Electronic Supplementary Material (ESI) for Dalton Transactions

Functionalization of lanthanide complexes via microwave-enhanced Cu(I)-catalysed azide-alkyne cycloaddition

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Experimental

General Procedures. ^{1}H NMR (300 MHz, 400 MHz or 500 MHz) and ^{13}C NMR (75 MHz, 100 MHz or 125 MHz) spectra were recorded on a Varian 300, a Varian 400 or a Bruker 500 MHz instrument, respectively. Spectra were collected in $CDCl_3$ unless noted otherwise. Chemical shifts were referenced to residual solvent peaks and are given as follows: chemical shift (δ, ppm), multiplicity (s, singlet; br, broad singlet; d, doublet, t, triplet; q, quartet; m, multiplet), coupling constant (Hz), integration. Absorption spectra and fluorescence spectra were collected at room temperature in the solvent indicated at the experiment. HR-ESI-MS analyses were performed on a Bruker MicroTOF ESI mass spectrometer. For accurate mass determination of Eu complexes the ^{153}Eu-isotope was used. Compounds **S1**,[1] **B**,[2] **F**,[3] **1**,[4] **Ln3**,[5] 2-azidoethanol,[6] **10**[7] and **12**[8] were synthesized following literature methods. All other chemicals were from commercial sources and used as received. Microwave heating was performed with a Biotage Initiator instrument. For temperatures and reaction times see Tables 1–2 and the detailed experimental procedures. **CAUTION: Low molecular weight azides are potentially explosive.**

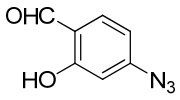
Chromatography. Preparative chromatography was performed using silica (230–400 mesh). Thin layer chromatography was performed on silica-coated aluminum plates. Samples were visualized by UV-light (254 and 356 nm), or staining with $KMnO_4/K_2CO_3$ or cerium ammonium molybdate. The purity of cycloaddition products was assesed by RP-HPLC on an Agilent Technologies 1200 system using an Agilent Eclipse XDB-C18 column (5 μm x 4.6 mm x 150 mm) with water:CH_3CN eluent system (ramping from 100% water to 100% CH_3CN).

Photophysical measurements. UV-Vis absorption spectroscopy was performed

on a Varian Cary 300 instrument; (sh) denotes shoulder to a peak. Steady state and time-resolved emission spectra were collected at room temperature using a Horiba Scientific FluoroMax 4 instrument equipped with a flash lamp. Hydration states (q) were determined according to the method developed by Horrocks,[9] and Parker and Beeby.[10] Emission intensity changes were fitted to single exponential decays using the instrument´s software (FluorEssence Version 3.5.1.20, based on Origin 8.1090). Fitting to double exponential was less suitable based on R^2 and chi.

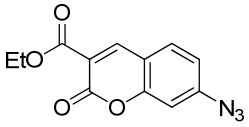
^{1}H NMR experiment for reaction optimization. An NMR-tube was charged with **La2** (20.8 mg, 0.04 mmol), azide **B** (9.8 mg, 0.042 mmol), the Cu-source, ligand, reducing agent and additive in the amounts indicated in Table S1, and DMSO-d_6 (400 μL) was added. The tube was capped, and immersed in a pre-heated oil-bath. ^{1}H NMR spectra were recorded at several time points (10 min, 30 min, 1 h, 2 h, 4 h, 6 h, 12 h, 18 h, 24 h). Conversion was estimated by integrating the signals at 5.50 ppm (s, C$\underline{H}_2$N$_3$, starting material **B**) and at 6.62 ppm (br, C$\underline{H}_2$-triazole **La2B**).

Additional syntheses.



S2. A solution of **S1**[1] (2.69 g, 19.9 mmol) in CH_3CN (50 mL) was treated consecutively with Et_3N (10.7 mL), $MgCl_2$ (2.84 g, 29.9 mmol), and paraformaldehyde (4.11 g, 137 mmol). The mixture was heated at 85 °C for 18 h, and then was allowed to cool back to room temperature. The mixture was diluted with EtOAc, and aqueous HCl (1 M) was added until all solids dissolved. The phases were separated, and the aqueous layer was extracted twice with EtOAc. The combined organic layer was washed with H_2O, and dried ($MgSO_4$). The solvent was removed at reduced pressure, and the dark oily

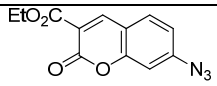
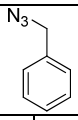
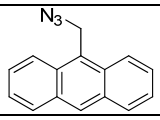
residue was purified by column chromatography [silica, pentane/EtOAc (10:1 → 6:1)] yielding a pale brown solid (2.32 g, 72%): ^{1}H NMR (500 MHz, $CDCl_3$) 6.61 (d, J = 2.1 Hz, 1H), 6.64–6.66 (m, 1H), 7.52 (d, J = 8.4 Hz, 1H), 9.80 (d, J = 0.5 Hz, 1H), 11.31 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) 107.2, 111.3, 118.1, 135.5, 149.0, 163.3, 194.9; HR-ESI-MS calcd 162.0309 obsd 162.0302 [$(M + H)^+$, M = $C_7H_4N_3O_2$].



S3. A mixture of **S2** (322 mg, 1.98 mmol), diethyl malonate (948 mg, 5.93 mmol) and 3Å molecular sieves (1 g) in CH_3CN (3.5 mL) was treated with piperidine (34 mg, 0.39 mmol). The mixture was heated at 65 °C for 24 h. The reaction mixture was cooled back to room temperature, filtered, concentrated, and the dark orange oily residue was purified by column chromatography [silica, pentane/EtOAc (3:1)] affording an orange solid (318 mg, 62%): ^{1}H NMR (500 MHz, $CDCl_3$) 1.41 (t, *J* = 7.2 Hz, 3H), 4.41 (q, *J* = 7.2 Hz, 2H), 6.96–7.00 (m, 2H), 7.58 (d, *J* = 9.0 Hz, 1H), 8.49 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) 14.3, 62.1, 106.8, 115.0, 116.2, 116.8, 131.0, 146.8, 148.1, 156.3, 156.5, 163.1; HR-ESI-MS calcd 282.0485 obsd 282.0500 [$(M + Na)^+$, M = $C_{12}H_9N_3O_4$].

La2B. ^{1}H NMR (500 MHz, DMSO-d_6) 2.07–4.11 (m, 24H), 6.74 (br, 2H), 7.57 (t, J = 7.6 Hz, 2H), 7. 62 (s, 1H), 7.70 (t, J = 7.1 Hz, 2H), 8.14 (d, J = 8.5 Hz, 2H), 8.51 (d, J = 8.6 Hz, 2H), 8.68 (s, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 46.9–54.3 (br m), 122.8, 125.3, 127.7, 129.3, 129.9, 130.7, 131.6.

Table S1. Screening of cycloaddition reaction conditions.

Azide					
Lanthanide complex	**Ln1**	**Ln2**	**Ln1**	**Ln2**	**Ln2**
Cu-source, additives	*Solvent, temperature, heating method, observations*				
$CuSO_4 \cdot 5H_2O$, NaAsc,TBTA	$H_2O/^tBuOH$/THF, RT	$H_2O/^tBuOH$/THF, 85 °C (benzoic acid instead of TBTA)			
	MeOH, RT				
$Cu(OAc)_2$, NaAsc, TBTA	$H_2O/^tBuOH$/THF, RT				
	MeOH, RT				
CuCl, TBTA	$H_2O/^tBuOH$/THF, RT		DMF, 85 °C (MW)		
	MeOH, RT				
CuCl, benzoic acid		$H_2O/^tBuOH$/THF, 120 °C (MW)			
Cu_2O, benzoic acid		$H_2O/^tBuOH$/THF, 120 °C (MW)			
CuBr, thioanisole		$H_2O/^tBuOH$/THF, 120 °C (MW)			
CuOAc, TBTA	$H_2O/^tBuOH$/THF, 100 °C (MW)	$H_2O/^tBuOH$/THF, 120 °C (MW)	$H_2O/^tBuOH$/THF RT at 85 °C (MW)	$H_2O/^tBuOH$/THF, 100 °C (MW)	
$Cu(acac)_2$, DIPEA, TMEDA	DMSO, 85 °C	DMF, 100 °C (MW)	DMSO, RT	DMF, 100 °C (MW)	
	DMF, 85 °C		DMF, RT		
	DMSO, 100 °C (MW)		DMF, 100 °C (2h, MW)		
	DMF, 100 °C (MW)				
	DMF + benzoic acid, 100 °C (MW)				
$Cu(OAc)_4$, NaAsc					$H_2O/^tBuOH$/DMF (1:1:1), RT, 26 h
CuI (10%)					DMSO, 80 °C, 3 h (*NMR exp*)
					DMSO, DIPEA (70 μL), 85 °C, overnight (39% conversion, *NMR exp*)
$Cu(OAc)_4$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), 85 °C, 21 h (50% conversion, *NMR exp*)
$CuSO_4 \cdot 5H_2O$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), TMEDA (10%) 85 °C, 21 h (63% conversion, *NMR exp*)
$CuBr_2$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), TMEDA (10%) 85 °C, 21 h (81% conversion, *NMR*

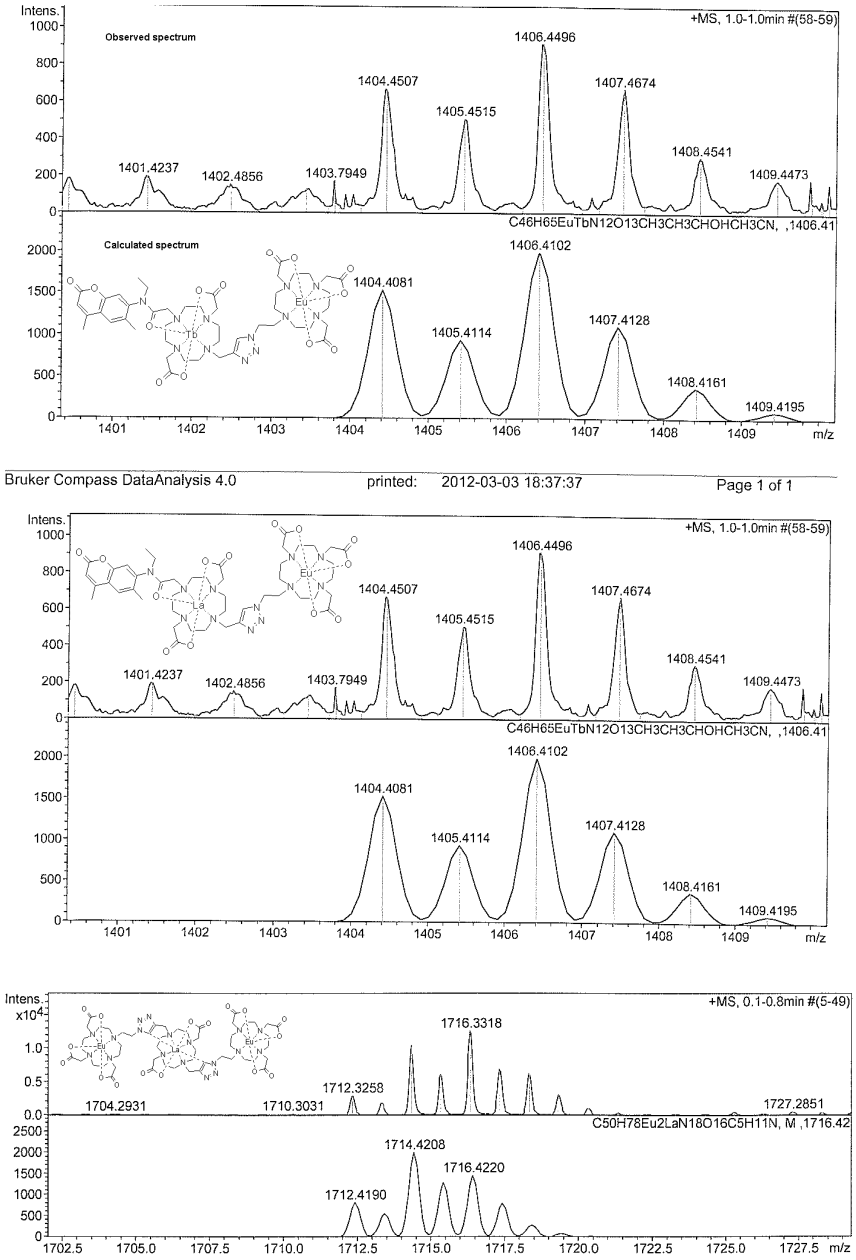
					exp); attempted isolation gave poor yield
$CuSO_4$ (anhydrous, 10%), NaAsc (25%)					DMSO, DIPEA (70 μL), TMEDA (10%) 85 °C, 21 h (65% conversion, *NMR exp*)
$Cuacac_2$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), TMEDA (10%) 85 °C, 21 h (55% conversion, *NMR exp*)
$Cuacac_2$ (10%), NaAsc (25%)					DMSO, TMEDA (10%) 85 °C, 12 h (immediate precipitate formation, **B** only seen, *NMR exp*)
$Cuacac_2$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), 85 °C, 21 h (61% conversion, *NMR exp*)
$Cuacac_2$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), TMEDA (10%) 85 °C, 21 h (55% conversion, *NMR exp*)
$Cuacac_2$ (10%), NaAsc (25%)					DMSO, DIPEA (70 μL), phen (12%) 85 °C, 20 h (68% conversion, *NMR exp*)
CuI (5%),				$^iPr_2NH$:CH_3CN (4:1), 80 °C, oil bath, 22 h, incomplete conv.	

Colour code Product isolated in good yield. Product detected by MS, low conversion (not isolated). Conversion determined by integration of starting material and product peaks. Trace amounts of product after 1 h, < 30% conversion after 4 h as determined by TLC and ESI-MS analysis of the reaction mixture. In all other cases: No product observed by ESI-MS and TLC analysis.

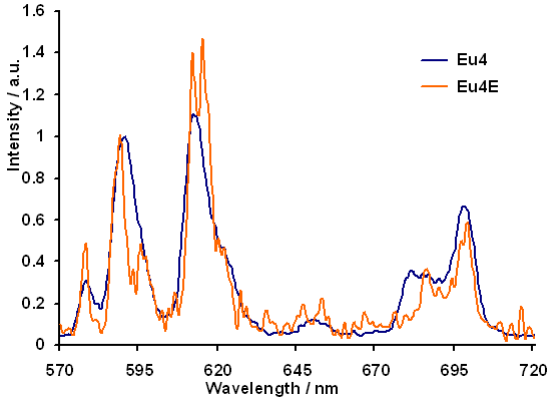
Table S2 Optimisation of cycloaddition reaction.

Entry	Reactants	Cu-source	Additive	Solvent	T (°C)	Time	Conversion[a] (yield)[b]
1	**La2** + **B**	$Cu(OAc)_2$[c]	$Et_2{}^iPrN$	H_2O / ^{t}BuOH / DMF	r.t.	22 h	44%
2	**La2** + **B**	$Cu(acac)_2$[c]	$Et_2{}^iPrN$, TMEDA	DMSO	85	4h	63%
3	**La2** + **B**	CuI	-	DMSO	80	3 h	0%
4	**La2** + **B**	CuI	$Et_2{}^iPrN$	DMSO	85	16 h	0%
5	**La2** + **A**	CuI	Piperidine	CH_3CN	100	20 min	100%[d] (62%)
6	**La2** + **A**	CuI	Piperidine	CH_3CN	100	10 min	100%[d] (67%)
7	**La2** + **A**	CuI	Piperidine	CH_3CN	100	5 min	100%[d] (72%)
8	**La2** + **A**	CuI	$^iPr_2NH$	CH_3CN	100	5 min	(61%)[d]
9	**La2** + **A**	CuI	Et_3N	CH_3CN	100	5 min	(55%)[d]
10	**La2** + **A**	CuI	$Et_2{}^iPrN$	CH_3CN	100	5 min	0%[e]

[a] Conversion determined by ^{1}H NMR analysis. [b] Isolated yield after precipitation. [c] With NaAsc. [d] Some starting material observed by ESI-MS analysis. [e] **La2** recovered.



Simulated and observed HR-ESI-MS spectra of biheterometallic compelxes **EuTb17** and **LaEu17**, and triheterometallic complex **Eu_2La18**.



Normalized emission spectra of **Eu4**(λ_{ex} = 397 nm, delay time 0.05 ms) and **Eu4E**. (λ_{ex} = 280 nm, delay time 0.05 ms).

References

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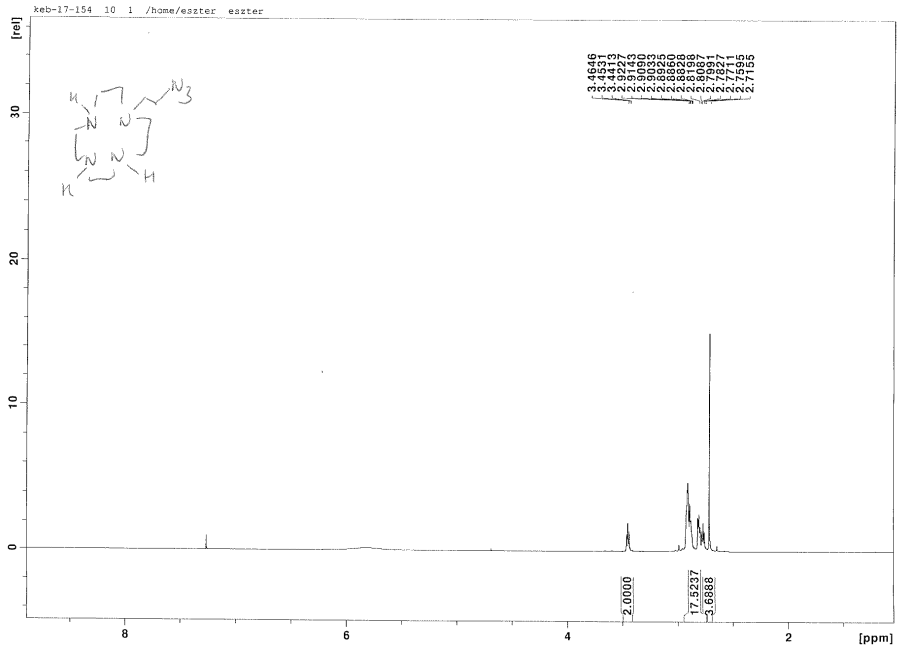
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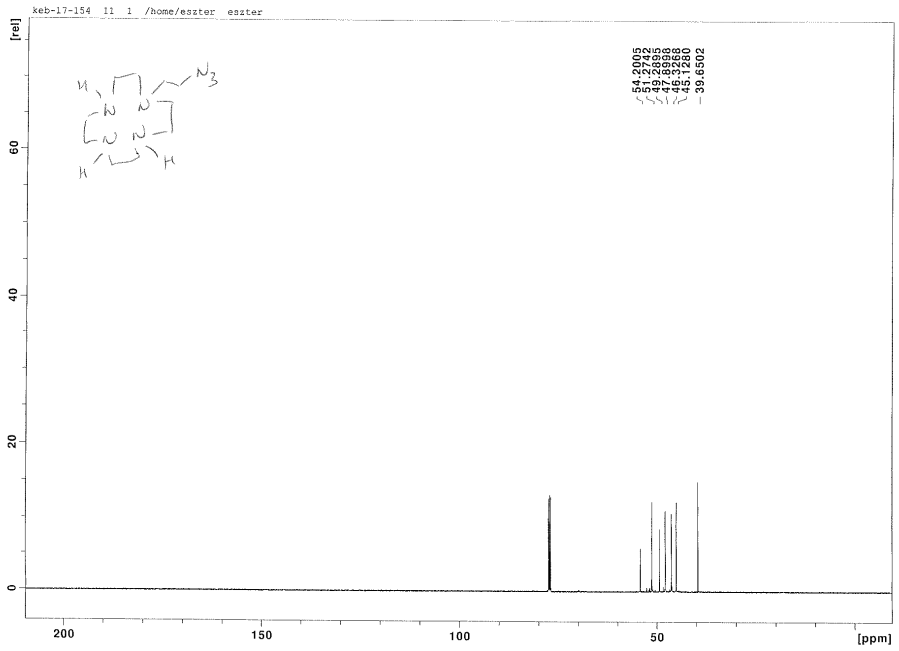
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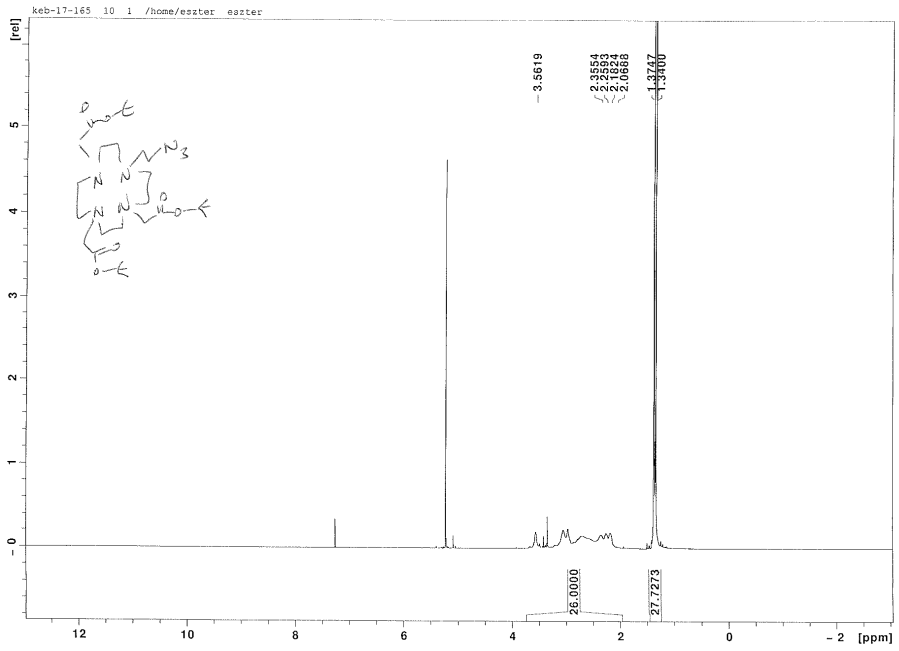
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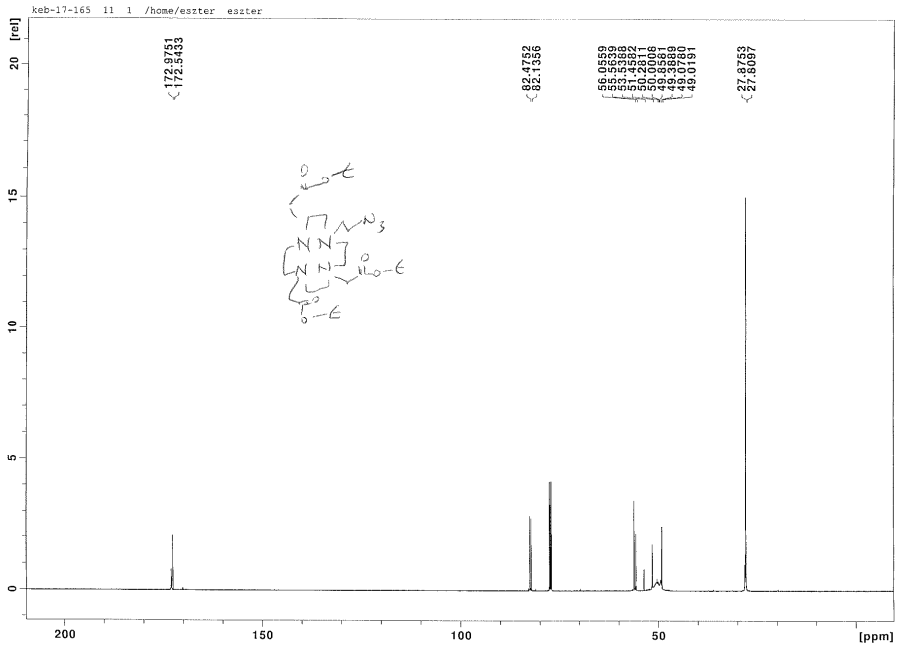
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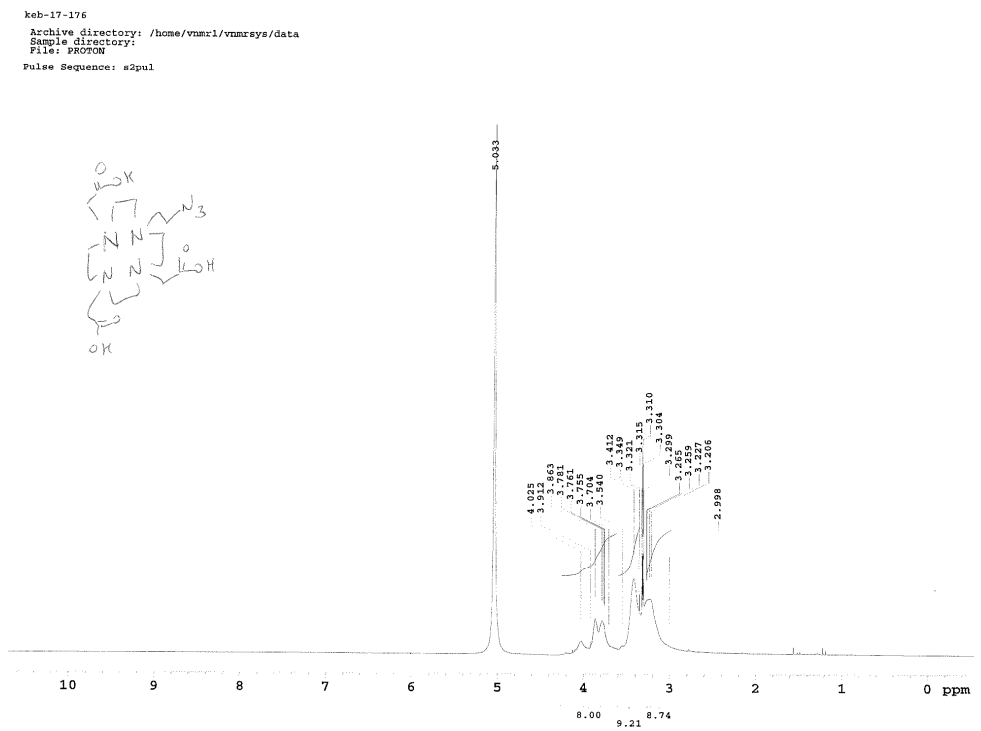
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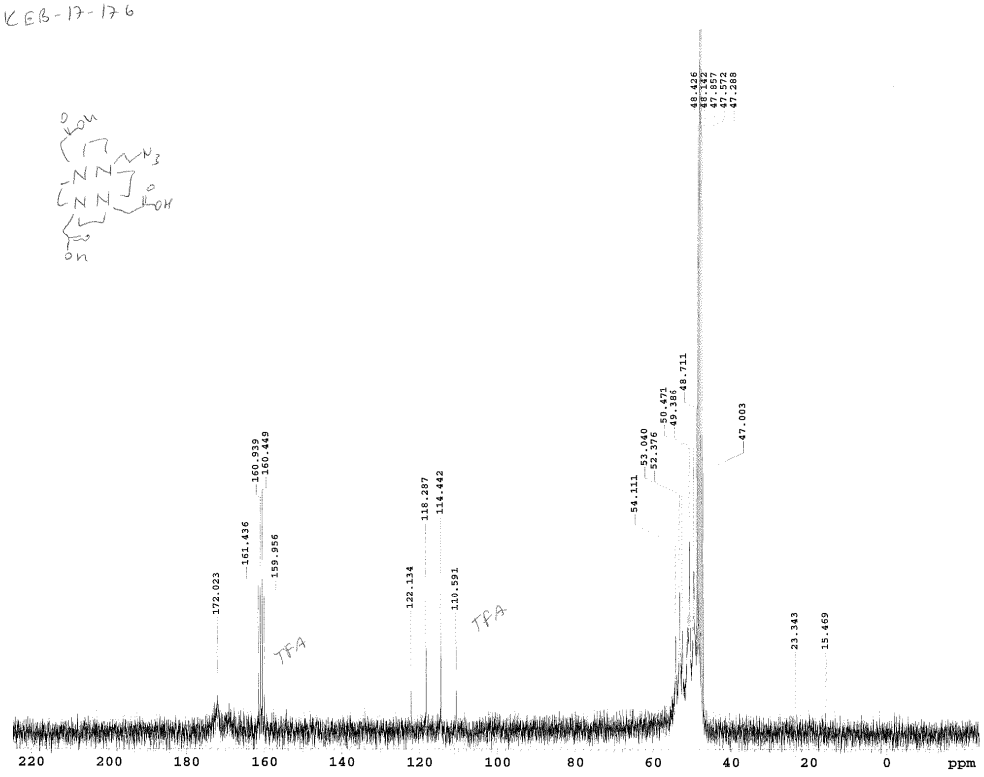
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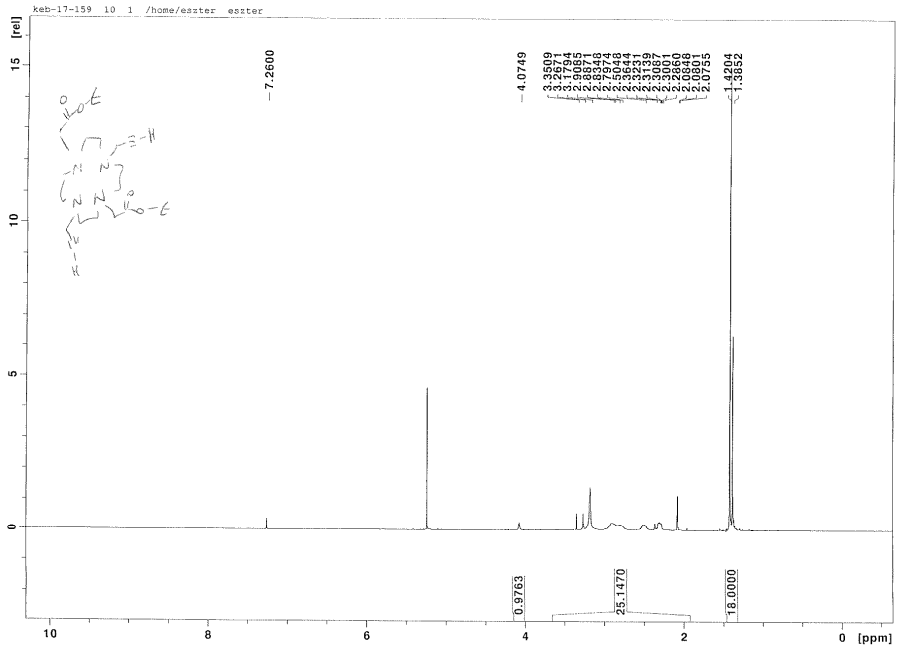
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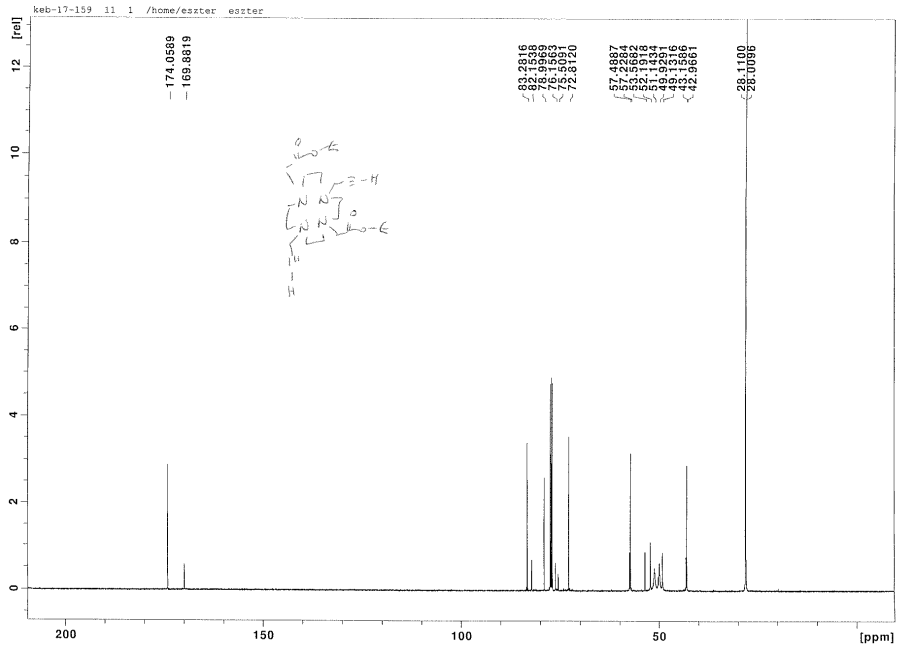
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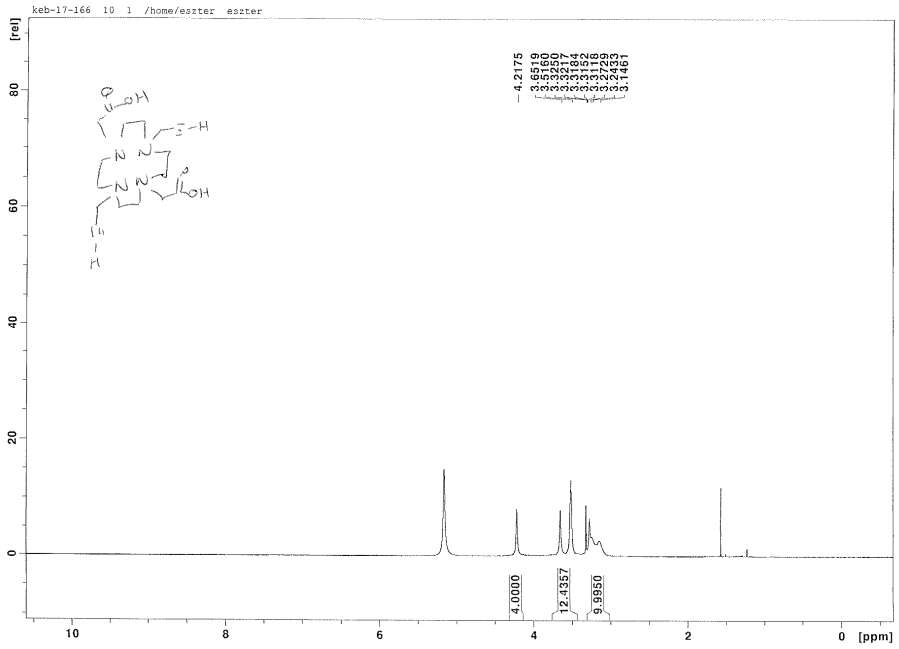
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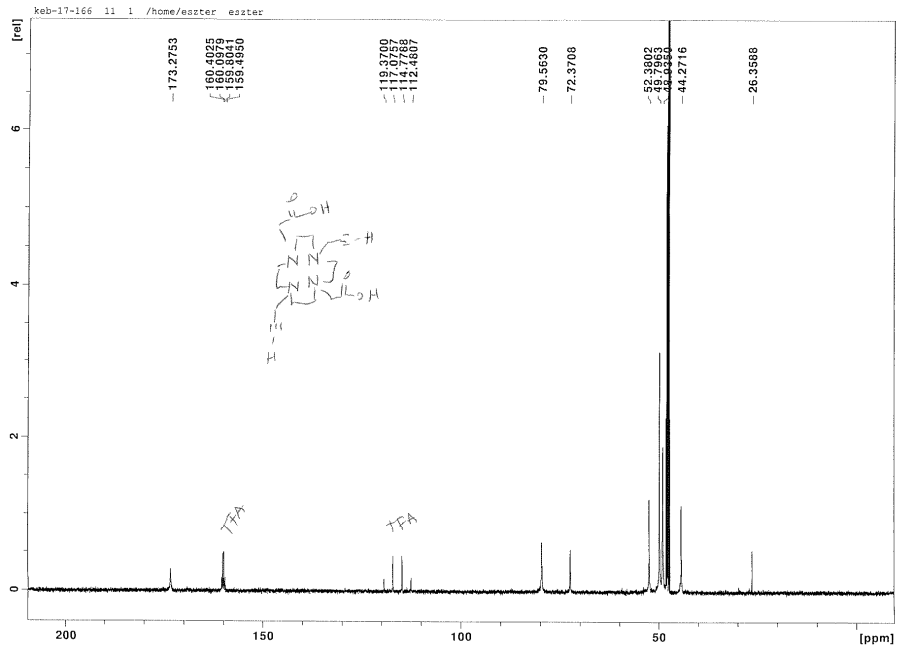
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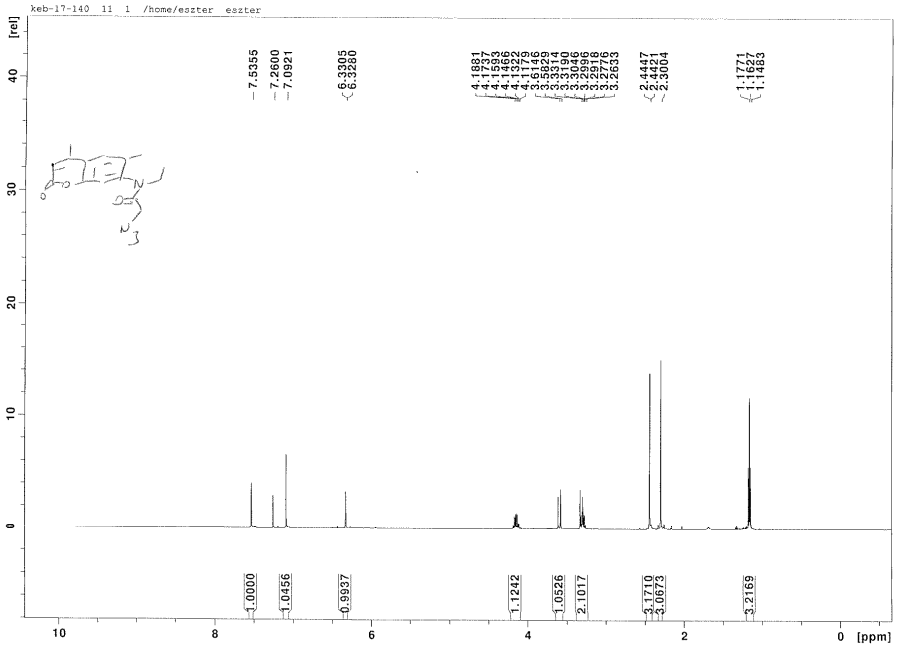
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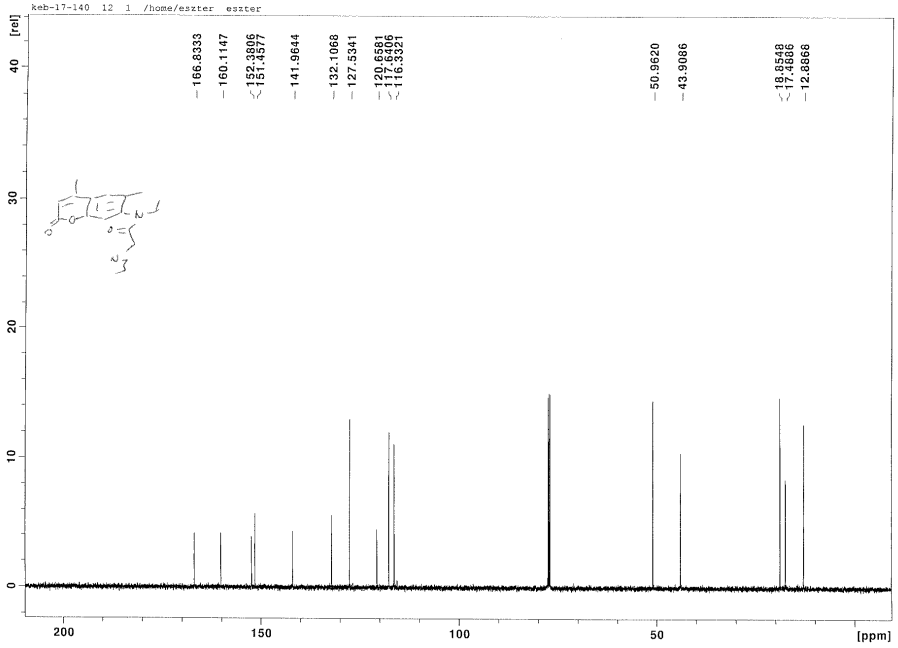
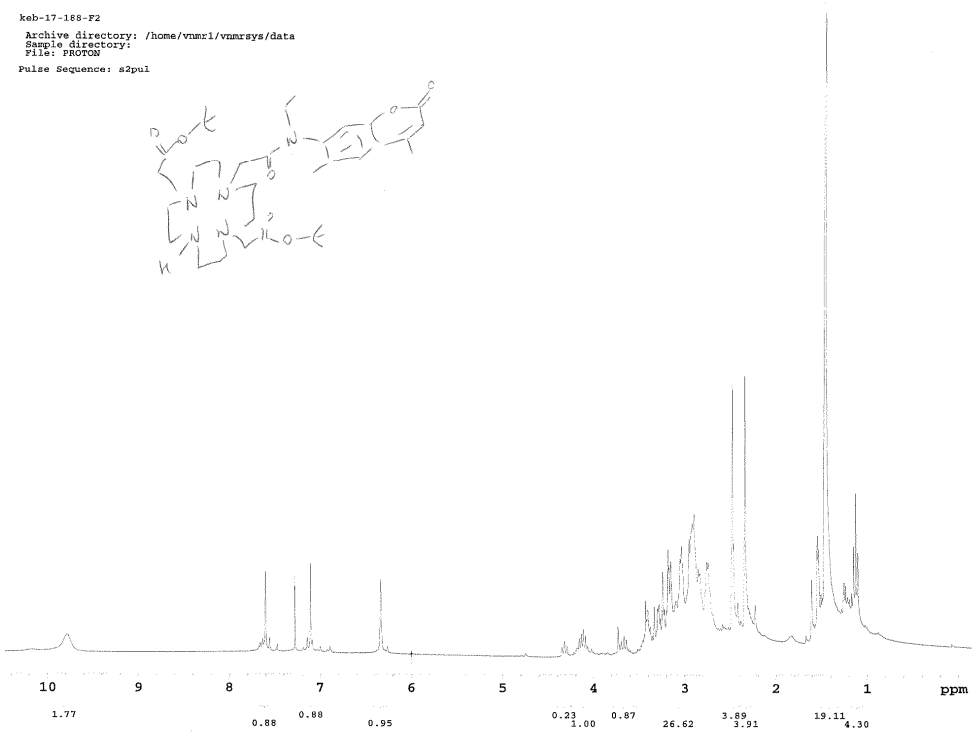
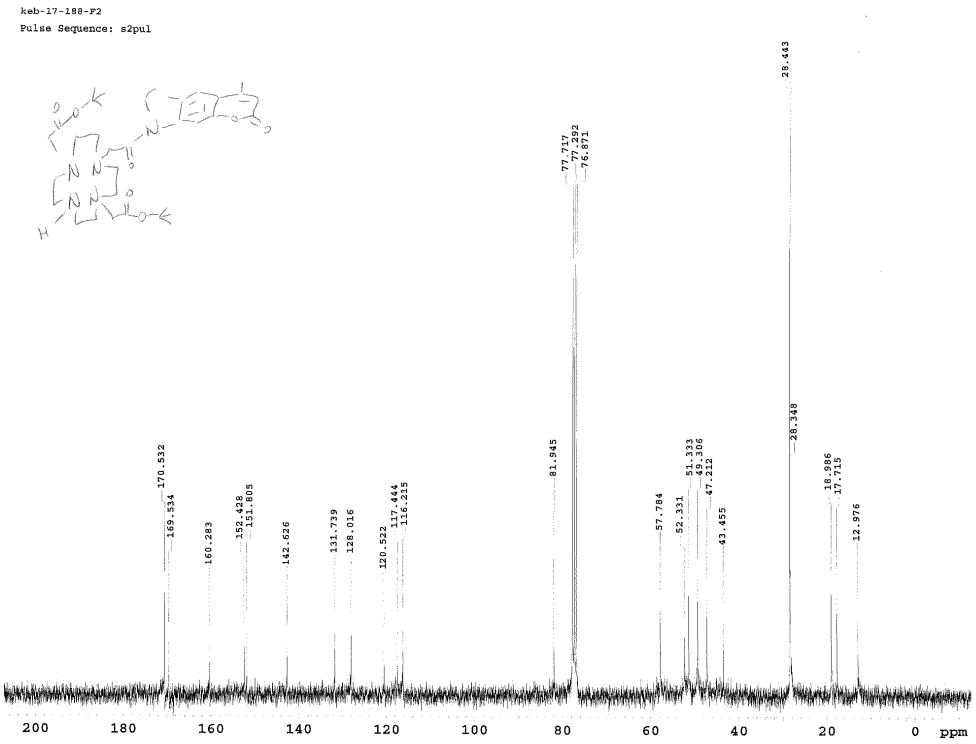
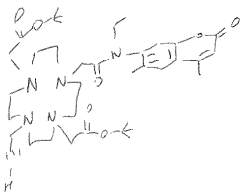
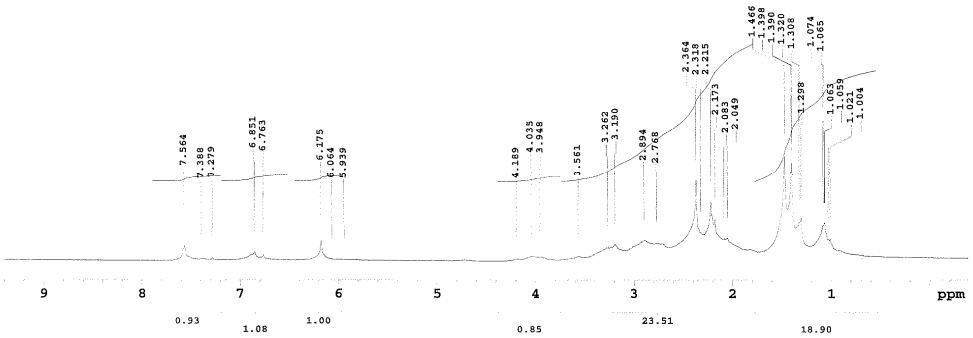

keb-17-140 12 1 /home/eszter eszter
[rel]
166.8333
160.1147
152.3806
151.4577
141.9644
132.1068
127.5341
120.6581
117.6406
116.3321
50.9620
43.9086
19.8548
17.4886
12.8868
[ppm]


keb-17-188-F2
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
10
9
8
7
6
5
4
3
2
1
ppm
1.77
0.88
0.88
0.95
0.23
1.00
0.87
26.62
3.89
3.91
19.11
4.30

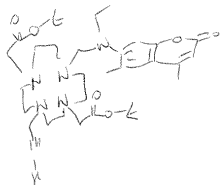

keb-17-188-F2
Pulse Sequence: s2pul
170.532
169.534
160.283
152.428
151.805
142.626
131.739
128.016
120.522
117.444
116.215
81.945
77.717
77.292
76.871
57.784
52.331
51.333
49.306
47.212
43.455
28.443
28.348
18.986
17.715
12.976
200
180
160
140
120
100
80
60
40
20
0
ppm

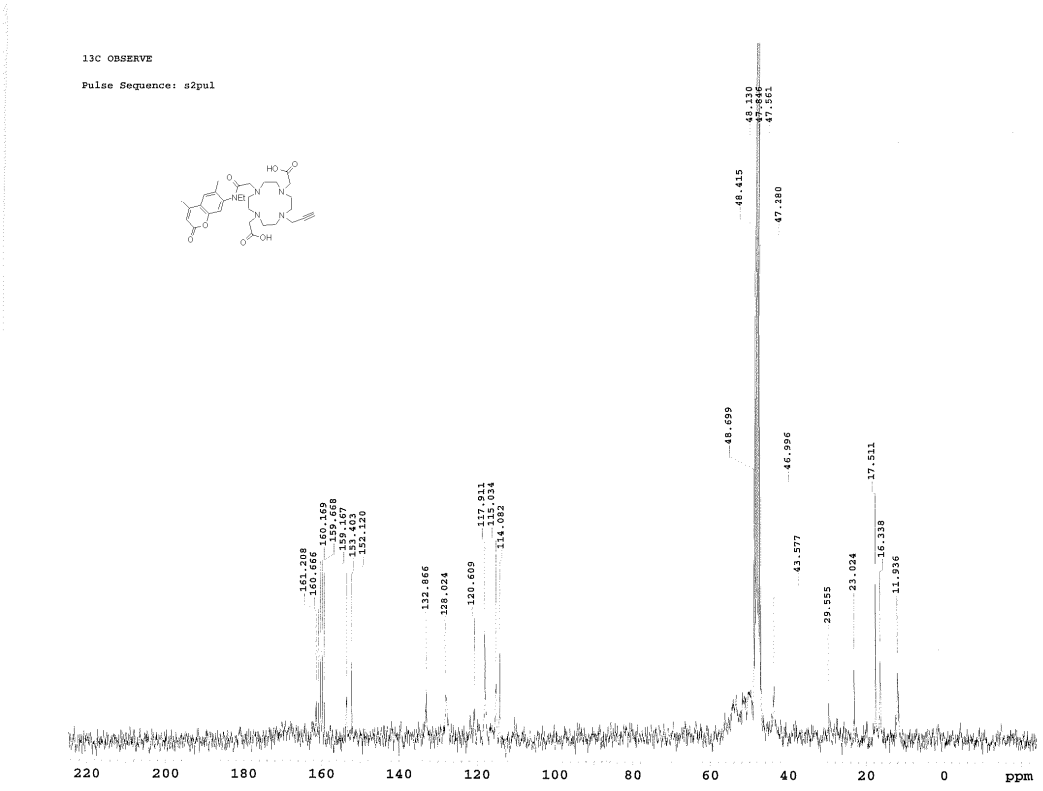
tetraalkyl
Pulse Sequence: s2pul

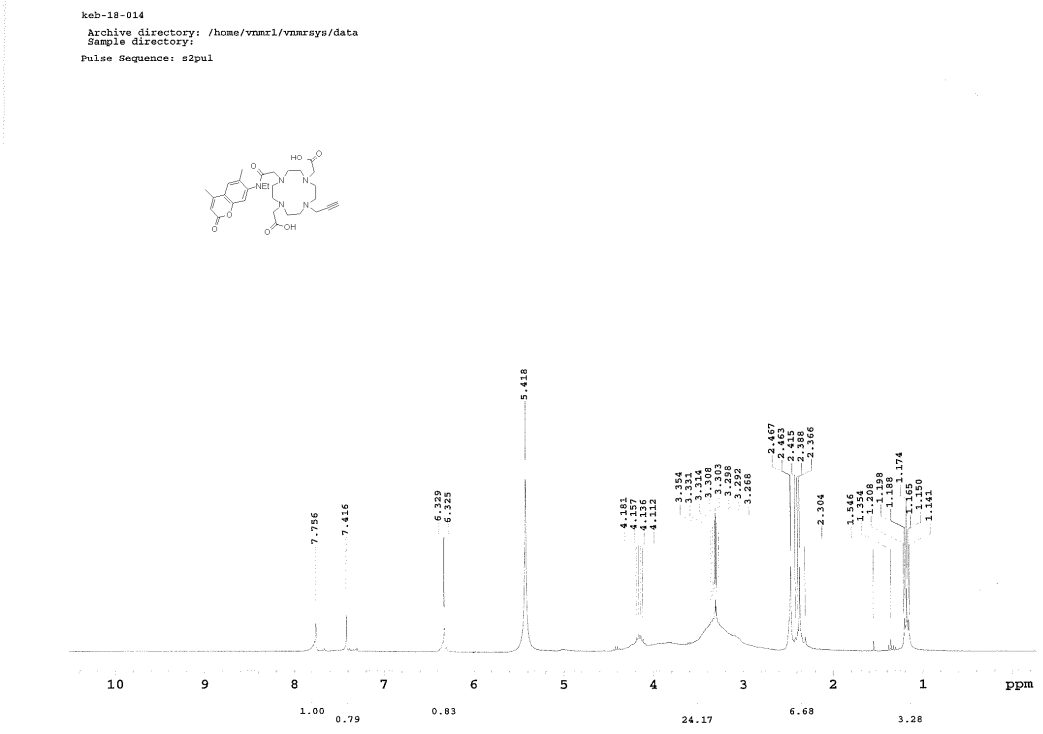


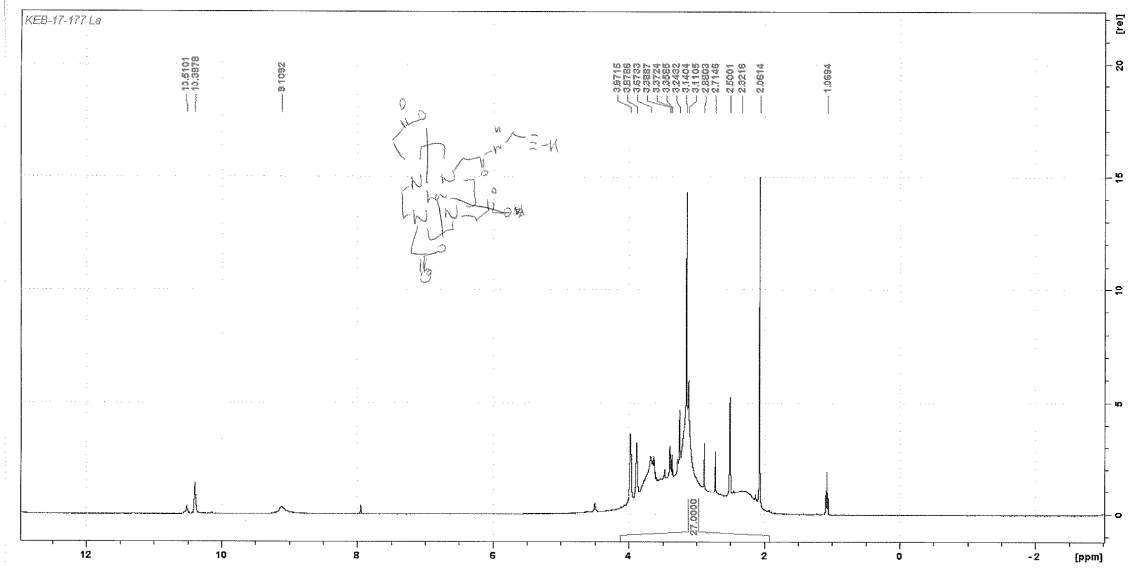


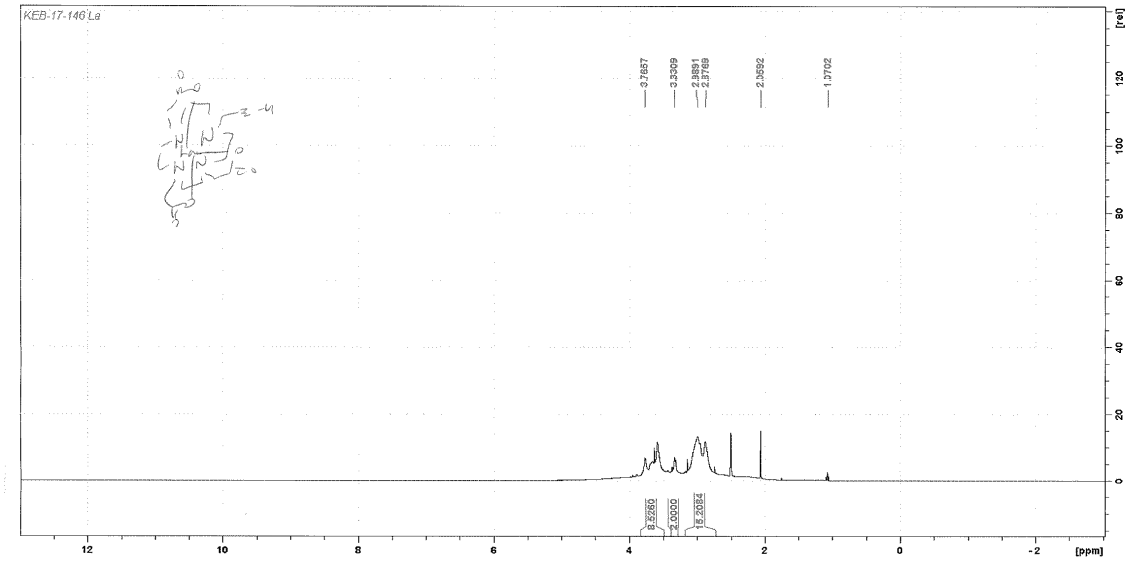
keb-17-194
Pulse Sequence: s2pul

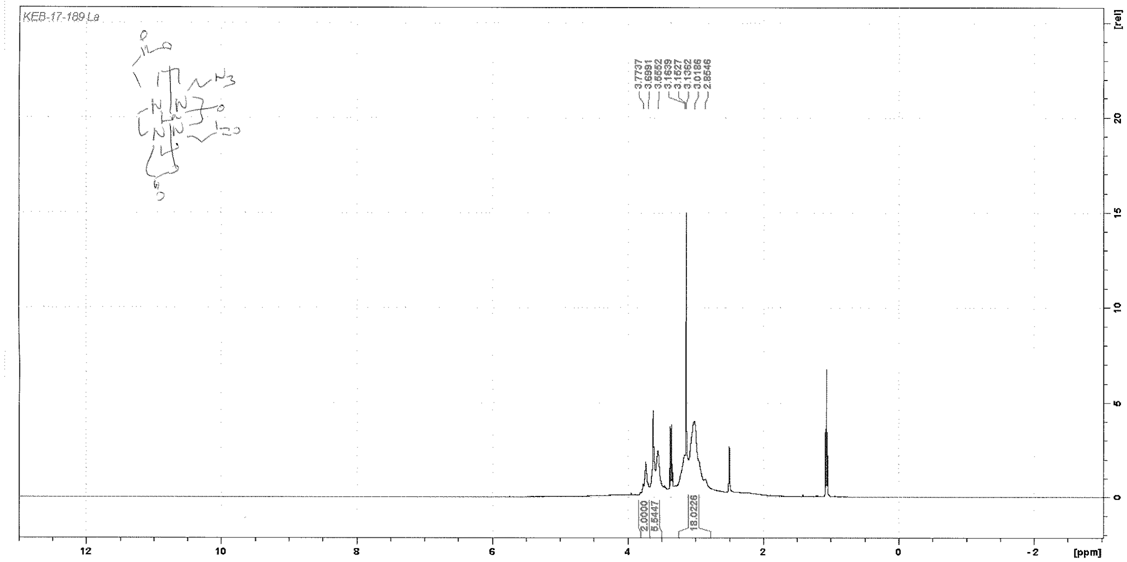


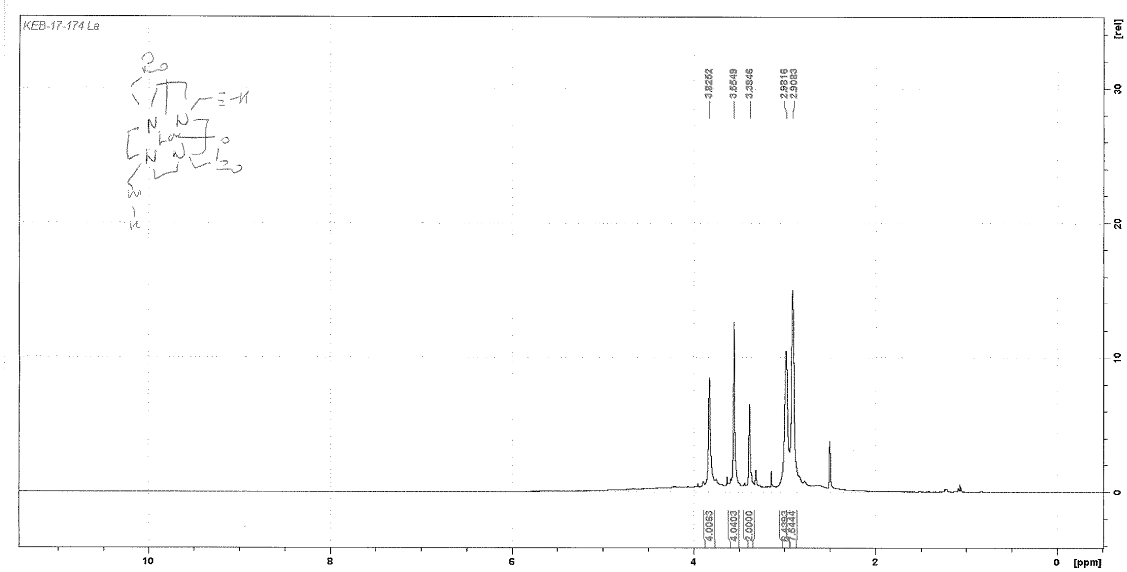

13C OBSERVE
Pulse Sequence: s2pul
48.130
47.846
47.561
48.415
47.280
48.699
46.996
17.511
16.338
11.936
43.577
29.555
23.024
161.208
160.666
160.169
159.668
159.167
133.403
152.120
132.866
128.024
120.609
117.911
115.034
114.002
220
200
180
160
140
120
100
80
60
40
20
0
ppm

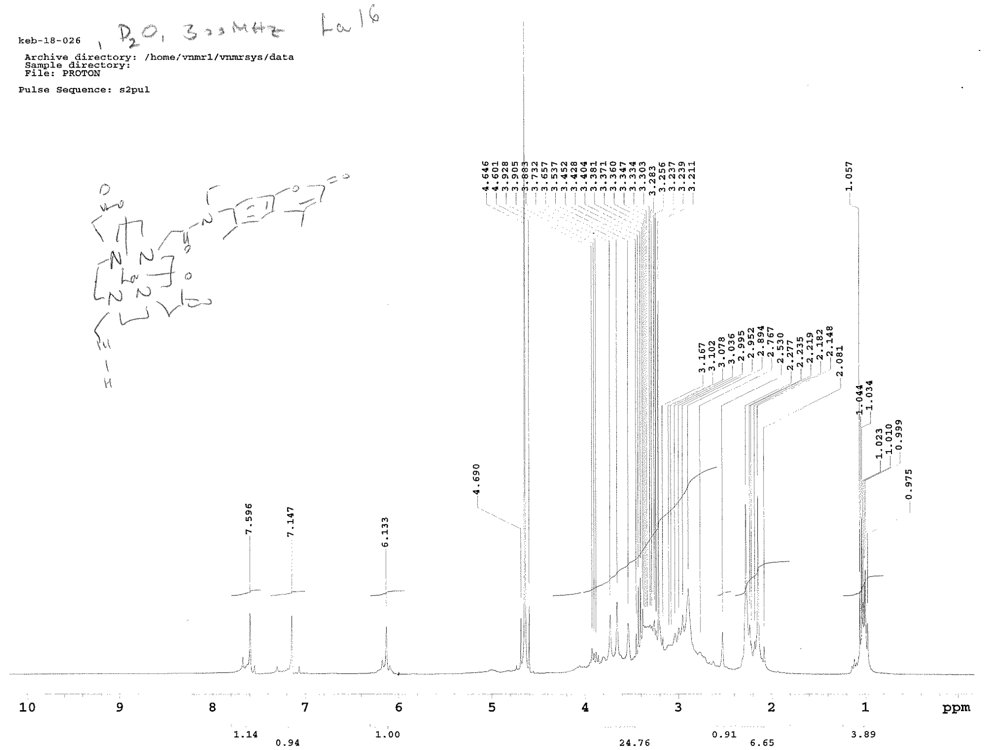

keb-18-014
Archive directory: /home/vnmrl/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
5.418
7.756
7.416
6.329
6.325
4.161
4.157
4.136
4.112
3.354
3.331
3.314
3.310
3.303
3.298
3.292
3.266
2.467
2.465
2.415
2.388
2.366
2.304
1.566
1.354
1.208
1.198
1.188
1.174
1.165
1.150
1.141
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.79
0.83
24.17
6.68
3.28

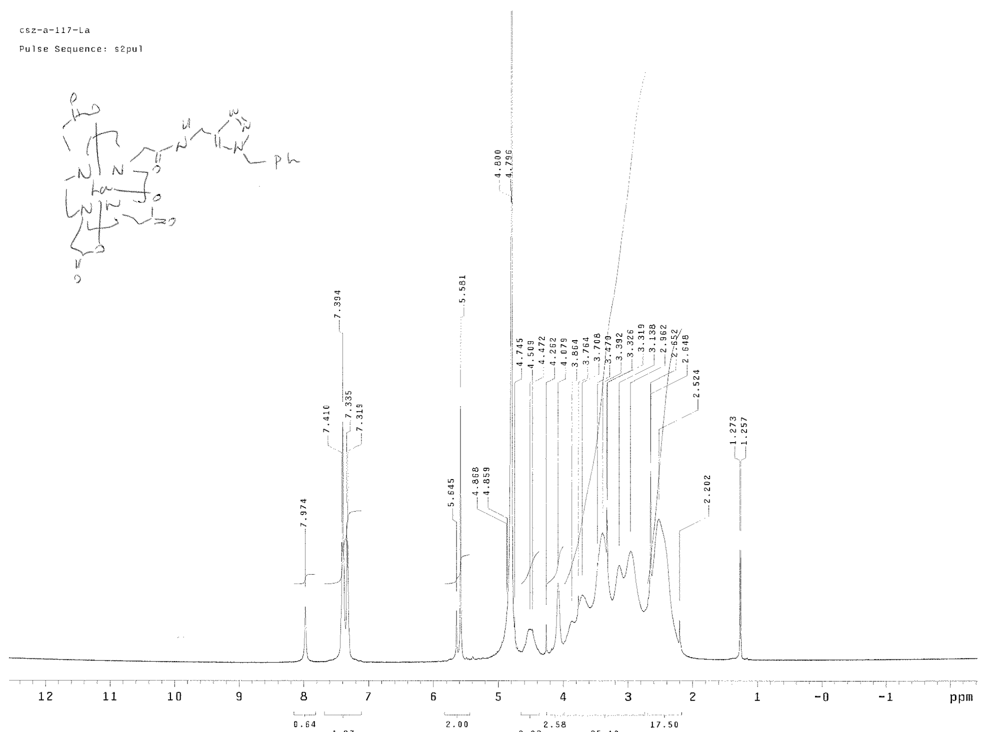

KEB-17-177 La
10.5101
10.3878
9.1092
3.8715
3.8736
3.8733
3.8897
3.8774
3.3585
3.2482
3.1104
2.8803
2.7146
2.5001
2.3216
2.0614
1.0694
27.0000
12
10
8
6
4
2
0
-2
[ppm]
[rel]

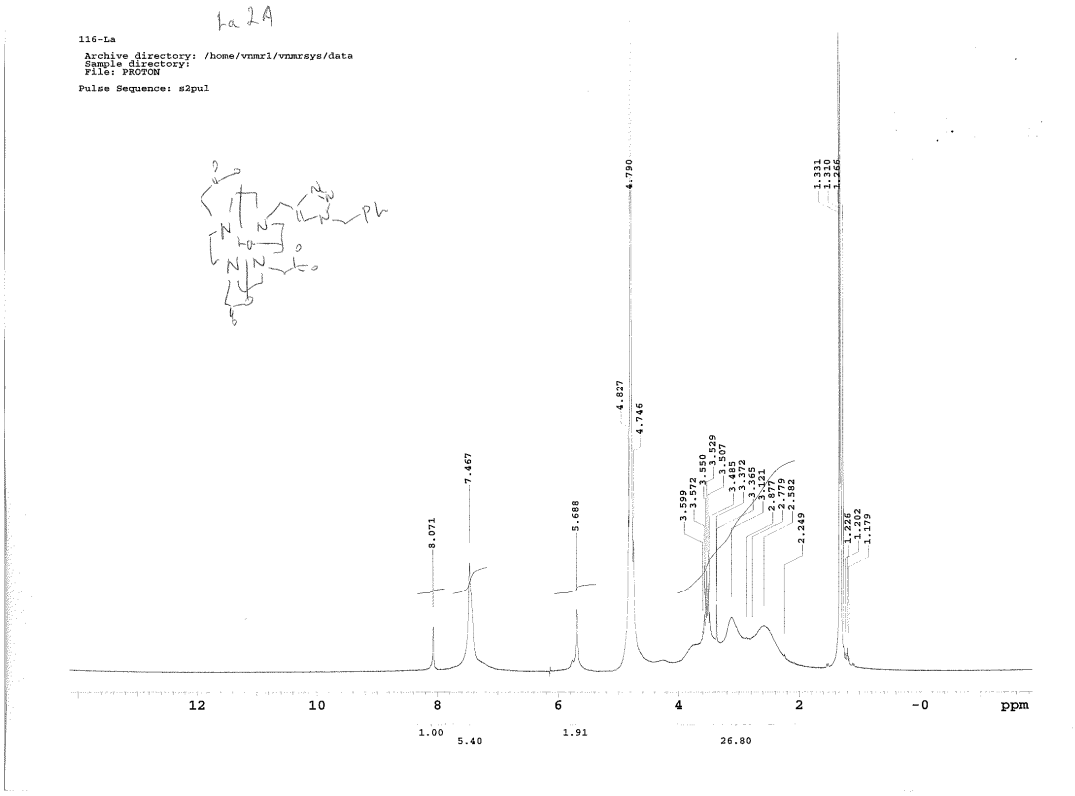

KEB-17-146 La
3.7857
3.3369
2.3891
2.3788
2.1592
1.3702
8.2260
2.0000
5.3084
12
10
8
6
4
2
0
-2
[ppm]
[rel]

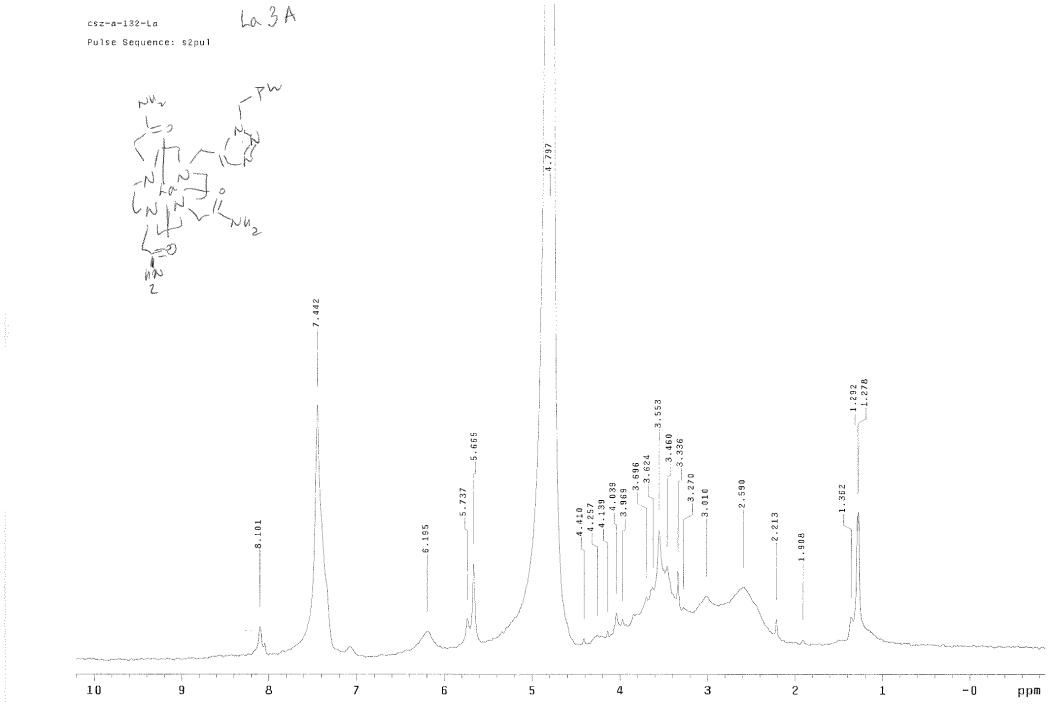


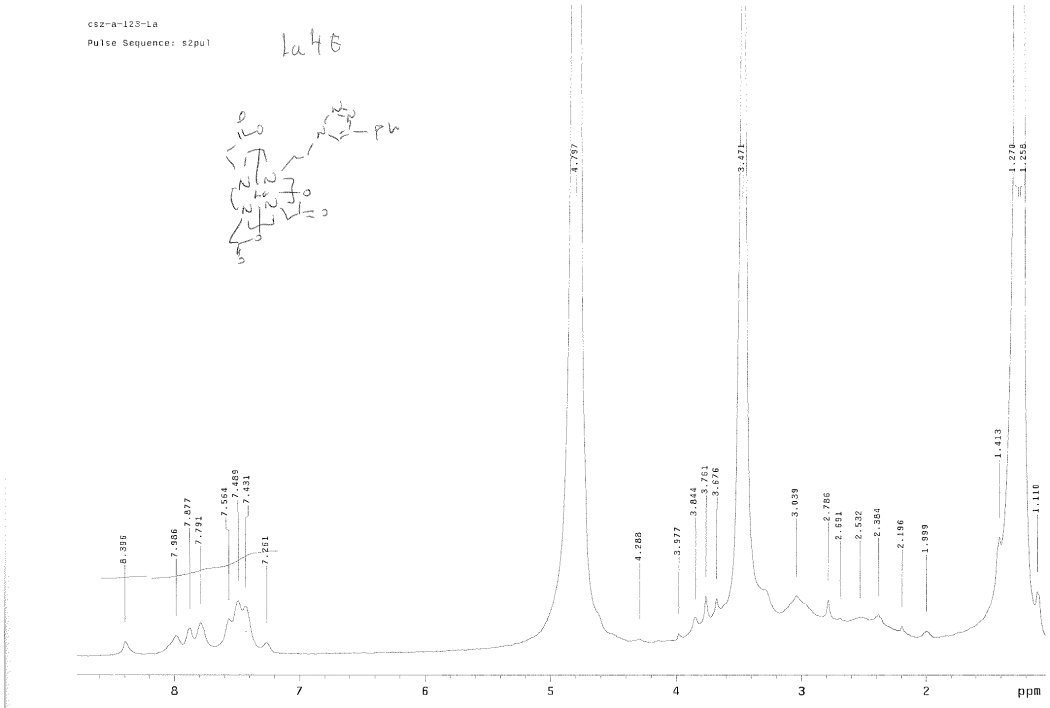


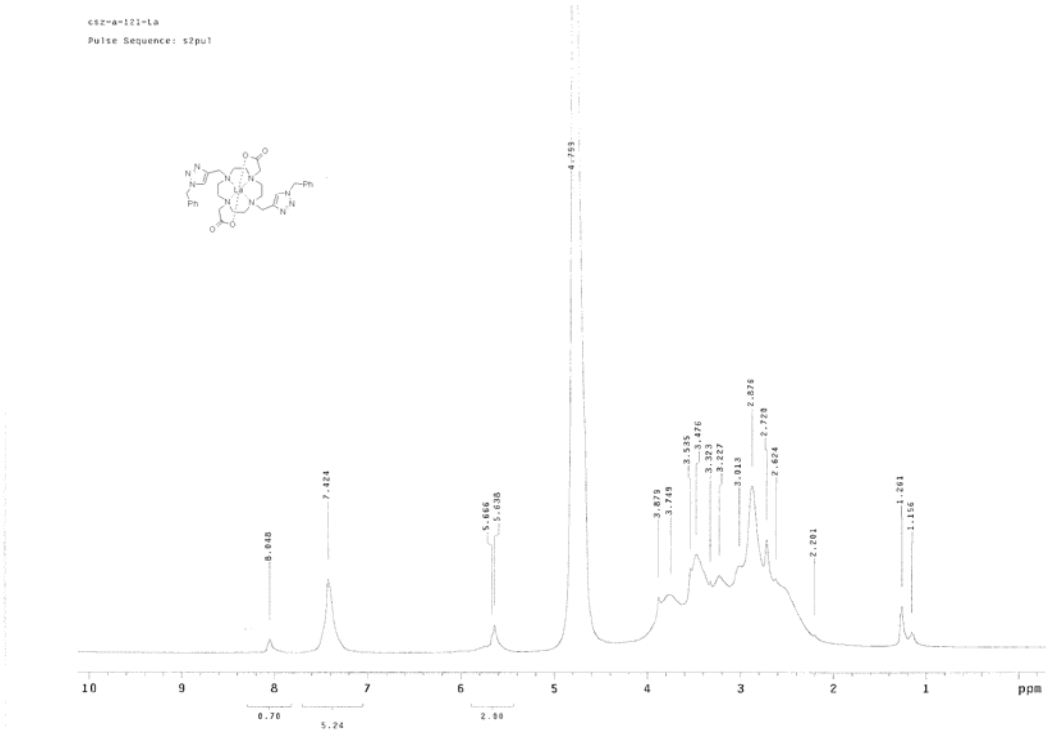

keb-18-026
D2O, 300MHz
La 16
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
ppm

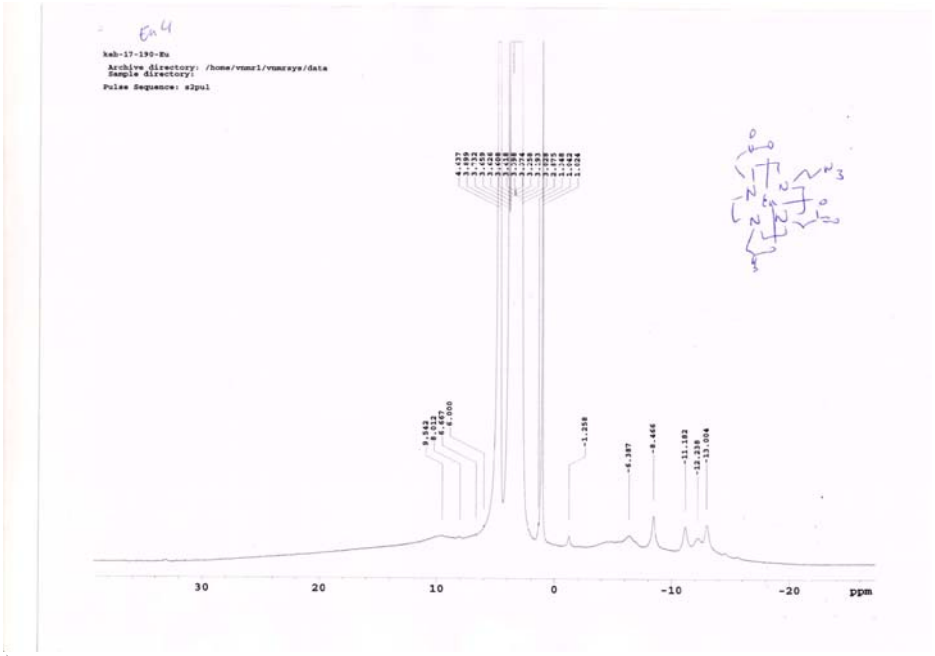

csz-a-117-La
Pulse Sequence: s2pul
Ph
ppm

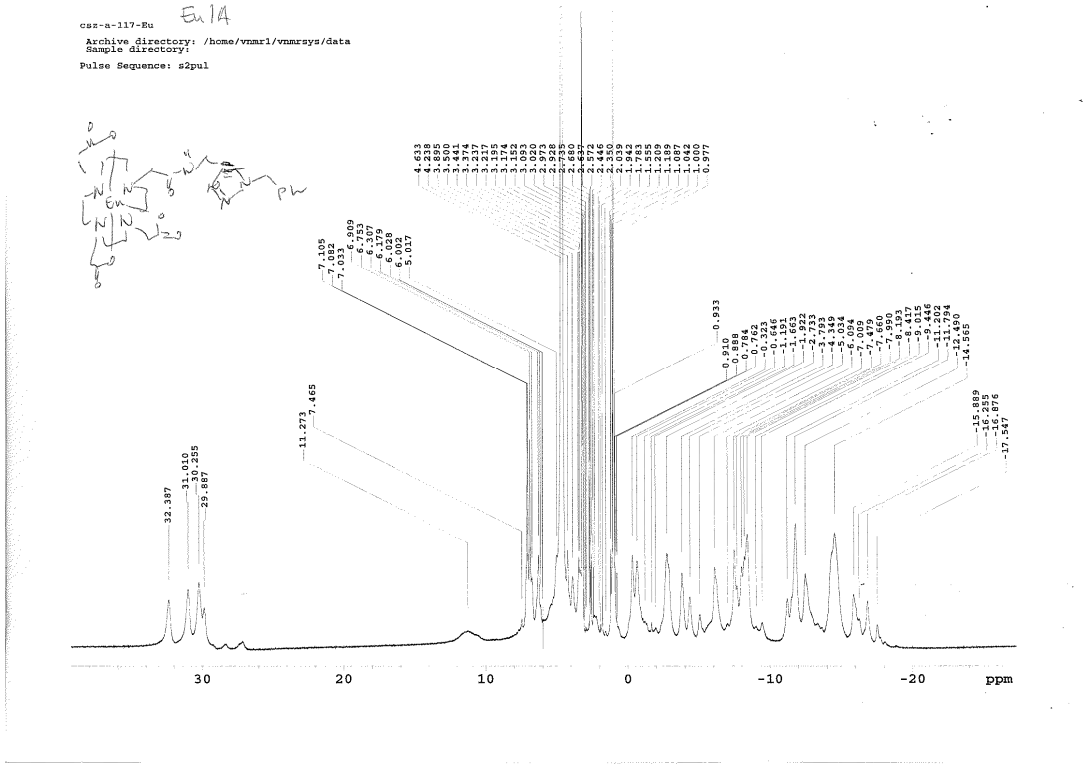

La 2A
116-La
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
Ph
8.071
7.467
5.688
4.827
4.790
4.746
3.599
3.572
3.550
3.529
3.507
3.485
3.372
3.365
3.141
2.779
2.582
2.249
1.331
1.310
1.286
1.226
1.202
1.179
12
10
8
6
4
2
-0
ppm
1.00
5.40
1.91
26.80

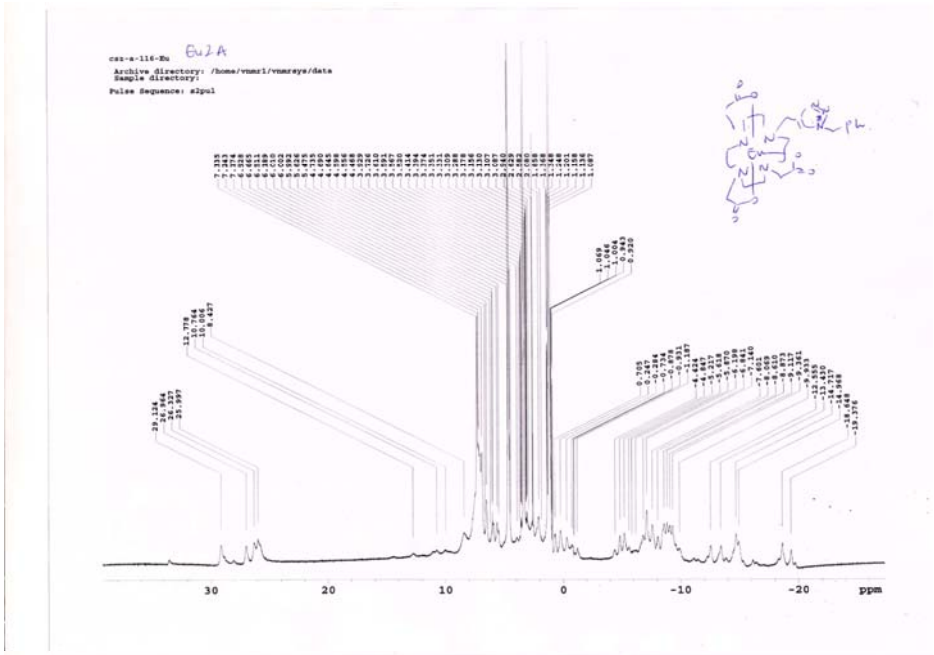

La 3A
csz-a-132-La
Pulse Sequence: s2pul
Ph
NH2
8.101
7.442
6.195
5.737
5.665
4.797
4.410
4.257
4.130
4.039
3.969
3.696
3.624
3.553
3.460
3.336
3.270
3.016
2.590
2.213
1.908
1.382
1.293
1.278
10
9
8
7
6
5
4
3
2
1
-0
ppm

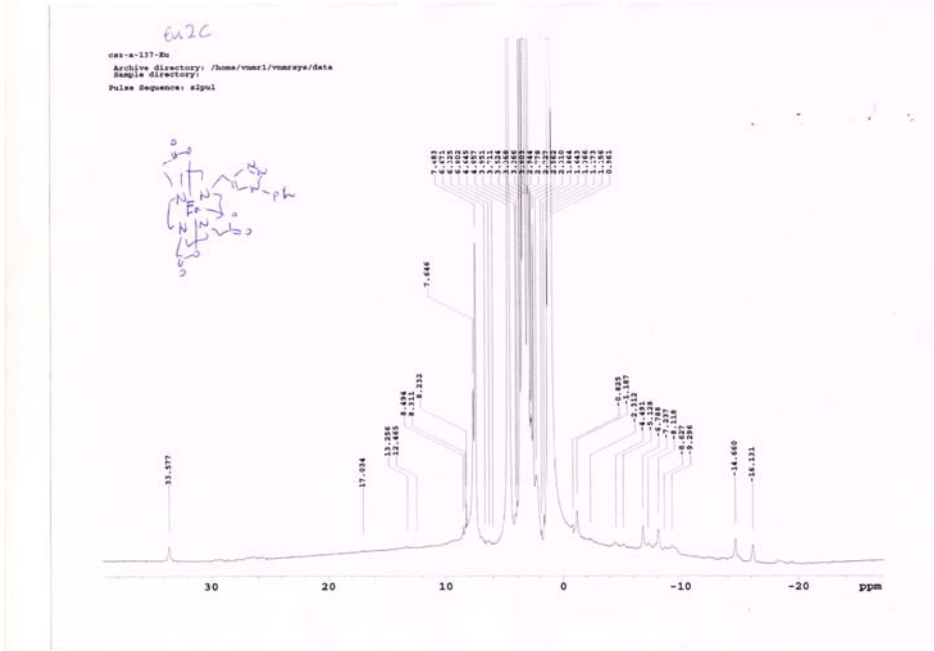

csz-a-123-La
Pulse Sequence: s2pul
La40
8.356
7.986
7.877
7.791
7.564
7.489
7.431
7.261
4.797
4.288
3.977
3.844
3.761
3.676
3.471
3.039
2.786
2.691
2.532
2.384
2.196
1.999
1.413
1.270
1.258
1.110
8
7
6
5
4
3
2
ppm

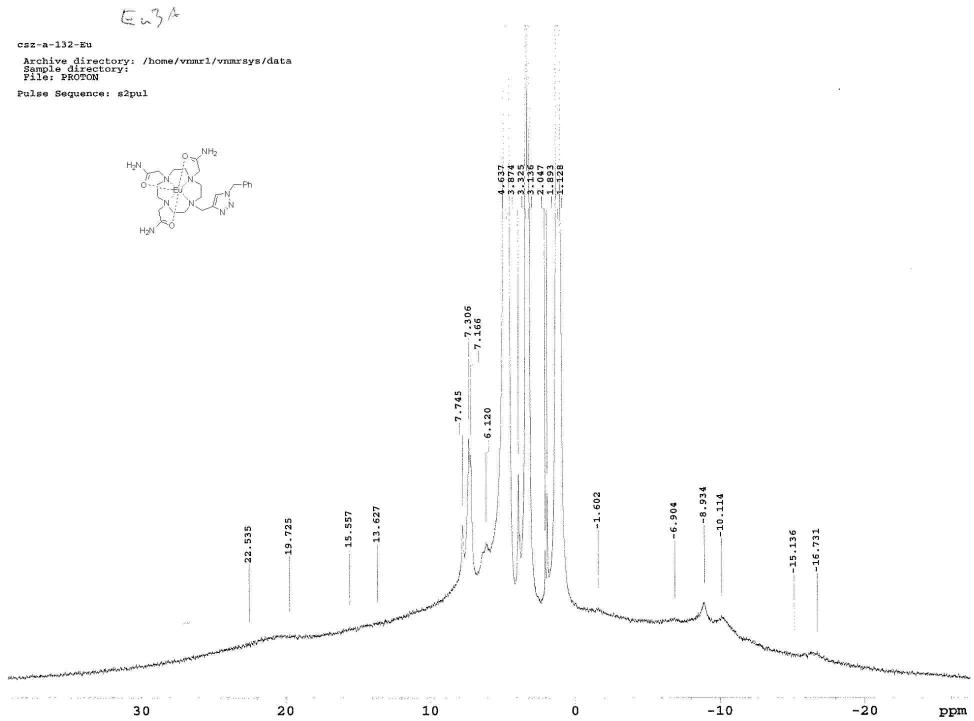

csz-a-121-La
Pulse Sequence: s2pul
8.648
7.424
5.666
5.638
4.765
3.879
3.749
3.535
3.476
3.323
3.227
3.013
2.875
2.728
2.624
2.281
1.261
1.156
10
9
8
7
6
5
4
3
2
1
ppm
0.70
5.24
2.86

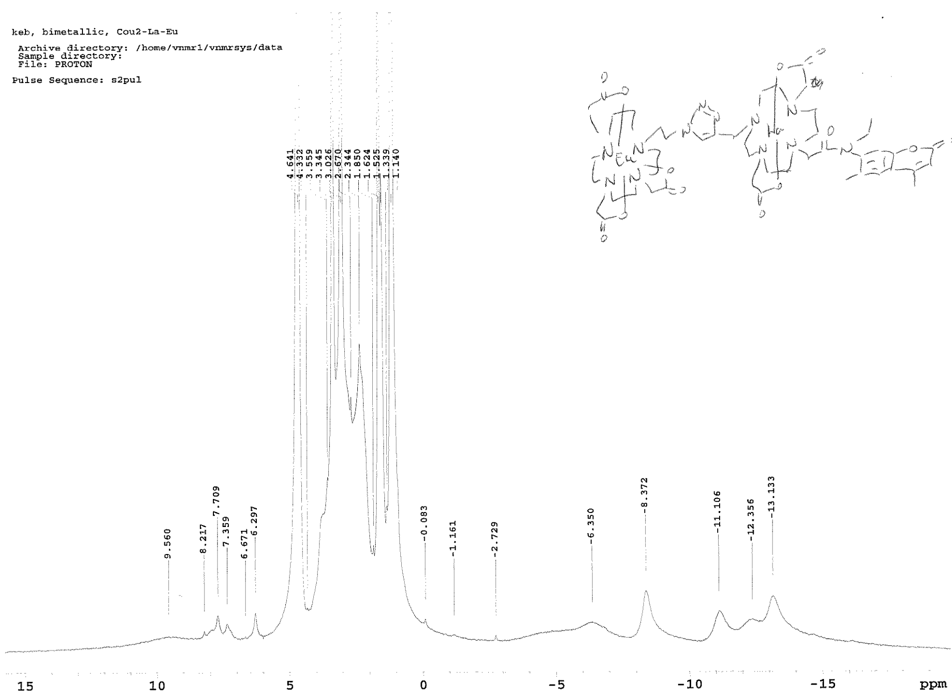

Eu4
kab-17-190-Eu
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
30
20
10
0
-10
-20
ppm

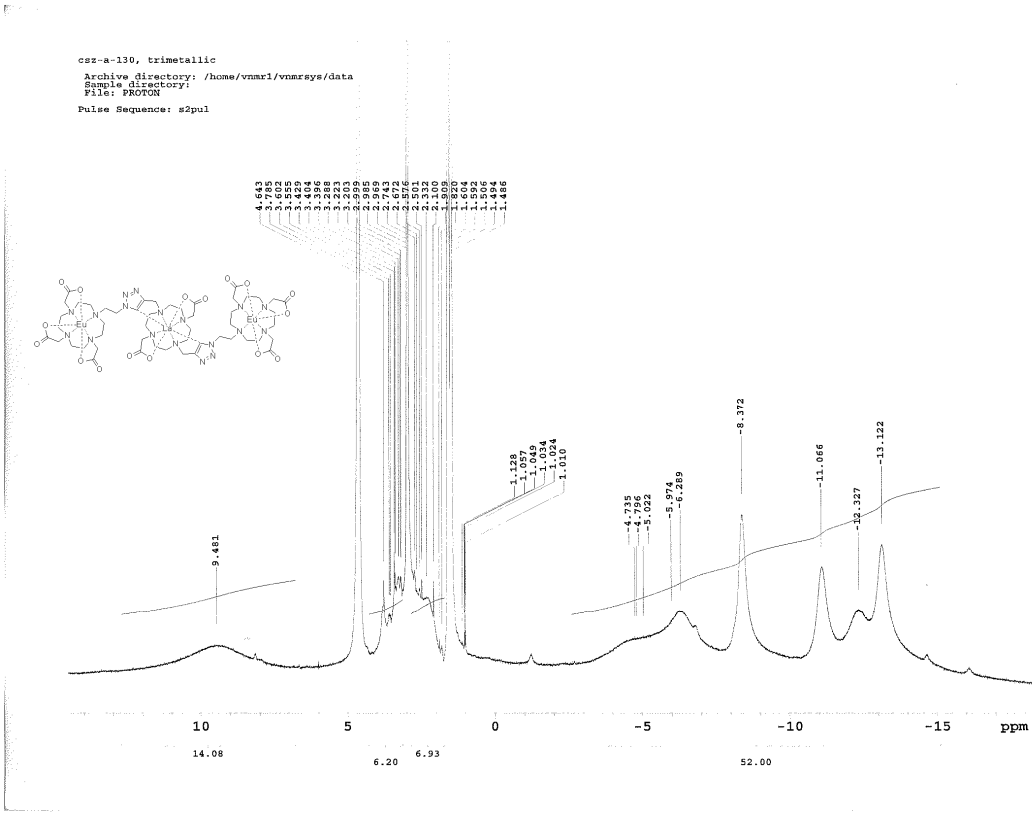

Eu14
csz-a-117-Eu
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
30
20
10
0
-10
-20
ppm






Eu3A
csz-a-132-Eu
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
22.535
19.725
15.557
13.627
7.745
7.306
7.166
6.120
4.637
3.874
3.325
3.136
2.047
1.893
1.128
-1.602
-6.904
-8.934
-10.114
-15.136
-16.731
30
20
10
0
-10
-20
ppm


keb, bimetallic, Cou2-La-Eu
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
9.560
8.217
7.709
7.359
6.671
6.297
4.641
4.332
3.559
3.145
3.026
2.670
2.344
1.850
1.624
1.525
1.339
1.140
-0.083
-1.161
-2.729
-6.350
-8.372
-11.106
-12.356
-13.133
15
10
5
0
-5
-10
-15
ppm


csz-a-130, trimetallic
Archive directory: /home/vnmr1/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
4.643
3.785
3.602
3.555
3.429
3.404
3.396
3.288
3.263
3.205
2.999
2.985
2.959
2.743
2.672
2.651
2.501
2.332
2.100
2.008
1.820
1.694
1.593
1.506
1.494
1.486
9.481
1.128
1.057
1.049
1.034
1.024
1.010
-4.735
-4.796
-5.022
-5.974
-6.289
-8.372
-11.066
-12.327
-13.122
10
5
0
-5
-10
-15
ppm
14.08
6.20
6.93
52.00