

Low background FRET- substrates for lipases and esterases suitable for high-throughput screening under basic (pH 11) conditions

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Supporting Information

- Rate data for all enzyme/substrate pairs tested (Table S1)
- Cocktail assay rate (Figure S1) and HPLC traces (Figure S2)
- NMR, MS spectra or all compounds

Table S1. (Next page) Reactions of substrates **1a-15a** with lipases and esterases. For each enzyme/substrate pair, the first number is the apparent initial reaction rate in nM/s measured by fluorescence at 10 μ M substrate, 50 μ g/ml enzyme, 25°C in aq. PBS with 30% v/v DMSO, as shown in Figure 1. The second percentage number gives the conversion of each substrate under cocktail condition (50 μ M cocktail **1a-15a**, 50 μ g/ml enzyme in aq. PBS with 30% v/v DMSO, 25°C, 2 h) as determined by HPLC analysis of products **1b-15b**, as shown in Figure 3. Error margins are ± 10 % of value. Enzymes codes: L9: Roche *Mucor miehei* lipase; L8: Roche *Thermocycles lanuginose* lipase; ANL: *Aspergillus niger* lipase 1.00 U/mg (Fluka 62294); HrLE: horse liver esterase 250 U/mg (Fluka 46058); E1: Roche pig liver esterase; E2: Roche pig liver esterase; L7: Roche pig pancreas lipase; PSBL2: *Pseudomonas sp.* Type B lipoprotein lipase 223 U/mg (Fluka 62336); MML: *Mucor miehei* lipase 1.3 U/mg (Fluka 62298); L2: Roche *Candida Antarctica* lipase fraction B; L6: Roche *Pseudomonas sp.* lipase; PSL: *Pseudomonas sp.* Lipoprotein lipase 1560 U/mg (Fluka 62335); RAL: *Rhizopus arrhizus* lipase 2 U/g (Fluka 62305); MME: *Mucor miehei* esterase 1.2 U/mg (Fluka 46059); PSBL1: *Pseudomonas sp.* Type B lipoprotein lipase 85,000 U/mg (SIGMA L-9656); CVL: *Chromobacterium visc.* lipoprotein lipase 2832 U/mg (Fluka 62333); RML: *Rhizomucor miehei* lipase 0.51 U/mg (Fluka 62291); L3p: Roche *Candida rugosa* lipase purified; HPL: hog pancreatic lipase 16.8 U/mg (Fluka 62300); L10: Roche *Alcaligines sp.* lipase; CAL: *Candida Antarctica* 3.2 U/mg (Fluka 62299); HLE: horse liver esterase 0.74 U/mg (Fluka 46069); MJL: *Mucor javanicus* lipase 4.9 U/mg (Fluka 62304); BTE: *Bacillus thermoglucodasium* esterase 0.082 U/mg (Fluka 46054); CLE: *Candida lipolytica* esterase 0.10 U/mg (Fluka 46056); CCL: *Candida cyclindraceae* lipase 2.4 U/mg (Fluka 62316); PCL: *Pseudomonas cepacia* lipase 48 U/mg (Fluka 62309); L5: Roche *Candida Antarctica* lipase. The following enzymes did not show significant reactivity with **1a-15a**: L3: Roche *Candida rugosa* lipase; TBE: *Thermoanaerobium brockii* esterase 0.15 U/mg (Fluka 46061); BSE: *Bacillus sp.* Esterase 0.15 U/mg (Fluka 46062); SCE: *Saccharomyces cerevisiae* esterase 2.2 U/g (Fluka 46071); WGL: Wheat germ lipase 0.08 U/mg (Fluka 62306); CLL: *Candida lipolytica* lipase 1.0 U/g (Fluka 62303); PRL: *Penicillium roquefortii* lipase 151 U/g (Fluka 62308); RNL: *Rhizopus niveus* lipase 2.6 U/mg (Fluka 62310).

Enzyme	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15															
L9	27.9	78%	36.3	81%	41.8	88%	30.9	77%	26.2	92%	52.1	80%	44.1	77%	47.7	84%	13.4	82%	17.9	77%	0.0	0%	16.7	87%	15.3	89%	17.1	95%	9.4	84%
L8	21.9	77%	34.7	82%	31.4	88%	27.4	84%	22.5	88%	46.0	78%	38.8	71%	45.2	76%	11.0	80%	14.5	70%	0.0	0%	12.5	82%	10.8	80%	9.3	88%	5.1	74%
ANL	26.1	90%	30.8	90%	30.5	93%	23.6	83%	16.3	100%	35.5	81%	2.1	81%	37.7	81%	12.0	95%	12.6	93%	0.0	0%	16.4	91%	14.6	87%	19.7	94%	9.3	79%
HrLE	27.7	79%	28.5	88%	11.6	79%	23.3	88%	12.5	82%	24.8	73%	9.0	73%	21.8	64%	8.5	79%	8.1	72%	1.3	38%	4.9	64%	5.1	70%	4.1	65%	2.5	59%
E1	5.8	79%	9.0	80%	5.1	77%	6.0	79%	7.0	70%	4.9	74%	6.6	66%	5.3	65%	4.5	83%	5.2	71%	0.3	6%	1.9	54%	3.4	70%	1.3	47%	1.0	47%
E2	4.9	68%	7.8	66%	4.4	65%	4.0	73%	4.7	65%	4.3	64%	7.1	66%	5.5	59%	3.7	71%	3.8	58%	0.5	19%	2.0	62%	3.1	68%	1.3	48%	0.9	41%
L7	6.3	68%	23.3	77%	12.2	86%	3.2	52%	5.5	74%	6.9	58%	4.0	50%	0.9	20%	2.9	64%	5.3	56%	0.0	0%	1.3	33%	2.6	44%	1.0	48%	0.8	31%
PSBL2	7.4	60%	6.6	59%	3.7	69%	2.1	52%	2.6	56%	3.0	43%	7.5	60%	3.9	53%	1.9	60%	5.5	62%	0.0	0%	2.3	67%	2.2	63%	1.9	60%	0.8	49%
MML	26.0	51%	21.6	37%	17.5	43%	4.8	25%	10.7	31%	9.1	29%	12.2	35%	7.9	34%	4.2	36%	6.6	53%	0.0	0%	2.8	37%	2.9	39%	2.7	42%	1.4	30%
L2	0.9	17%	5.8	30%	5.9	51%	20.8	51%	9.0	57%	30.8	49%	0.3	8%	0.6	12%	7.3	59%	3.3	28%	0.0	9%	0.2	0%	5.6	59%	1.9	45%	2.4	56%
L6	1.4	49%	1.3	37%	1.6	51%	0.7	23%	1.2	40%	1.9	30%	4.9	59%	2.4	49%	1.5	51%	4.6	55%	0.0	0%	2.5	53%	2.3	49%	1.2	50%	0.9	32%
PSL	1.0	31%	7.3	68%	1.8	54%	2.4	50%	2.4	64%	2.5	53%	0.2	6%	0.9	21%	1.5	59%	1.6	43%	0.0	0%	0.2	4%	1.0	39%	0.5	43%	0.3	28%
RAL	1.5	38%	4.7	43%	2.3	46%	1.5	26%	2.2	33%	2.0	23%	1.3	44%	0.8	29%	1.5	27%	4.5	59%	0.0	0%	2.1	45%	1.9	35%	2.1	51%	1.1	29%
MME	16.5	13%	19.1	15%	17.1	13%	8.0	16%	16.4	9%	16.4	10%	16.2	12%	16.8	10%	1.5	11%	6.7	20%	0.0	0%	1.0	8%	1.4	14%	0.8	13%	0.5	9%
PSBL1	1.0	20%	6.8	60%	2.0	54%	2.5	50%	2.3	51%	2.5	48%	0.3	7%	1.0	17%	1.5	48%	1.7	43%	0.0	0%	0.2	0%	1.0	40%	0.6	39%	0.3	32%
CVL	1.0	23%	3.2	56%	1.6	44%	1.4	30%	1.6	37%	1.7	21%	1.0	7%	1.0	13%	1.0	47%	1.8	44%	0.0	0%	0.5	17%	1.0	38%	0.6	35%	0.3	27%
RML	10.5	26%	7.4	18%	5.9	24%	1.5	15%	8.3	10%	6.9	8%	7.4	17%	7.2	11%	1.5	23%	2.4	20%	0.0	0%	1.2	15%	2.1	18%	1.7	21%	0.9	15%
L3p	1.6	17%	3.1	15%	4.1	22%	1.8	11%	2.2	19%	2.4	16%	3.5	23%	3.2	21%	1.5	16%	2.3	31%	0.0	0%	1.0	20%	1.7	15%	3.1	23%	0.4	13%
HPL	1.3	18%	4.1	27%	4.0	43%	1.2	14%	2.6	19%	2.6	18%	0.9	17%	0.2	0%	1.0	16%	1.0	13%	0.0	0%	0.4	4%	1.3	10%	0.6	7%	0.4	4%
L10	0.7	7%	3.0	27%	1.3	18%	1.4	15%	1.0	26%	1.6	7%	0.3	0%	0.9	8%	1.3	21%	5.1	25%	0.0	0%	0.4	0%	1.8	14%	0.7	15%	0.5	9%
CAL	0.3	16%	0.0	9%	0.5	16%	0.5	8%	0.9	9%	0.7	12%	1.0	12%	1.2	13%	0.8	16%	0.8	9%	0.0	0%	0.7	4%	0.5	11%	0.4	7%	0.3	10%
HLE	0.8	24%	1.0	23%	0.6	13%	0.7	11%	1.2	12%	0.6	13%	0.8	11%	0.3	0%	0.5	16%	0.3	7%	0.0	0%	0.2	0%	0.4	8%	0.1	11%	0.2	7%
MJL	1.2	12%	1.2	8%	0.6	4%	0.3	3%	0.8	3%	0.5	0%	1.5	19%	1.0	6%	0.5	4%	0.8	6%	0.0	0%	1.2	9%	0.9	0%	0.8	5%	0.5	4%
BTE	0.1	9%	0.2	5%	0.1	5%	0.2	5%	0.3	3%	0.2	0%	0.3	0%	0.2	0%	0.2	0%	0.4	7%	0.0	0%	0.2	4%	0.3	5%	0.4	8%	0.2	6%
CLE	0.2	4%	0.3	6%	0.2	0%	0.3	3%	0.4	0%	0.2	4%	0.2	0%	0.1	3%	0.2	5%	0.2	8%	0.0	0%	0.1	0%	0.2	0%	0.1	2%	0.1	13%
CCL	0.1	0%	0.2	0%	0.2	0%	0.2	5%	0.3	0%	0.0	0%	0.2	0%	0.2	0%	0.1	3%	0.3	0%	0.0	0%	0.1	6%	0.3	6%	0.2	16%	0.1	0%
PCL	0.2	4%	0.6	6%	0.5	2%	0.6	2%	0.7	4%	0.6	2%	0.0	3%	0.0	3%	0.3	3%	0.3	0%	0.0	0%	0.0	0%	0.2	3%	0.3	0%	0.0	0%
TBE	0.2	5%	0.4	10%	0.2	5%	0.3	7%	0.2	5%	0.0	7%	0.0	0%	0.0	0%	0.0	0%	0.0	0%	0.0	0%	0.0	0%	0.0	0%	0.0	0%	0.0	0%
L5	0.0	0%	0.5	7%	0.2	4%	0.3	6%	0.4	1%	0.4	0%	0.0	0%	0.1	1%	0.2	3%	0.1	0%	0.0	0%	0.0	0%	0.2	0%	0.2	4%	0.1	0%

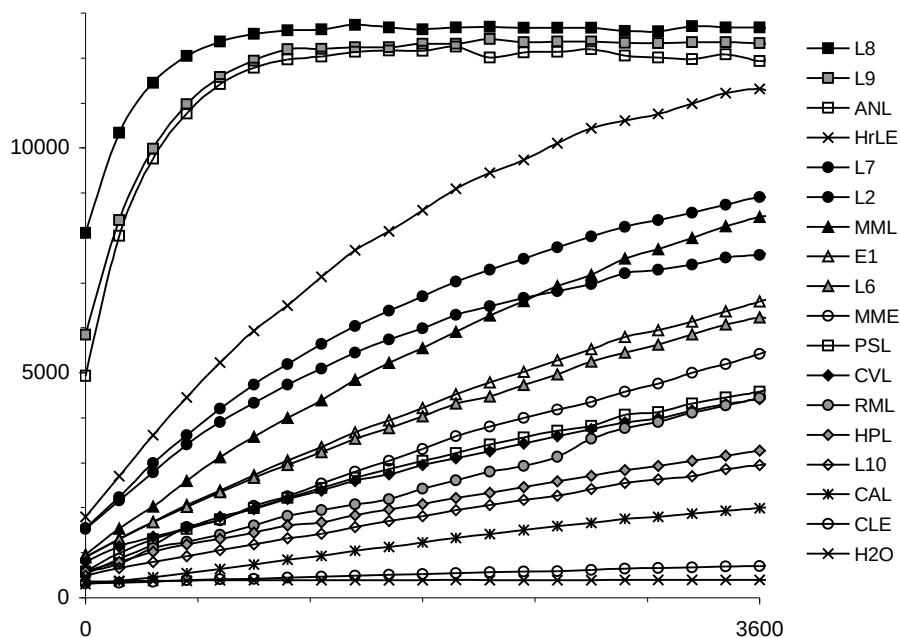


Figure S1. Fluorescence assay of selected lipases and esterases under cocktails conditions with **1a-15a**. Conditions: 30 μ M cocktail (2 μ M of each of **1a-15a**) in aq. PBS pH 7.4 with 30% v/v DMSO, 50 μ g/ml enzyme, 25°C. See also legend of Figure 1.

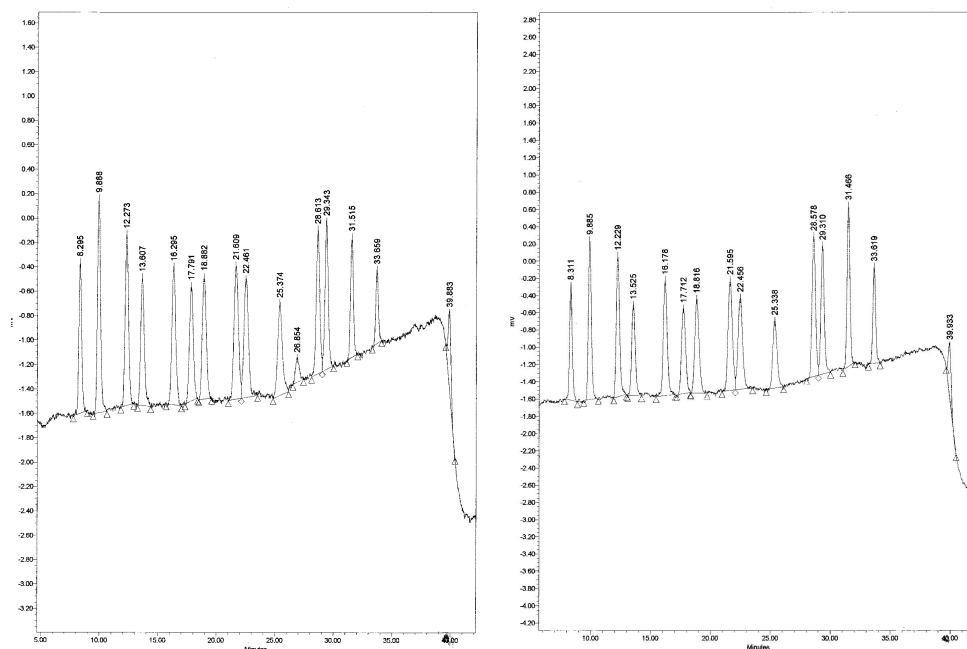
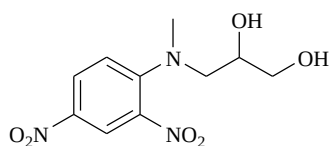
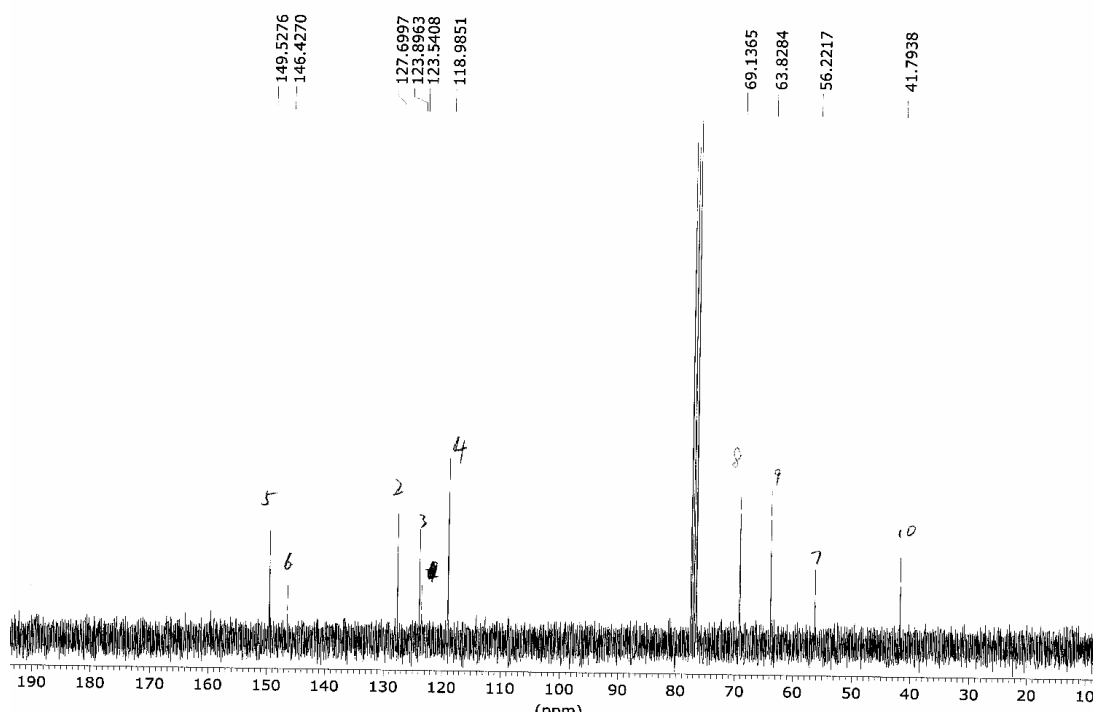
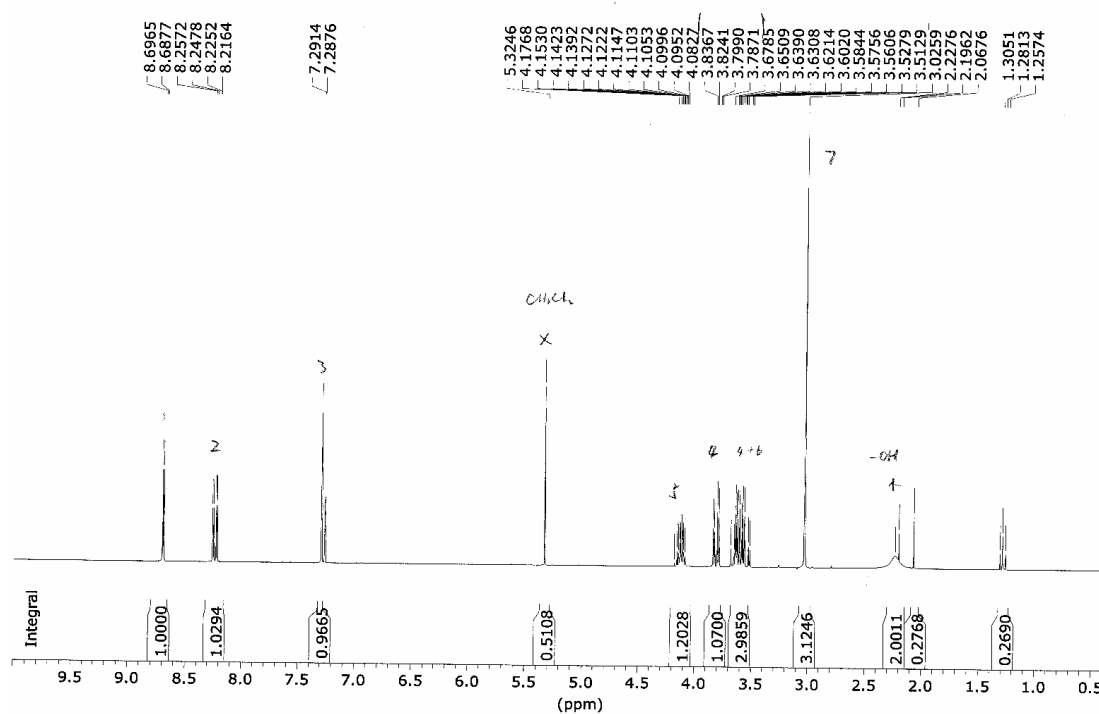


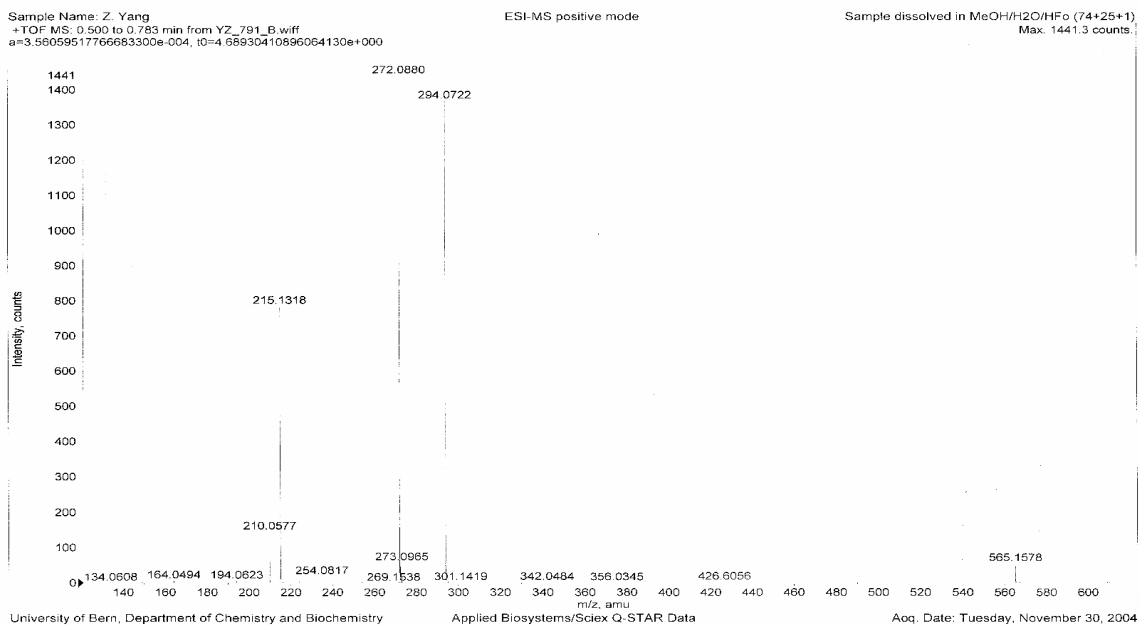
Figure S2. RP-HPLC analysis of reaction products **1b-15b** after enzyme reactions. Left trace: reaction with horse liver esterase (HrLE), which converts all 15 substrates **1a-15a** (products **1b-15b** are numbered by elution order). Right trace: reaction with Chiralzyme L9 (note that **11b** is not formed). Assay conditions: 50 μ M cocktail **1a-15a** in aq. PBS with 30% v/v DMSO, 25°C, 2h. HPLC conditions see experimental section.

1b.

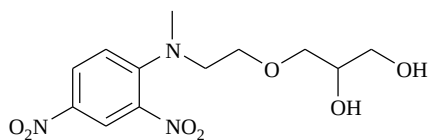
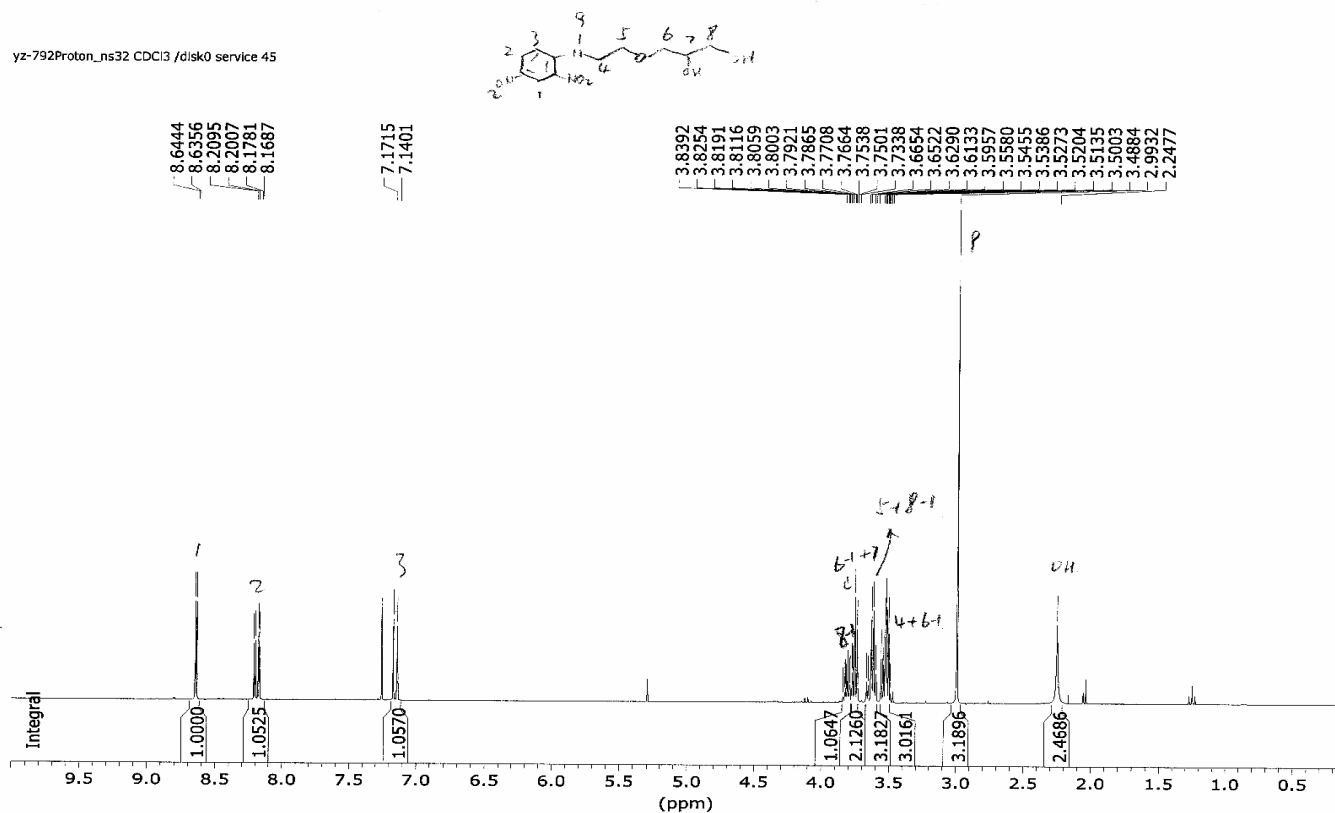
C₁₀H₁₃N₃O₆ Mol. Wt.: 271.23

yz-791Proton_ns32 CDCl3 /disk0 service 44

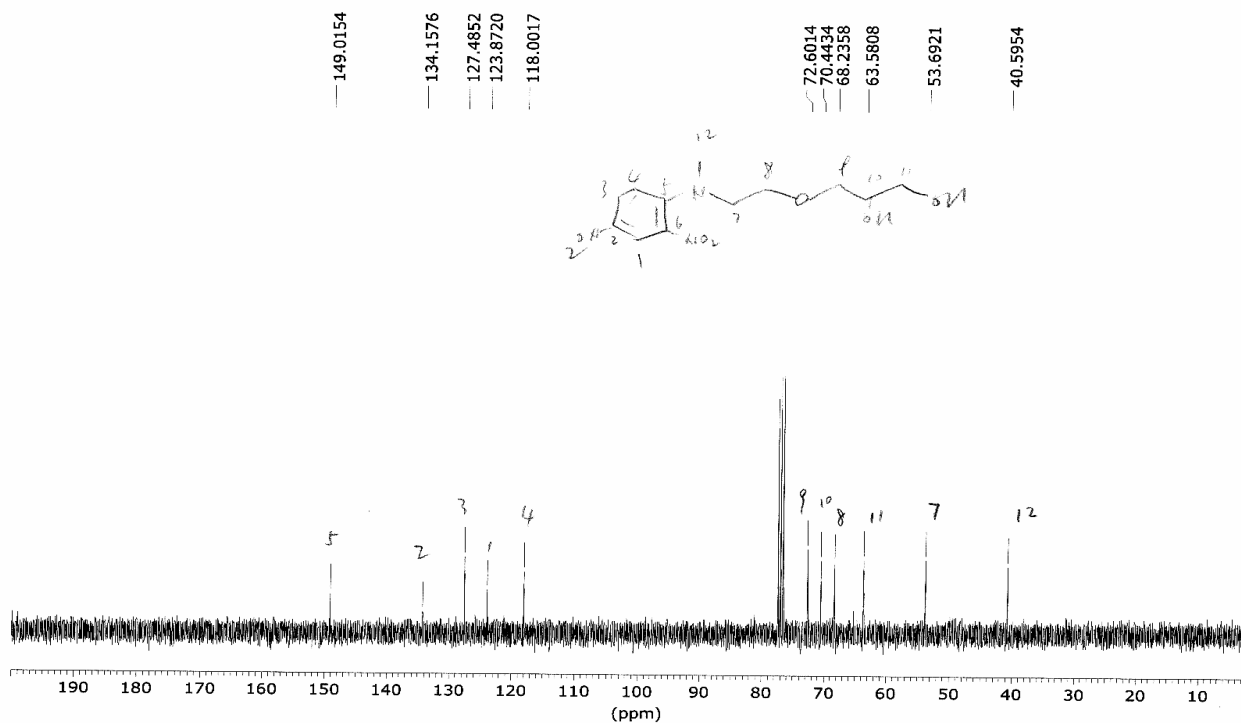




2b.


 $C_{12}H_{17}N_3O_7$ Mol. Wt.: 315.28
yz-792Proton_ns32 CDCl₃ /disk0 service 45

yz-792Carbon_ns256 CDCl3 /disk0 service 45



Sample Name: Z. Yang

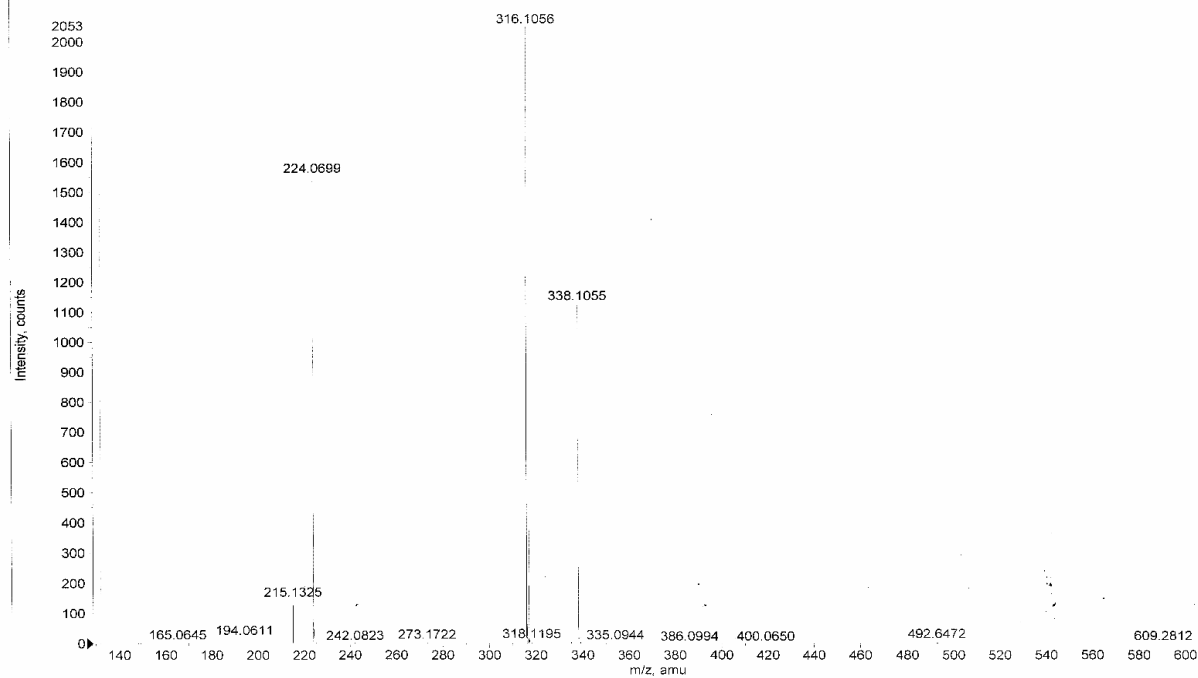
ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+)

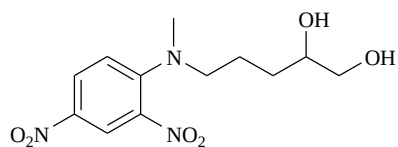
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Max: 2053.4 count

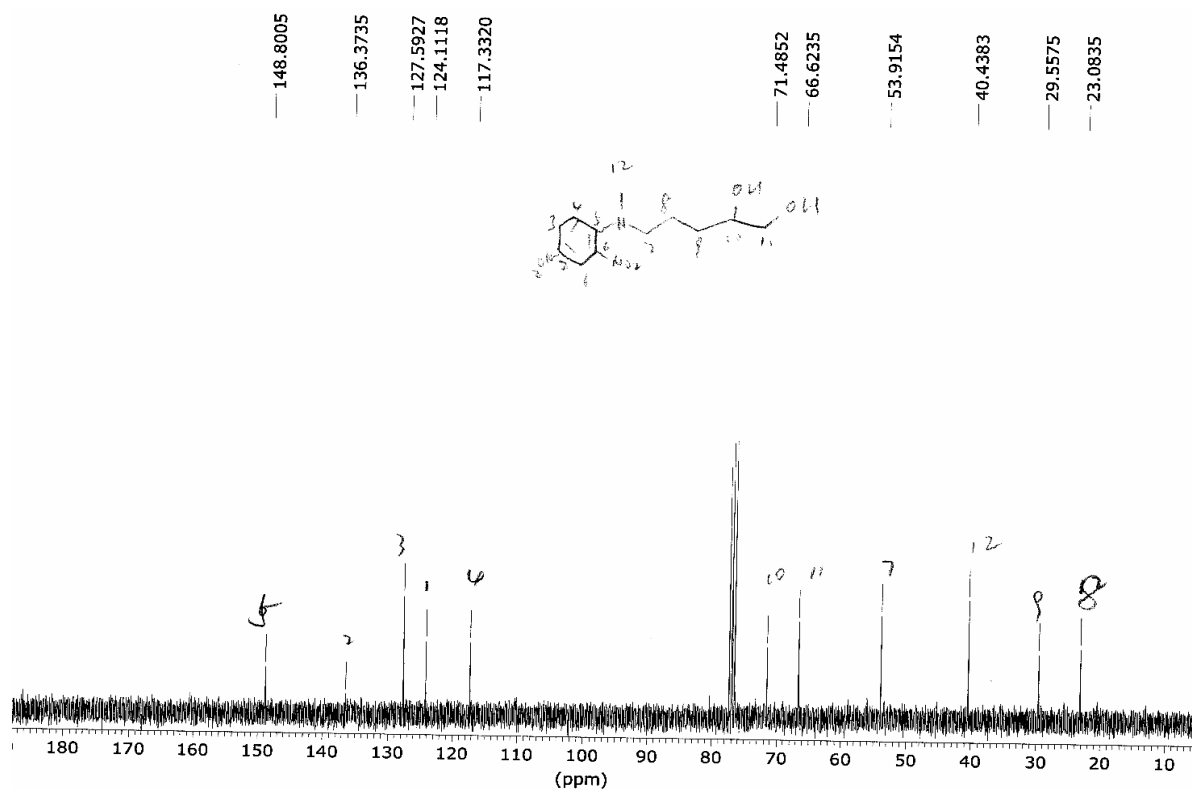
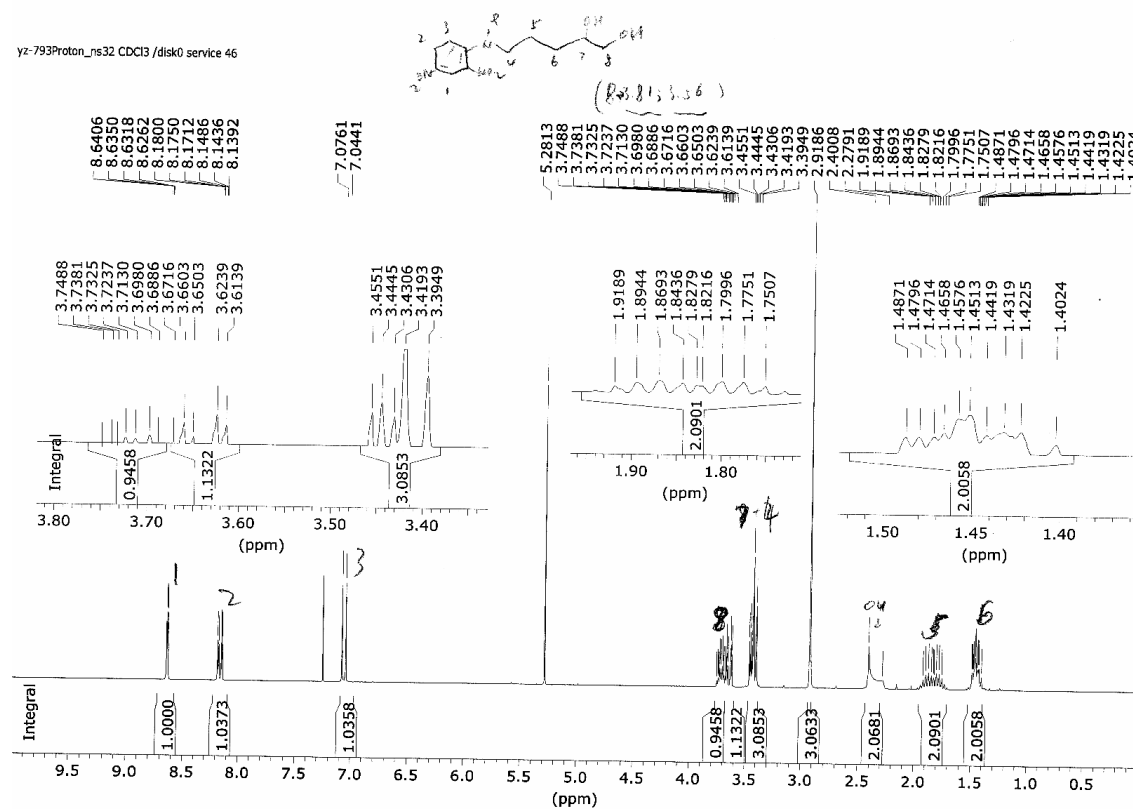
a=3.56058115291520550e-004, t0=4.68930410896064130e+000



3b.

C₁₂H₁₇N₃O₆ Mol. Wt.: 299.28

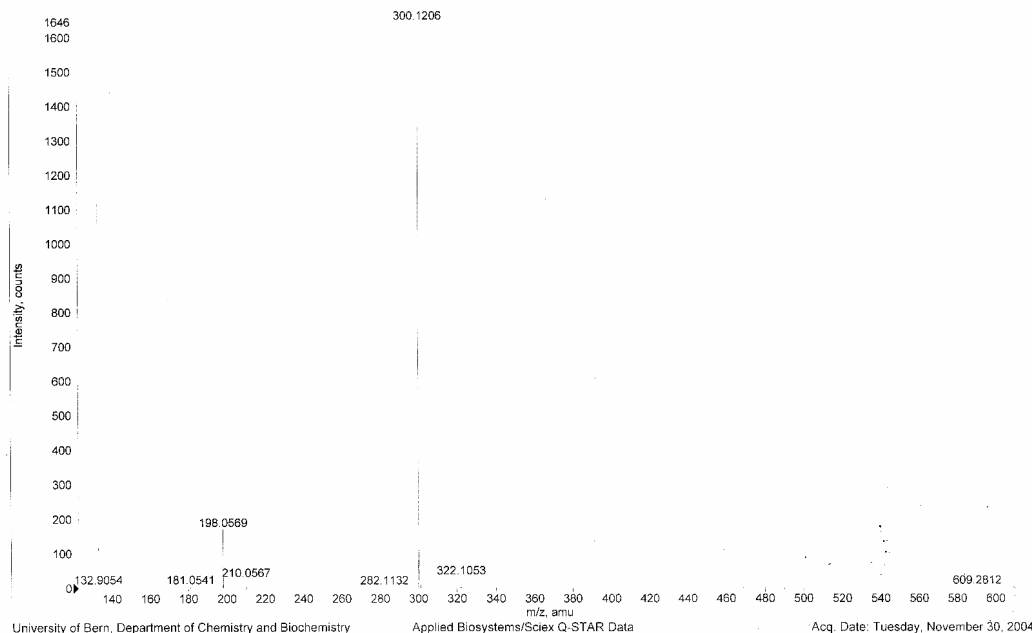
yz-793Proton_ns32 CDC13 /disk0 service 46



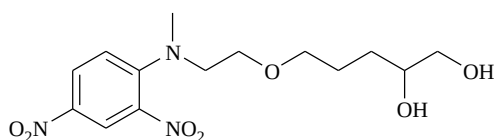
Sample Name: Z. Yang
 +TOF MS: 6.951 to 7.017 min from YZ_793_B.wiff
 a=3.58055751735064200e-004, t0=-4.2973806091670400e+000

ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+1)
 Max. 1645.6 counts.

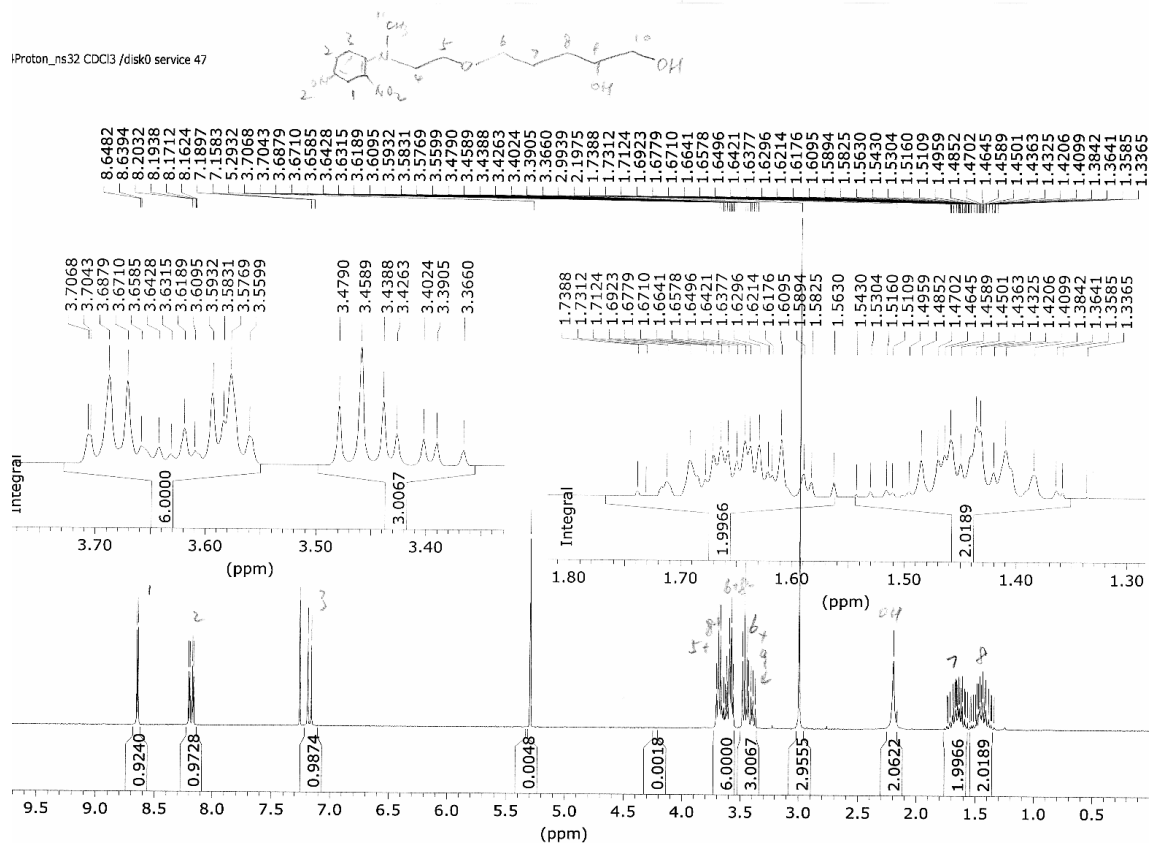


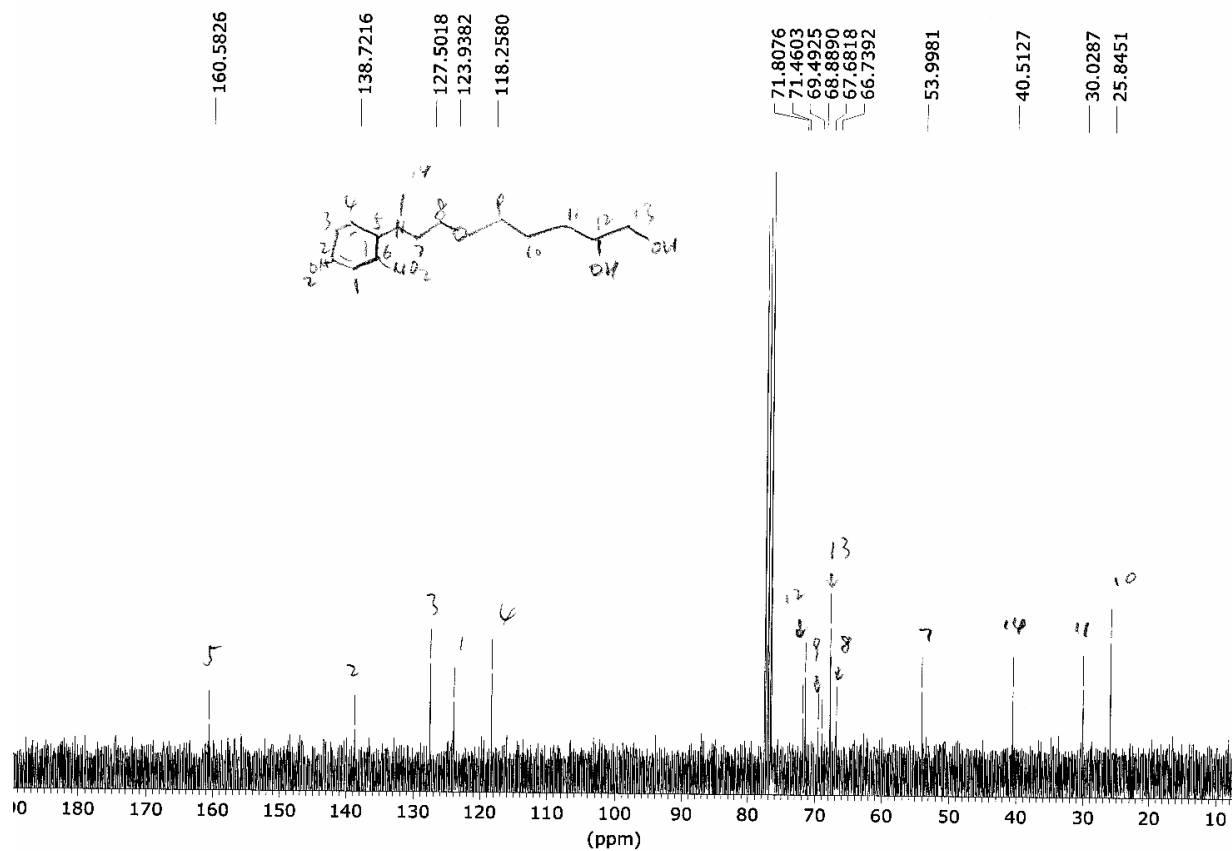
4b.



$C_{14}H_{21}N_3O_7$ Mol. Wt.: 343.33

1Proton_ns32 CDCl3 /disk0 service 47

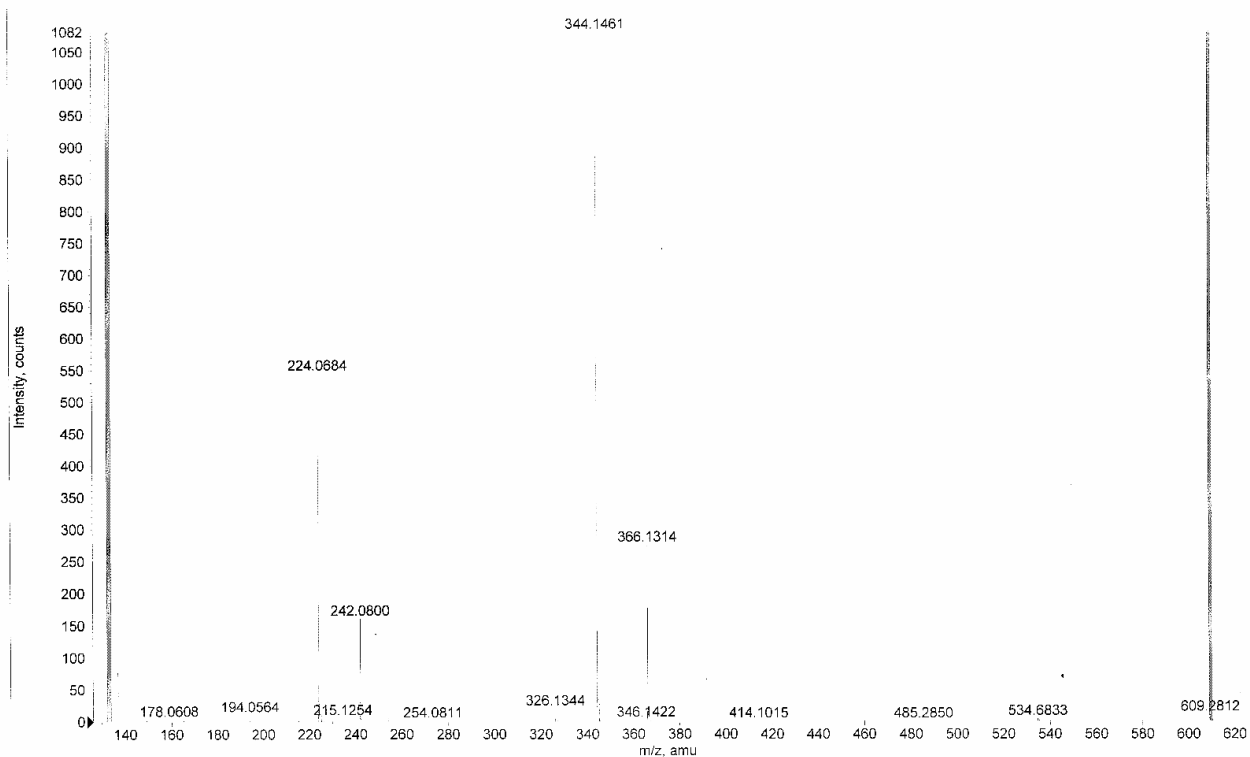




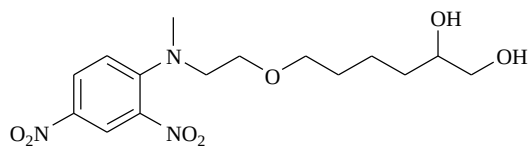
Sample Name: Z. Yang
 +TOF MS: 2.584 to 2.700 min from YZ_794_B.wiff
 a=3.56059627359171270e-004, t0=4.79597350423136960e+000

ESI-MS positive mode

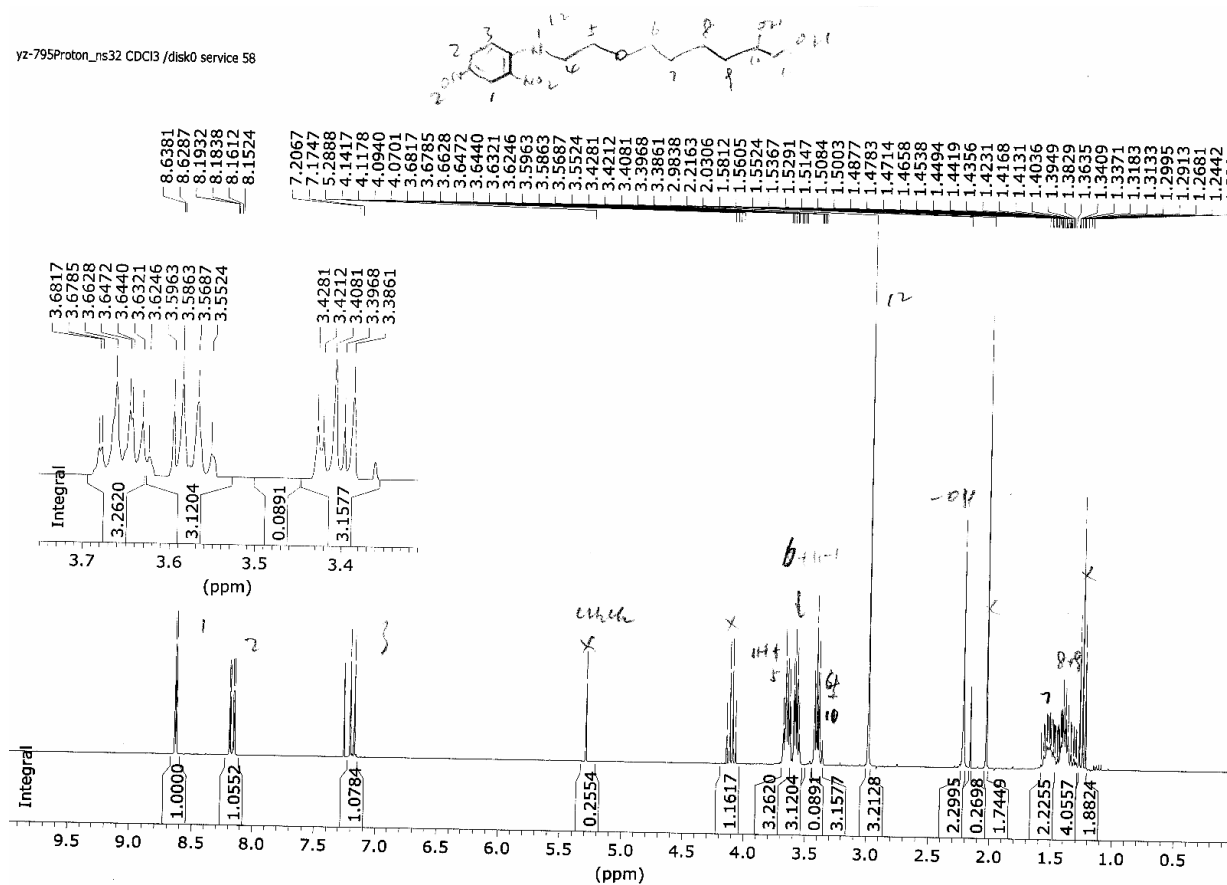
Sample dissolved in MeOH/H₂O/HFo (74+25+1)
 Max. 1081.5 counts.

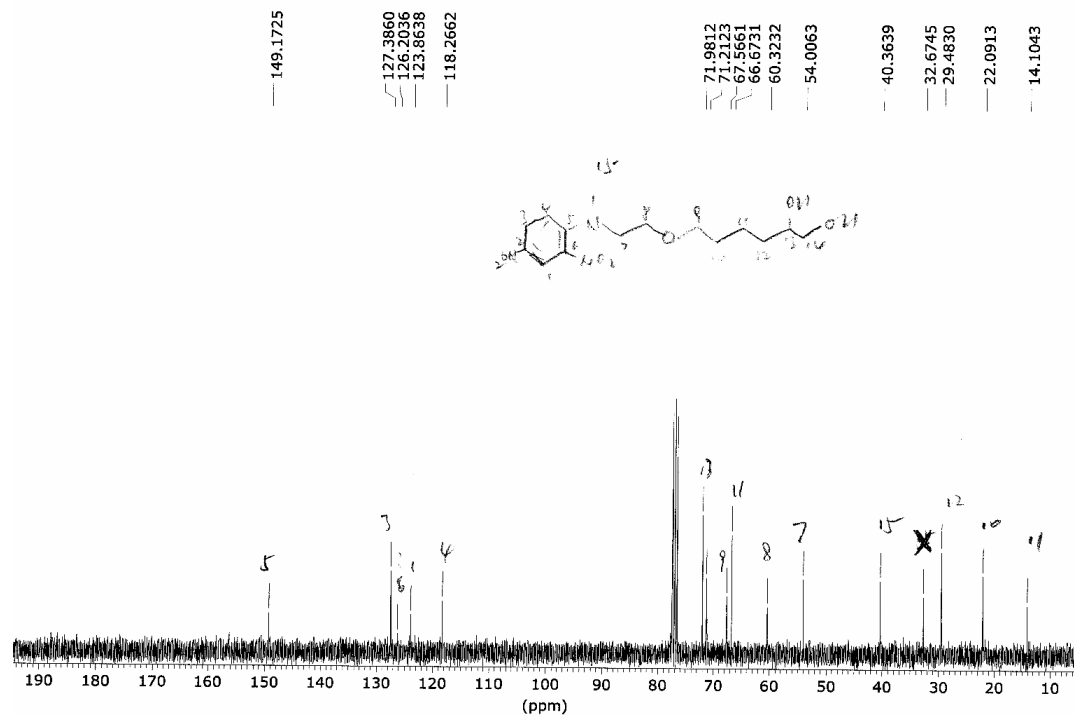


5b.



$C_{15}H_{23}N_3O_7$ Mol. Wt.: 357.36

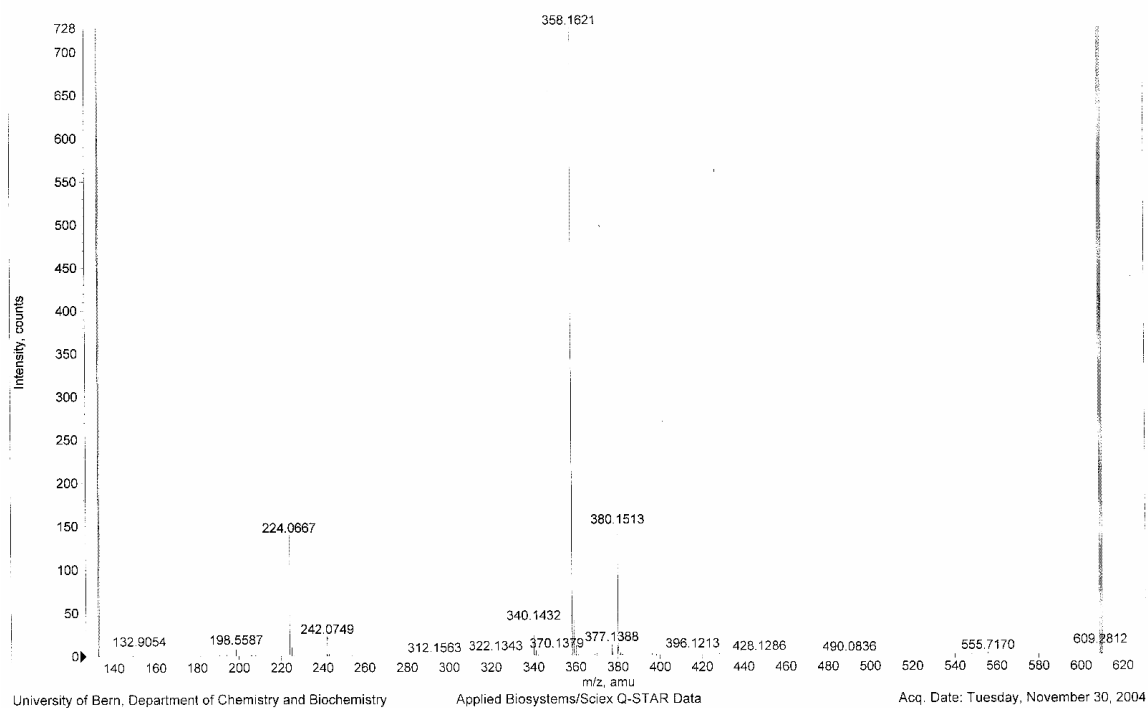




Sample Name: Z. Yang
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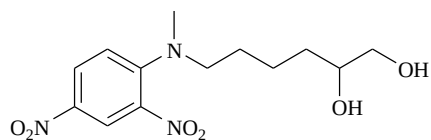
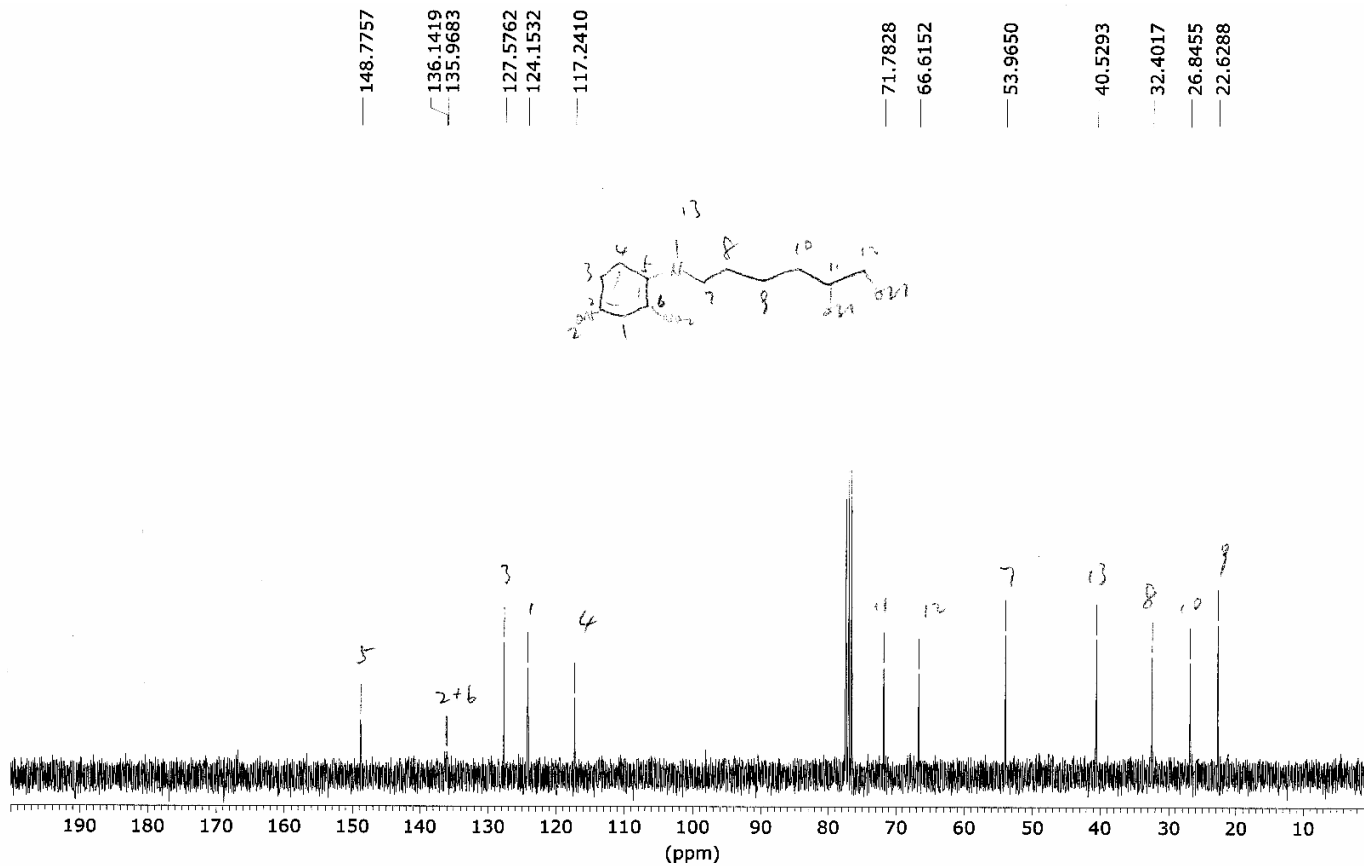
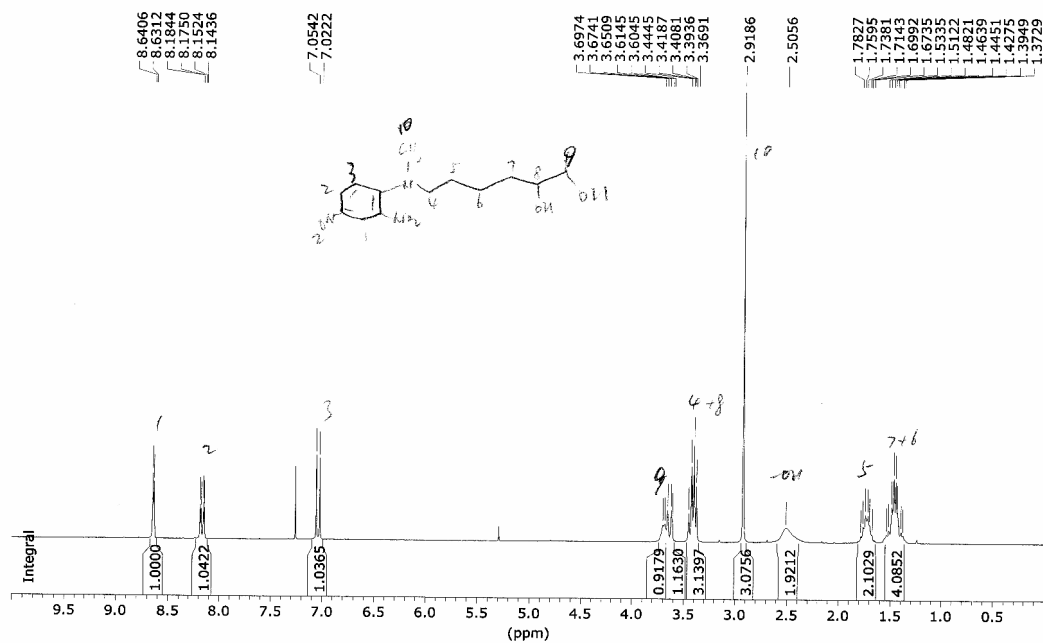
ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74*25+1)
 Max. 728.3 counts.



S13

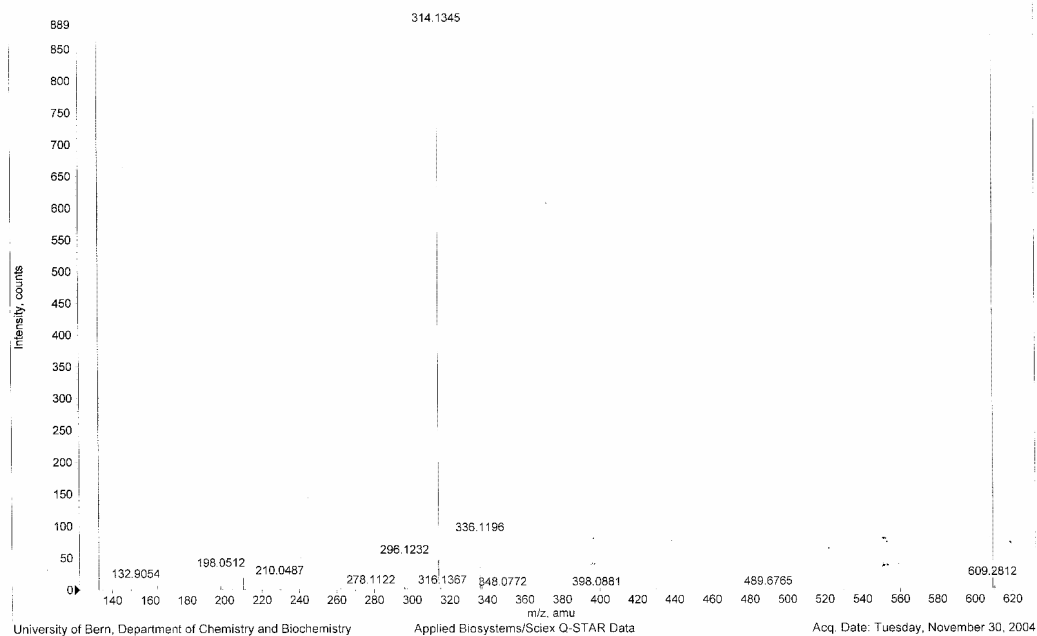
6b.

 $C_{13}H_{19}N_3O_6$ Mol. Wt.: 313.31

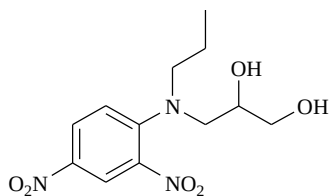
Sample Name: Z. Yang
+TOF MS: 3.834 to 4.000 min from YZ_796_B.wiff
a=3.56061426784864790e-004, f0=5.20320572169657680e+000

ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+1)
Max. 888.9 counts.



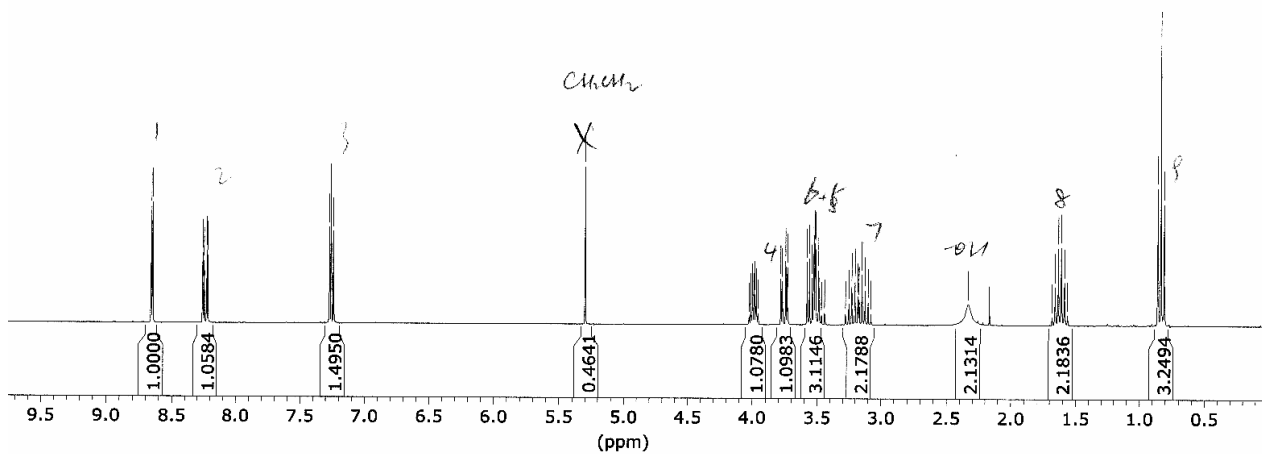
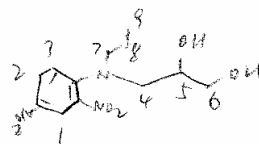
7b.

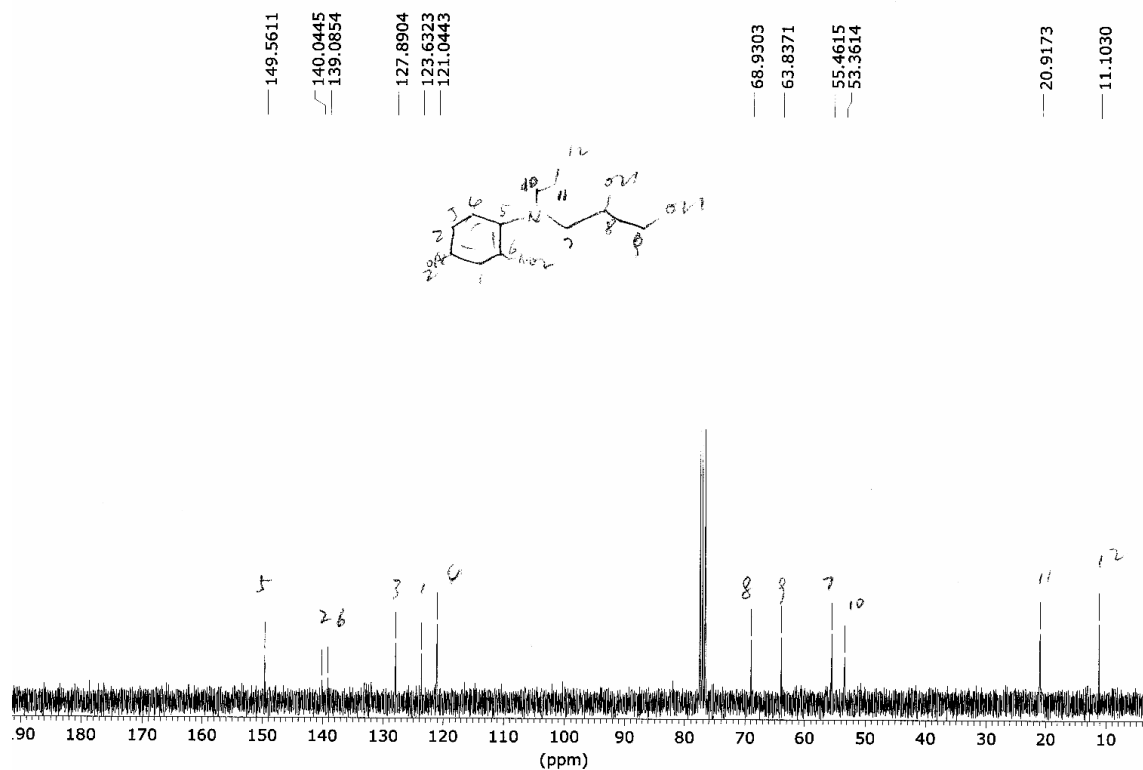

 $C_{12}H_{17}N_3O_6$ Mol. Wt.: 299.28

8.6544
8.6456
8.2541
8.2446
8.2227
8.2139

7.2707
7.2399

5.2964
4.0249
4.0130
4.0093
3.9980
3.9936
3.9860
3.9816
3.9703
3.9666
3.9540
3.7789
3.7670
3.7413
3.7294
3.5744
3.5580
3.5367
3.5242
3.5204
3.5110
3.5091
3.4846
3.4633
3.4363
3.2731
3.2493
3.2261
3.2016
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3.1708
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3.0993
3.0754
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1.6308
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1.5825
1.5586
0.8583
0.8338
0.8093

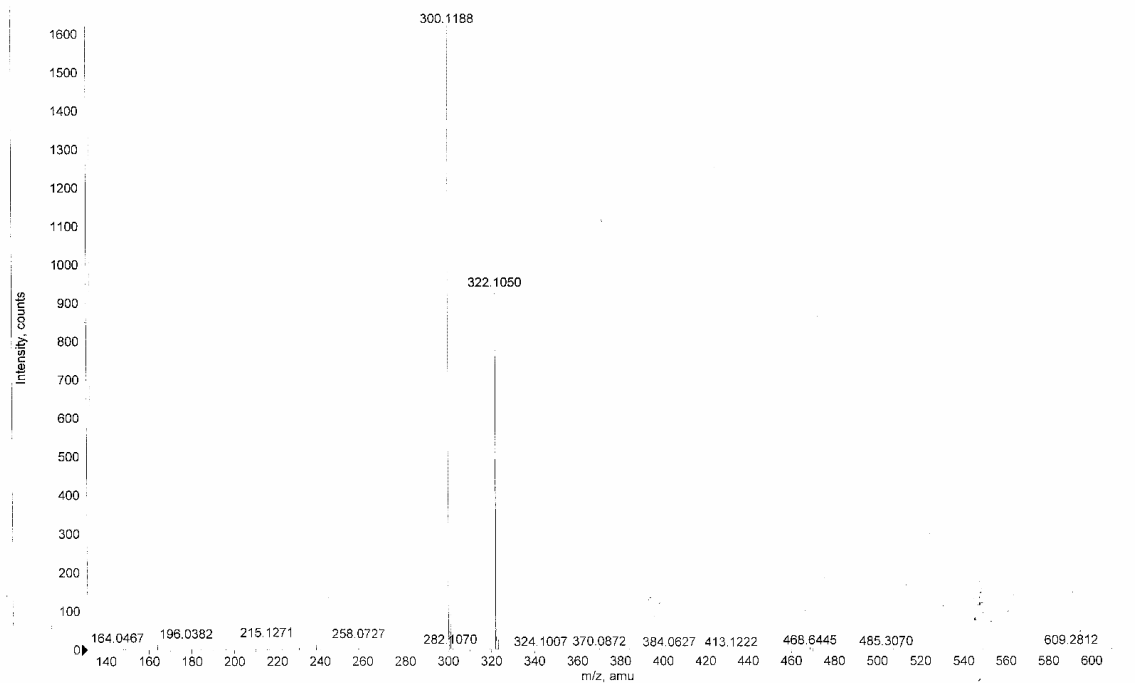




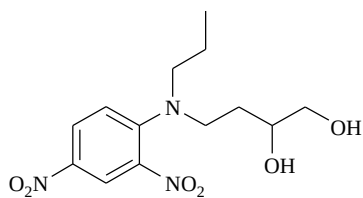
Sample Name: Z. Yang
+TOF MS: 0.250 to 0.467 min from YZ_797_B.wiff
a=3.56058407472479450e-004, t0=4.68930410898084130e+000

ESI-MS positive mode

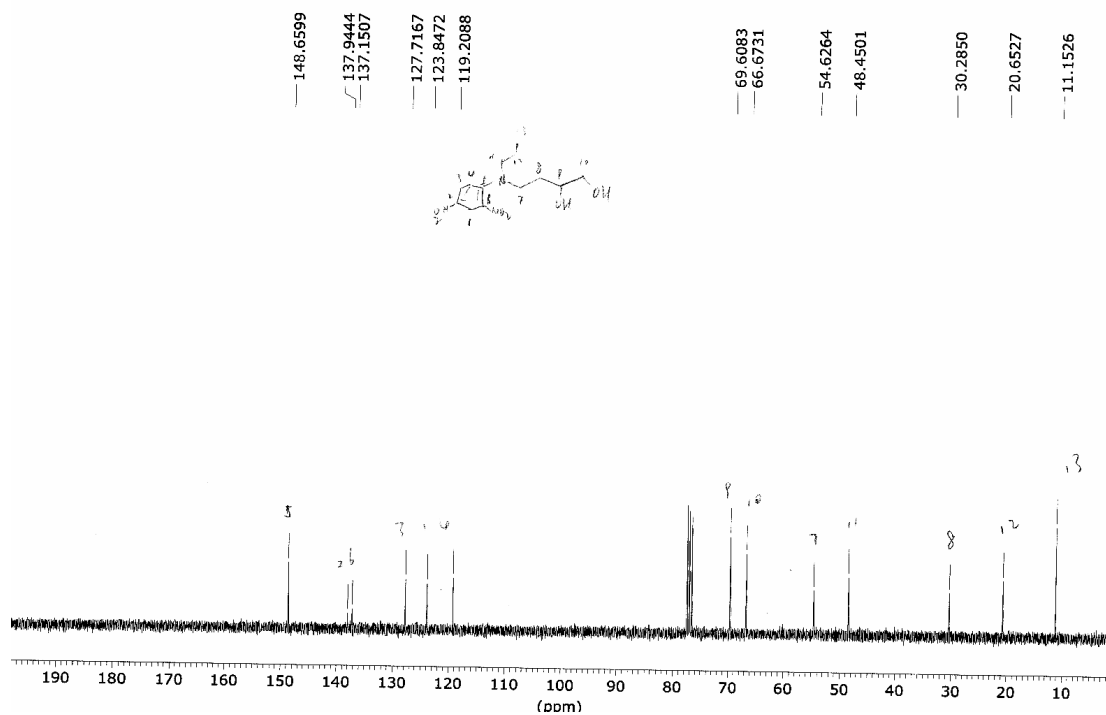
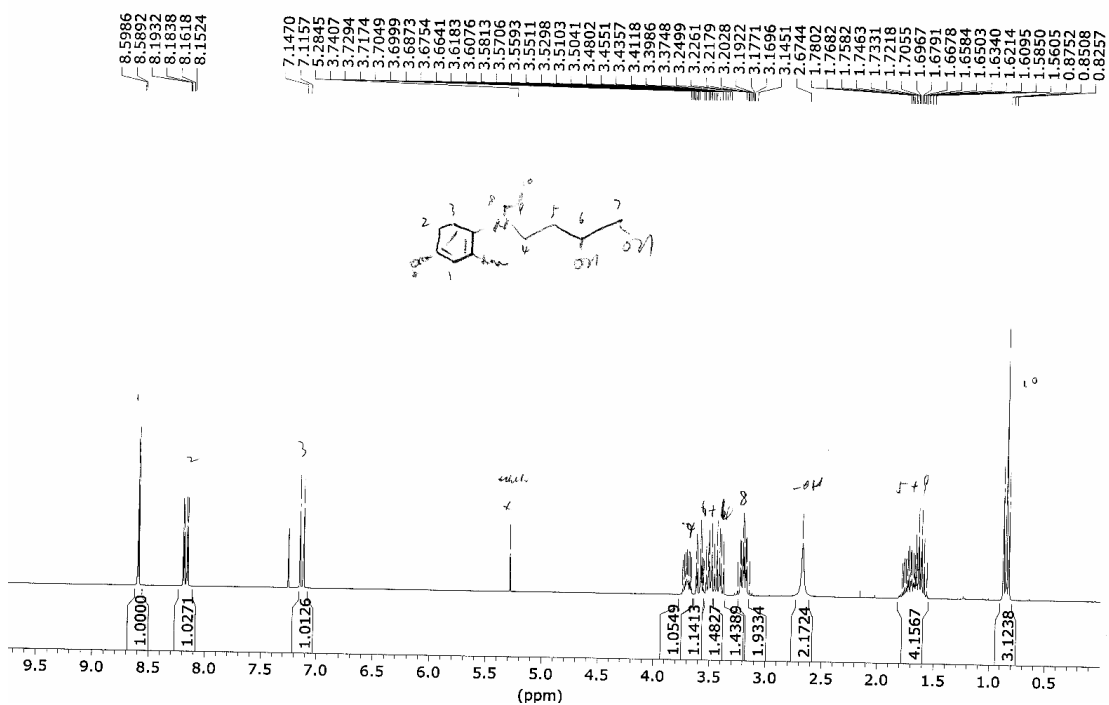
Sample dissolved in MeOH/H₂O/HFo (74+25+1)
Max: 1621.9 counts.



8b.

 $C_{13}H_{19}N_3O_6$

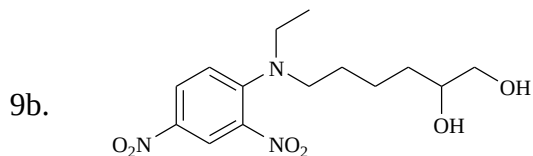
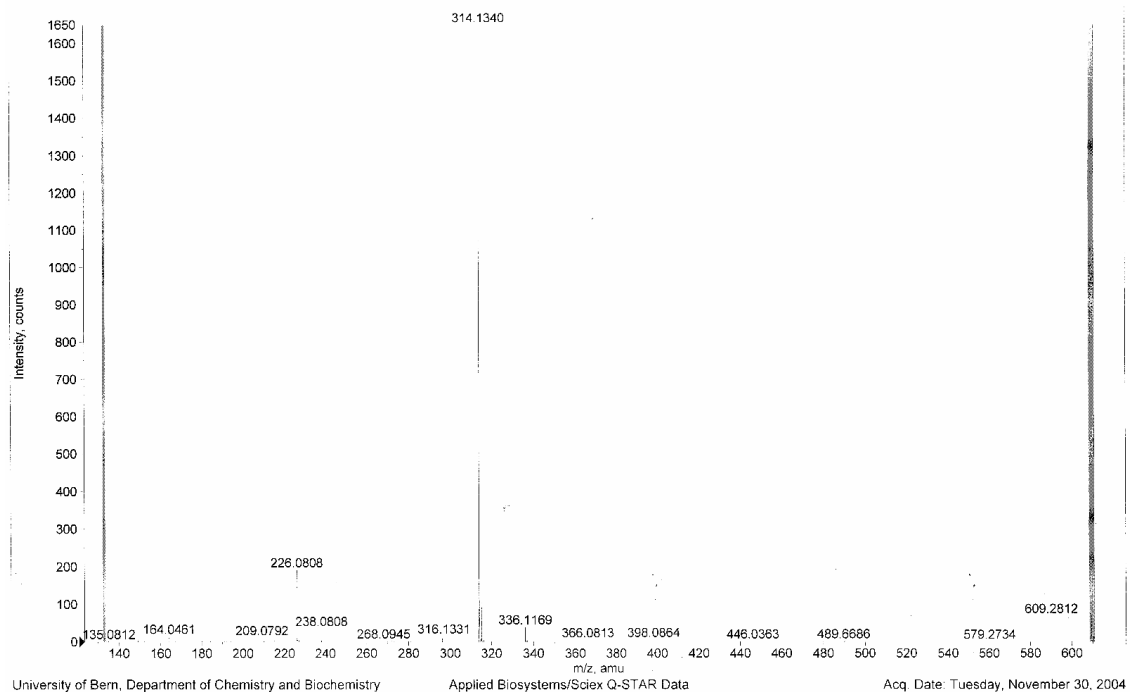
Mol. Wt.: 313.31



Sample Name: Z. Yang
 +TOF MS: 2.467 to 2.667 min from YZ_798_B.wiff
 a=3.56072646223432000e-004, ID=4.81046054991384150e+000

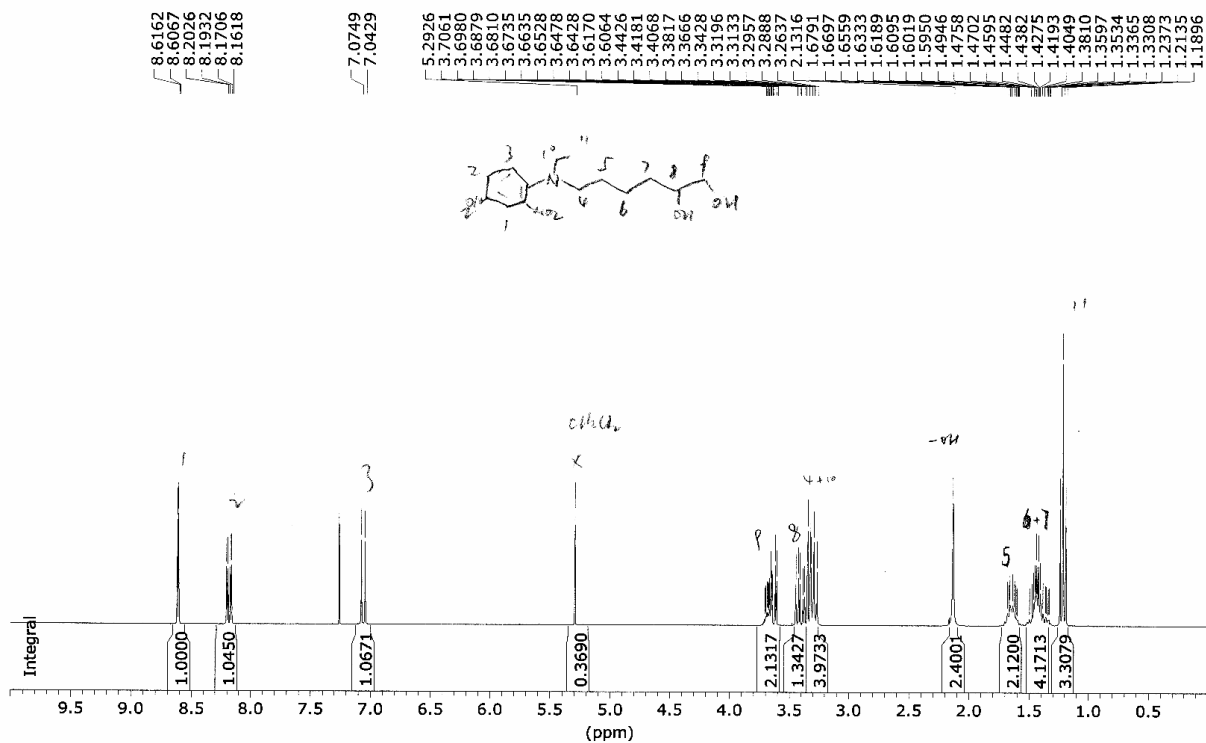
ESI-MS positive mode

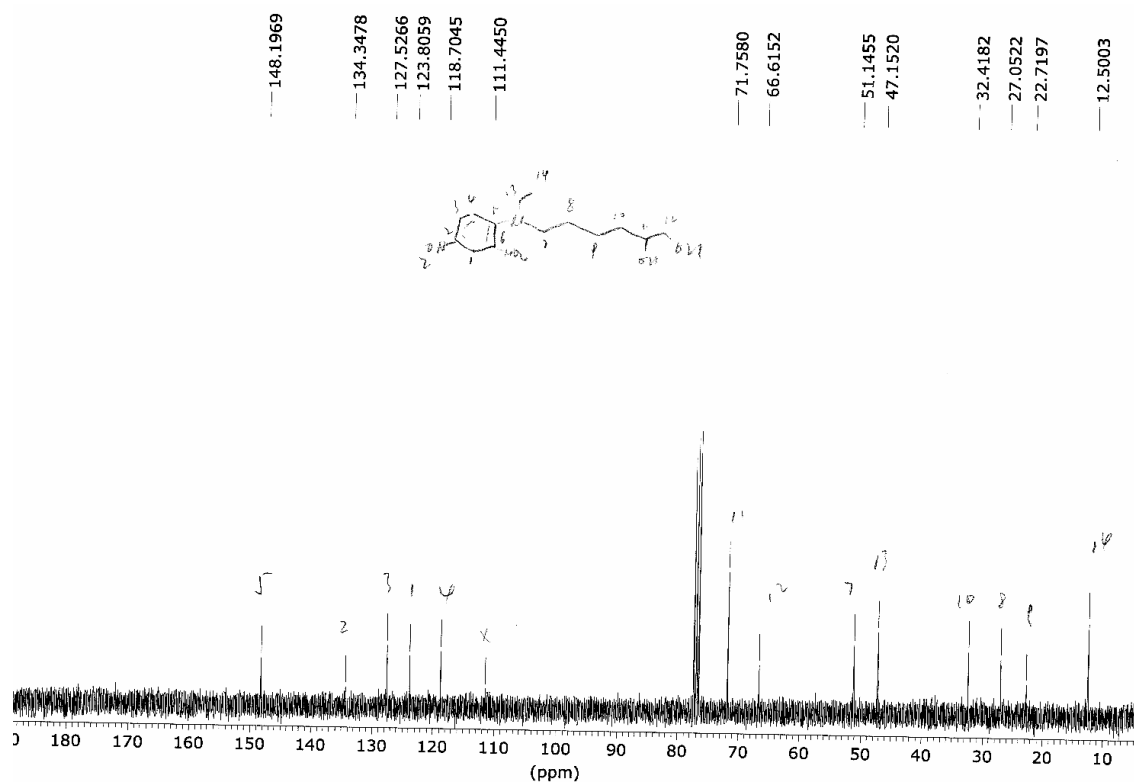
Sample dissolved in MeOH/H₂O/HFo (74+25+1)
 Max. 1650.3 counts.



$C_{14}H_{21}N_3O_6$ Mol. Wt.: 327.33

yz-799Proton_ns32 CDCl₃ /disk0 service 51

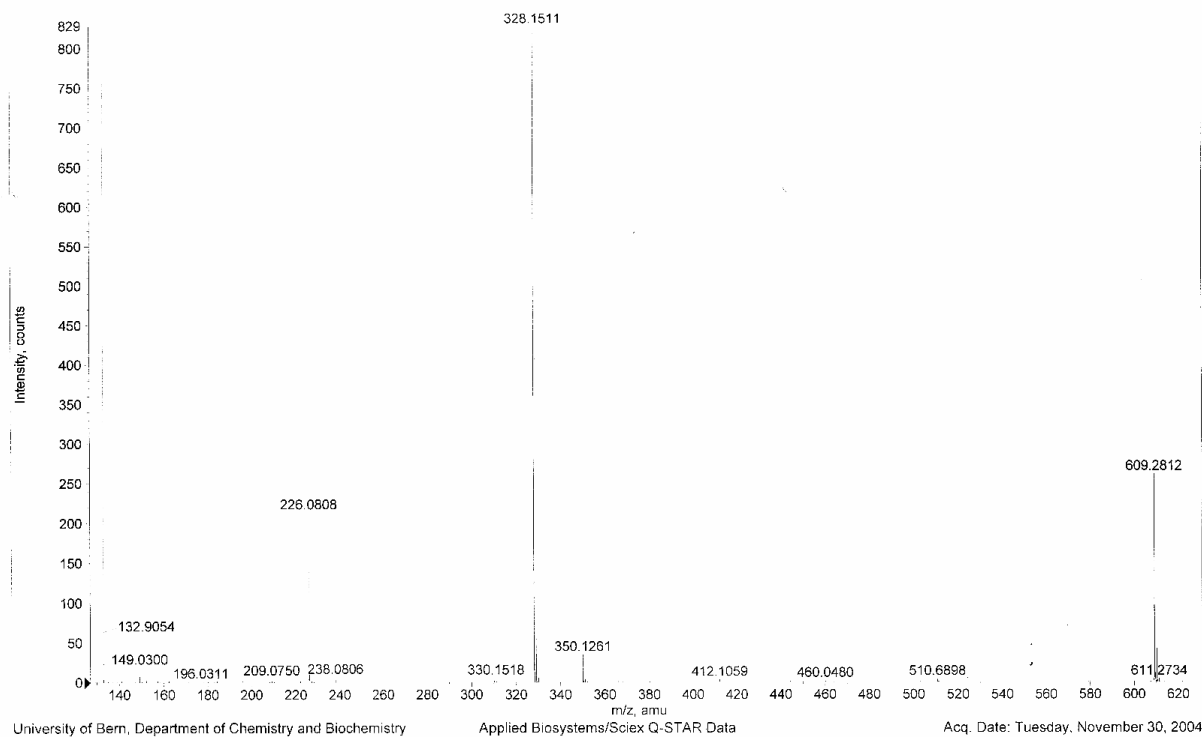




Sample Name: Z. Yang
+TOF MS: 8.452 to 8.502 min from YZ_800_B.wiff
a=3.56071714120299150e-004, t0=4.72570674708185830e+000

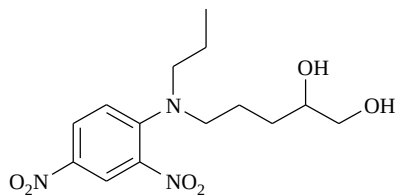
ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+1)
Max. 828.8 counts



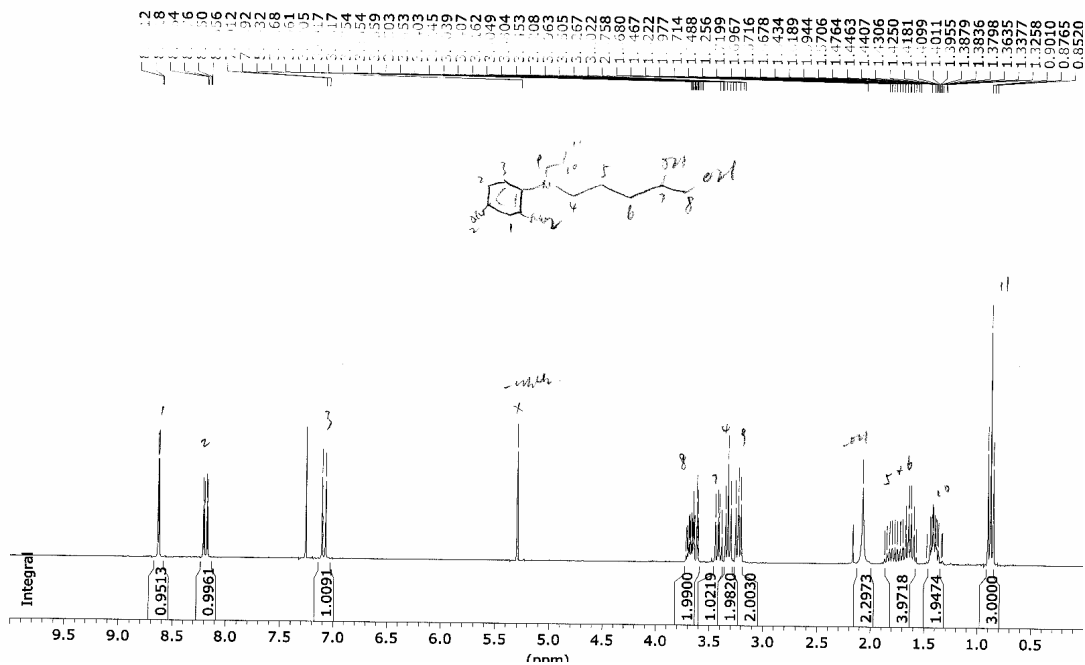
S19

10b.

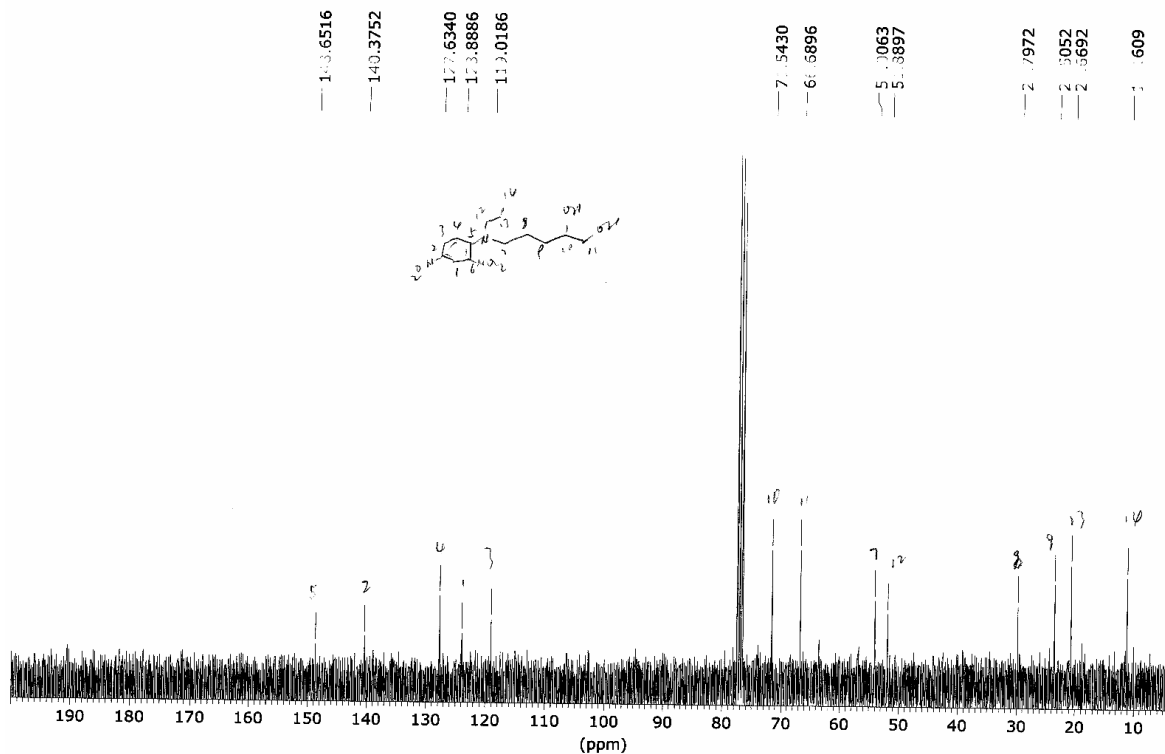


$C_{14}H_{21}N_3O_6$ Mol. Wt.: 327.33

yz-800Proton_ns32 CDCl3 /disk0 service 52



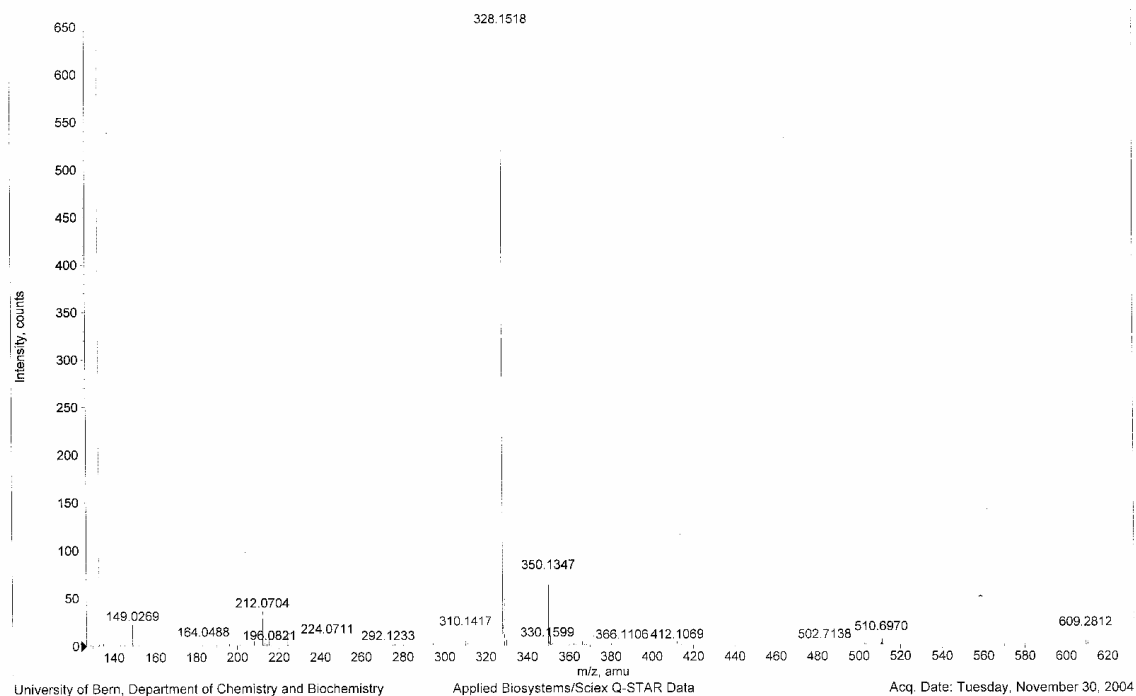
yz-800Carbon_ns256 CDCl3 /disk0 service 52



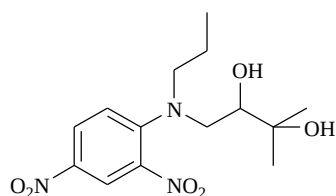
Sample Name: Z. Yang
 *TOF MS: 3.534 to 3.684 min from YZ_799_B.wiff
 a=3.56073134324555380e-004, 10=4.68930410996084130e+000

ESI-MS positive mode

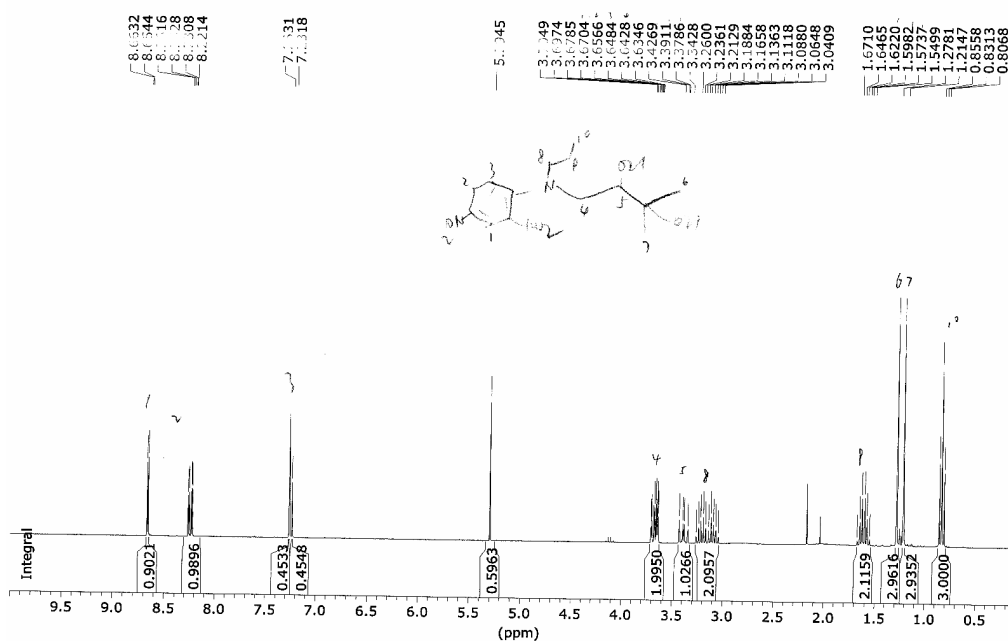
Sample dissolved in MeOH/H₂O/HFO (74+25+1)
 Max: 650.4 counts.



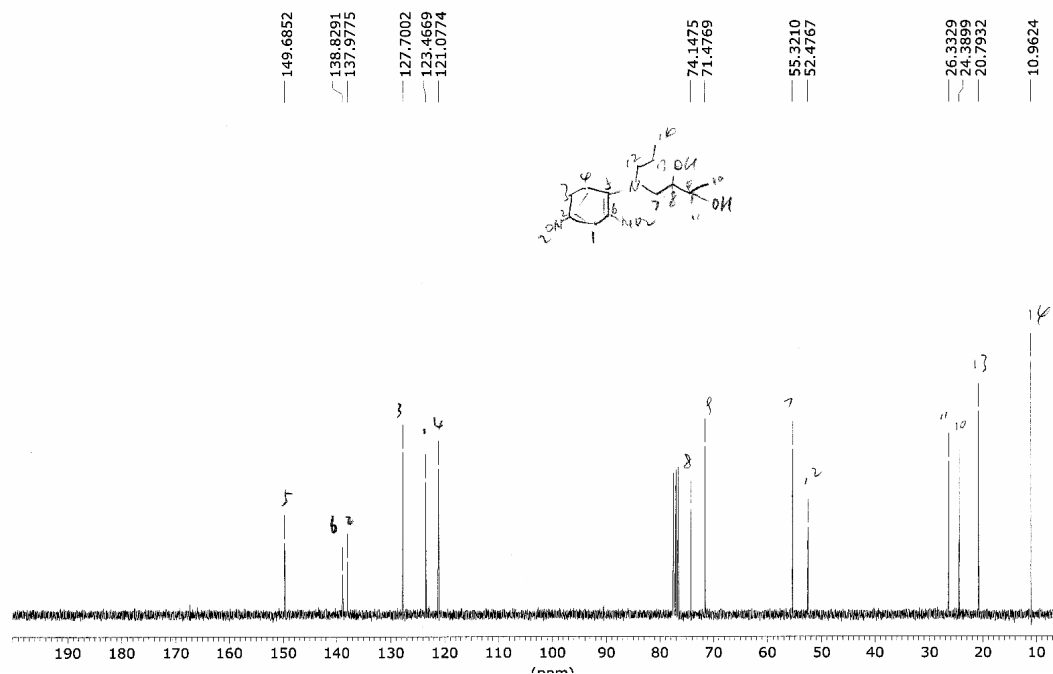
11b.



C₁₄H₂₁N₃O₆ Mol. Wt.: 327.33

yz-801Proton_ns32 CDCl₃ /disk0 service 53

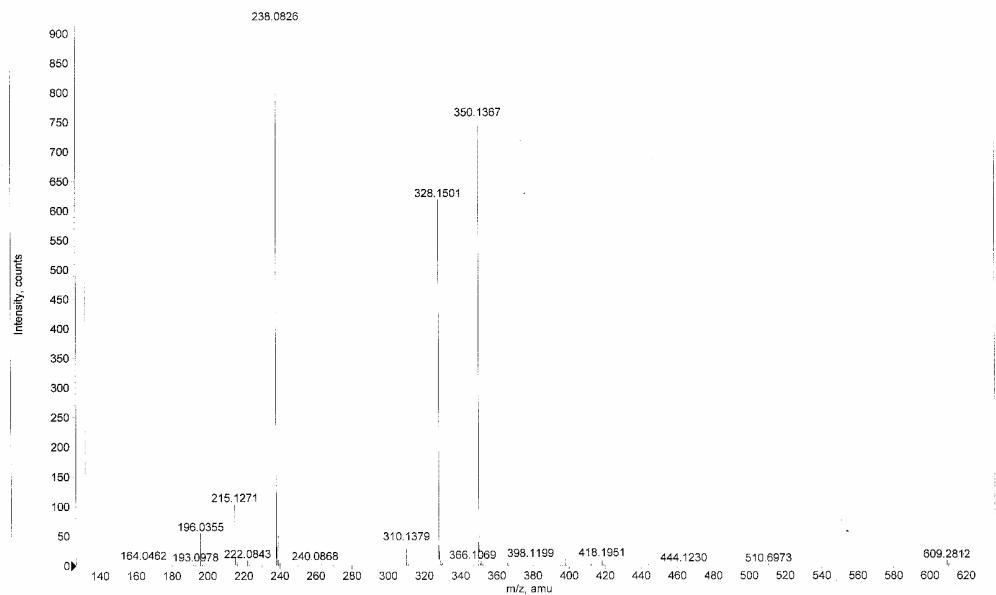
yz-801Carbon_ms512 CDCl3 /disk0 service 102



Sample Name: Z. Yang
 +TOF MS: 1.200 to 1.334 min from YZ_801_B.wiff
 a=3.56072170010476540e-004, t0=4.68930410896064130e+000

ESI-MS positive mode

Sample dissolved in MeOH/H2O/HFo (74+25+1)
 Max. 1315.6 counts.



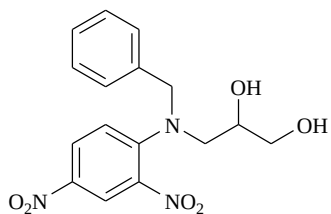
University of Bern, Department of Chemistry and Biochemistry

Applied Biosystems/Sciex Q-STAR Data

Acq. Date: Tuesday, November 30, 2004

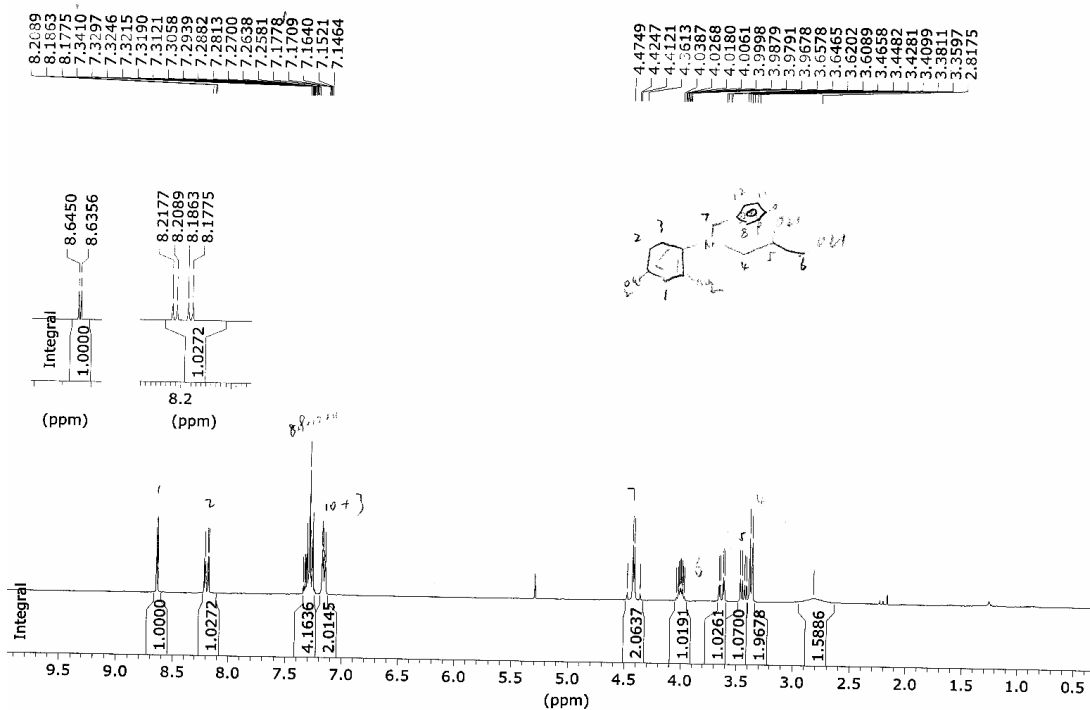
S22

12b.

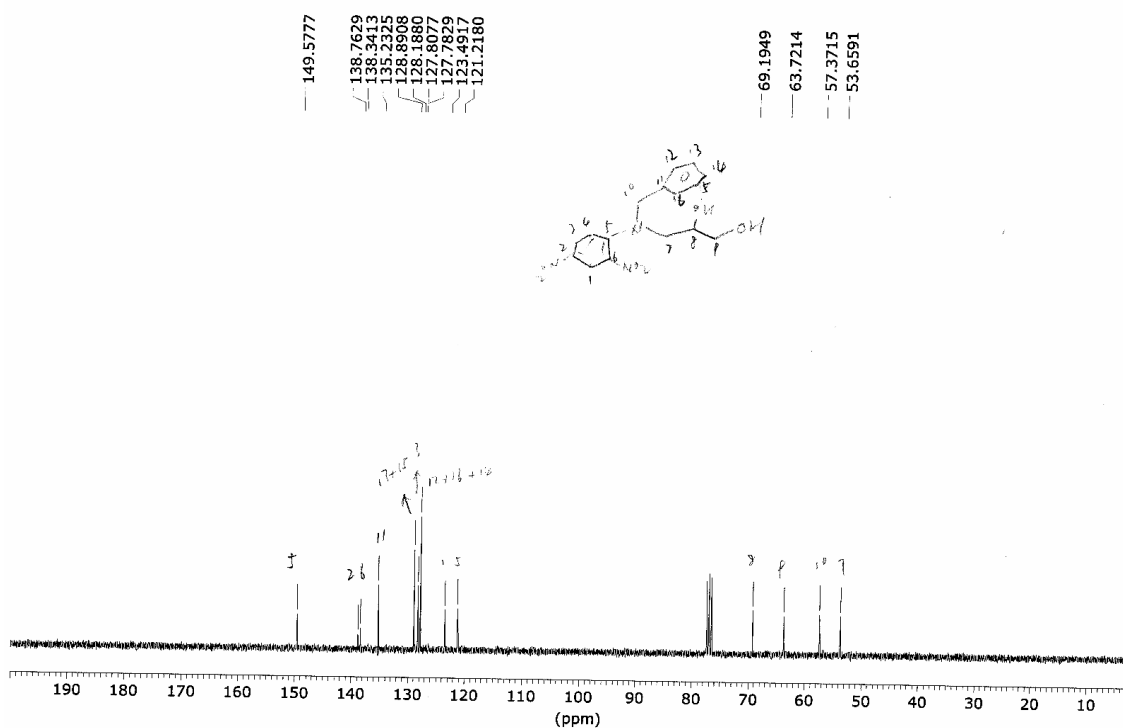


$C_{16}H_{17}N_3O_6$ Mol. Wt.: 347.32

yz-802Proton_ns32 CDCl3 /disk0 service 54



yz-802Carbon_ns255 CDCl3 /disk0 service 54

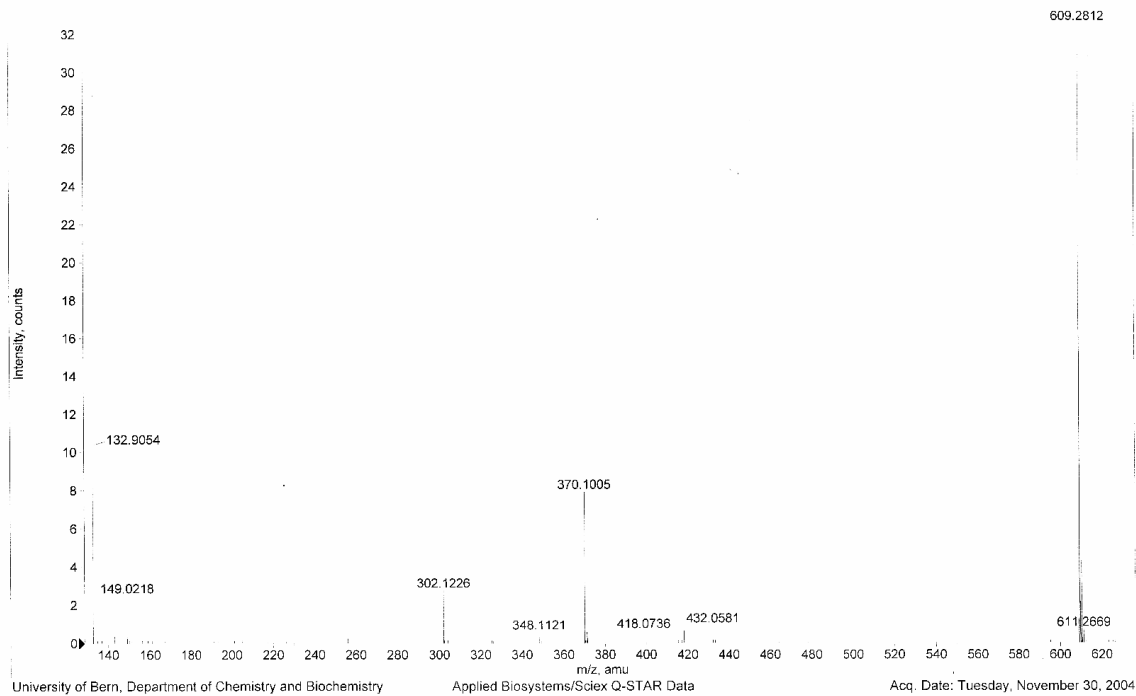


S23

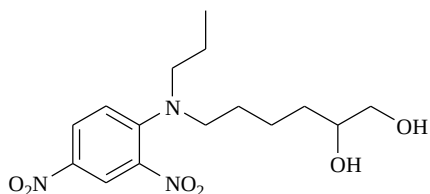
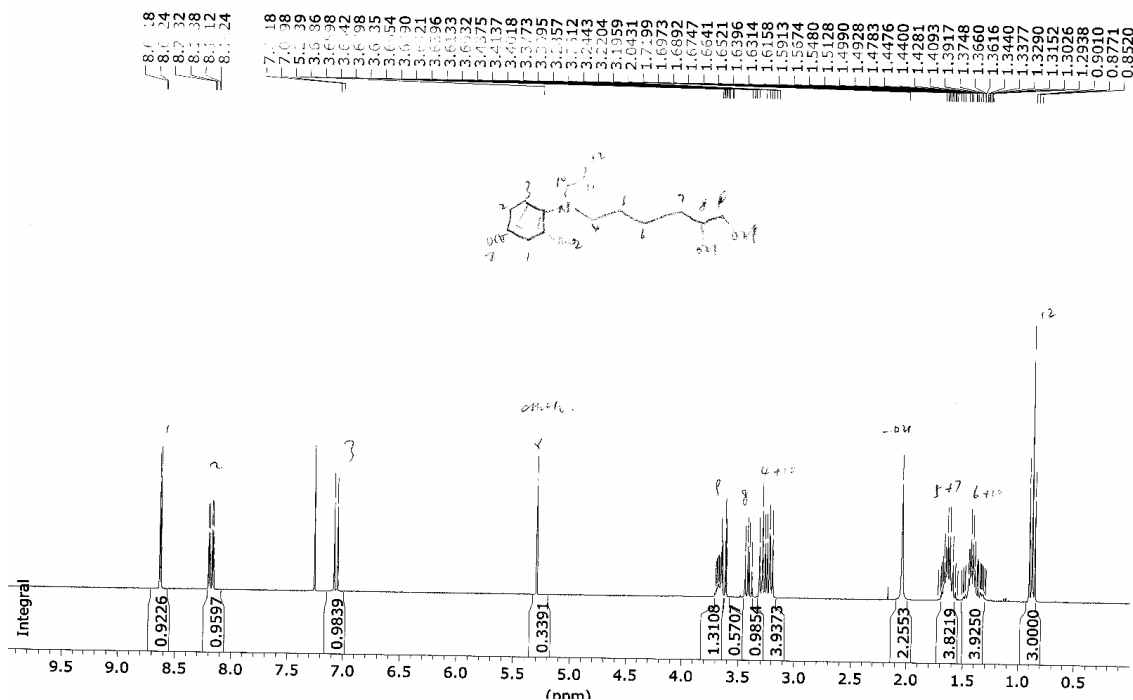
Sample Name: Z. Yang
 *TOF MS 2.267 to 2.484 min from Sample 2 (Z. Yang) of YZ_802_B.wiff
 a=3.56072176639810660e-004, t0=4.71903971608480790e+000

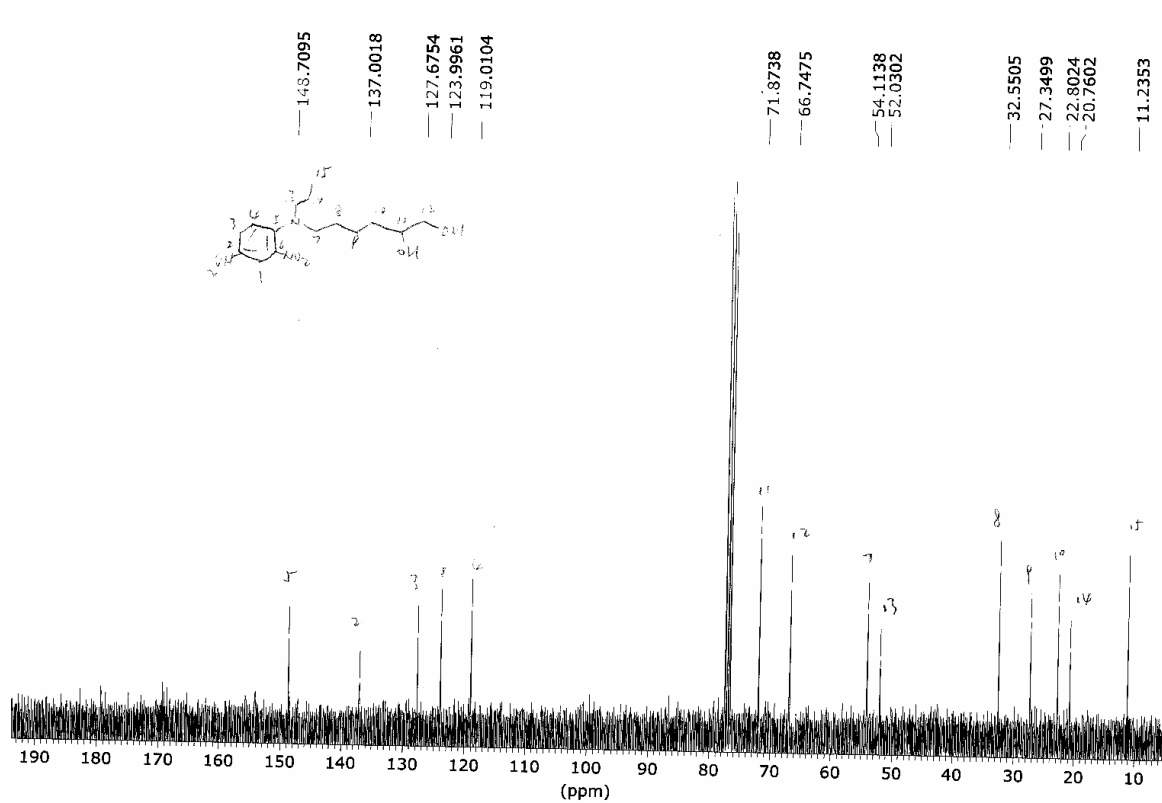
ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+1)
 Max. 32 6 counts.



13b.

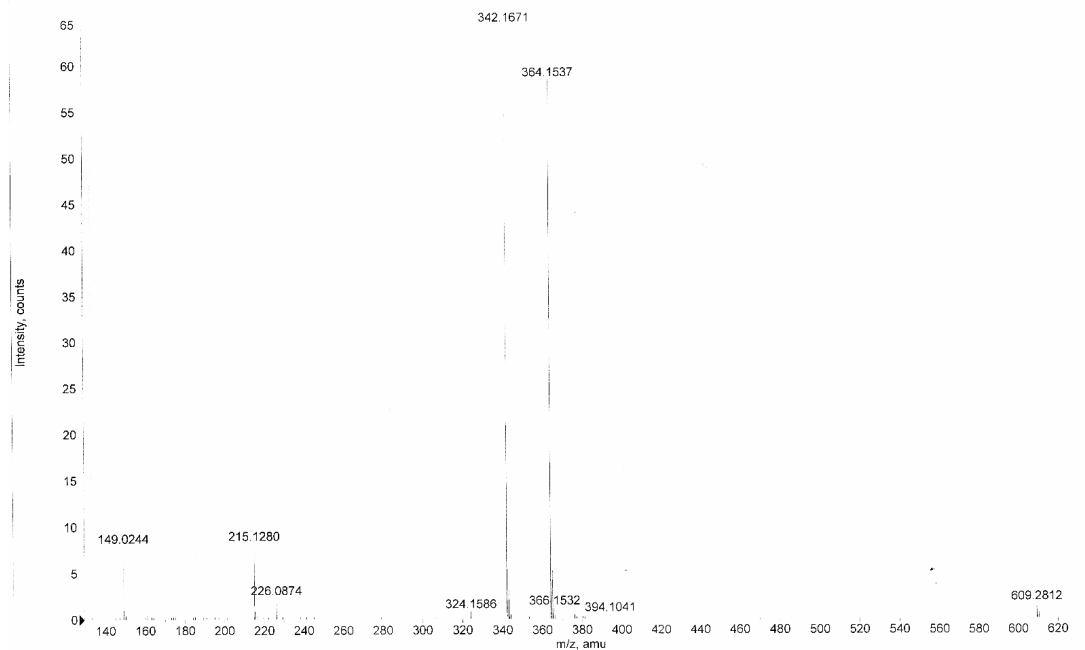

 $C_{15}H_{23}N_3O_6$ Mol. Wt.: 341.36




Sample Name: Z. Yang
+TOF MS: 0.350 to 0.467 min from YZ_803_B.wiff
a=3.56074040202493520e-004, t0=4.68930410896064130e+000

ESI-MS positive mode

Sample dissolved in MeOH/H₂O/HFo (74+25+1)
Max 64.5 counts.

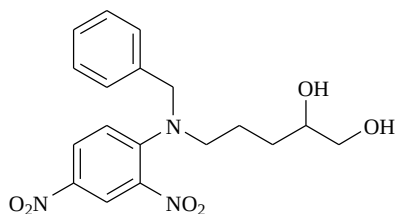


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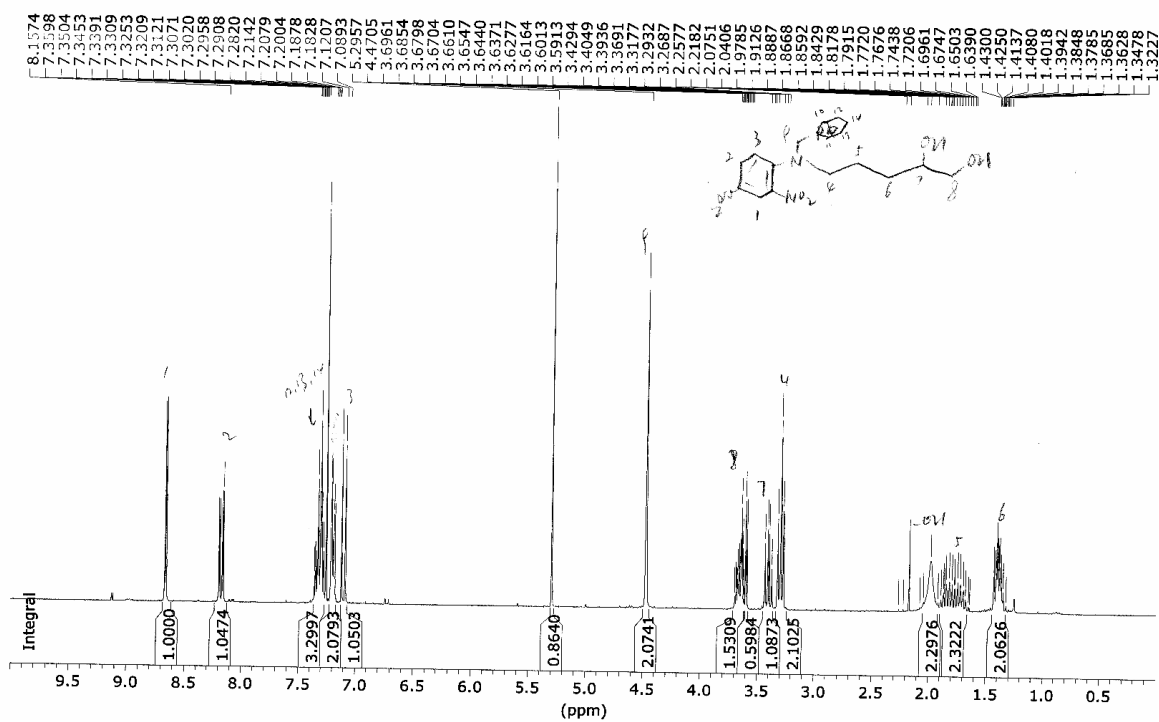
Acq. Date: Tuesday, November 30, 2004

14b.

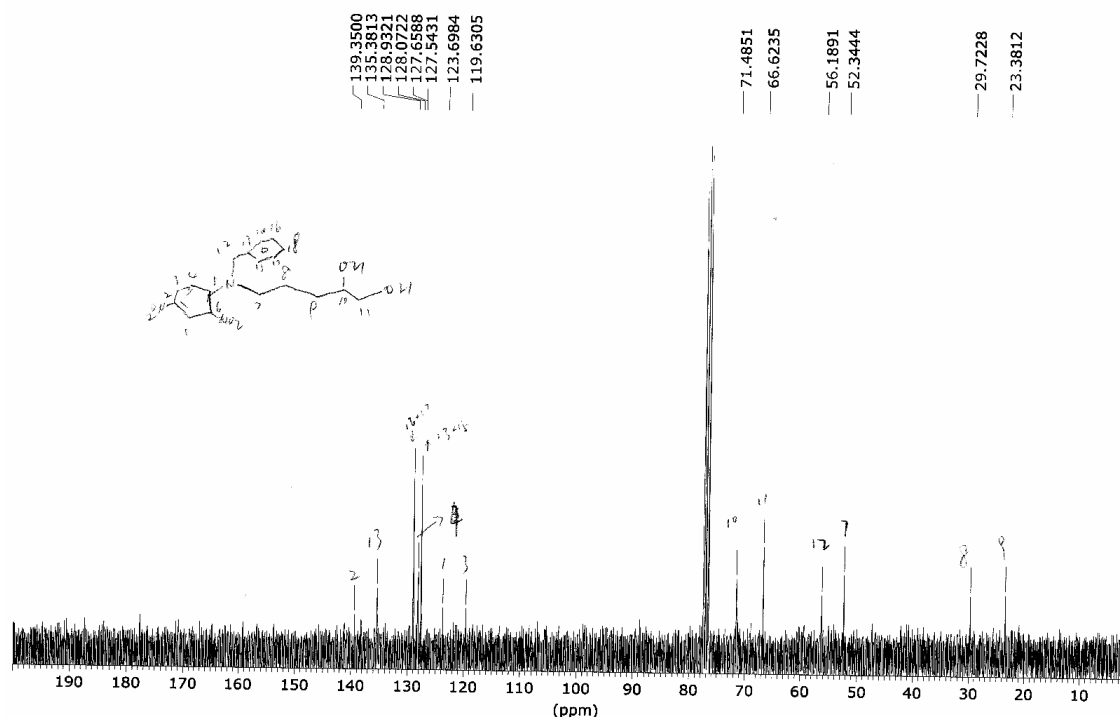


C₁₈H₂₁N₃O₆ Mol. Wt.: 375.38

yz-804Proton_ns32 CDCl3 /disk0 service 56



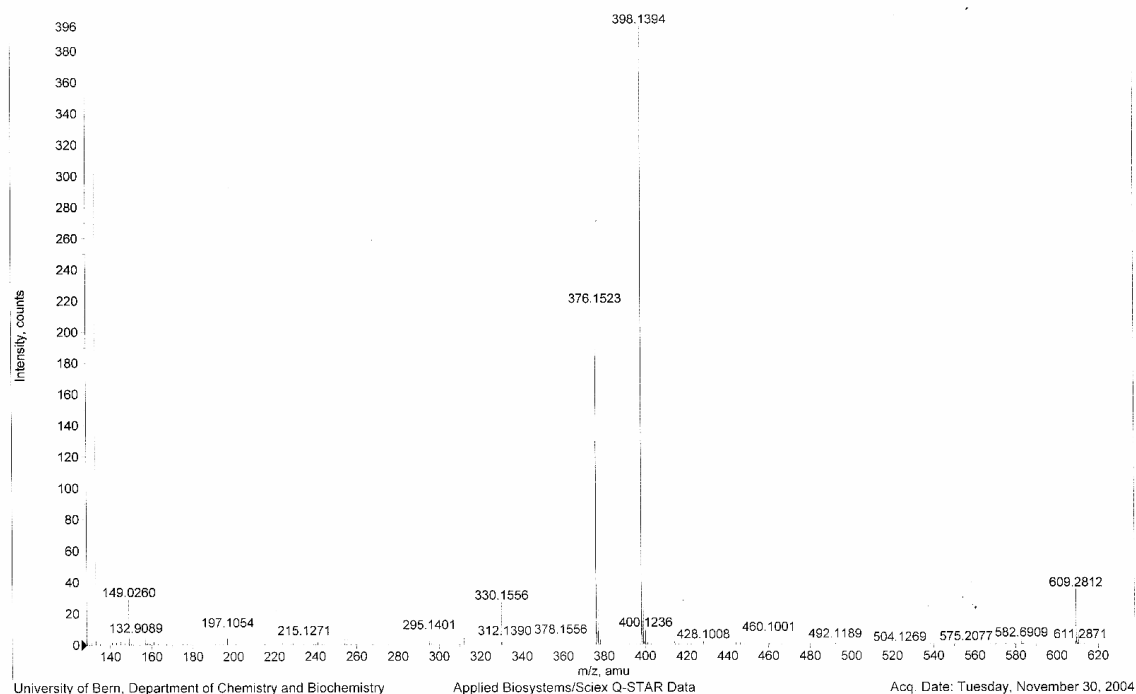
yz-804Carbon_ns256 CDCl3 /disk0 service 56



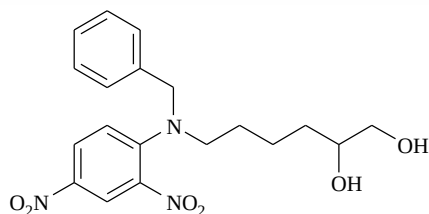
Sample Name: Z. Yang
 +TOF MS: 2.351 to 2.534 min from YZ_804_B.wiff
 a=3.56071731688485200e-004, t0=4.68930410896064130e+000

ESI-MS positive mode

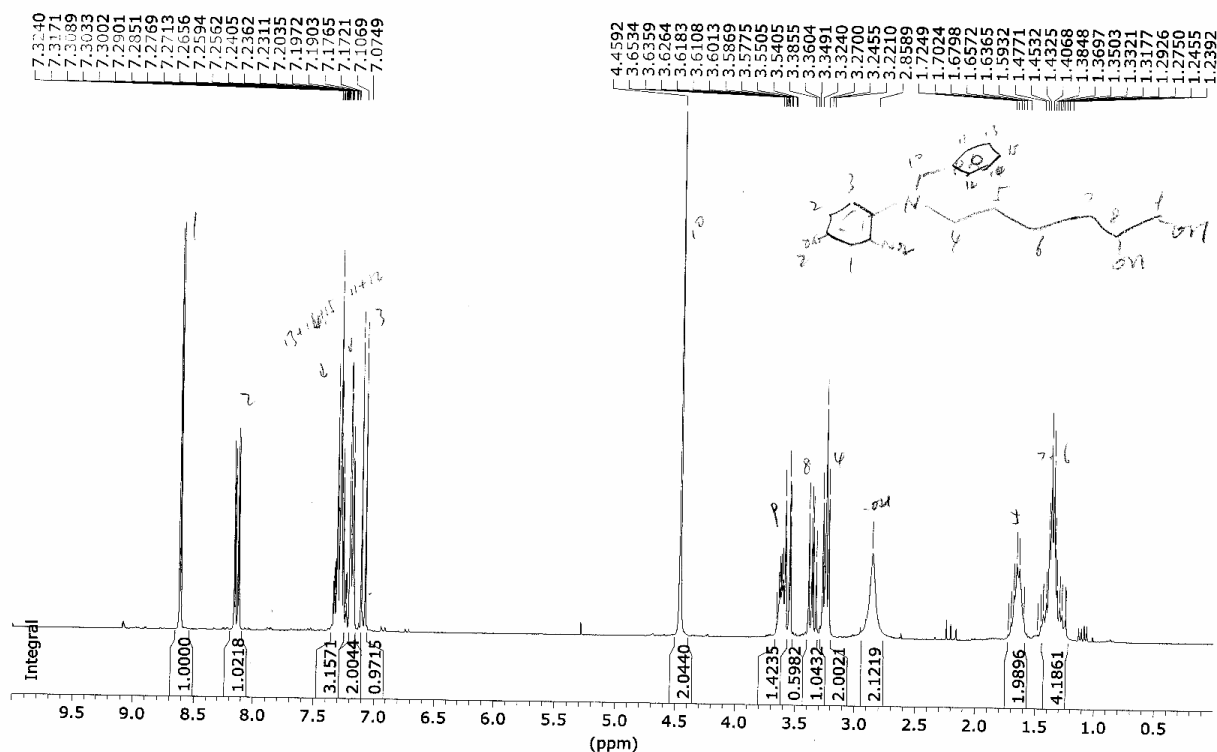
Sample dissolved in MeOH/H₂O/HF₃ (74+25+1)
 Max. 395.8 counts.

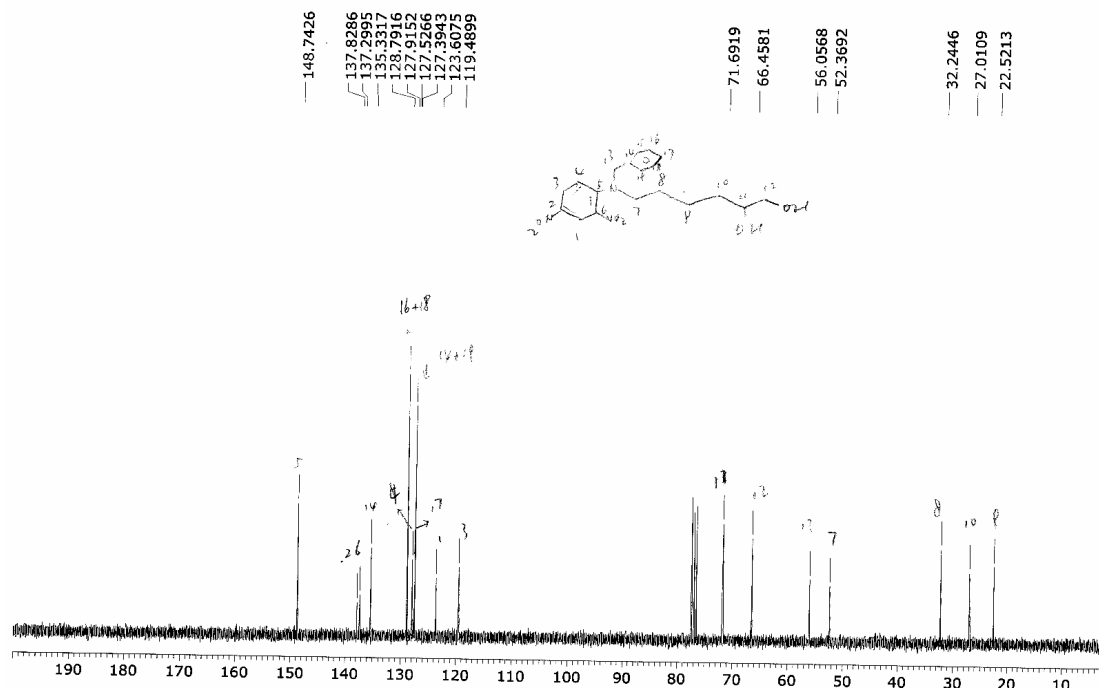


15b.



C₁₉H₂₃N₃O₆ Mol. Wt.: 389.4

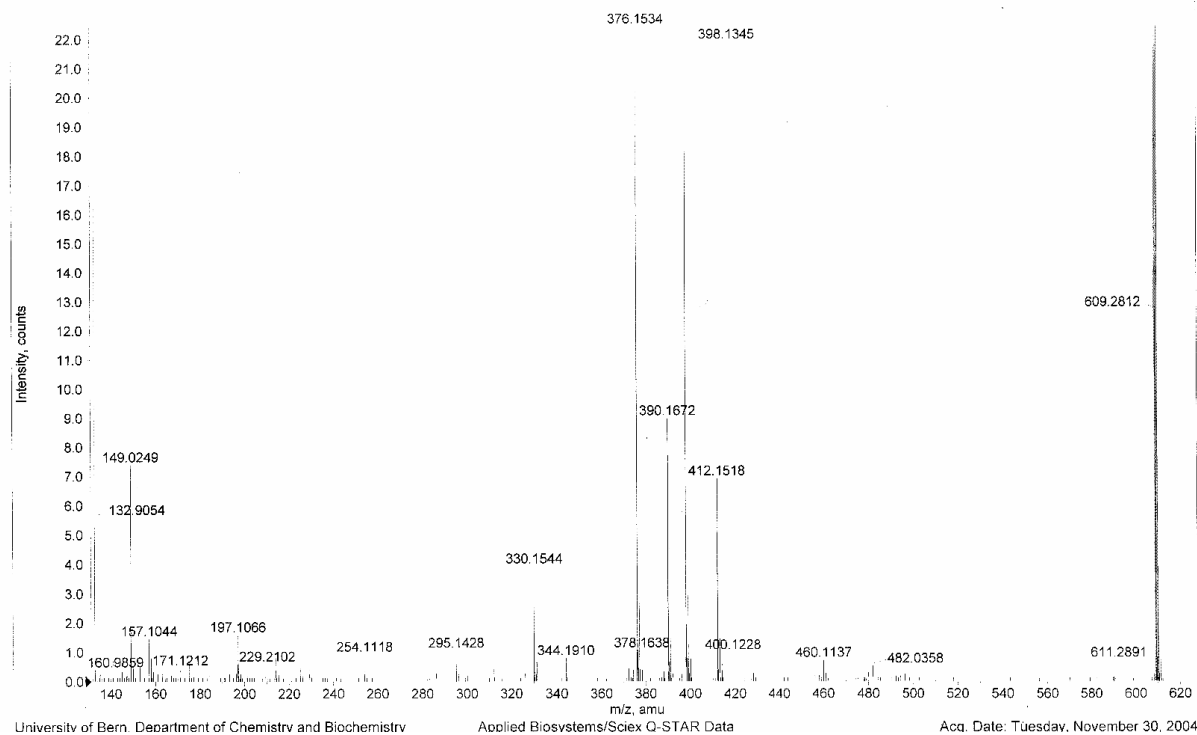
yz-805Proton_ns32 CDCl₃ /disk0 service 57



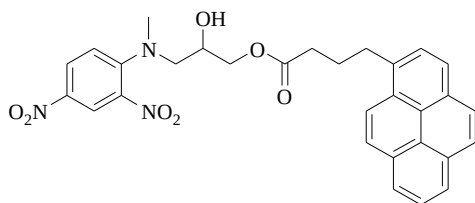
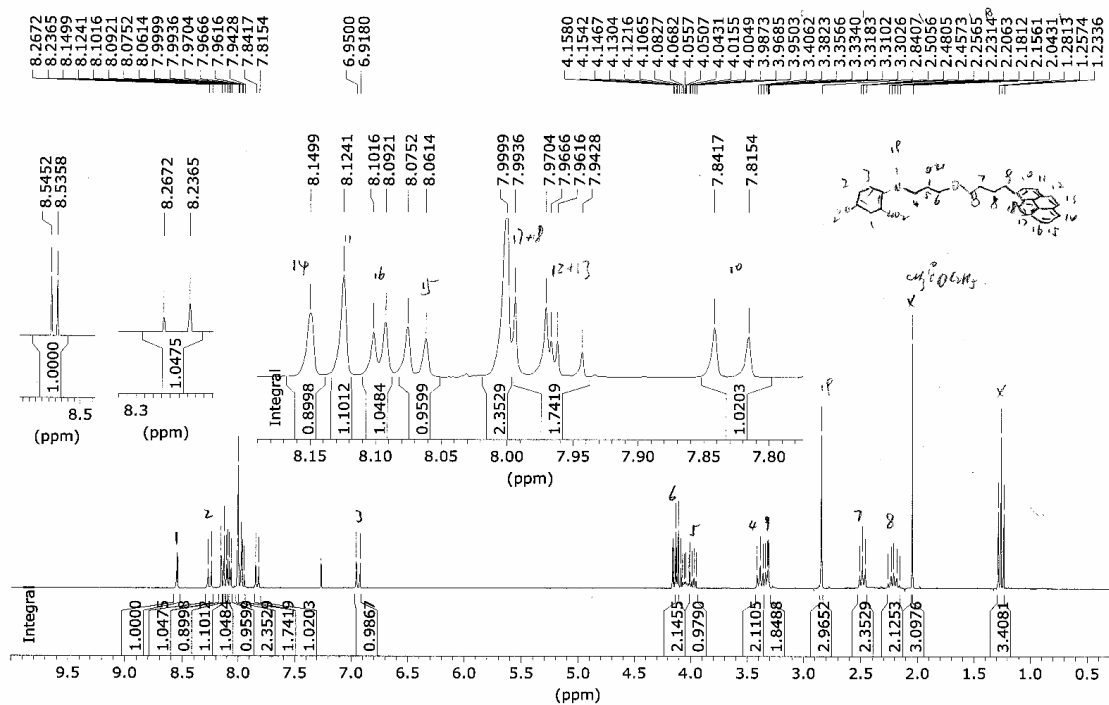
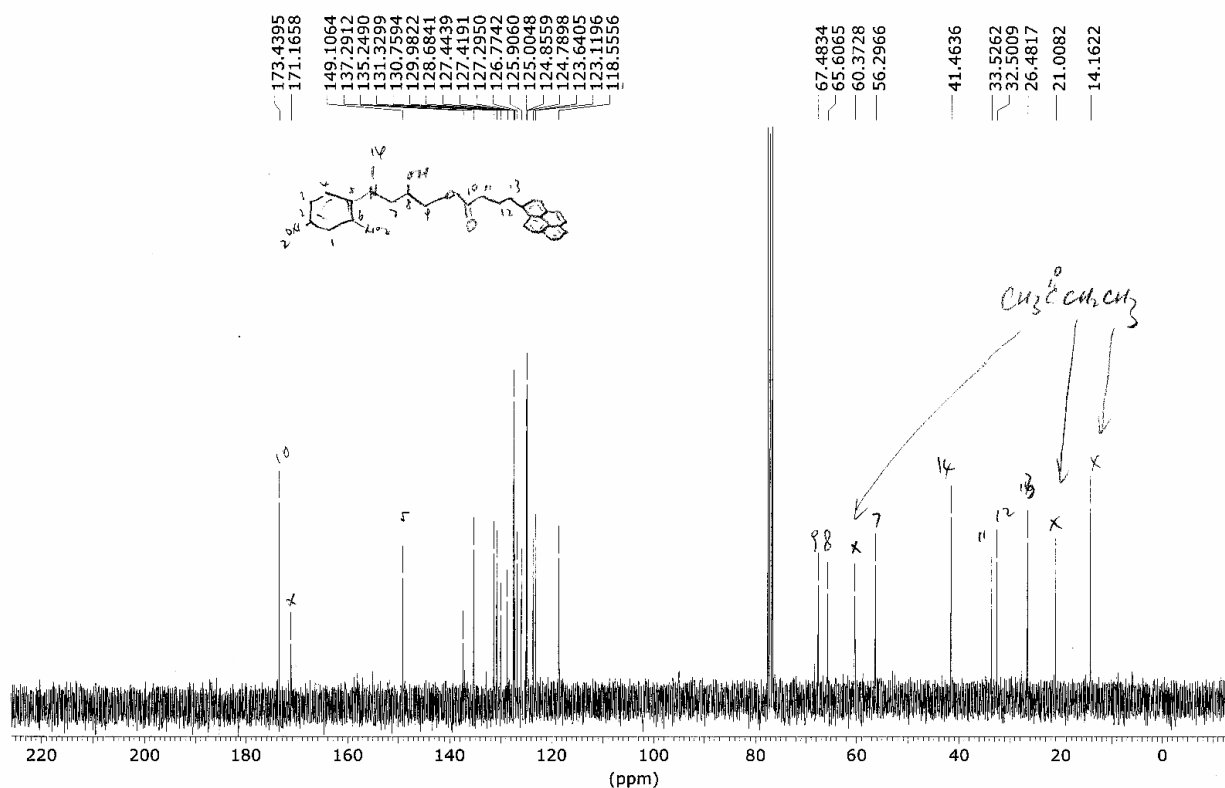
Sample Name: Z. Yang
 +TOF MS: 3.668 to 3.901 min from YZ_805_B.wiff
 a=3.56074800750573060e-004, t0=4.94546106209800110e+000

ESI-MS positive mode

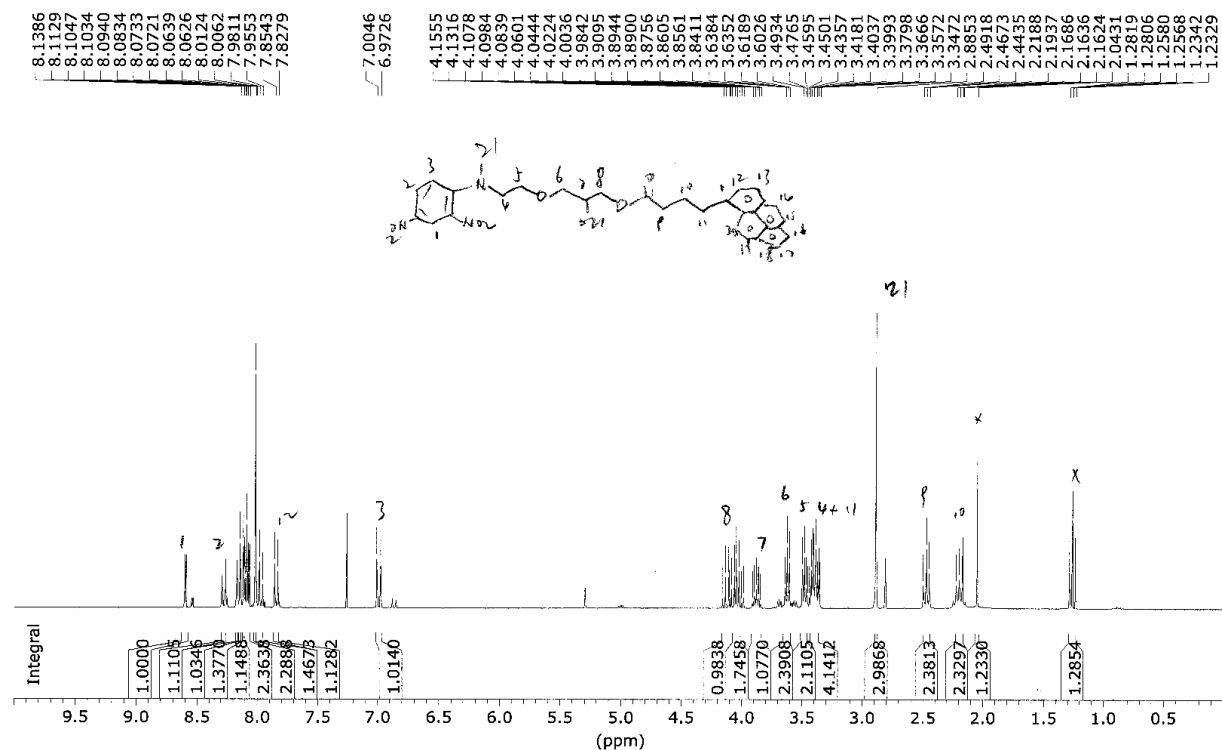
Sample dissolved in MeOH/H₂O/HFo (74+25+1)
 Max. 23.3 counts.



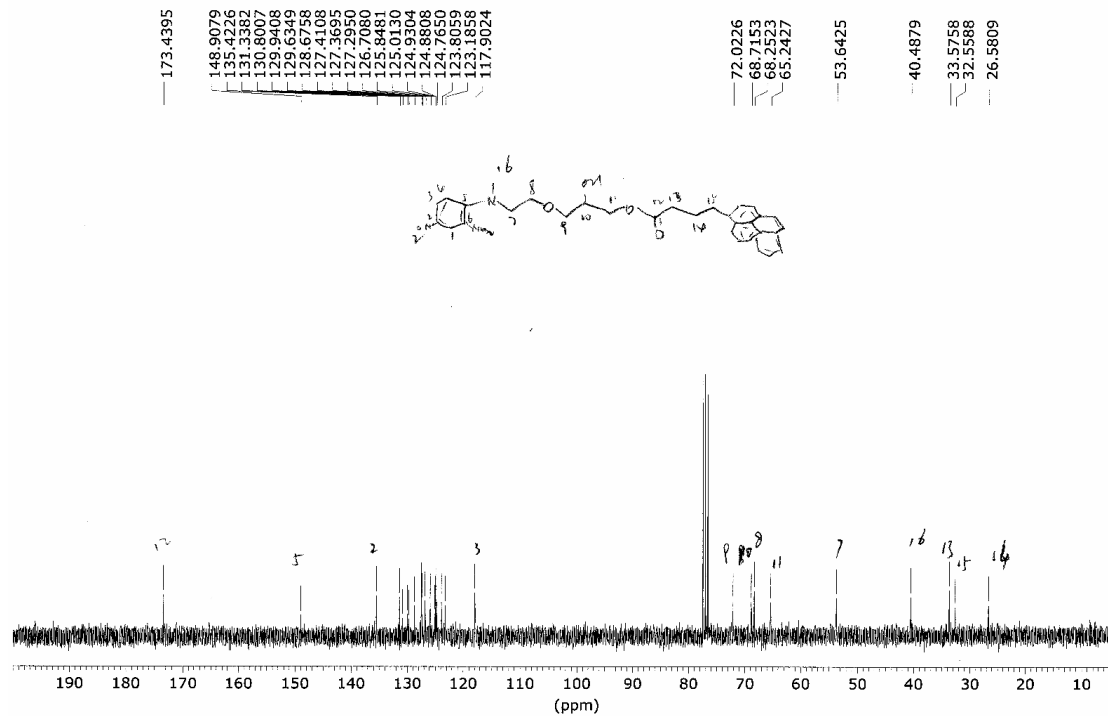
1a.

C₃₀H₂₇N₃O₇ Mol. Wt.: 541.55yz-806-2Proton_ns32 CDCl₃ /disk0 service 113yz-806-2Carbon_ns512 CDCl₃ /disk0 service 113

2a.

Cc1ccc(cc1)N(CCOCCOC(=O)c2ccccc2)Cc3ccccc3 C₃₂H₃₁N₃O₈ Mol. Wt.: 585.6

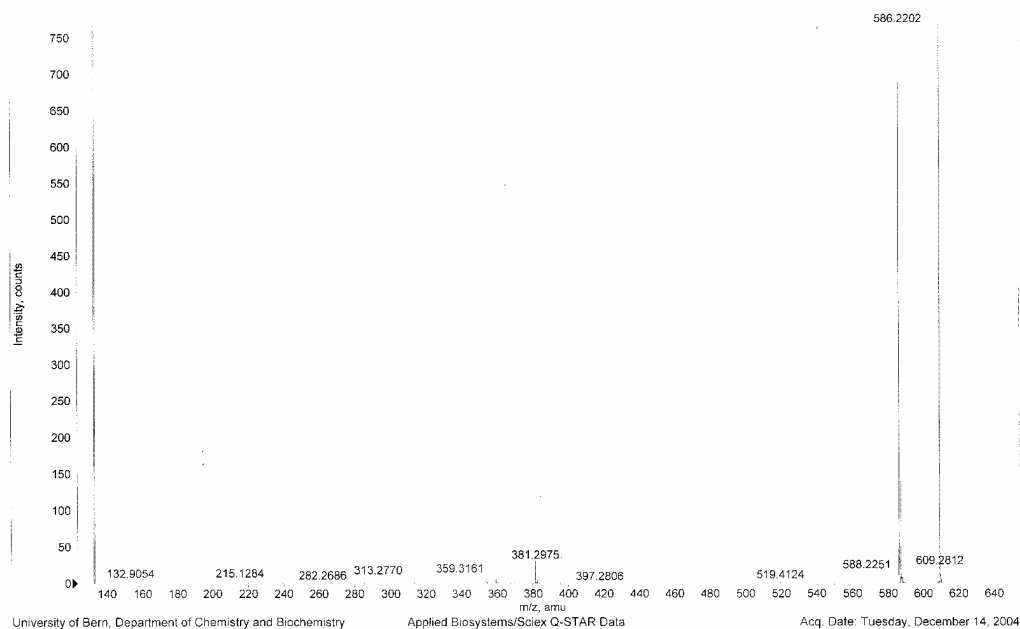
yz-807Carbon_ns512 CDCl3 /disk0 service 59



Sample Name: Z. Yang
+TOF MS: 1.667 to 1.800 min from YZ_807_N_B.wiff
a=3.55798380700148220e-004, t0=4.66883675069402670e+000

ESI-MS positive mode

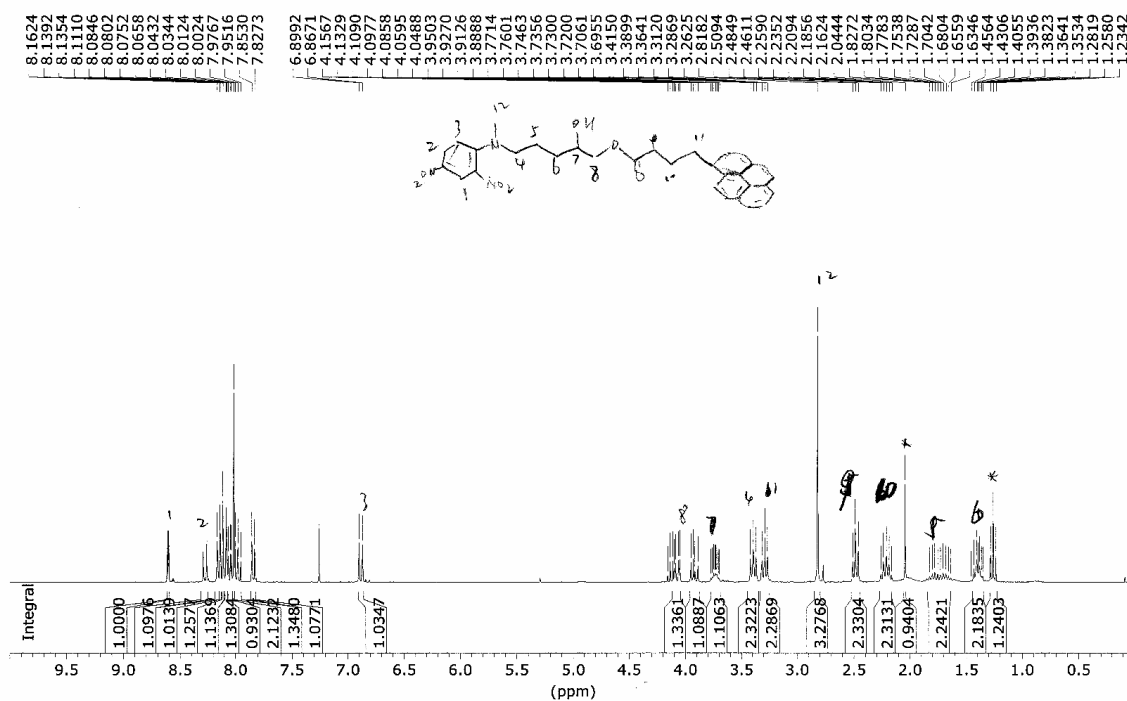
Sample dissolved in H₂O/ACN (1+1)
Max. 767.7 counts.



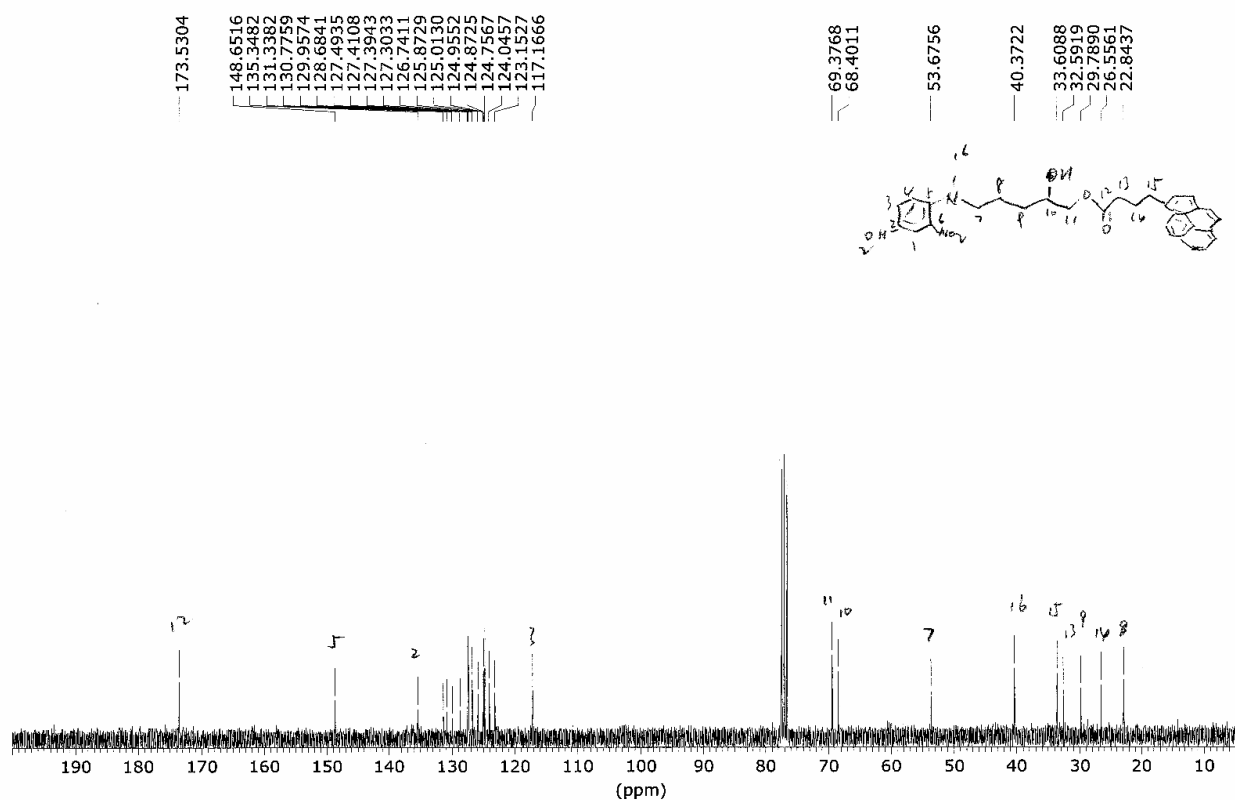
3a



yz-808-2Proton_ns32.CDCI3 /disk0 service 115



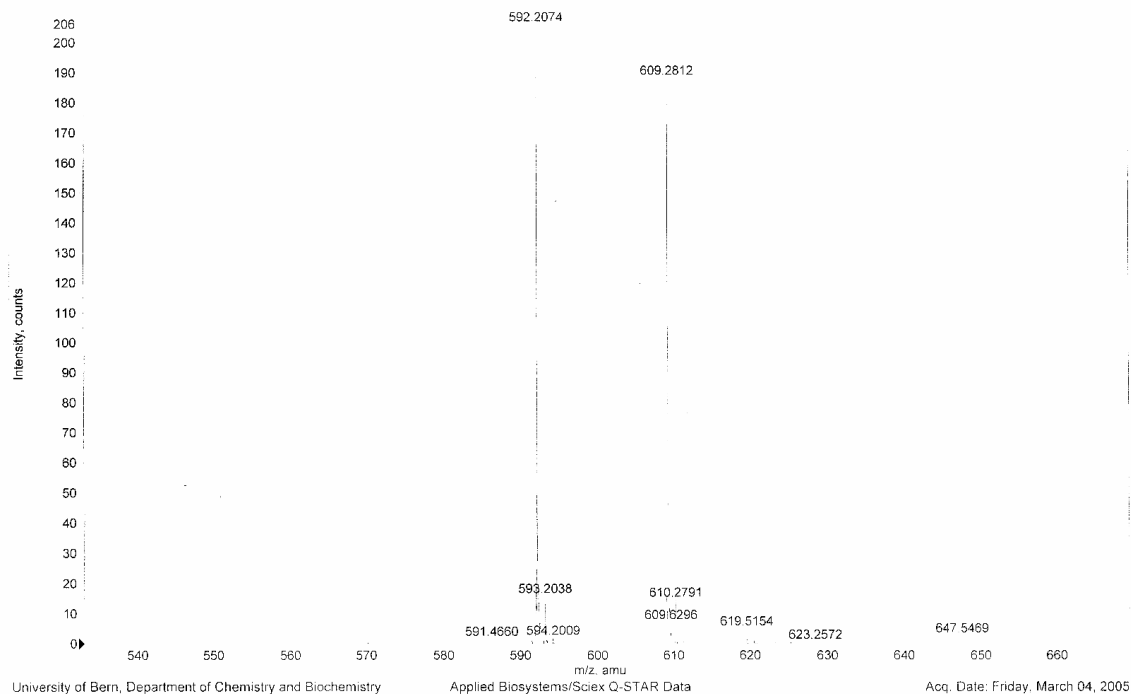
yz-808-2Carbon_ns512 CDCl3 /disk0 service 115



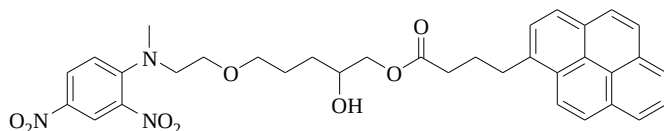
Sample Name: Yang
 +TOF MS: 0.017 to 0.150 min from YZ808_2_MeOH_B.wiff
 a=3.55830854144618550e-004, t0=5.03067418562932290e+000

ESI-MS positive mode

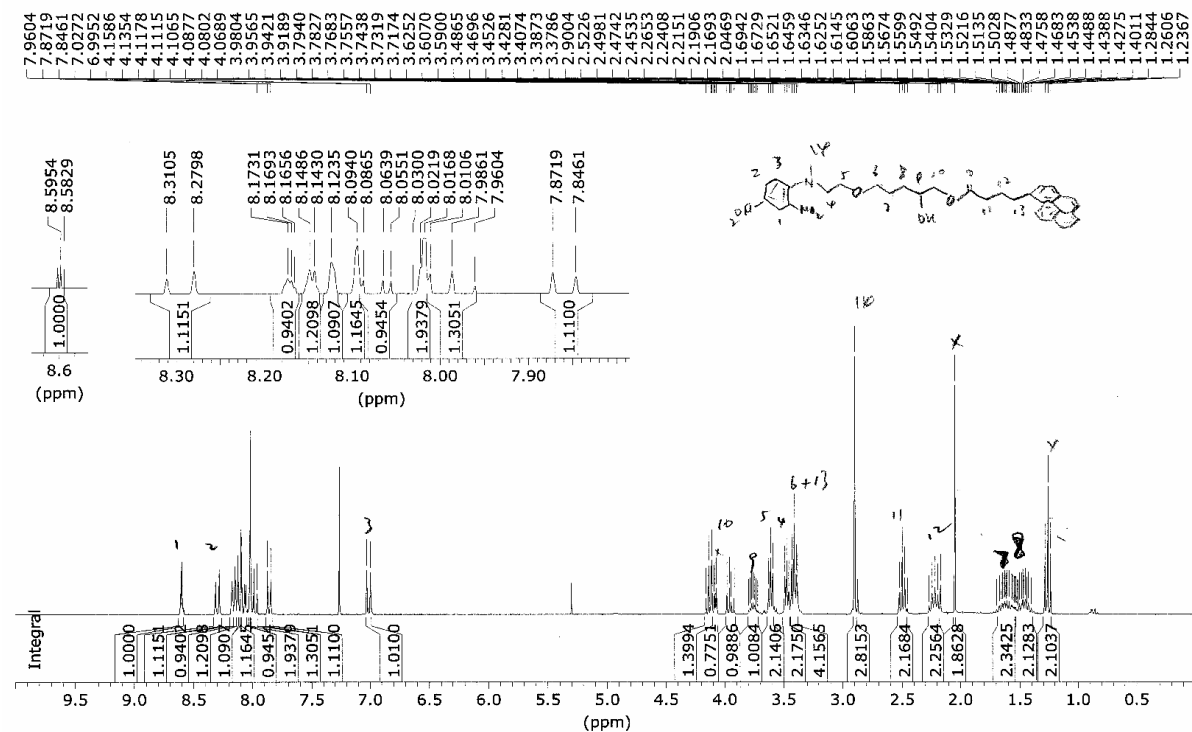
Sample dissolved in MeOH
 Max. 1668.9 counts.

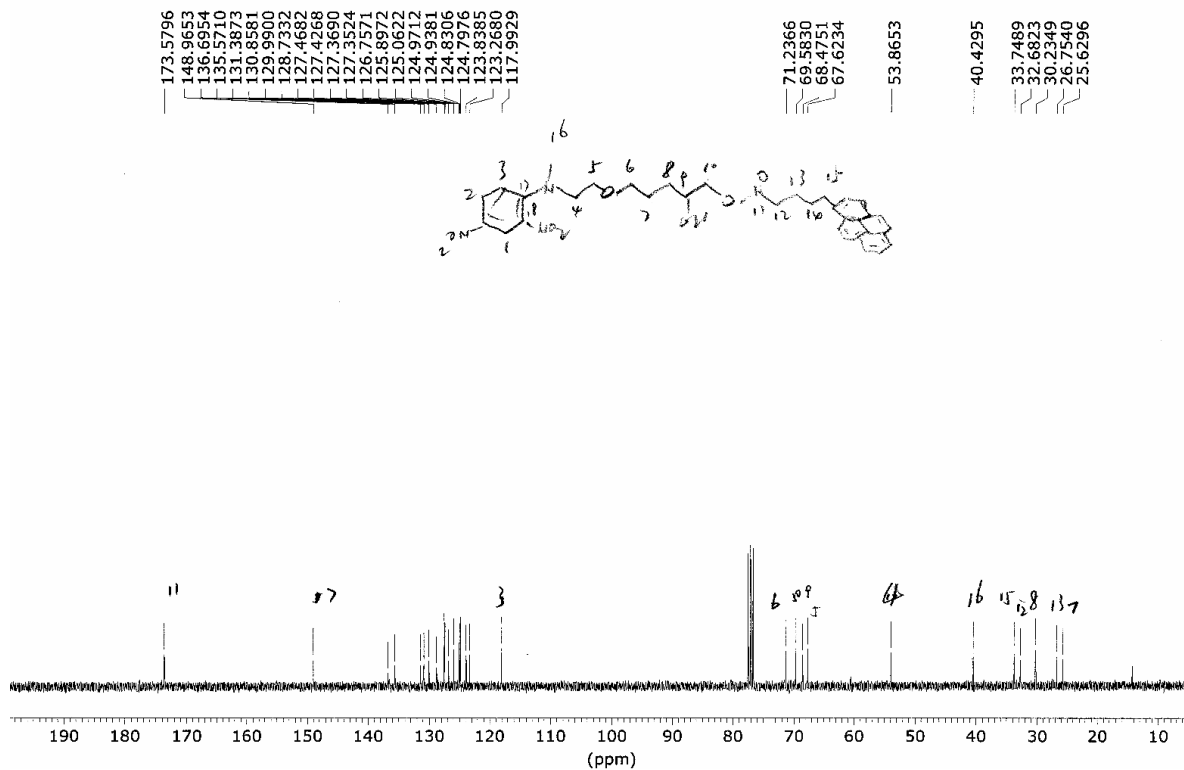


4a


 $C_{34}H_{35}N_3O_8$ Mol. Wt.: 613.66

yz-809Proton_ns32 CDCl3 /disk0 service 85





Sample Name: Z. Yang
+TOF MS: 4.434 to 4.568 min from YZ_809_N_B.wiff
a=3.55810430898526360e-004, t0=5.79430630949718760e+000

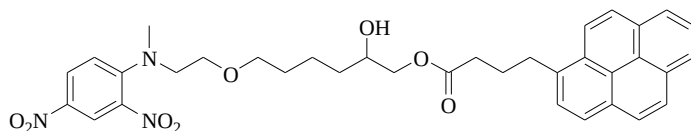
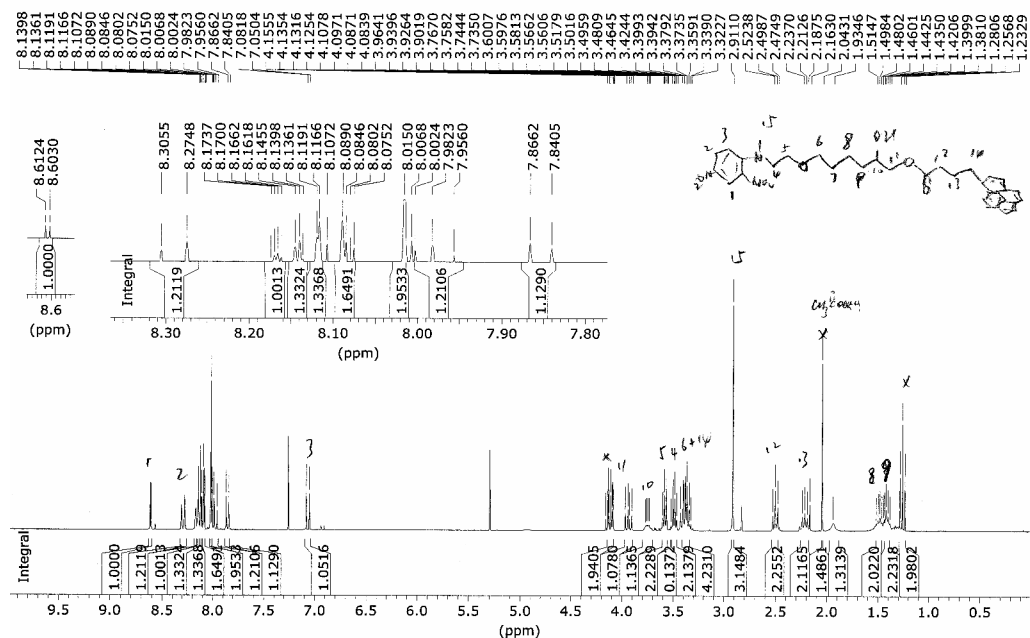
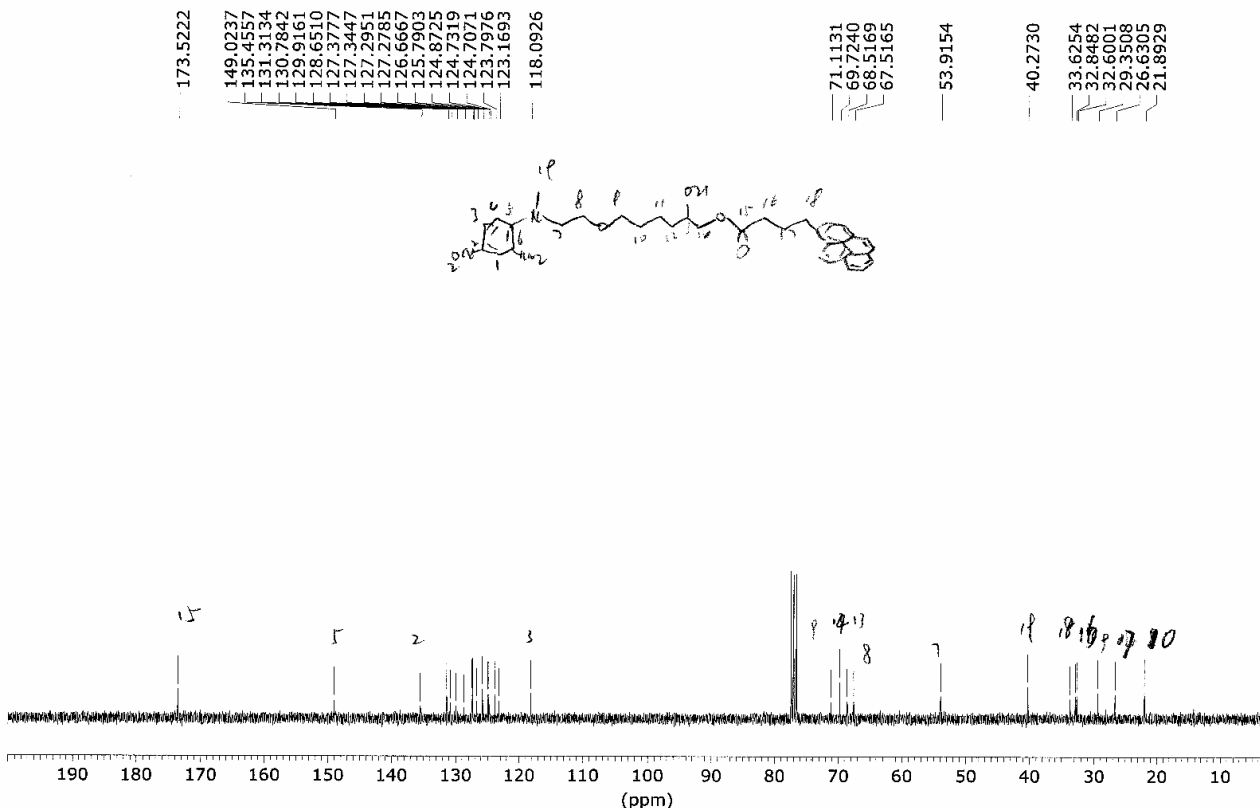
ESI-MS positive mode

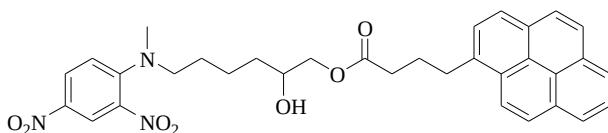
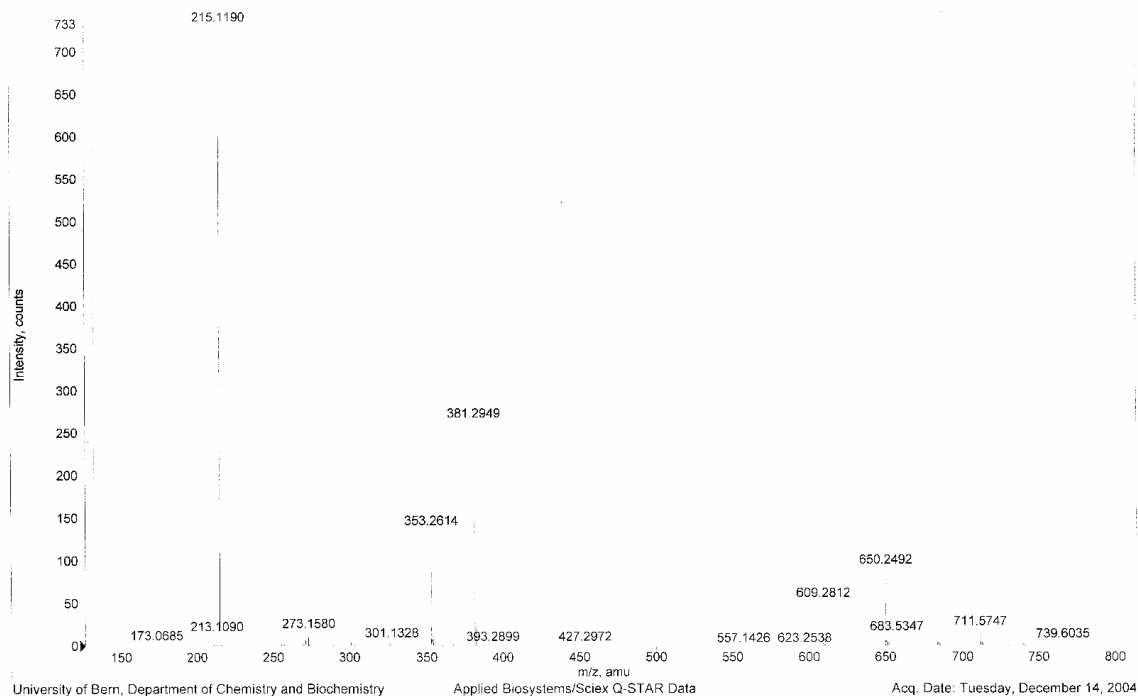
Sample dissolved in H₂O/ACN (1+1)
Max. 1241.6 counts.



S34

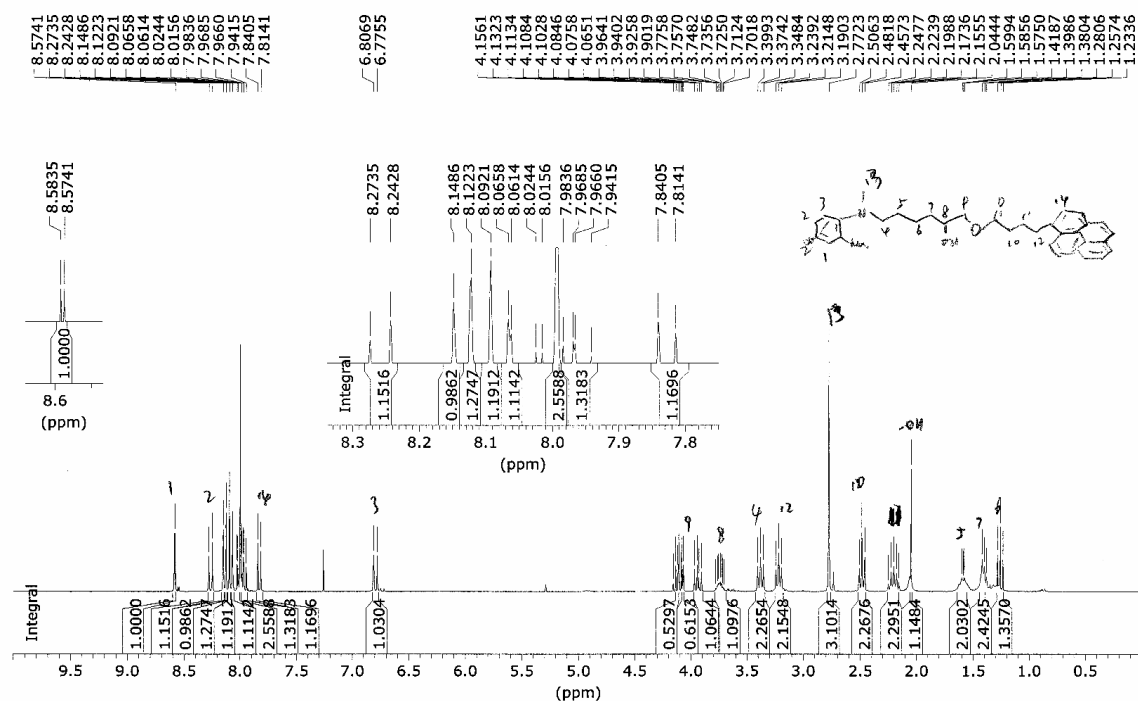
5a

C₃₅H₃₇N₃O₈ Mol. Wt.: 627.68yz-810Proton_ns32 CDCl₃/disk0 service 60yz-810Carbon_ns512 CDCl₃/disk0 service 60

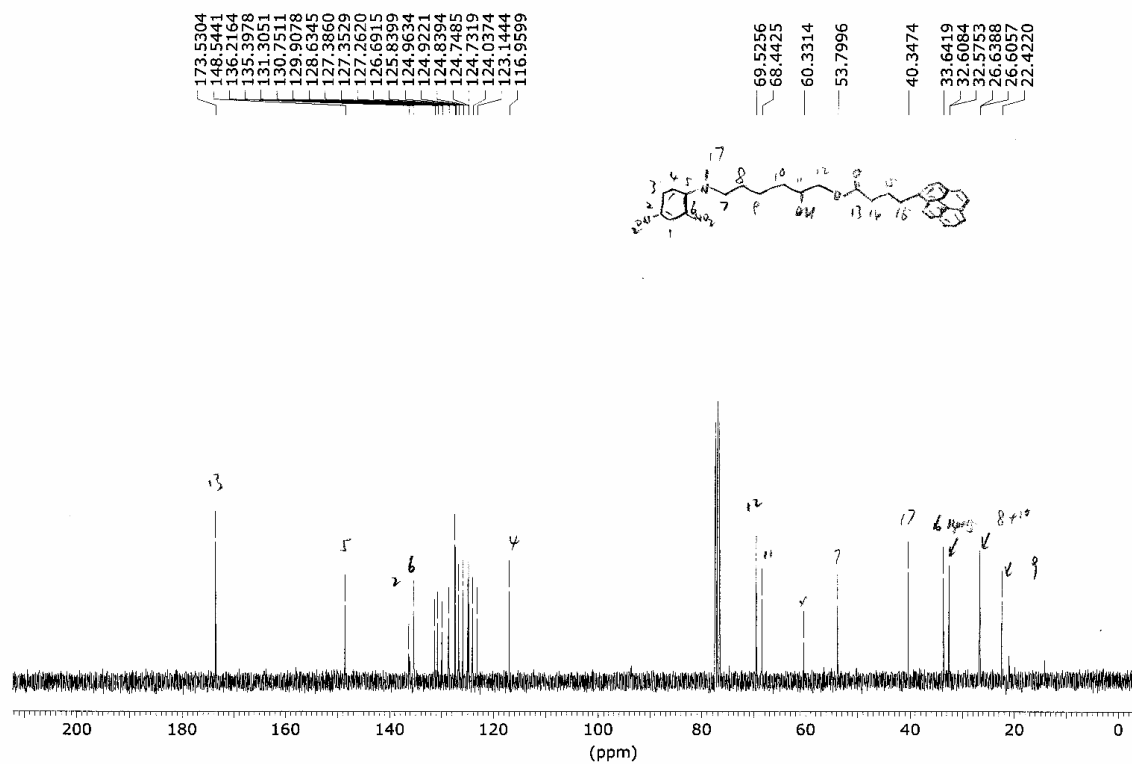


$C_{33}H_{33}N_3O_7$ Mol. Wt.: 583.63

yz-811Proton_ns32 CDCl3 /disk0 service 86



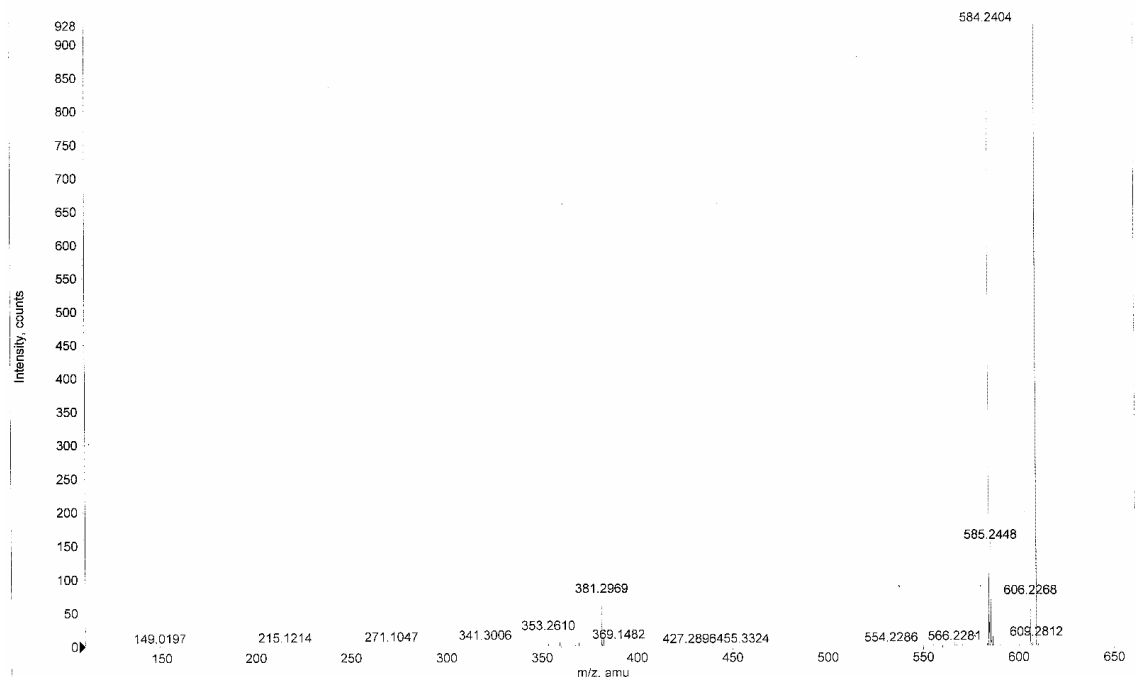
yz-B11Carbon_ns512 CDCl3 /disk0 service 86



Sample Name: Z. Yang
+TOF MS: 0.917 to 1.084 min from YZ_B11_N_B.wiff
a=3.55801612566747430e-004, t0=5.79430630949718760e+000

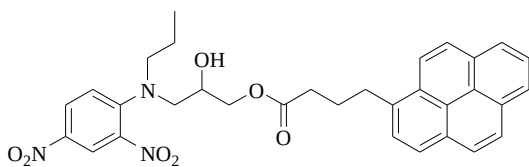
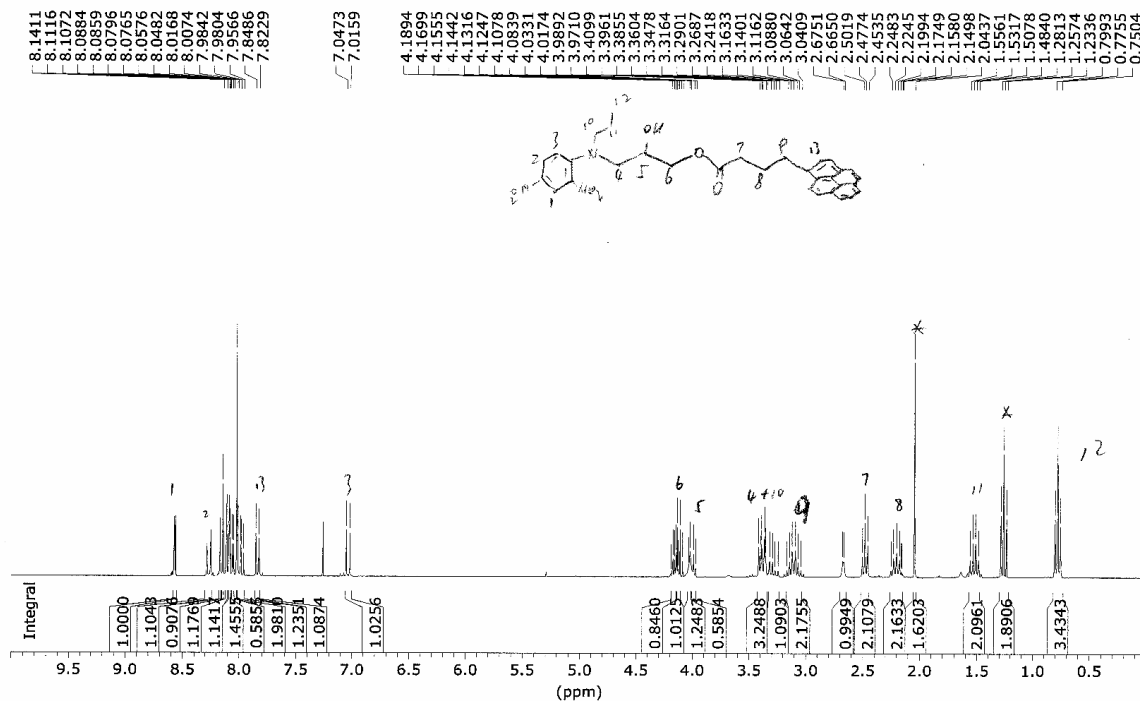
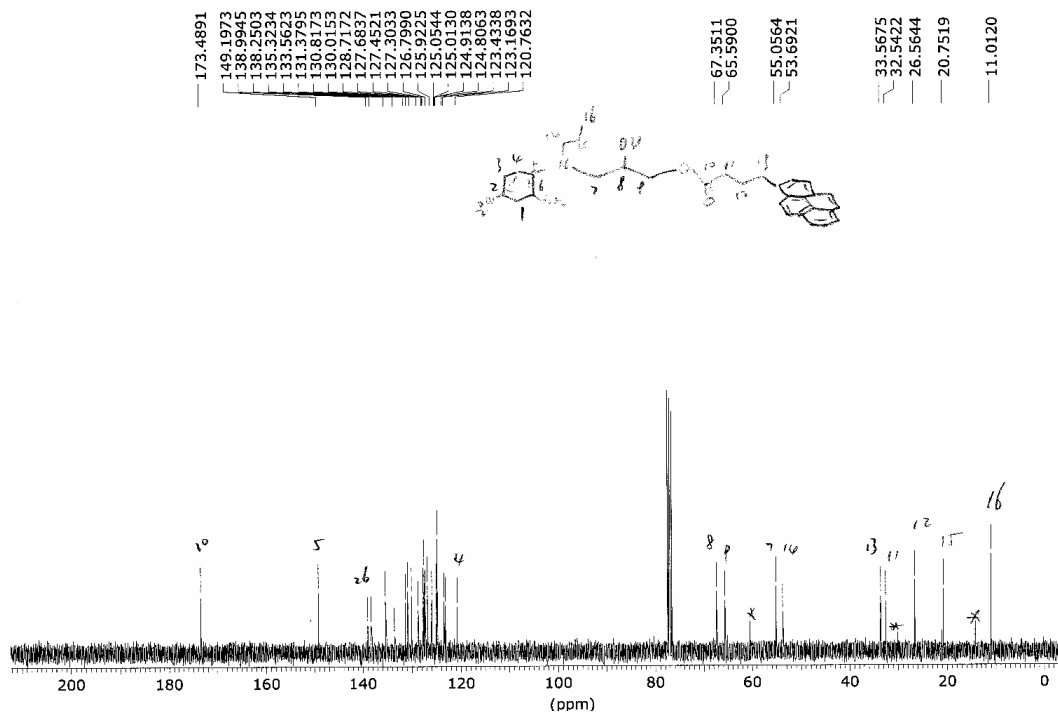
ESI-MS positive mode

Sample dissolved in H₂O/ACN (1+1)
Max. 928.4 counts

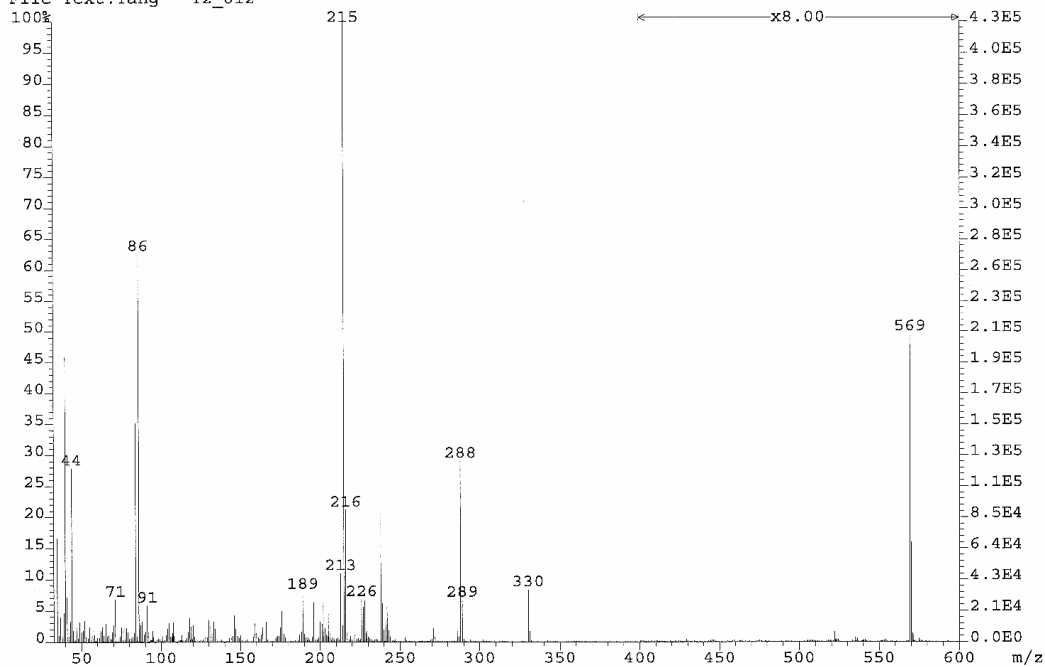


S37

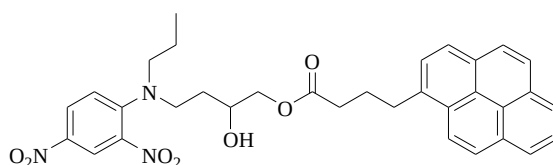
7a

C₃₂H₃₁N₃O₇ Mol. Wt.: 569.6yz-812Proton_ns32 CDCl₃ /disk0 service 74yz-812Carbon_ns512 CDCl₃ /disk0 service 74

File:Y2_812 Ident:105_127-1_12 Win 90PPM Acq:13-DEC-2004 10:19:50 +4:54 Cal:CEI121304_1
 AutoSpecQ EI+ Magnet BpM:215 BpI:425630 TIC:4339977 Flags:HALL
 File Text:Yang Y2_812

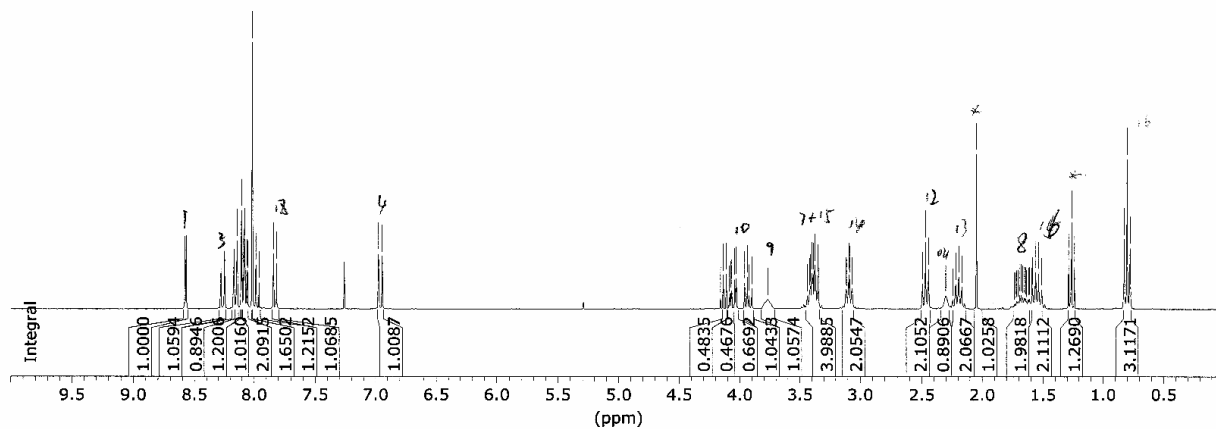
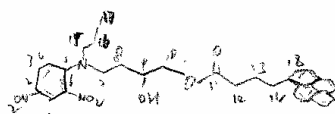


8a

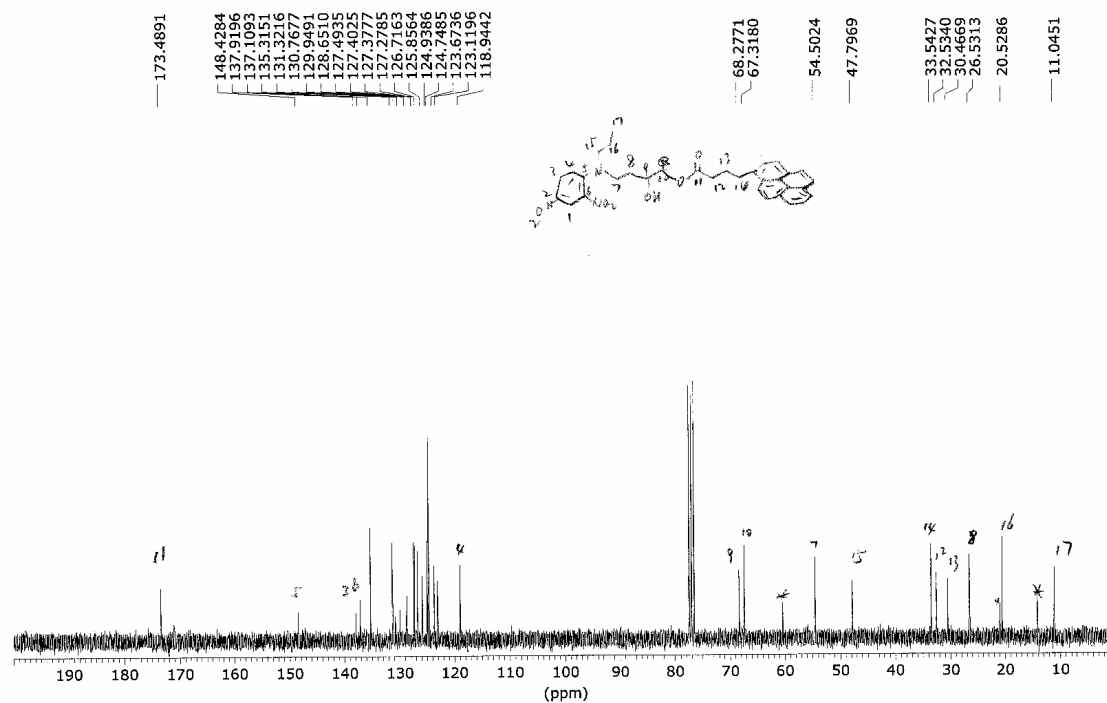


$C_{33}H_{33}N_3O_7$ Mol. Wt.: 583.63

yz-813Proton_ns32 CDCl3 /disk0 service 75



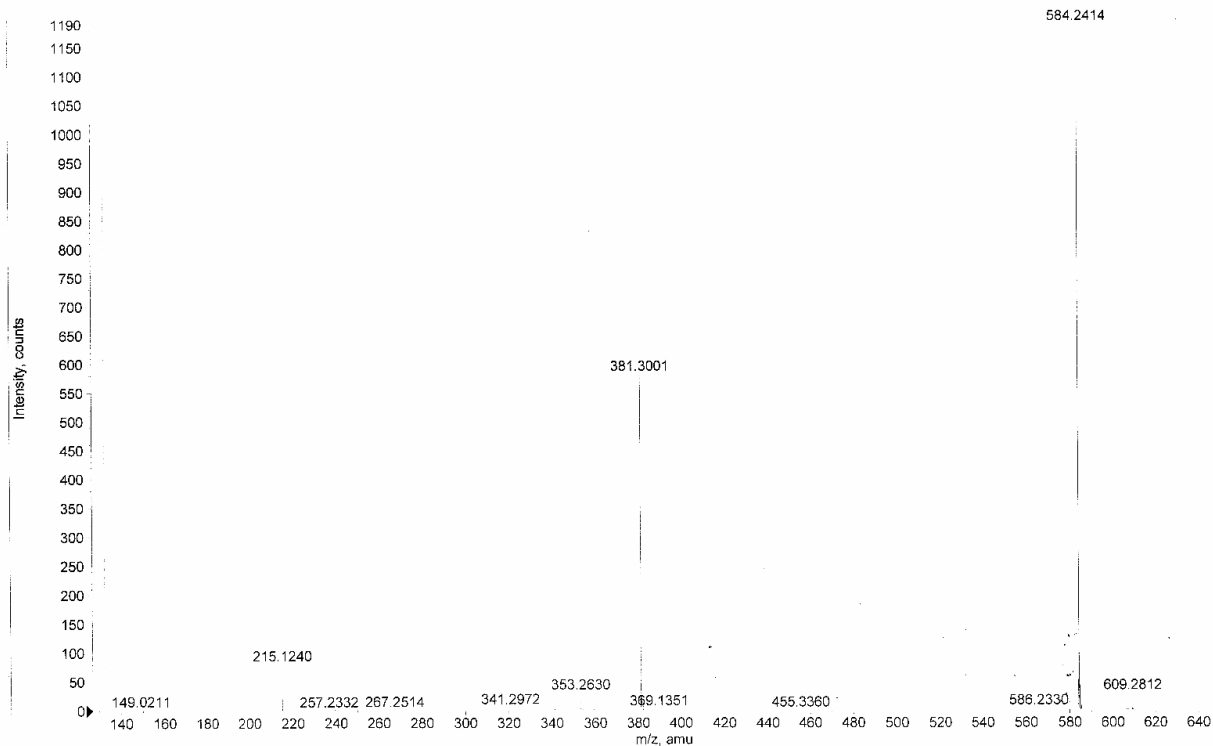
yz-813 Carbon_n5512 CDCl3 /disk0 service 75



Sample Name: Z. Yang
+TOF MS: 0.167 to 0.333 min from YZ_813_N_B.wiff
a=3.55801437374745240e-004, t0=5.79430630949718780e+000

ESI-MS positive mode

Sample dissolved in H₂O/ACN (1+1)
Max. 1190.4 counts.



University of Bern, Department of Chemistry and Biochemistry

Applied Biosystems/Sciex Q-STAR Data

Acq. Date: Monday, December 13, 200

9a



¹H NMR spectrum (CDCl₃) of compound 10.

Chemical structure of compound 10: O=C1C(=O)N(C1)C2=CC=C(C=C2)C3=CC=CC=C3

Peak list (ppm): 9.572, 8.0486, 8.0256, 8.0162, 7.9891, 7.9691, 7.9673, 7.9428, 7.8386, 7.8129, 6.8703, 6.8383, 4.1561, 4.1323, 4.1094, 4.1004, 4.0899, 4.0846, 4.0626, 4.0519, 3.9559, 3.9321, 3.9176, 3.8938, 3.7601, 3.7419, 3.7331, 3.7200, 3.7093, 3.6961, 3.6848, 3.3949, 3.3949, 3.3704, 3.3440, 3.3355, 3.3166, 3.1878, 3.1551, 3.1551, 3.1426, 3.1319, 3.1181, 2.5000, 2.4749, 2.4510, 2.2481, 2.2481, 2.1927, 2.1680, 2.1504, 2.1429, 2.0933, 2.0437, 2.0437, 1.5436, 1.5298, 1.5185, 1.4866, 1.4866, 1.4193, 1.3949, 1.3748, 1.3572, 1.2869, 1.2800, 1.2631, 1.2562, 1.2323, 1.2323, 1.1183, 1.0943, 1.0685, 1.0447, 0.9618, 0.1391.

Integration values: 1.0000, 1.1719, 1.3166, 1.1455, 1.0187, 1.1344, 2.3367, 0.9431, 1.1515, 0.9916, 1.1719, 0.9785, 1.3166, 1.1455, 1.0185, 1.1344, 2.3367, 0.9431, 0.9431, 1.1515, 1.1515, 7.8386, 7.8129.

—173.5387

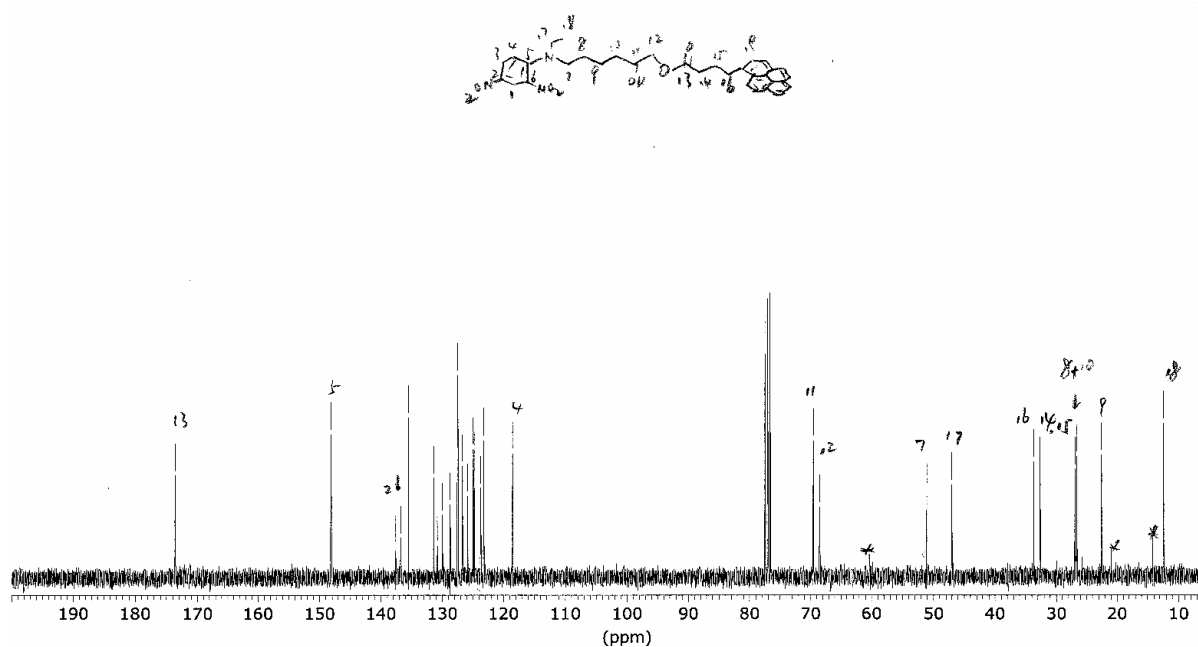
148.0729
137.5062
136.7124
135.4309
131.3134
130.7677
129.9160
128.6427
127.3943
127.3529
127.2702
126.6832
125.8316
124.9717
124.9056
124.8559
124.7485
124.7368
123.7149
123.1610
118.4977

69.5173
68.4425

51.0546
46.9784

33.6336
32.6332
32.5836
26.8786
26.6057
22.5378

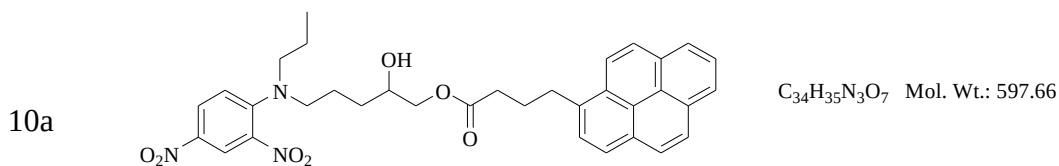
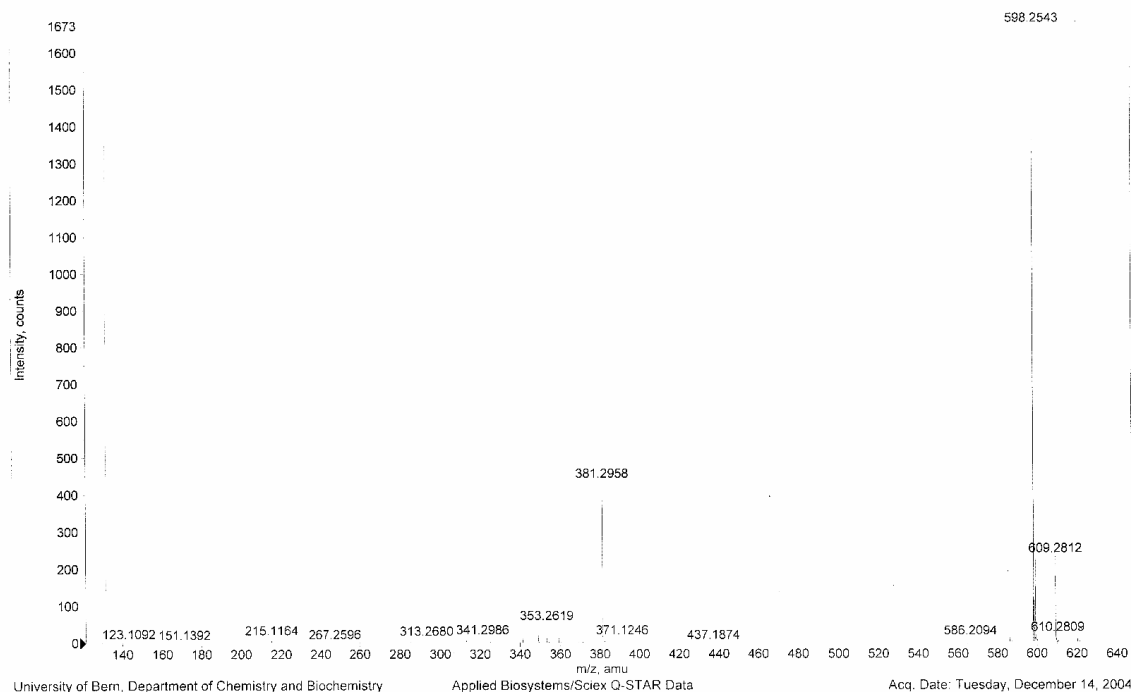
12.4011



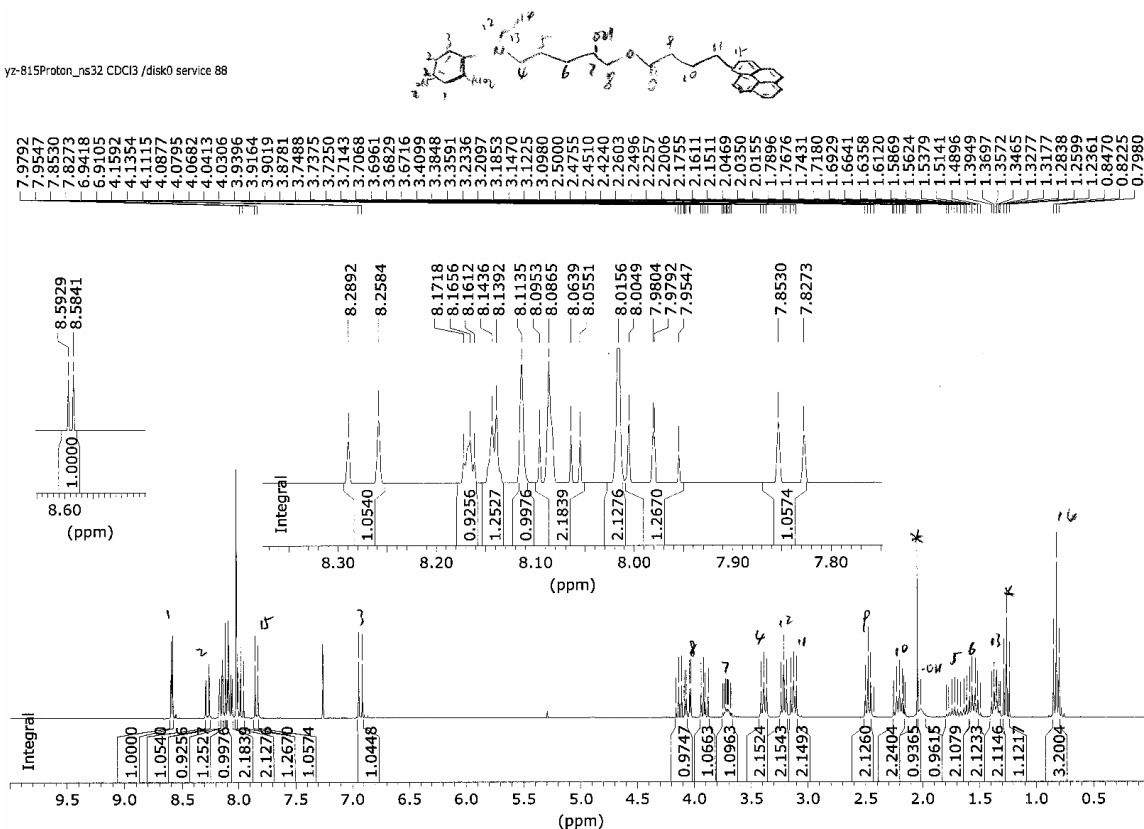
Sample Name: Z. Yang
+TOF MS: 1.434 to 1.594 min from YZ_814_N_B.wiff
a=3.55805174860253280e-004, tD=5.79430630949718760e+000

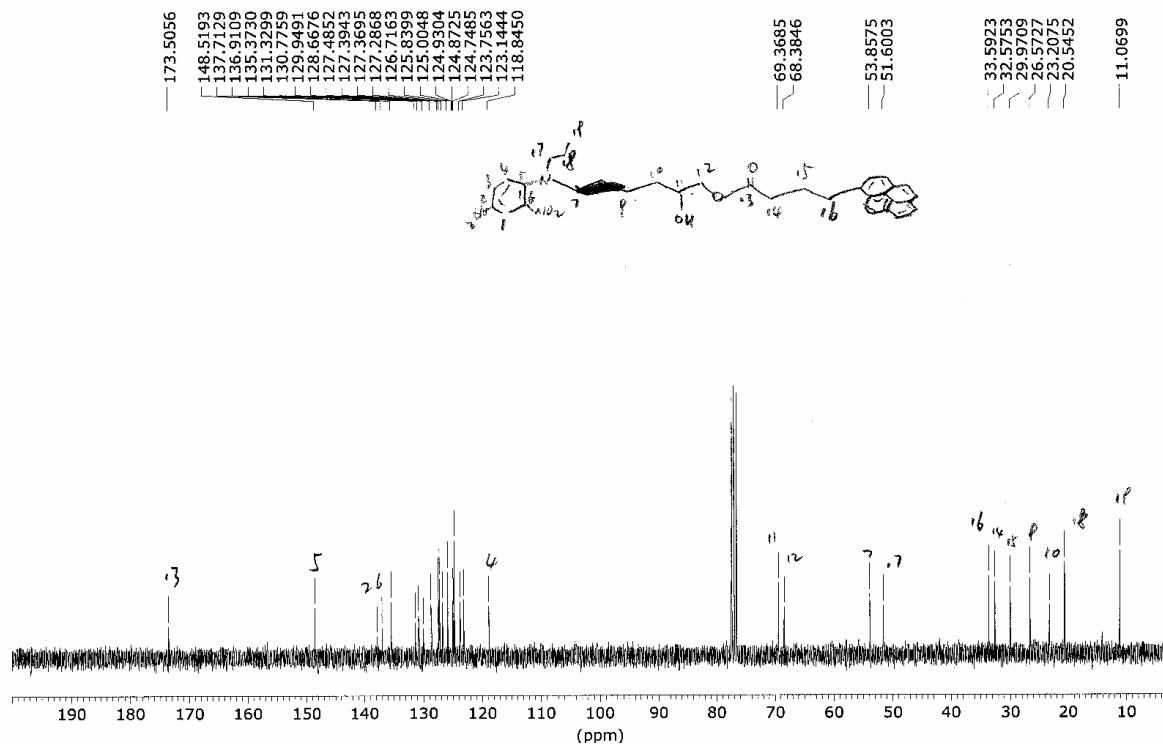
ESI-MS positive mode

Sample dissolved in H₂O/ACN (1+1)
Max. 1672.8 counts.



yz-815Proton_ns32 CDCl₃ / disk0 service 88

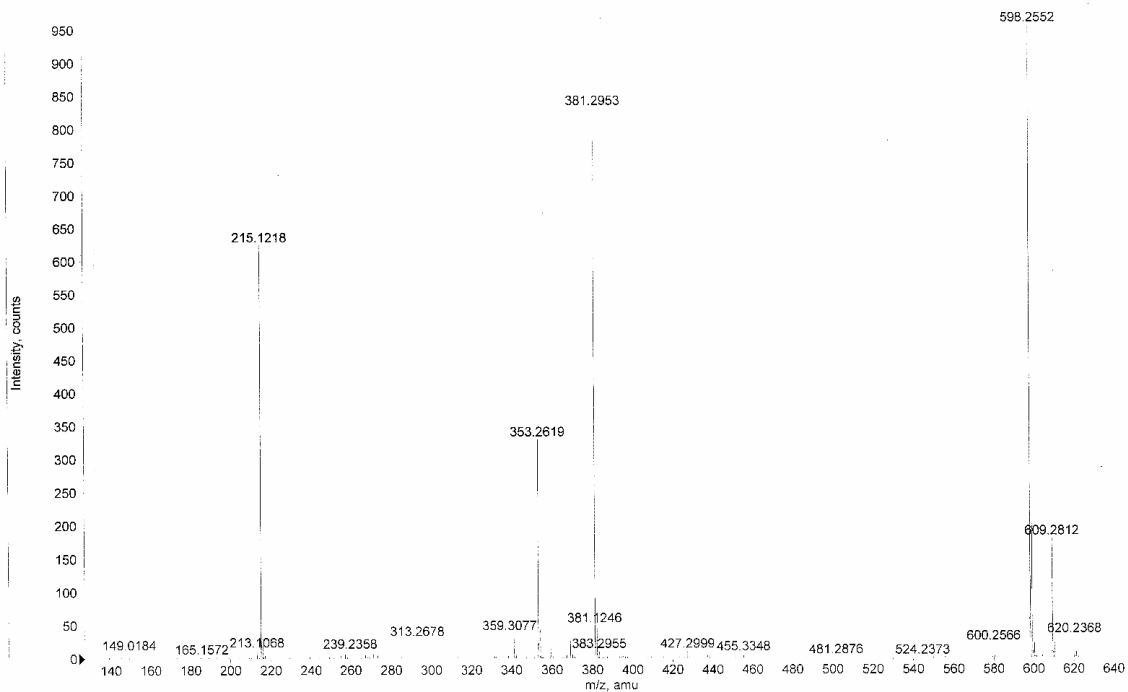




Sample Name: Z. Yang
 *TOF MS: 2.684 to 2.817 min from YZ_B15_N.B.wiff
 a=3.55806780847235250e-004, t0=5.79430630949718760e+000

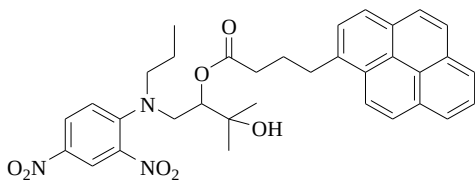
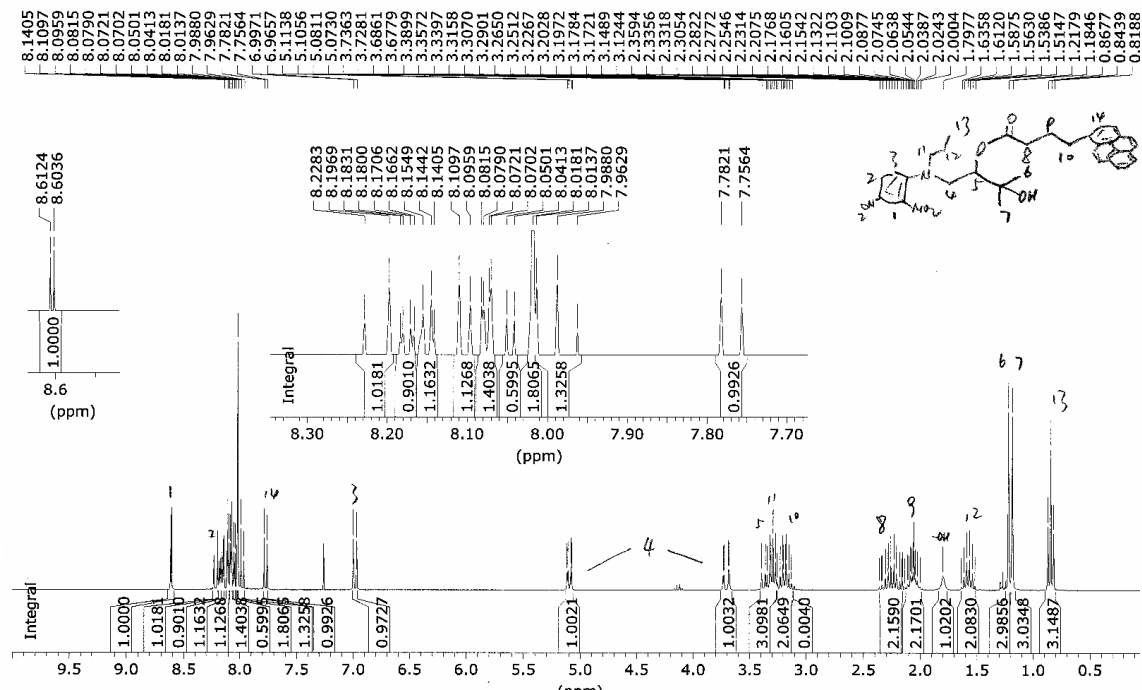
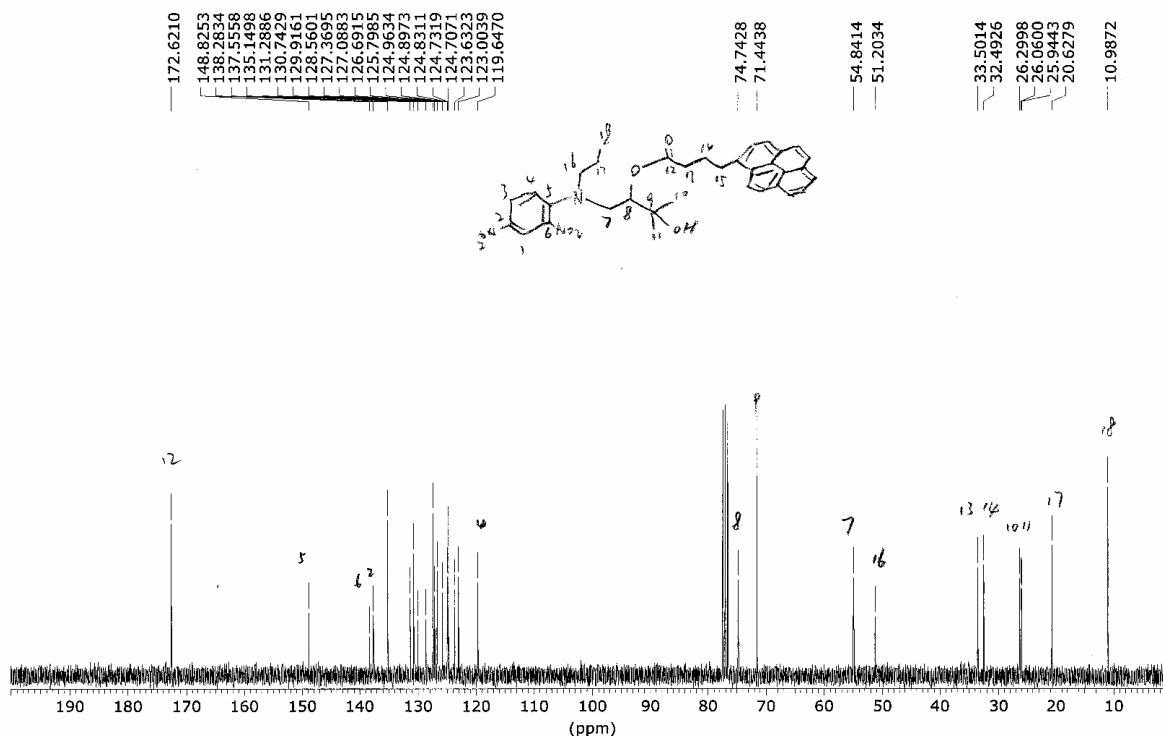
ESI-MS positive mode

Sample dissolved in H₂O/ACN (1+1)
 Max. 956.2 counts.



S43

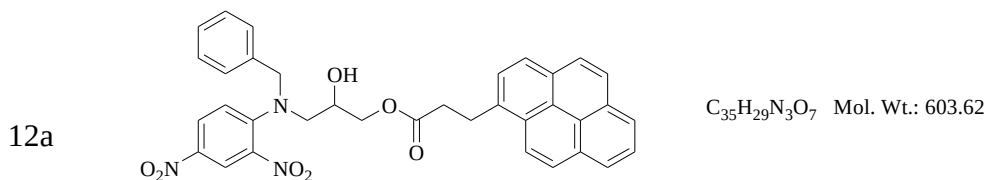
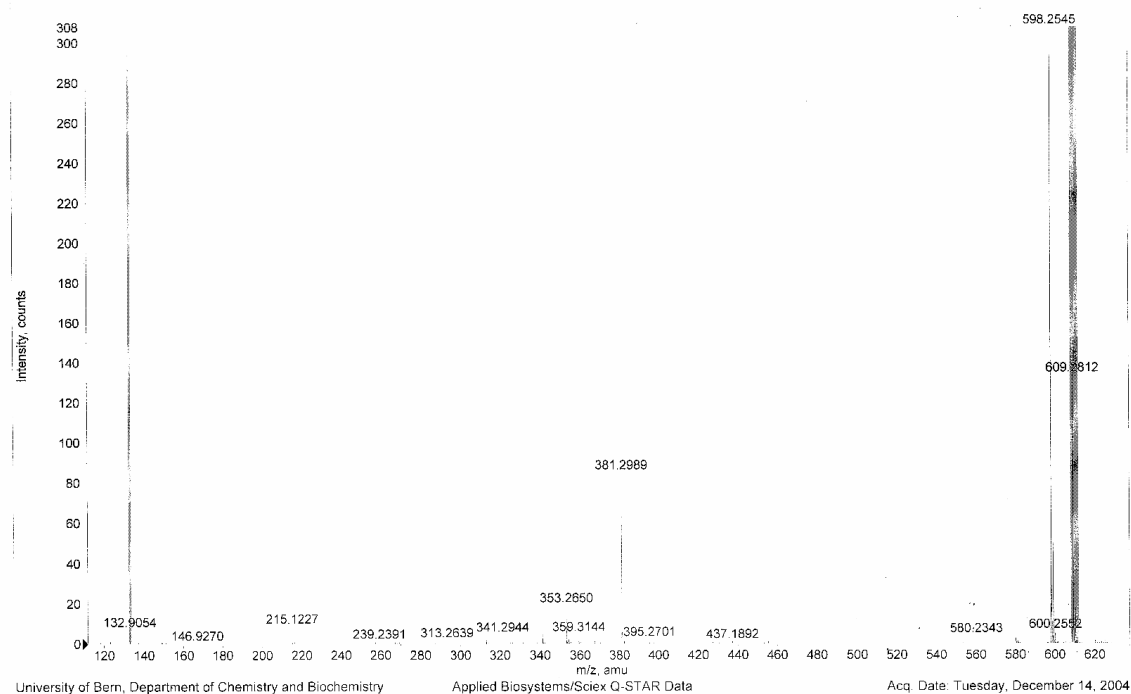
11a

C₃₄H₃₅N₃O₇ Mol. Wt.: 597.66yz-816Proton_ns32 CDCl₃ /disk0 service 103yz-816Carbon_ns512 CDCl₃ /disk0 service 103

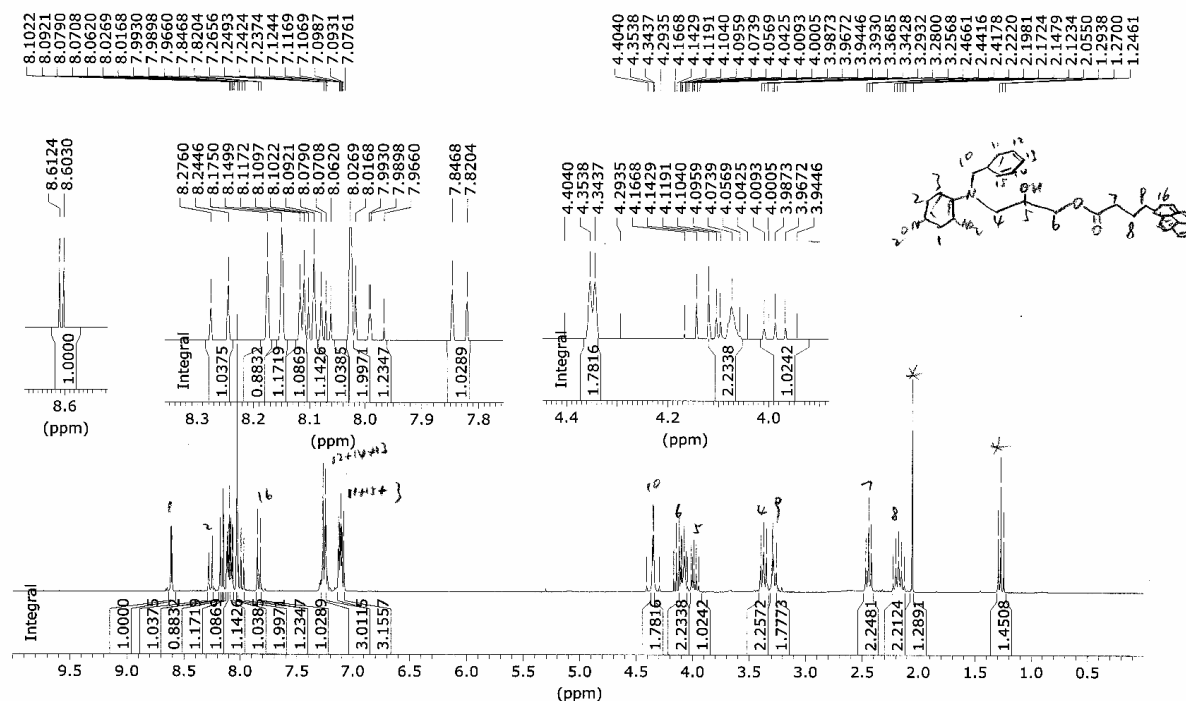
Sample Name: Z. Yang
+TOF MS: 4.234 to 4.334 min from YZ_816_N_B.wiff
a=3.55803463078336600e-004, t0=4.97092404794966570e+000

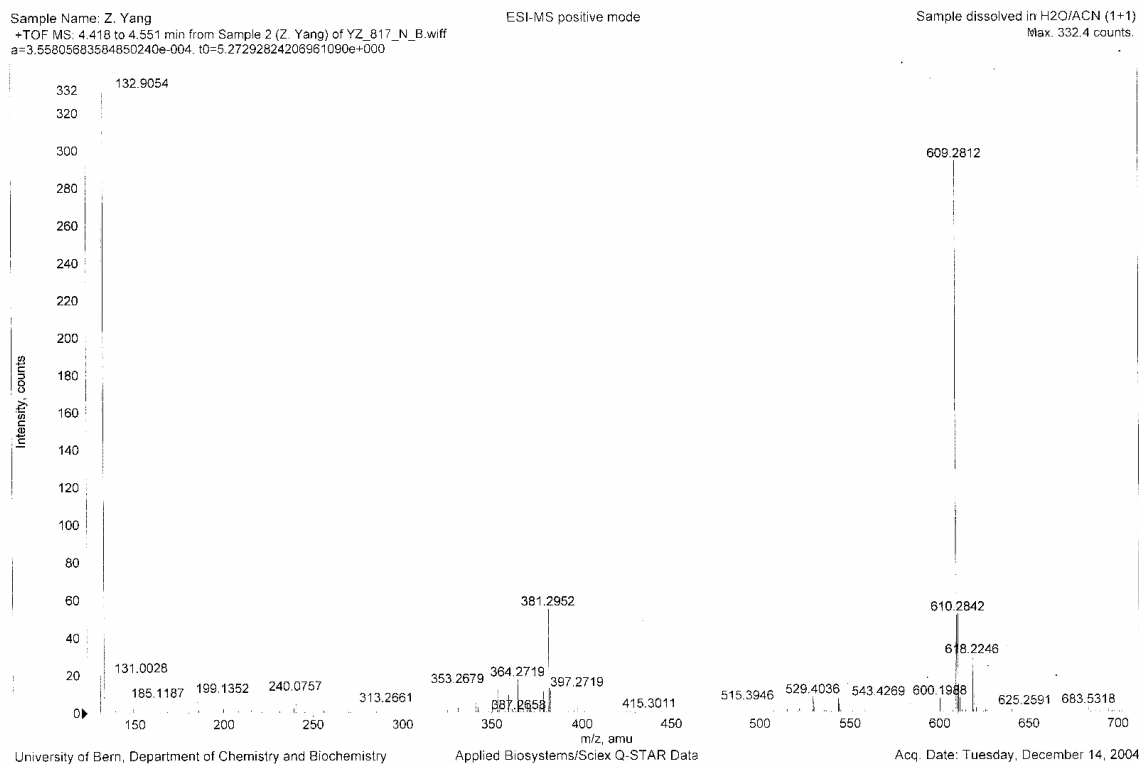
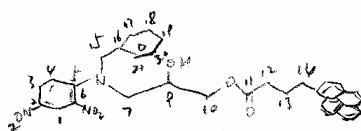
ESI-MS positive mode

Sample dissolved in H₂O/ACN (1+1)
Max. 307.9 counts



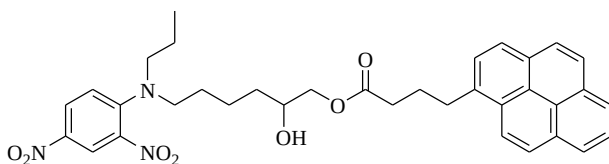
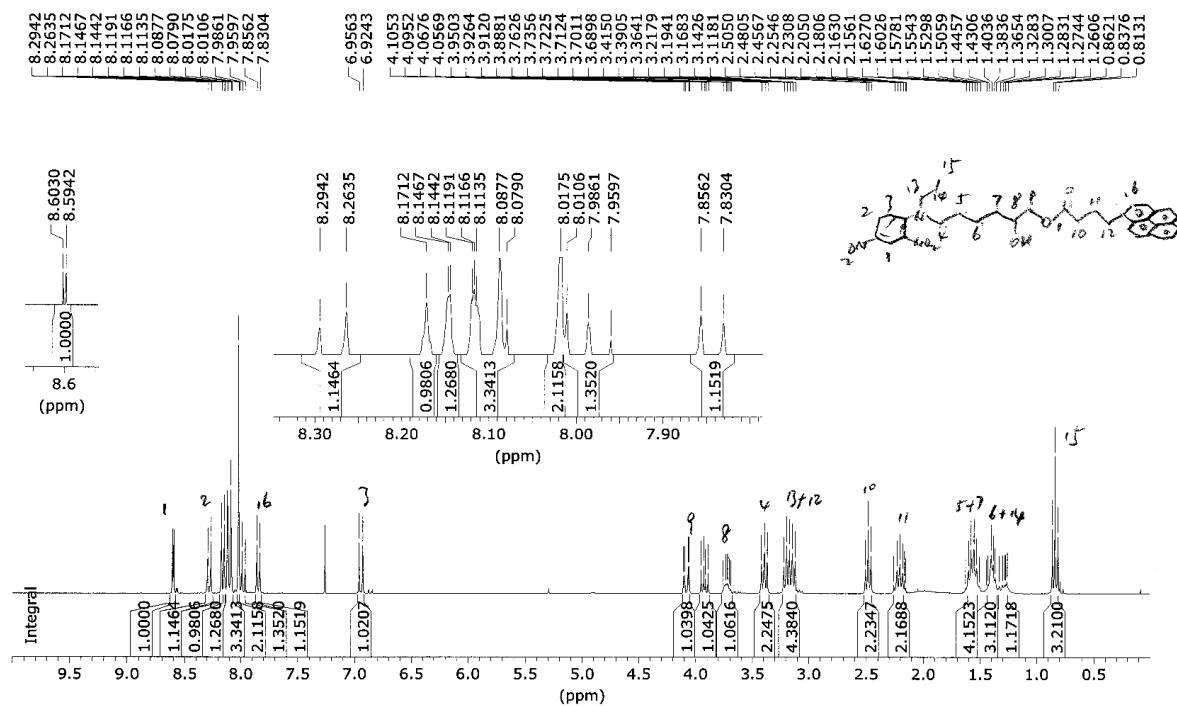
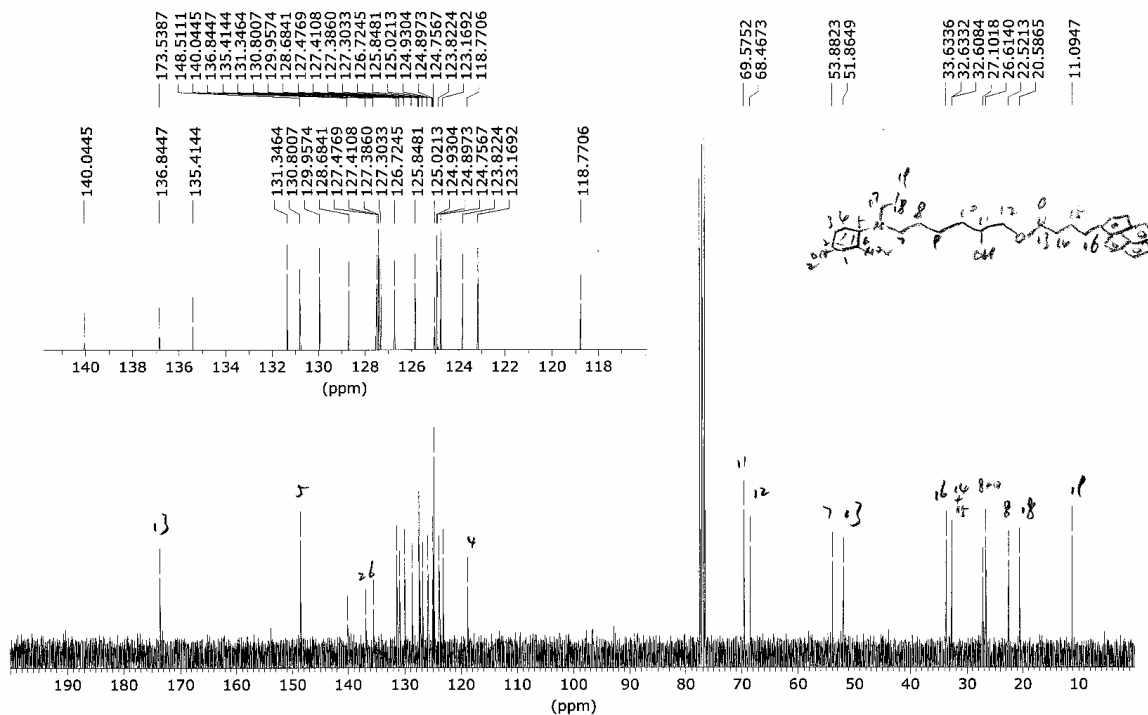
yz-817Proton ns32 CDCl3 /disk0 service 59

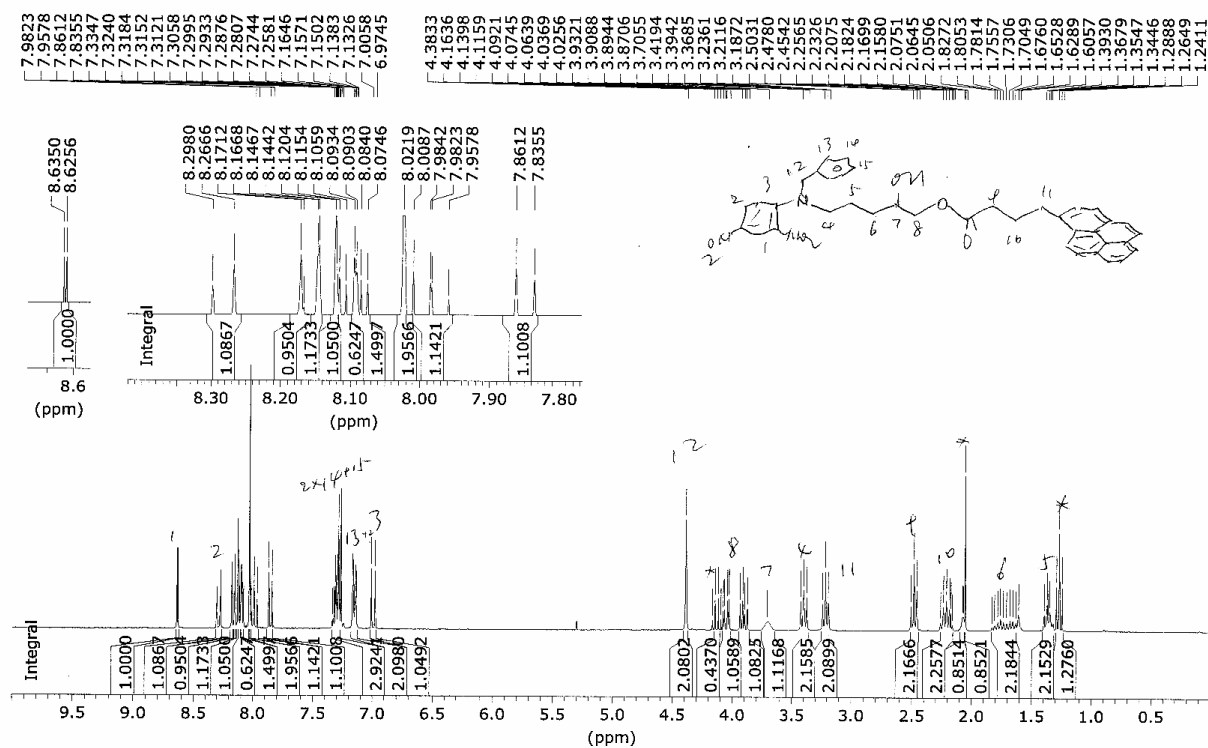
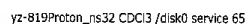




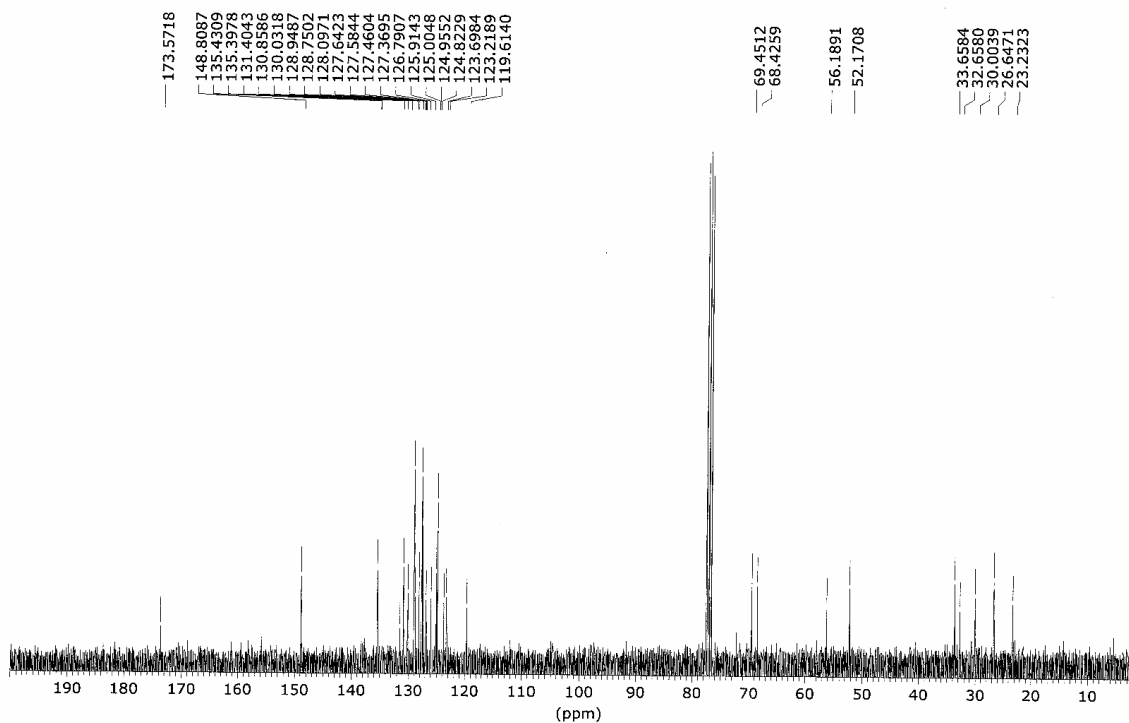
S46

13a

C₃₅H₃₇N₃O₇ Mol. Wt.: 611.68yz-818-2Proton_ns32 CDCl₃ /disk0 service 31yz-818-2Carbon_ns512 CDCl₃ /disk0 service 31



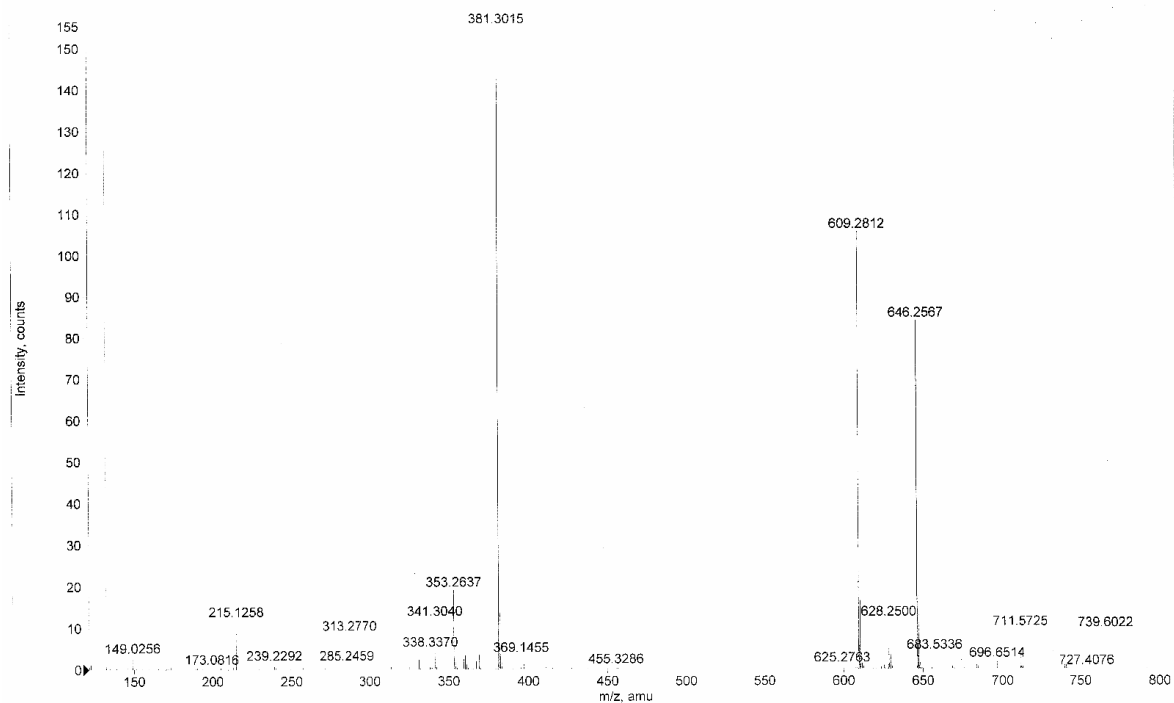
yz-819Carbon_ns512 CDCl3 /disk0 service 65



Sample Name: Z. Yang
+TOF MS: 1.050 to 1.284 min from YZ_819_N_B.wiff
a=3.55800654093799560e-004, t0=4.40045264344735190e+000

ESI-MS positive mode

Sample dissolved in H2O/ACN (1+1)
Max. 155.4 counts.



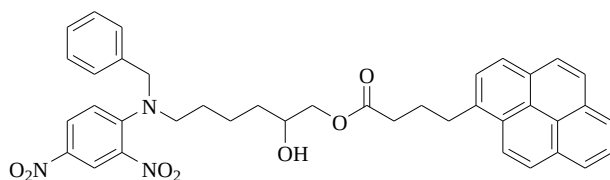
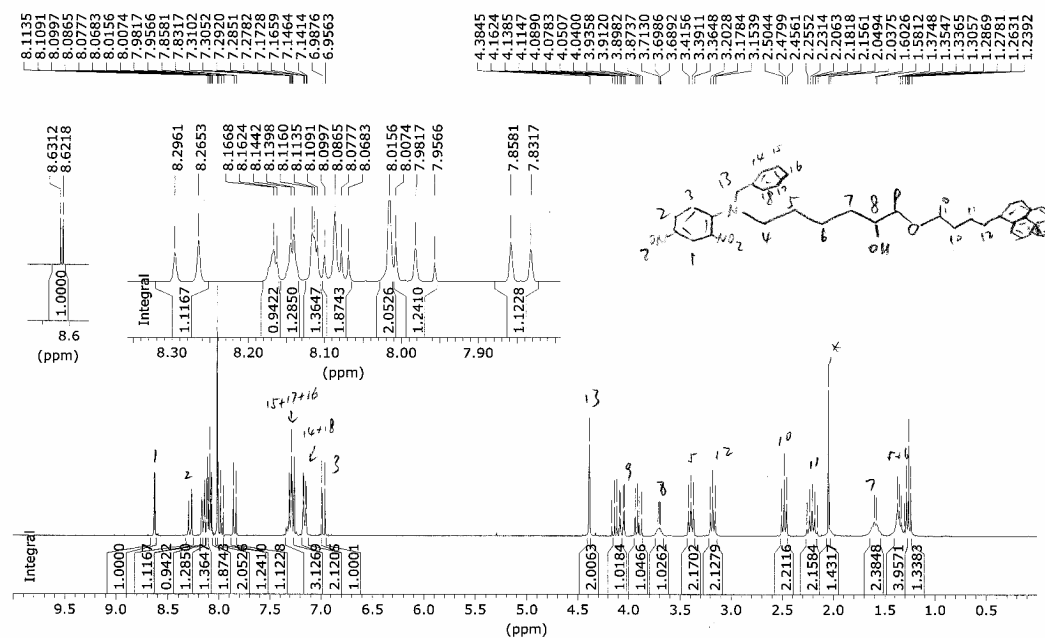
University of Bern, Department of Chemistry and Biochemistry

Applied Biosystems/Sciex Q-STAR Data

Acq. Date: Tuesday, December 14, 2004

S49

15a

C₃₉H₃₇N₃O₇ Mol. Wt.: 659.73yz-820Proton_ns32 CDCl₃/disk0 service 66yz-820Carbon_ns512 CDCl₃/disk0 service 66