

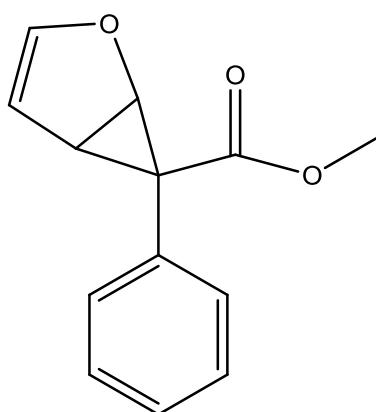
Universitetet i Oslo

Det matematisk-naturvitenskaplige fakultet

Exam in: KJM3000
Day of exam: 2013-06-07
Exam hours: 14.30 – 18.30 (4 hours)
This examination paper consists of 2 page(s).
Appendices: 3 (1, 9 and 1 pages respectively)
Permitted materials: Ruler, calculator and molecular modelling kit

Make sure that your copy of this examination paper is complete before answering

Task 1 (30%)

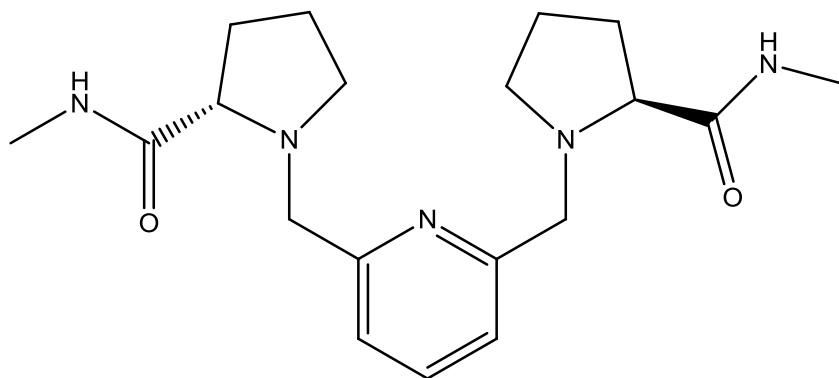


1

- a.** The following data are given for compound **1**: $^1\text{H NMR}$ (CDCl_3 , 500 MHz): δ 7.29-7.23 (m, 3H), 7.19 (m, 2H), 5.90 (d, $J = 2.5$ Hz, 1H), 5.22 (t, $J = 2.5$ Hz, 1H), 5.13 (d, $J = 5.5$ Hz, 1H), 3.61 (s, 3H), 3.30 (dd, $J = 5.5$ og 2,5 Hz, 1H). Assign the signals and explain briefly the coupling pattern.
- b.** There is a total of 5 sp^3 hybridized C-H bonds in compound **1**. Three of them have an IR stretching frequency around $2800\text{-}2950\text{ cm}^{-1}$, while two have IR stretching frequencies above 3000 cm^{-1} . Explain briefly this difference.

- c. Identify the compound whose mass spectrum are found in attachment 1, page 2. Draw structural equations with arrows for the market ions and explain briefly your reasoning.

Task 2 (30%)



III

Spectroscopic data for compound **III** are found in attachment 2.

- Label the carbons and protons in compound **III** and assign as many of the signals as possible in the attached ^1H - and ^{13}C -NMR spectra by setting up a table with values for: Chemical shift, multiplicity (coupling), coupling constants, and number of atoms (integrals).
- Write structural equations with arrows to account for the following values in the *two* attached mass spectra: Ms-spectrum 1 (Electrospray ionization): 360 og 361. Ms-spectrum 2 (EI,70 eV): 301, 302 og 233.

Task 3 (40%).

Identify the compound whose spectroscopic data are found in attachment 3. Assign as many of the signals in the ^{13}C - and ^1H -NMR spectra as possible and give a brief explanation. Comment briefly on the given EA/MS-, IR- og UV-data.

Elemental analysis: C:83.53; H:9.51; N:6.96

HRMS (EI): 201.1517

UV: $\lambda_{\text{max}} \approx 210 \text{ nm}$, $\epsilon_{\text{max}} \approx 7000$.

IR: 3100 (m), 2950 (s), 2250 (m).

^1H -NMR (500 MHz, CDCl_3) δ = 6.87 (s, 2H) 3.76 (d, J = 10.4 Hz, 1H), 2.39 (s, 6H), 2.39-2.33 (m, 1H, J = 10.4, 6.7 and 6.6 Hz), 2.26 (s, 3H), 1.30 (d, J = 6.6 Hz, 3H), 0.77 (d, J = 6.7 Hz, 3H).

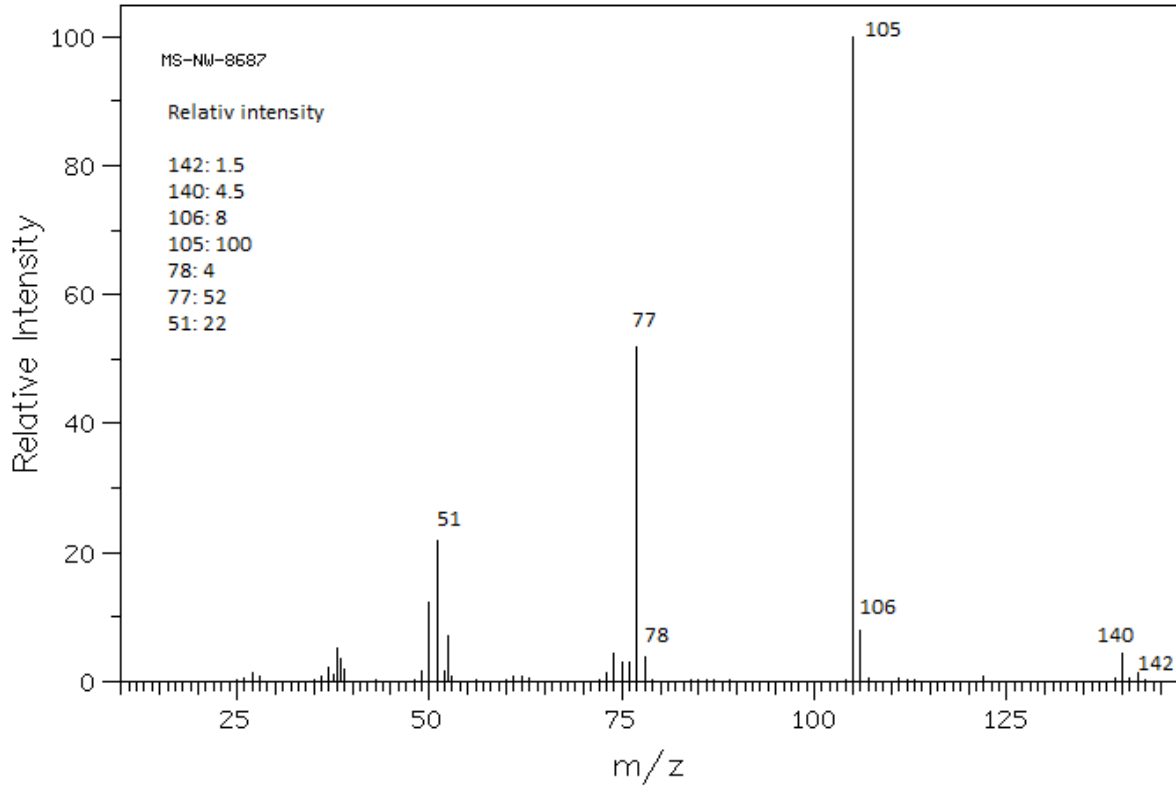
^{13}C -NMR (125 MHz, CDCl_3) δ = 137.4(s), 136.4(s), 130.3(s), 128.6(d), 120.5(s), 38.7(d), 30.9(d), 21.9(q), 20.9(q), 20.7(q), 19.7(q).

Vedlegg 1 / Attachment 1

Table 4.3 Atomic weights and approximate natural abundance of some isotopes

<i>Isotope</i>	<i>Atomic weight</i> (¹² C = 12.000 000)	<i>Natural abundance</i> (%)
¹ H	1.007 825	99.985
² H	2.014 102	0.015
¹² C	12.000 000	98.9
¹³ C	13.003 354	1.1
¹⁴ N	14.003 074	99.64
¹⁵ N	15.000 108	0.36
¹⁶ O	15.994 915	99.8
¹⁷ O	16.999 133	0.04
¹⁸ O	17.999 160	0.2
¹⁹ F	18.998 405	100
²⁸ Si	27.976 927	92.2
²⁹ Si	28.976 491	4.7
³⁰ Si	29.973 761	3.1
³¹ P	30.973 763	100
³² S	31.972 074	95.0
³³ S	32.971 461	0.76
³⁴ S	33.967 865	4.2
³⁵ Cl	34.968 855	75.8
³⁷ Cl	36.965 896	24.2
⁷⁹ Br	78.918 348	50.5
⁸¹ Br	80.916 344	49.5
¹²⁷ I	126.904 352	100

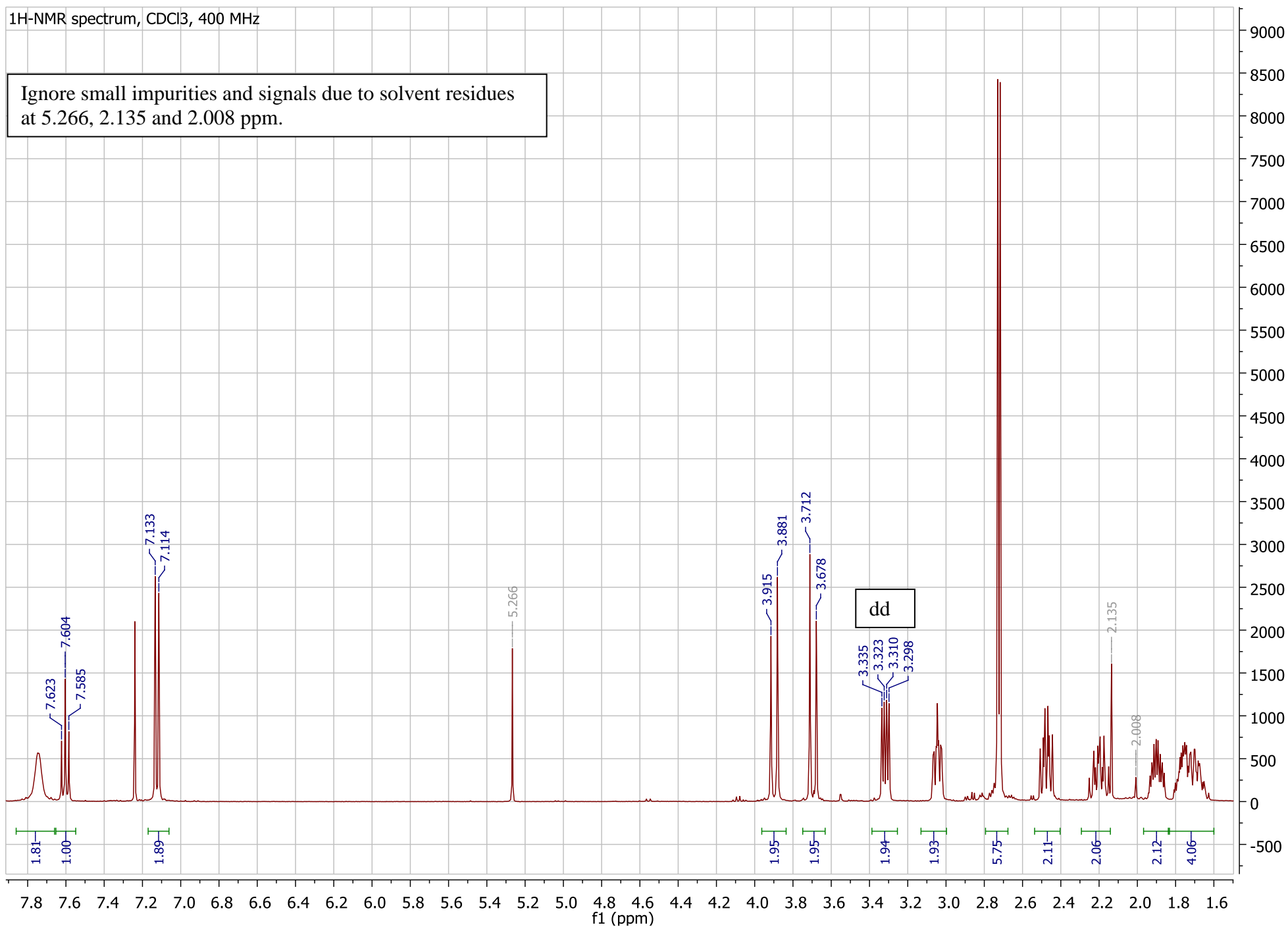
Task 1c: MS (EI, 70 eV):



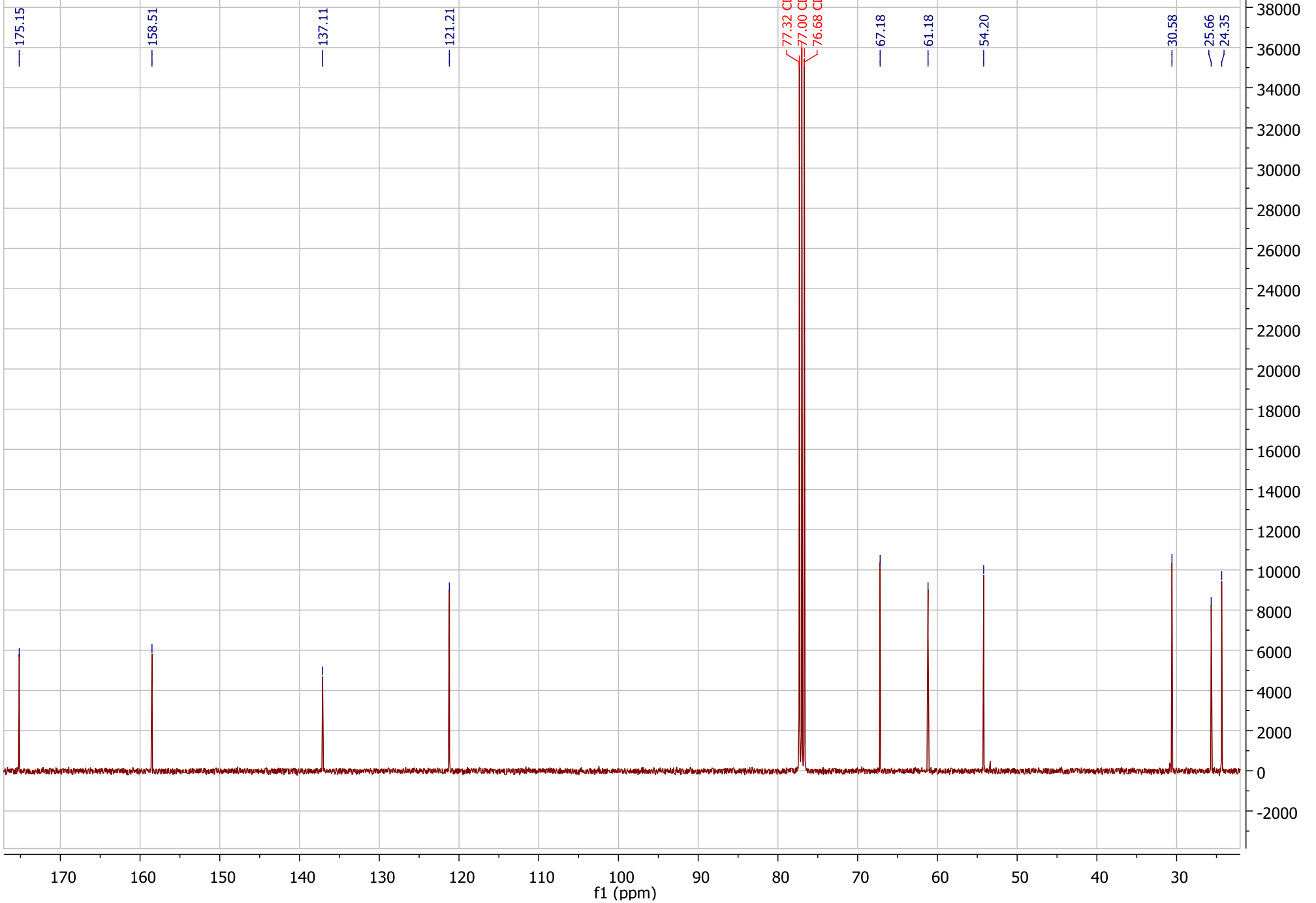
Vedlegg 2 / Attachment 2

MS-data and NMR-data for compound **III**.

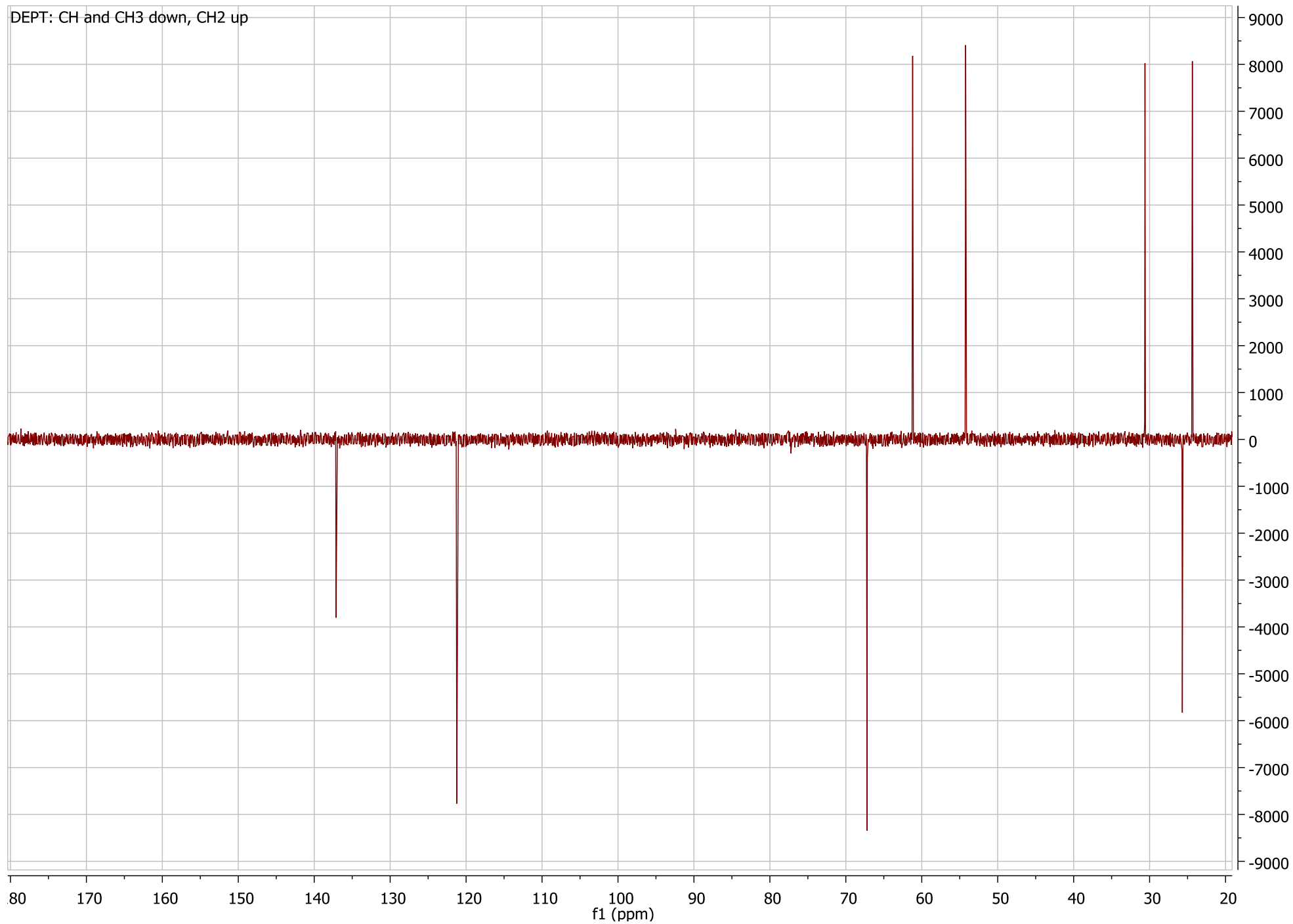
Ignore small impurities and signals due to solvent residues at 5.266, 2.135 and 2.008 ppm.

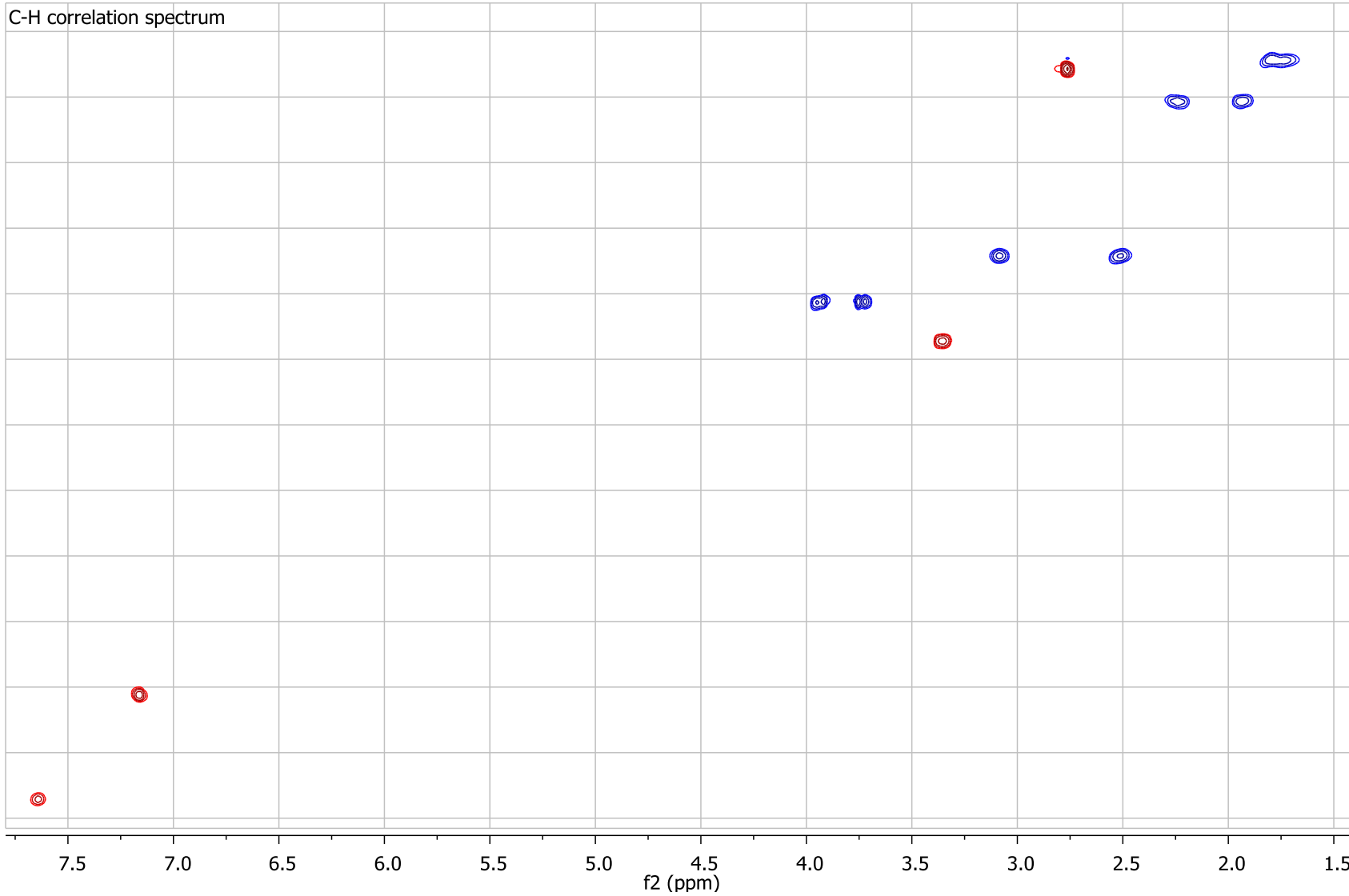


13C-NMR, 100MHz



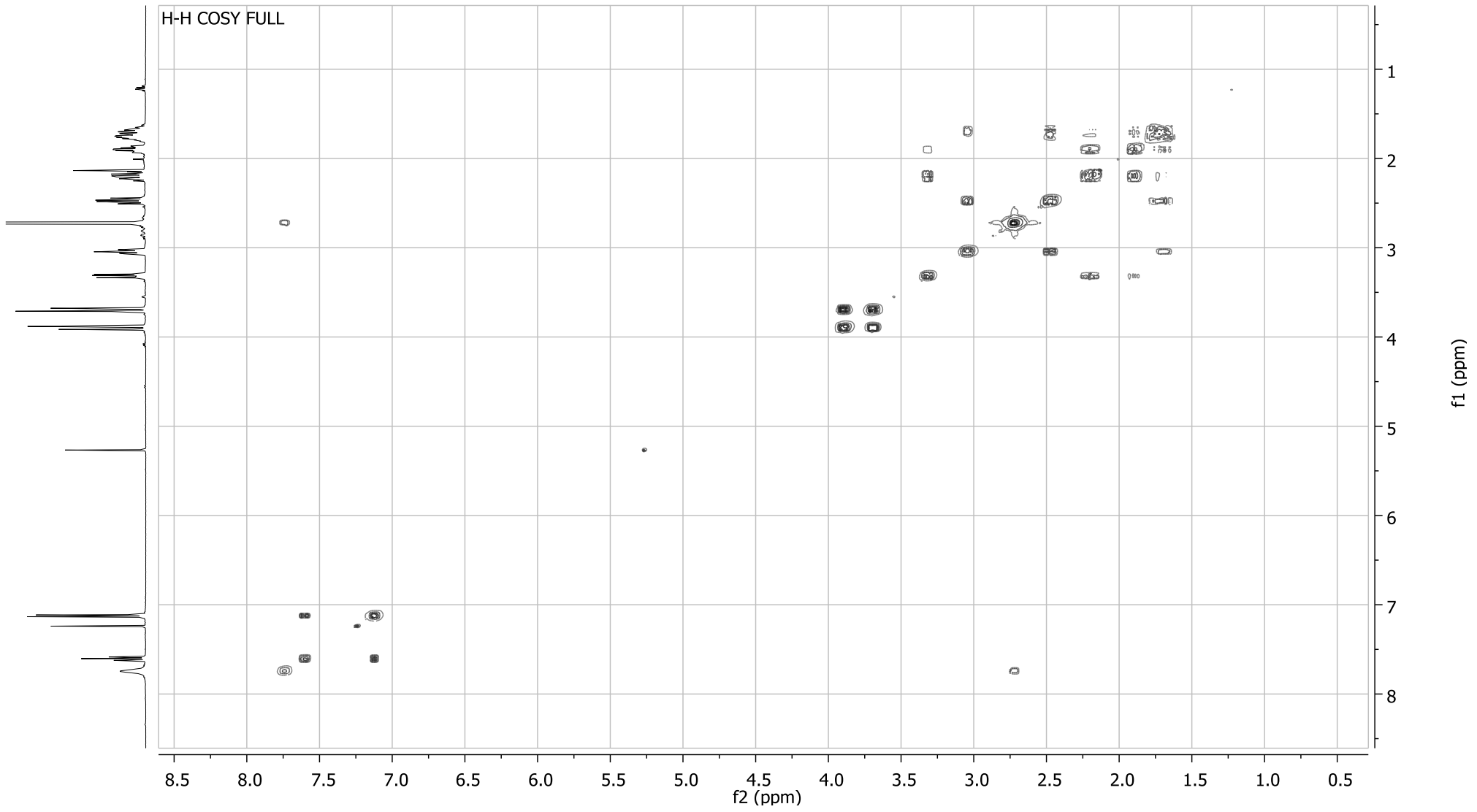
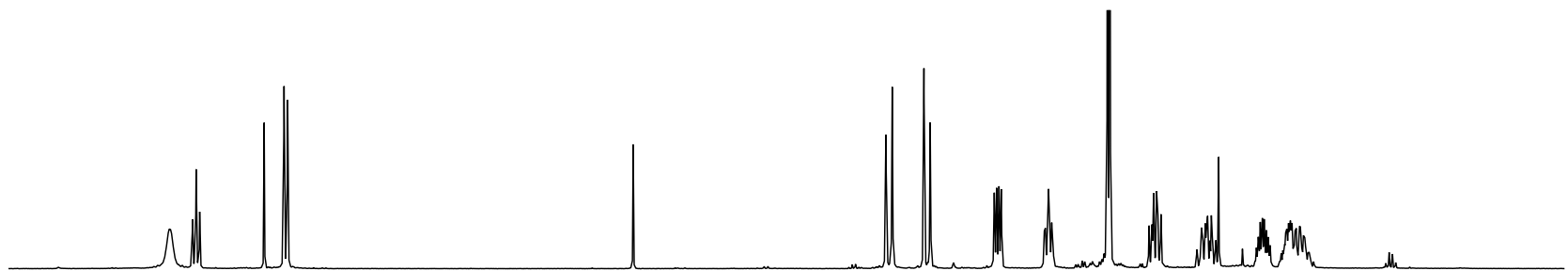
DEPT: CH and CH3 down, CH2 up

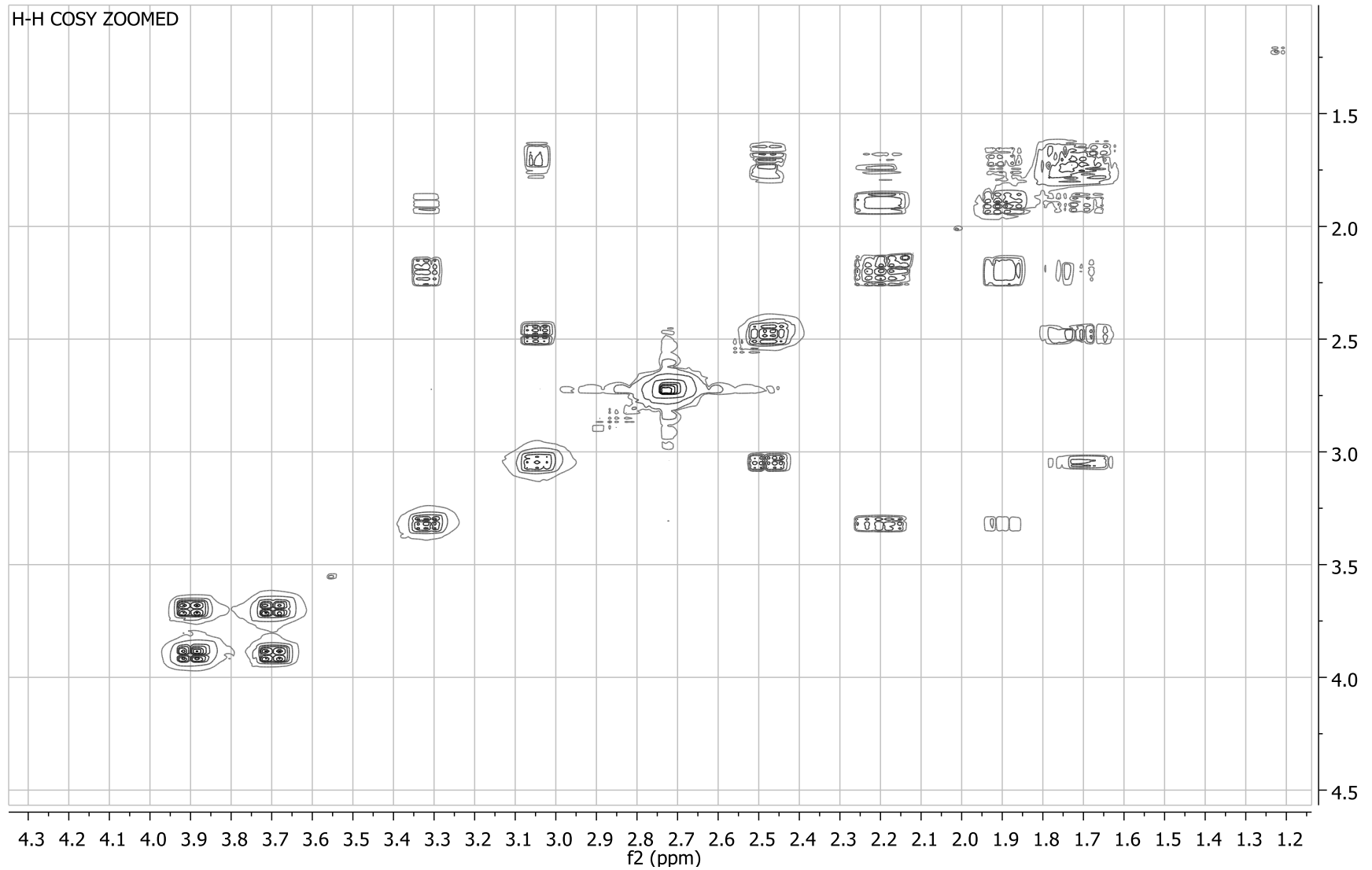
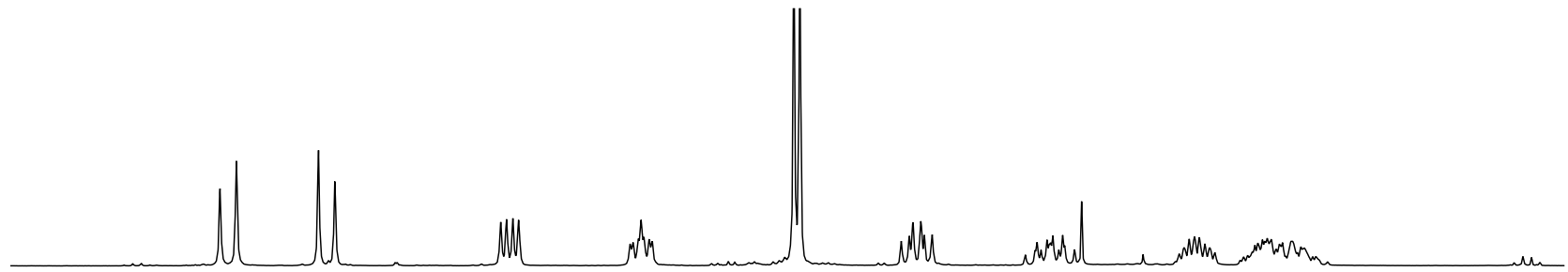




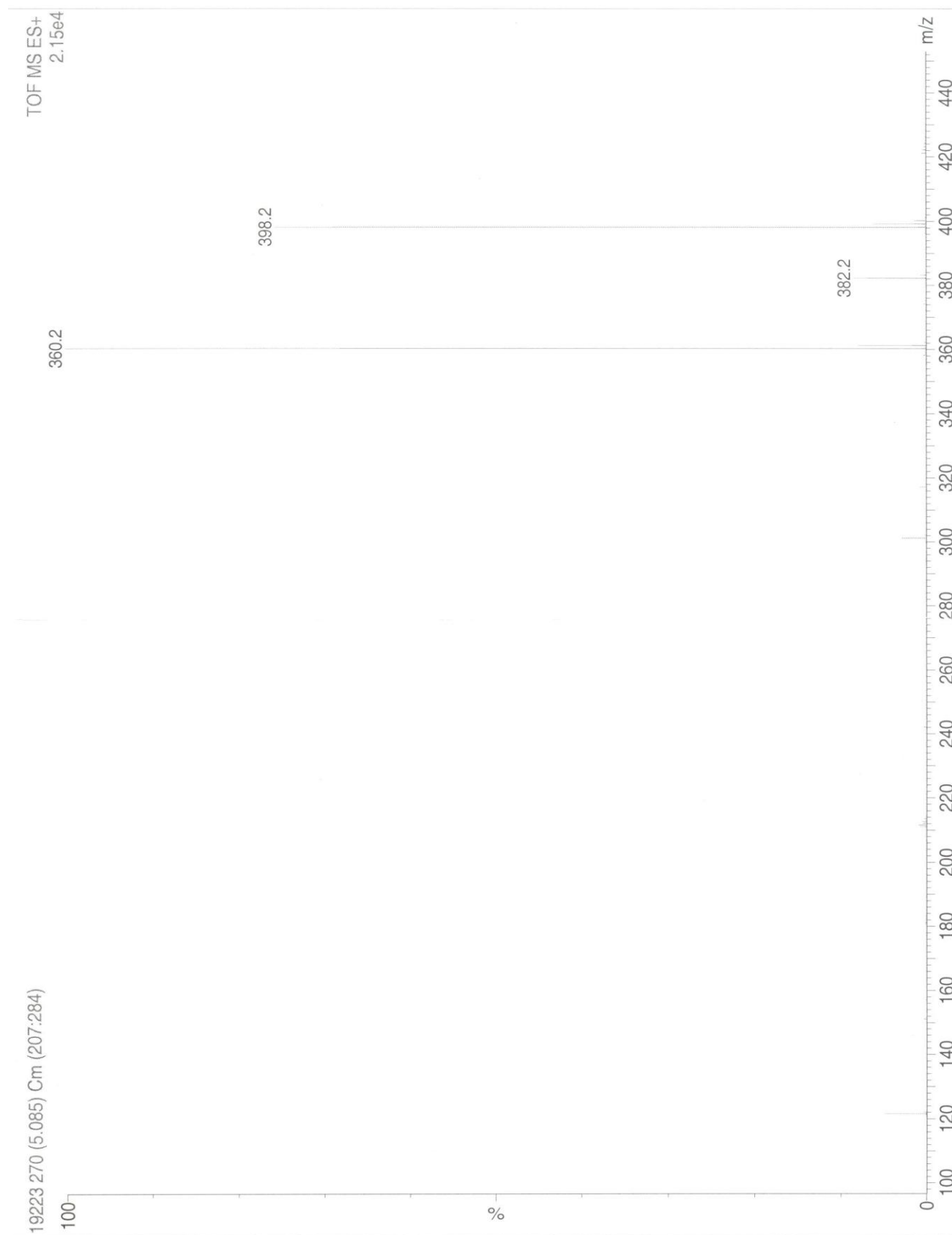
f2 (ppm)

f1 (ppm)

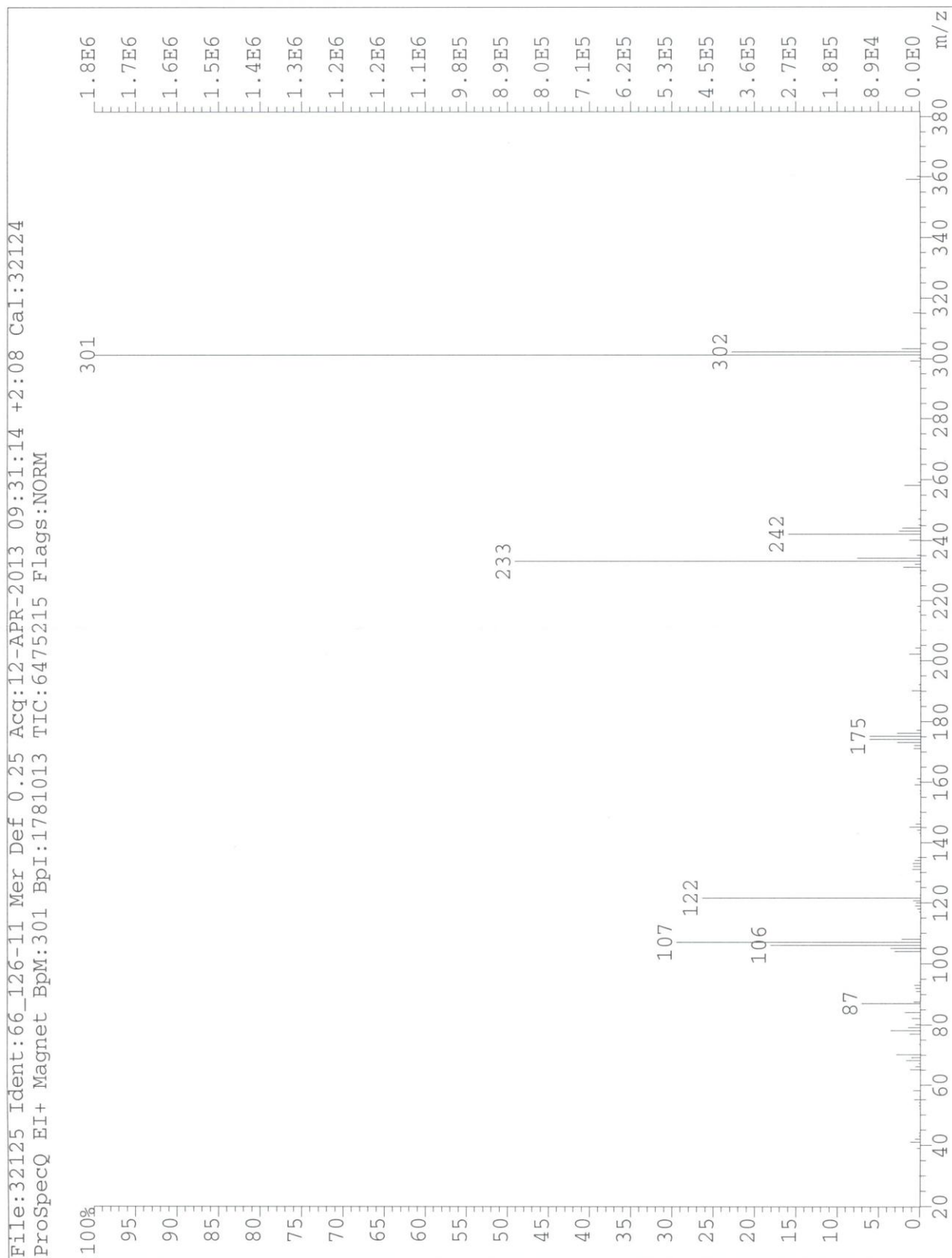




LR-MS (Electrospray) **III**: Ignore peaks at 382 and 398.



LR-MS (EI, 70 eV) III:



Vedlegg 3 / Attachment 3

