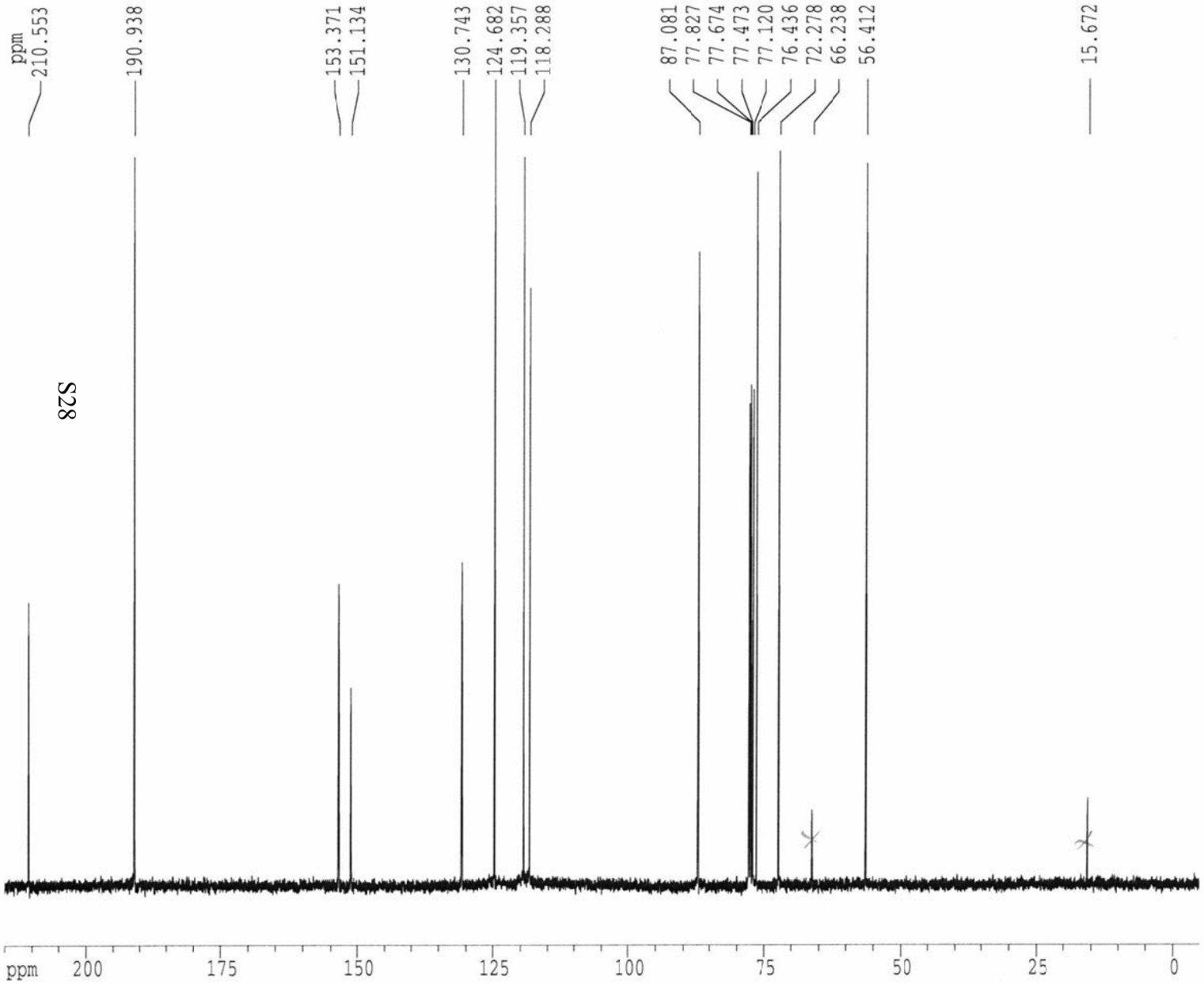


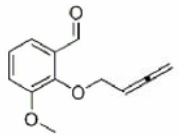
/Data/UNI_FR/BIRB1433_ESI/3/pdata/1 FTMS USER Thu May 18 13:45:48 2006


c:\first\data\galaxy\tmp245.ras Fri May 12 14:29:38:56 2006
6b
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
2962.48
2870.87
2746.47
1958.59
1688.57
1596.96
1478.35
1395.41
1364.55
1237.26
1202.54
1163.00
1115.75
980.74
848.62


6c
colonne ether pentane, f1170101
10.4402
2676.73
2672.64
2667.65
2663.10
2659.02
7.40
(ppm)
2570.01
2565.01
7.14
7.12
(ppm)
7.4327
7.4213
7.4075
7.3948
7.3835
7.2687
7.1363
7.1225
1952.84
1946.03
1938.77
1931.95
1925.14
5.40
(ppm)
5.4226
5.4037
5.3835
5.3646
5.3457
1718.06
1711.70
1693.54
1686.27
(ppm)
4.7707
4.7530
4.7026
4.6824
3.8905
3.4920
3.4731
3.4542
3.4340
1.2133
1.1932
1.1742
Integral
0.9059
1.9379
0.9287
0.8966
1.8755
1.9286
3.0000
(ppm)

colonne ether pentane
C13CPD CDCl3 u frbi 18





9c

Current Data Parameters
NAME f1170101
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 21.05
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PROBHD 5 mm SEI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 21645.021 Hz
FIDRES 0.330277 Hz
AQ 1.5139316 sec
RG 1625.5
DW 23.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 13.60 usec
PL1 -6.00 dB
SFO1 90.5637800 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 16.45 dB
PL13 120.00 dB
SFO2 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5547250 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
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F1 19469.27 Hz
F2P -5.000 ppm
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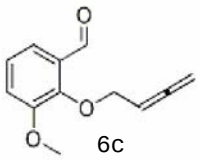
PPMCM 11.00000 ppm/cm
HZCM 996.10199 Hz/cm



6c

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F11701

XMASS Mass Analysis for /Data/UNI_FR/BIRB1340_ESI/5/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

Ion mass = 227.0677510

Charge = +1

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*** Mass Analysis for mass 227.0677510							
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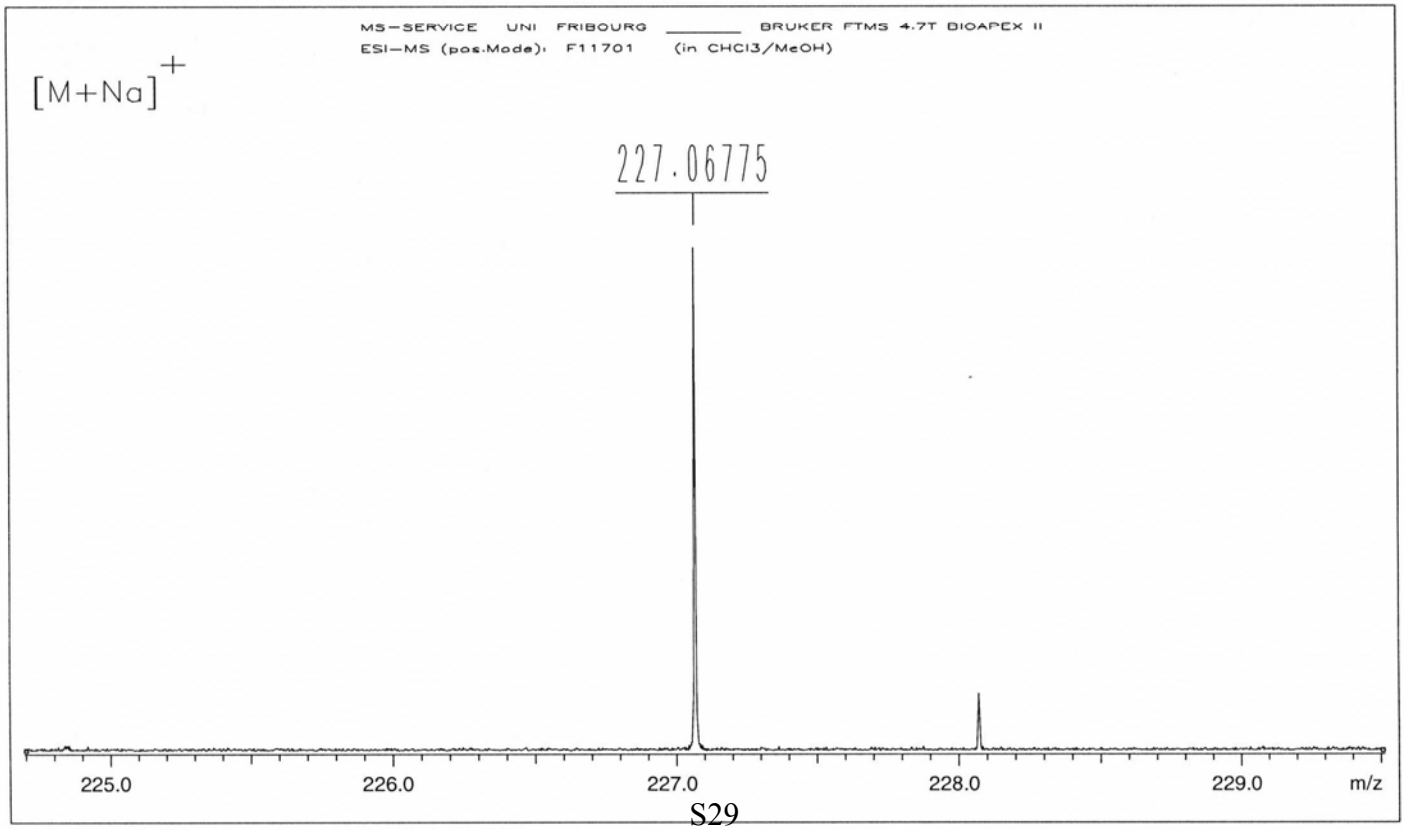
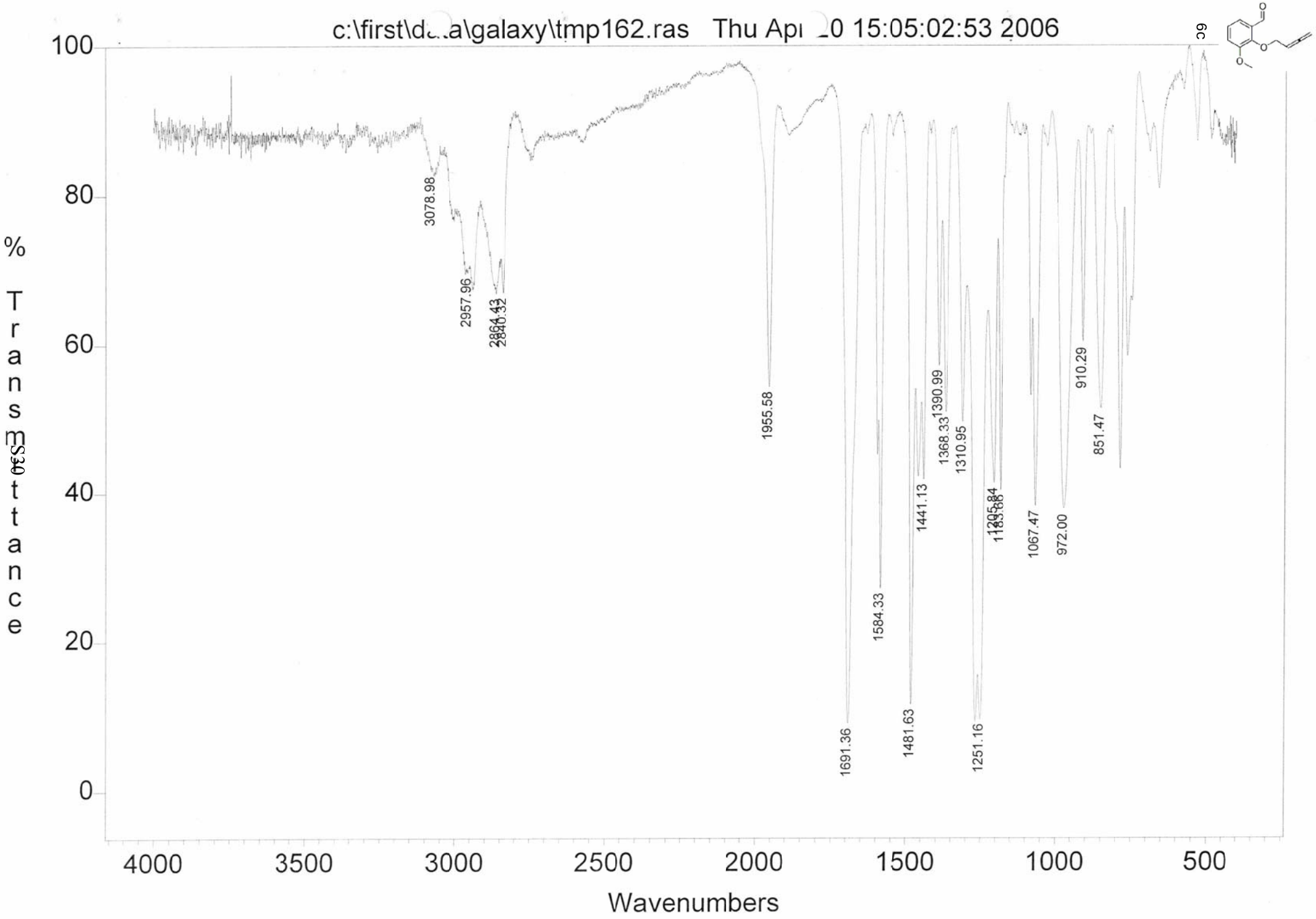
2	14	11	3	0	227.0702706	9.5	2.520e-03
3	5	16	8	1	227.0737386	-2.5	5.988e-03
4	7	15	8	0	227.0761439	0.5	8.393e-03
5	10	11	6	0	227.0550145	5.5	1.274e-02
6	8	12	6	1	227.0526092	2.5	1.514e-02
7	17	7	1	0	227.0491413	14.5	1.861e-02
8	15	8	1	1	227.0467360	11.5	2.102e-02
9	9	16	5	1	227.0889947	1.5	2.124e-02
10	11	15	5	0	227.0914000	4.5	2.365e-02



/Data/UNI_FR/BIRB1340_ESI/5/pdata/1 FTMS USER Wed Apr 26 16:11:46 2006


c:\first\d..a\galaxy\tmp162.ras Thu Ap 0 15:05:02:53 2006
% Transmittance
Wavenumbers
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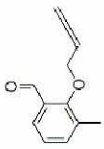
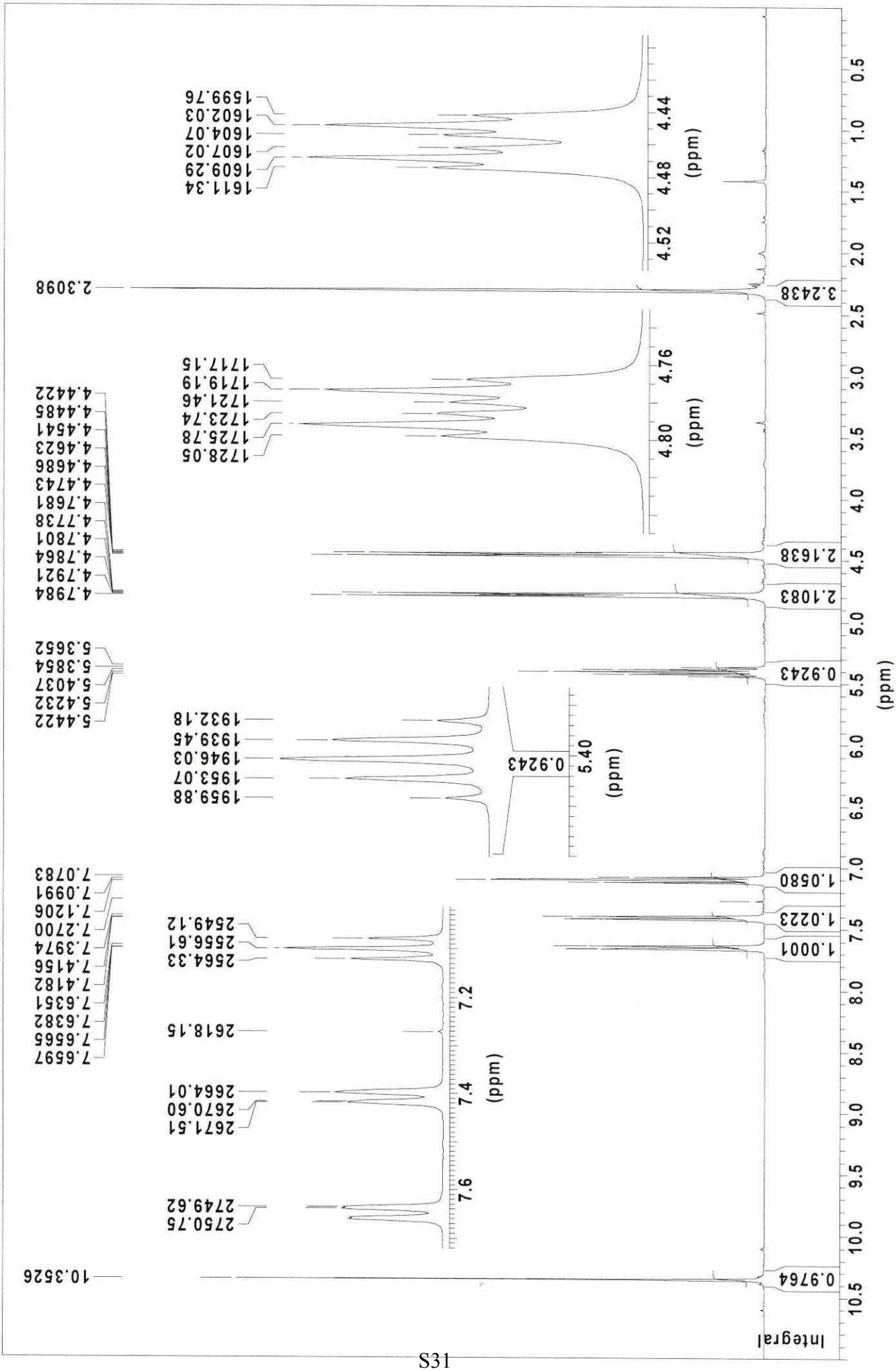
4000
3500
3000
2500
2000
1500
1000
500
3078.98
2957.96
2864.43
2840.32
1955.58
1691.36
1584.33
1481.63
1441.13
1390.99
1368.33
1310.95
1251.16
1067.47
972.00
910.29
851.47



6d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7.6351
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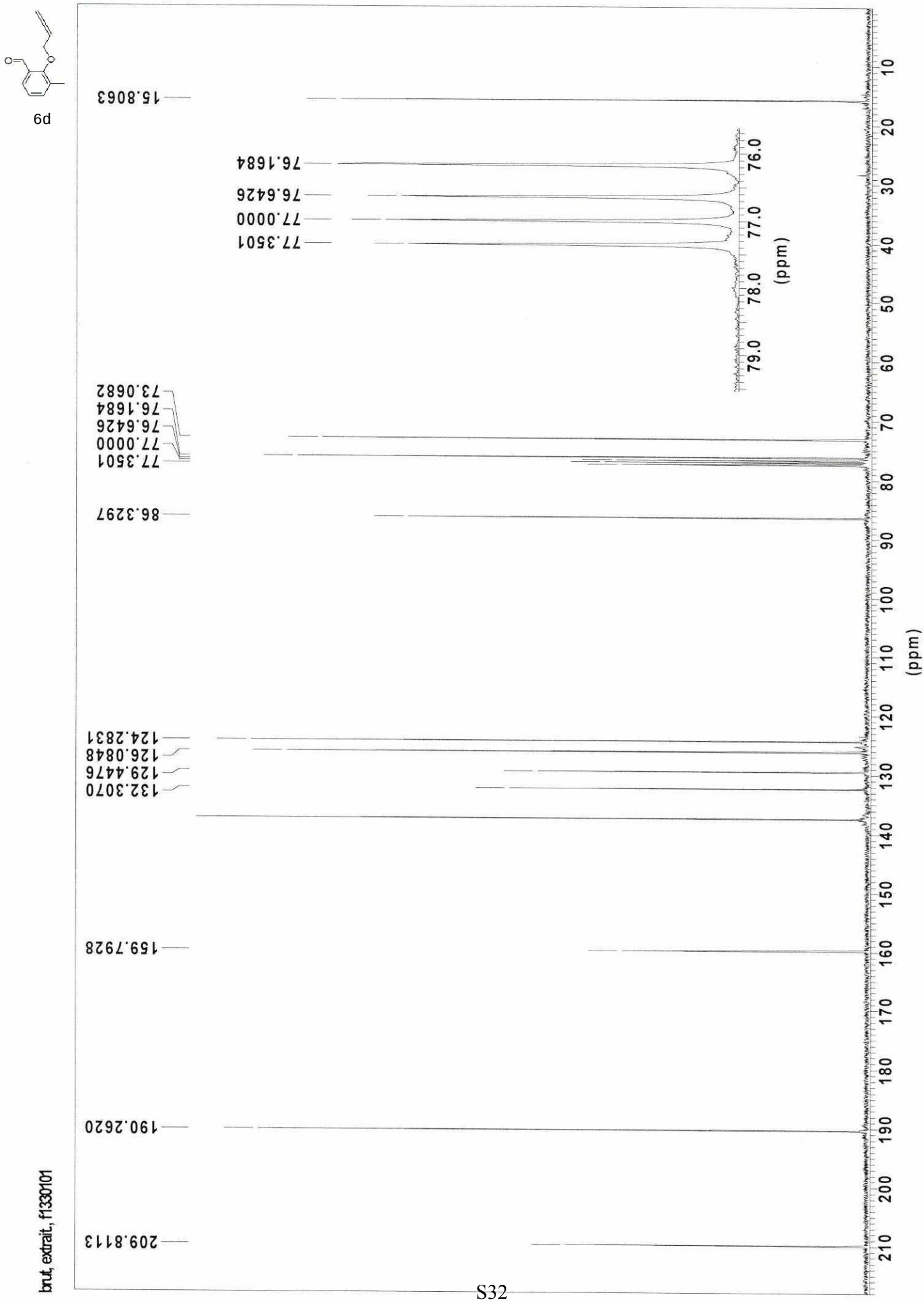
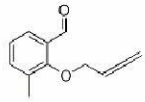
7.4156
7.3974
7.2700
7.1206
7.0991
7.0783
5.4422
5.4232
5.4037
5.3854
5.3652
4.7984
4.7921
4.7864
4.7801
4.7738
4.7681
4.7443
4.4686
4.4623
4.4541
4.4485
4.4422
2.3098
2750.75
2749.62
2671.51
2670.60
2664.01
2618.15
2564.33
2556.61
2549.12
1959.88
1953.07
1946.03
1939.45
1932.18
1728.05
1725.78
1723.74
1721.46
1719.19
1717.15
1611.34
1609.29
1607.02
1604.07
1602.03
1599.76
0.9764
1.0001
1.0223
1.0580
0.9243
2.1083
2.1638
3.2438
Integral
(ppm)


6d
brut, extrait, f1330101
209.8113
190.2620
159.7928
132.3070
129.4476
126.0848
124.2831
86.3297
77.3501
77.0000
76.6426
76.1684
73.0682
15.8063
(ppm)
76.1684
76.6426
77.0000
77.3501
(ppm)



6d

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

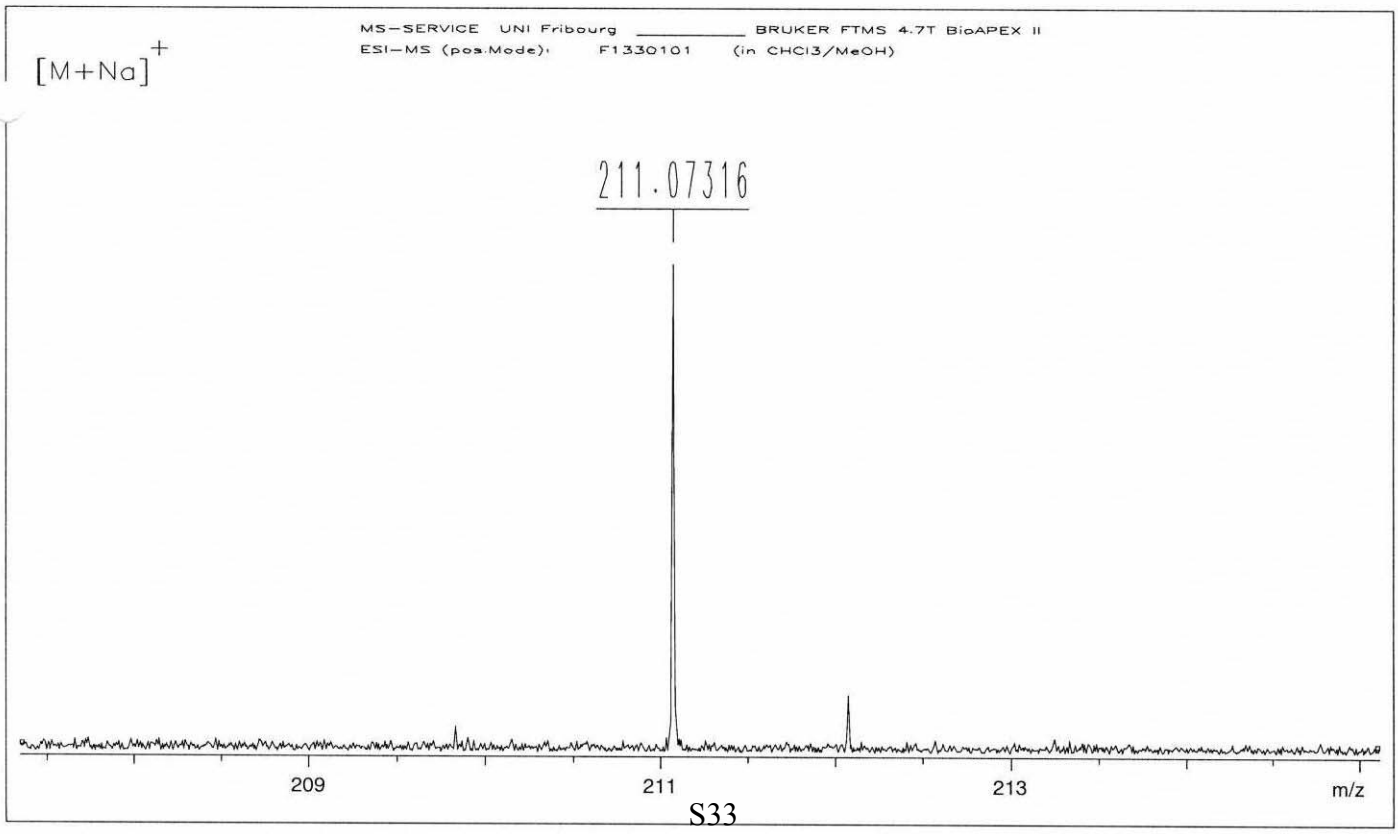
ESI-MS: F1330101

XMASS Mass Analysis for /Data/UNI_FR/BIRB1793_ESI/3/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

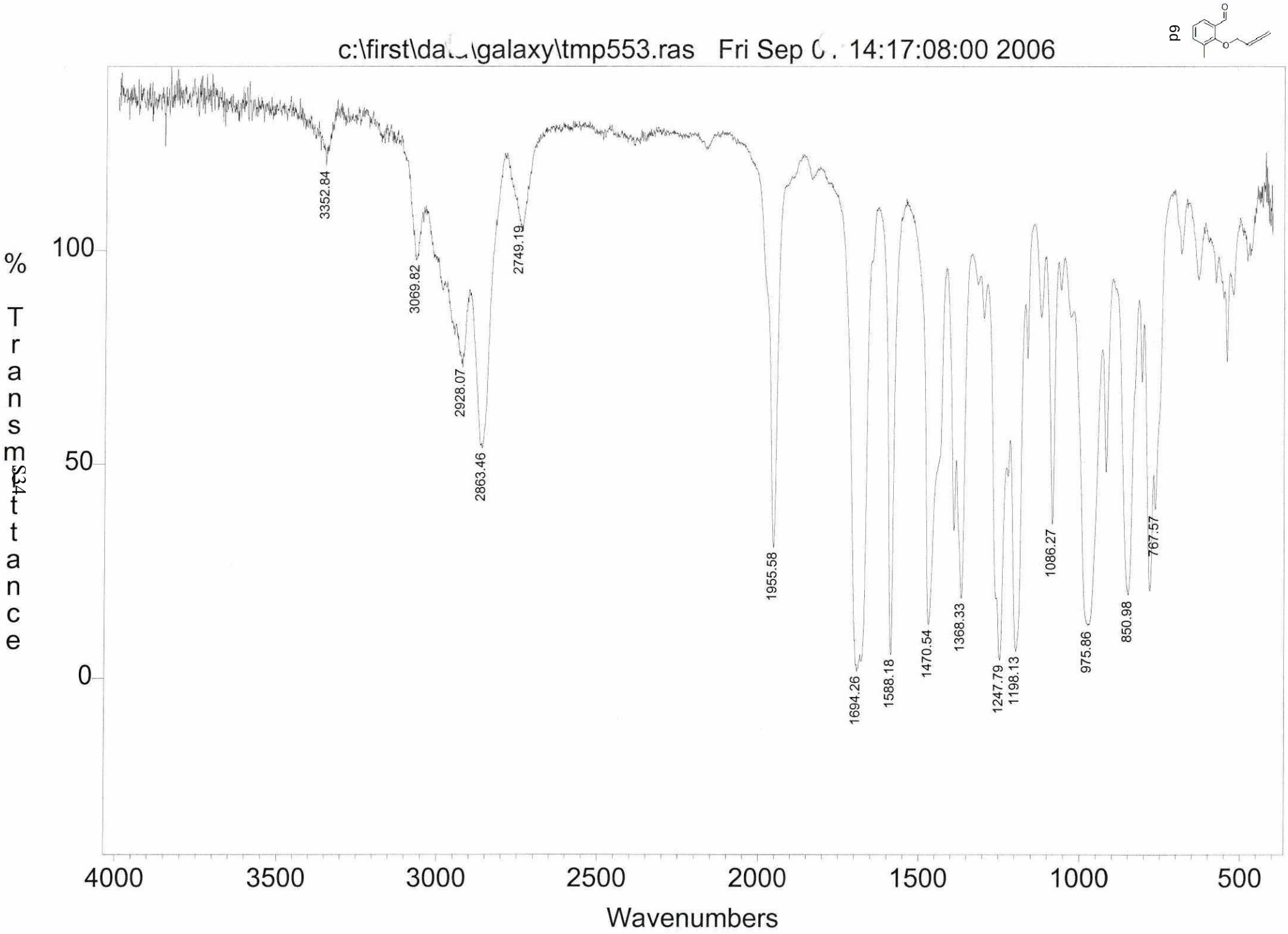
Ion mass = 211.0731630

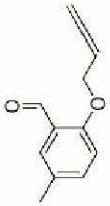
Charge = +1

#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 211.0731630							
1	12	12	2	1	211.0729507	6.5	2.123e-04
2	14	11	2	0	211.0753560	9.5	2.193e-03
3	5	16	7	1	211.0788240	-2.5	5.661e-03
4	7	15	7	0	211.0812293	0.5	8.066e-03
5	10	11	5	0	211.0600999	5.5	1.306e-02
6	8	12	5	1	211.0576946	2.5	1.547e-02
7	9	16	4	1	211.0940801	1.5	2.092e-02
8	11	15	4	0	211.0964854	4.5	2.332e-02
9	6	11	8	0	211.0448437	1.5	2.832e-02
10	13	7	3	0	211.0389705	10.5	3.419e-02

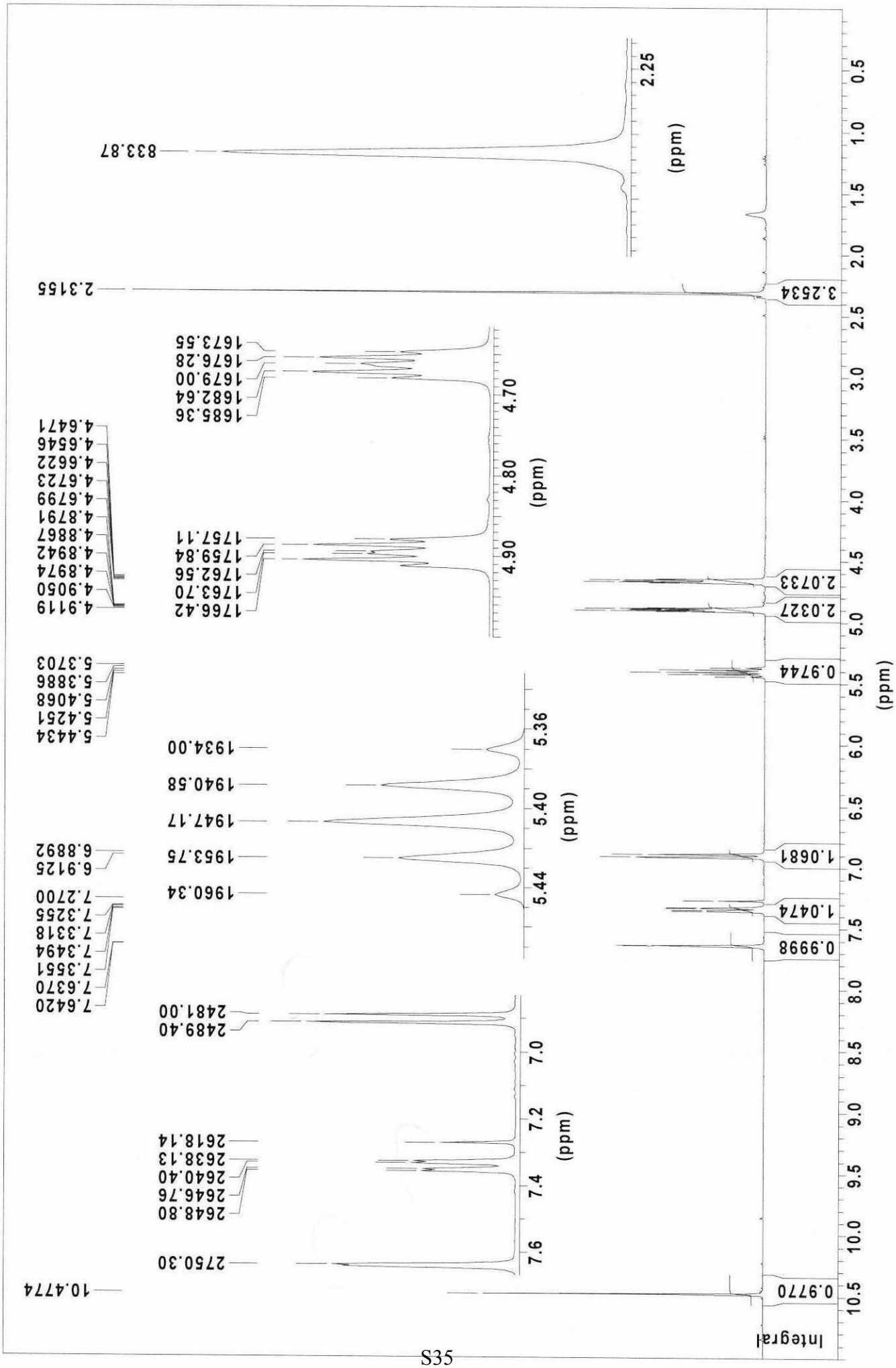


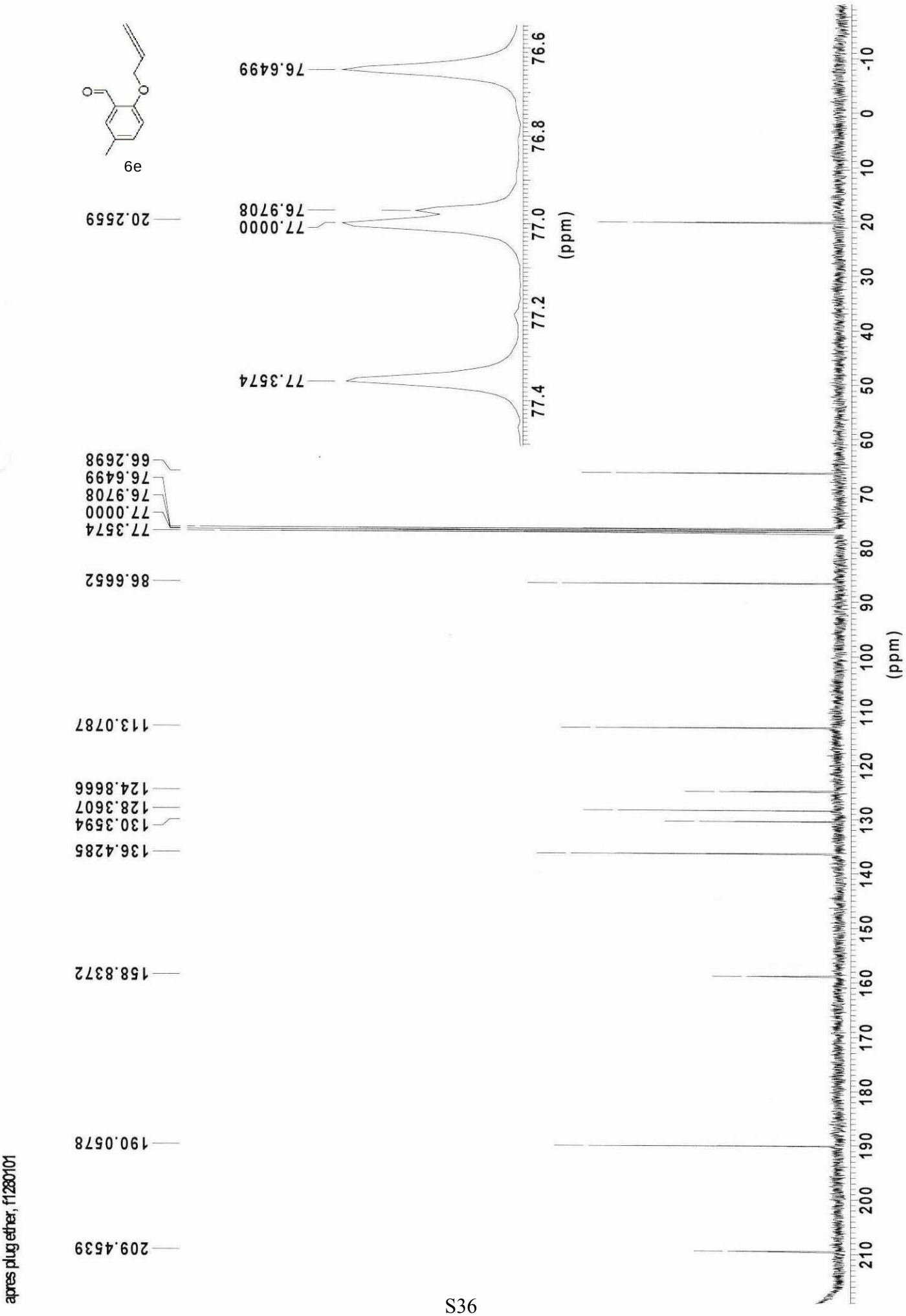
/Data/UNI_FR/BIRB1793_ESI/3/pdata/1 FTMS USER Fri Sep 8 09:50:21 2006

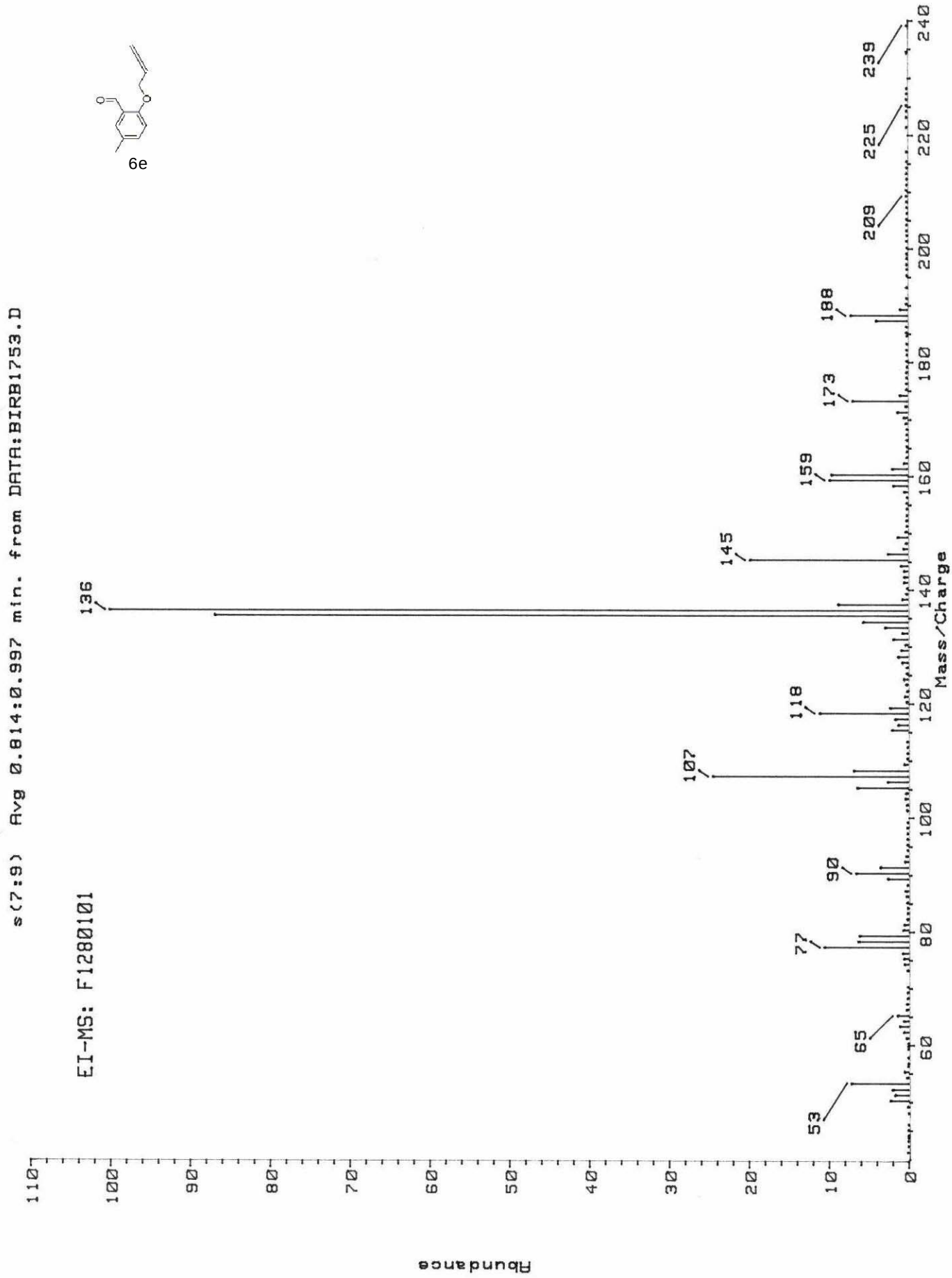

c:\first\data\galaxy\tmp553.ras Fri Sep 0 . 14:17:08:00 2006
6d
% Transmittance
100
50
0
3352.84
3069.82
2928.07
2863.46
2749.19
1955.58
1694.26
1588.18
1470.54
1368.33
1247.79
1198.13
1086.27
975.86
850.98
767.57
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers

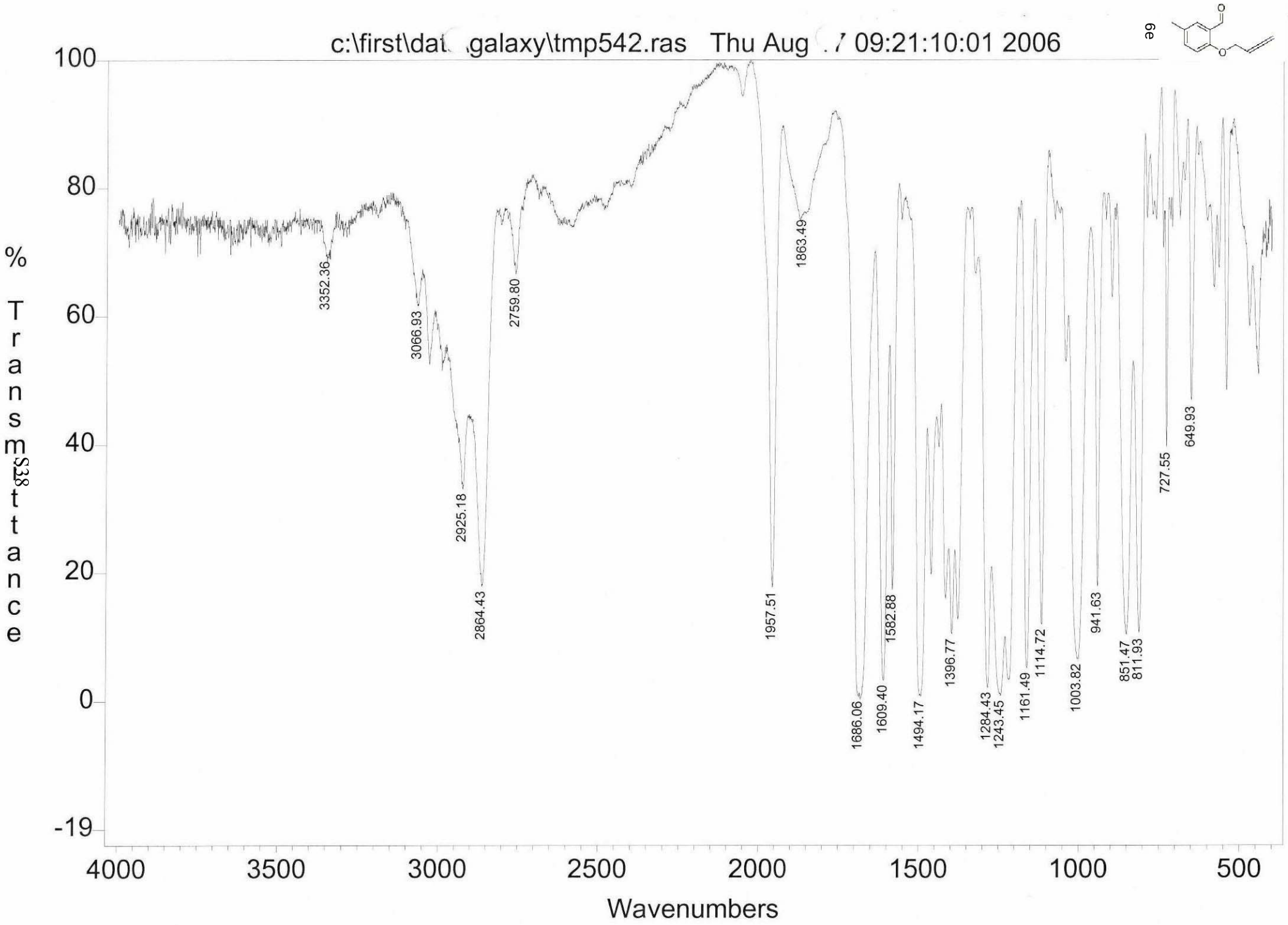


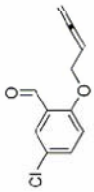
6e


apres plug ether, f1280101
10.4774
2750.30
2648.80
2646.76
2640.40
2638.13
2618.14
2489.40
2481.00
7.6420
7.6370
7.3551
7.3494
7.3318
7.3255
7.2700
6.9125
6.8892
1960.34
1953.75
1947.17
1940.58
1934.00
5.4434
5.4251
5.4068
5.3886
5.3703
4.9119
4.9050
4.8974
4.8942
4.8867
4.8791
4.6799
4.6723
4.6622
4.6546
4.6471
1766.42
1763.70
1762.56
1759.84
1757.11
1685.36
1682.64
1679.00
1676.28
1673.55
2.3155
833.87
(ppm)
Integral
0.9770
0.9998
1.0474
1.0681
0.9744
2.0327
2.0733
3.2534

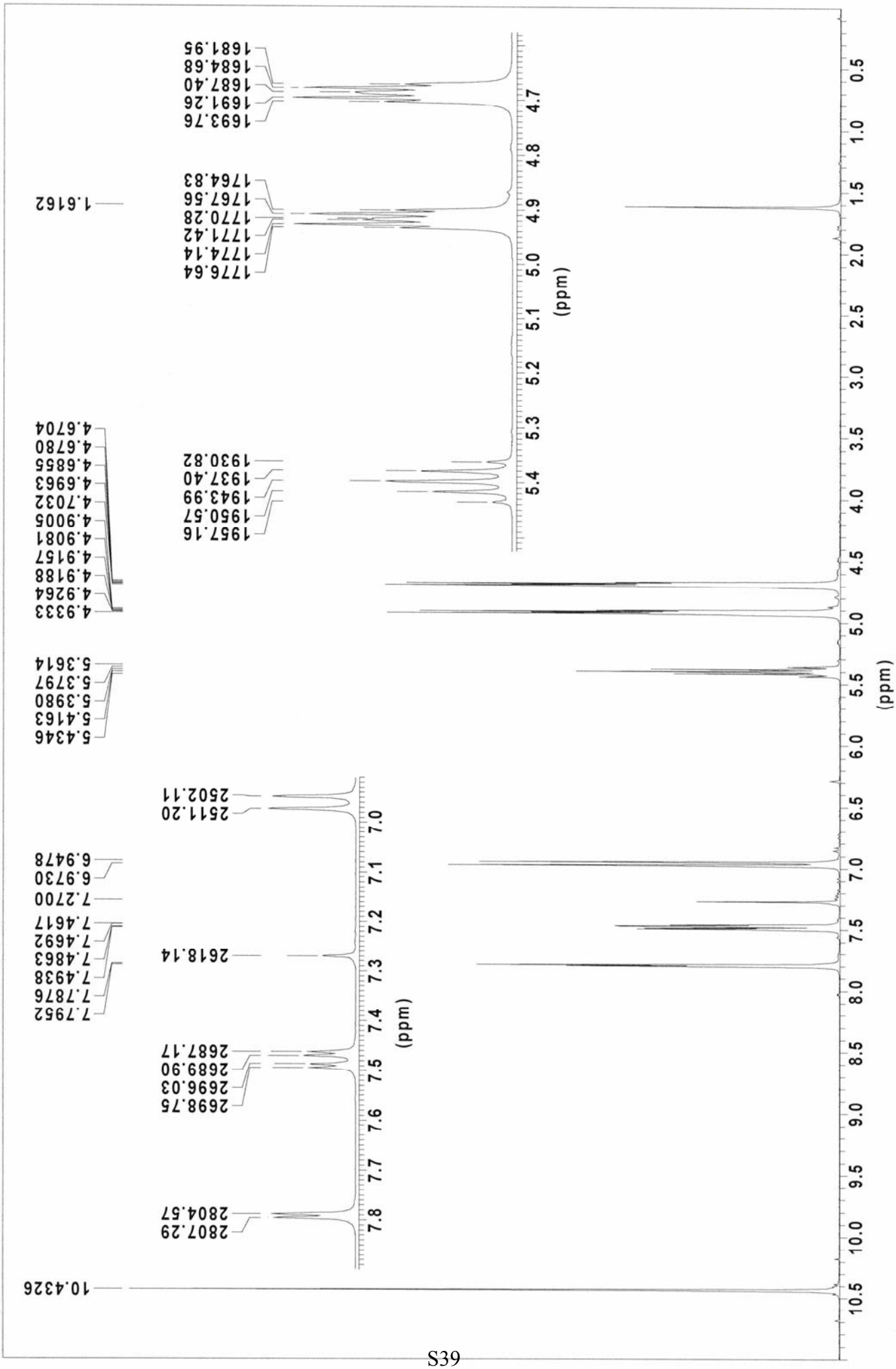

6e
20.2559
66.2698
76.6499
76.9708
77.0000
77.3574
86.6652
113.0787
124.8666
128.3607
130.3594
136.4285
158.8372
190.0578
209.4539
77.3574
77.0000
76.9708
76.6499
(ppm)
(ppm)
apres plug ether, f1280101


6e
s(7:9) Avg 0.814:0.997 min. from DATA:BIRB1753.D
EI-MS: F1280101
Mass/Charge
Abundance
136
145
159
173
188
209
225
239
118
107
90
77
65
53

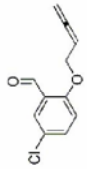

c:\first\dat galaxy\tmp542.ras Thu Aug 7 09:21:10:01 2006
6e
% Transmittance
Wavenumbers
100
80
60
40
20
0
-19
4000
3500
3000
2500
2000
1500
1000
500
3352.36
3066.93
2925.18
2864.43
2759.80
1957.51
1863.49
1686.06
1609.40
1582.88
1494.17
1396.77
1284.43
1243.45
1161.49
1114.72
1003.82
941.63
851.47
811.93
727.55
649.93



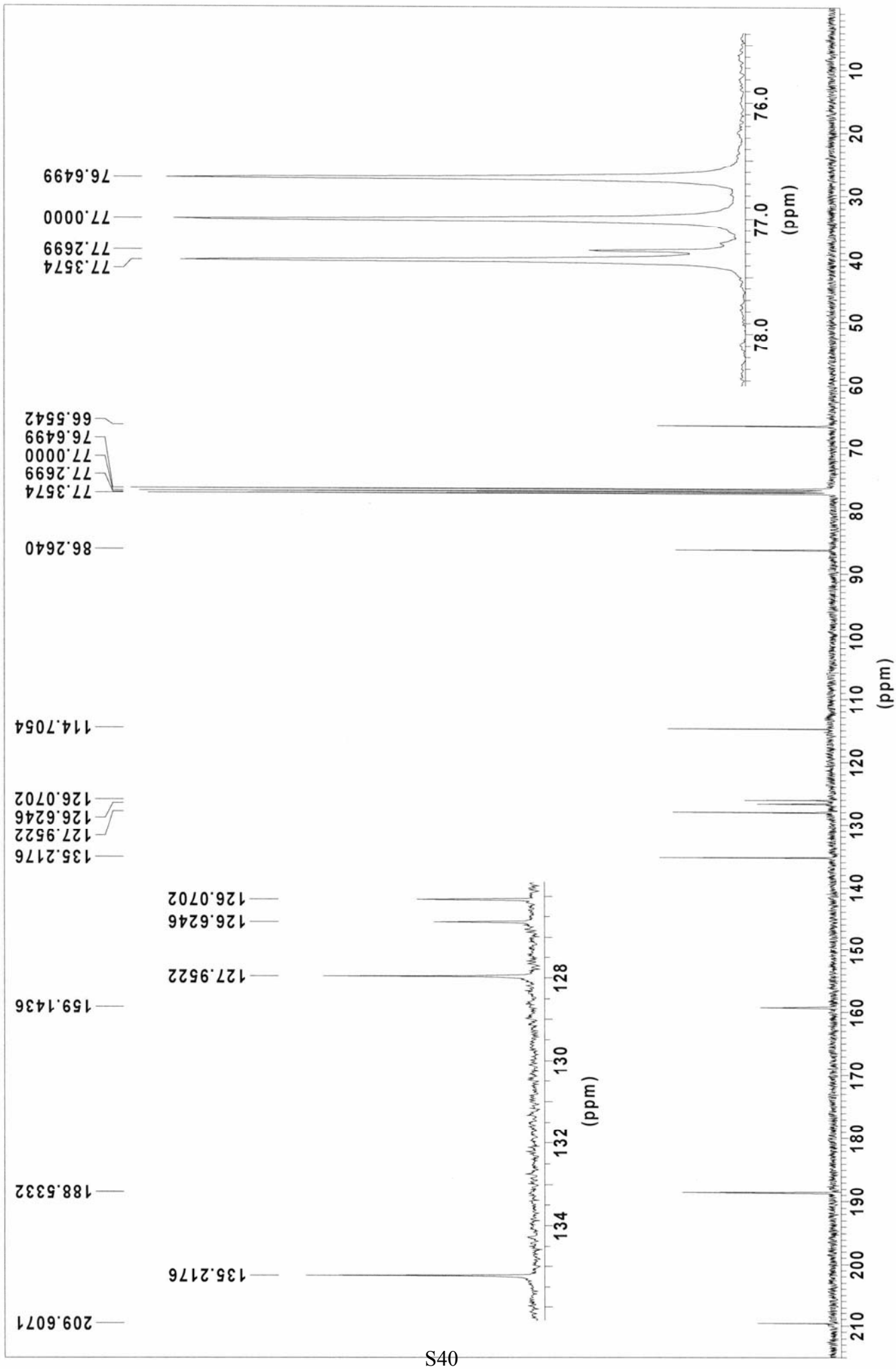
6f

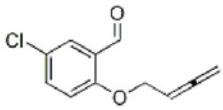


idem 0101, 3 semaine congelateur, f1190201



6f


f1190201
209.6071
188.5332
159.1436
135.2176
126.0702
126.6246
127.9522
135.2176
126.0702
126.6246
127.9522
114.7054
86.2640
77.3574
77.2699
77.0000
76.6499
66.5542
76.6499
77.0000
77.2699
77.3574
(ppm)
(ppm)
(ppm)



6f

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

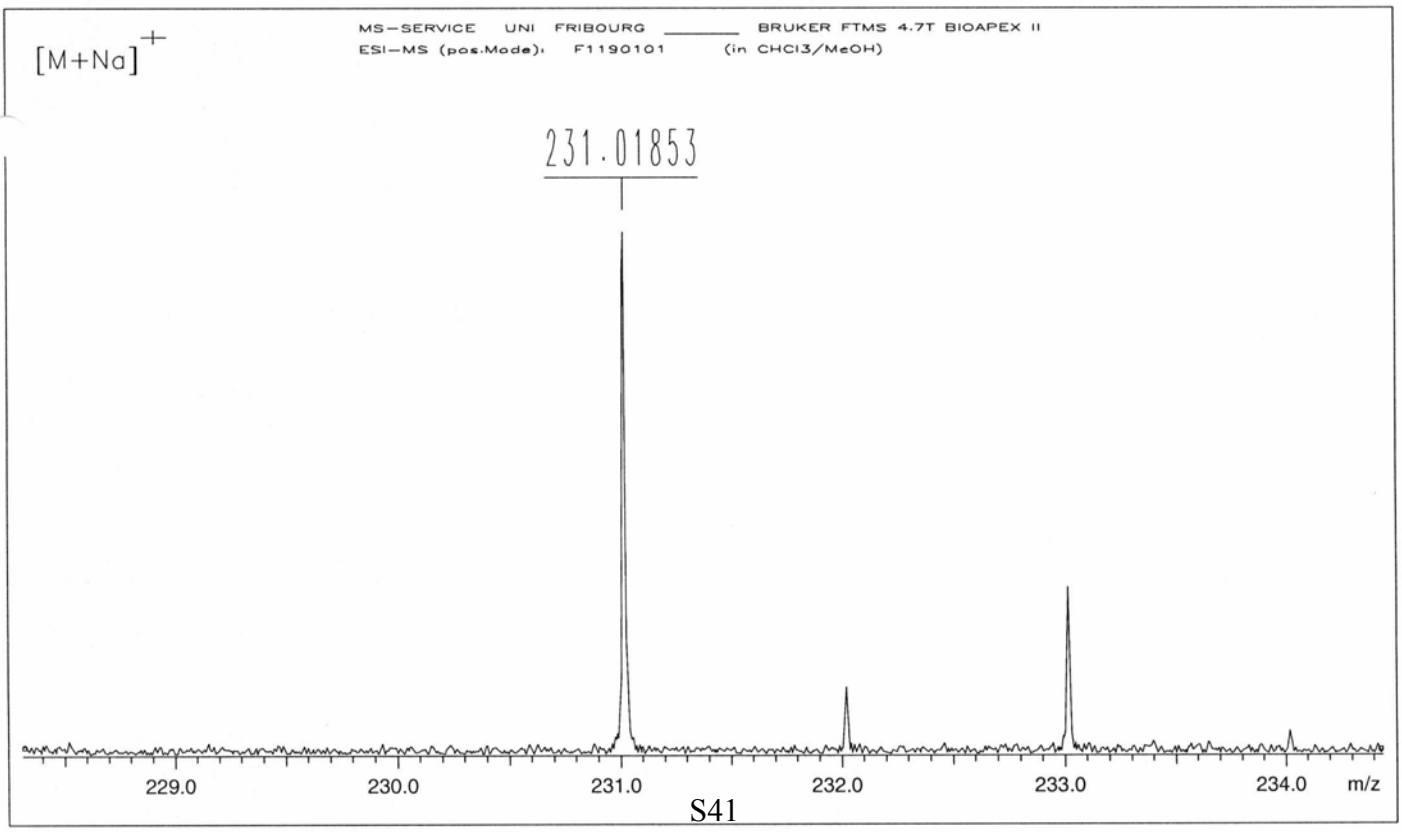
ESI-MS: F1190101

XMASS Mass Analysis for /Data/UNI_FR/BIRB1666_ESI/4/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

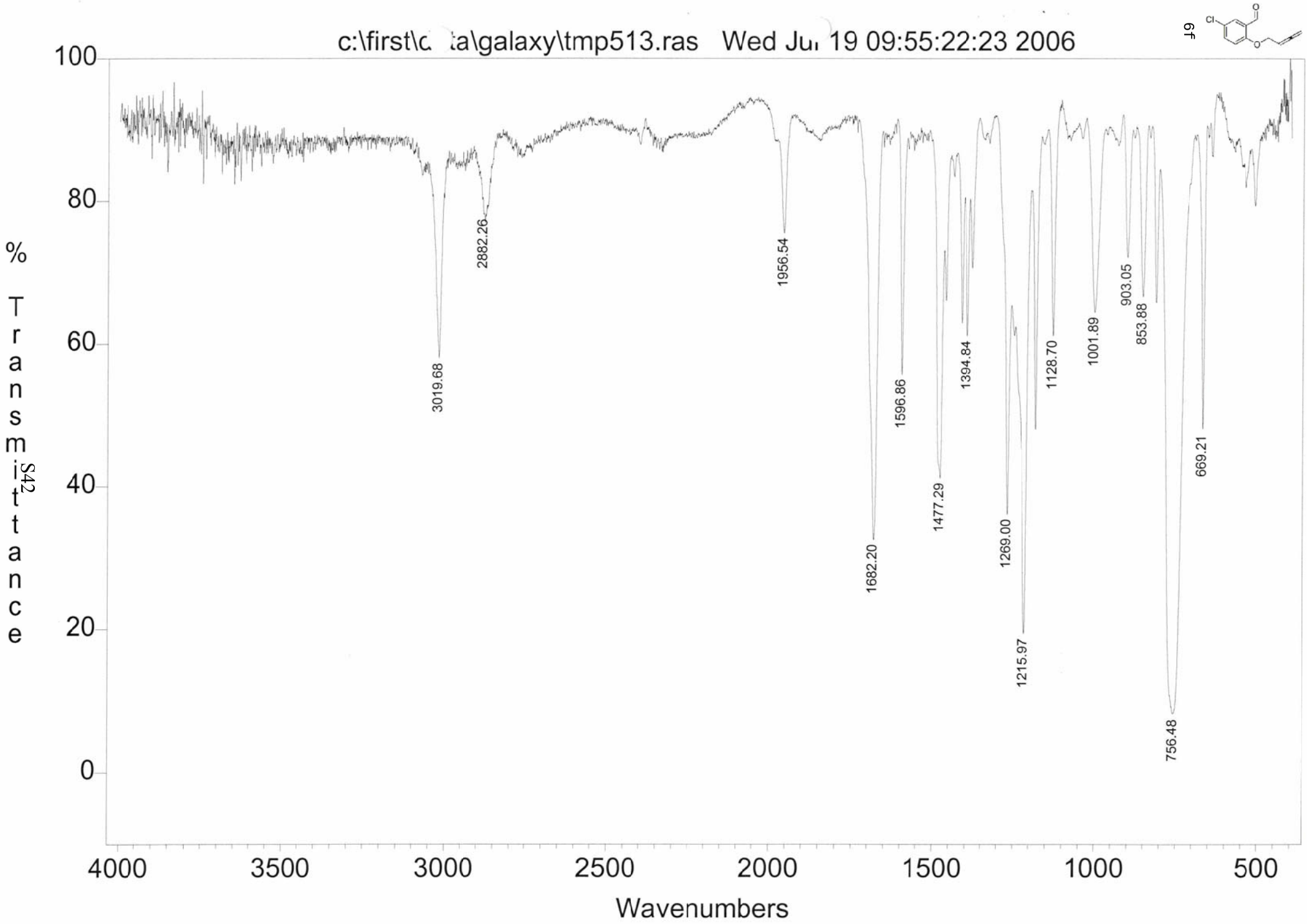
Ion mass = 231.0185350

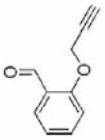
Charge = +1

#	C	H	35Cl	O	Na	mass	DBE	error
*** Mass Analysis for mass 231.0185350								
1	11	9	1	2	1	231.0183283	6.5	2.067e-04
2	13	8	1	2	0	231.0207336	9.5	2.199e-03
3	10	8	0	5	1	231.0263945	6.5	7.859e-03
4	12	7	0	5	0	231.0287998	9.5	1.026e-02
5	15	3	0	3	0	231.0076704	14.5	1.086e-02
6	9	8	1	5	0	231.0054775	5.5	1.306e-02
7	13	4	0	3	1	231.0052651	11.5	1.327e-02
8	7	9	1	5	1	231.0030722	2.5	1.546e-02
9	8	13	1	4	1	231.0394577	1.5	2.092e-02
10	14	8	0	2	1	231.0416506	10.5	2.312e-02

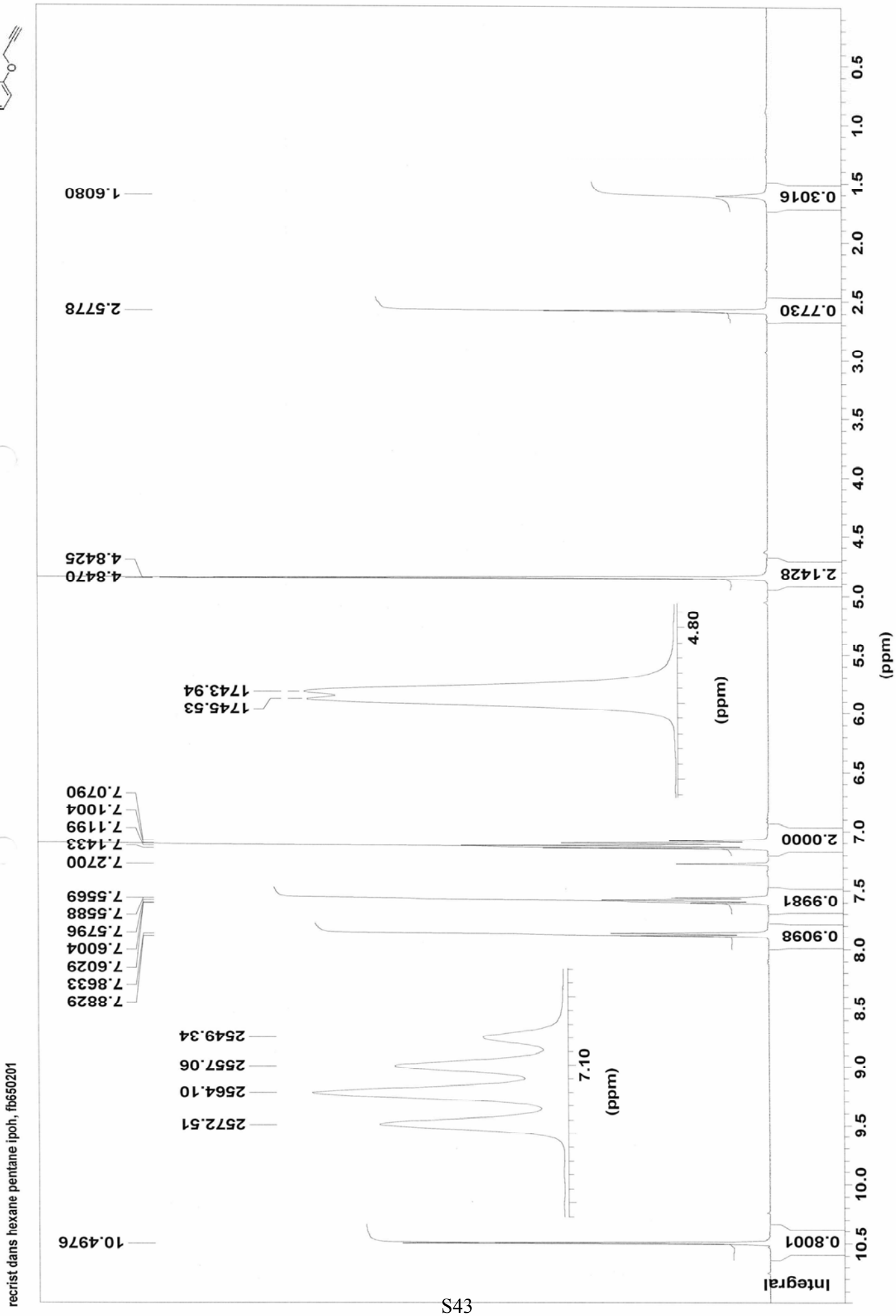


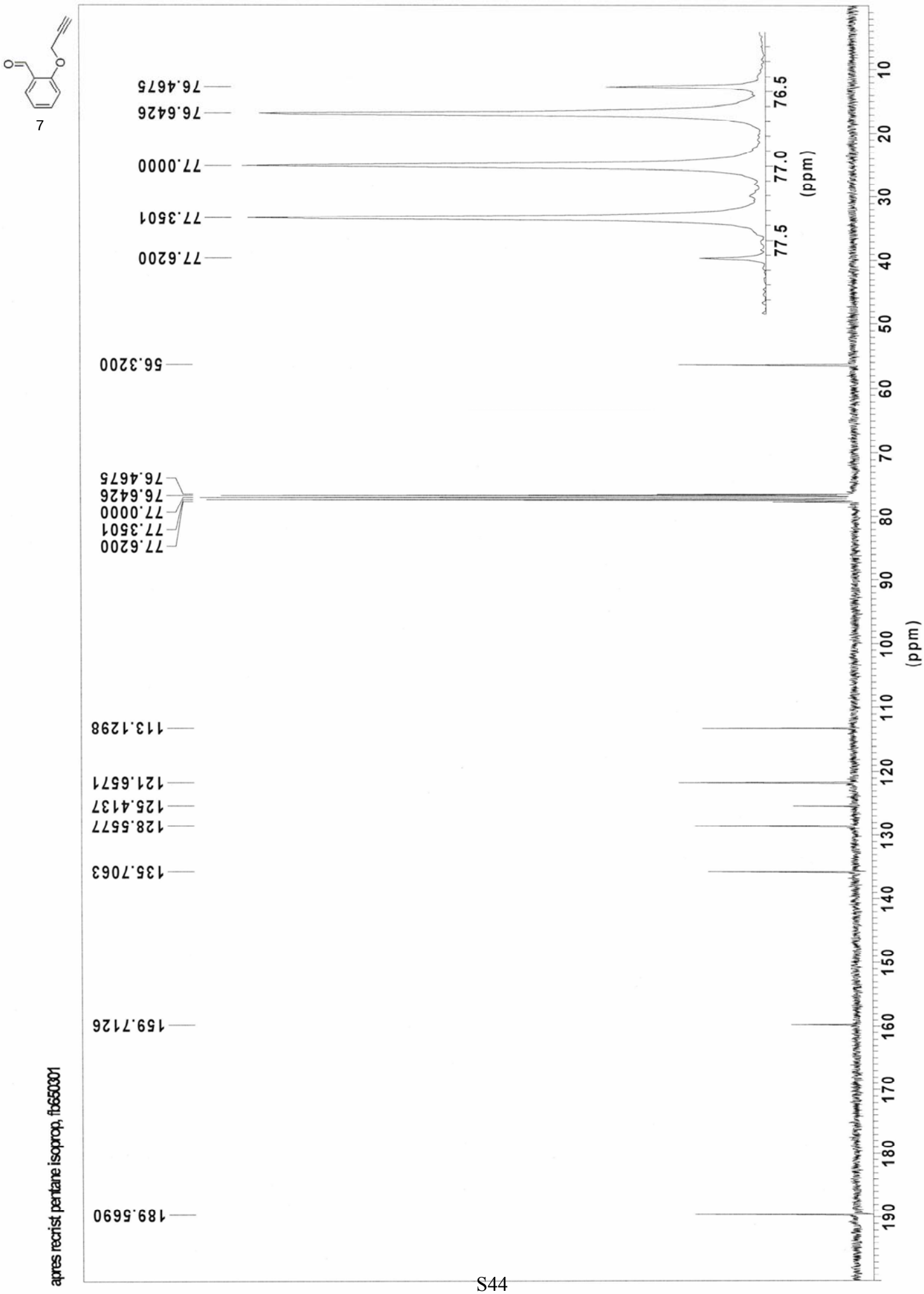
/Data/UNI_FR/BIRB1666_ESI/4/pdata/1 FTMS USER Wed Jul 19 16:19:23 2006

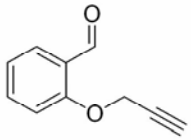

c:\first\c ta\galaxy\tmp513.ras Wed Ju 19 09:55:22:23 2006
6f
% Transmittance
100
80
60
40
20
0
3019.68
2882.26
1956.54
1682.20
1596.86
1477.29
1394.84
1269.00
1215.97
1128.70
1001.89
903.05
853.88
756.48
669.21
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



7


recrist dans hexane pentane ipoh, fb650201
10.4976
7.8829
7.8633
7.6029
7.6004
7.5796
7.5588
7.5569
7.2700
7.1433
7.1199
7.1004
7.0790
4.8470
4.8425
2.5778
1.6080
2572.51
2564.10
2557.06
2549.34
1745.53
1743.94
7.10
(ppm)
4.80
(ppm)
Integral
0.8001
0.9098
0.9981
2.0000
2.1428
0.7730
0.3016
(ppm)


7
apres recrist pentane isoprop, fb650301
189.5690
159.7126
135.7063
128.5577
125.4137
121.6571
113.1298
77.6200
77.3501
77.0000
76.6426
76.4675
56.3200
77.6200
77.3501
77.0000
76.6426
76.4675
(ppm)
(ppm)



7

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

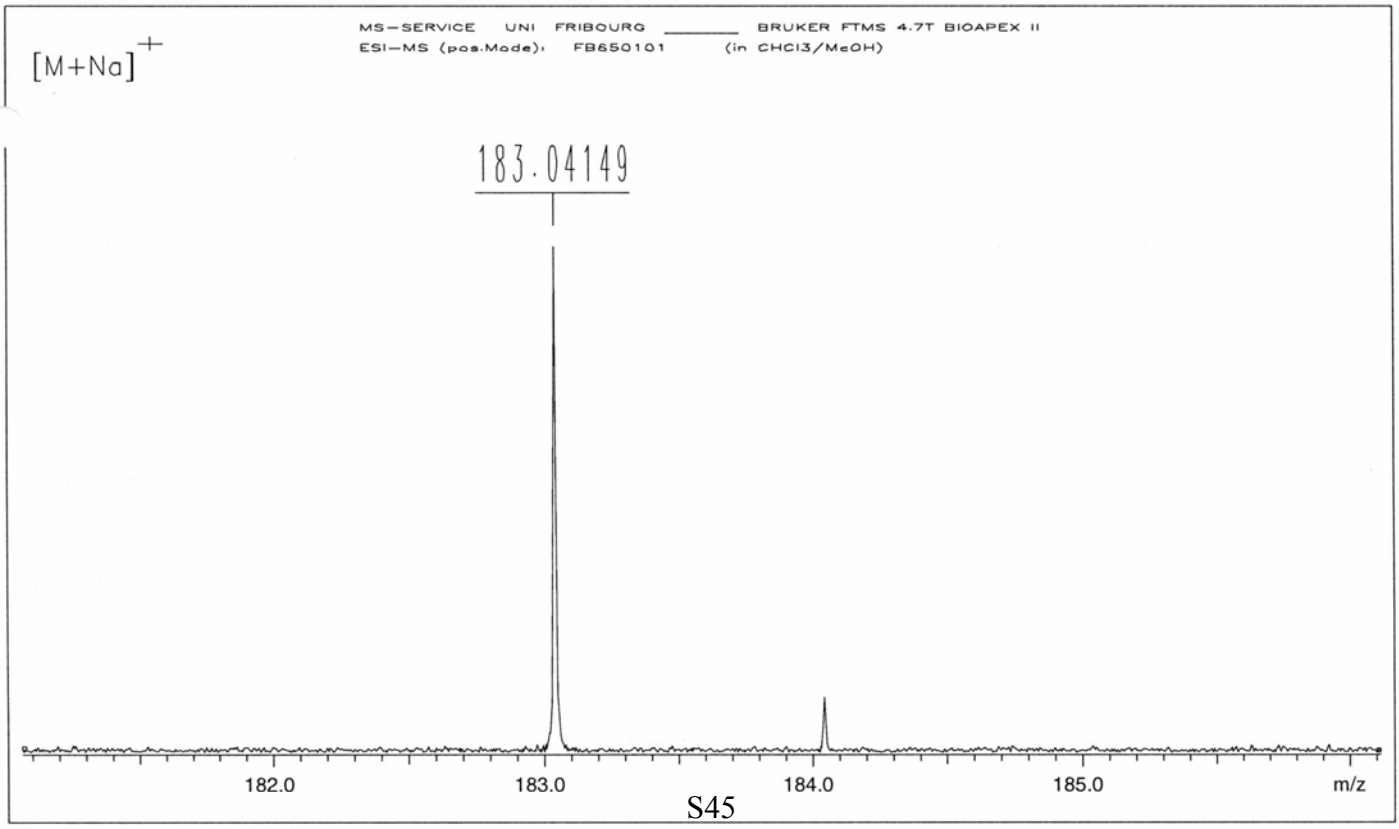
ESI-MS: FB650101

XMASS Mass Analysis for /Data/UNI_FR/BIRB1667_ESI/2/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

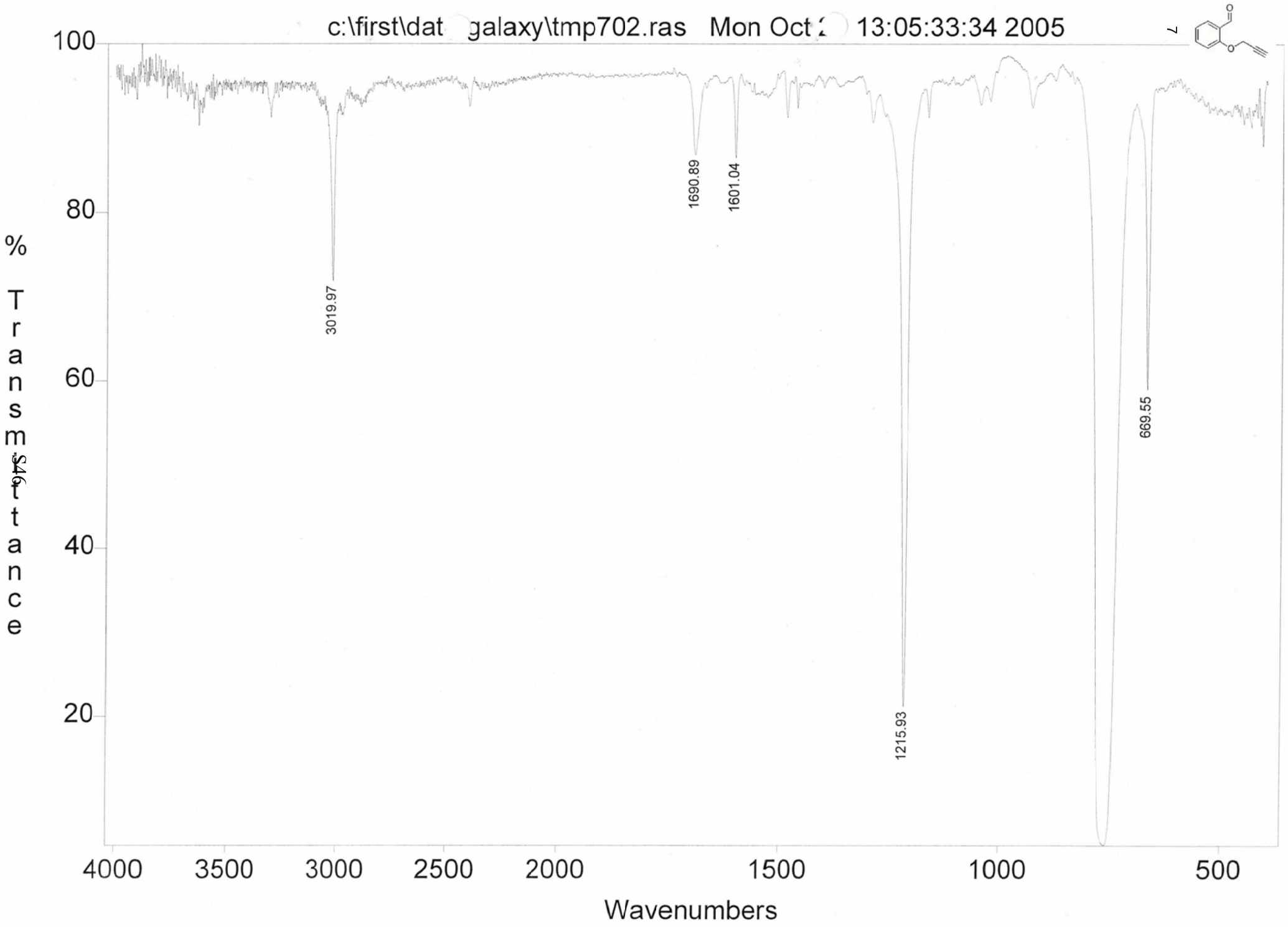
Ion mass = 183.0414950

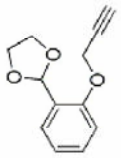
Charge = +1

#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 183.0414950							
1	10	8	2	1	183.0416506	6.5	1.556e-04
2	12	7	2	0	183.0440559	9.5	2.561e-03
3	5	11	7	0	183.0499291	0.5	8.434e-03
4	8	7	5	0	183.0287998	5.5	1.270e-02
5	6	8	5	1	183.0263945	2.5	1.510e-02
6	7	12	4	1	183.0627800	1.5	2.128e-02
7	9	11	4	0	183.0651853	4.5	2.369e-02
8	11	3	3	0	183.0076704	10.5	3.382e-02
9	9	4	3	1	183.0052651	7.5	3.623e-02
10	11	12	1	1	183.0780361	5.5	3.654e-02

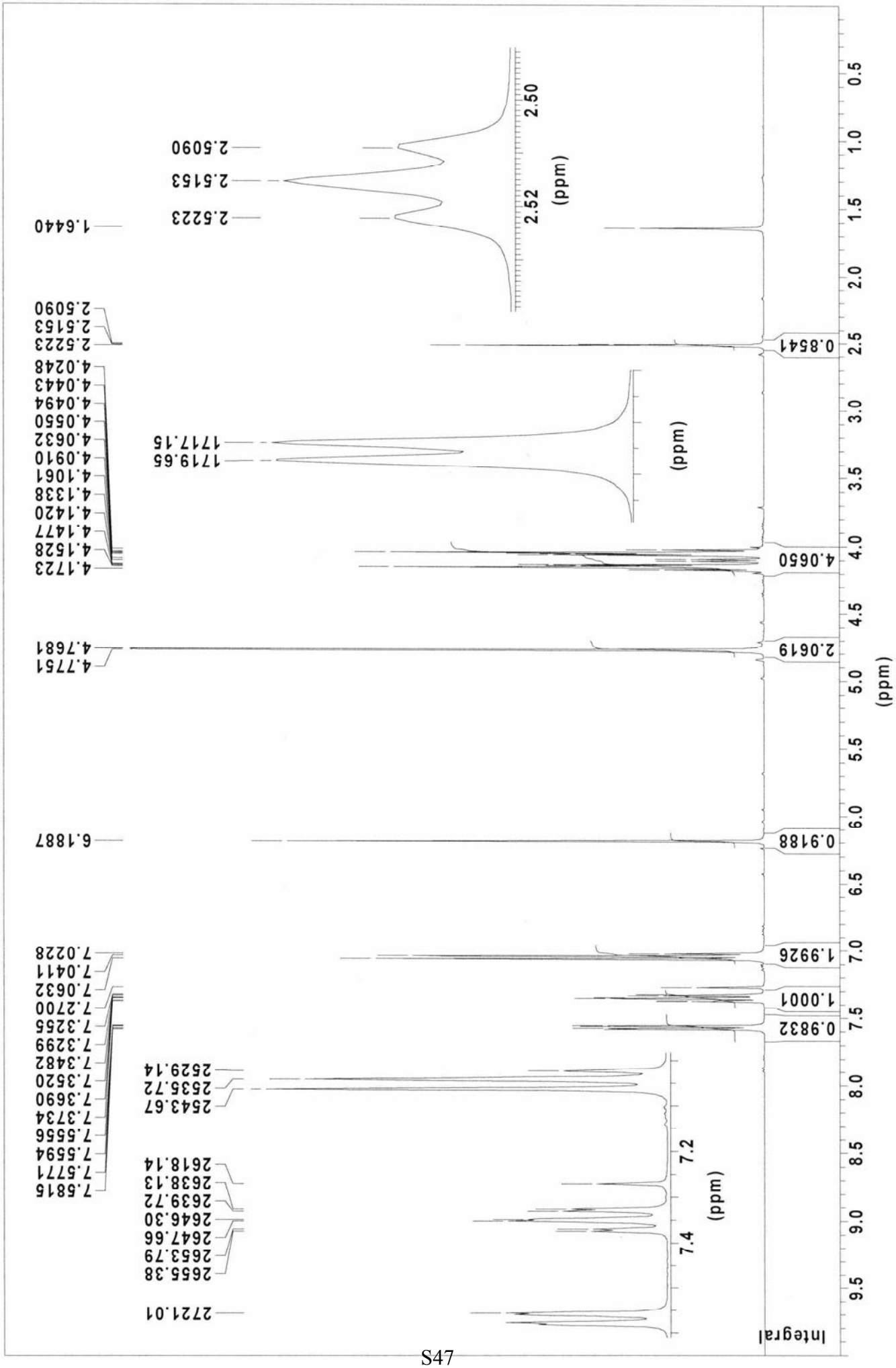


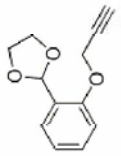
/Data/UNI_FR/BIRB1667_ESI/2/pdata/1 FTMS USER Wed Jul 19 16:12:16 2006


c:\first\dat galaxy\tmp702.ras Mon Oct 2 13:05:33:34 2005
7
100
80
60
40
20
% Transmittance
3019.97
1690.89
1601.04
1215.93
669.55
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers

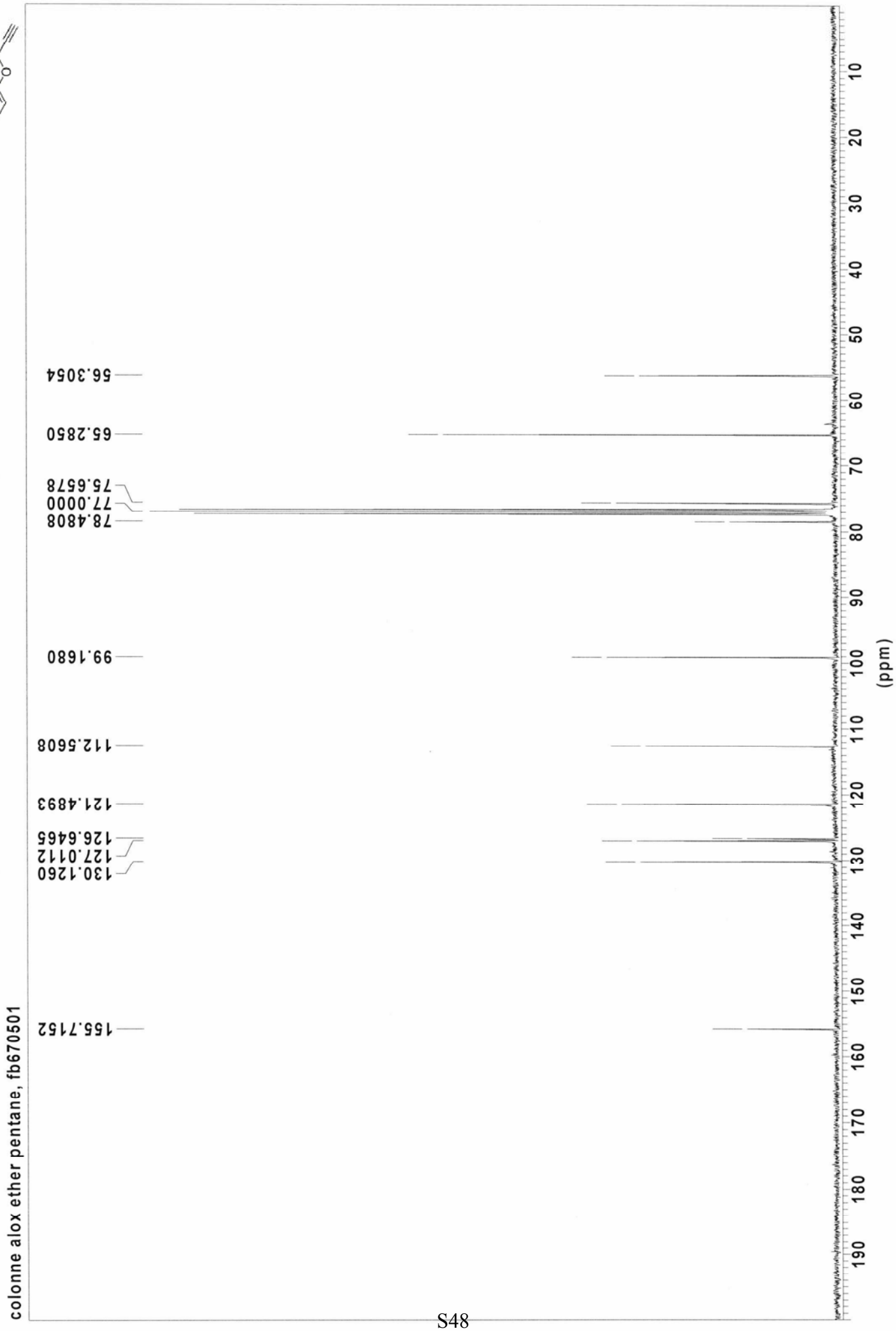


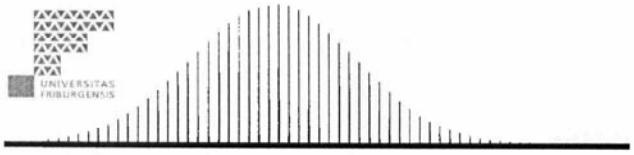
8

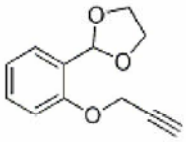

colonne alox ether pentane, fb670501
Integral
(ppm)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
7.4
7.2
2.52
2.50
2721.01
2655.38
2653.79
2647.66
2646.30
2639.72
2638.13
2618.14
2543.67
2535.72
2529.14
7.5815
7.5771
7.5594
7.5556
7.3734
7.3690
7.3520
7.3482
7.3299
7.3255
7.2700
7.0632
7.0411
7.0228
6.1887
4.7751
4.7681
4.1723
4.1528
4.1477
4.1420
4.1338
4.1061
4.0910
4.0632
4.0550
4.0494
4.0443
4.0248
2.5223
2.5153
2.5090
1.6440
2.5223
2.5153
2.5090
1719.65
1717.15
0.8541
4.0650
2.0619
0.9188
1.9926
1.0001
0.9832



8


colonne alox ether pentane, fb670501
155.7152
130.1260
127.0112
126.6465
121.4893
112.5608
99.1680
78.4808
77.0000
75.6578
65.2850
56.3054
(ppm)





8

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

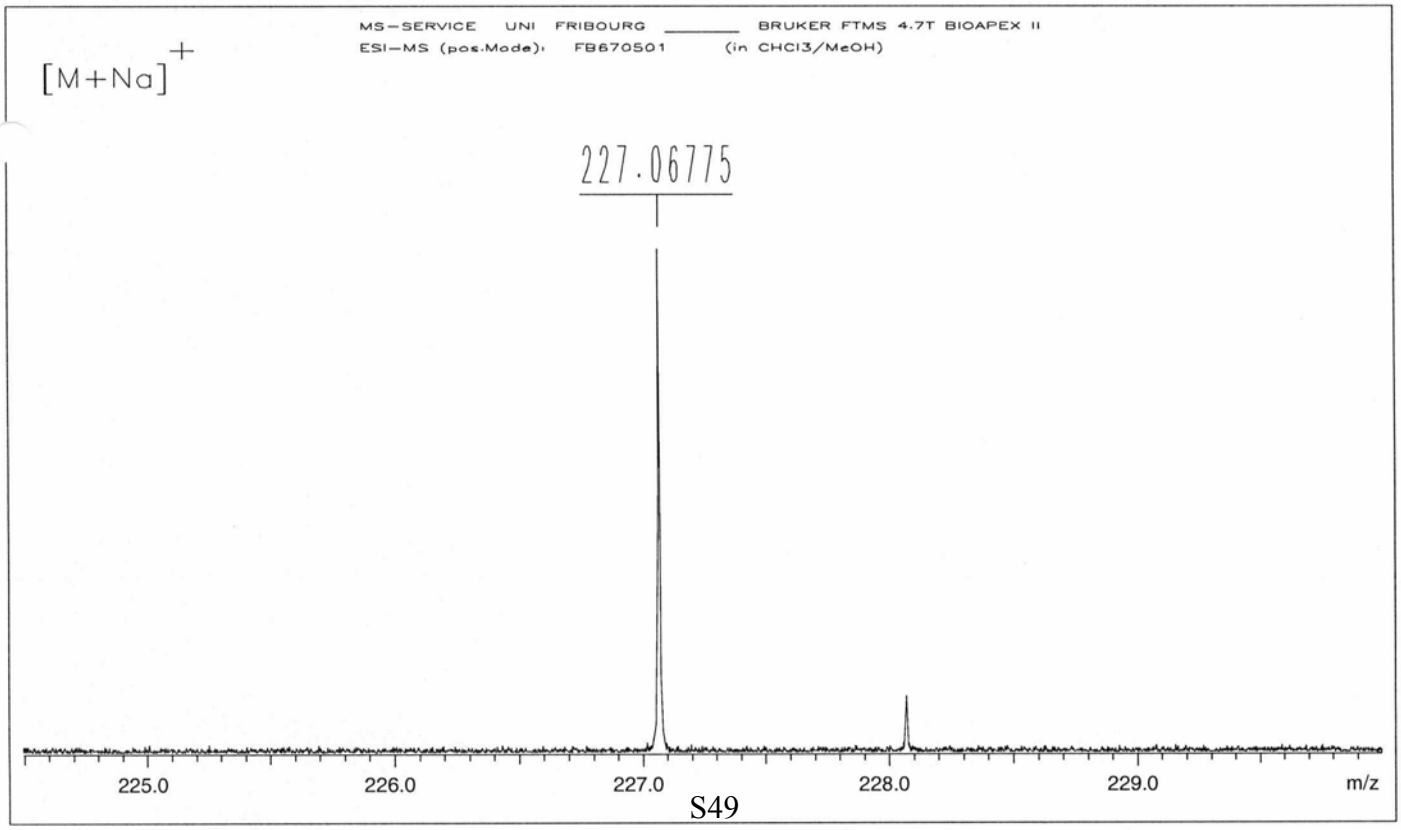
ESI-MS: FB670501

XMASS Mass Analysis for /Data/UNI_FR/BIRB1659_ESI/2/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

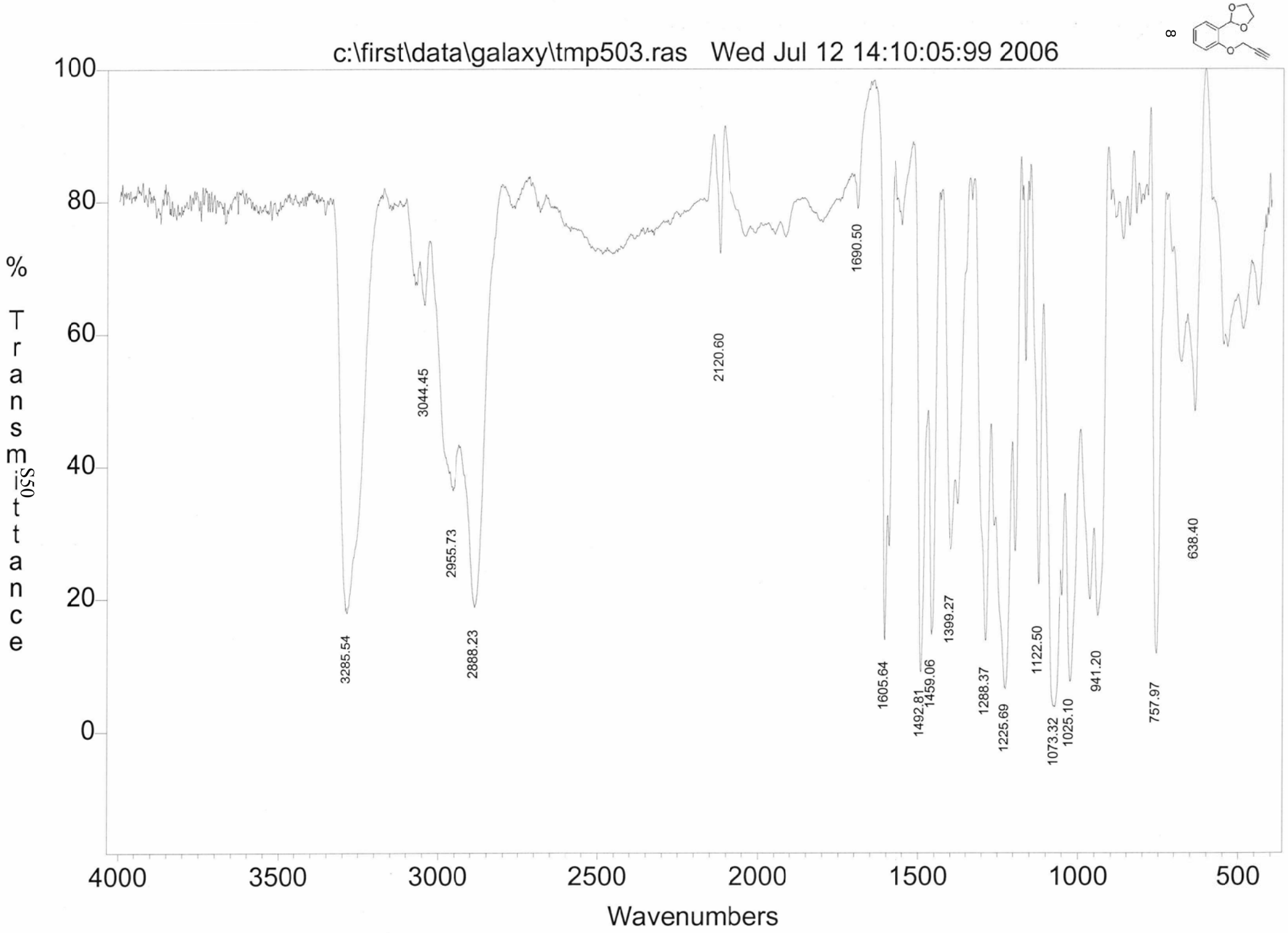
Ion mass = 227.0677490

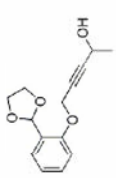
Charge = +1

#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 227.0677490							
1	12	12	3	1	227.0678653	6.5	1.163e-04
2	14	11	3	0	227.0702706	9.5	2.522e-03
3	5	16	8	1	227.0737386	-2.5	5.990e-03
4	7	15	8	0	227.0761439	0.5	8.395e-03
5	10	11	6	0	227.0550145	5.5	1.273e-02
6	8	12	6	1	227.0526092	2.5	1.514e-02
7	17	7	1	0	227.0491413	14.5	1.861e-02
8	15	8	1	1	227.0467360	11.5	2.101e-02
9	9	16	5	1	227.0889947	1.5	2.125e-02
10	11	15	5	0	227.0914000	4.5	2.365e-02

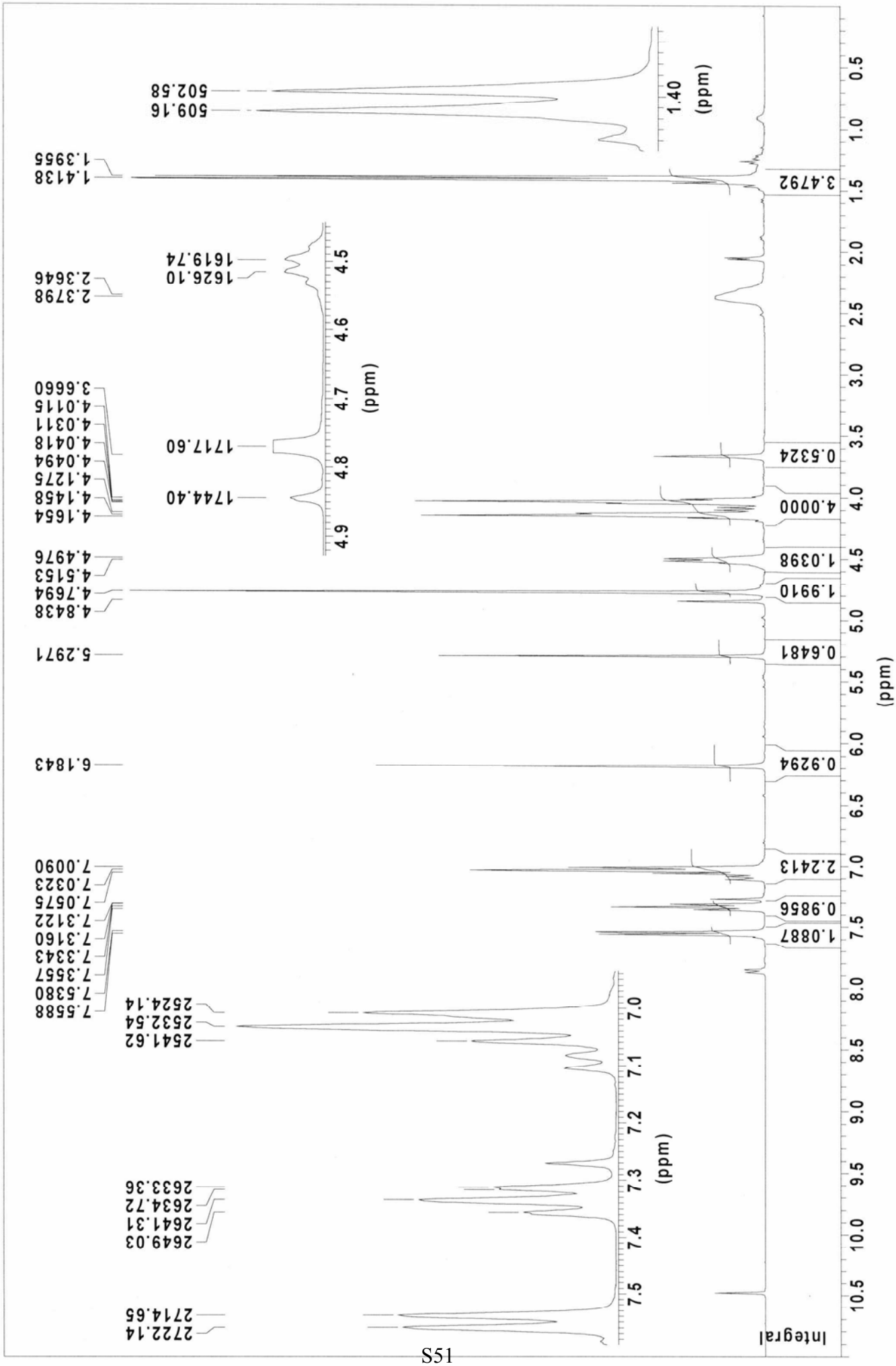


/Data/UNI_FR/BIRB1659_ESI/2/pdata/1 FTMS USER Wed Jul 19 09:19:32 2006

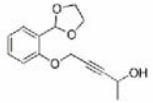

c:\first\data\galaxy\tmp503.ras Wed Jul 12 14:10:05:99 2006
8
% Transmittance
100
80
60
40
20
0
3285.54
3044.45
2955.73
2888.23
2120.60
1690.50
1605.64
1492.81
1459.06
1399.27
1288.37
1225.69
1122.50
1073.32
1025.10
941.20
757.97
638.40
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



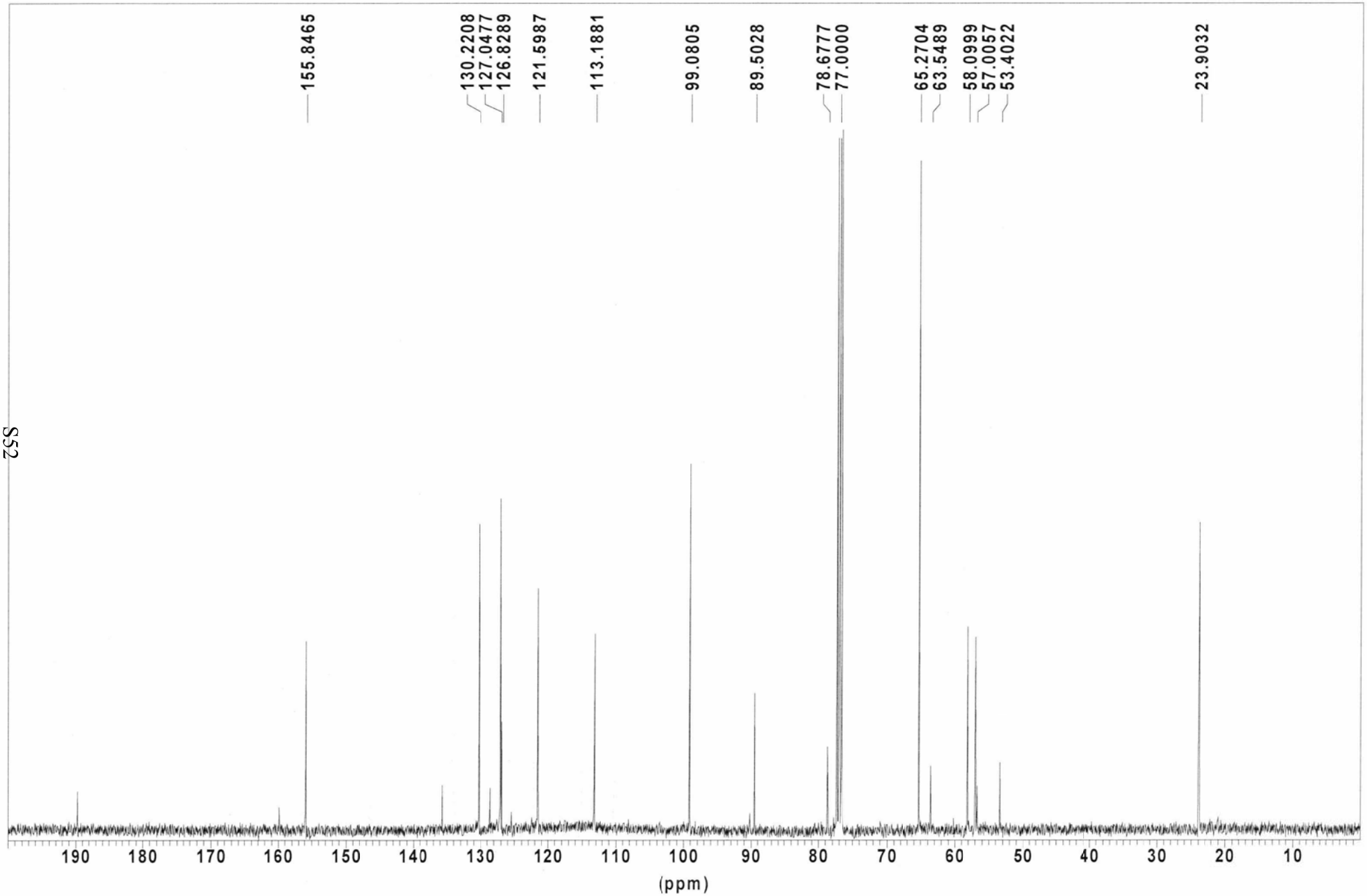
9

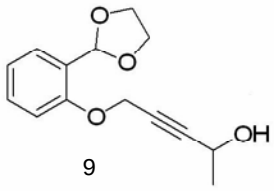

plug alox EtOAc Ether DichlO, fb790201
Integral
(ppm)
1.40
3.4792
0.5324
4.0000
1.0398
1.9910
0.6481
0.9294
2.2413
0.9856
1.0887
1.4138
1.3955
509.16
502.58
2.3798
2.3646
1626.10
1619.74
1744.40
1717.60
4.1654
4.1458
4.1275
4.0494
4.0418
4.0311
4.0115
3.6660
5.2971
4.8438
4.7694
4.5153
4.4976
6.1843
7.5588
7.5380
7.3557
7.3343
7.3160
7.3122
7.0575
7.0323
7.0090
2541.62
2532.54
2524.14
2649.03
2641.31
2634.72
2633.36
2722.14
2714.65

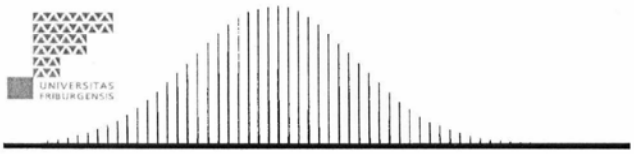
plug alox EtOAc Ether Dichlo, fb790201



9







FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

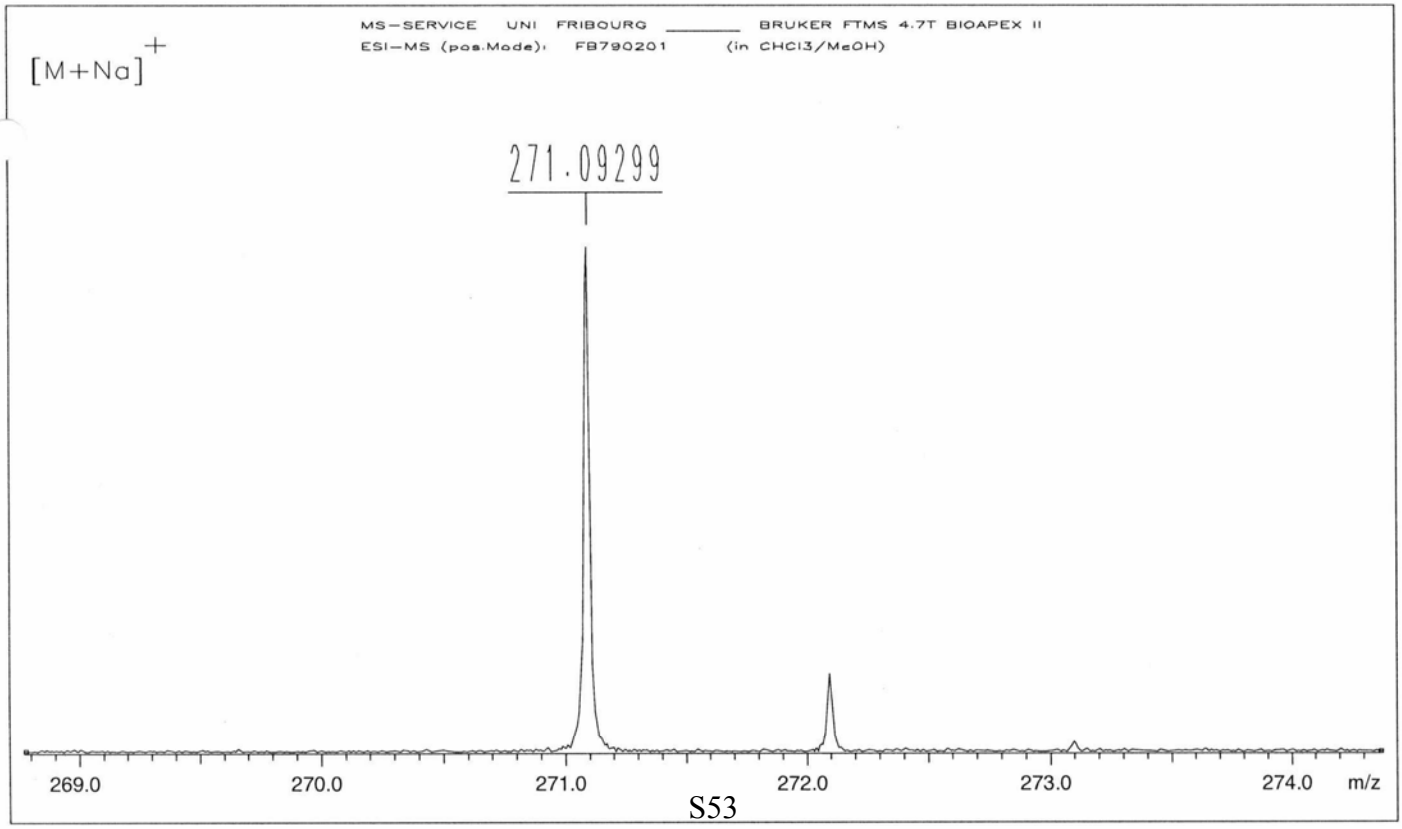
ESI-MS: FB790201

XMASS Mass Analysis for /Data/UNI_FR/BIRB1678_ESI/20/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

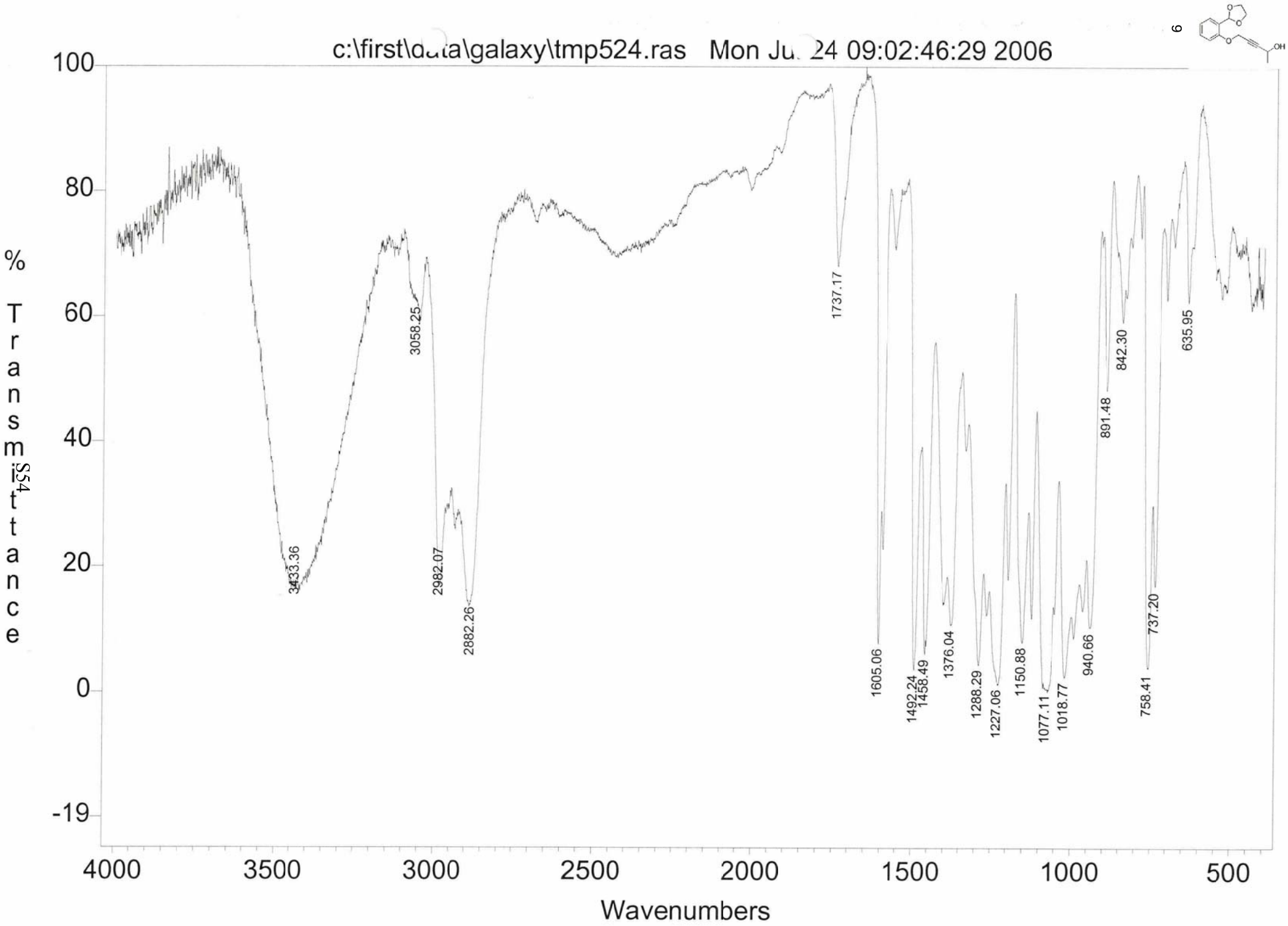
Ion mass = 271.0929900

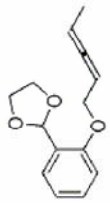
Charge = +1

#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 271.0929900							
1	14	16	4	1	271.0940801	6.5	1.090e-03
2	16	15	4	0	271.0964854	9.5	3.495e-03
3	7	20	9	1	271.0999533	-2.5	6.963e-03
4	9	19	9	0	271.1023586	0.5	9.369e-03
5	12	15	7	0	271.0812293	5.5	1.176e-02
6	10	16	7	1	271.0788240	2.5	1.417e-02
7	18	16	1	1	271.1093362	10.5	1.635e-02
8	19	11	2	0	271.0753560	14.5	1.763e-02
9	20	15	1	0	271.1117415	13.5	1.875e-02
10	17	12	2	1	271.0729507	11.5	2.004e-02



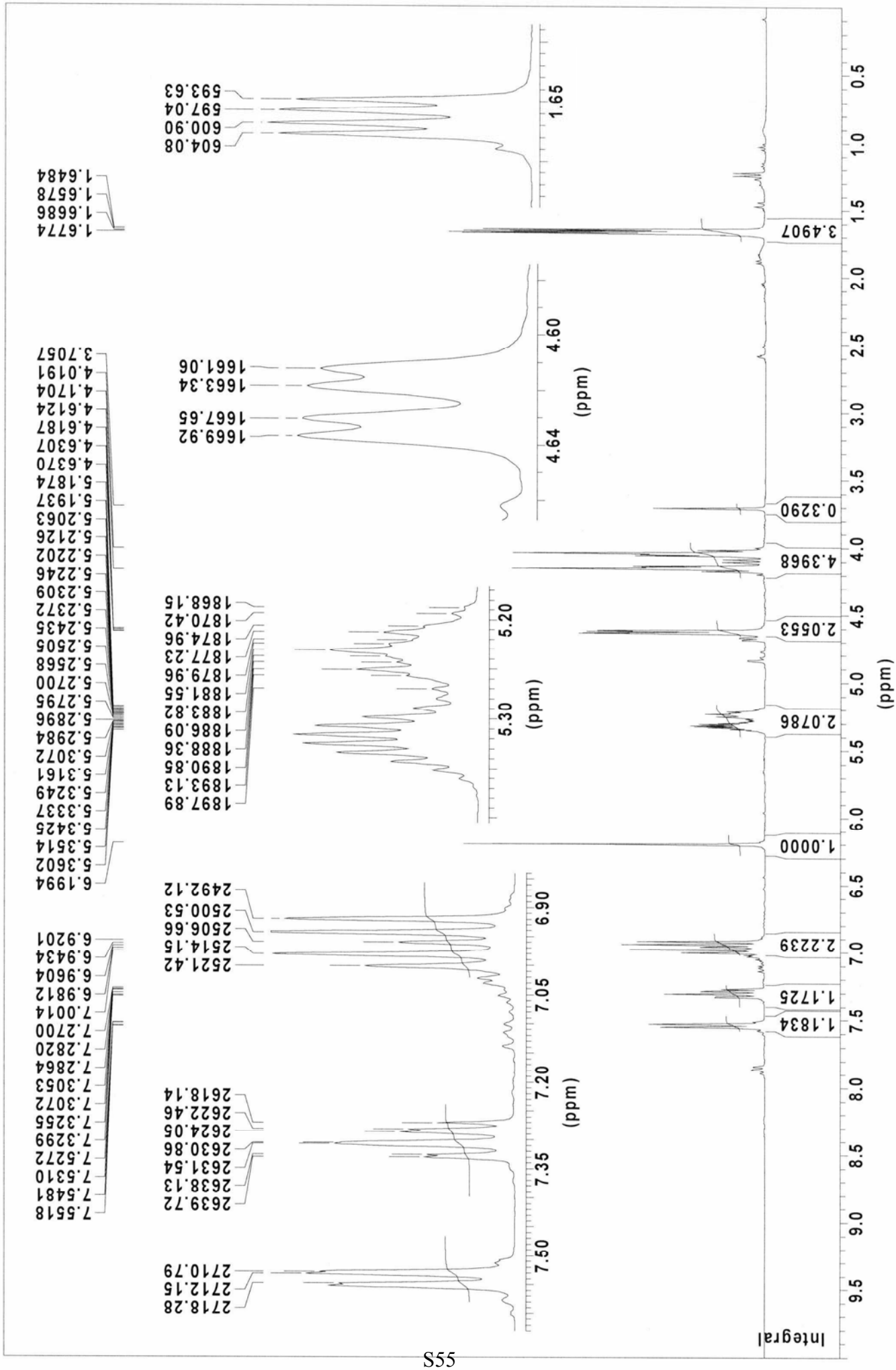
/Data/UNI_FR/BIRB1678_ESI/20/pdata/1 FTMS USER Mon Jul 24 15:27:39 2006

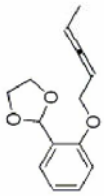

c:\first\data\galaxy\tmp524.ras Mon Ju. 24 09:02:46:29 2006
9
OH
% Transmittance
100
80
60
40
20
0
-19
3433.36
3058.25
2982.07
2882.26
1737.17
1605.06
1492.24
1458.49
1376.04
1288.29
1227.06
1150.88
1077.11
1018.77
940.66
891.48
842.30
758.41
737.20
635.95
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



10

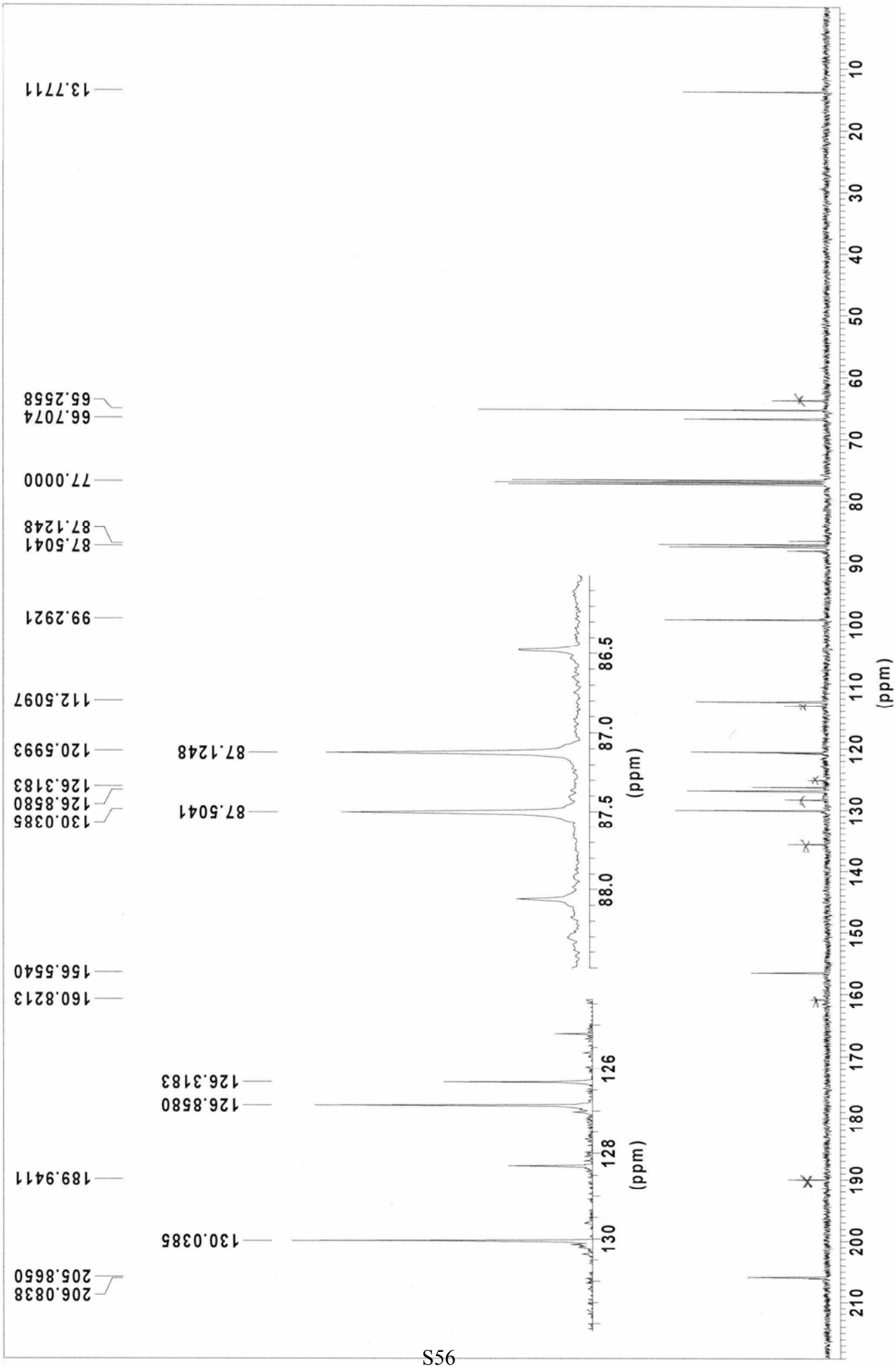
colonne ether pentane 3 spots UV 1 KMnO4, fb810201

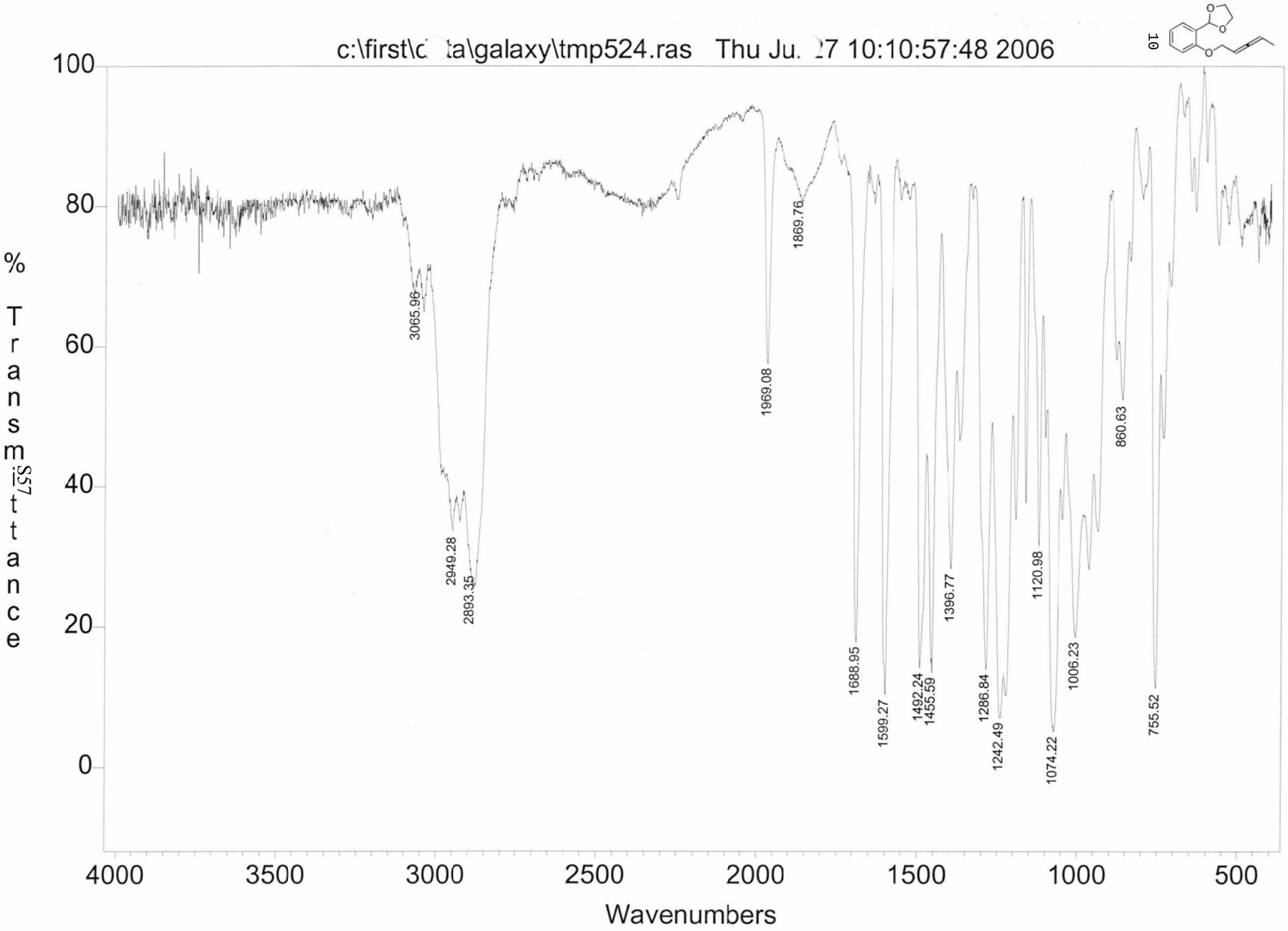


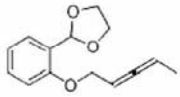


10

colonne ether pentane 3 spots UV 1 KMnO4, fb810201




c:\first\c ta\galaxy\tmp524.ras Thu Ju. 27 10:10:57:48 2006
10
% Transmittance
100
80
60
40
20
0
3065.96
2949.28
2893.35
1969.08
1869.76
1688.95
1599.27
1492.24
1455.59
1396.77
1286.84
1242.49
1120.98
1074.22
1006.23
860.63
755.52
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



10

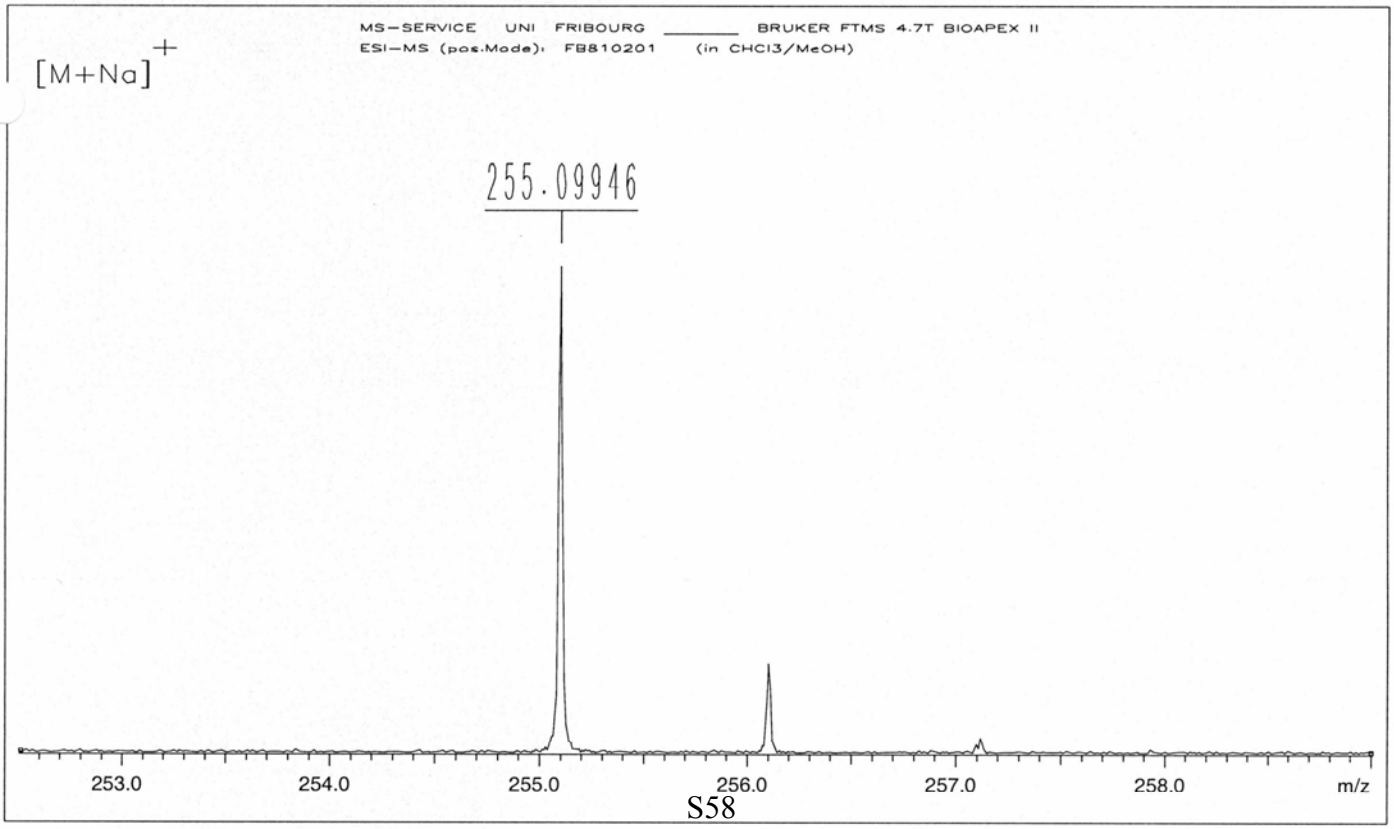
ESI-MS: FB810201

XMASS Mass Analysis for /Data/UNI_FR/BIRB1724_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

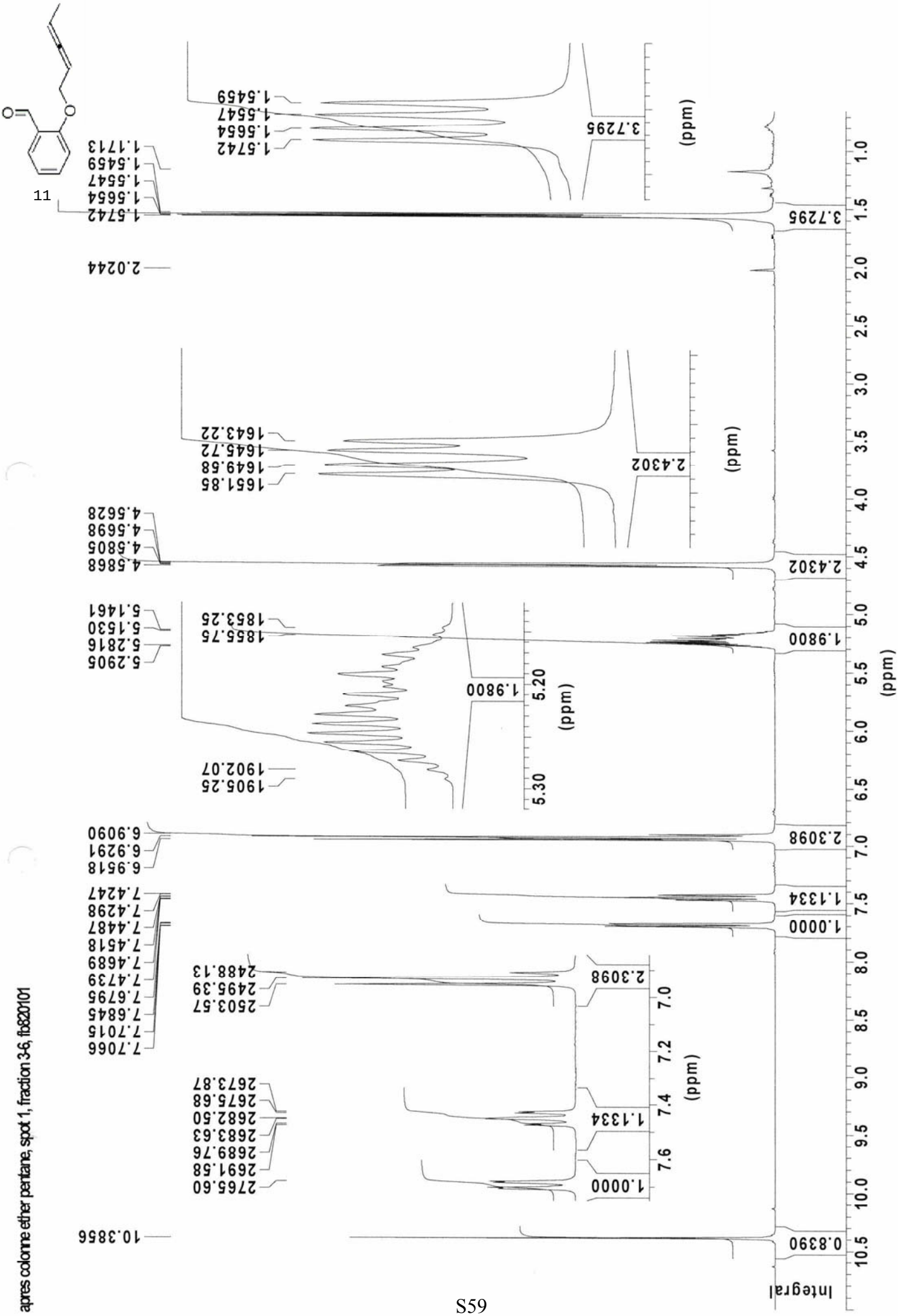
Ion mass = 255.0994640

Charge = +1

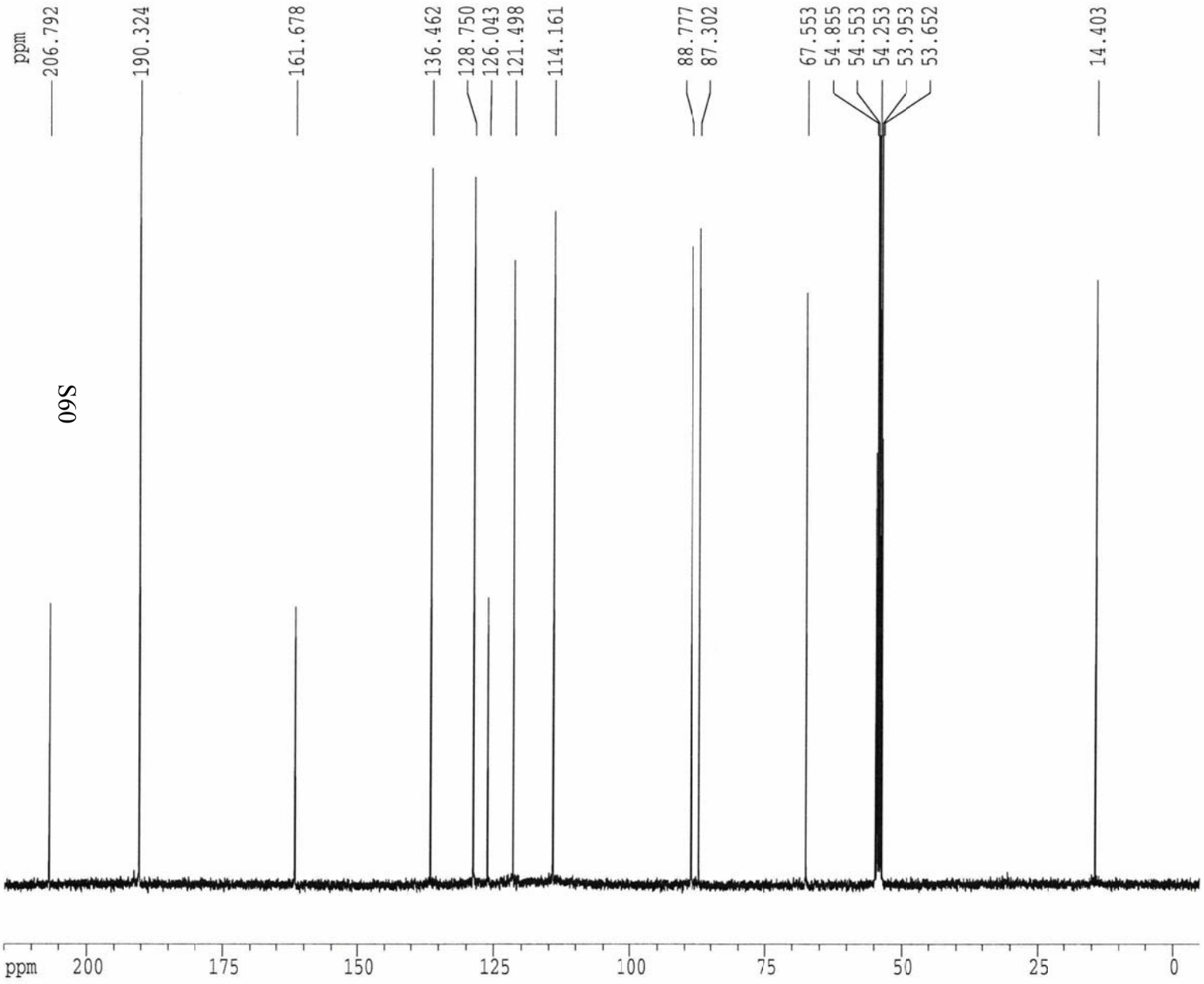
#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 255.0994640							
1	14	16	3	1	255.0991655	6.5	2.985e-04
2	16	15	3	0	255.1015708	9.5	2.107e-03
3	7	20	8	1	255.1050387	-2.5	5.575e-03
4	9	19	8	0	255.1074440	0.5	7.980e-03
5	12	15	6	0	255.0863146	5.5	1.315e-02
6	10	16	6	1	255.0839093	2.5	1.555e-02
7	19	11	1	0	255.0804414	14.5	1.902e-02
8	11	20	5	1	255.1202948	1.5	2.083e-02
9	17	12	1	1	255.0780361	11.5	2.143e-02
10	13	19	5	0	255.1227001	4.5	2.324e-02



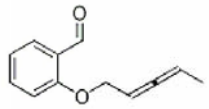
/Data/UNI_FR/BIRB1724_ESI/1/pdata/1 FTMS USER Tue Aug 22 07:50:43 2006


11
apres colonne ether pentane, spot 1, fraction 3-6, fb820101
Integral
(ppm)
10.3856
0.8390
1.0000
1.1334
2.3098
1.9800
2.4302
3.7295
2.0244
1.5742
1.5654
1.5547
1.5459
1.1713
4.5868
4.5805
4.5698
4.5628
5.2905
5.2816
5.1530
5.1461
6.9518
6.9291
6.9090
7.7066
7.7015
7.6845
7.6795
7.4739
7.4689
7.4518
7.4487
7.4298
7.4247
2765.60
2691.58
2689.76
2683.63
2682.50
2675.68
2673.87
2503.57
2495.39
2488.13
1905.25
1902.07
1855.75
1853.25
1651.85
1649.58
1645.72
1643.22

fb820101-13C CPD
C13CPD CD2Cl2 u asth 25



11



Current Data Parameters
NAME fb820101
EXPNO 2
PROCNO 1

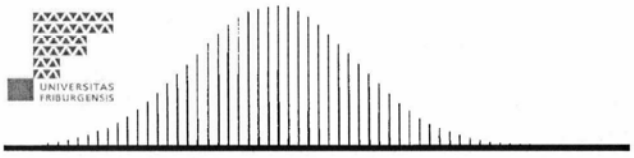
F2 - Acquisition Parameters
Date_ 20050906
Time 1.09
INSTRUM dpx360
PROBHD 5 mm SEI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 2048
DS 4
SWH 21645.021 Hz
FIDRES 0.330277 Hz
AQ 1.5139316 sec
RG 1625.5
DW 23.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

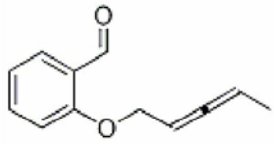
======== CHANNEL f1 ========
NUC1 13C
P1 13.60 usec
PL1 -6.00 dB
SFO1 90.5637800 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 16.45 dB
PL13 120.00 dB
SFO2 360.1314405 MHz

F2 - Processing parameters
SI 32768
SF 90.5546888 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 19469.26 Hz
F2P -5.000 ppm
F2 -452.77 Hz
PPMCM 11.00000 ppm/cm
HZCM 996.10156 Hz/cm





11

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

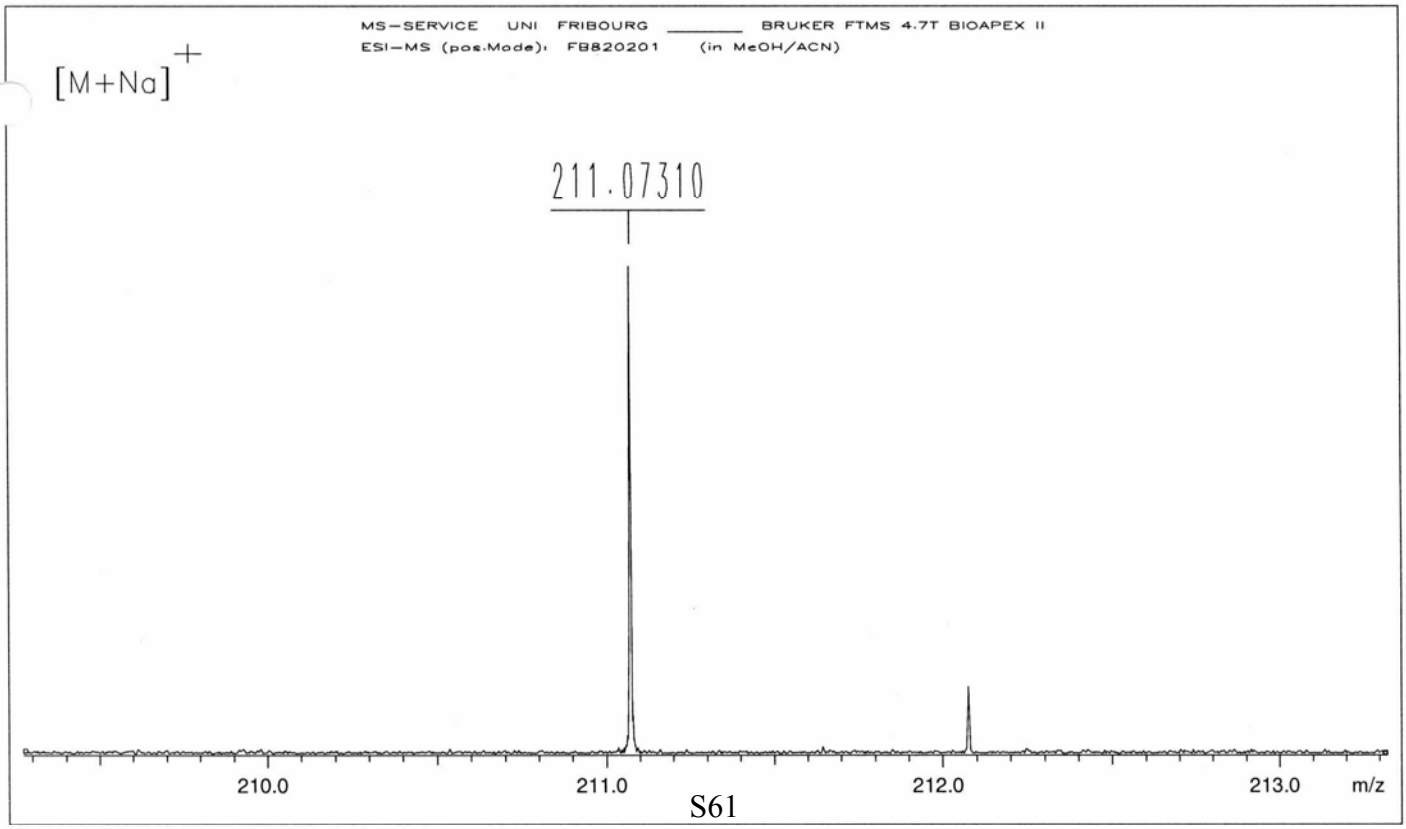
ESI-MS: FB820201

XMASS Mass Analysis for /Data/UNI_FR/BIRB1044_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

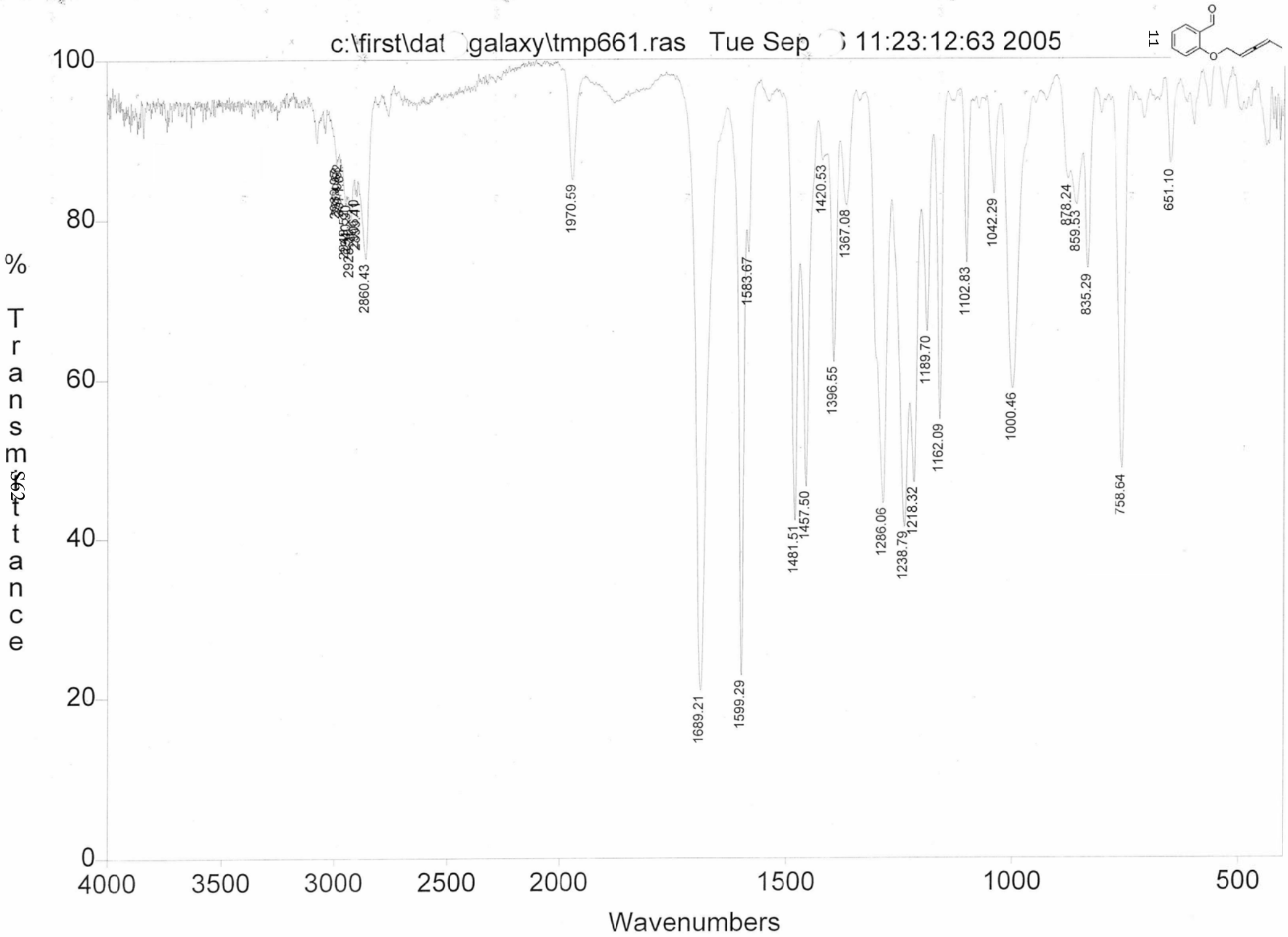
Ion mass = 211.0730960

Charge = +1

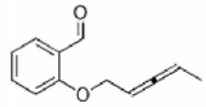
#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 211.0730960							
1	12	12	2	1	211.0729507	6.5	1.453e-04
2	14	11	2	0	211.0753560	9.5	2.260e-03
3	5	16	7	1	211.0788240	-2.5	5.728e-03
4	7	15	7	0	211.0812293	0.5	8.133e-03
5	10	11	5	0	211.0600999	5.5	1.300e-02
6	8	12	5	1	211.0576946	2.5	1.540e-02
7	9	16	4	1	211.0940801	1.5	2.098e-02
8	11	15	4	0	211.0964854	4.5	2.339e-02
9	6	11	8	0	211.0448437	1.5	2.825e-02
10	13	7	3	0	211.0389705	10.5	3.413e-02



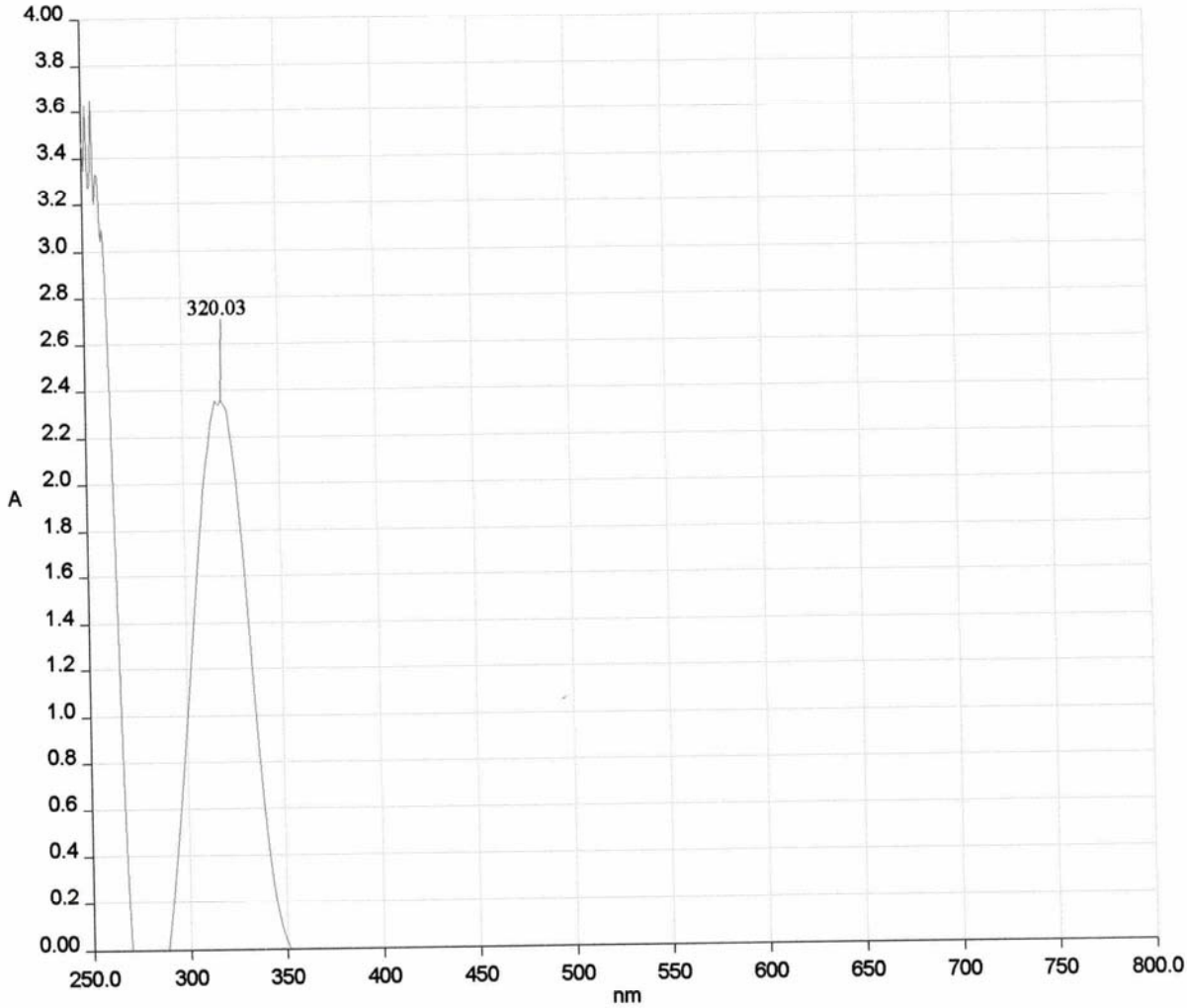
/Data/UNI_FR/BIRB1044_ESI/1/pdata/1 FTMS USER Wed Jan 18 16:14:09 2006


c:\first\dat galaxy\tmp661.ras Tue Sep 11:23:12:63 2005
11
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
2860.43
1970.59
1689.21
1599.29
1583.67
1481.51
1457.50
1420.53
1396.55
1367.08
1286.06
1238.79
1218.32
1189.70
1162.09
1102.83
1042.29
1000.46
878.24
859.53
835.29
758.64
651.10

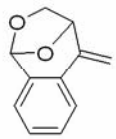
Date: 06.09.2005 Time: 17:25:26



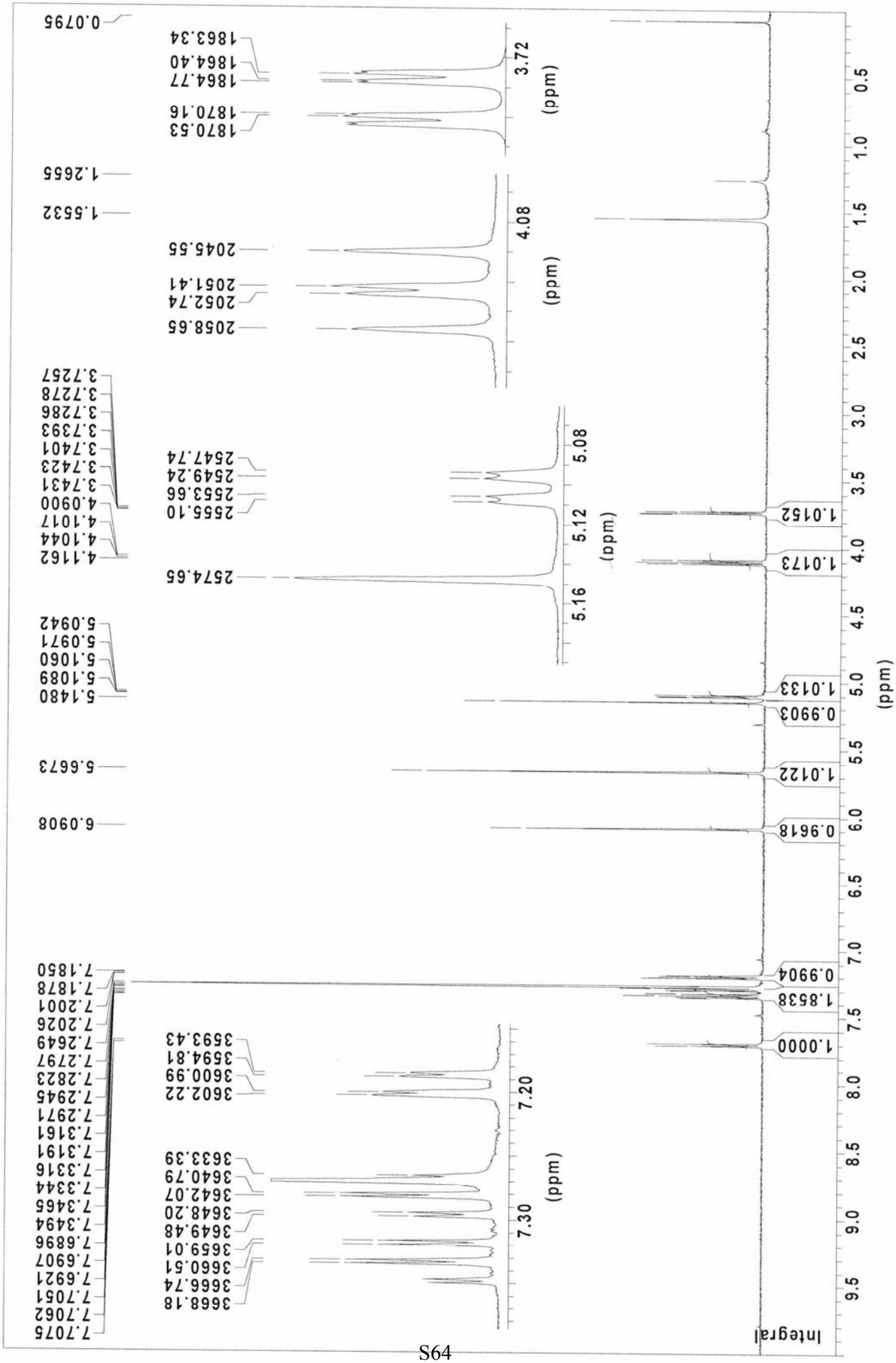
11

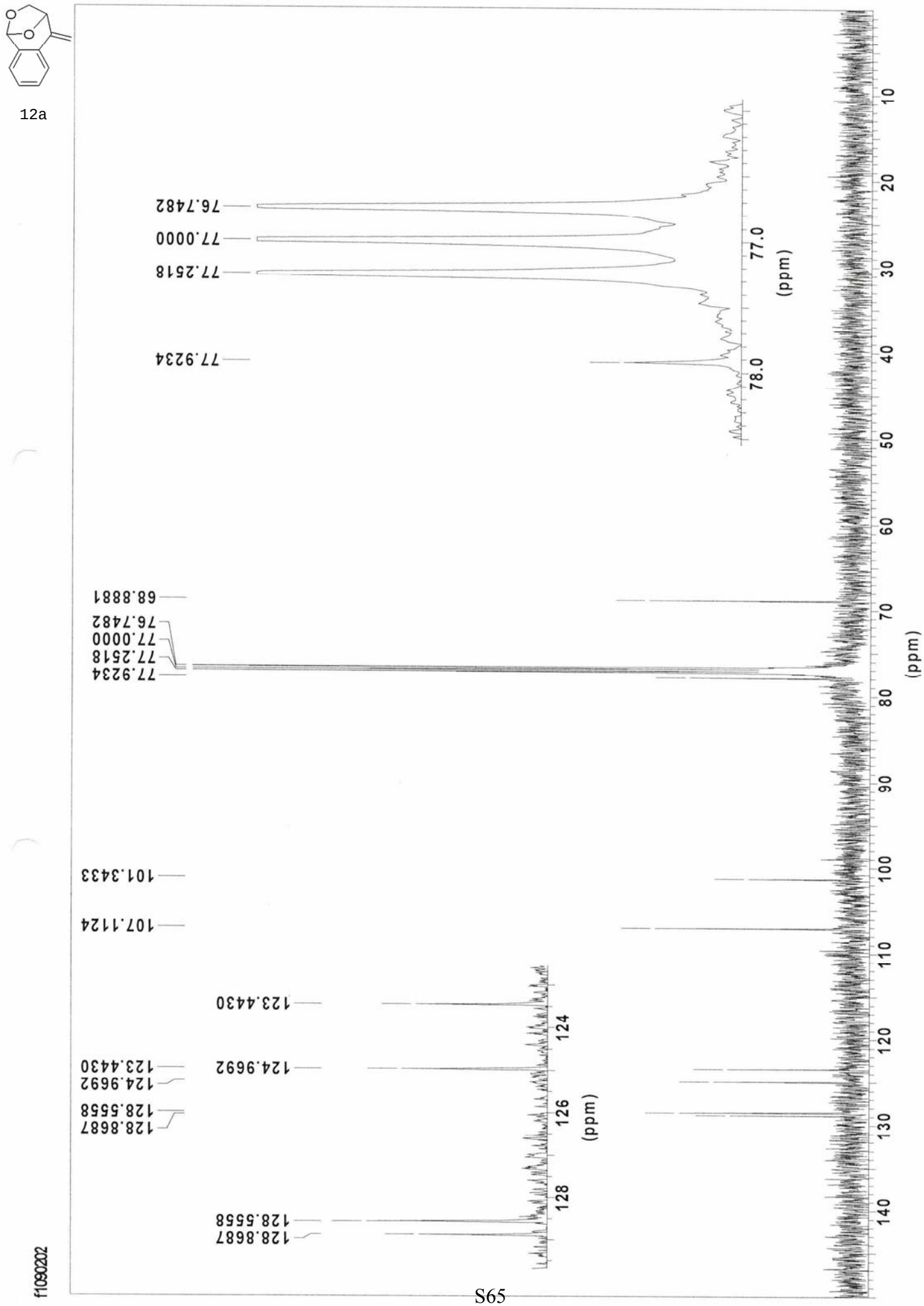


FB820203.SP - 06.09.2005

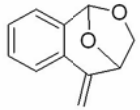


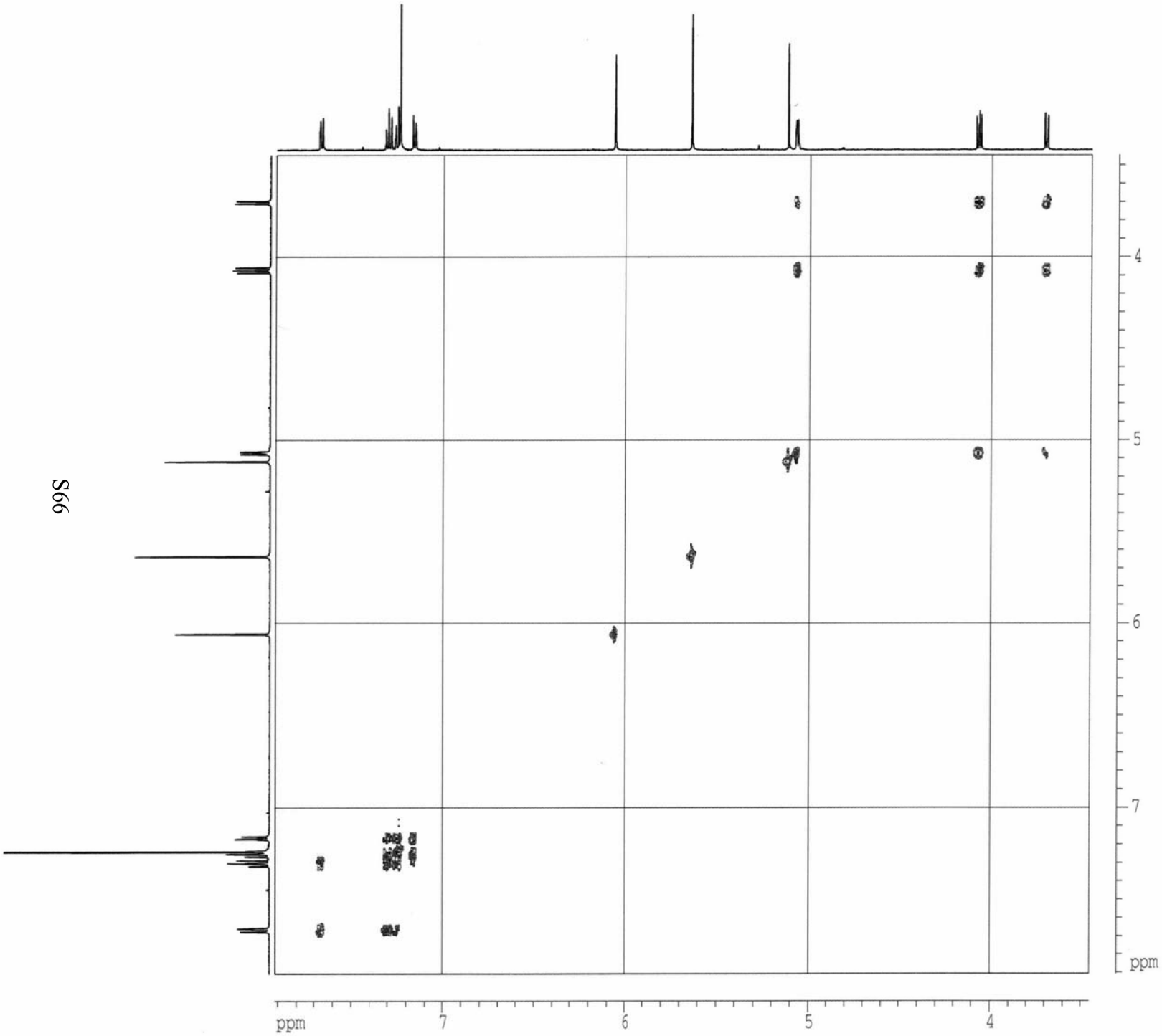
12a


f1080202
7.7075
7.7062
7.7051
7.6921
7.6907
7.6896
7.3494
7.3465
7.3344
7.3316
7.3191
7.3161
7.2971
7.2945
7.2823
7.2797
7.2649
7.2026
7.2001
7.1878
7.1850
6.0908
5.6673
5.1480
5.1089
5.1060
5.0971
5.0942
4.1162
4.1044
4.1017
4.0900
3.7431
3.7423
3.7401
3.7393
3.7286
3.7278
3.7257
1.2655
1.5532
0.0795
3668.18
3666.74
3660.51
3659.01
3649.48
3648.20
3642.07
3640.79
3633.39
3602.22
3600.99
3594.81
3593.43
7.30
7.20
(ppm)
2574.65
2555.10
2553.66
2549.24
2547.74
5.16
5.12
5.08
(ppm)
2058.65
2052.74
2051.41
2045.55
4.08
(ppm)
1870.53
1870.16
1864.77
1864.40
1863.34
3.72
(ppm)
Integral
1.0000
1.8538
0.9904
0.9618
1.0122
0.9903
1.0133
1.0173
1.0152
(ppm)


12a
f1090202
77.9234
77.2518
77.0000
76.7482
68.8881
101.3433
107.1124
123.4430
124.9692
128.8687
128.5558
(ppm)

12a





Current Data Parameters

NAME	birb2713.bbo
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20050207
Time	10.31
INSTRUM	drx500
PROBHD	5 mm Multinucl
PULPROG	cosygs
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	2232.143 Hz
FIDRES	1.089913 Hz
AQ	0.4588020 sec
RG	4096
DW	224.000 usec
DE	6.00 usec
TE	293.0 K
d0	0.00000300 sec
D1	2.00000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
IN0	0.00044800 sec

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

NUC1	1H
P0	6.00 usec
P1	9.00 usec
PL1	-3.00 dB
SFO1	500.1328621 MHz

====== GRADIENT CHANNEL =====

P16	6000.00 usec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	500.1329 MHz
FIDRES	8.719308 Hz
SW	4.463 ppm

F2 - Processing parameters

SI	2048
SF	500.1300200 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

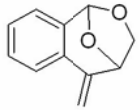
F1 - Processing parameters

SI	512
MC2	QF
SF	500.1300232 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

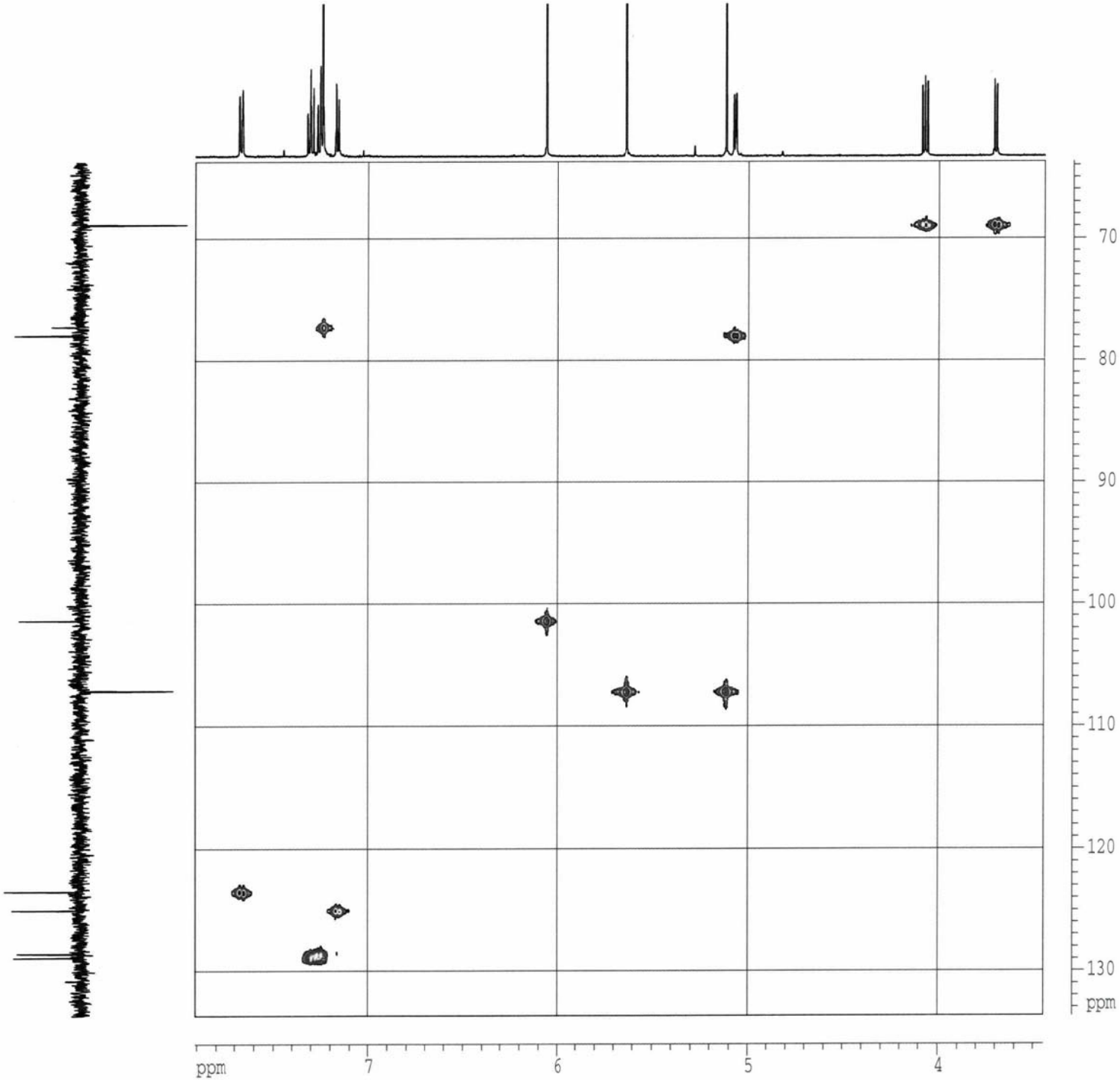
2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	7.914 ppm
F2LO	3958.17 Hz
F2PHI	3.451 ppm
F2HI	1726.03 Hz
F1PLO	7.908 ppm
F1LO	3954.99 Hz
F1PHI	3.445 ppm
F1HI	1722.85 Hz
F2PPMCM	0.29754 ppm/cm
F2HZCM	148.80952 Hz/cm
F1PPMCM	0.29754 ppm/cm
F1HZCM	148.80952 Hz/cm

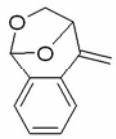
12a



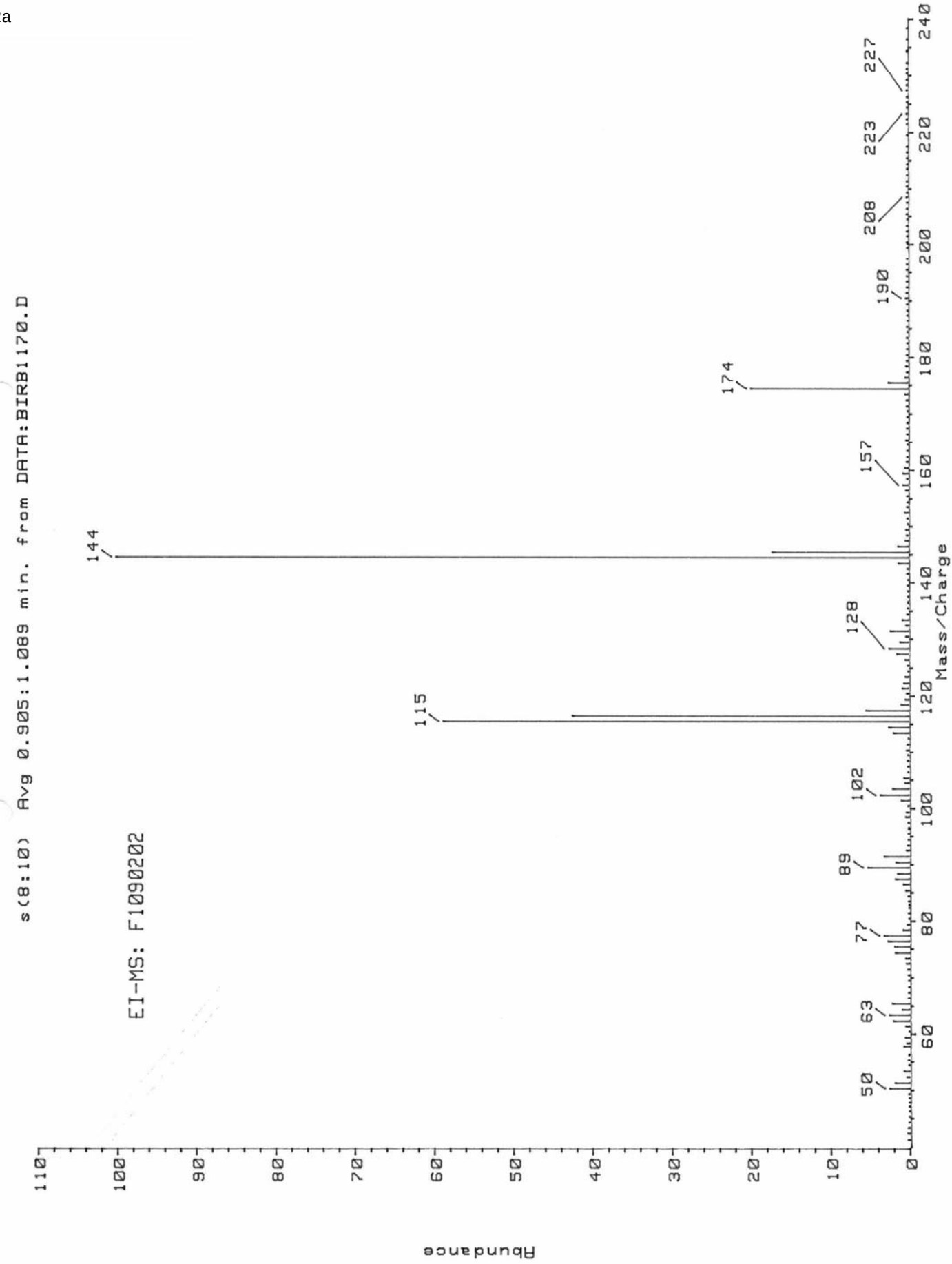
HETCOR

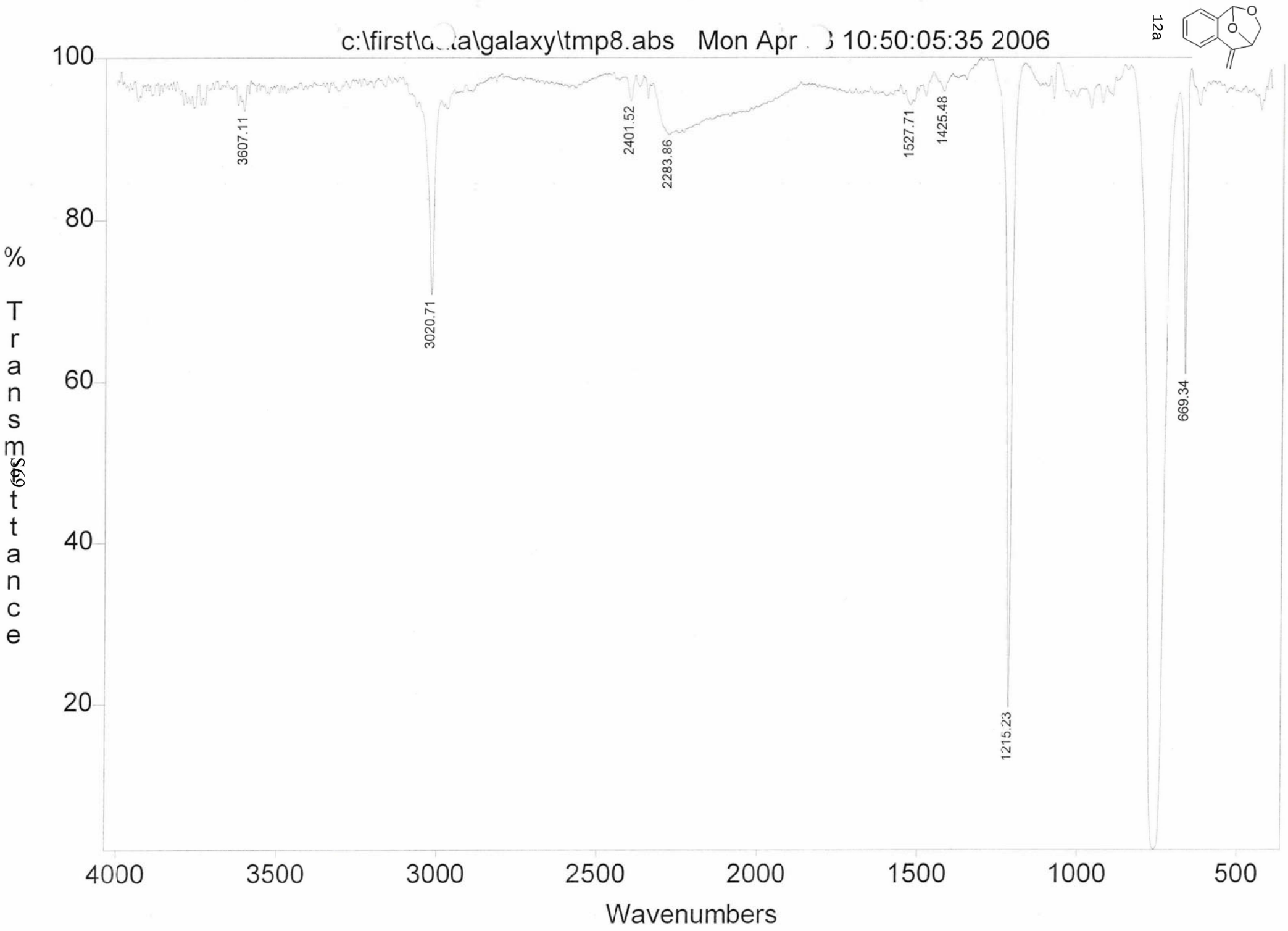


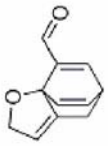

```
        Current Data Parameters
NAME        birb2713.bbo
EXPNO                  5
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20060207
Time               8.36
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          inv4gs
TD                 2048
SOLVENT           CDCl3
NS                   16
DS                   16
SWH            2232.143 Hz
FIDRES         1.089913 Hz
AQ            0.4588020 sec
RG              20642.5
DW              224.000 usec
DE                6.00 usec
TE               293.0 K
CNST2        145.0000000
d0            0.00000300 sec
D1            2.00000000 sec
d2            0.00344828 sec
d12           0.00002000 sec
d13           0.00000300 sec
D16           0.00020000 sec
d20           0.00192528 sec
IN0           0.00005680 sec

     ======== CHANNEL f1 ========
NUC1                  1H
P1                 8.20 usec
p2                16.40 usec
PL1               -3.00 dB
SFO1         500.1328621 MHz

     ======== CHANNEL f2 ========
CPDPRG2             garp
NUC2                 13C
P3                 7.00 usec
PCPD2             88.00 usec
PL2               -1.00 dB
PL12              17.00 dB
SFO2         125.7701921 MHz

     ====== GRADIENT CHANNEL =====
P16              1500.00 usec

      F1 - Acquisition parameters
ND0                    2
TD                   173
SFO1           125.7678 MHz
FIDRES         50.883335 Hz
SW               69.993 ppm

      F2 - Processing parameters
SI                  2048
SF          500.1300232 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00

      F1 - Processing parameters
SI                  2048
MC2                   QF
SF          125.7554348 MHz
WDW                 SINE
SSB                    2
LB                  0.00 Hz
GB                     0

        2D NMR plot parameters
CX2               15.00 cm
CX1               15.00 cm
F2PLO             7.908 ppm
F2LO             3954.99 Hz
F2PHI             3.445 ppm
F2HI             1722.85 Hz
F1PLO            133.691 ppm
F1LO            16812.31 Hz
F1PHI            63.692 ppm
F1HI             8009.66 Hz
F2PPMCM          0.29754 ppm/cm
F2HZCM         148.80952 Hz/cm
F1PPMCM          4.66655 ppm/cm
F1HZCM         586.84344 Hz/cm
```



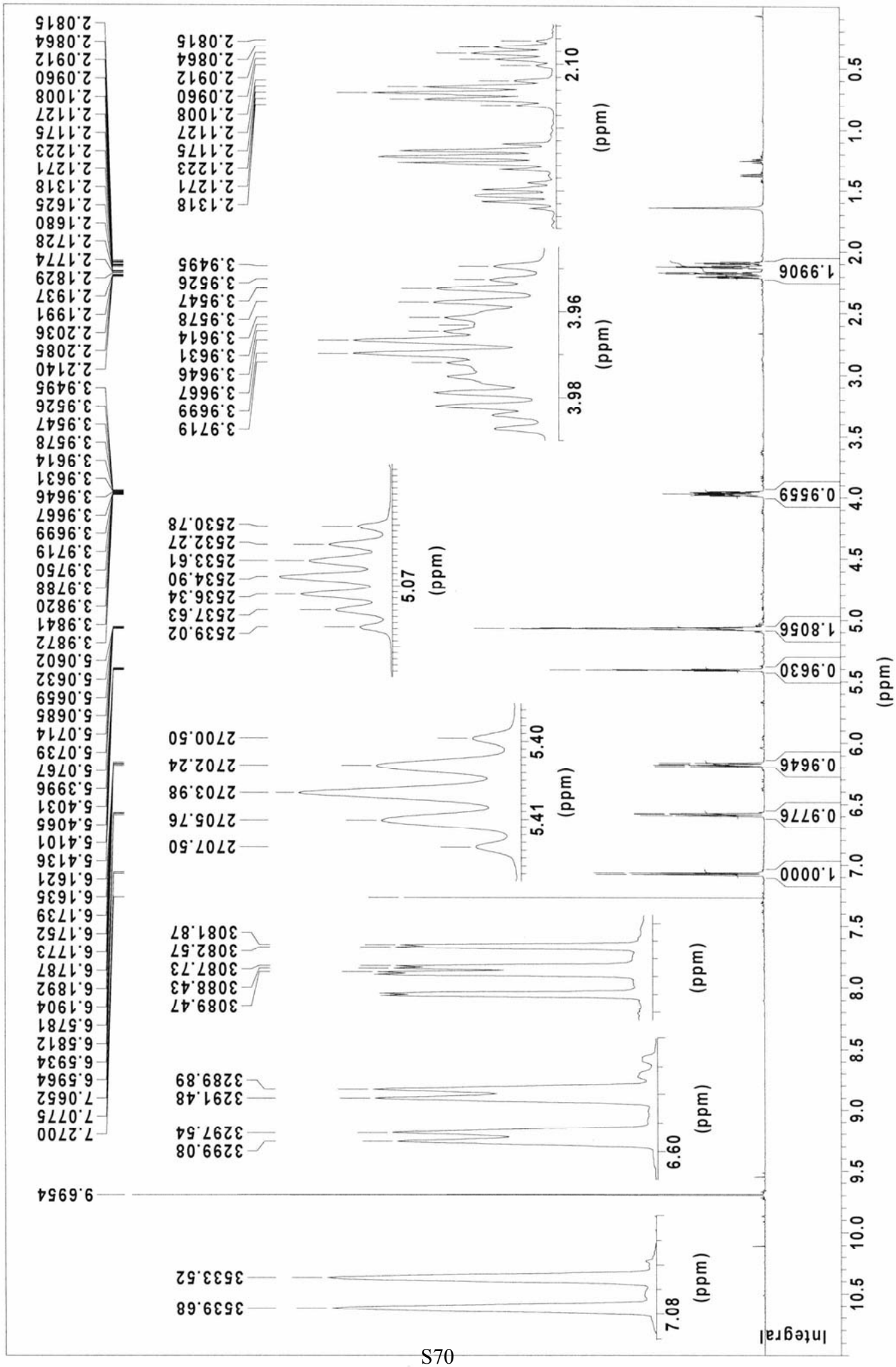
12a


s(8:10) Avg 0.905:1.089 min. from DATA:BIRB1170.D
EI-MS: F1090202
Mass/Charge
Abundance
144
115
174
157
128
102
89
77
63
50
190
208
223
227

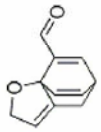

c:\first\data\galaxy\tmp8.abs Mon Apr 3 10:50:05:35 2006
12a
100
80
60
40
20
% Transmittance
3607.11
3020.71
2401.52
2283.86
1527.71
1425.48
1215.23
669.34
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



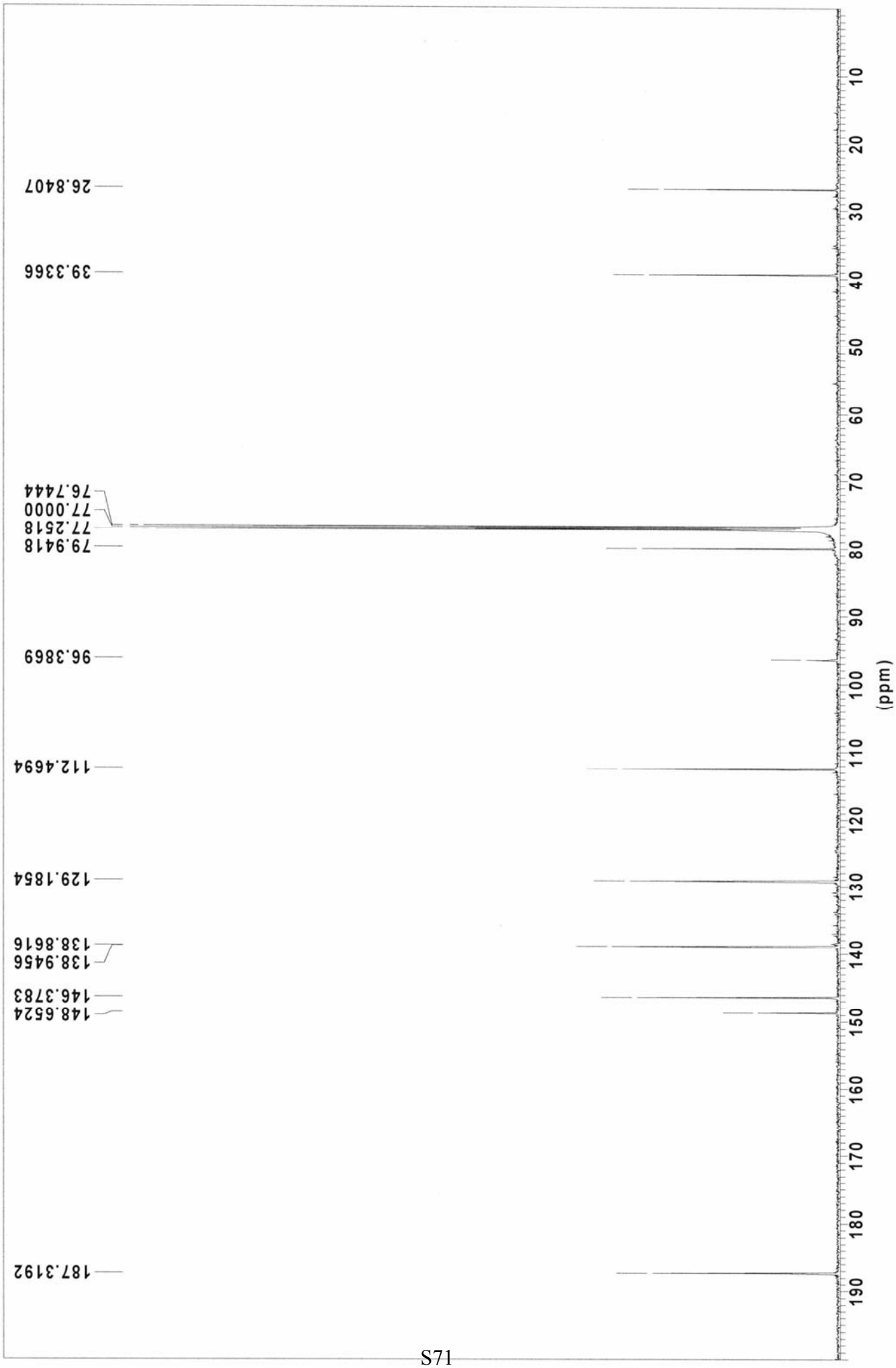
13a

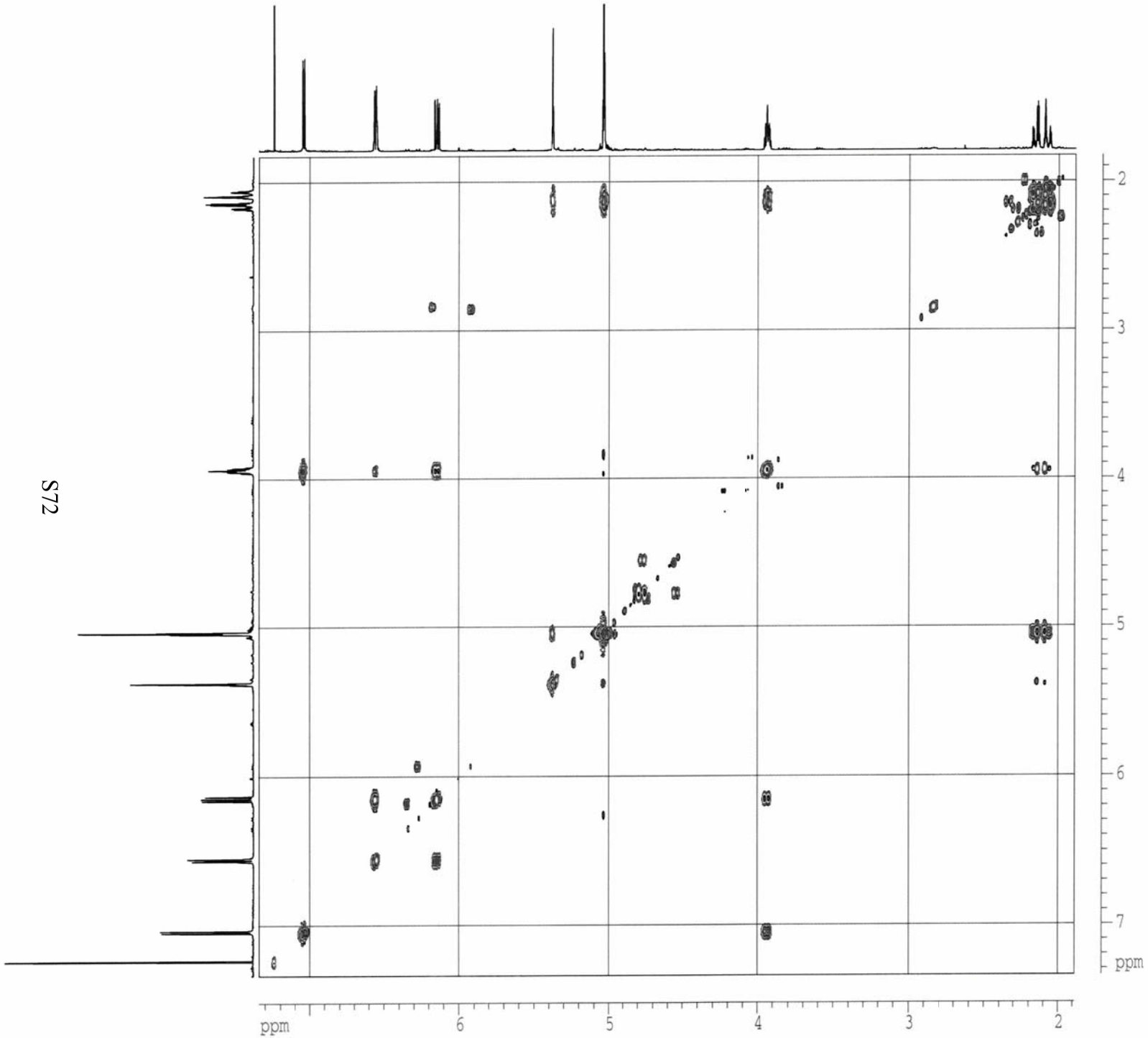


f1140102

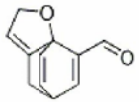


13a


26.8407
39.3366
76.7444
77.0000
77.2518
79.9418
96.3869
112.4694
129.1854
138.8616
138.9456
146.3783
148.6524
187.3192
(ppm)
f1140102



13a



Current Data Parameters

NAME	birb2768.bbo
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20060327
Time	13.58
INSTRUM	drx500
PROBHD	5 mm Multinucl
PULPROG	cosygs
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	4664.179 Hz
FIDRES	2.277431 Hz
AQ	0.2195956 sec
RG	574.7
DW	107.200 usec
DE	6.00 usec
TE	275.0 K
d0	0.00000300 sec
D1	2.00000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
IN0	0.00021440 sec

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

NUC1	1H
P0	6.00 usec
P1	9.00 usec
PL1	-3.00 dB
SFO1	500.1328449 MHz

====== GRADIENT CHANNEL =====

P16	6000.00 usec

F1 - Acquisition parameters

ND0	1
TD	256
SFO1	500.1328 MHz
FIDRES	18.219450 Hz
SW	9.326 ppm

F2 - Processing parameters

SI	2048
SF	500.1300232 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

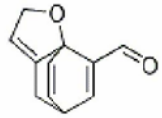
F1 - Processing parameters

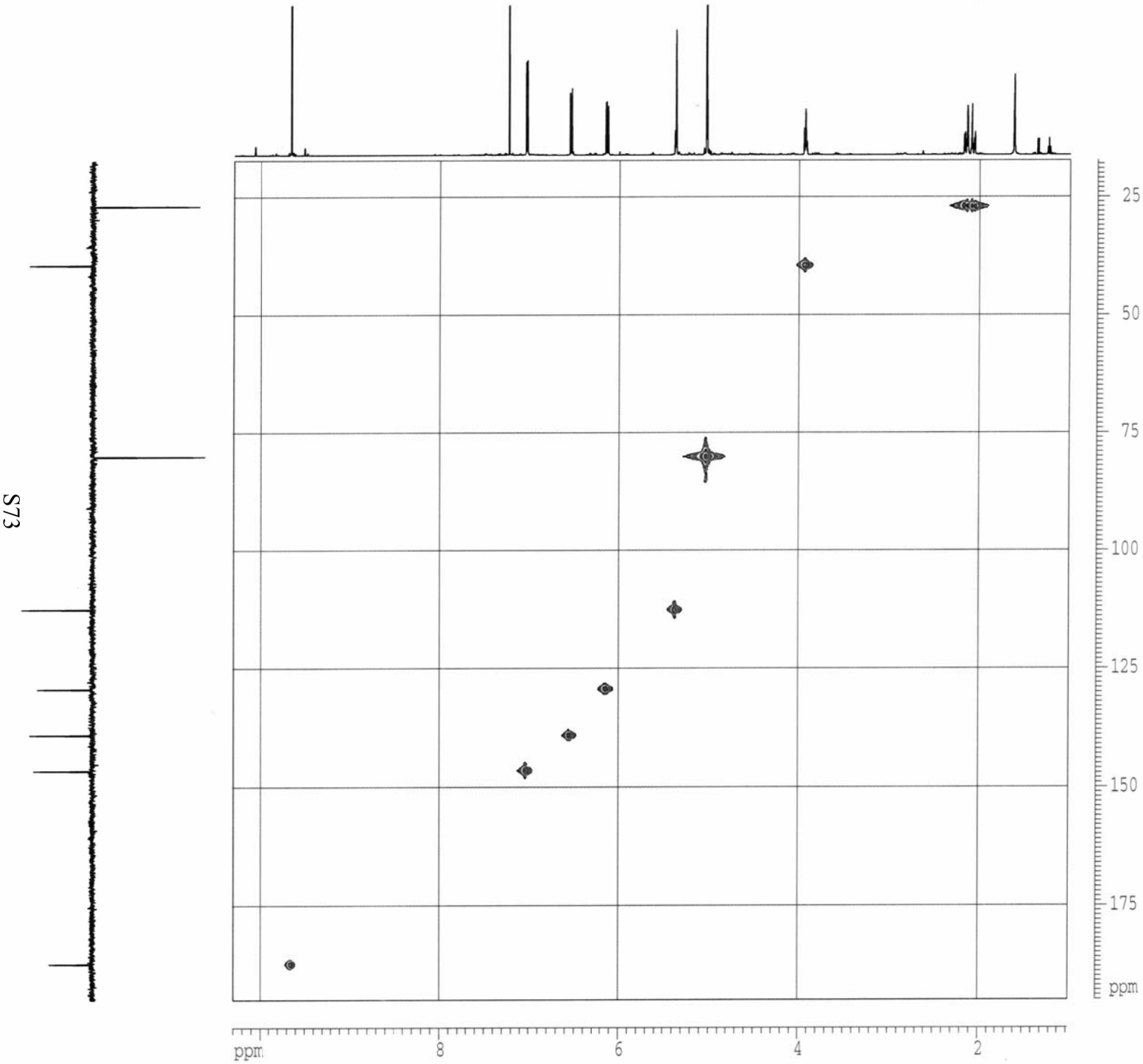
SI	512
MC2	QF
SF	500.1300193 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

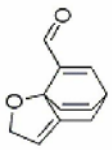
CX2	15.00 cm
CX1	15.00 cm
F2PLO	7.340 ppm
F2LO	3671.20 Hz
F2PHI	1.885 ppm
F2HI	942.84 Hz
F1PLO	7.344 ppm
F1LO	3672.78 Hz
F1PHI	1.825 ppm
F1HI	912.53 Hz
F2PPMCM	0.36369 ppm/cm
F2HZCM	181.89084 Hz/cm
F1PPMCM	0.36794 ppm/cm
F1HZCM	184.01643 Hz/cm

13a

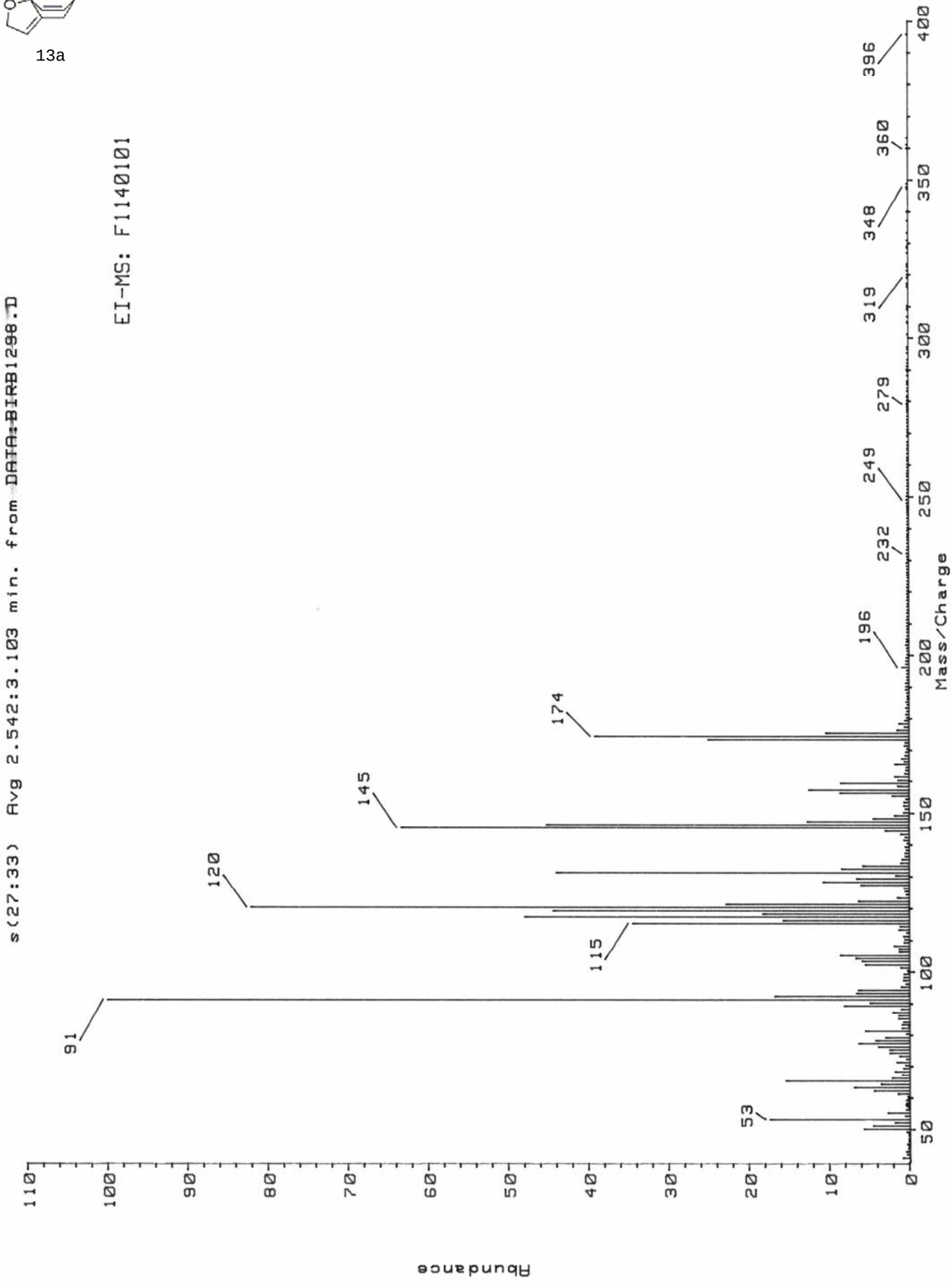


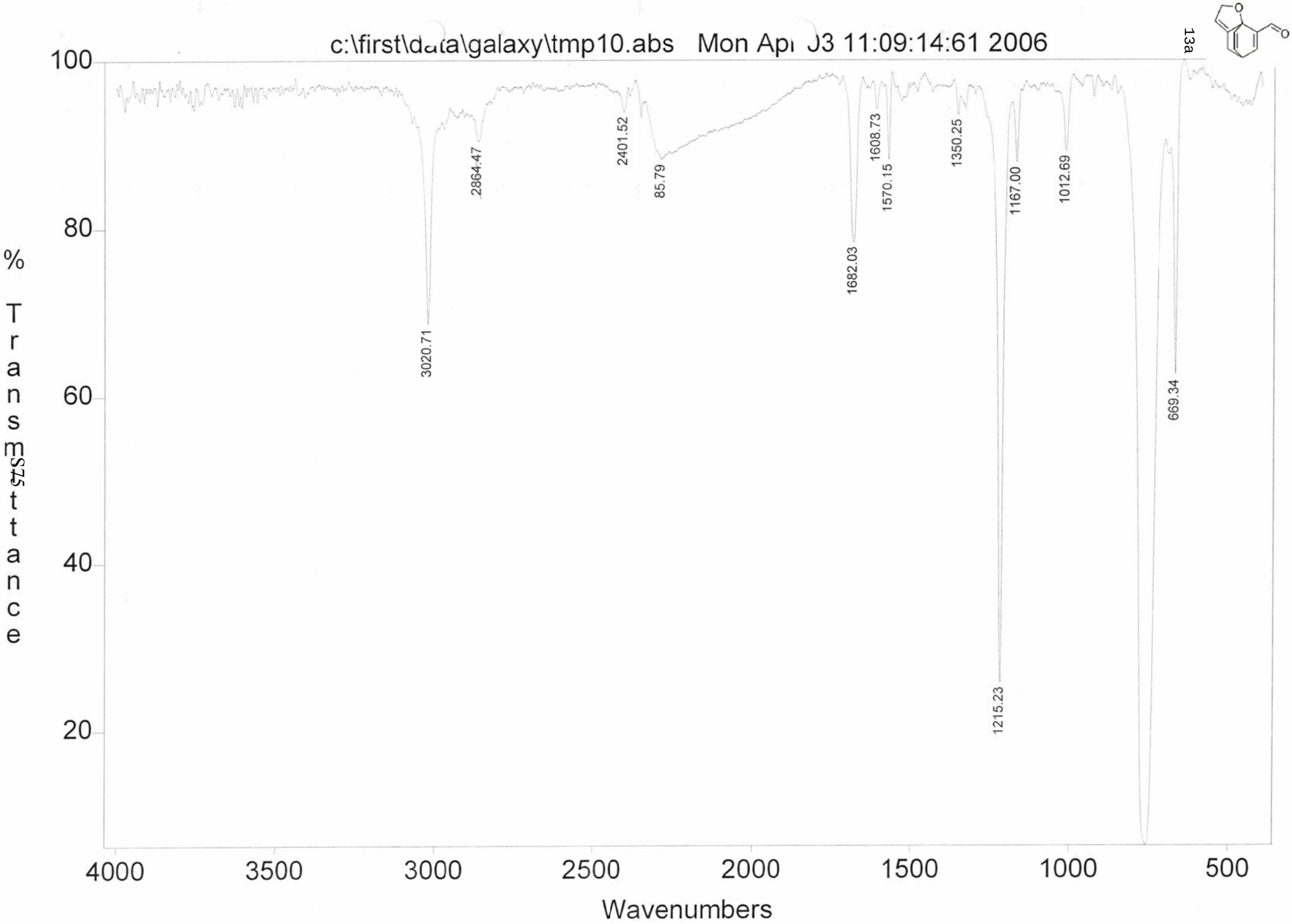


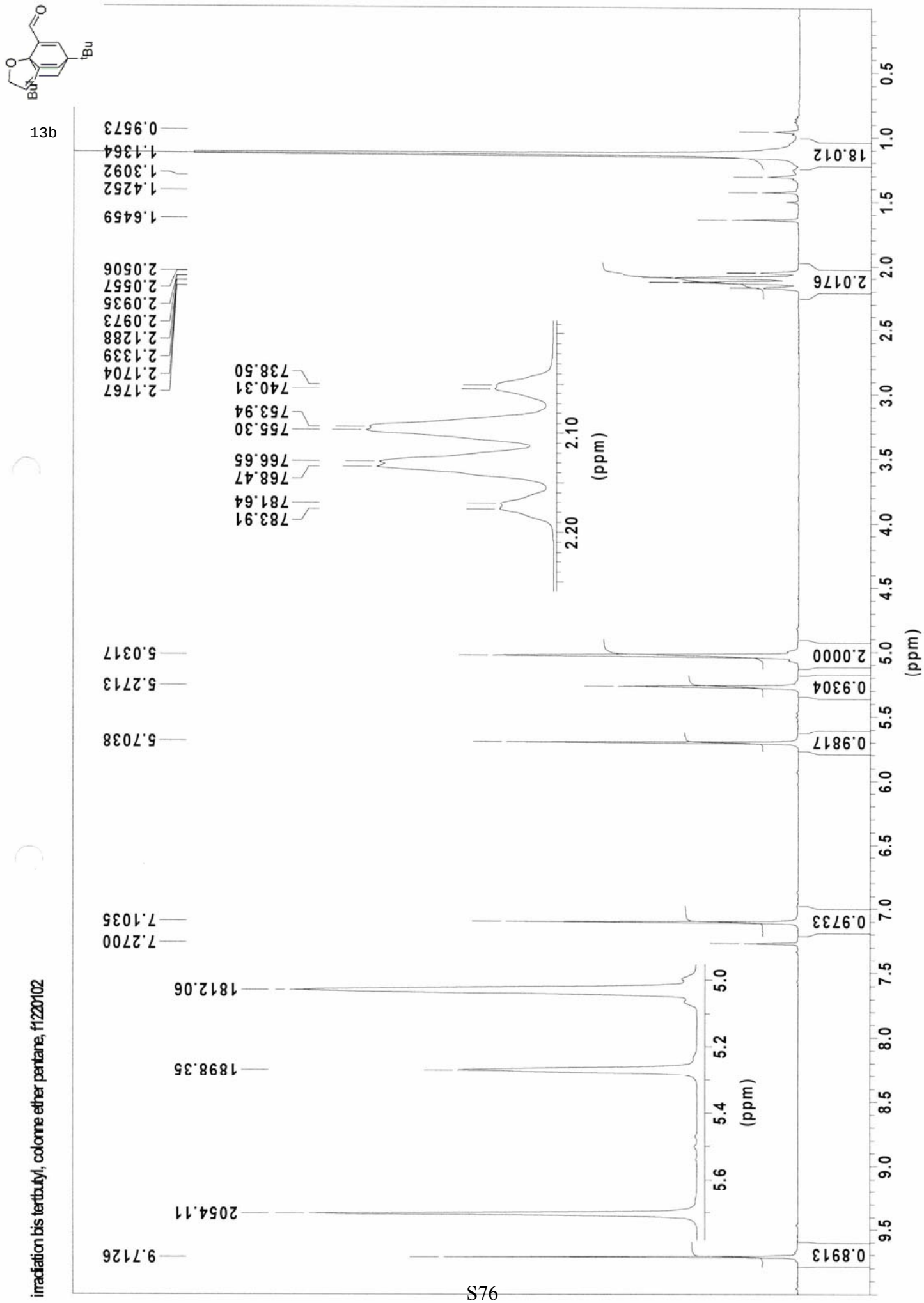

```
        Current Data Parameters
NAME        birb2768.bbo
EXPNO                  5
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20060327
Time              15.16
INSTRUM           drx500
PROBHD   5 mm Multinucl
PULPROG           inv4gs
TD                  2048
SOLVENT            CDCl3
NS                     8
DS                    16
SWH            4664.179 Hz
FIDRES         2.277431 Hz
AQ            0.2195956 sec
RG                 26008
DW              107.200 usec
DE                6.00 usec
TE               275.0 K
CNST2        145.0000000
d0            0.00000300 sec
D1            2.00000000 sec
d2            0.00344828 sec
d12           0.00002000 sec
d13           0.00000300 sec
D16           0.00020000 sec
d20           0.00192528 sec
IN0           0.00002235 sec

     ======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
p2                 16.40 usec
PL1                -3.00 dB
SFO1        500.1318449 MHz

     ======== CHANNEL f2 ========
CPDPRG2             garp
NUC2                 13C
P3                  7.00 usec
PCPD2              88.00 usec
PL2                -1.00 dB
PL12               17.00 dB
SFO2        125.7711247 MHz

     ====== GRADIENT CHANNEL =====
P16              1500.00 usec

      F1 - Acquisition parameters
ND0                    2
TD                   210
SFO1           125.7678 MHz
FIDRES        106.530312 Hz
SW             177.878 ppm

      F2 - Processing parameters
SI                  2048
SF          500.1300232 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00

      F1 - Processing parameters
SI                   512
MC2                   QF
SF          125.7544990 MHz
WDW                 SINE
SSB                    2
LB                  0.00 Hz
GB                     0

         2D NMR plot parameters
CX2                15.00 cm
CX1                15.00 cm
F2PLO            10.305 ppm
F2LO             5153.81 Hz
F2PHI              0.979 ppm
F2HI              489.63 Hz
F1PLO            195.081 ppm
F1LO             24532.38 Hz
F1PHI              17.184 ppm
F1HI              2161.01 Hz
F2PPMCM           0.62173 ppm/cm
F2HZCM         310.94528 Hz/cm
F1PPMCM          11.85981 ppm/cm
F1HZCM        1491.42432 Hz/cm
```

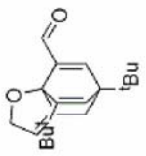


13a

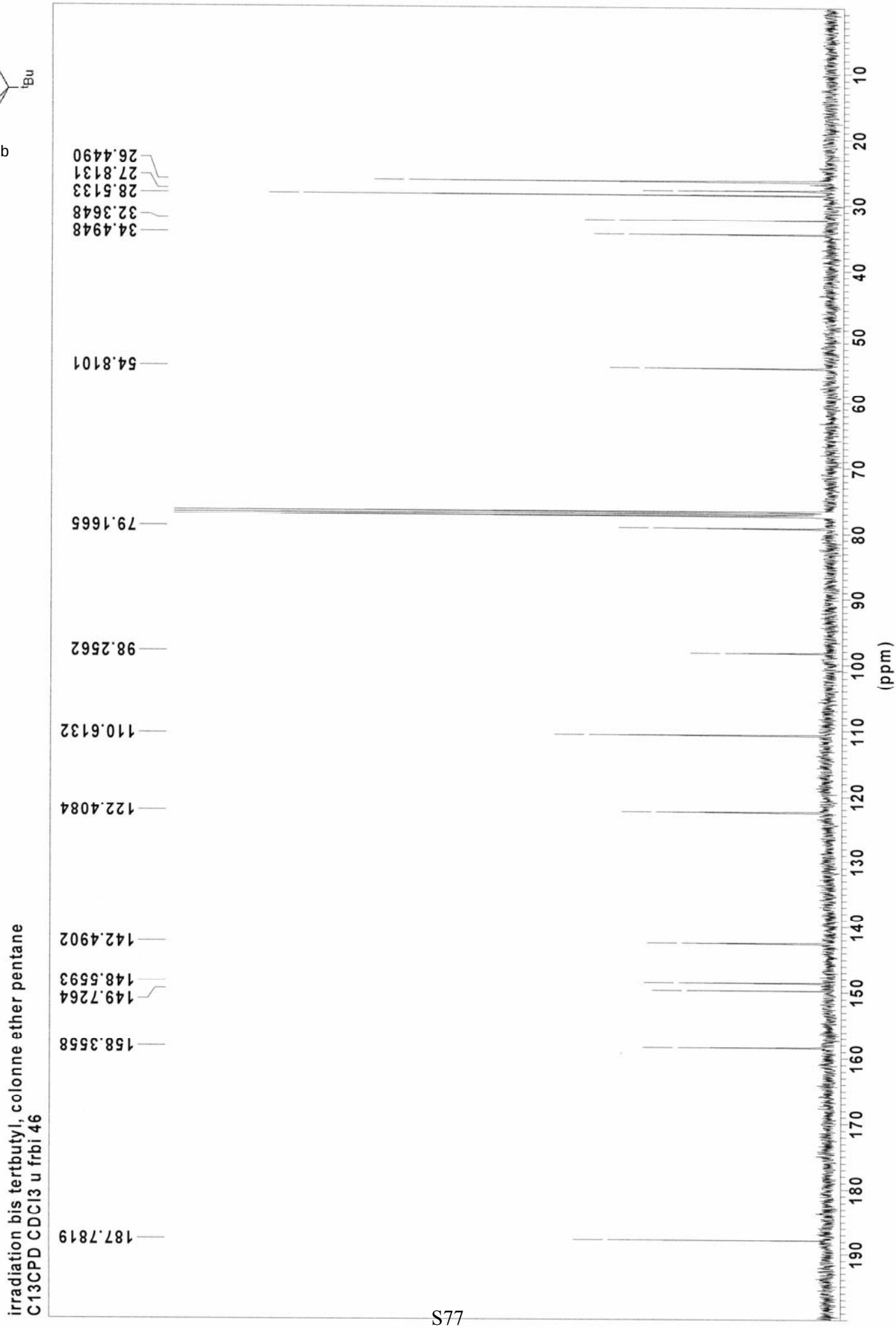

s(27:33) Avg 2.542:3.103 min. from DATA:BIRB1298.D
EI-MS: F1140101
Abundance
Mass/Charge
91
120
145
174
196
232
249
279
319
348
360
396
53
115


c:\first\data\galaxy\tmp10.abs Mon Apr 03 11:09:14:61 2006
13a
% Transmittance
100
80
60
40
20
3020.71
2864.47
2401.52
85.79
1682.03
1608.73
1570.15
1350.25
1215.23
1167.00
1012.69
669.34
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers

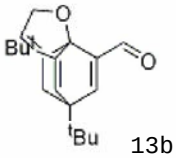

13b
irradiation bis tertbutyl, colonne ether pentane, f1220102
(ppm)
9.7126
2054.11
1898.35
1812.06
7.2700
7.1035
5.7038
5.2713
5.0317
2.1767
2.1704
2.1339
2.1288
2.0973
2.0935
2.0557
2.0506
1.6459
1.4252
1.3092
1.1364
0.9573
783.91
781.64
768.47
766.65
755.30
753.94
740.31
738.50
0.8913
18.012
2.0176
2.0000
0.9304
0.9817
0.9733



13b



irradiation bis tertbutyl, colonne ether pentane
C13CPD CDCl3 u frbi 46



13b

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F1220102

XMASS Mass Analysis for /Data/UNI_FR/BIRB1475_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

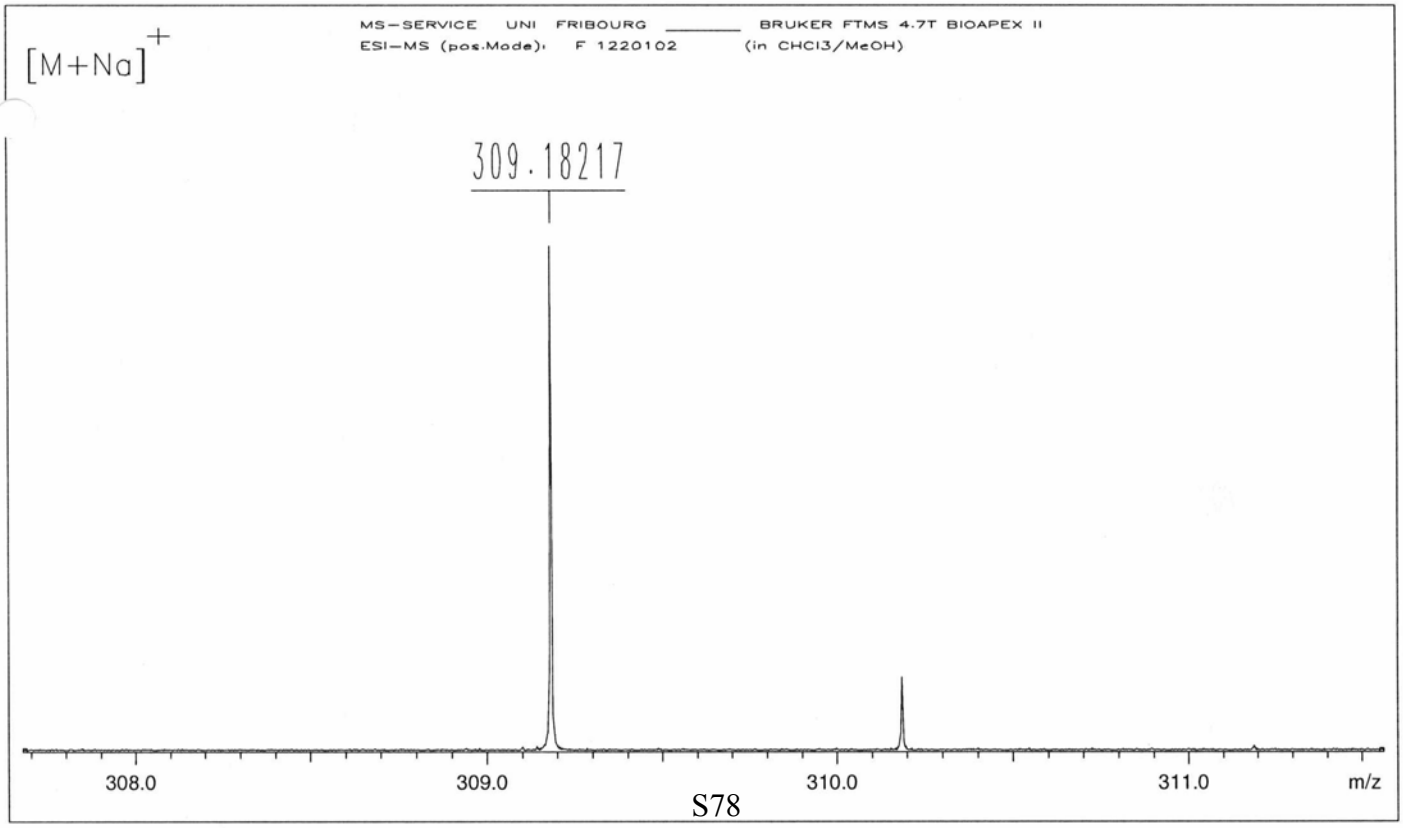
Ion mass = 309.1821710

Charge = +1

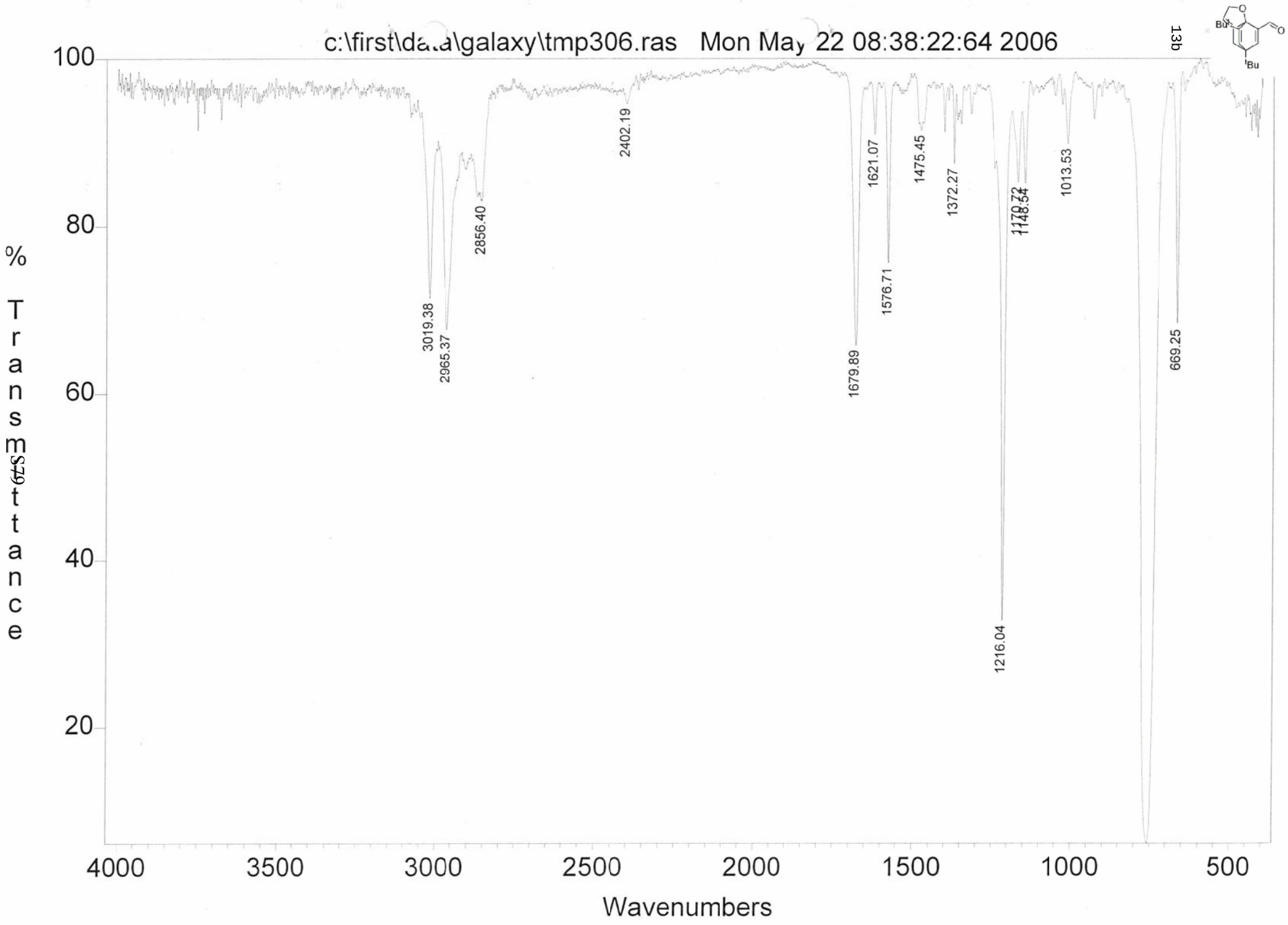
#	C	H	O	Na	mass	DBE	error

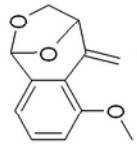
*** Mass Analysis for mass 309.1821710

1	19	26	2	1	309.1825012	6.5	3.302e-04
2	21	25	2	0	309.1849065	9.5	2.735e-03
3	17	25	5	0	309.1696503	5.5	1.252e-02
4	15	26	5	1	309.1672450	2.5	1.493e-02
5	16	30	4	1	309.2036305	1.5	2.146e-02
6	18	29	4	0	309.2060358	4.5	2.386e-02
7	20	21	3	0	309.1485210	10.5	3.365e-02
8	18	22	3	1	309.1461157	7.5	3.606e-02
9	16	21	6	0	309.1332648	6.5	4.891e-02

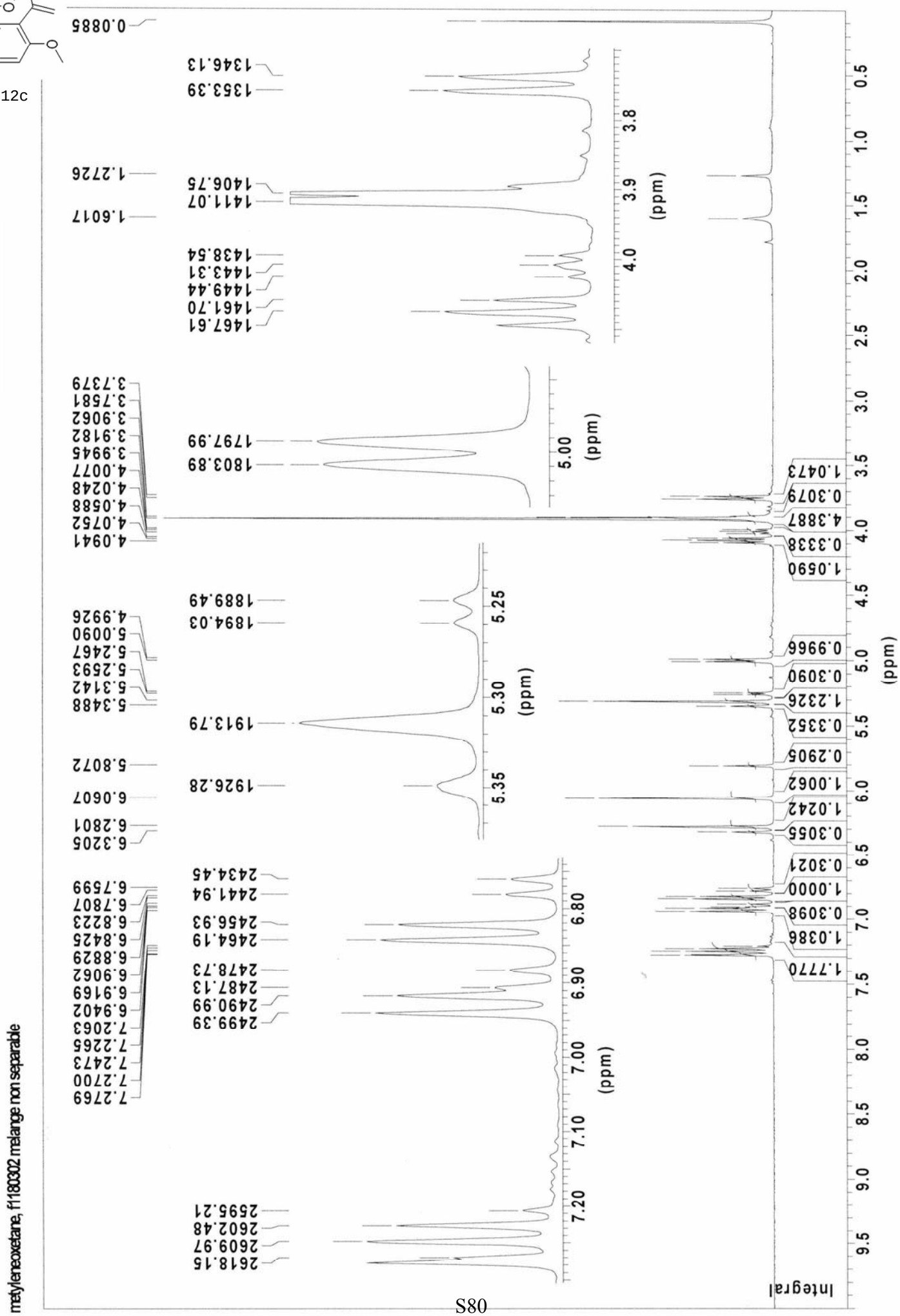


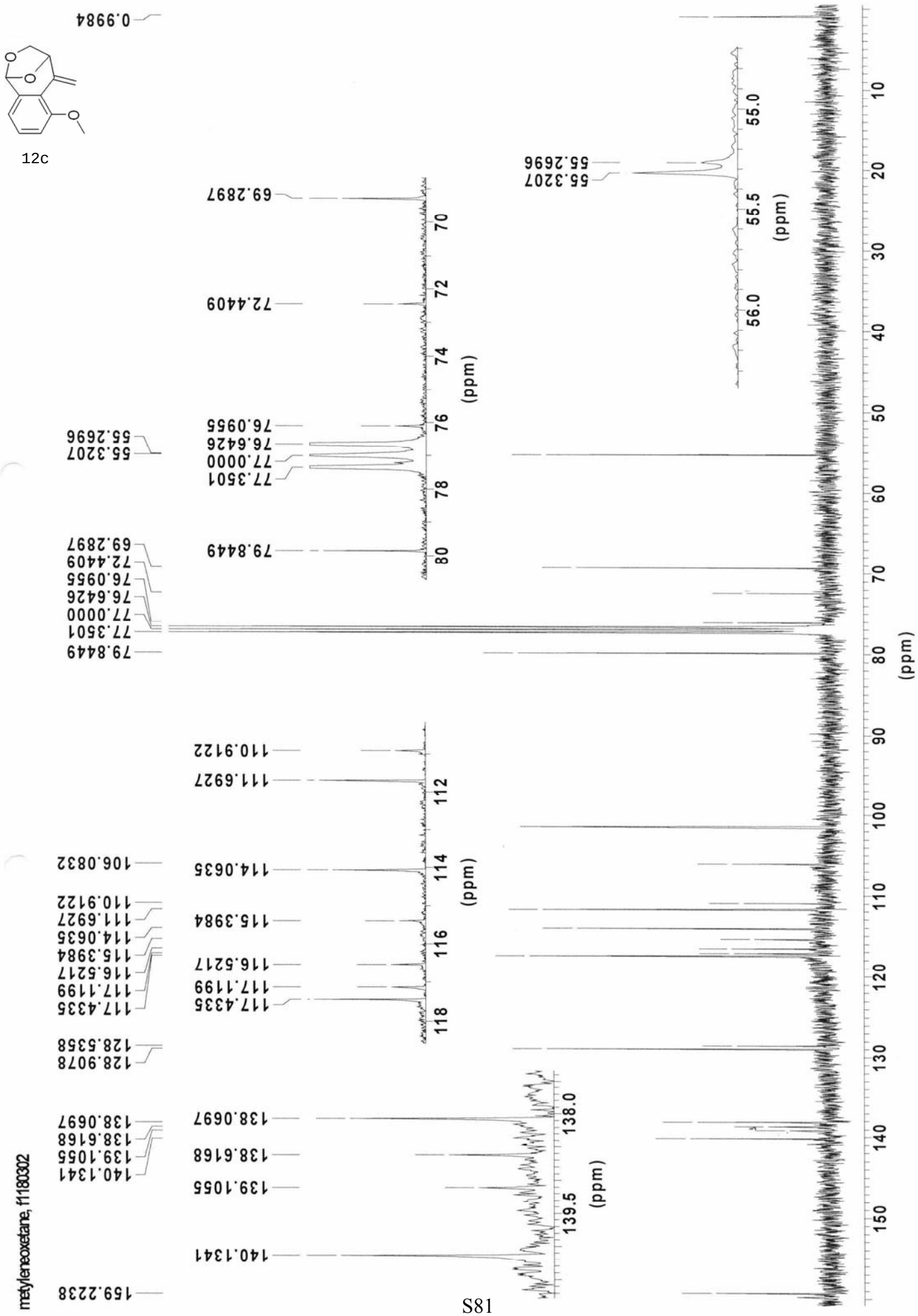
/Data/UNI_FR/BIRB1475_ESI/1/pdata/1 FTMS USER Tue May 23 14:20:24 2006

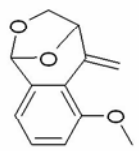

c:\first\data\galaxy\tmp306.ras Mon May 22 08:38:22:64 2006
13b
% Transmittance
Wavenumbers
100
80
60
40
20
4000
3500
3000
2500
2000
1500
1000
500
3019.38
2965.37
2856.40
2402.19
1679.89
1621.07
1576.71
1475.45
1372.27
1216.04
1170.72
1148.54
1013.53
669.25



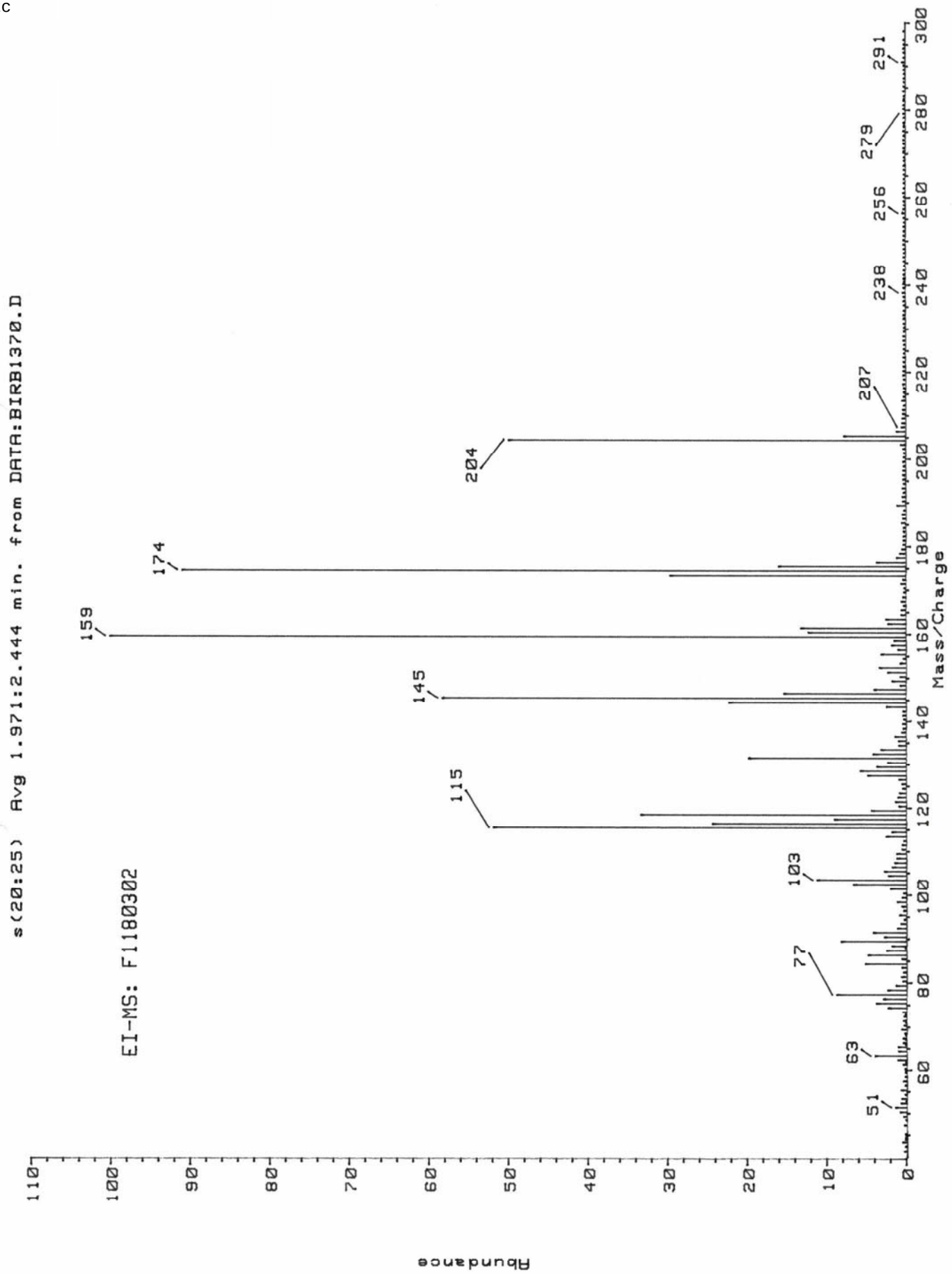
12c

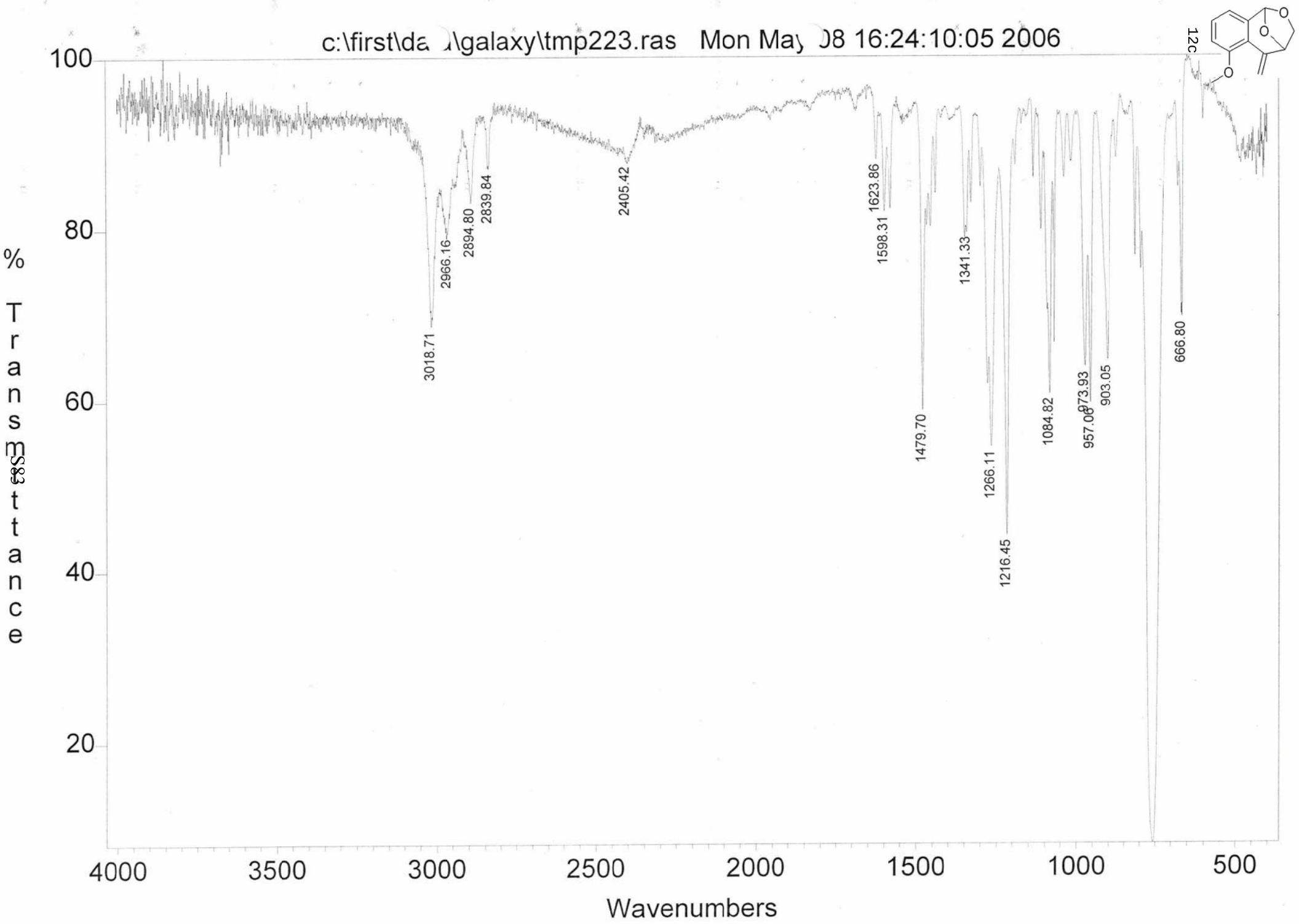

methylenedioxetane, f1180302 melange non separable
(ppm)
Integral

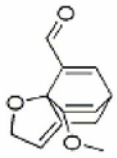

12c
methylenoxetane, f1180302
159.2238
140.1341
139.1055
138.6168
138.0697
128.9078
128.5358
117.4335
117.1199
116.5217
115.3984
114.0635
111.6927
110.9122
106.0832
79.8449
77.3501
77.0000
76.6426
76.0955
72.4409
69.2897
55.3207
55.2696
0.9984
(ppm)



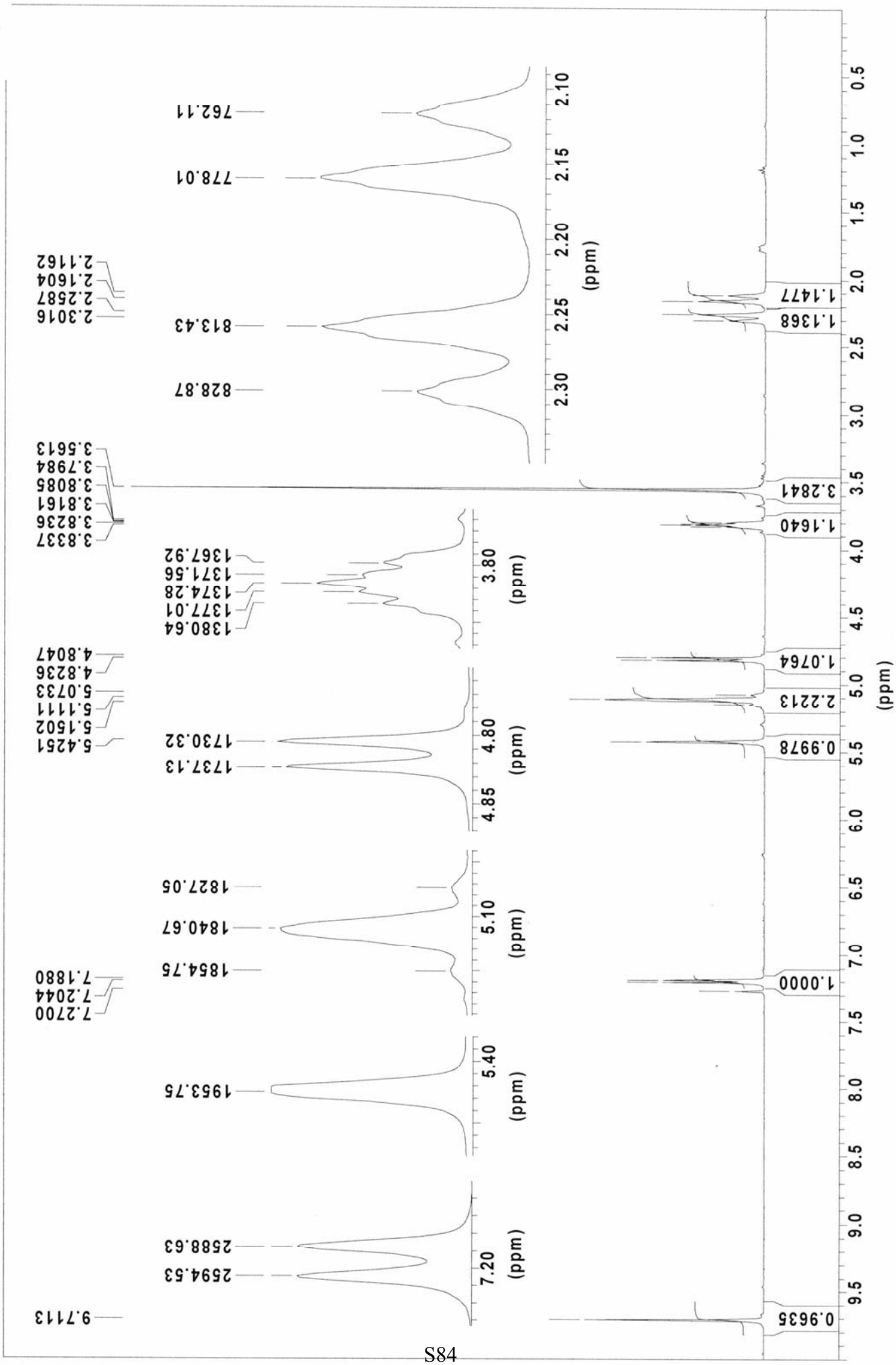
12c


s(20:25) Avg 1.971:2.444 min. from DATA:BIRB1370.D
EI-MS: F1180302
Mass/Charge
Abundance
51
63
77
103
115
145
159
174
204
207
238
256
279
291

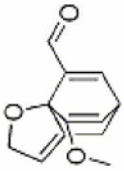

c:\first\da \galaxy\tmp223.ras Mon May 08 16:24:10:05 2006
12c
% Transmittance
100
80
60
40
20
3018.71
2966.16
2894.80
2839.84
2405.42
1623.86
1598.31
1479.70
1341.33
1266.11
1216.45
1084.82
957.08
973.93
903.05
666.80
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers



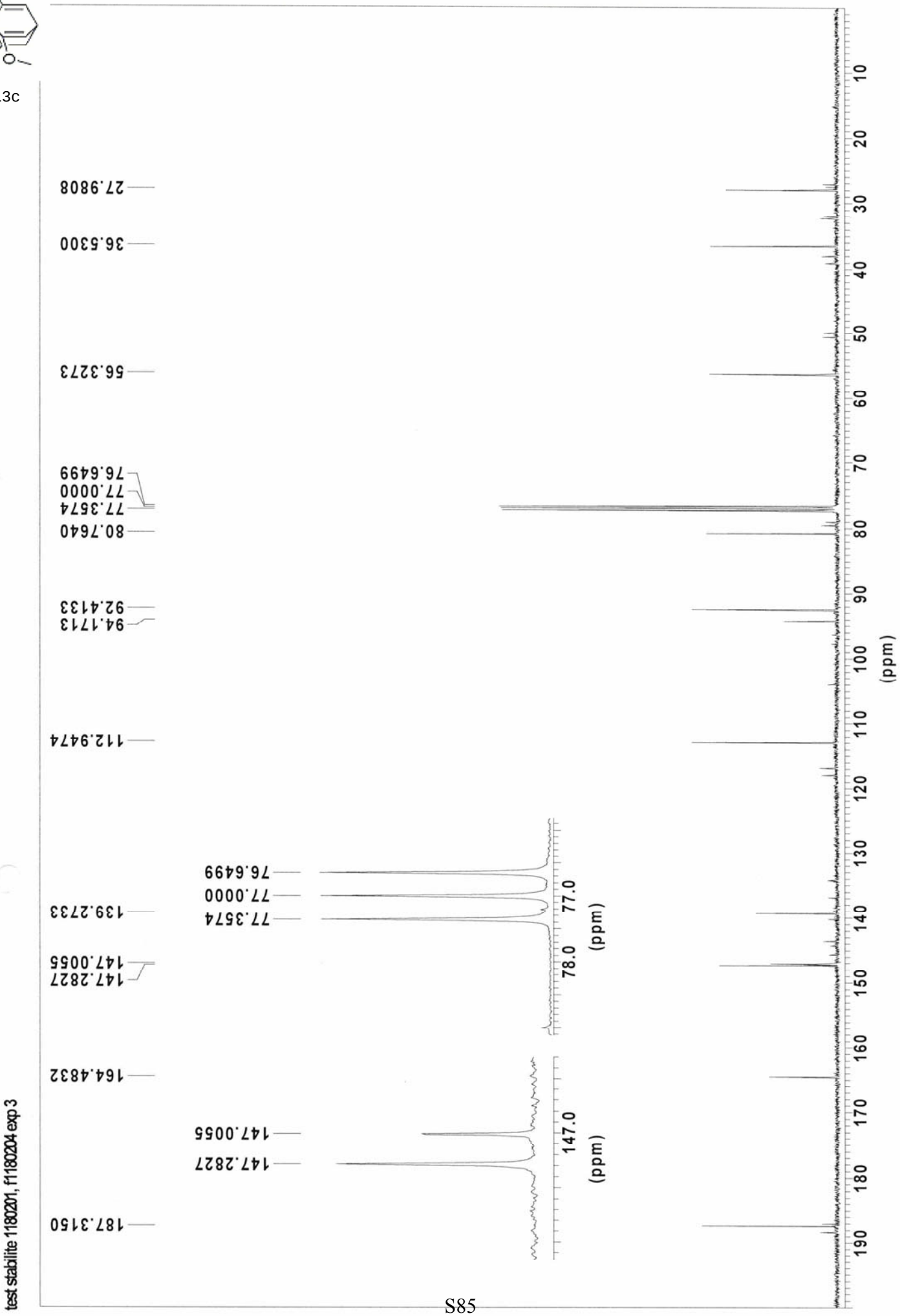
13c



tubes 30-42, colonne ether pentane, f1180202

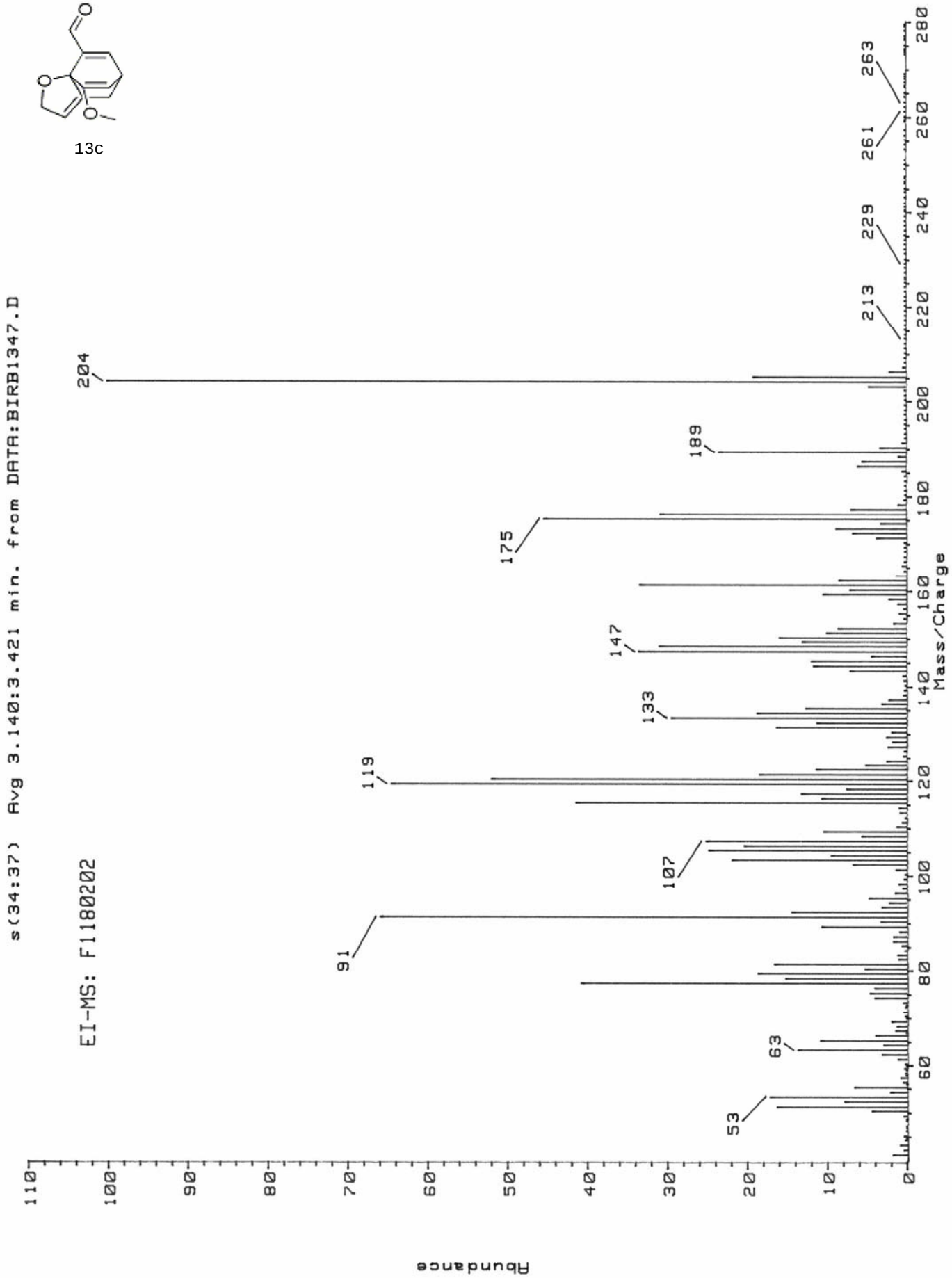


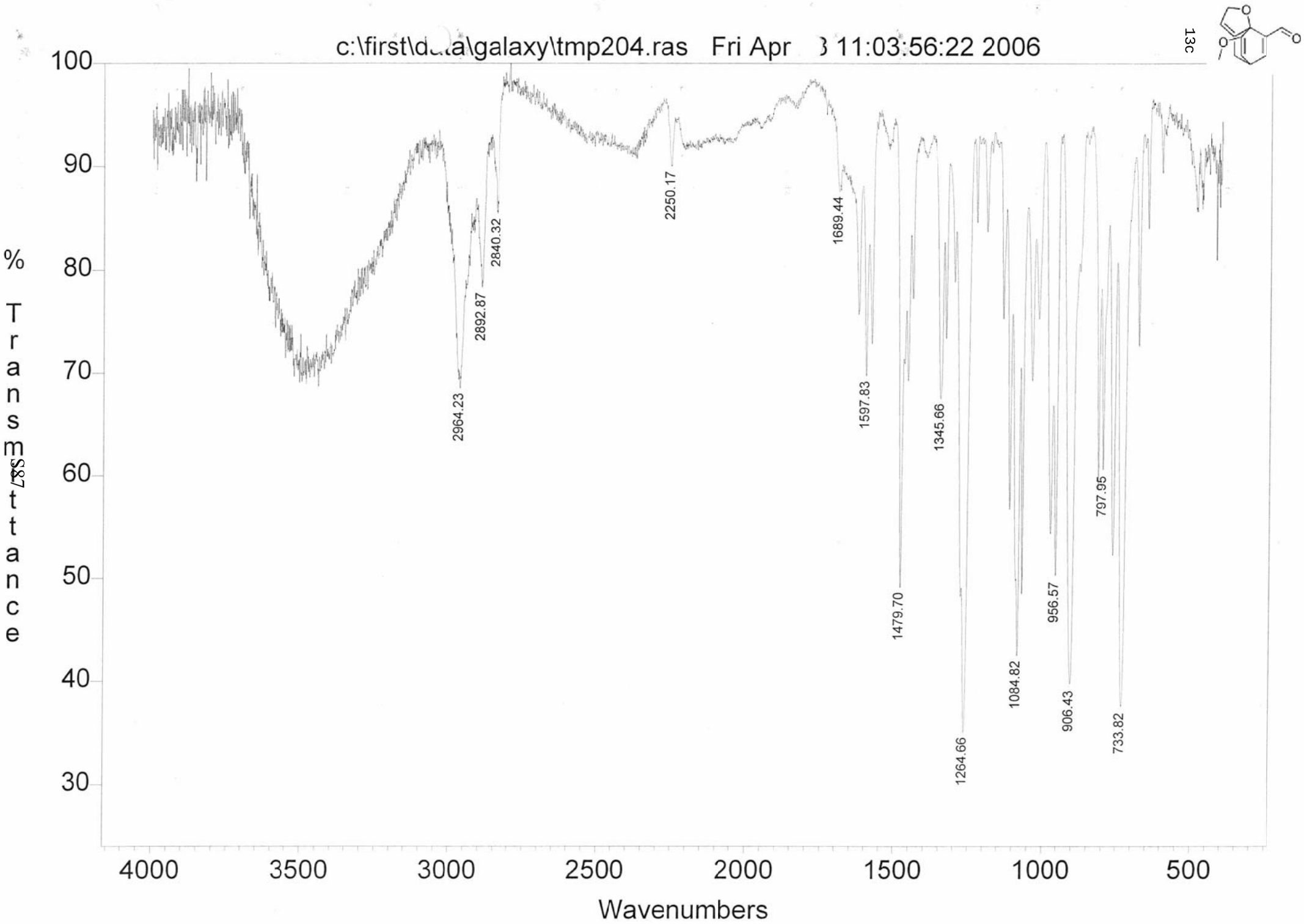
13c

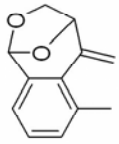


test stabilite 1180201, f1180204 exp 3

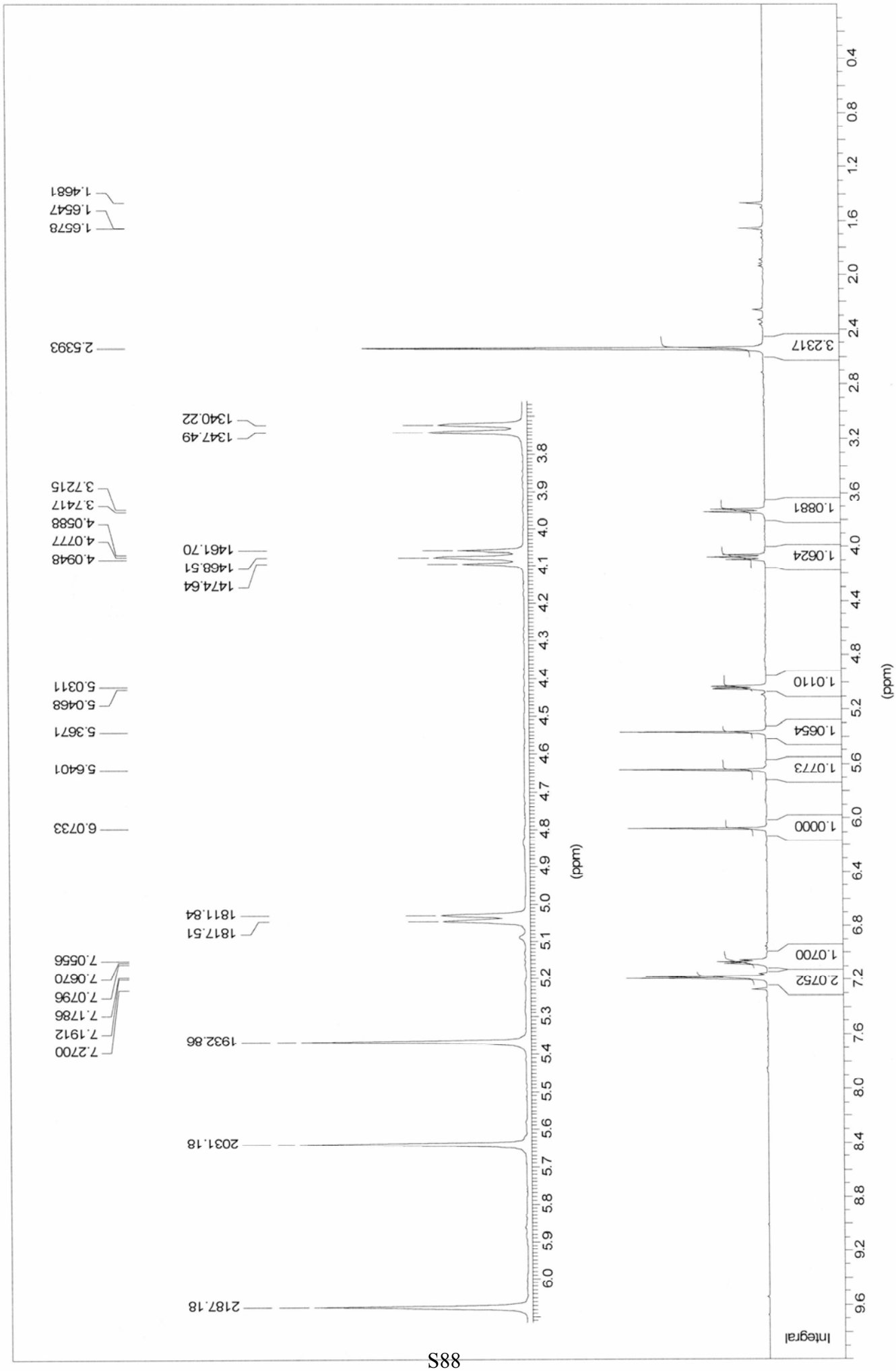
13c

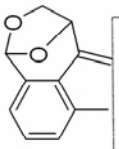

s(34:37) Avg 3.140:3.421 min. from DATA:BIRB1347.D
EI-MS: F1180202
204
189
175
147
133
119
107
91
63
53
213
229
261
263
Mass/Charge
Abundance


c:\first\data\galaxy\tmp204.ras Fri Apr 3 11:03:56:22 2006
13c
% Transmittance
Wavenumbers
100
90
80
70
60
50
40
30
4000
3500
3000
2500
2000
1500
1000
500
2964.23
2892.87
2840.32
2250.17
1689.44
1597.83
1479.70
1345.66
1264.66
1084.82
956.57
906.43
797.95
733.82

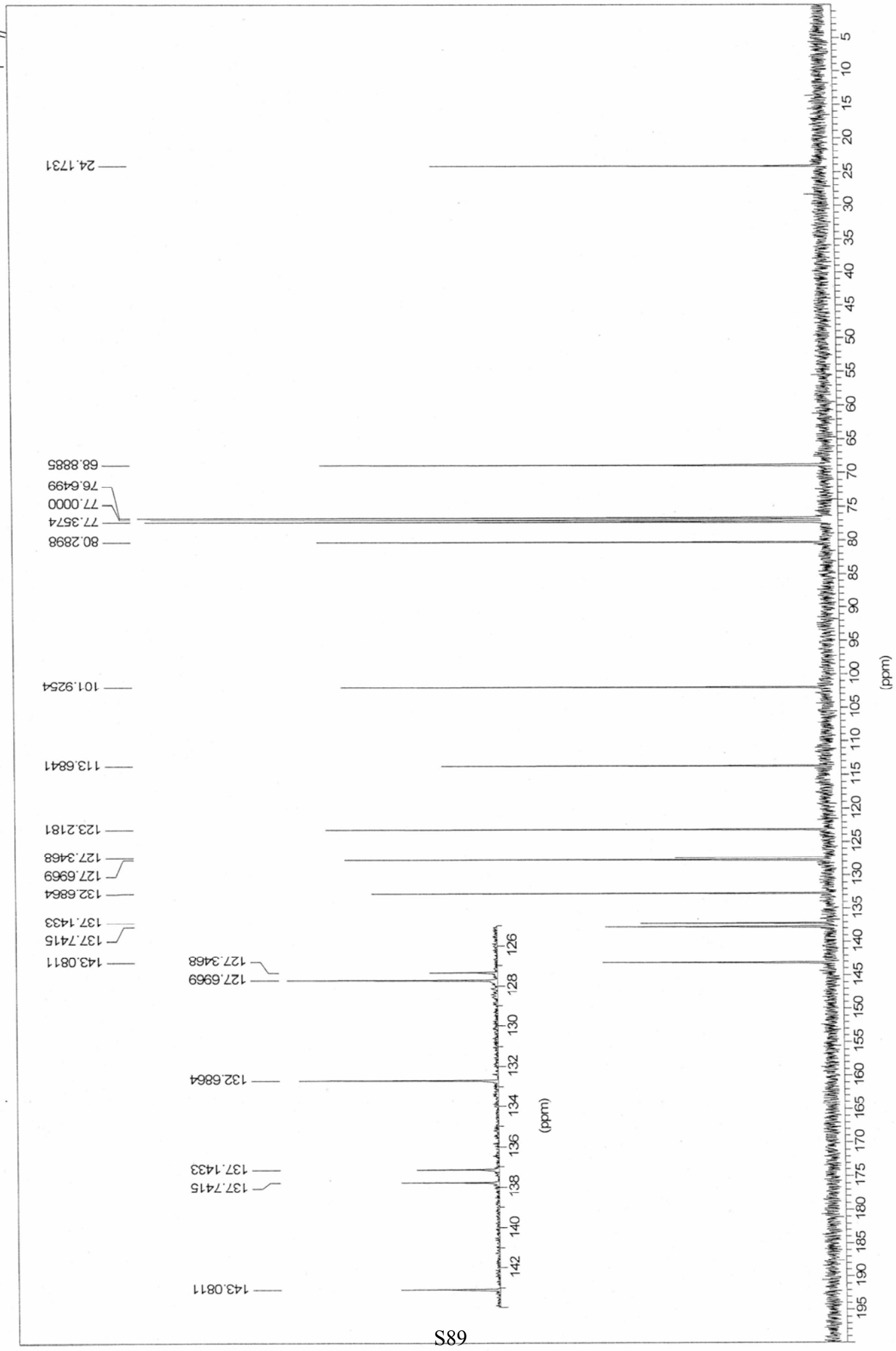


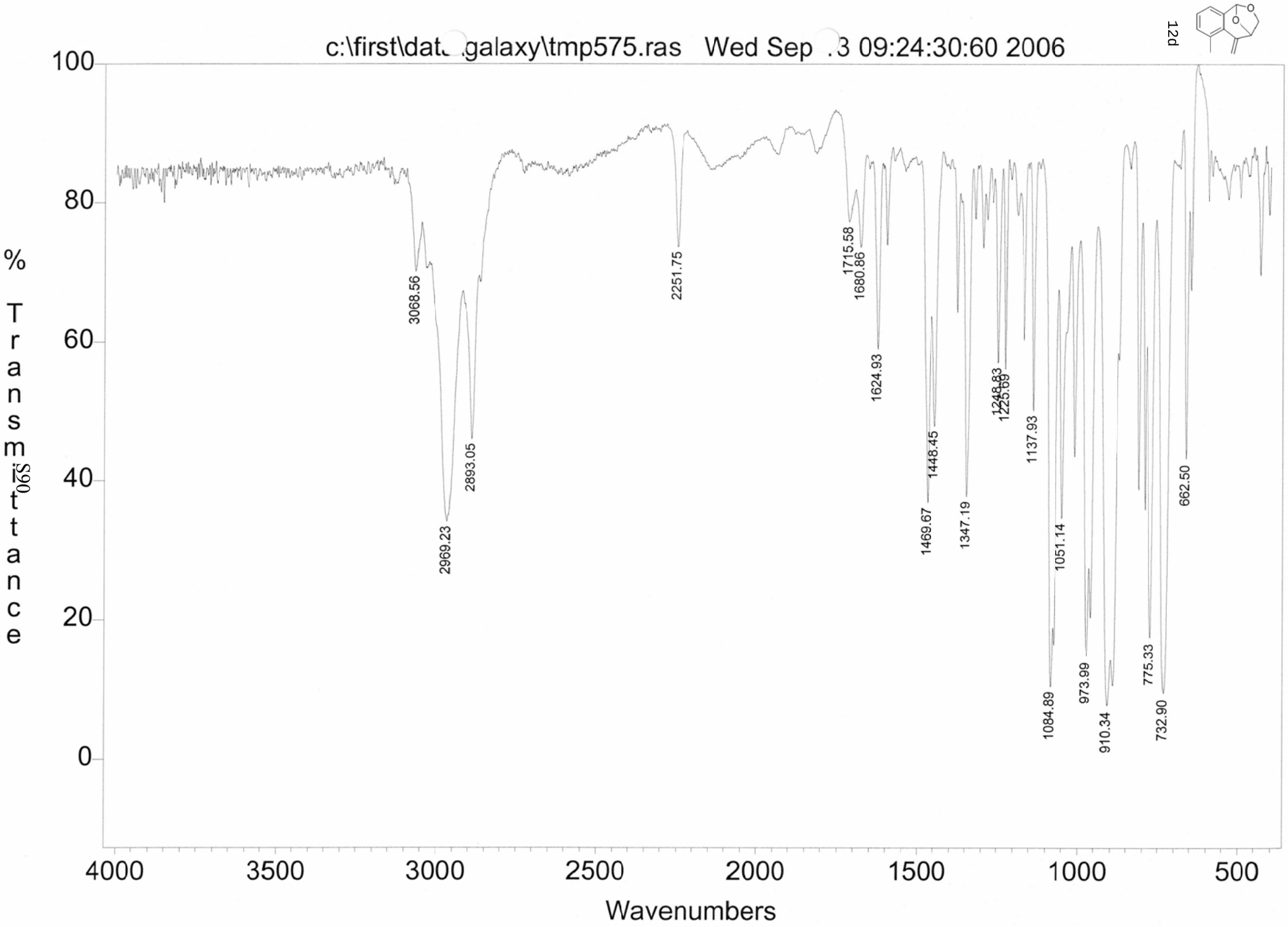
12d

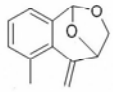

colonne ether pentane spot 2, f1340102
(ppm)
Integral
3.2317
1.0881
1.0624
1.0110
1.0654
1.0773
1.0000
1.0700
2.0752
2.5393
1.4681
1.6547
1.6578
7.2700
7.1912
7.1786
7.0796
7.0670
7.0556
6.0733
5.6401
5.3671
5.0468
5.0311
4.0948
4.0777
4.0588
3.7417
3.7215
2187.18
2031.18
1932.86
1817.51
1811.84
1474.64
1468.51
1461.70
1347.49
1340.22



12d


24.1731
68.8885
76.6499
77.0000
77.3574
80.2898
101.9254
113.6841
123.2181
127.3468
127.6969
132.6864
137.1433
137.7415
143.0811
(ppm)
colonne ether pentane spot 2, f1340102


c:\first\dat galaxy\tmp575.ras Wed Sep 3 09:24:30:60 2006
12d
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
3068.56
2969.23
2893.05
2251.75
1715.58
1680.86
1624.93
1469.67
1448.45
1347.19
1248.83
1225.69
1137.93
1084.89
1051.14
973.99
910.34
775.33
732.90
662.50



12d

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

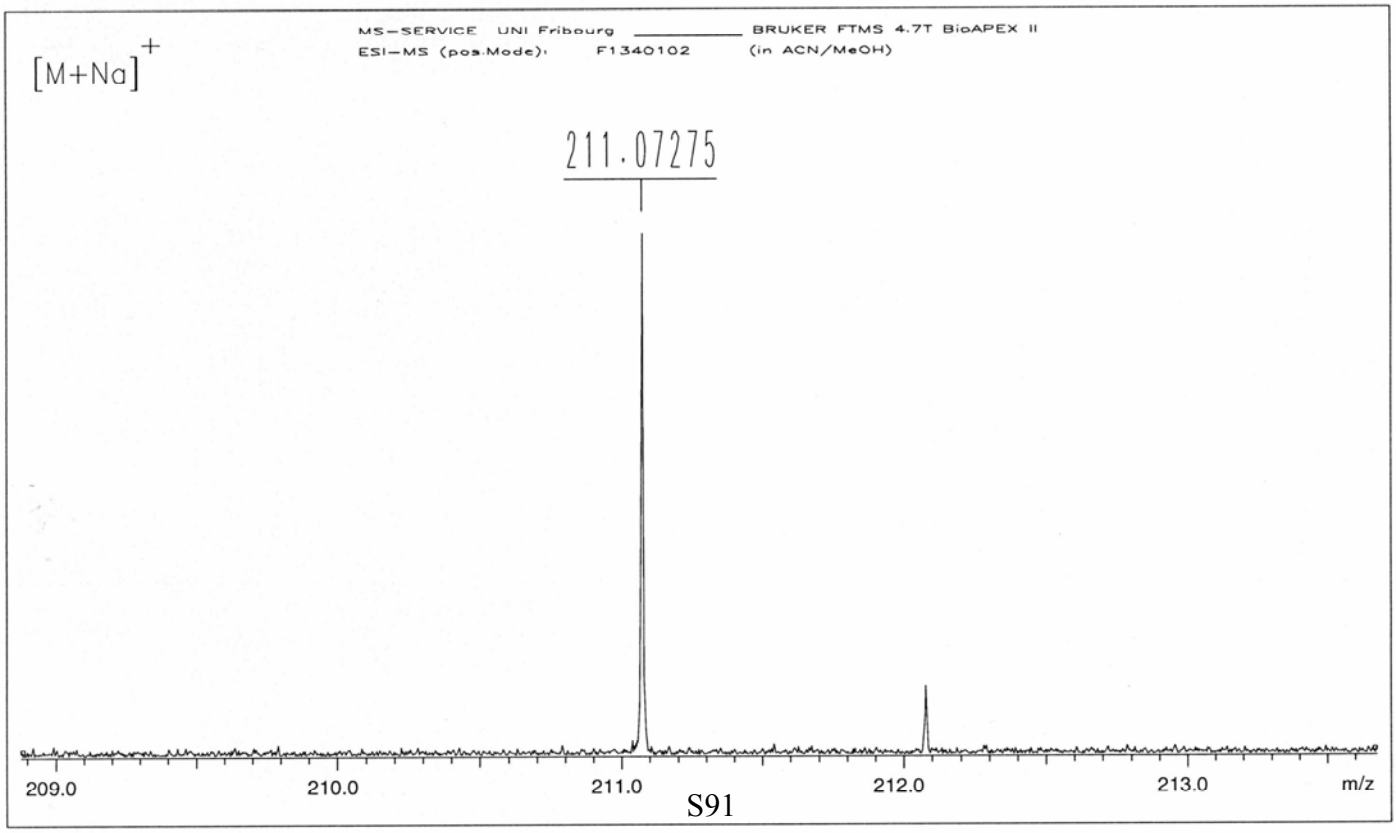
ESI-MS: F1340102

XMASS Mass Analysis for /Data/UNI_FR/BIRB1810_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

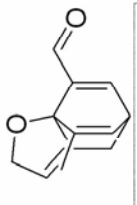
Ion mass = 211.0727510

Charge = +1

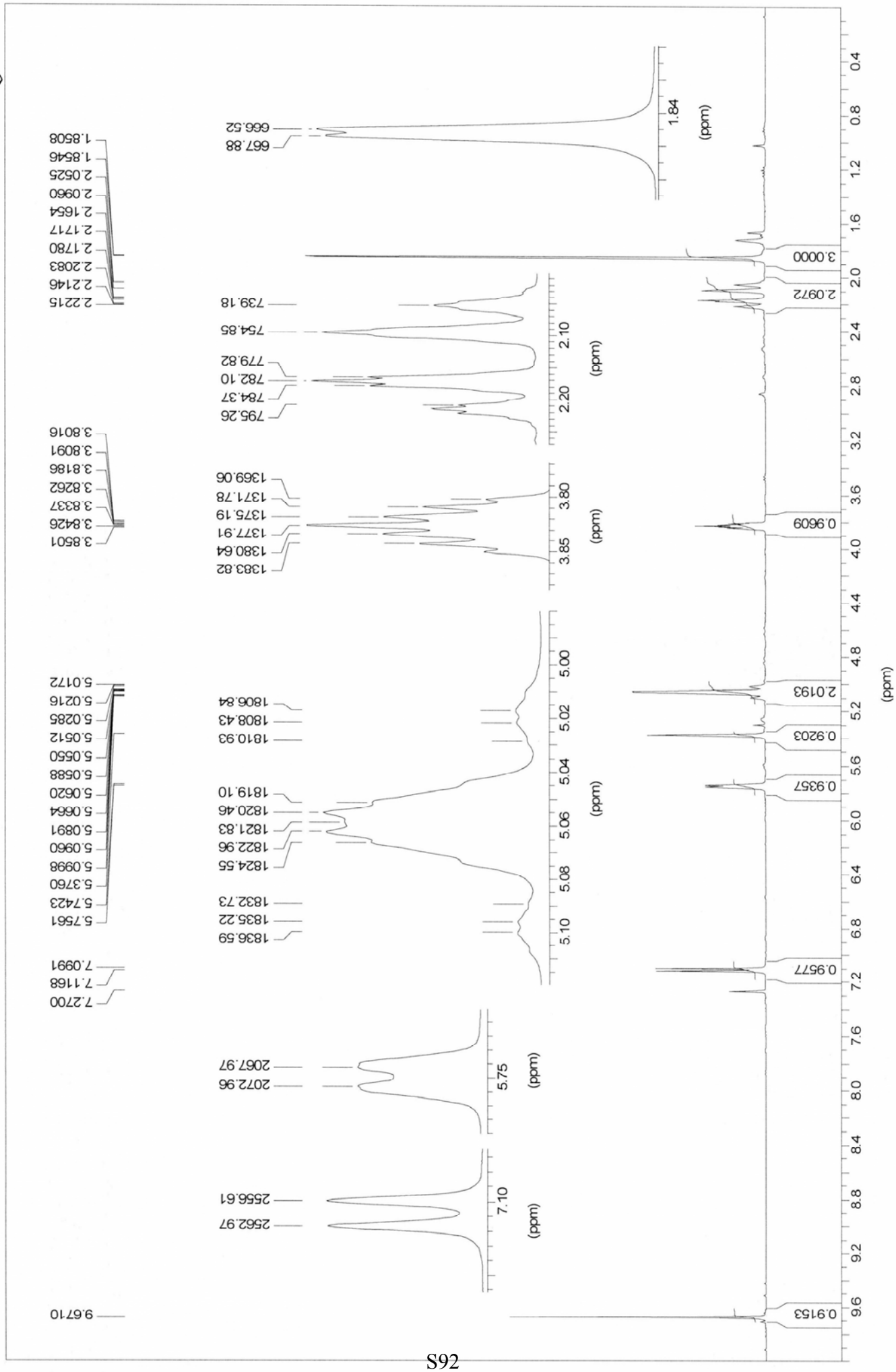
#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 211.0727510							
1	12	12	2	1	211.0729507	6.5	1.997e-04
2	14	11	2	0	211.0753560	9.5	2.605e-03
3	11	15	4	0	211.0964854	4.5	2.373e-02
4	13	7	3	0	211.0389705	10.5	3.378e-02
5	11	8	3	1	211.0365652	7.5	3.619e-02
6	13	16	1	1	211.1093362	5.5	3.659e-02
7	15	15	1	0	211.1117415	8.5	3.899e-02

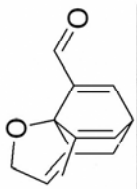


/Data/UNI_FR/BIRB1810_ESI/1/pdata/1 FTMS USER Wed Sep 20 16:08:44 2006

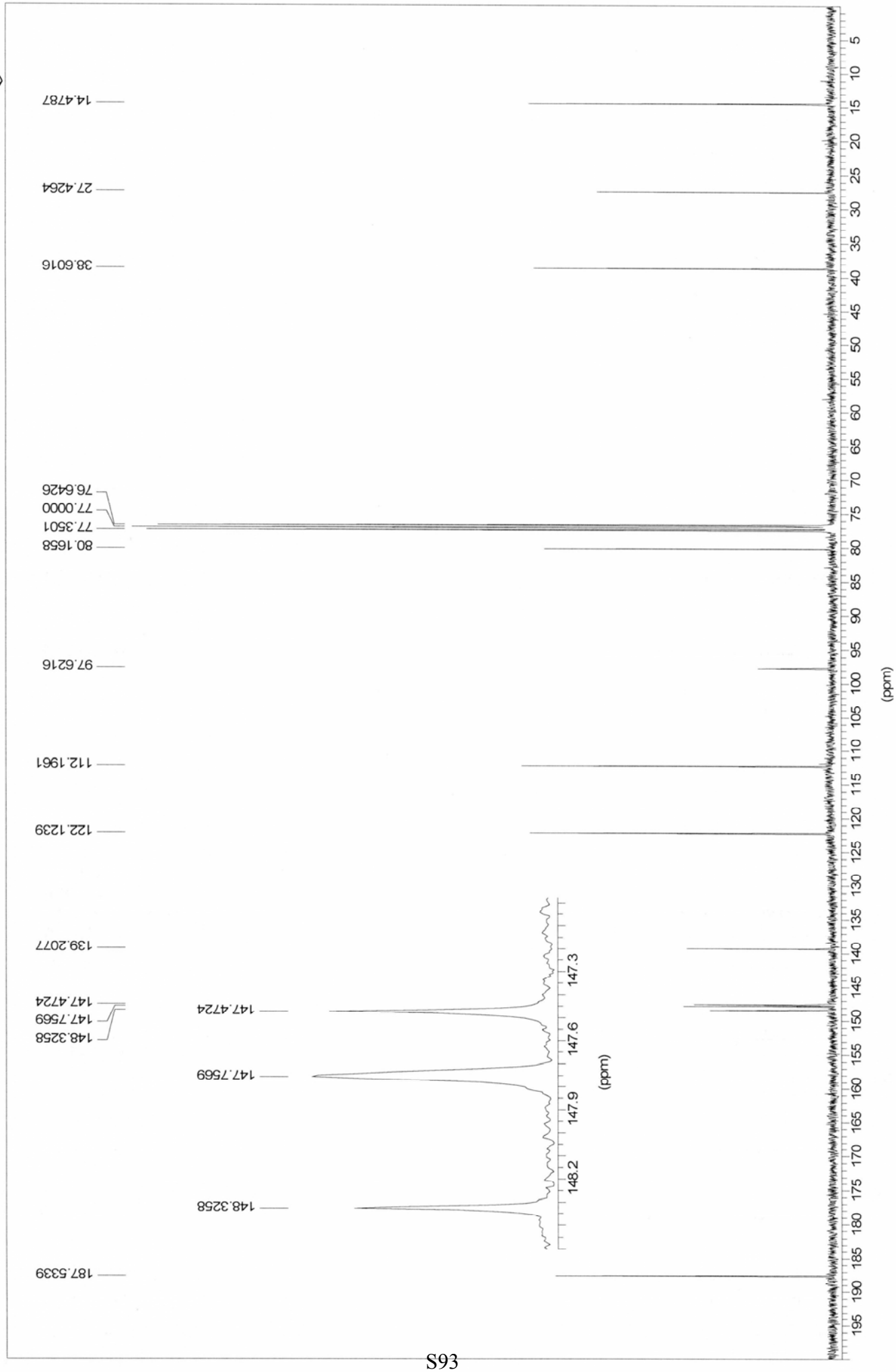


13d

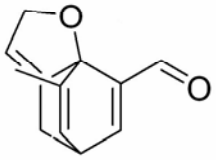

colonne 2 spot 1, f1340105
9.6710
7.2700
7.1168
7.0991
5.7561
5.7423
5.3760
5.0998
5.0960
5.0891
5.0664
5.0620
5.0588
5.0550
5.0512
5.0285
5.0216
5.0172
3.8501
3.8426
3.8337
3.8262
3.8186
3.8091
3.8016
2.2215
2.2146
2.2083
2.1780
2.1717
2.1654
2.0960
2.0525
1.8546
1.8508
2562.97
2556.61
2072.96
2067.97
1836.59
1835.22
1832.73
1824.55
1822.96
1821.83
1820.46
1819.10
1810.93
1808.43
1806.84
1383.82
1380.64
1377.91
1375.19
1371.78
1369.06
795.26
784.37
782.10
779.82
754.85
739.18
667.88
666.52
7.10
5.75
5.10
5.06
5.04
5.02
5.00
3.85
3.80
2.20
2.10
1.84
(ppm)
0.9153
0.9577
0.9357
0.9203
2.0193
0.9609
2.0972
3.0000



13d



colonne 2 spot 1, f1340105



13d

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F1340105

XMASS Mass Analysis for /Data/UNI_FR/BIRB1811_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

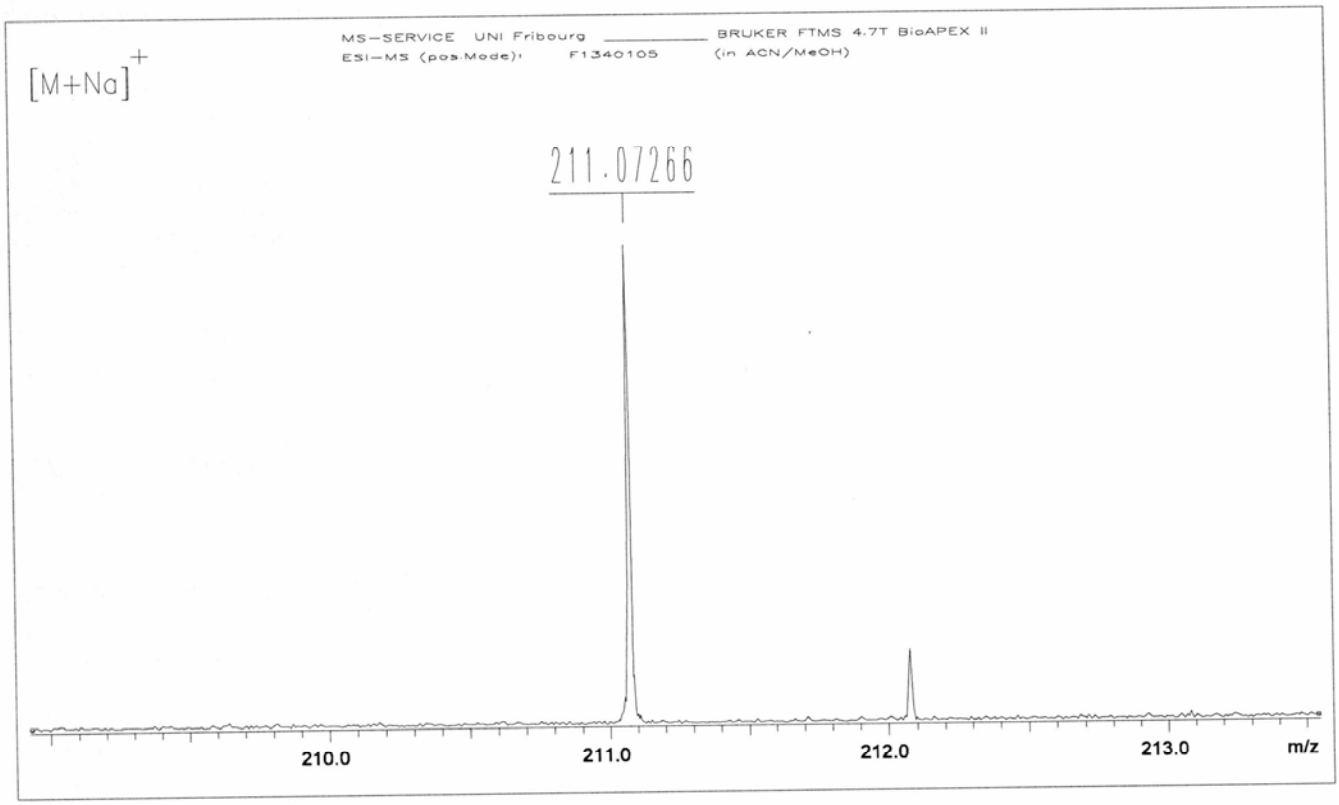
Ion mass = 211.0726640

Charge = +1

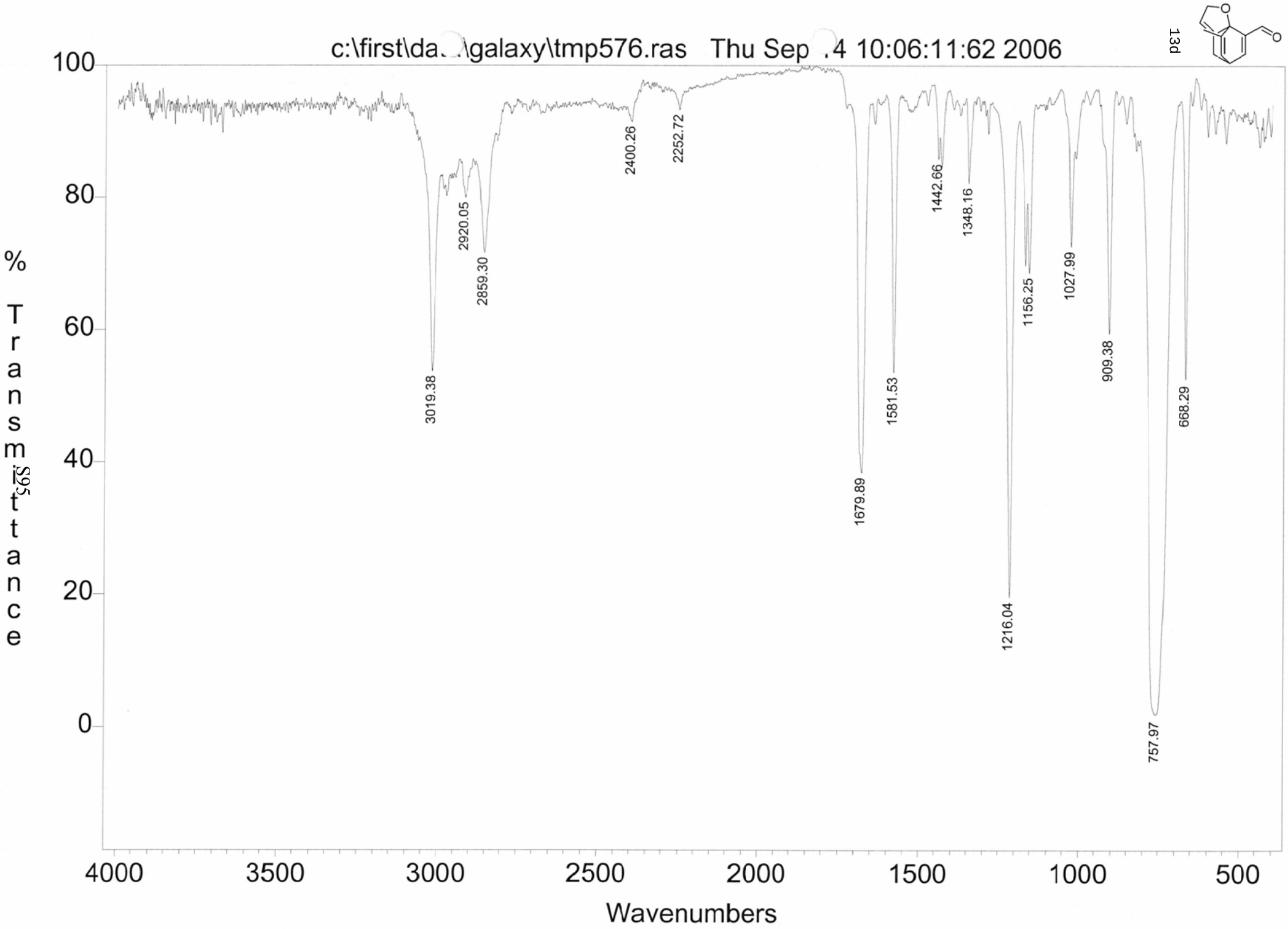
#	C	H	O	Na	mass	DBE	error

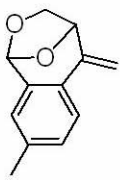
*** Mass Analysis for mass 211.0726640

1	12	12	2	1	211.0729507	6.5	2.867e-04
2	14	11	2	0	211.0753560	9.5	2.692e-03
3	11	15	4	0	211.0964854	4.5	2.382e-02
4	13	7	3	0	211.0389705	10.5	3.369e-02
5	11	8	3	1	211.0365652	7.5	3.610e-02
6	13	16	1	1	211.1093362	5.5	3.667e-02
7	15	15	1	0	211.1117415	8.5	3.908e-02

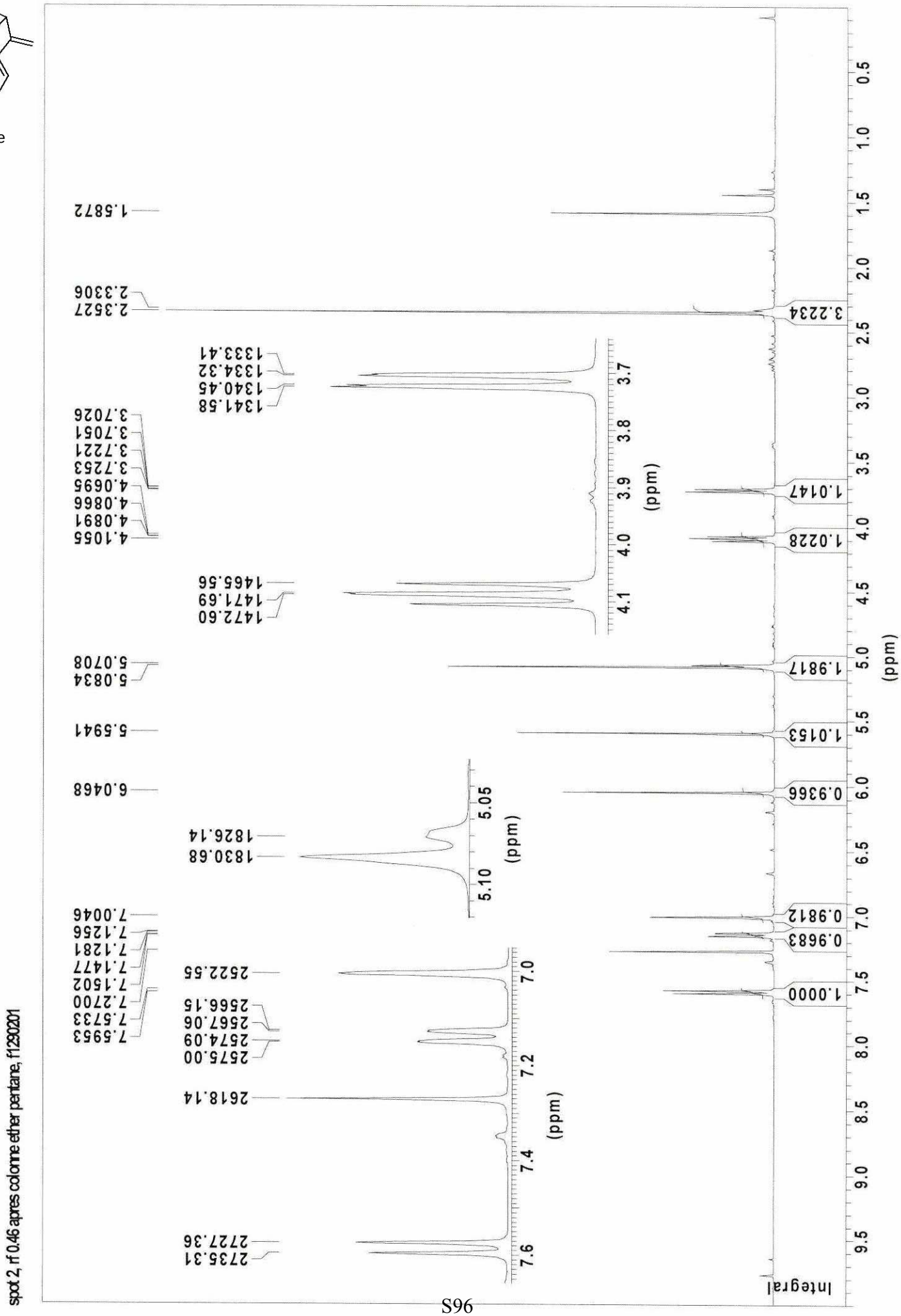


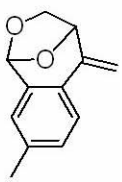
/Data/UNI_FR/BIRB1811_ESI/1/pdata/1 FTMS USER Wed Sep 20 16:12:39 2006


c:\first\da...\galaxy\tmp576.ras Thu Sep .4 10:06:11:62 2006
13d
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
3019.38
2920.05
2859.30
2400.26
2252.72
1679.89
1581.53
1442.66
1348.16
1216.04
1156.25
1027.99
909.38
757.97
668.29

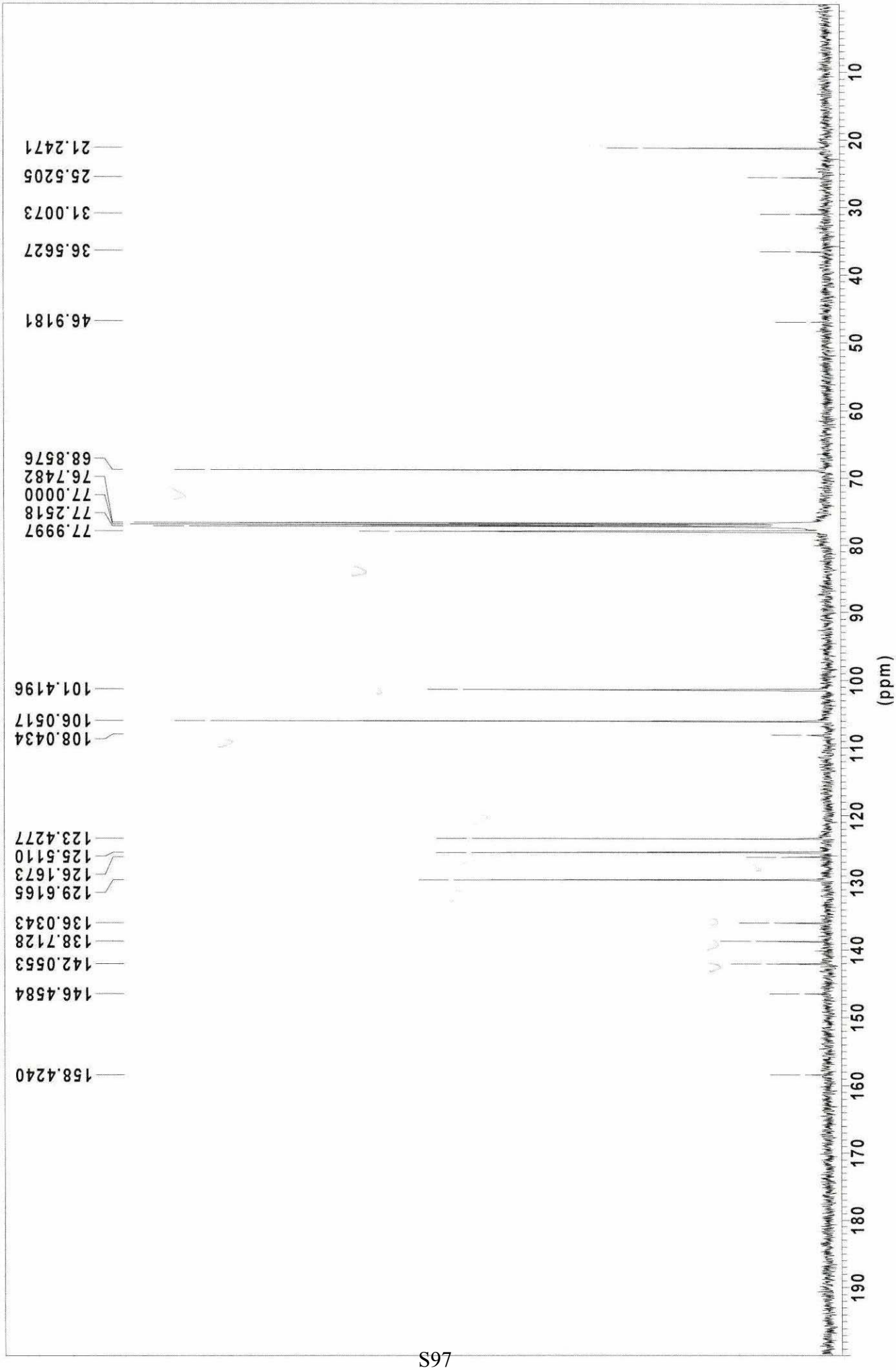


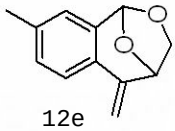
12e


spot 2, rf 0.46 apres colonne ether pentane, f1290201
2735.31
2727.36
2618.14
2575.00
2574.09
2567.06
2566.15
2522.55
7.5953
7.5733
7.2700
7.1502
7.1477
7.1281
7.1256
7.0046
1830.68
1826.14
6.0468
5.5941
5.0834
5.0708
1472.60
1471.69
1465.56
4.1055
4.0891
4.0866
4.0695
3.7253
3.7221
3.7051
3.7026
1341.58
1340.45
1334.32
1333.41
2.3527
2.3306
1.5872
(ppm)
Integral
3.2234
1.0147
1.0228
1.9817
1.0153
0.9366
0.9812
0.9683
1.0000



12e


158.4240
146.4584
142.0553
138.7128
136.0343
129.6165
126.1673
125.5110
123.4277
108.0434
106.0517
101.4196
77.9997
77.2518
77.0000
76.7482
68.8576
46.9181
36.5627
31.0073
25.5205
21.2471
(ppm)
f1290201



12e

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F1290201

XMASS Mass Analysis for /Data/UNI_FR/BIRB1792_ESI/5/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

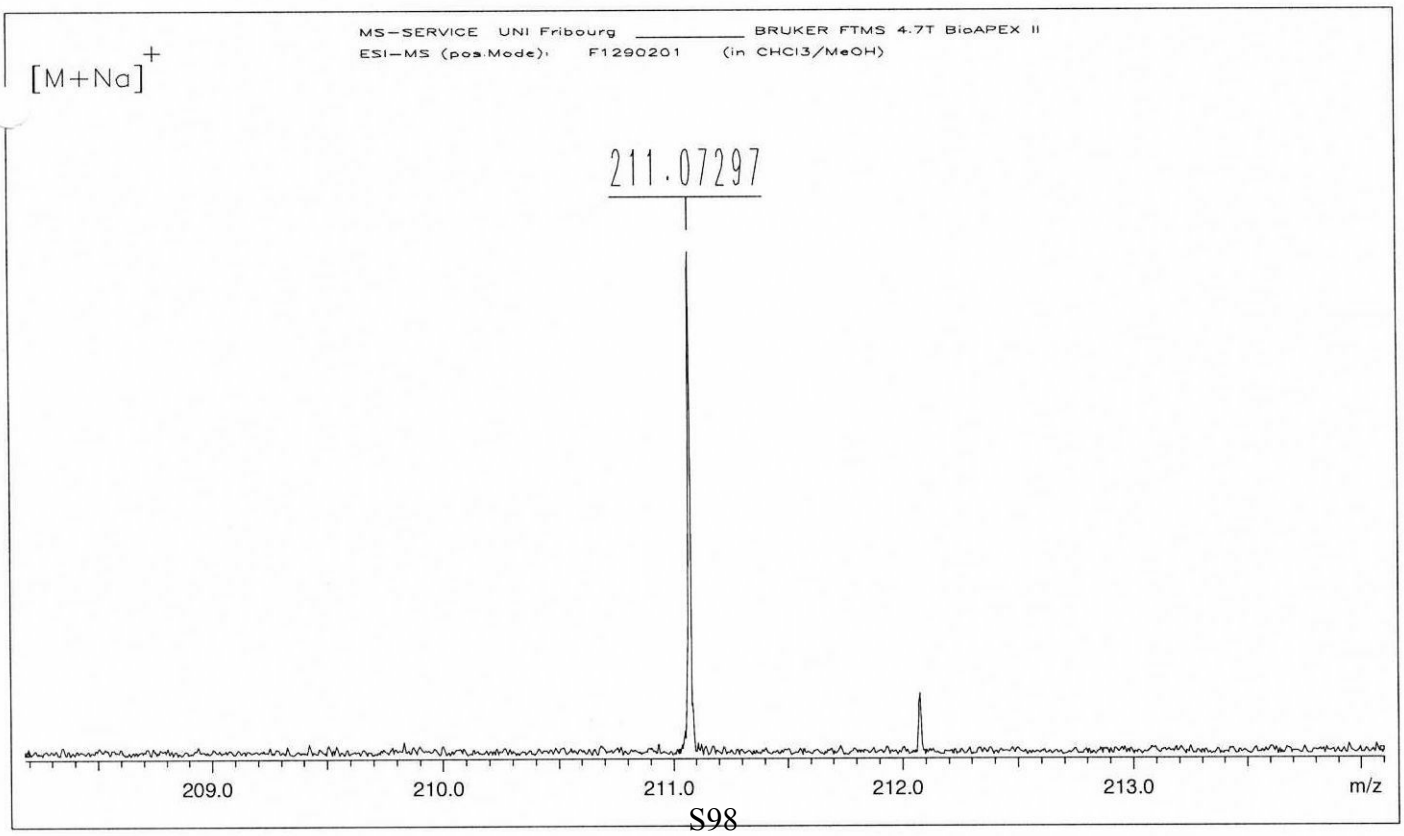
Ion mass = 211.0729670

Charge = +1

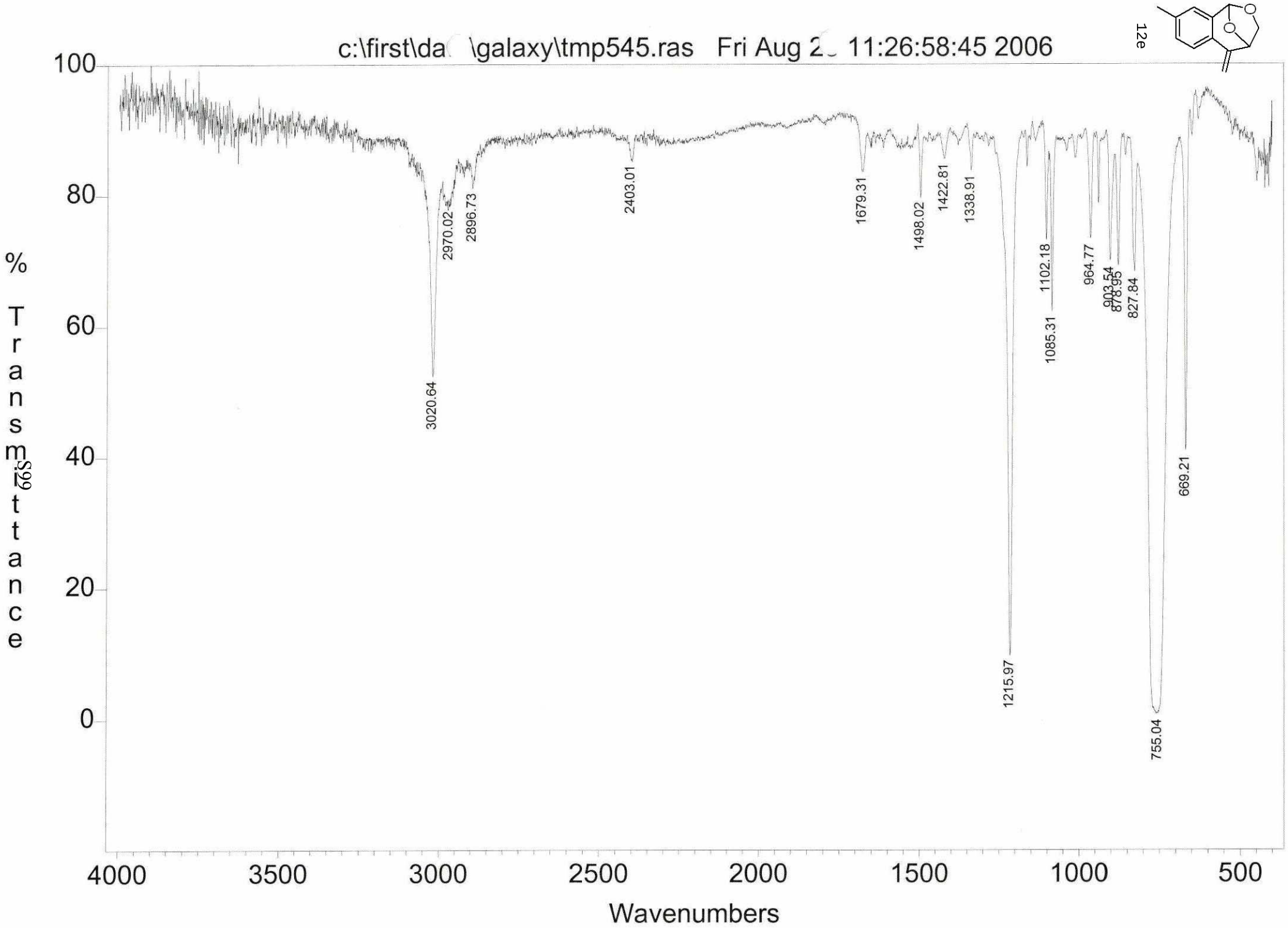
#	C	H	O	Na	mass	DBE	error

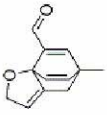
*** Mass Analysis for mass 211.0729670

1	12	12	2	1	211.0729507	6.5	1.628e-05
2	14	11	2	0	211.0753560	9.5	2.389e-03
3	5	16	7	1	211.0788240	-2.5	5.857e-03
4	7	15	7	0	211.0812293	0.5	8.262e-03
5	10	11	5	0	211.0600999	5.5	1.287e-02
6	8	12	5	1	211.0576946	2.5	1.527e-02
7	9	16	4	1	211.0940801	1.5	2.111e-02
8	11	15	4	0	211.0964854	4.5	2.352e-02
9	6	11	8	0	211.0448437	1.5	2.812e-02
10	13	7	3	0	211.0389705	10.5	3.400e-02

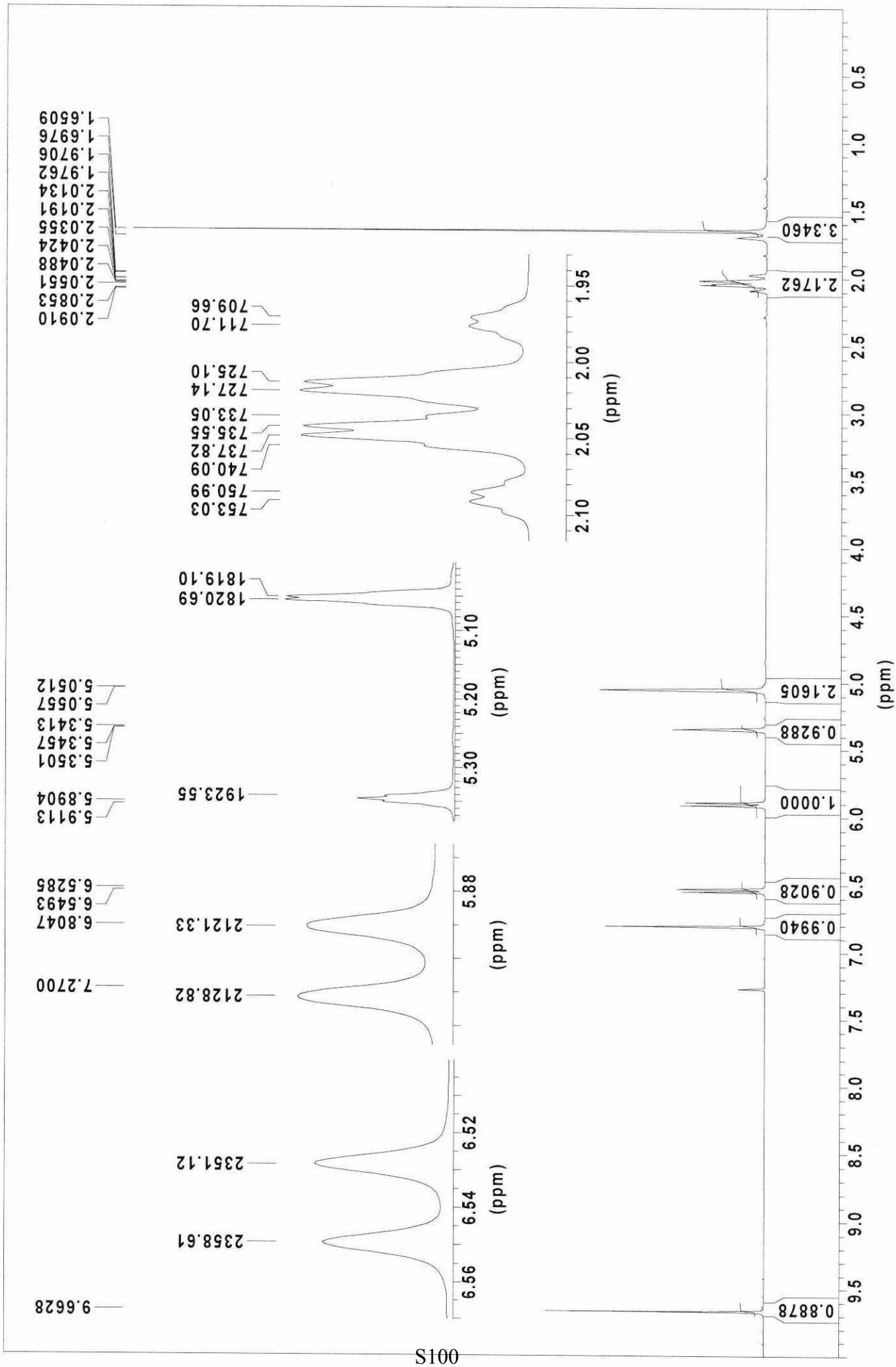


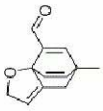
/Data/UNI_FR/BIRB1792_ESI/5/pdata/1 FTMS USER Fri Sep 8 09:55:32 2006


c:\first\da \galaxy\tmp545.ras Fri Aug 2 11:26:58:45 2006
12e
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
3020.64
2970.02
2896.73
2403.01
1679.31
1498.02
1422.81
1338.91
1215.97
1102.18
1085.31
964.77
903.54
878.95
827.84
755.04
669.21

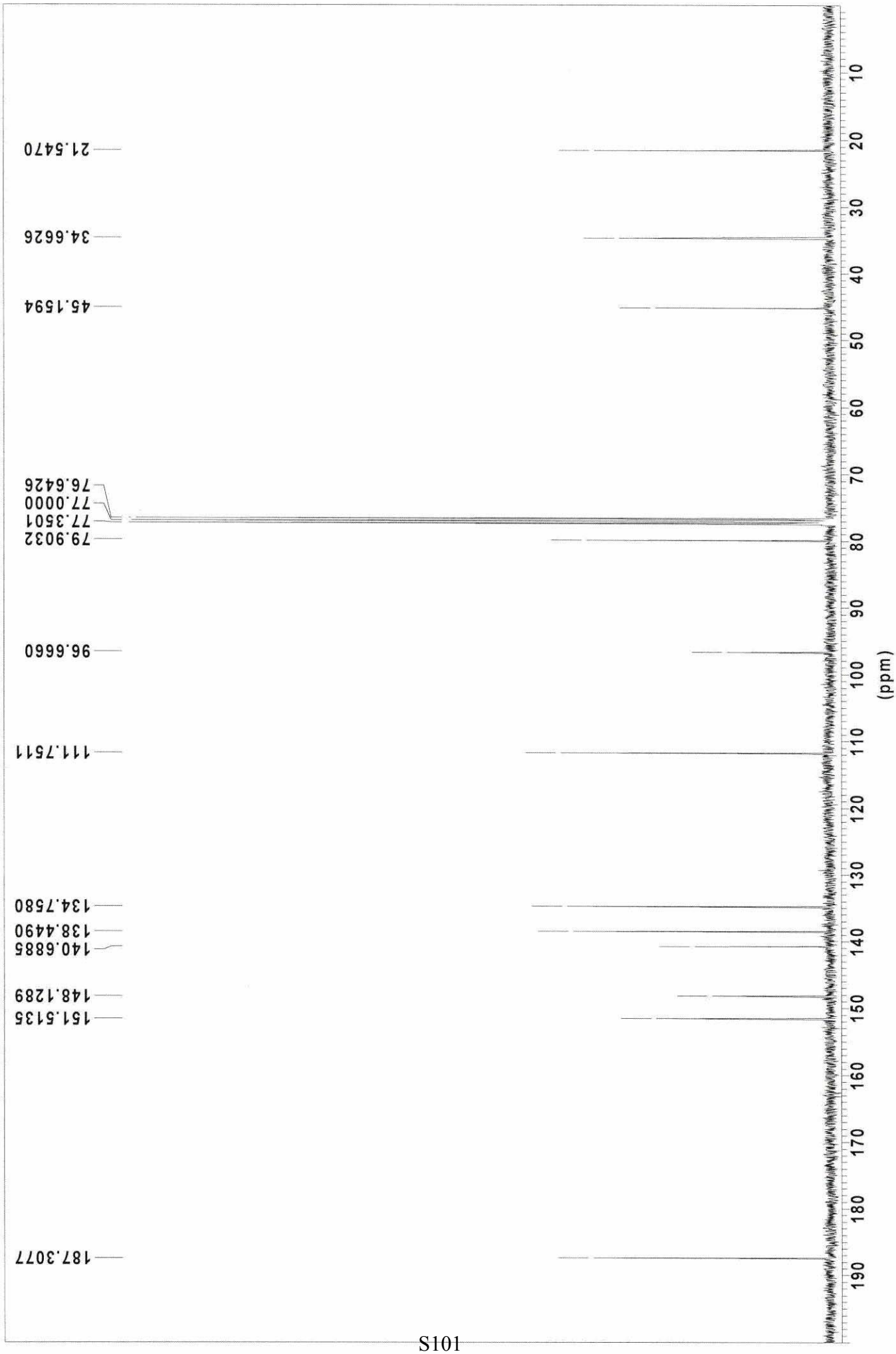


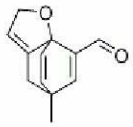
13e


spot 3, rf 0.28 colonne ether pentane, f1290202
9.6628
2358.61
2351.12
7.2700
2128.82
2121.33
6.8047
6.5493
6.5285
5.9113
5.8904
1923.55
5.3501
5.3457
5.3413
5.0557
5.0512
1820.69
1819.10
753.03
750.99
740.09
737.82
735.55
733.05
727.14
725.10
711.70
709.66
2.0910
2.0853
2.0551
2.0488
2.0424
2.0355
2.0191
2.0134
1.9762
1.9706
1.6976
1.6509
(ppm)
0.8878
0.9940
0.9028
1.0000
0.9288
2.1605
3.3460
2.1762



13e


21.5470
34.6626
45.1594
76.6426
77.0000
77.3501
79.9032
96.6660
111.7511
134.7580
138.4490
140.6885
148.1289
151.5135
187.3077
(ppm)
spot 3, f1290202



13e

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

ESI-MS: F1290202

XMASS Mass Analysis for /Data/UNI_FR/BIRB1794_ESI/3/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

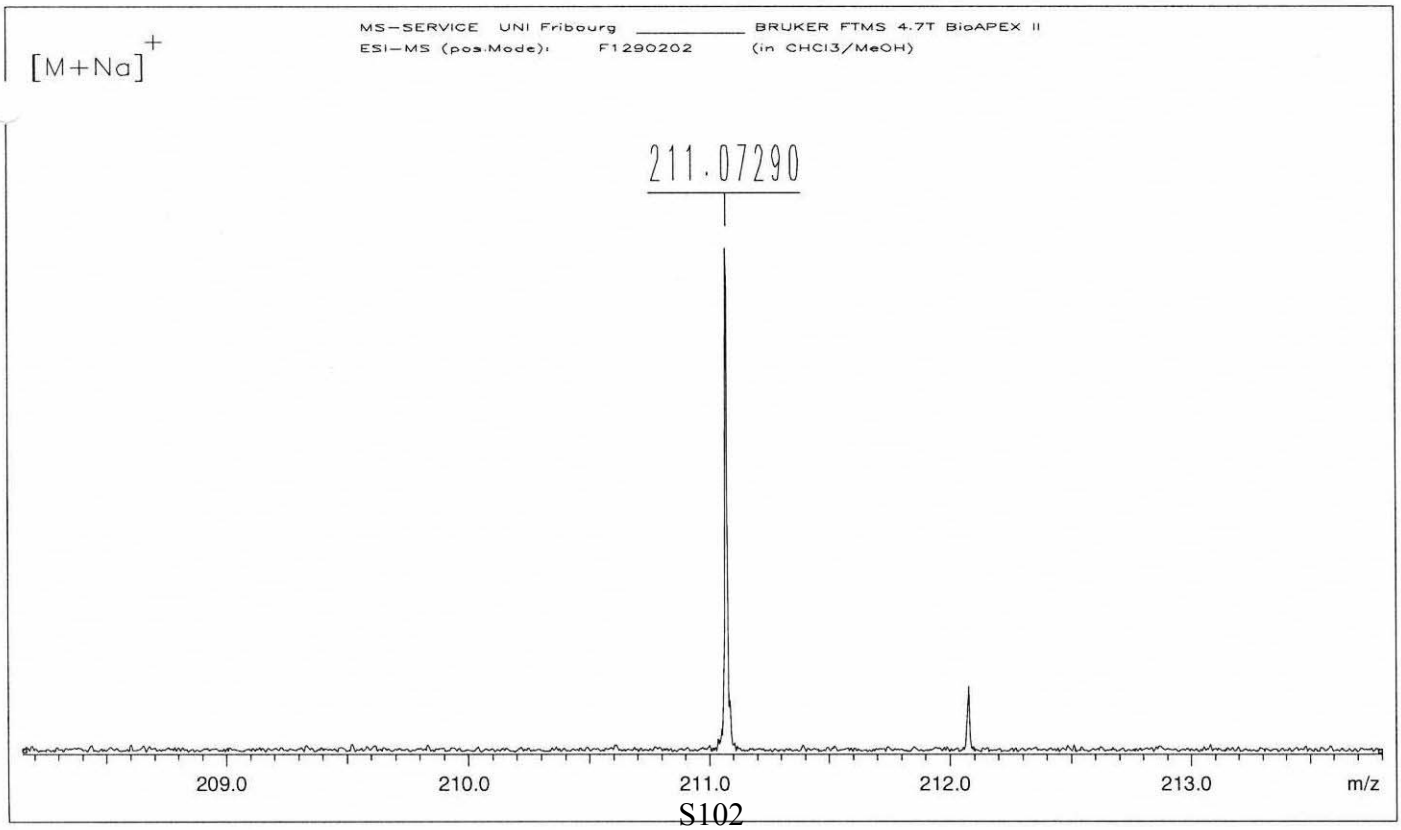
Ion mass = 211.0729030

Charge = +1

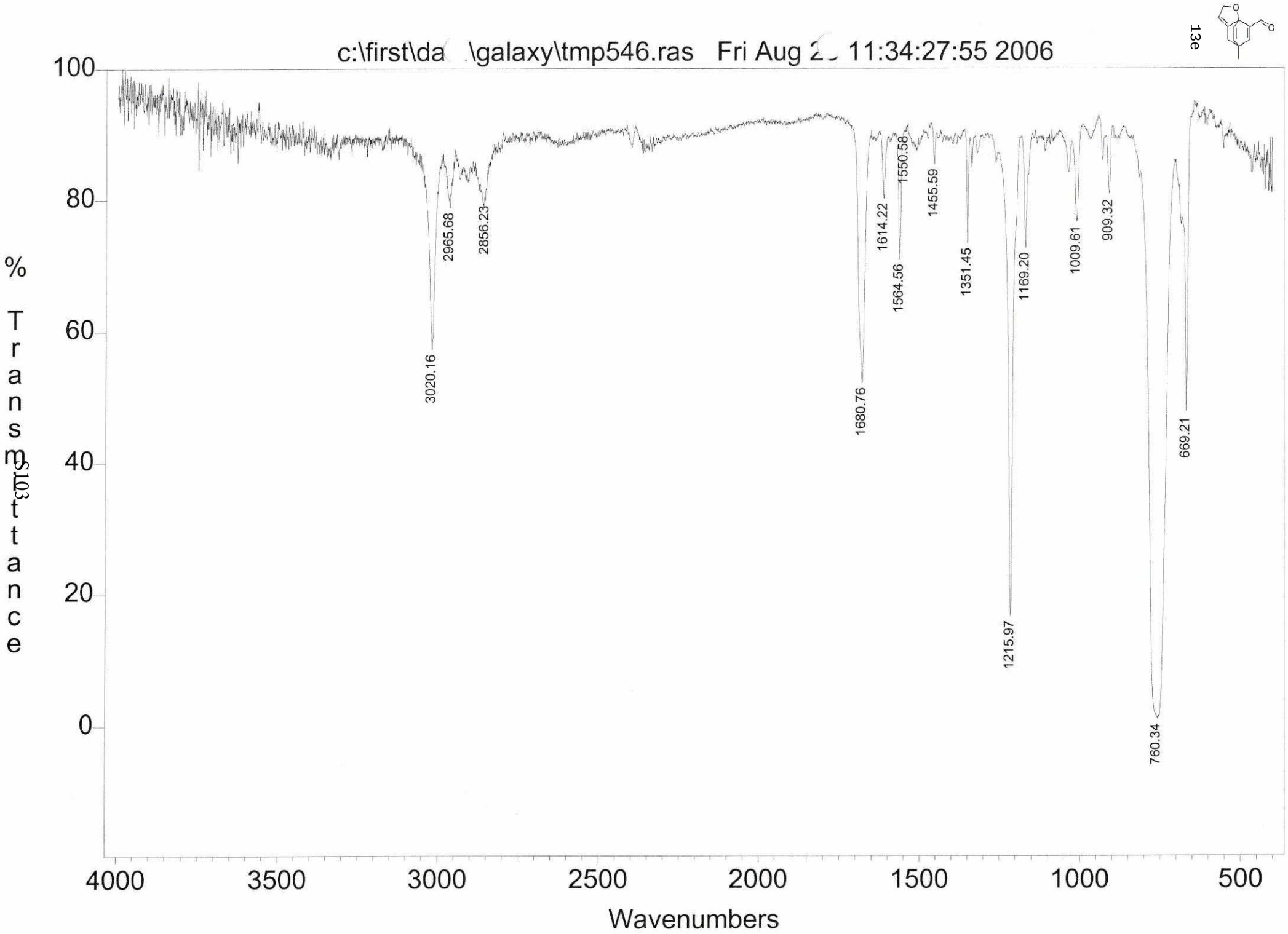
#	C	H	O	Na	mass	DBE	error

*** Mass Analysis for mass 211.0729030

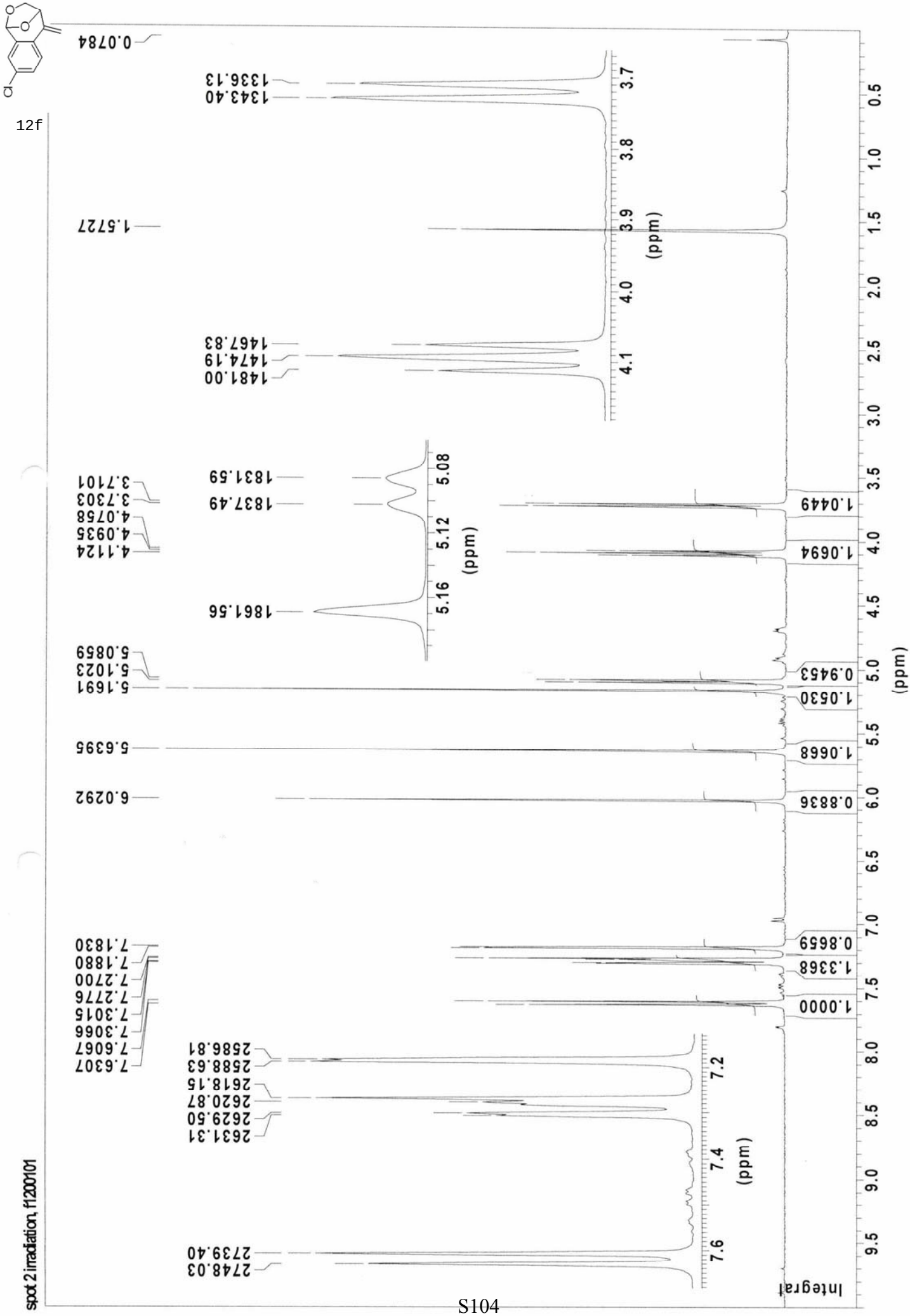
1	12	12	2	1	211.0729507	6.5	4.772e-05
2	14	11	2	0	211.0753560	9.5	2.453e-03
3	5	16	7	1	211.0788240	-2.5	5.921e-03
4	7	15	7	0	211.0812293	0.5	8.326e-03
5	10	11	5	0	211.0600999	5.5	1.280e-02
6	8	12	5	1	211.0576946	2.5	1.521e-02
7	9	16	4	1	211.0940801	1.5	2.118e-02
8	11	15	4	0	211.0964854	4.5	2.358e-02
9	6	11	8	0	211.0448437	1.5	2.806e-02
10	13	7	3	0	211.0389705	10.5	3.393e-02

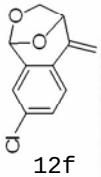


/Data/UNI_FR/BIRB1794_ESI/3/pdata/1 FTMS USER Fri Sep 8 10:01:59 2006

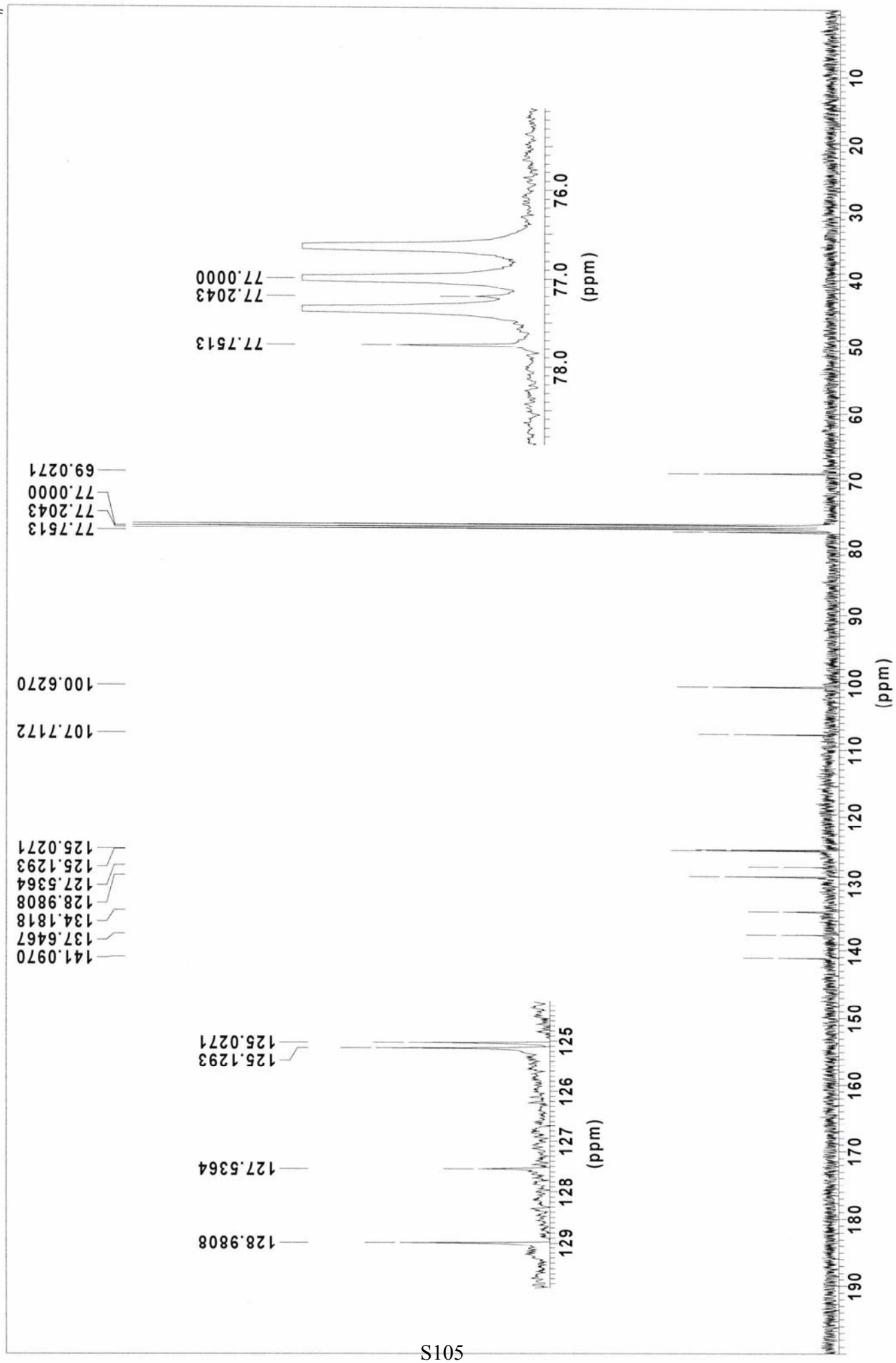

c:\first\da \galaxy\tmp546.ras Fri Aug 2 11:34:27:55 2006
13e
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3500
3000
2500
2000
1500
1000
500
3020.16
2965.68
2856.23
1680.76
1614.22
1564.56
1550.58
1455.59
1351.45
1215.97
1169.20
1009.61
909.32
760.34
669.21

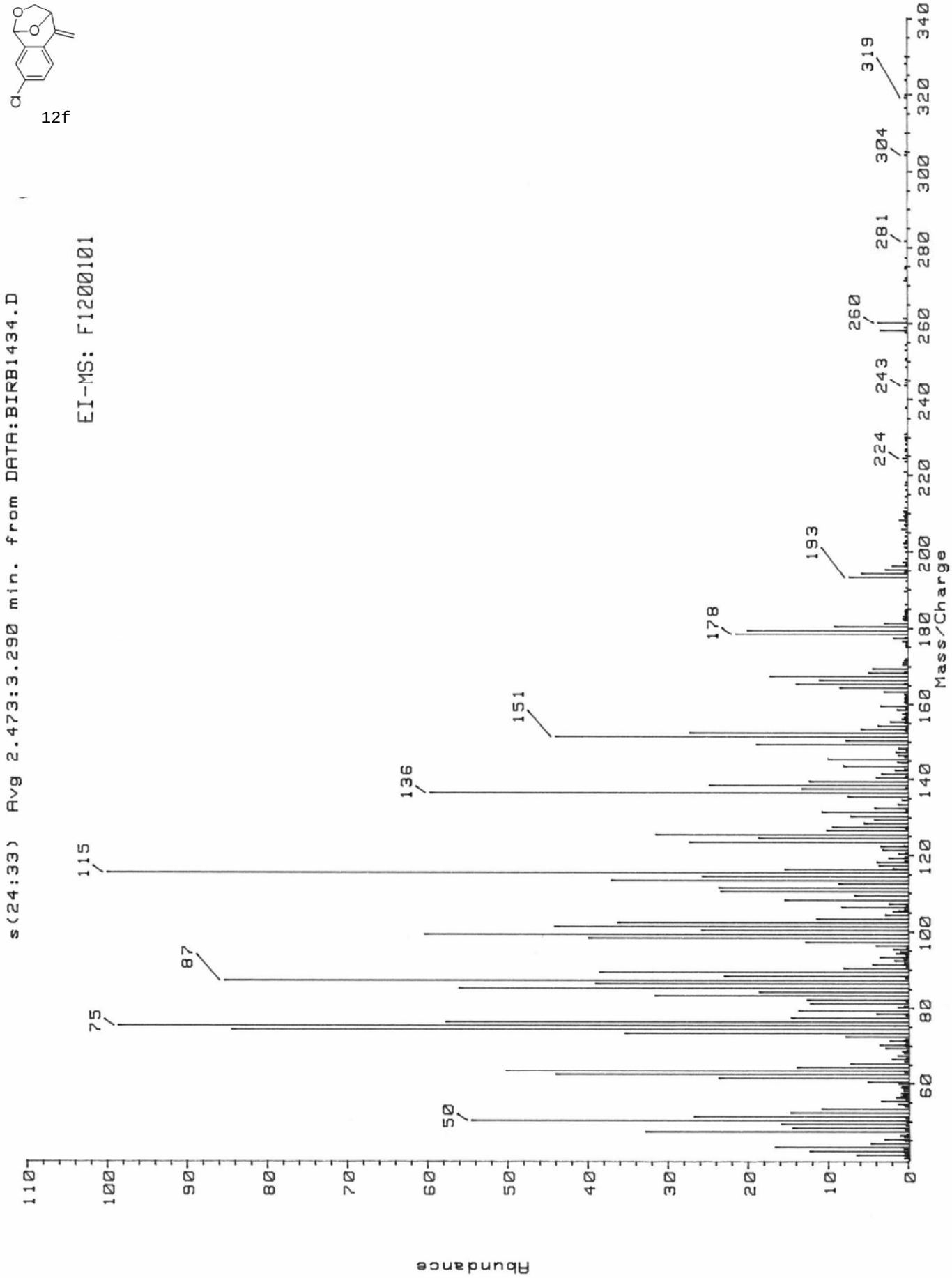
S103

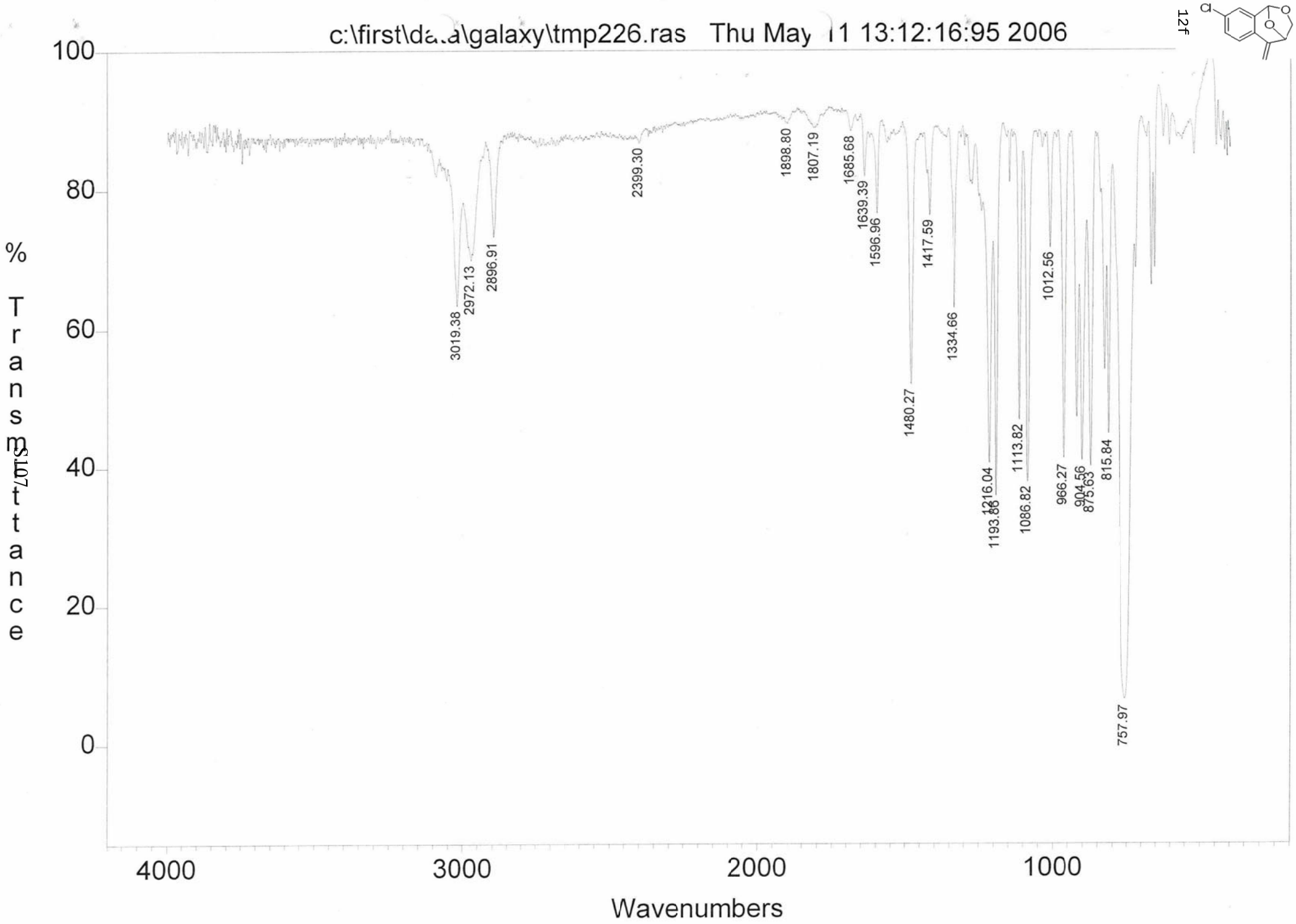

12f
spot 2 irradiation, f12001101
Integral
(ppm)
0.0784
1.5727
1343.40
1336.13
1481.00
1474.19
1467.83
1831.59
1837.49
1861.56
3.7101
3.7303
4.0758
4.0935
4.1124
5.0859
5.1023
5.1691
5.6395
6.0292
7.1830
7.1880
7.2700
7.2776
7.3015
7.3066
7.6067
7.6307
2586.81
2588.63
2618.15
2620.87
2629.50
2631.31
2739.40
2748.03
1.0000
1.3368
0.8659
0.8836
1.0668
1.0530
0.9453
1.0694
1.0449

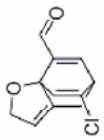


12f

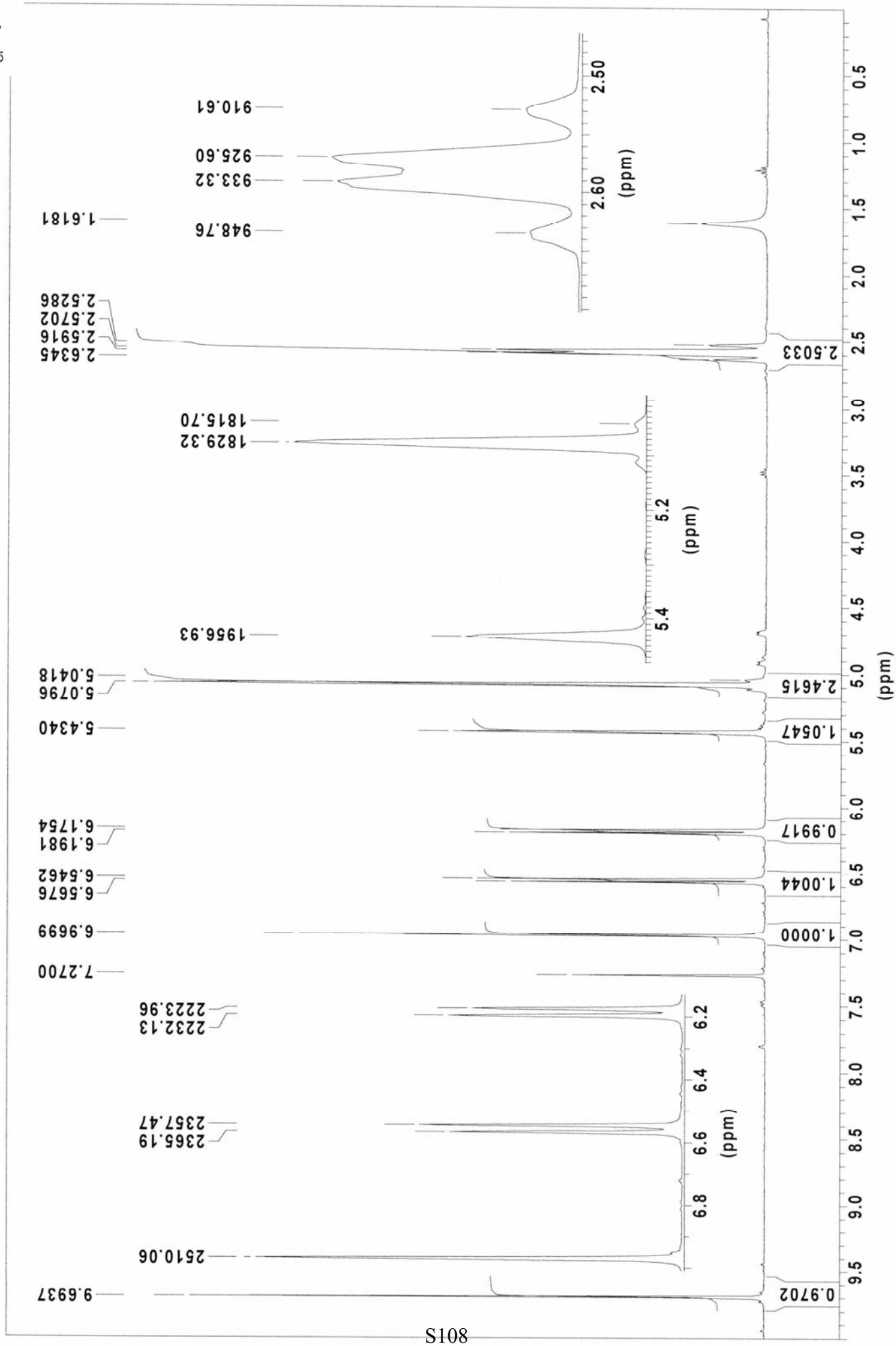

141.0970
137.6467
134.1818
128.9808
127.5364
125.1293
125.0271
125.0271
125.1293
127.5364
128.9808
107.7172
100.6270
77.7513
77.2043
77.0000
69.0271
77.7513
77.2043
77.0000
(ppm)
(ppm)
idem 01, f1200102


12f
s(24:33) Avg 2.473:3.290 min. from DATA:BIRB1434.D
EI-MS: F1200101
Mass/Charge
Abundance
50
75
87
115
136
151
178
193
224
243
260
281
304
319

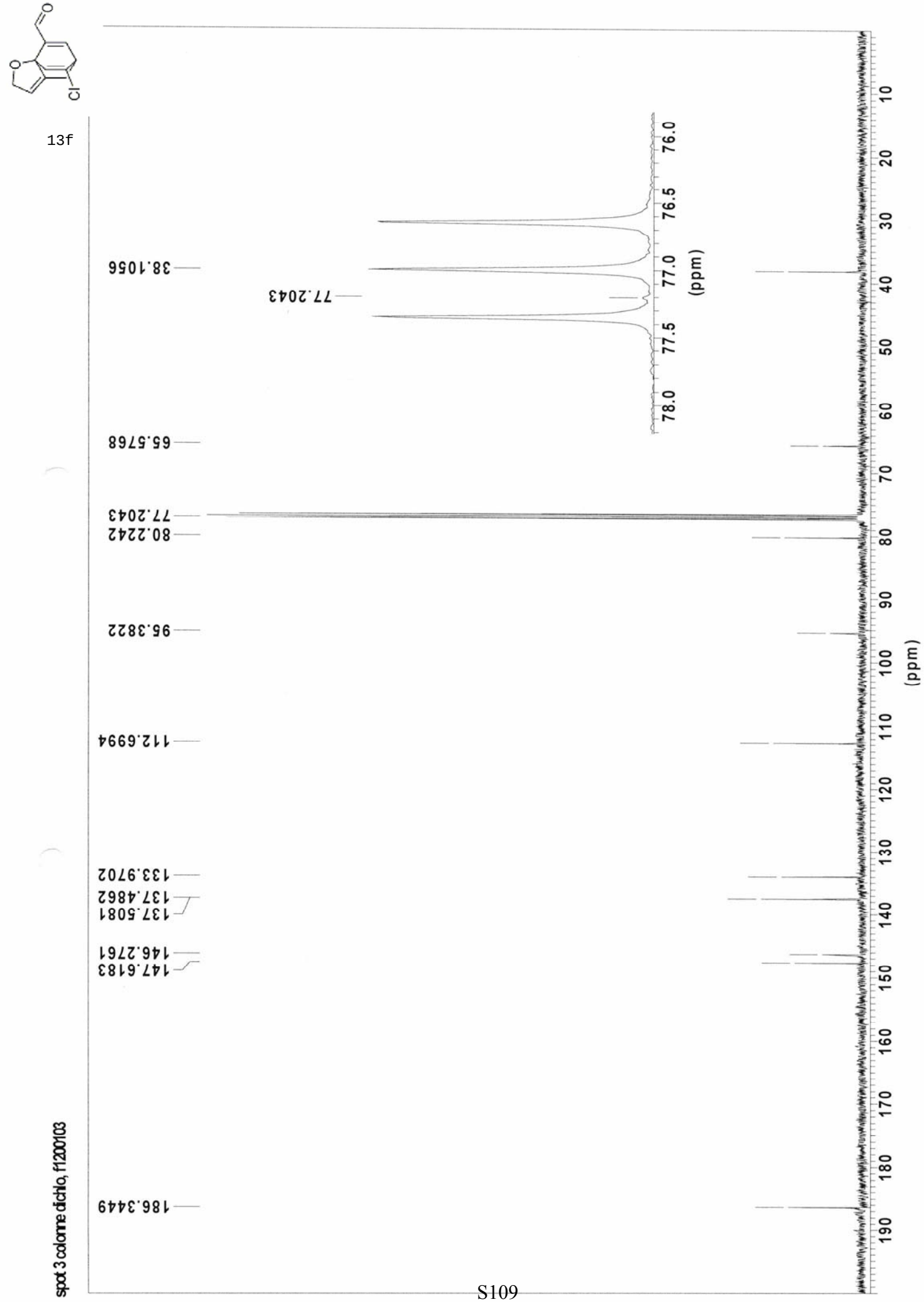

c:\first\data\galaxy\tmp226.ras Thu May 11 13:12:16:95 2006
12f
% Transmittance
Wavenumbers
100
80
60
40
20
0
4000
3000
2000
1000
3019.38
2972.13
2896.91
2399.30
1898.80
1807.19
1685.68
1639.39
1596.96
1480.27
1417.59
1334.66
1216.04
1193.88
1113.82
1086.82
1012.56
966.27
904.56
875.63
815.84
757.97

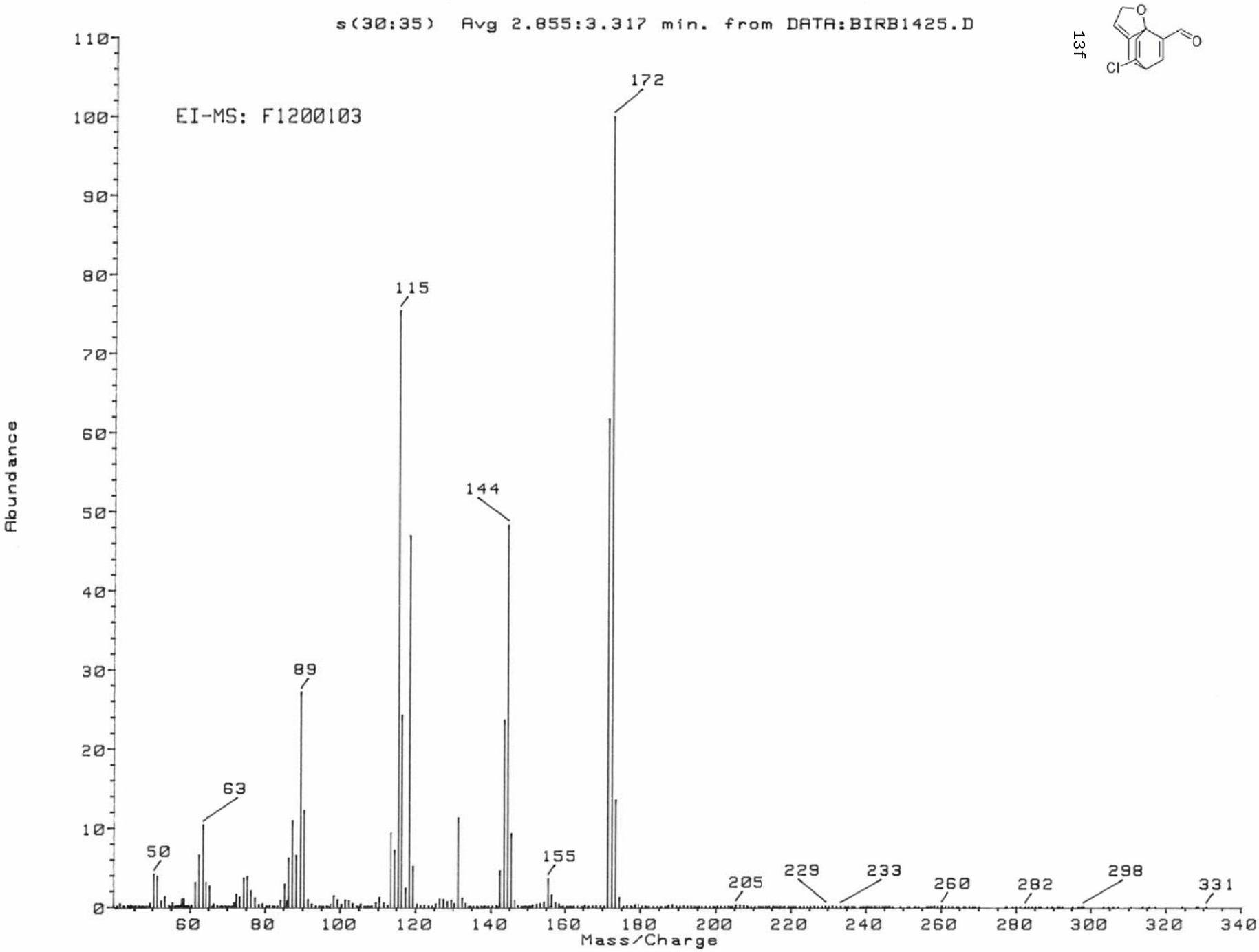


13f

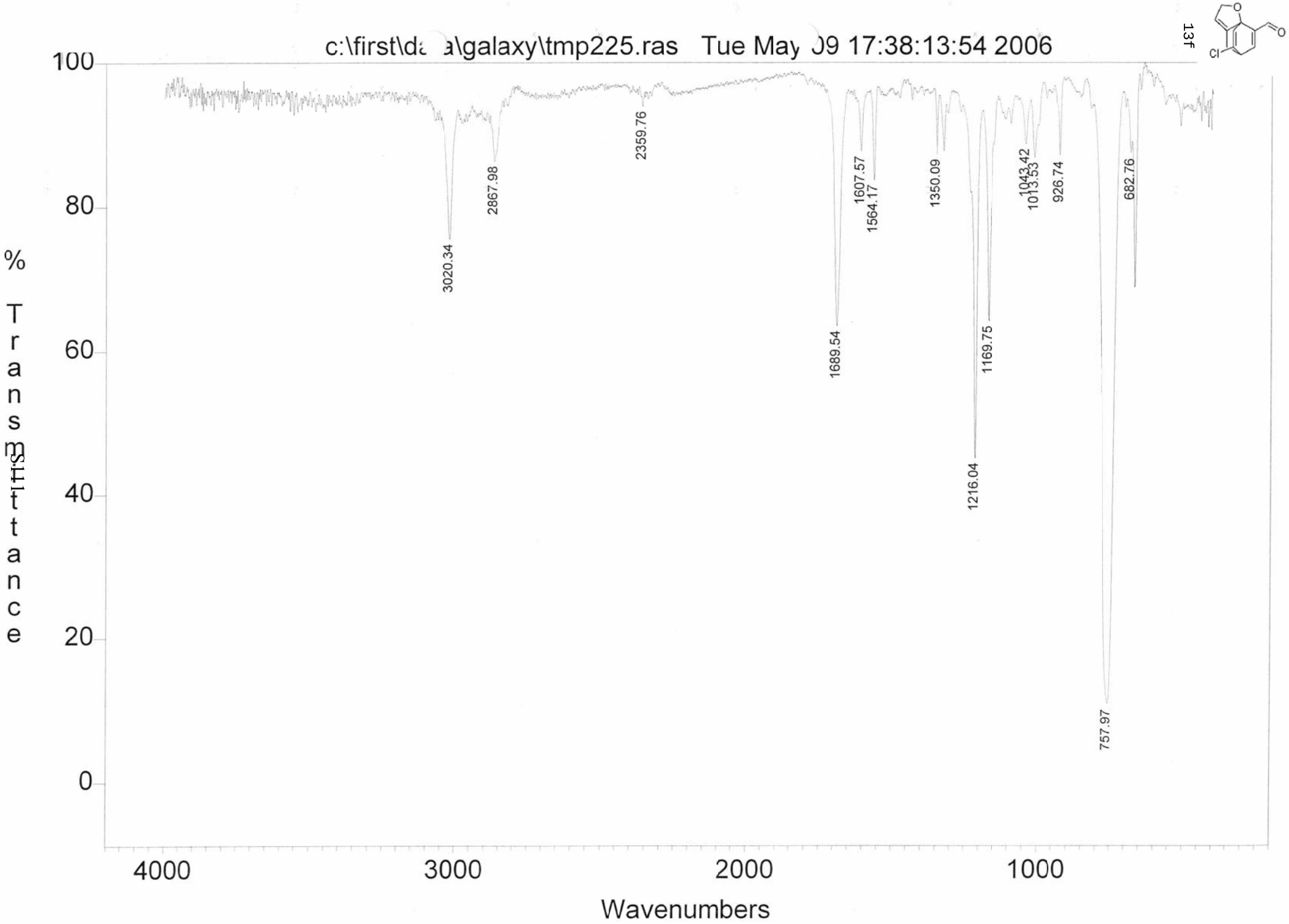


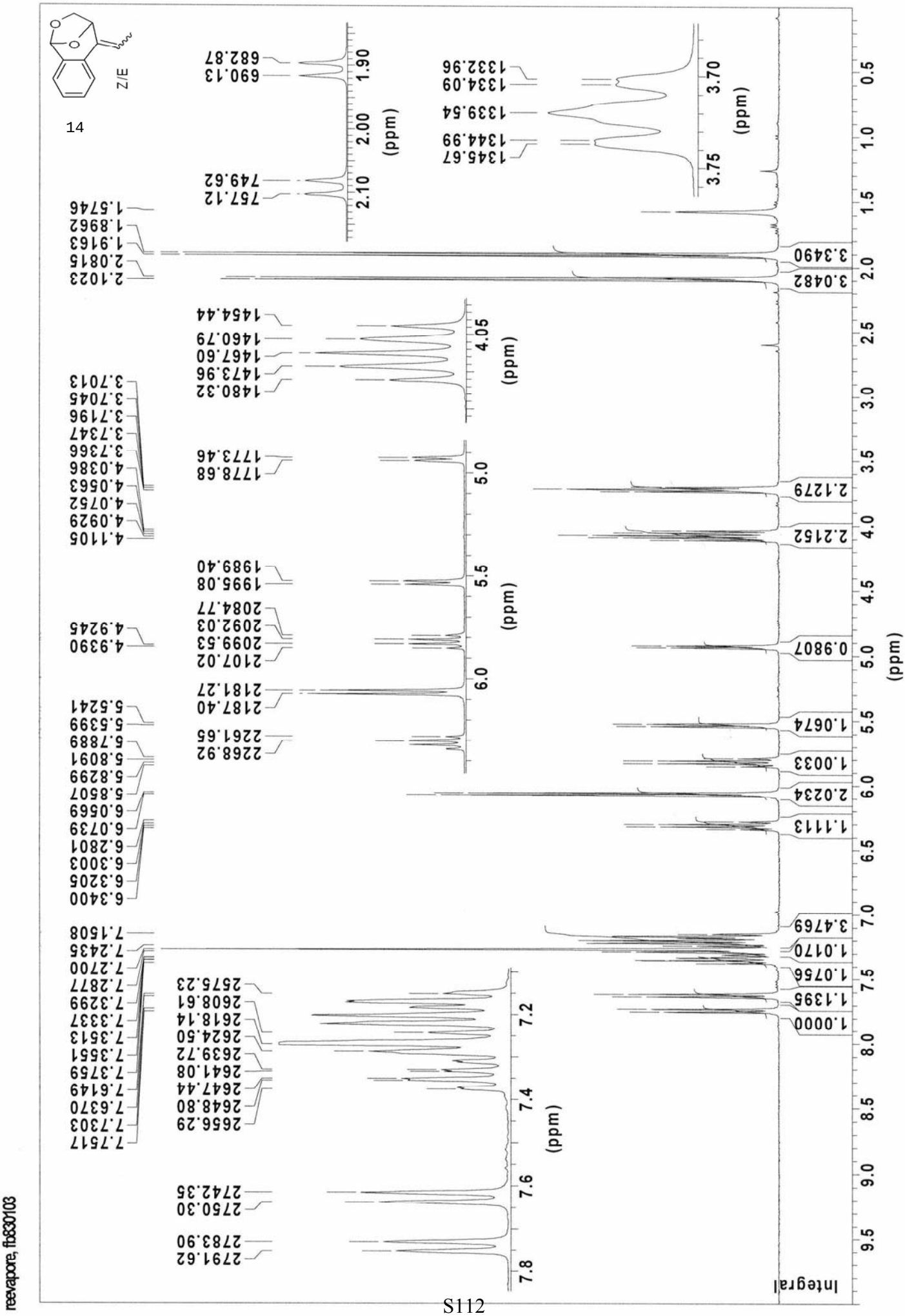
spot 3 colonne dichlo, f12000103

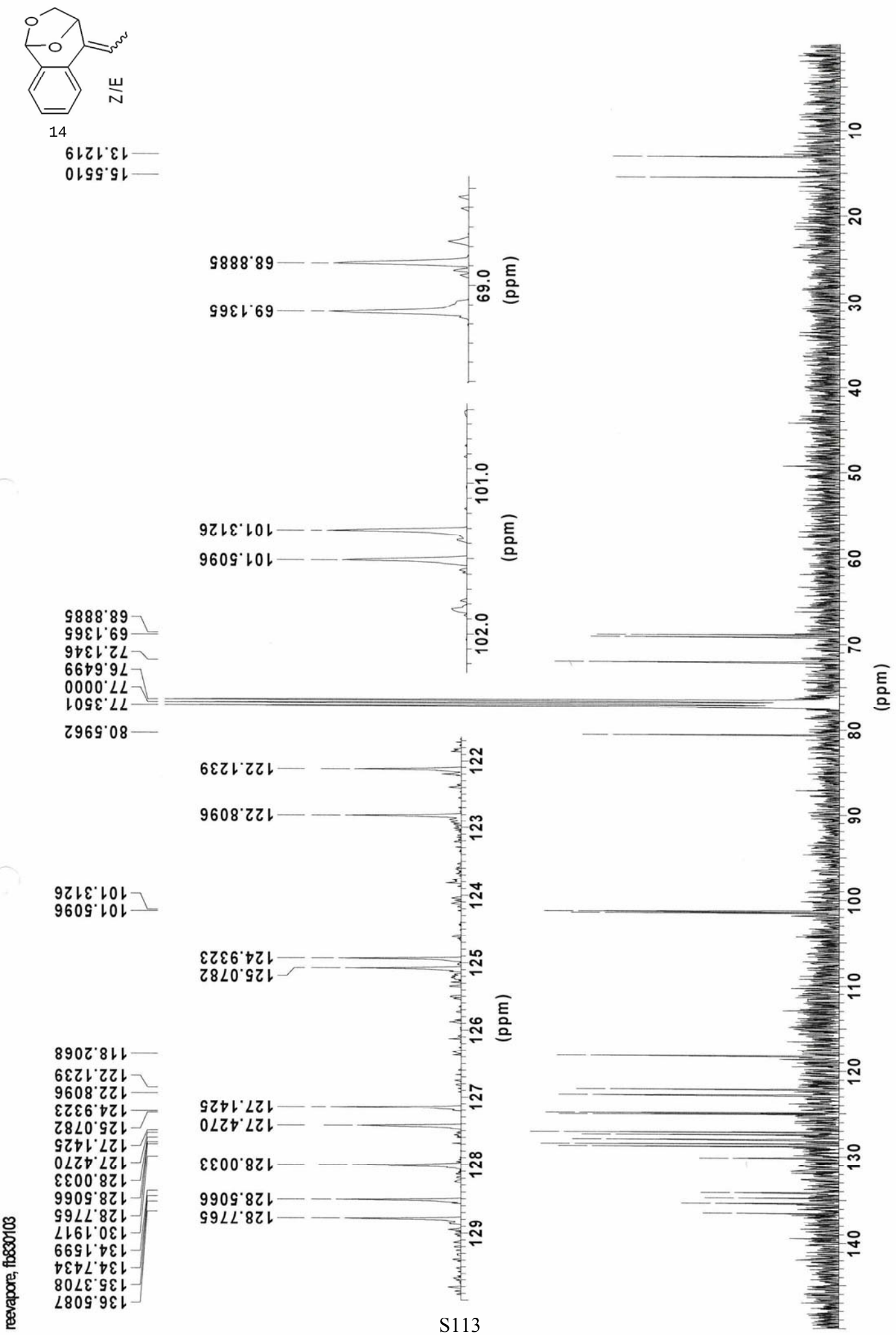

13f
spot 3 colonne dichlo, f1200103
186.3449
147.6183
146.2761
137.5081
137.4862
133.9702
112.6994
95.3822
80.2242
77.2043
65.5768
38.1056
77.2043
(ppm)
(ppm)

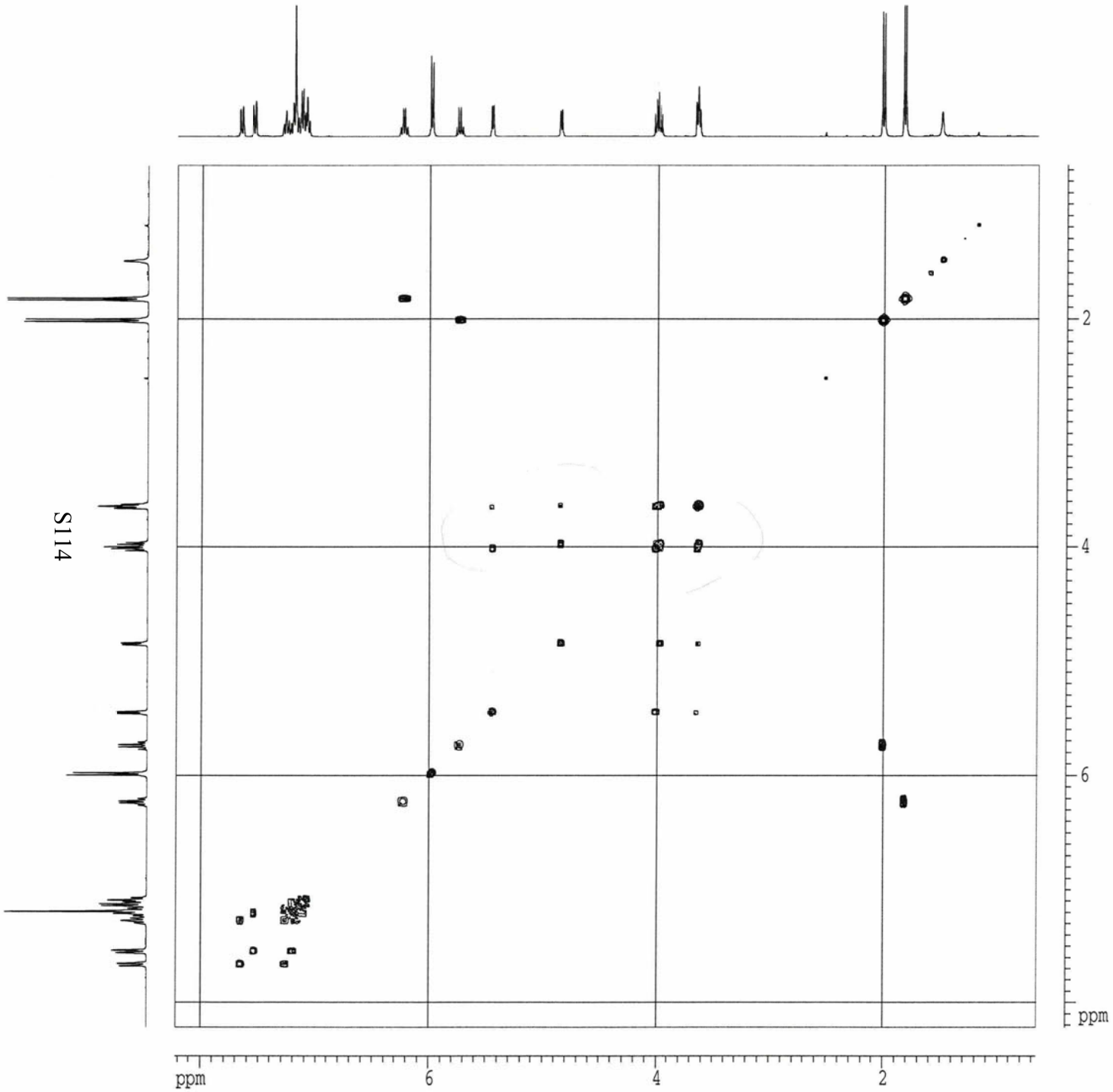

s(30:35) Avg 2.855:3.317 min. from DATA:BIRB1425.D
EI-MS: F1200103
Abundance
Mass/Charge
172
115
144
89
63
50
155
205
229
233
260
282
298
331

13f


c:\first\da a\galaxy\tmp225.ras Tue May 09 17:38:13:54 2006
13f
% Transmittance
100
80
60
40
20
0
3020.34
2867.98
2359.76
1689.54
1607.57
1564.17
1350.09
1216.04
1169.75
1043.42
1013.53
926.74
757.97
682.76
4000
3000
2000
1000
Wavenumbers

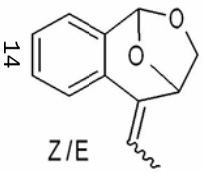



Z/E
14
13.1219
15.5510
68.8885
69.1365
101.3126
101.5096
68.8885
69.1365
72.1346
76.6499
77.0000
77.3501
80.5962
122.1239
122.8096
101.5096
101.3126
124.9323
125.0782
118.2068
122.1239
122.8096
124.9323
125.0782
127.1425
127.4270
128.0033
127.4270
127.1425
128.5066
128.7765
130.1917
134.1599
134.7434
135.3708
136.5087
128.7765
128.5066
128.0033
127.4270
127.1425
125.0782
124.9323
122.8096
122.1239
80.5962
101.5096
101.3126
69.1365
68.8885
15.5510
13.1219
(ppm)
reevapone, fb830103



reevapore, fb830103
COSYGPSW CDCl3 u frbi 5(

14



Z/E

Current Data Parameters

NAME	fb830103
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20050907
Time	16.17
INSTRUM	dpx360
PROBHD	5 mm SEI 1H-13
PULPROG	cosygpqf
TD	2048
SOLVENT	CDCl3
NS	1
DS	8
SWH	2723.312 Hz
FIDRES	1.329742 Hz
AQ	0.3760628 sec
RG	1448.2
DW	183.600 usec
DE	6.00 usec
TE	300.0 K
d0	0.00000300 sec
D1	1.32468998 sec
d13	0.00000400 sec
D16	0.00030000 sec
IN0	0.00036720 sec
MCREST	0.00000000 sec
MCWRK	1.32468998 sec

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

NUC1	1H
P0	6.80 usec
P1	6.80 usec
PL1	-4.00 dB
SFO1	360.1316378 MHz

====== GRADIENT CHANNEL =====

GPNAM1	SINE.100
GPNAM2	SINE.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	10.00 %
GPZ2	10.00 %
P16	1500.00 usec

F1 - Acquisition parameters

ND0	1
TD	128
SFO1	360.1316 MHz
FIDRES	21.275871 Hz
SW	7.562 ppm

F2 - Processing parameters

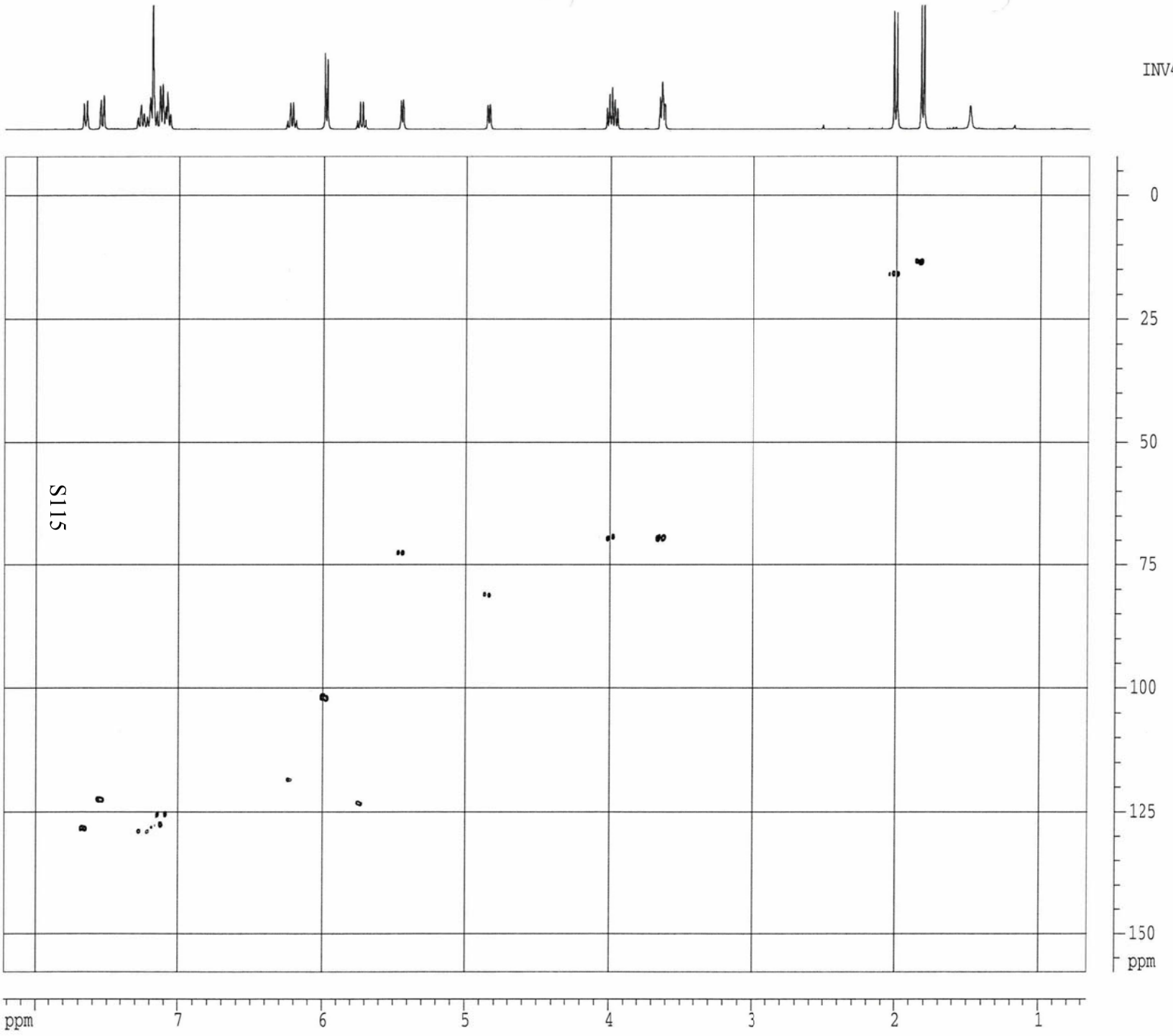
SI	1024
SF	360.1300391 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

F1 - Processing parameters

SI	1024
MC2	QF
SF	360.1300391 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

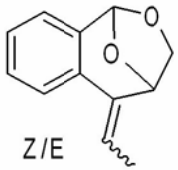
2D NMR plot parameters

CX2	15.00 cm
CX1	15.00 cm
F2PLO	8.220 ppm
F2LO	2960.40 Hz
F2PHI	0.658 ppm
F2HI	237.09 Hz
F1PLO	8.220 ppm
F1LO	2960.40 Hz
F1PHI	0.658 ppm
F1HI	237.09 Hz
F2PPMCM	0.50413 ppm/cm
F2HZCM	181.55409 Hz/cm
F1PPMCM	0.50413 ppm/cm
F1HZCM	181.55409 Hz/cm



reevapore, fb830103
INV4GPSW CDCl3 u frbi 5(

14



Z/E

Current Data Parameters

NAME	fb830103
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20050908
Time	6.13
INSTRUM	dpx360
PROBHD	5 mm SEI 1H-13
PULPROG	inv4gpqf
TD	1024
SOLVENT	CDCl3
NS	4
DS	16
SWH	2723.312 Hz
FIDRES	2.659484 Hz
AQ	0.1880564 sec
RG	26008
DW	183.600 usec
DE	6.00 usec
TE	300.0 K
CNST2	145.0000000
d0	0.00000300 sec
D1	1.45351005 sec
d2	0.00344828 sec
d12	0.00002000 sec
d13	0.00000400 sec
D16	0.00030000 sec
d20	0.00192428 sec
IN0	0.00003330 sec
MCREST	0.00000000 sec
MCWRK	1.45351005 sec

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

NUC1	1H
P1	6.80 usec
p2	13.60 usec
PL1	-4.00 dB
SFO1	360.1316378 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	13C
P3	13.60 usec
PCPD2	63.00 usec
PL2	-6.00 dB
PL12	7.00 dB
SFO2	90.5615161 MHz

====== GRADIENT CHANNEL =====

GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPX1	0.00 %
GPX2	0.00 %
GPX3	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPY3	0.00 %
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1500.00 usec

F1 - Acquisition parameters

ND0	2
TD	128
SFO1	90.56152 MHz
FIDRES	117.304802 Hz
SW	165.799 ppm

F2 - Processing parameters

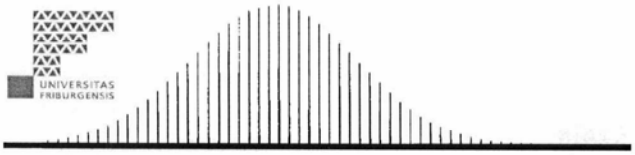
SI	1024
SF	360.1300391 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

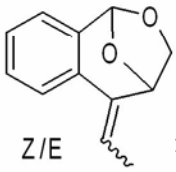
F1 - Processing parameters

SI	1024
MC2	QF
SF	90.5547250 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

2D NMR plot parameters

CX2	20.00 cm
CX1	15.00 cm
F2PLO	8.220 ppm
F2LO	2960.40 Hz
F2PHI	0.658 ppm





14

FTMS 4.7T BioAPEX II MS-Service UNI-Fribourg

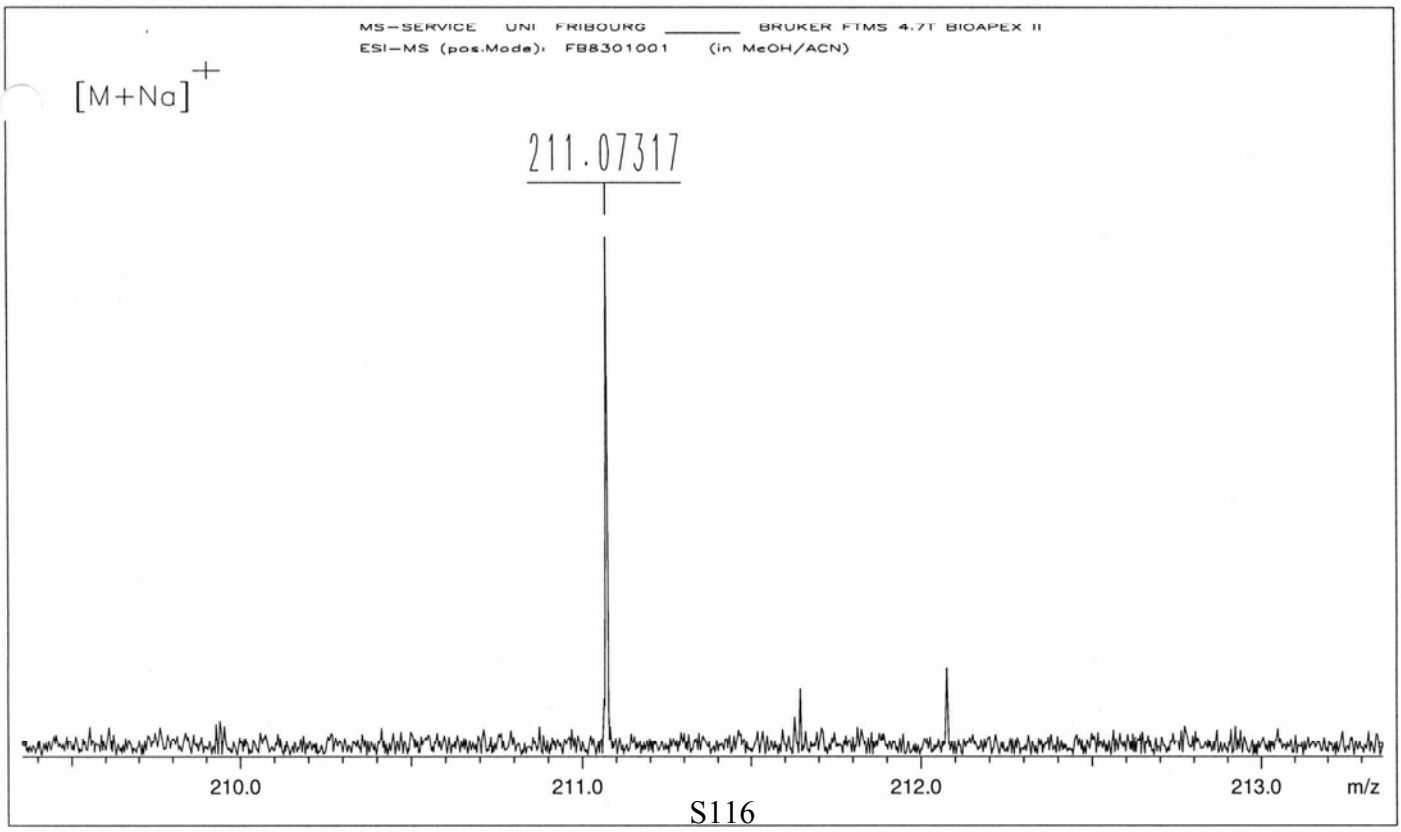
ESI-MS: FB8301001

XMASS Mass Analysis for /Data/UNI_FR/BIRB1045_ESI/1/pdata/1/massanal.res:
XMASS Mass Analysis Constraints

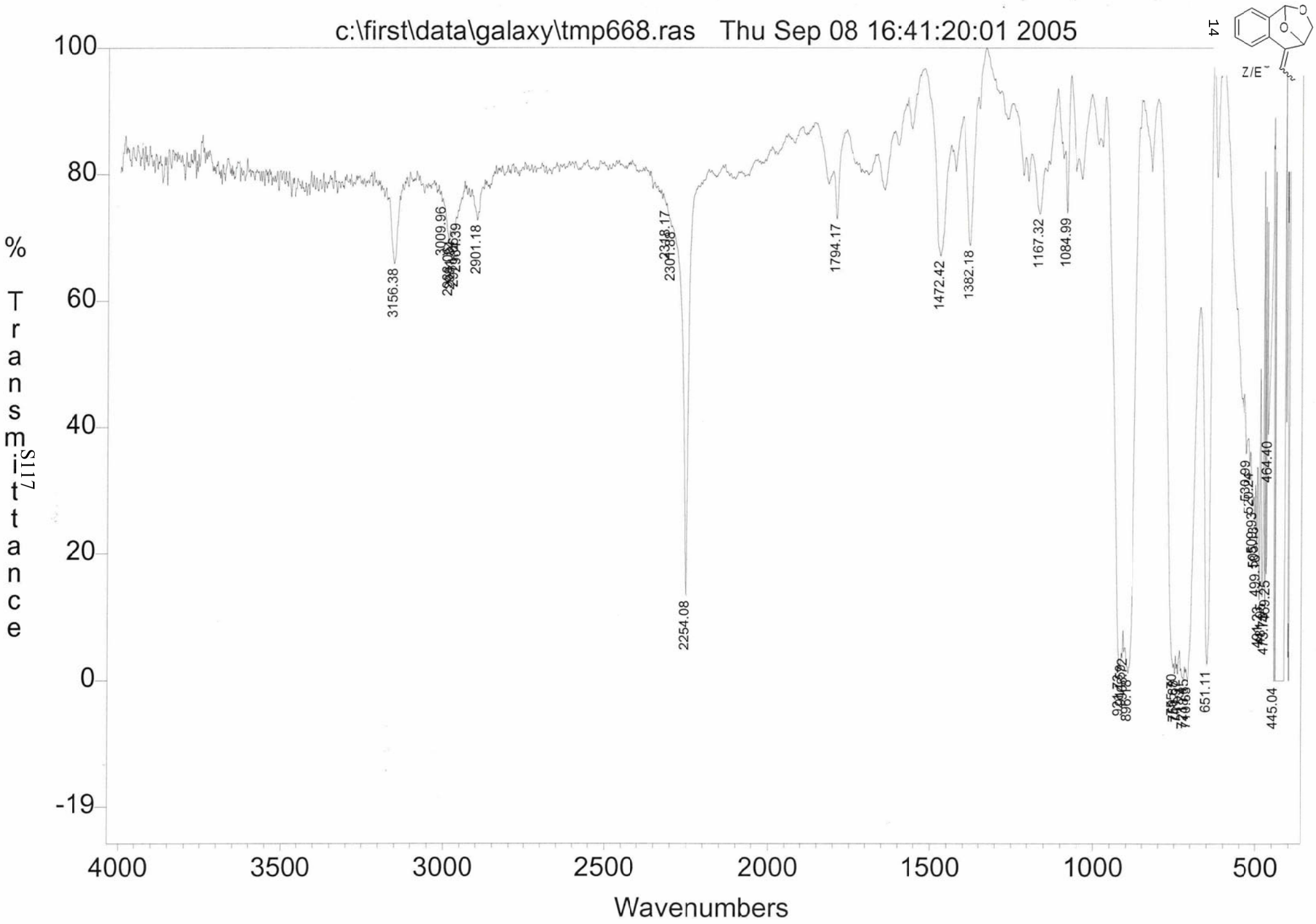
Ion mass = 211.0731750

Charge = +1

#	C	H	O	Na	mass	DBE	error
*** Mass Analysis for mass 211.0731750							
1	12	12	2	1	211.0729507	6.5	2.243e-04
2	14	11	2	0	211.0753560	9.5	2.181e-03
3	5	16	7	1	211.0788240	-2.5	5.649e-03
4	7	15	7	0	211.0812293	0.5	8.054e-03
5	10	11	5	0	211.0600999	5.5	1.308e-02
6	8	12	5	1	211.0576946	2.5	1.548e-02
7	9	16	4	1	211.0940801	1.5	2.091e-02
8	11	15	4	0	211.0964854	4.5	2.331e-02
9	6	11	8	0	211.0448437	1.5	2.833e-02
10	13	7	3	0	211.0389705	10.5	3.420e-02



/Data/UNI_FR/BIRB1045_ESI/1/pdata/1 FTMS USER Wed Jan 18 16:20:27 2006


c:\first\data\galaxy\tmp668.ras Thu Sep 08 16:41:20:01 2005
14
Z/E
% Transmittance
Wavenumbers
100
80
60
40
20
0
-19
4000
3500
3000
2500
2000
1500
1000
500
3156.38
3009.96
2901.18
2318.17
2301.88
2254.08
1794.17
1472.42
1382.18
1167.32
1084.99
651.11
464.40
445.04

Crystallography for compound 12d

A colourless crystal of compound **12d** was mounted on a Stoe Mark II-Imaging Plate Diffractometer System[1] equipped with a graphite-monochromator. Data collection was performed at –100˚C using Mo-Kα radiation (λ = 0.71073 Å). 45 exposures (5 min per exposure) were obtained at an image plate distance of 100 mm with φ= 0˚ and 0 < ω < 90˚ and with the crystal oscillating through 2˚ in ω. The resolution was D_{min} - D_{max} 17.78 - 0.72 Å.

This compound crystallised in an orthorhombic non-centrosymmetric cell ($P2_12_12_1$) with one molecule per asymmetric unit. The absolute structure can not be derived based on the absence of heavy atoms in the structure. The molecular formula of this compound is [$C_{12}H_{12}O_2$].

The structure was solved by direct methods using the program SHELXS-97[2] and refined by full matrix least squares on F^2 with SHELXL-97[2]. The hydrogen atoms were included in calculated positions and treated as riding atoms using SHELXL-97 default parameters. All non-hydrogen atoms were refined anisotropically. No absorption correction was applied. The drawing was done using PLATON[3] and show thermal ellipsoids of 50% probability. Tables 1-9 show experimental and crystallographic details of compound 12d.

[1] Stoe & Cie (2002). *X-Area V1.17 & X-RED32 V1.04 Software*. Stoe & Cie GmbH, Darmstadt, Germany.

[2] Sheldrick, G.M. (2008). *Acta Cryst.* **A64**, 112–122.

[3] Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7-13.

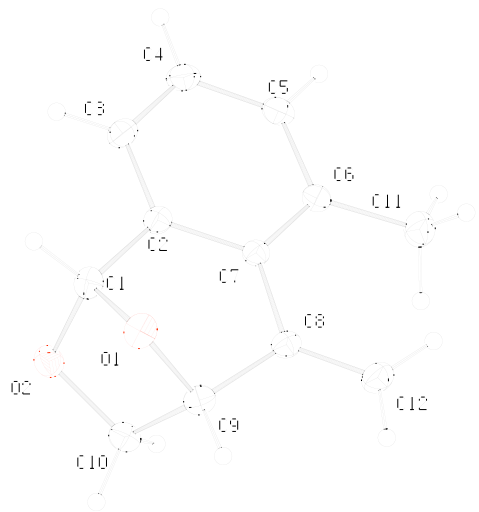


Table 1. Crystal data table for 12d.

Identification code	fb40201
Crystal shape	block
Crystal colour	colourless
Crystal size	.45 x .40 x .35 mm
Empirical formula	C12 H12 O2
Formula weight	188.22
Crystal system	Orthorhombic
Space group	P 21 21 21
Unit cell dimensions	a = 7.9458(6) A alpha = 90 deg. b = 9.3400(7) A beta = 90 deg. c = 12.5560(12) A gamma = 90 deg.
Volume	931.83(13) A^3
Cell refinement parameters	
Reflections	17530
Angle range	1.62 < theta < 29.54
Z	4
Density (calculated)	1.342 g/cm^3
Radiation used	MoK\a
Wavelength	0.71073 A
Linear absorption coefficient	0.090 mm^-1
Temperature	173(2) K

Table 2. Data Collection Details for 12d.

Diffractometer	STOE IPDS 2
Scan method	rotation method
Number of Reflections measured	15942
Number of Independent reflections	2515
Number of observed reflections	2382
Criterion for recognizing	>2sigma(I)

R(int) =	0.1014
Theta range for data collection	2.72 to 29.22 deg.
Index ranges	-10<=h<=10, -12<=k<=12, -17<=l<=17
F(000)	400

Table 3. Refinement Details for 12d.

Refinement method	Full-matrix least-squares on F^2
Final R indices [I>2sigma(I)]	R1 = 0.0368, wR2 = 0.0978
R indices (all data)	R1 = 0.0388, wR2 = 0.0991
R1 [=SUM(||Fo|-|Fc||)/SUM|Fo|]	
wR^2 {=[SUM(w(Fo^2-Fc^2)^2)/SUM(wFo^4)]^1/2}	
H-locating and refining Method	constr
Number of reflections used	2515
Number of L.S. restraints	0
Number of refined Parameters	127
Goodness-of-fit on F^2	1.072
S {=[SUM w(Fo^2-Fc^2)^2]/(n-p)^1/2},	n= number of reflections, p= Parameters used.
w=1/[\s^2^(Fo^2^)+(0.0483P)^2^+0.1026P]	calc where P=(Fo^2^+2Fc^2^)/3
Maximum delta/sigma	0.000
Maximum e-density	0.240 e.A^-3
Minimun e-density	-0.188 e.A^-3

Table 4. Computer Programs used for 12d.

Data collection program	STOE X-AREA
Cell refinement program	STOE X-AREA
Data reduction program	STOE X-RED
Structure Solving Program	SHELXS-97 (Sheldrick, 1990)
Structure Refinement Program	SHELXL-97 (Sheldrick, 1997)
Absolute structure parameter	0.4(10)

Table 5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 12d. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	3147(2)	2319(1)	6489(1)	28(1)
C(2)	4389(1)	1425(1)	5870(1)	25(1)
C(3)	3842(2)	668(1)	4984(1)	30(1)
C(4)	4945(2)	-227(2)	4445(1)	33(1)
C(5)	6571(2)	-371(2)	4816(1)	30(1)
C(6)	7169(1)	376(1)	5703(1)	27(1)
C(7)	6068(1)	1325(1)	6229(1)	23(1)
C(8)	6517(2)	2251(1)	7157(1)	25(1)
C(9)	4999(2)	2869(1)	7713(1)	28(1)
C(10)	3823(2)	1726(1)	8186(1)	30(1)
C(11)	8947(2)	88(2)	6056(1)	40(1)
C(12)	8038(2)	2651(2)	7486(1)	34(1)
O(1)	3932(1)	3536(1)	6925(1)	32(1)
O(2)	2545(1)	1529(1)	7385(1)	32(1)

Table 6. Bond lengths [A] and angles [deg] for 12d.

C(1)-O(1)	1.4080(15)
C(1)-O(2)	1.4273(15)
C(1)-C(2)	1.5089(18)
C(1)-H(1A)	1.0000
C(2)-C(3)	1.3875(17)
C(2)-C(7)	1.4116(15)
C(3)-C(4)	1.388(2)
C(3)-H(3A)	0.9500
C(4)-C(5)	1.3801(19)
C(4)-H(4A)	0.9500
C(5)-C(6)	1.3971(18)
C(5)-H(5A)	0.9500
C(6)-C(7)	1.4097(16)
C(6)-C(11)	1.5046(18)
C(7)-C(8)	1.4934(16)
C(8)-C(12)	1.3310(17)
C(8)-C(9)	1.5089(17)
C(9)-O(1)	1.4446(15)
C(9)-C(10)	1.5374(17)
C(9)-H(9A)	1.0000
C(10)-O(2)	1.4415(16)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-H(11C)	0.9800
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(12)-H(12A)	0.9500
C(12)-H(12B)	0.9500

O(1)-C(1)-O(2)	105.04(10)
O(1)-C(1)-C(2)	110.93(10)
O(2)-C(1)-C(2)	109.85(9)
O(1)-C(1)-H(1A)	110.3
O(2)-C(1)-H(1A)	110.3
C(2)-C(1)-H(1A)	110.3
C(3)-C(2)-C(7)	121.27(11)
C(3)-C(2)-C(1)	119.32(10)
C(7)-C(2)-C(1)	119.35(11)
C(2)-C(3)-C(4)	119.99(12)
C(2)-C(3)-H(3A)	120.0
C(4)-C(3)-H(3A)	120.0
C(5)-C(4)-C(3)	119.02(12)
C(5)-C(4)-H(4A)	120.5
C(3)-C(4)-H(4A)	120.5
C(4)-C(5)-C(6)	122.61(12)
C(4)-C(5)-H(5A)	118.7
C(6)-C(5)-H(5A)	118.7
C(5)-C(6)-C(7)	118.48(11)
C(5)-C(6)-C(11)	117.68(11)
C(7)-C(6)-C(11)	123.83(11)
C(6)-C(7)-C(2)	118.54(11)
C(6)-C(7)-C(8)	125.57(10)
C(2)-C(7)-C(8)	115.89(10)
C(12)-C(8)-C(7)	128.40(12)
C(12)-C(8)-C(9)	118.35(11)
C(7)-C(8)-C(9)	113.08(10)
O(1)-C(9)-C(8)	108.46(10)
O(1)-C(9)-C(10)	102.00(10)
C(8)-C(9)-C(10)	113.49(10)
O(1)-C(9)-H(9A)	110.8
C(8)-C(9)-H(9A)	110.8
C(10)-C(9)-H(9A)	110.8
O(2)-C(10)-C(9)	104.31(10)
O(2)-C(10)-H(10A)	110.9
C(9)-C(10)-H(10A)	110.9
O(2)-C(10)-H(10B)	110.9
C(9)-C(10)-H(10B)	110.9
H(10A)-C(10)-H(10B)	108.9
C(6)-C(11)-H(11C)	109.5
C(6)-C(11)-H(11A)	109.5
H(11C)-C(11)-H(11A)	109.5
C(6)-C(11)-H(11B)	109.5
H(11C)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
C(8)-C(12)-H(12A)	120.0
C(8)-C(12)-H(12B)	120.0
H(12A)-C(12)-H(12B)	120.0
C(1)-O(1)-C(9)	100.24(9)
C(1)-O(2)-C(10)	104.36(9)

Symmetry transformations used to generate equivalent atoms:

Table 7. Torsion-angles for 12d.

O(1)-C(1)-C(2)-C(3)	-147.52(11)
O(2)-C(1)-C(2)-C(3)	96.79(13)

O(1)-C(1)-C(2)-C(7)	35.24(15)
O(2)-C(1)-C(2)-C(7)	-80.45(13)
C(7)-C(2)-C(3)-C(4)	1.13(18)
C(1)-C(2)-C(3)-C(4)	-176.06(12)
C(2)-C(3)-C(4)-C(5)	1.30(19)
C(3)-C(4)-C(5)-C(6)	-1.5(2)
C(4)-C(5)-C(6)-C(7)	-0.77(18)
C(4)-C(5)-C(6)-C(11)	177.80(13)
C(5)-C(6)-C(7)-C(2)	3.12(16)
C(11)-C(6)-C(7)-C(2)	-175.35(12)
C(5)-C(6)-C(7)-C(8)	-177.26(11)
C(11)-C(6)-C(7)-C(8)	4.26(19)
C(3)-C(2)-C(7)-C(6)	-3.36(17)
C(1)-C(2)-C(7)-C(6)	173.83(10)
C(3)-C(2)-C(7)-C(8)	176.99(11)
C(1)-C(2)-C(7)-C(8)	-5.83(15)
C(6)-C(7)-C(8)-C(12)	19.0(2)
C(2)-C(7)-C(8)-C(12)	-161.33(13)
C(6)-C(7)-C(8)-C(9)	-165.84(11)
C(2)-C(7)-C(8)-C(9)	13.79(14)
C(12)-C(8)-C(9)-O(1)	124.60(12)
C(7)-C(8)-C(9)-O(1)	-51.06(12)
C(12)-C(8)-C(9)-C(10)	-122.81(13)
C(7)-C(8)-C(9)-C(10)	61.53(13)
O(1)-C(9)-C(10)-O(2)	20.46(11)
C(8)-C(9)-C(10)-O(2)	-95.99(11)
O(2)-C(1)-O(1)-C(9)	49.87(11)
C(2)-C(1)-O(1)-C(9)	-68.76(12)
C(8)-C(9)-O(1)-C(1)	78.19(11)
C(10)-C(9)-O(1)-C(1)	-41.86(11)
O(1)-C(1)-O(2)-C(10)	-36.61(11)
C(2)-C(1)-O(2)-C(10)	82.75(11)
C(9)-C(10)-O(2)-C(1)	8.81(12)

Table 8. Anisotropic displacement parameters (A^2 x 10^3) for 12d.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	28(1)	25(1)	33(1)	3(1)	-2(1)	1(1)
C(2)	26(1)	23(1)	24(1)	6(1)	-2(1)	-2(1)
C(3)	30(1)	34(1)	26(1)	3(1)	-6(1)	-5(1)
C(4)	39(1)	36(1)	23(1)	-3(1)	-1(1)	-8(1)
C(5)	35(1)	32(1)	25(1)	-2(1)	6(1)	-3(1)
C(6)	27(1)	29(1)	24(1)	3(1)	2(1)	-1(1)
C(7)	25(1)	22(1)	21(1)	4(1)	0(1)	-3(1)
C(8)	30(1)	23(1)	23(1)	3(1)	-2(1)	-3(1)
C(9)	33(1)	23(1)	28(1)	-2(1)	-2(1)	0(1)
C(10)	33(1)	29(1)	28(1)	-1(1)	4(1)	-1(1)
C(11)	30(1)	48(1)	43(1)	-9(1)	-1(1)	7(1)
C(12)	34(1)	36(1)	31(1)	-1(1)	-6(1)	-7(1)
O(1)	35(1)	20(1)	40(1)	1(1)	-3(1)	2(1)
O(2)	29(1)	34(1)	33(1)	0(1)	4(1)	-4(1)

Table 9. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 12d.

	x	y	z	U(eq)
H(1A)	2186	2609	6022	34
H(3A)	2712	763	4747	36
H(4A)	4586	-734	3829	39
H(5A)	7313	-1003	4454	37
H(9A)	5345	3575	8270	34
H(10A)	4439	822	8318	36
H(10B)	3322	2061	8864	36
H(11C)	8982	9	6833	60
H(11A)	9674	877	5826	60
H(11B)	9342	-808	5737	60
H(12A)	9011	2338	7113	40
H(12B)	8151	3248	8094	40

Crystallography for compound 13b

A colourless crystal of compound **13b** was mounted on a Stoe Imaging Plate Diffractometer System[1] equipped with a one-circle φ goniometer and a graphite-monochromator. Data collection was performed at –100˚C using Mo-Kα radiation (λ = 0.71073 Å). 200 exposures (3 min per exposure) were obtained at an image plate distance of 70 mm with 0 < φ < 200˚ and with the crystal oscillating through 1˚ in φ. The resolution was D_{min} - D_{max} 12.45 - 0.81 Å.

This compound crystallised in a monoclinic cell ($P2_1/n$) with one molecule in the asymmetric unit. The molecular formula of this compound is [$C_{19}H_{26}O_2$].

The structure was solved by direct methods using the program SHELXS-97[2] and refined by full matrix least squares on F^2 with SHELXL-97[2]. The hydrogen atoms were included in calculated positions and treated as riding atoms using SHELXL-97 default parameters. No absorption correction was applied. The drawing was done using PLATON[3] and show thermal ellipsoids of 50% probability. Tables 1-9 show experimental and crystallographic details of compound 13b.

[1] Stoe & Cie (2002). *X-Area V1.17 & X-RED32 V1.04 Software*. Stoe & Cie GmbH, Darmstadt, Germany.

[2] Sheldrick, G.M. (2008). *Acta Cryst.* **A64**, 112–122.

[3] Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7-13.

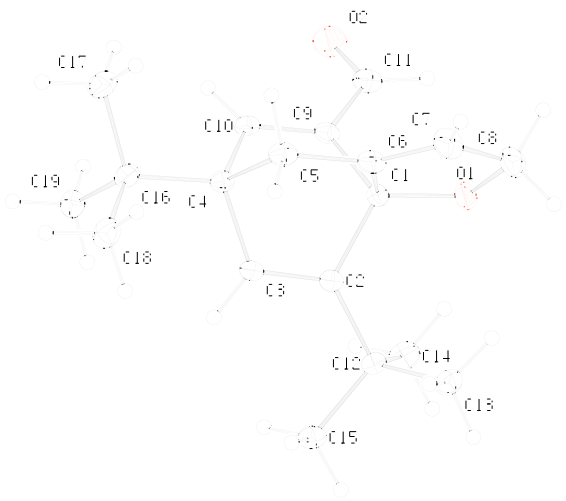


Table 1. Crystal data table for 13b.

Identification code	13b
Crystal shape	block
Crystal colour	colourless
Crystal size	0.45 x 0.40 x 0.35 mm
Empirical formula	C19 H26 O2
Formula weight	286.40
Crystal system	Monoclinic
Space group	P21/n
Unit cell dimensions	a = 14.004(3)A alpha= 90 deg. b = 6.9416(8)A beta= 110.82(2) deg. c = 18.083(4)A gamma= 90 deg.
Volume	1643.1(5) A^3
Cell refinement parameters	
Reflections	6240
Angle range	2.15 < theta < 25.75
Z	4
Density (calculated)	1.158 g/cm^3
Radiation used	MoK\a
Wavelength	0.71073 A
Linear absorption coefficient	0.073 mm^-1
Temperature	173(2) K

Table 2. Data Collection Details for 13b.

Diffractometer	STOE IPDS
Scan method	phi oscillation
Number of Reflections measured	12006
Number of Independent reflections	3198
Number of observed reflections	2195
Criterion for recognizing	>2sigma(I)

R(int) =	0.0562
Theta range for data collection	2.28 to 25.97 deg.
Index ranges	-17<=h<=17, -8<=k<=8, -22<=l<=21
Number of standards	0
Intensity variation	0 %
F(000)	624

Table 3. Refinement Details for 13b.

Refinement method	Full-matrix least-squares on F^2
Final R indices [I>2sigma(I)]	R1 = 0.0382, wR2 = 0.0919
R indices (all data)	R1 = 0.0615, wR2 = 0.0981
R1 [=SUM(||Fo|-|Fc||)/SUM|Fo|]	
wR^2 {=[SUM(w(Fo^2-Fc^2)^2)/SUM(wFo^4)]^1/2}	
H-locating and refining Method	constr
Number of reflections used	3198
Number of L.S. restraints	0
Number of refined Parameters	196
Goodness-of-fit on F^2	0.985
S {=[SUM w(Fo^2-Fc^2)^2]/(n-p)^1/2},	n= number of reflections, p= Parameters used.
w=1/[\s^2^(Fo^2^)+(0.0565P)^2^+0.0000P]	calc where P=(Fo^2^+2Fc^2^)/3
Maximum delta/sigma	0.000
Maximum e-density	0.275 e.A^-3
Minimun e-density	-0.174 e.A^-3

Table 4. Computer Programs used for 13b.

Data collection program	EXPOSE (Stoe IPDS Software, 2000)
Cell refinement program	CELL (Stoe IPDS Software, 2000)
Data reduction program	INTEGRATE (Stoe IPDS Software, 2000)
Structure Solving Program	SHELXS-97 (Sheldrick, 1990)

Structure Refinement Program	SHELXL-97 (Sheldrick, 1997)
Pictures drawn with	PLATON99 (Spek, 1990)
Tables made with	SHELXL-97 (Sheldrick, 1997)

Table 5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 13b. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2048(1)	4396(2)	-445(1)	23(1)
C(2)	2273(1)	3522(2)	-1147(1)	21(1)
C(3)	1405(1)	3099(2)	-1735(1)	22(1)
C(4)	398(1)	3533(2)	-1615(1)	23(1)
C(5)	442(1)	5774(2)	-1452(1)	28(1)
C(6)	1396(1)	6169(2)	-763(1)	28(1)
C(7)	1825(1)	7656(2)	-314(1)	39(1)
C(8)	2783(1)	7074(2)	334(1)	40(1)
C(9)	1345(1)	3027(2)	-221(1)	23(1)
C(10)	491(1)	2611(2)	-829(1)	24(1)
C(11)	1570(1)	2425(2)	604(1)	32(1)
C(12)	3354(1)	3227(2)	-1142(1)	25(1)
C(13)	3923(1)	5155(2)	-1031(1)	33(1)
C(14)	3925(1)	1841(2)	-465(1)	36(1)
C(15)	3338(1)	2339(2)	-1920(1)	34(1)
C(16)	-569(1)	2901(2)	-2319(1)	28(1)
C(17)	-1547(1)	3390(3)	-2153(1)	41(1)
C(18)	-620(1)	3915(2)	-3091(1)	38(1)
C(19)	-542(1)	717(2)	-2438(1)	34(1)
O(1)	2898(1)	5044(1)	216(1)	30(1)
O(2)	1160(1)	1110(2)	809(1)	45(1)

Table 6. Bond lengths [A] and angles [deg] for 13b.

C(1)-O(1)	1.4278(16)
C(1)-C(6)	1.5183(18)
C(1)-C(9)	1.5223(18)
C(1)-C(2)	1.5373(19)
C(2)-C(3)	1.3326(18)
C(2)-C(12)	1.5242(18)
C(3)-C(4)	1.5309(18)
C(3)-H(3A)	0.9500
C(4)-C(10)	1.5207(19)
C(4)-C(16)	1.5571(19)
C(4)-C(5)	1.5806(18)
C(5)-C(6)	1.492(2)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.319(2)
C(7)-C(8)	1.489(2)

C(7)-H(7A)	0.9500
C(8)-O(1)	1.4432(17)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(10)	1.3369(19)
C(9)-C(11)	1.4714(19)
C(10)-H(10A)	0.9500
C(11)-O(2)	1.2052(17)
C(11)-H(11A)	0.9500
C(12)-C(15)	1.530(2)
C(12)-C(13)	1.5339(19)
C(12)-C(14)	1.5384(19)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15C)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15A)	0.9800
C(16)-C(19)	1.534(2)
C(16)-C(17)	1.541(2)
C(16)-C(18)	1.541(2)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800

O(1)-C(1)-C(6)	105.74(10)
O(1)-C(1)-C(9)	113.50(11)
C(6)-C(1)-C(9)	104.37(11)
O(1)-C(1)-C(2)	117.46(11)
C(6)-C(1)-C(2)	106.27(11)
C(9)-C(1)-C(2)	108.39(10)
C(3)-C(2)-C(12)	126.76(12)
C(3)-C(2)-C(1)	110.38(12)
C(12)-C(2)-C(1)	122.86(11)
C(2)-C(3)-C(4)	117.94(12)
C(2)-C(3)-H(3A)	121.0
C(4)-C(3)-H(3A)	121.0
C(10)-C(4)-C(3)	105.83(10)
C(10)-C(4)-C(16)	114.00(11)
C(3)-C(4)-C(16)	113.90(11)
C(10)-C(4)-C(5)	104.81(10)
C(3)-C(4)-C(5)	104.07(11)
C(16)-C(4)-C(5)	113.26(10)
C(6)-C(5)-C(4)	107.49(11)
C(6)-C(5)-H(5A)	110.2
C(4)-C(5)-H(5A)	110.2
C(6)-C(5)-H(5B)	110.2
C(4)-C(5)-H(5B)	110.2
H(5A)-C(5)-H(5B)	108.5
C(7)-C(6)-C(5)	137.26(13)
C(7)-C(6)-C(1)	108.72(13)
C(5)-C(6)-C(1)	113.93(11)

C(6)-C(7)-C(8)	110.98(13)
C(6)-C(7)-H(7A)	124.5
C(8)-C(7)-H(7A)	124.5
O(1)-C(8)-C(7)	105.37(11)
O(1)-C(8)-H(8A)	110.7
C(7)-C(8)-H(8A)	110.7
O(1)-C(8)-H(8B)	110.7
C(7)-C(8)-H(8B)	110.7
H(8A)-C(8)-H(8B)	108.8
C(10)-C(9)-C(11)	125.34(13)
C(10)-C(9)-C(1)	112.95(12)
C(11)-C(9)-C(1)	121.43(12)
C(9)-C(10)-C(4)	115.55(12)
C(9)-C(10)-H(10A)	122.2
C(4)-C(10)-H(10A)	122.2
O(2)-C(11)-C(9)	125.05(14)
O(2)-C(11)-H(11A)	117.5
C(9)-C(11)-H(11A)	117.5
C(2)-C(12)-C(15)	111.00(11)
C(2)-C(12)-C(13)	110.66(11)
C(15)-C(12)-C(13)	107.93(12)
C(2)-C(12)-C(14)	108.86(11)
C(15)-C(12)-C(14)	108.29(12)
C(13)-C(12)-C(14)	110.07(11)
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(12)-C(14)-H(14A)	109.5
C(12)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(12)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(12)-C(15)-H(15C)	109.5
C(12)-C(15)-H(15B)	109.5
H(15C)-C(15)-H(15B)	109.5
C(12)-C(15)-H(15A)	109.5
H(15C)-C(15)-H(15A)	109.5
H(15B)-C(15)-H(15A)	109.5
C(19)-C(16)-C(17)	108.22(13)
C(19)-C(16)-C(18)	108.70(12)
C(17)-C(16)-C(18)	108.53(12)
C(19)-C(16)-C(4)	109.59(11)
C(17)-C(16)-C(4)	110.69(11)
C(18)-C(16)-C(4)	111.04(11)
C(16)-C(17)-H(17A)	109.5
C(16)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109.5
C(16)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(16)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(16)-C(19)-H(19A)	109.5

C(16)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(16)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(1)-O(1)-C(8)	109.15(11)

Symmetry transformations used to generate equivalent atoms:

Table 7. Torsion-angles for 13b.

O(1)-C(1)-C(2)-C(3)	-176.11(10)
C(6)-C(1)-C(2)-C(3)	-58.06(13)
C(9)-C(1)-C(2)-C(3)	53.62(14)
O(1)-C(1)-C(2)-C(12)	3.45(18)
C(6)-C(1)-C(2)-C(12)	121.50(13)
C(9)-C(1)-C(2)-C(12)	-126.81(12)
C(12)-C(2)-C(3)-C(4)	-179.53(12)
C(1)-C(2)-C(3)-C(4)	0.01(16)
C(2)-C(3)-C(4)-C(10)	-51.33(15)
C(2)-C(3)-C(4)-C(16)	-177.36(11)
C(2)-C(3)-C(4)-C(5)	58.84(14)
C(10)-C(4)-C(5)-C(6)	54.68(14)
C(3)-C(4)-C(5)-C(6)	-56.24(13)
C(16)-C(4)-C(5)-C(6)	179.54(11)
C(4)-C(5)-C(6)-C(7)	-173.86(17)
C(4)-C(5)-C(6)-C(1)	2.11(16)
O(1)-C(1)-C(6)-C(7)	-1.26(16)
C(9)-C(1)-C(6)-C(7)	118.73(13)
C(2)-C(1)-C(6)-C(7)	-126.81(13)
O(1)-C(1)-C(6)-C(5)	-178.37(11)
C(9)-C(1)-C(6)-C(5)	-58.38(14)
C(2)-C(1)-C(6)-C(5)	56.08(14)
C(5)-C(6)-C(7)-C(8)	176.23(16)
C(1)-C(6)-C(7)-C(8)	0.12(18)
C(6)-C(7)-C(8)-O(1)	1.05(18)
O(1)-C(1)-C(9)-C(10)	172.53(11)
C(6)-C(1)-C(9)-C(10)	57.90(14)
C(2)-C(1)-C(9)-C(10)	-55.06(15)
O(1)-C(1)-C(9)-C(11)	-1.75(17)
C(6)-C(1)-C(9)-C(11)	-116.38(13)
C(2)-C(1)-C(9)-C(11)	130.67(12)
C(11)-C(9)-C(10)-C(4)	175.20(12)
C(1)-C(9)-C(10)-C(4)	1.18(16)
C(3)-C(4)-C(10)-C(9)	49.99(15)
C(16)-C(4)-C(10)-C(9)	175.95(11)
C(5)-C(4)-C(10)-C(9)	-59.67(14)
C(10)-C(9)-C(11)-O(2)	20.1(2)
C(1)-C(9)-C(11)-O(2)	-166.33(14)
C(3)-C(2)-C(12)-C(15)	-0.32(19)
C(1)-C(2)-C(12)-C(15)	-179.81(11)
C(3)-C(2)-C(12)-C(13)	119.49(14)
C(1)-C(2)-C(12)-C(13)	-60.00(16)
C(3)-C(2)-C(12)-C(14)	-119.42(14)
C(1)-C(2)-C(12)-C(14)	61.09(16)
C(10)-C(4)-C(16)-C(19)	-60.77(15)
C(3)-C(4)-C(16)-C(19)	60.83(14)
C(5)-C(4)-C(16)-C(19)	179.50(12)

C(10)-C(4)-C(16)-C(17)	58.52(15)
C(3)-C(4)-C(16)-C(17)	-179.88(12)
C(5)-C(4)-C(16)-C(17)	-61.21(15)
C(10)-C(4)-C(16)-C(18)	179.13(11)
C(3)-C(4)-C(16)-C(18)	-59.27(15)
C(5)-C(4)-C(16)-C(18)	59.41(15)
C(6)-C(1)-O(1)-C(8)	1.91(14)
C(9)-C(1)-O(1)-C(8)	-111.90(12)
C(2)-C(1)-O(1)-C(8)	120.25(13)
C(7)-C(8)-O(1)-C(1)	-1.85(15)

Table 8. Anisotropic displacement parameters (A^2 x 10^3) for 13b.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	24(1)	22(1)	23(1)	-2(1)	9(1)	-1(1)
C(2)	25(1)	17(1)	24(1)	2(1)	12(1)	1(1)
C(3)	26(1)	20(1)	22(1)	1(1)	12(1)	2(1)
C(4)	23(1)	22(1)	26(1)	2(1)	11(1)	2(1)
C(5)	31(1)	22(1)	36(1)	3(1)	16(1)	7(1)
C(6)	36(1)	19(1)	34(1)	1(1)	20(1)	3(1)
C(7)	52(1)	21(1)	47(1)	-6(1)	21(1)	-1(1)
C(8)	51(1)	29(1)	41(1)	-13(1)	20(1)	-10(1)
C(9)	29(1)	18(1)	27(1)	-1(1)	15(1)	2(1)
C(10)	26(1)	20(1)	31(1)	0(1)	17(1)	2(1)
C(11)	36(1)	31(1)	33(1)	0(1)	19(1)	1(1)
C(12)	22(1)	26(1)	27(1)	1(1)	11(1)	1(1)
C(13)	30(1)	36(1)	36(1)	1(1)	15(1)	-6(1)
C(14)	30(1)	37(1)	41(1)	9(1)	12(1)	7(1)
C(15)	27(1)	42(1)	37(1)	-4(1)	16(1)	4(1)
C(16)	22(1)	33(1)	28(1)	2(1)	9(1)	-1(1)
C(17)	24(1)	55(1)	42(1)	-2(1)	12(1)	2(1)
C(18)	29(1)	49(1)	31(1)	7(1)	6(1)	-3(1)
C(19)	32(1)	37(1)	33(1)	-6(1)	11(1)	-8(1)
O(1)	33(1)	28(1)	27(1)	-7(1)	9(1)	-6(1)
O(2)	55(1)	47(1)	38(1)	6(1)	23(1)	-7(1)

Table 9. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 13b.

	x	y	z	U(eq)
H(3A)	1410	2546	-2214	26
H(5A)	451	6491	-1923	34
H(5B)	-163	6186	-1331	34
H(7A)	1559	8929	-393	47
H(8A)	3374	7805	302	48
H(8B)	2728	7310	857	48
H(10A)	-24	1794	-774	28
H(11A)	2077	3127	1005	38

H(13A)	3994	5701	-515	50
H(13B)	3537	6048	-1450	50
H(13C)	4602	4946	-1058	50
H(14A)	3570	599	-551	54
H(14B)	3945	2388	40	54
H(14C)	4624	1654	-452	54
H(15C)	4041	2162	-1902	51
H(15B)	2972	3199	-2360	51
H(15A)	2992	1088	-1998	51
H(17A)	-1586	4786	-2087	61
H(17B)	-1532	2736	-1669	61
H(17C)	-2146	2961	-2599	61
H(18A)	16	3682	-3187	56
H(18B)	-710	5304	-3042	56
H(18C)	-1197	3405	-3533	56
H(19A)	-539	54	-1959	51
H(19B)	76	376	-2544	51
H(19C)	-1146	325	-2887	51