Preparation and enzymatic resolution of 3-hydroxy fatty acid nicotinyl esters.

Justus T. Metternich and Stefan H. Hüttenhain *

Table of contents

IR Spectrum

1H Spectrum

13C Spectrum

Enzymatic resolution chromatogram

5) 4e

Characterization data

Chromatogramm

IR Spectrum

1H Spectrum

13C Spectrum

Enzymatic resolution chromatogram

6) 4f

Characterization data

Chromatogramm

IR Spectrum

1H Spectrum

13C Spectrum

Enzymatic resolution chromatogram

7) 4g

Characterization data

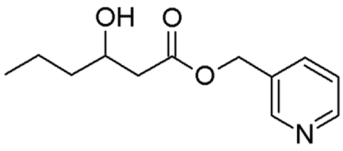
Chromatogramm

IR Spectrum

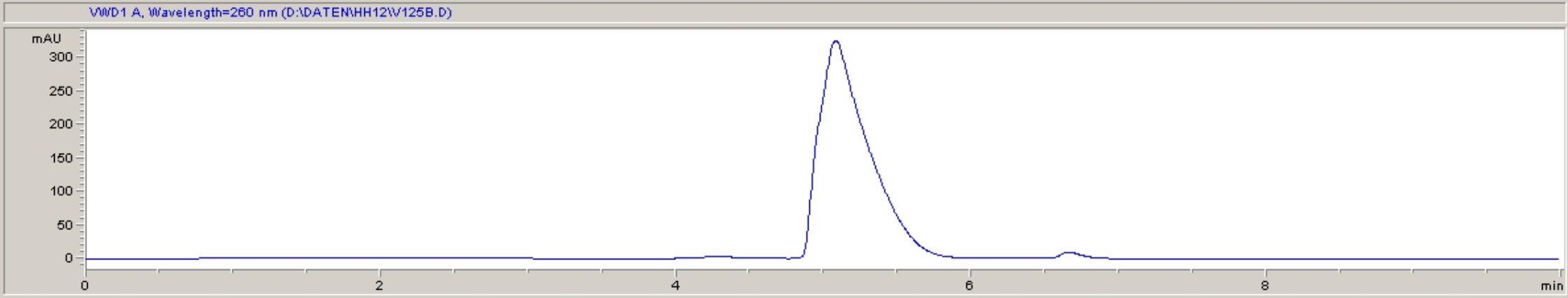
1H Spectrum

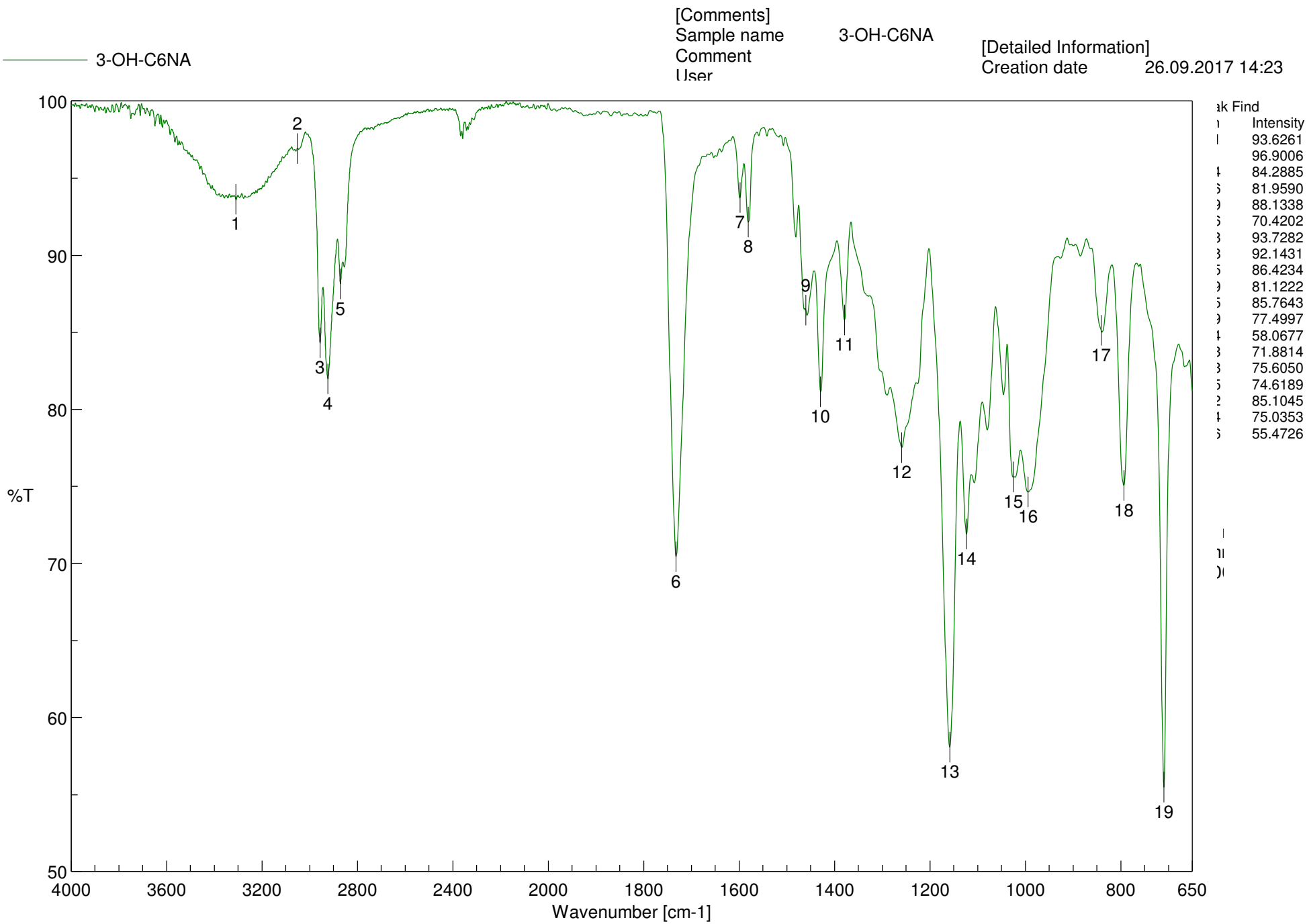
13C Spectrum

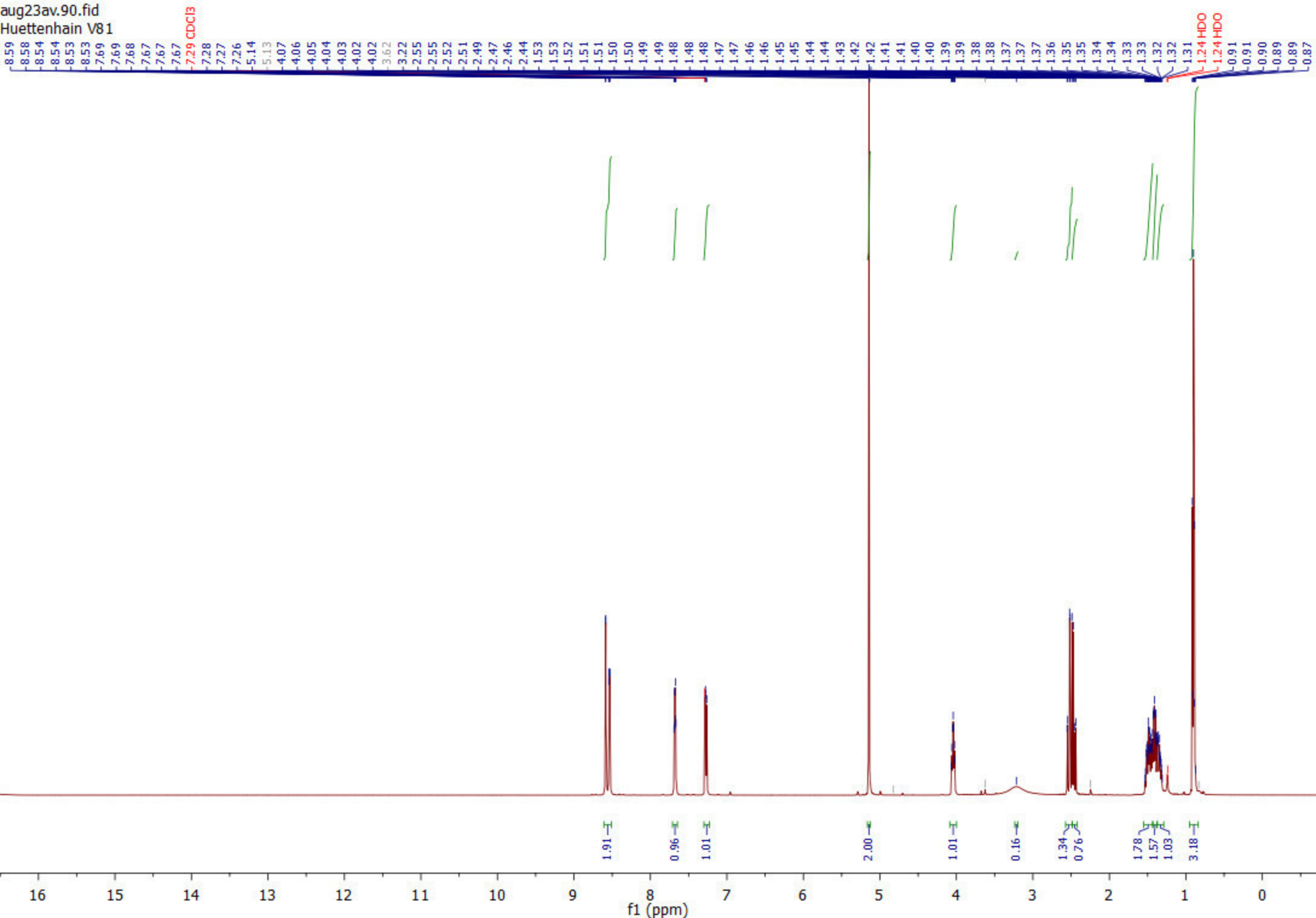
Enzymatic resolution chromatogram

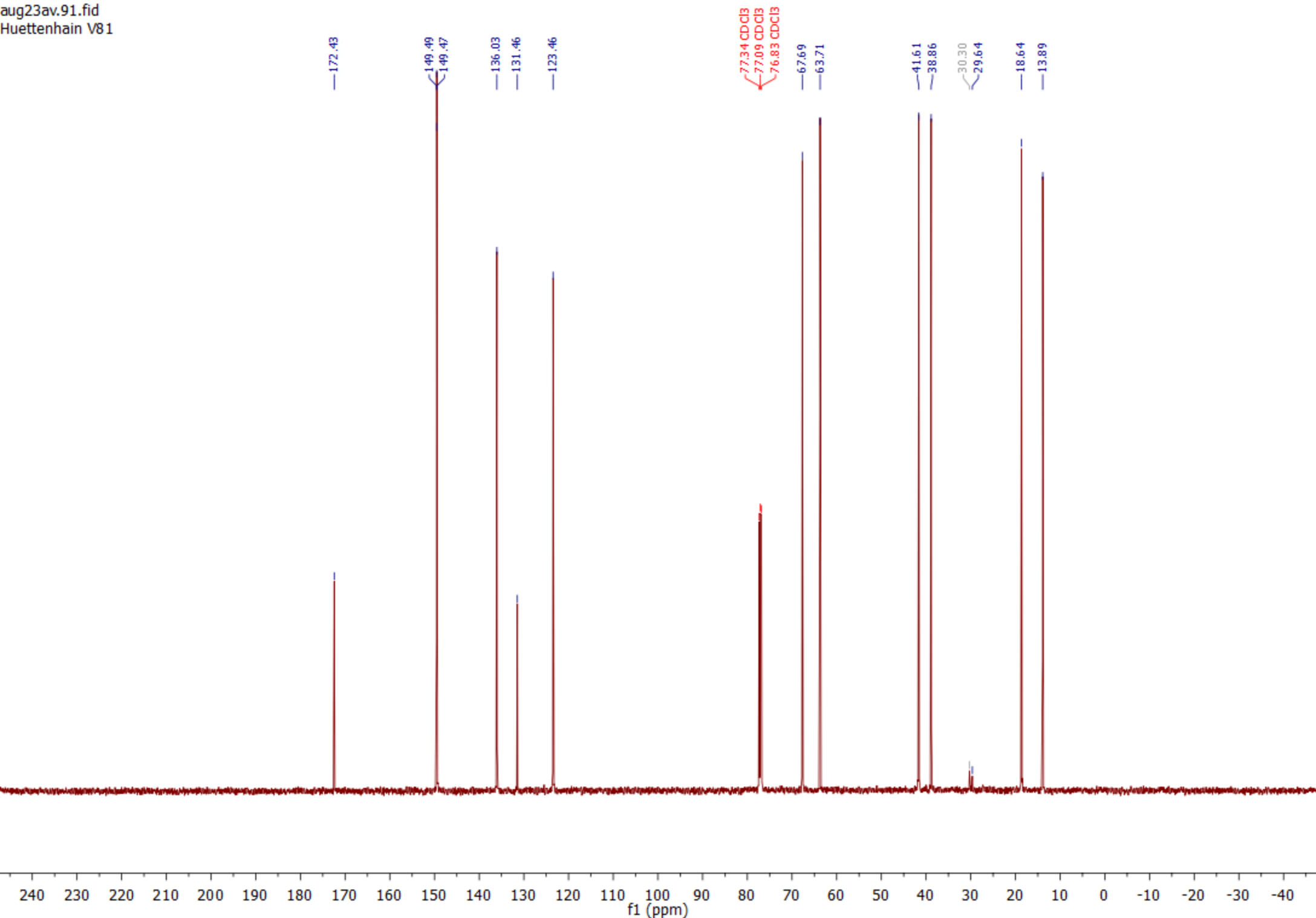


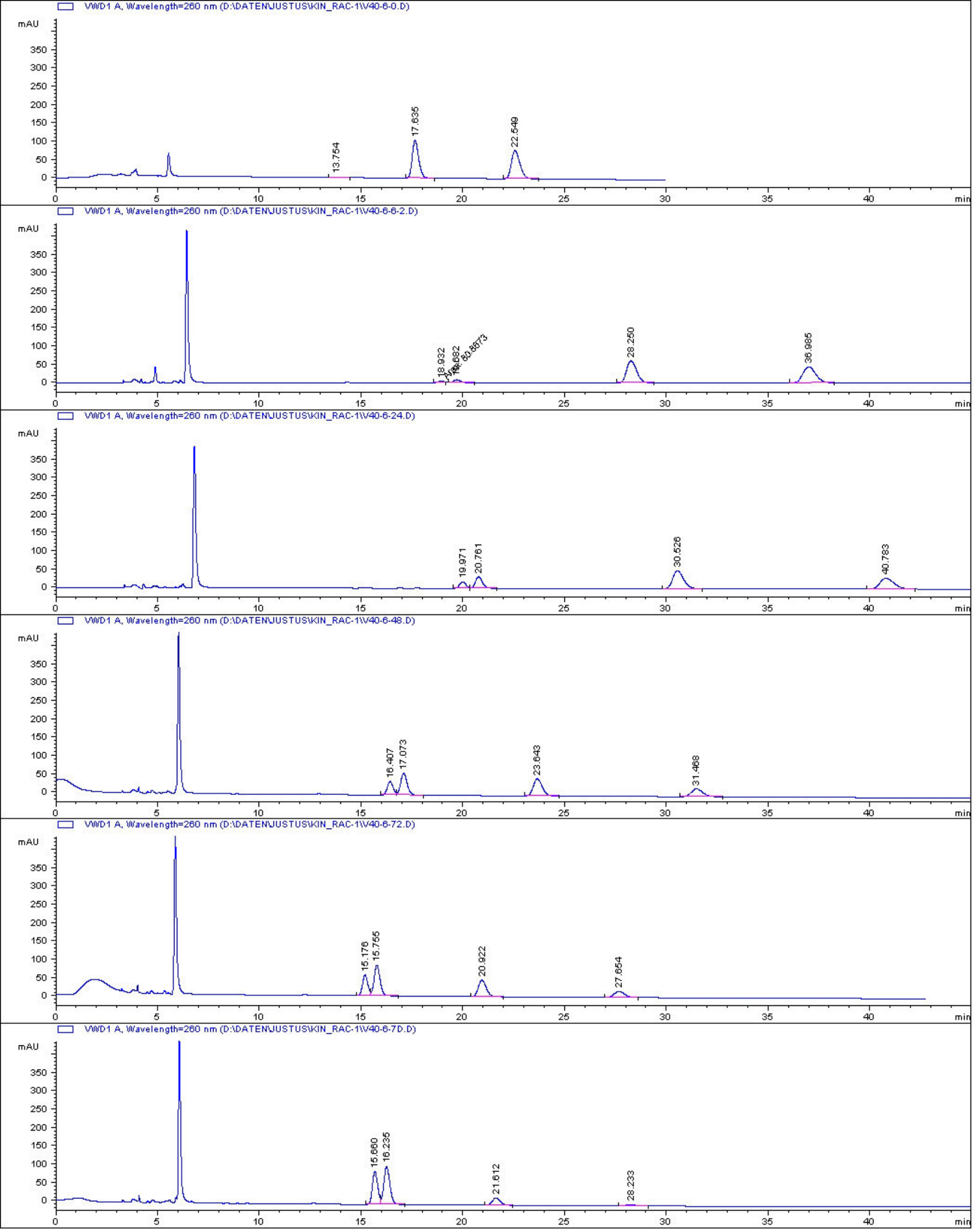
3-hydroxy hexanoic acid pyridin-3-ylmethylester **(4a).** Following the general procedure (A) from the substrate **3a** (221.3 mg, 0.1 mmol) the title molecule was obtained as a yellow oil (197 mg, 88% yield). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ (ppm): 8.61 - 8.51 (m, 2H), 7.68 (dt, J = 7.9, 2.0 Hz, 1H), 7.30 - 7.23 (m, 1H), 5.14 (s, 2H), 4.04 (tt, J = 8.1, 3.9 Hz, 1H), 2.57 - 2.49 (m, 1H), 2.49 - 2.42 (m, 1H), 1.55 - 1.43 (m, 2H), 1.43 - 1.37 (m, 2H), 1.37 - 1.29 (m, 1H), 0.90 (t, J = 7.0 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 126 MHz) δ (ppm): 172.4, 149.5, 149.4, 136.0, 131.5, 123.5, 67.69, 63.71, 41.6, 38.9, 30.3, 29.6, 18.6, 13.9; IR v: 3310; 3052; 2956; 2924; 2871; 1732; 1158; 1025; 794; 710 cm^{-1}; HPLC Waters SunFire^{TM} C18 3.5µm 2.1 x 100 mm; ACN: H_2O 90:10; 0.3 ml/min t_r = 6.044min; Anal. Calcd for $C_{12}H_{17}NO_3$: C, 64.55; H, 7.67; N, 6.27. Found: C, 64.15; H, 7.51; N, 6.08.

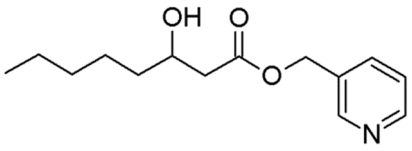

VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V125B.D)
mAU
300
250
200
150
100
50
0
0
2
4
6
8
min


3-OH-C6NA
[Comments]
Sample name 3-OH-C6NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 14:23
ak Find
Intensity
93.6261
96.9006
84.2885
81.9590
88.1338
70.4202
93.7282
92.1431
86.4234
81.1222
85.7643
77.4997
58.0677
71.8814
75.6050
74.6189
85.1045
75.0353
55.4726
%T
100
90
80
70
60
50
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19

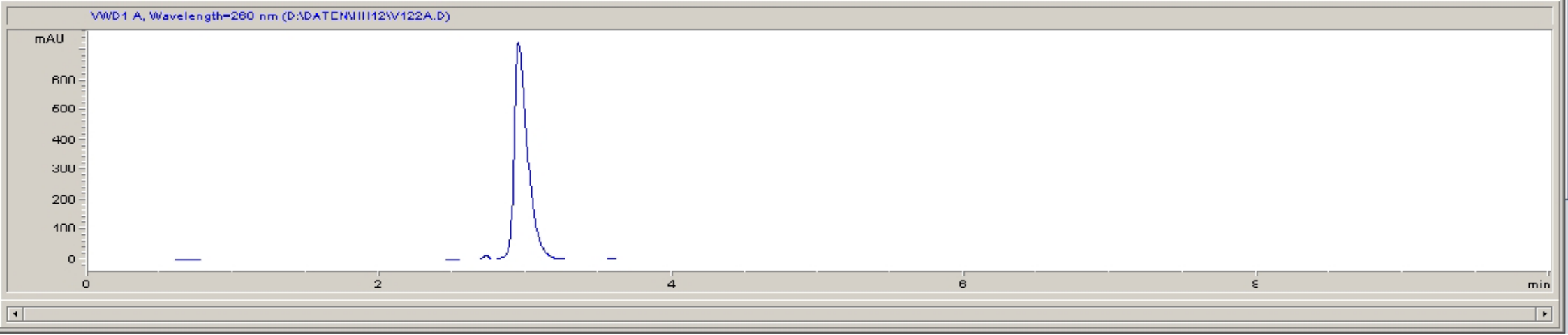

aug23av.90.fid
Huettenhain V81
CDCl3
HDO
HDO
1.91
0.96
1.01
2.00
1.01
0.16
1.34
0.76
1.78
1.57
1.03
3.18
f1 (ppm)

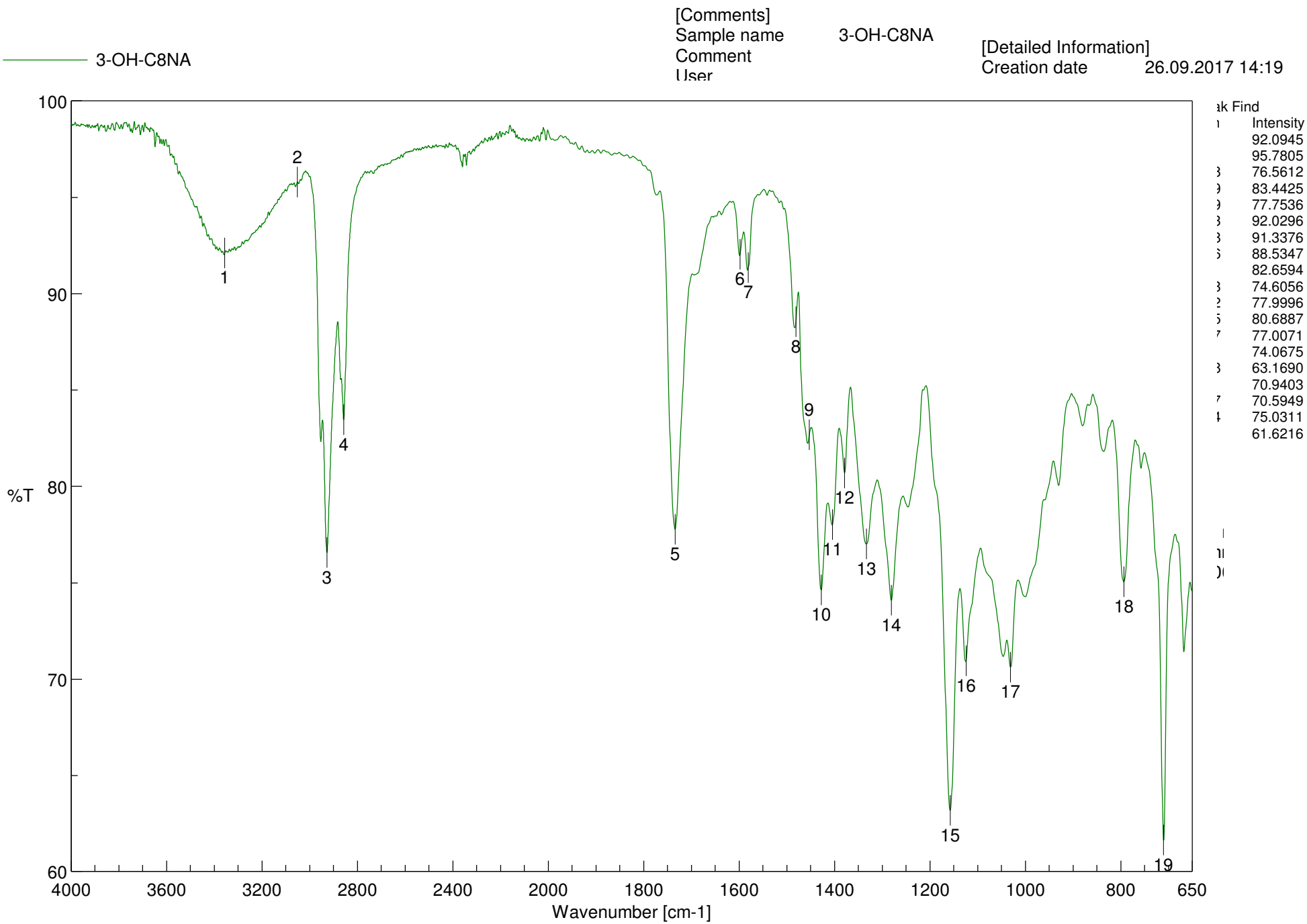

aug23av.91.fid
Huettenhain V81
172.43
149.49
149.47
136.03
131.46
123.46
77.34 CDCl3
77.09 CDCl3
76.83 CDCl3
67.69
63.71
41.61
38.86
30.30
29.64
18.64
13.89
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)


VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-0.D)
mAU
13.754
17.635
22.549
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-6-2.D)
mAU
18.932
19.682
20.873
28.250
36.985
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-24.D)
mAU
19.971
20.761
30.526
40.783
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-48.D)
mAU
16.407
17.073
23.643
31.468
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-72.D)
mAU
15.176
15.755
20.922
27.654
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KIN_RAC-1\V40-6-7D.D)
mAU
15.660
16.235
21.612
28.233
min

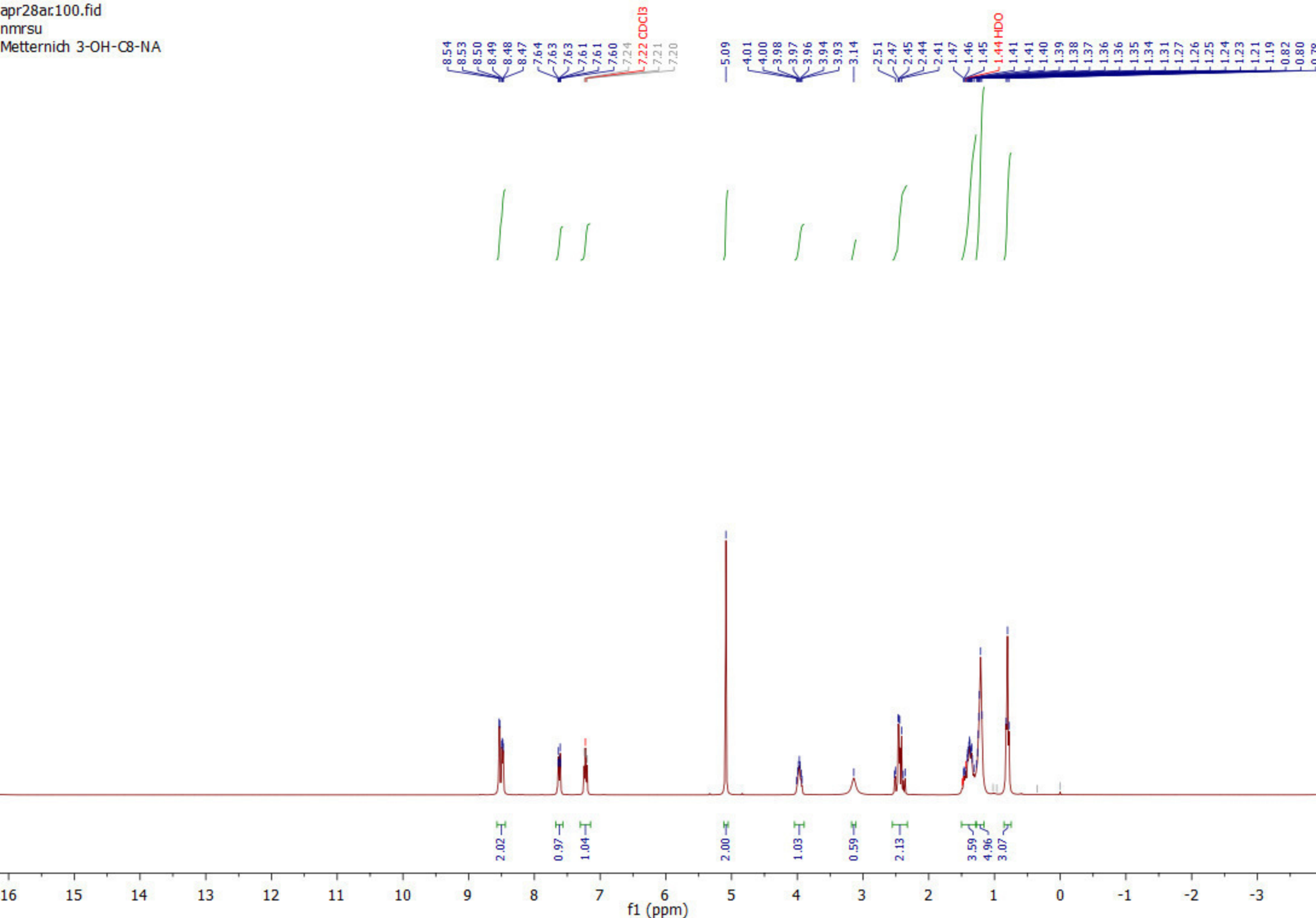


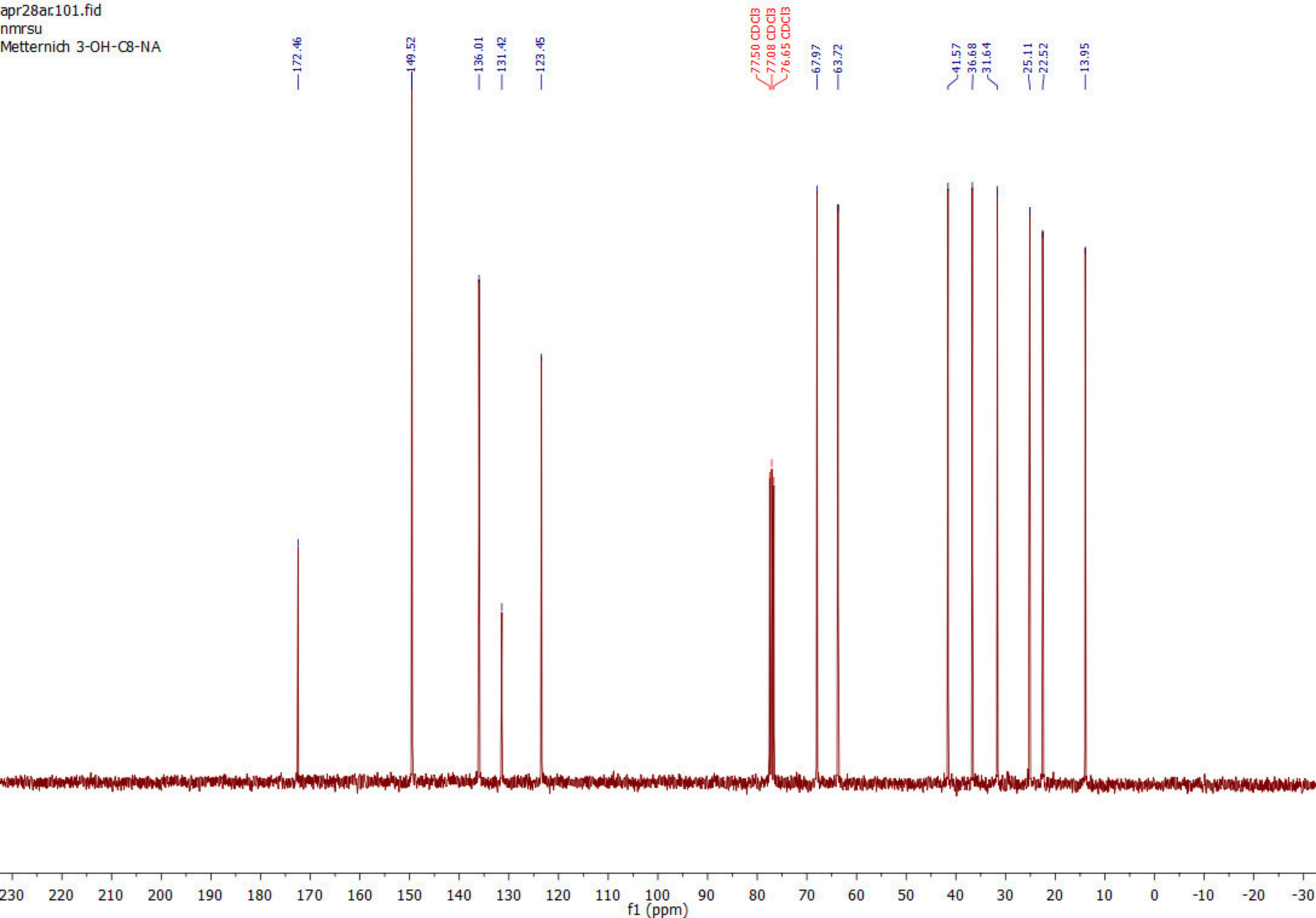
3-hydroxy octanoic acid pyridin-3-ylmethylester **(4b).** Following the general procedure (A) from the substrate **3b** (249.3 mg, 0.1 mmol) the title molecule was obtained as a yellow oil (189.6 mg, 76% yield). ^{1}H-NMR ($CDCl_3$, 300 MHz) δ (ppm) 8.57 - 8.44 (m, 2H), 7.62 (dt, J = 7.7, 2.0 Hz, 1H), 5.09 (s, 2H), 3.97 (tt, J = 7.8, 3.8 Hz, 1H), 3.14 (s, 1H), 2.55 - 2.33 (m, 2H), 1.50 - 1.28 (m, 4H), 1.22 (q, J = 5.9, 4.4 Hz, 5H), 0.85 - 0.75 (m, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz) δ (ppm): 172.5, 149.5, 136.0, 131.4, 123.5, 68.0, 63.7, 41.6, 36.7, 31.6, 25.1, 22.5, 14.0; IR v: 3357; 3052; 2928; 2858; 1734; 1157; 1031; 794; 711 cm^{-1}; HPLC: Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ; ACN:H_2O 50: 50; 1ml/min, (t_r = 2.94 min); Anal. Calcd for $C_{14}H_{21}NO_3$: C, 66.91; H, 8.42; N, 5.57. Found: C, 66.84; H, 8.504; N, 5.408.

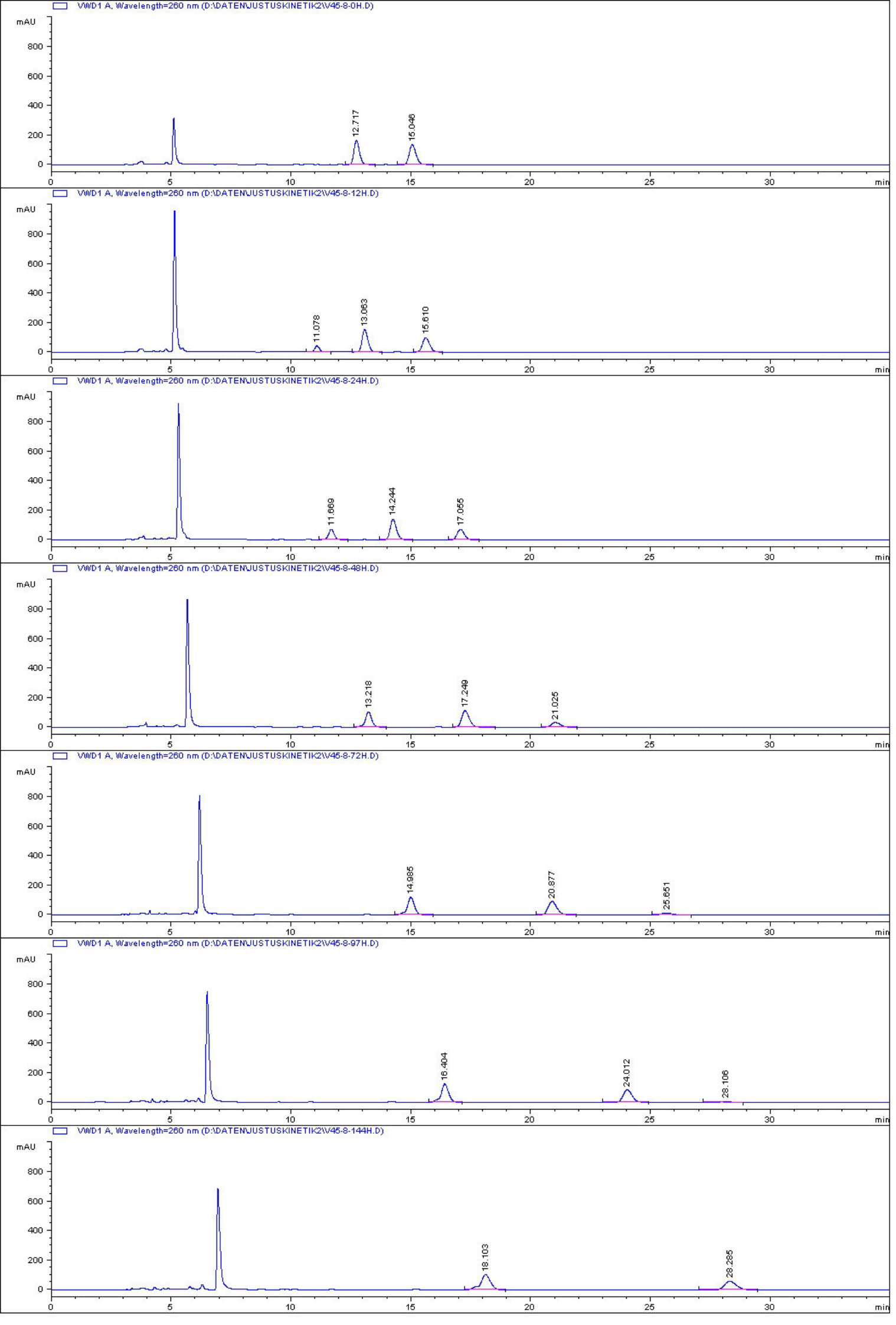

VWD1 A, Wavelength=260 nm (D:\DATEN\IIII12\V122A.D)
mAU
800
600
400
300
200
100
0
0
2
4
6
8
min

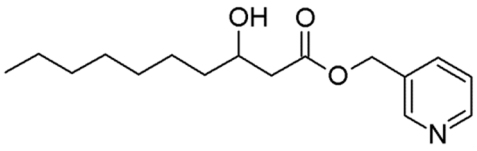

3-OH-C8NA
[Comments]
Sample name 3-OH-C8NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 14:19
ak Find
Intensity
92.0945
95.7805
76.5612
83.4425
77.7536
92.0296
91.3376
88.5347
82.6594
74.6056
77.9996
80.6887
77.0071
74.0675
63.1690
70.9403
70.5949
75.0311
61.6216
%T
100
90
80
70
60
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19

apr28ar.100.fid
nmrsu
Metternich 3-OH-C8-NA

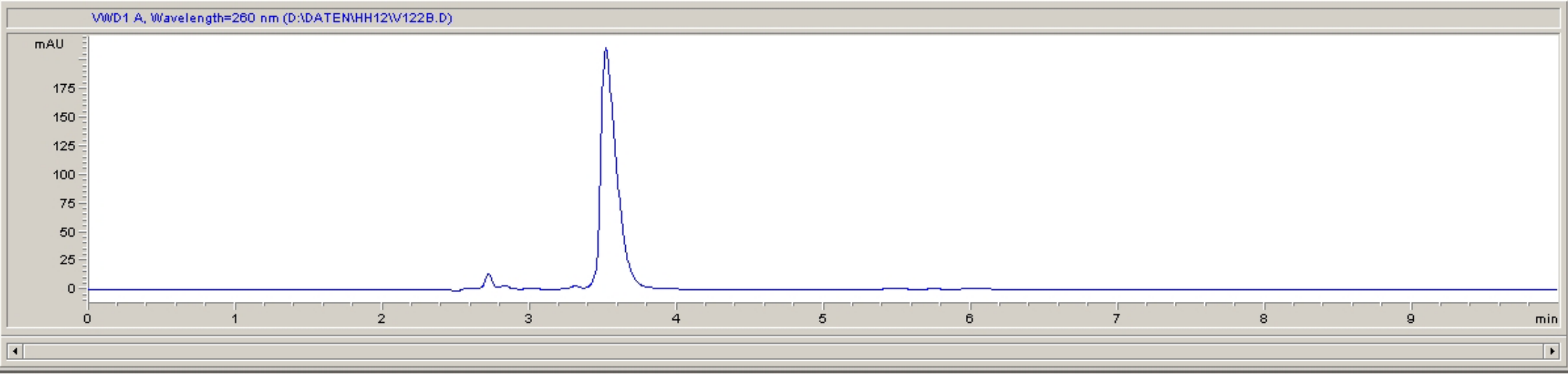


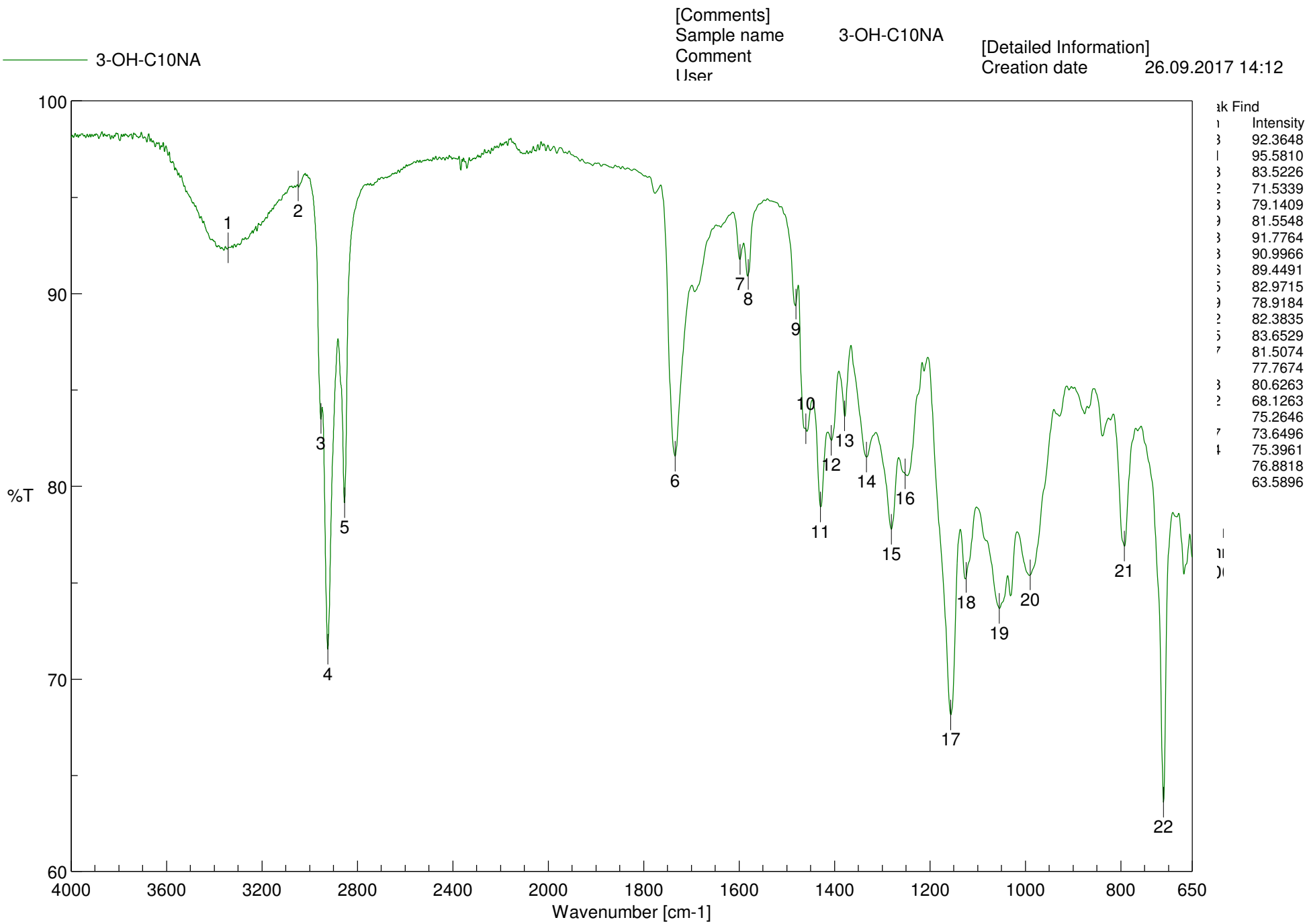

apr28ar.101.fid
nmrsu
Metternich 3-OH-C8-NA
172.46
149.52
136.01
131.42
123.45
77.50 CDCl3
77.08 CDCl3
76.65 CDCl3
67.97
63.72
41.57
36.68
31.64
25.11
22.52
13.95
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)


VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-0H.D)
mAU
12.717
15.046
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-12H.D)
11.078
13.063
15.610
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-24H.D)
11.669
14.244
17.055
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-48H.D)
13.218
17.249
21.025
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-72H.D)
14.985
20.877
25.651
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-97H.D)
16.404
24.012
28.106
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-8-144H.D)
18.103
28.285
min

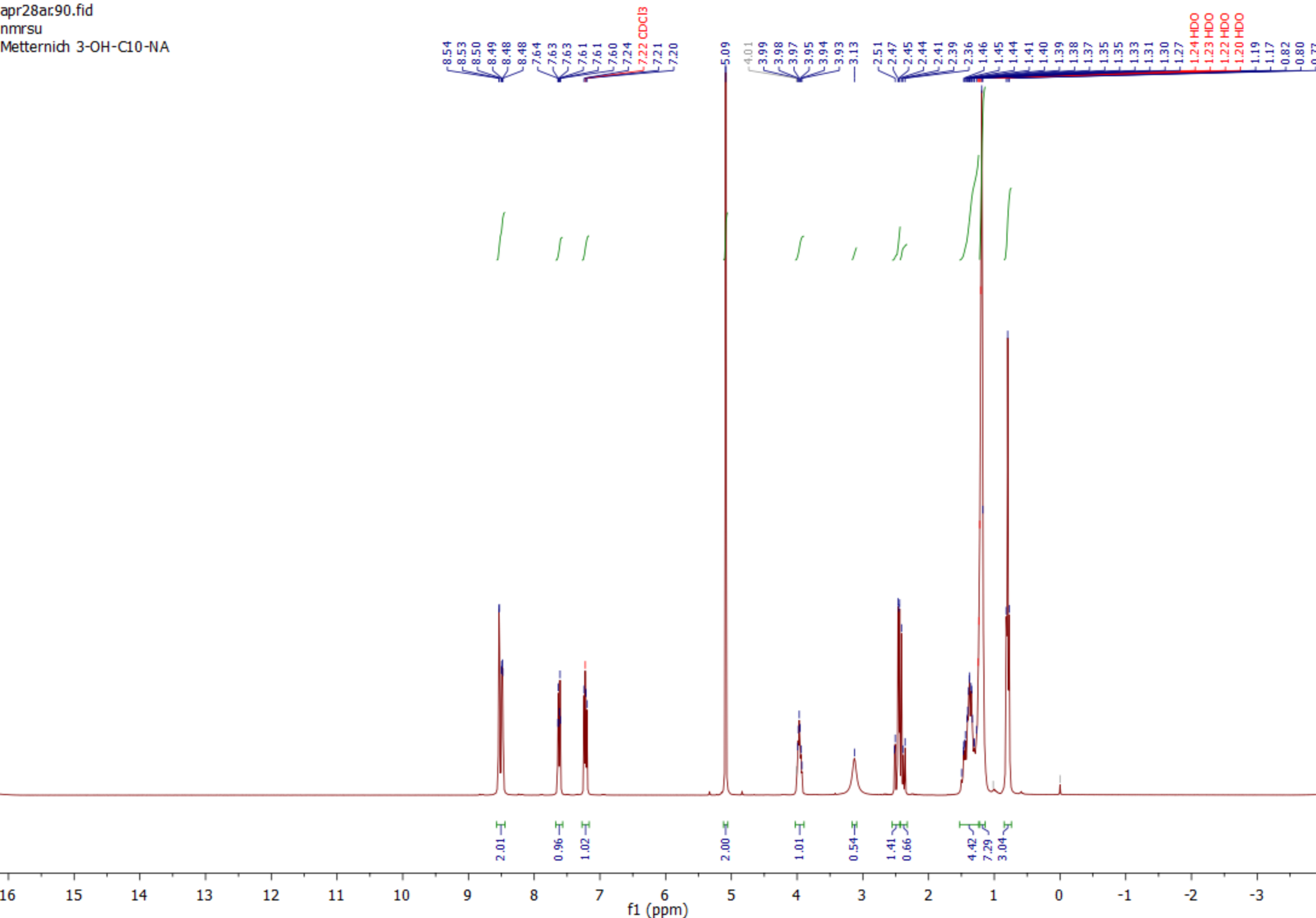


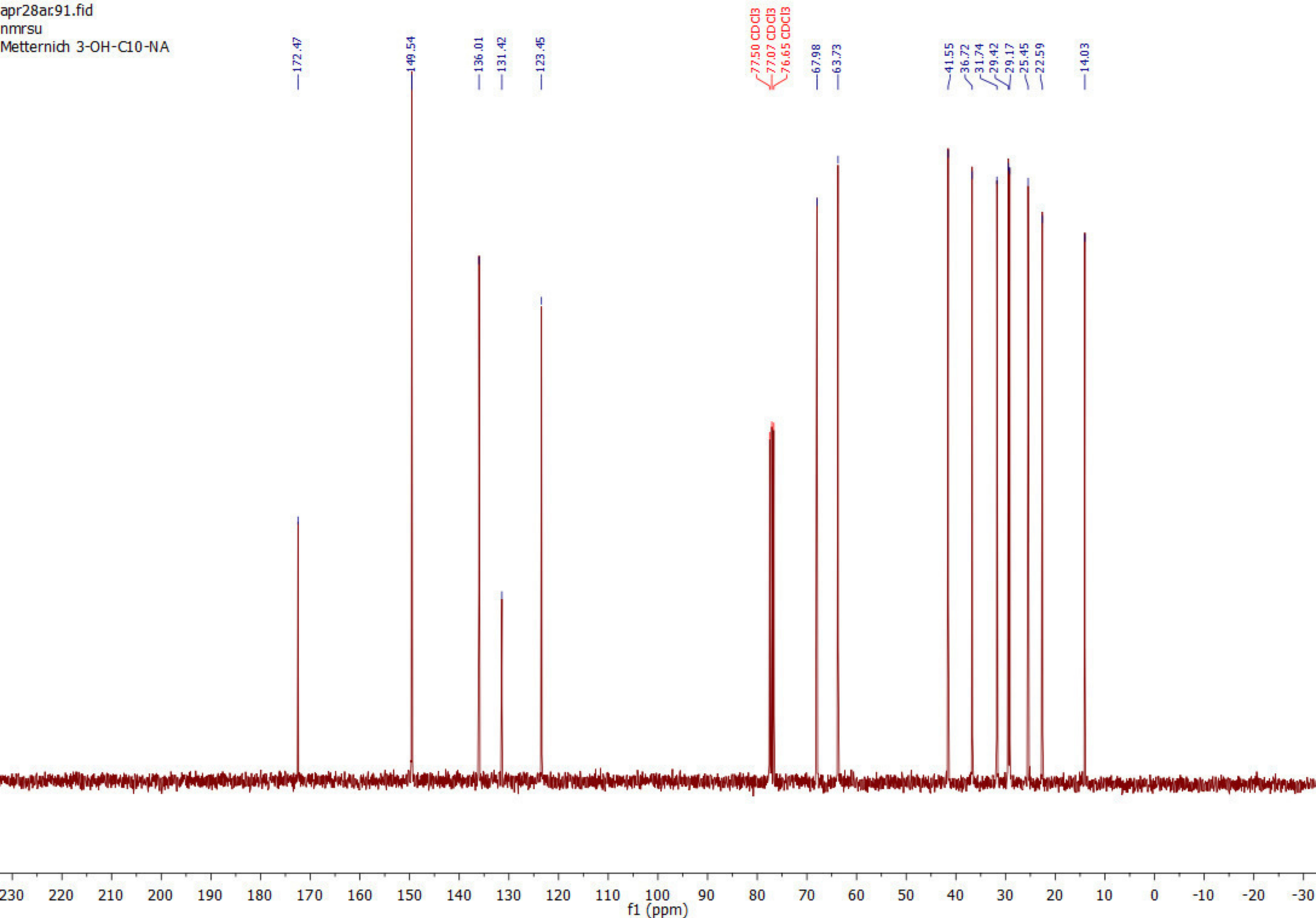
3-hydroxy decanoic acid pyridin-3-ylmethylester **(4c).** Following the general procedure (A) from the substrate **3c** (277.4 mg, 0.1 mmol) the title molecule was obtained as a yellow oil (209 mg, 74% yield). ^{1}H-NMR ($CDCl_3$, 300 MHz) δ (ppm): 8.57 - 8.44 (m, 2H), 7.62 (dt, J = 7.8, 2.0 Hz, 1H), 7.27 - 7.16 (m, 1H), 5.09 (s, 2H), 3.96 (dq, J = 8.0, 4.0 Hz, 1H), 3.13 (s, 1H), 2.55 - 2.43 (m, 1H), 2.38 (d, J = 16.2 Hz, 1H), 1.53 - 1.24 (m, 4H), 1.18 (d, J = 5.1 Hz, 7H), 0.85 - 0.74 (m, 3H); ^{13}C-NMR ($CDCl_3$, 75 MHz) δ (ppm): 172.5, 149.5, 136.0, 131.4, 123.5, 68.0, 63.7, 41.6, 36.7, 31.7, 29.4, 29.2, 25.5, 22.6, 14.0; IR v: 3324; 3049; 2952; 2925; 2854; 1734; 1156; 1055; 793; 711 cm^{-1}; HPLC: Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ; ACN:H_2O 50:50, 1ml/min (t_r = 3.52 min); Anal. Calcd for $C_{16}H_{25}NO_3$: C, 68.79; H, 9.02; N, 5.01. Found: C, 68.54; H, 8.886; N, 4.943.

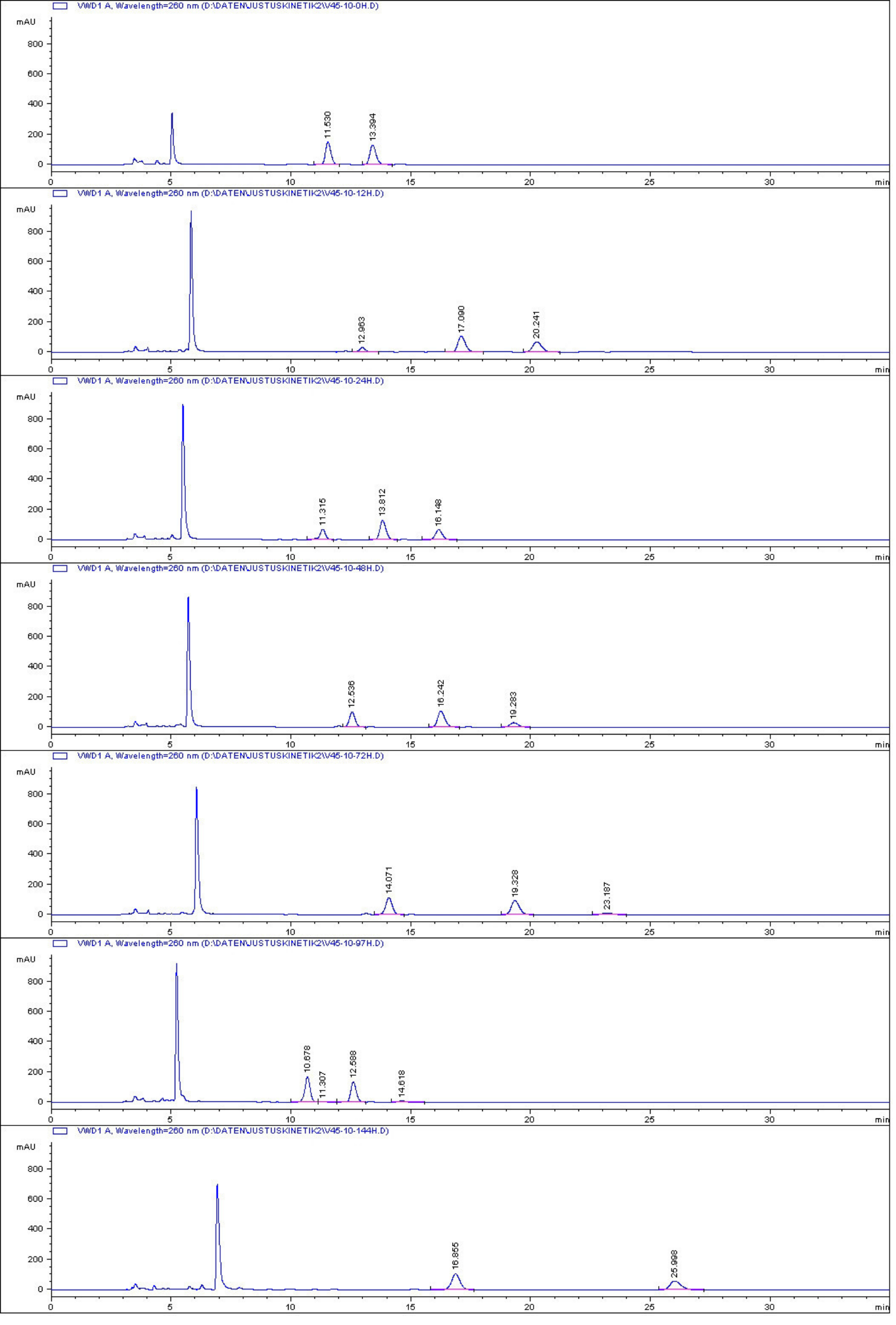

VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V122B.D)
mAU
175
150
125
100
75
50
25
0
0
1
2
3
4
5
6
7
8
9
min

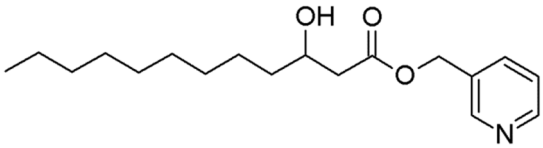

3-OH-C10NA
[Comments]
Sample name 3-OH-C10NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 14:12
ak Find
Intensity
92.3648
95.5810
83.5226
71.5339
79.1409
81.5548
91.7764
90.9966
89.4491
82.9715
78.9184
82.3835
83.6529
81.5074
77.7674
80.6263
68.1263
75.2646
73.6496
75.3961
76.8818
63.5896
%T
100
90
80
70
60
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22

apr28ar.90.fid
nmrsu
Metternich 3-OH-C10-NA

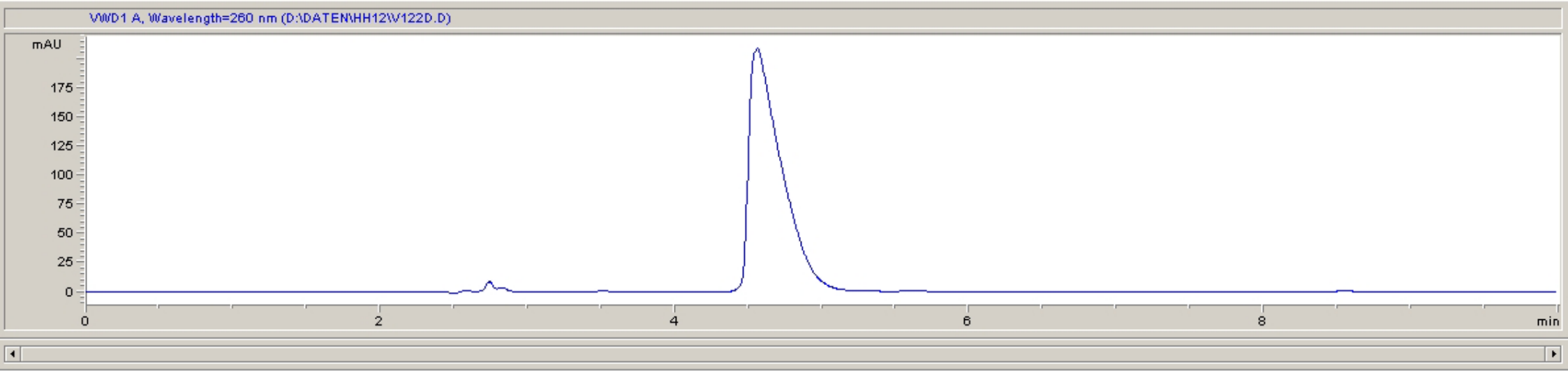


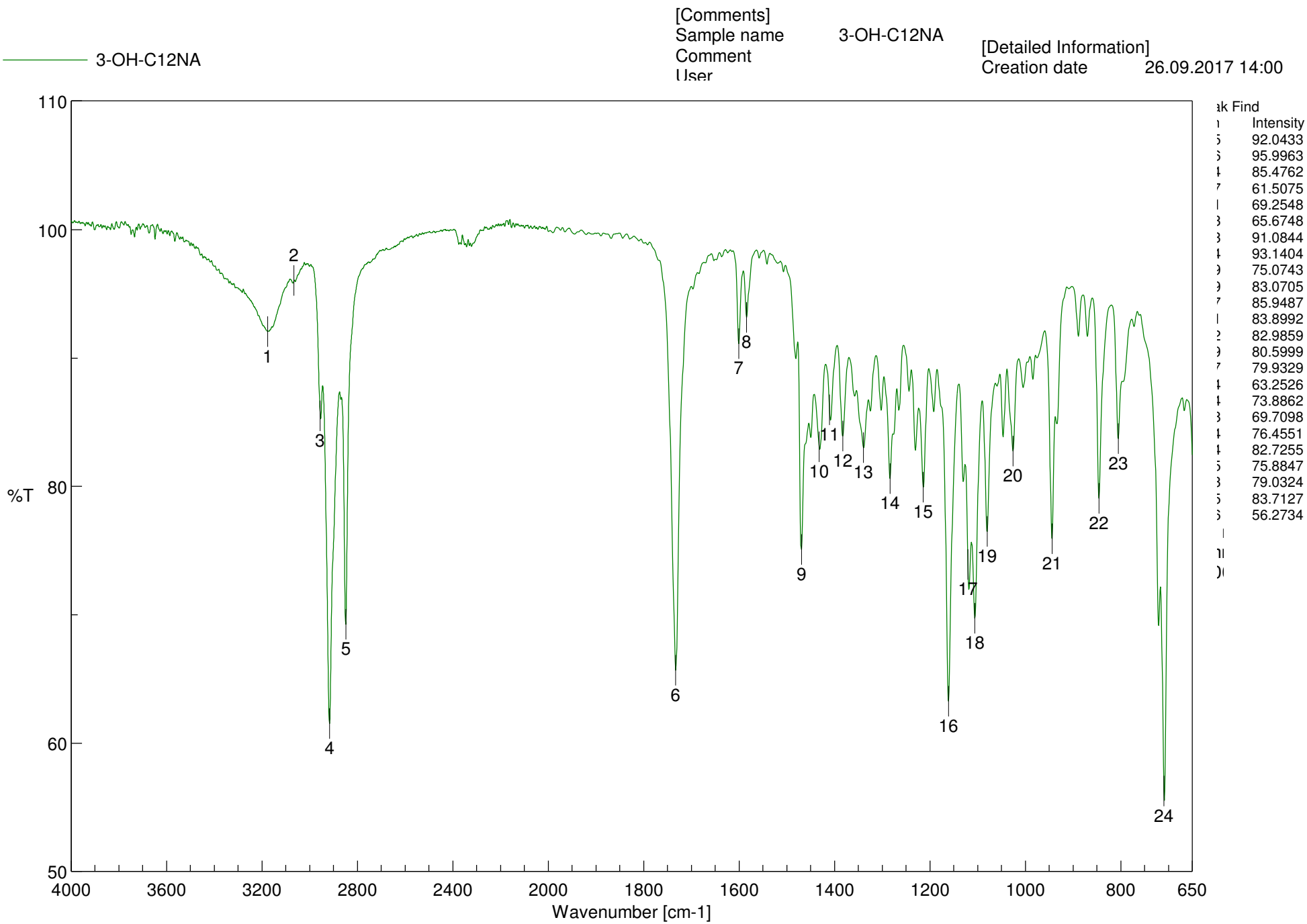

apr28ar.91.fid
nmrsu
Metternich 3-OH-C10-NA
172.47
149.54
136.01
131.42
123.45
77.50 CDCl3
77.07 CDCl3
76.65 CDCl3
67.98
63.73
41.55
36.72
31.74
29.42
29.17
25.45
22.59
14.03
f1 (ppm)

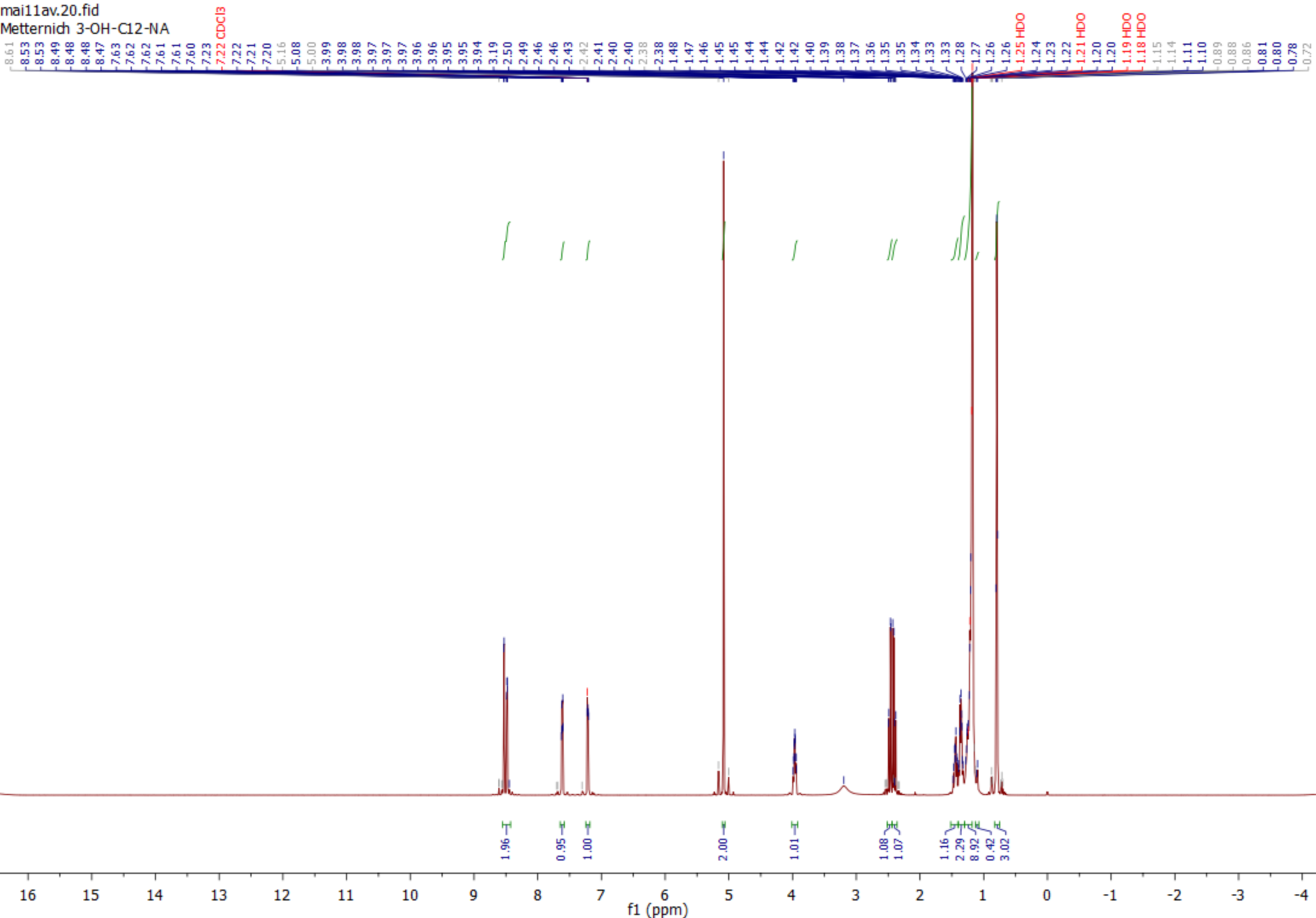

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-0H.D)
mAU
11.530
13.394
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-12H.D)
mAU
12.963
17.090
20.241
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-24H.D)
mAU
11.315
13.812
16.148
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-48H.D)
mAU
12.536
16.242
19.283
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-72H.D)
mAU
14.071
19.328
23.187
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-97H.D)
mAU
10.678
11.307
12.588
14.818
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\V45-10-144H.D)
mAU
16.855
25.998
min

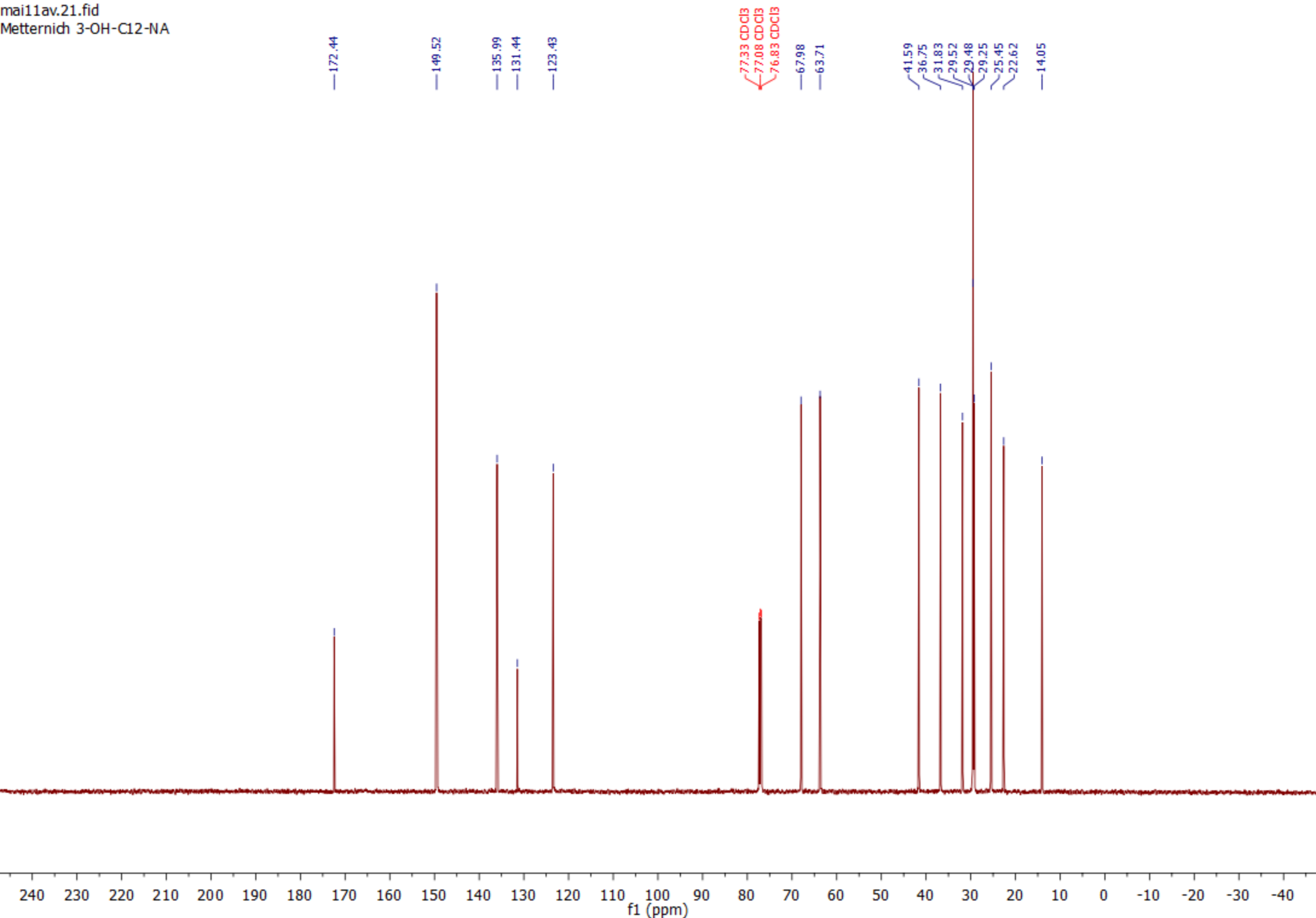


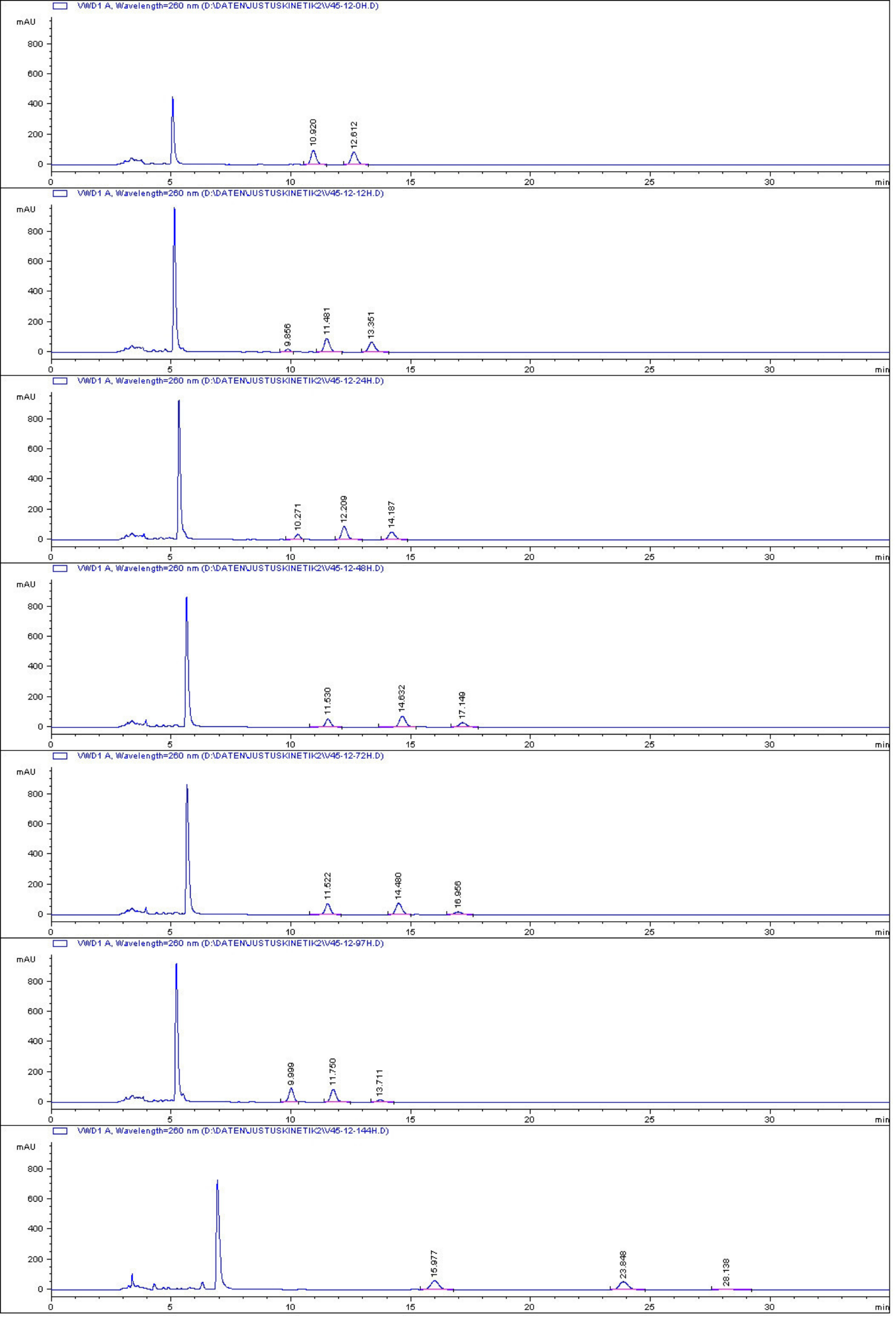
3-hydroxy dodecanoic acid pyridin-3-ylmethylester **(4d).** Following the general procedure (A) from the substrate **3d** (305.4 mg, 0.1 mmol) the title molecule was obtained as a yellow oil (274 mg, 89% yield). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ (ppm): 8.55 - 8.43 (m, 2H), 7.62 (dt, J = 7.8, 2.0 Hz, 1H), 7.21 (dd, J = 8.1, 5.1 Hz, 1H), 5.08 (s, 2H), 3.97 (tdd, J = 8.1, 4.5, 3.4 Hz, 1H), 2.48 (dd, J = 16.1, 3.5 Hz, 1H), 2.40 (dd, J = 16.1, 8.8 Hz, 1H), 1.52 - 1.40 (m, 1H), 1.40 - 1.30 (m, 2H), 1.30 - 1.18 (m, 9H), 1.10 (d, J = 5.3 Hz, 1H), 0.80 (t, J = 6.9 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 126 MHz) δ (ppm): 172.4, 149.5, 136.0, 131.4, 123.4, 68.0, 63.7, 41.6, 36.8, 31.8, 29.5, 29.5, 29.3, 25.5, 22.6, 14.1; IR v: 3177; 3066; 2956; 2918; 2849; 1733; 1161; 1020; 710 cm^{-1}; HPLC**:** Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ; ACN:H_2O 50:50 1ml/min,(t_r = 4.56 min); Anal. Calcd for $C_{18}H_{29}NO_3$: C, 70.32; H, 9.51; N, 4.56. Found: C, 70.54; H, 9.732; N, 4.706.

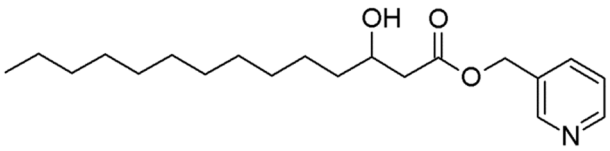

VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V122D.D)
mAU
175
150
125
100
75
50
25
0
0
2
4
6
8
min


3-OH-C12NA
[Comments]
Sample name 3-OH-C12NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 14:00
ak Find
Intensity
92.0433
95.9963
85.4762
61.5075
69.2548
65.6748
91.0844
93.1404
75.0743
83.0705
85.9487
83.8992
82.9859
80.5999
79.9329
63.2526
73.8862
69.7098
76.4551
82.7255
75.8847
79.0324
83.7127
56.2734
%T
110
100
80
60
50
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24

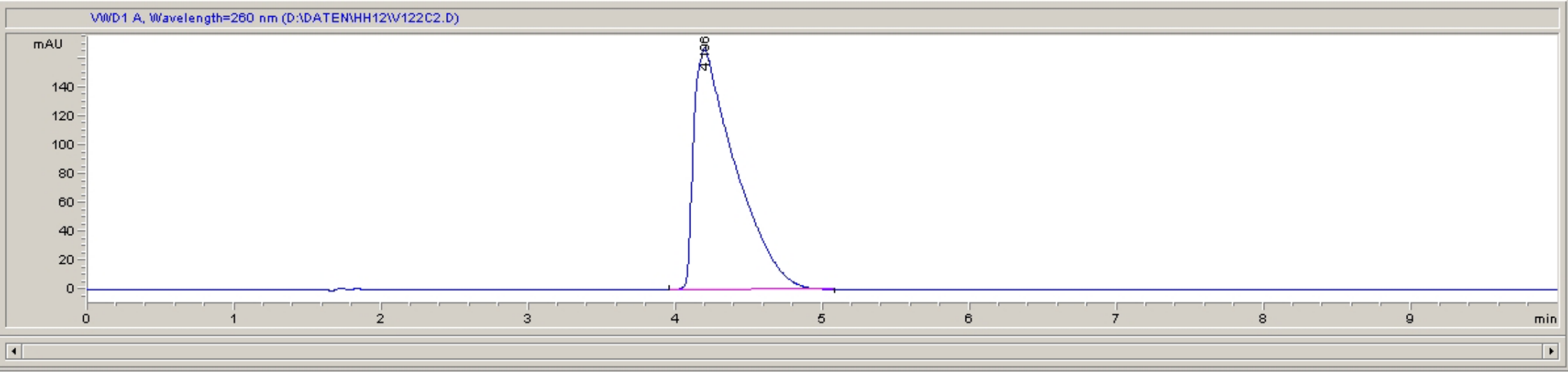

mai11av.20.fid
Metternich 3-OH-C12-NA
CDCl3
HDO
HDO
HDO
1.96
0.95
1.00
2.00
1.01
1.08
1.07
1.16
2.29
8.92
0.42
3.02
f1 (ppm)


mai11av.21.fid
Metternich 3-OH-C12-NA
172.44
149.52
135.99
131.44
123.43
77.33 CDCl3
77.08 CDCl3
76.83 CDCl3
67.98
63.71
41.59
36.75
31.83
29.52
29.48
29.25
25.45
22.62
14.05
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)


VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-0H.D)
mAU
10.920
12.612
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-12H.D)
mAU
9.856
11.481
13.351
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-24H.D)
mAU
10.271
12.209
14.187
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-48H.D)
mAU
11.530
14.632
17.149
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-72H.D)
mAU
11.522
14.480
16.956
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-97H.D)
mAU
9.999
11.750
13.711
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-12-144H.D)
mAU
15.977
23.848
28.138
min



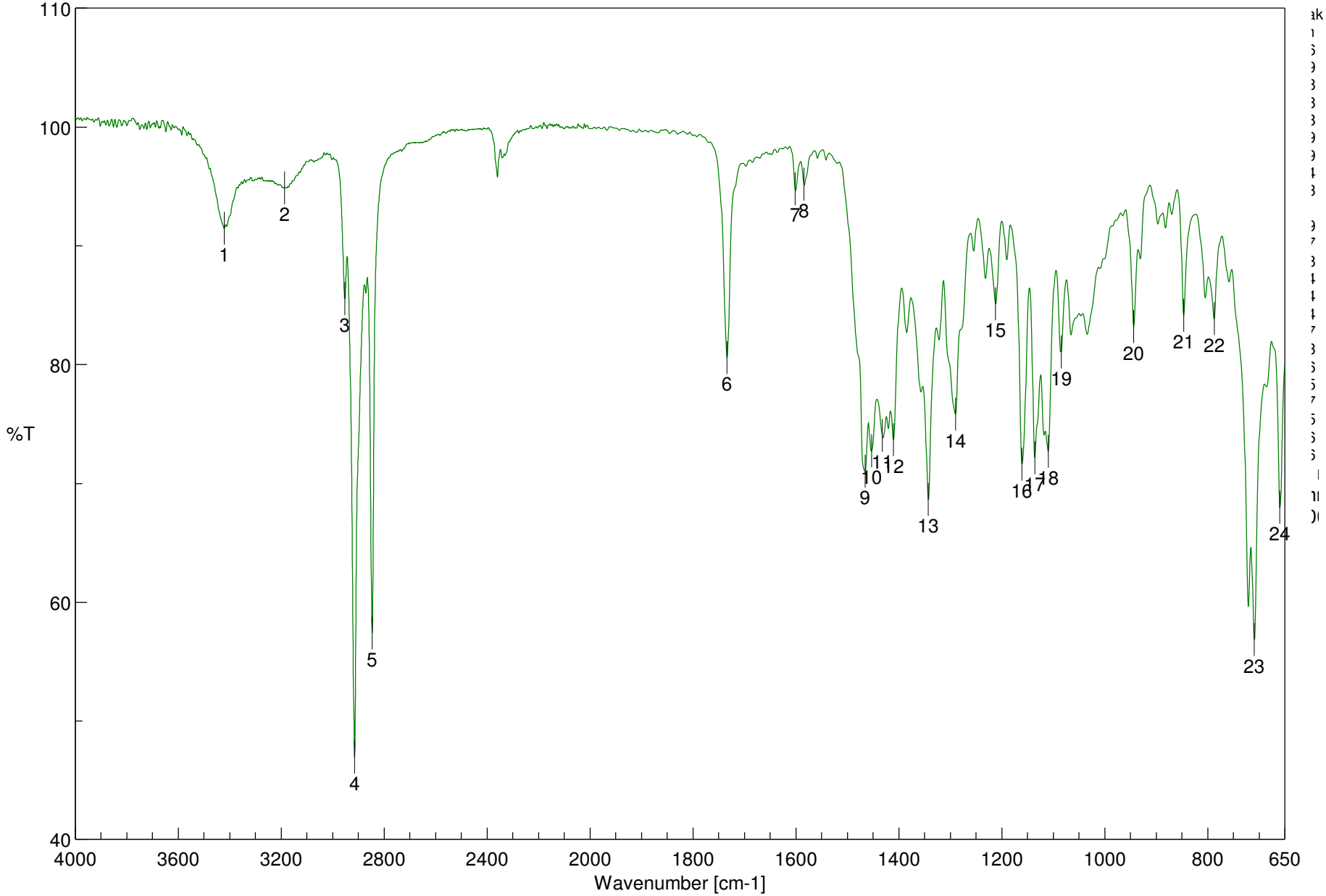
3-hydroxy tetradecanoic acid pyridin-3-ylmethylester **(4e).** Following the general procedure (A) from the substrate **3e** (333.5 mg, 0.1 mmol) the title molecule was obtained as a yellow solid (274.4 mg, 82% yield). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ (ppm) 8.54 (s, 1H), 8.50 (d, J = 4.9 Hz, 1H), 7.62 (d, J = 7.8 Hz, 1H), 7.22 (dd, J = 8.2, 5.1 Hz, 1H), 5.09 (s, 2H), 3.96 (tt, J = 7.9, 3.8 Hz, 1H), 2.48 (dd, J = 16.2, 3.4 Hz, 1H), 2.40 (dd, J = 16.2, 8.8 Hz, 1H), 1.46 (tt, J = 10.7, 4.5 Hz, 1H), 1.36 (dtd, J = 13.9, 9.7, 4.3 Hz, 2H), 1.19 (s, 15H), 1.11 (d, J = 5.7 Hz, 1H), 0.80 (t, J = 6.8 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 126 MHz) δ (ppm): 172.5, 149.6, 149.6, 136.0, 131.4, 123.5, 77.3, 77.1, 76.8, 68.0, 63.8, 41.5, 36.7, 31.9, 29.6, 29.6, 29.5, 29.5, 29.3, 25.5, 22.7, 14.1; IR v: 3422; 3187; 2952; 2915; 2847; 1734; 1161; 787; 710 cm^{-1}; HPLC: Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ;: ACN:H_2O 50:50 1.5ml/min (t_r = 4.20 min); Anal. Calcd for $C_{20}H_{33}NO_3$: C, 71.60; H, 9.91; N, 4.21. Found: C, 71.68; H, 10.24; N, 4.23.


VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V122C2.D)
mAU
140
120
100
80
60
40
20
0
4.196
0
1
2
3
4
5
6
7
8
9
min

3-OH-c14NA

[Comments]
Sample name
Comment
User

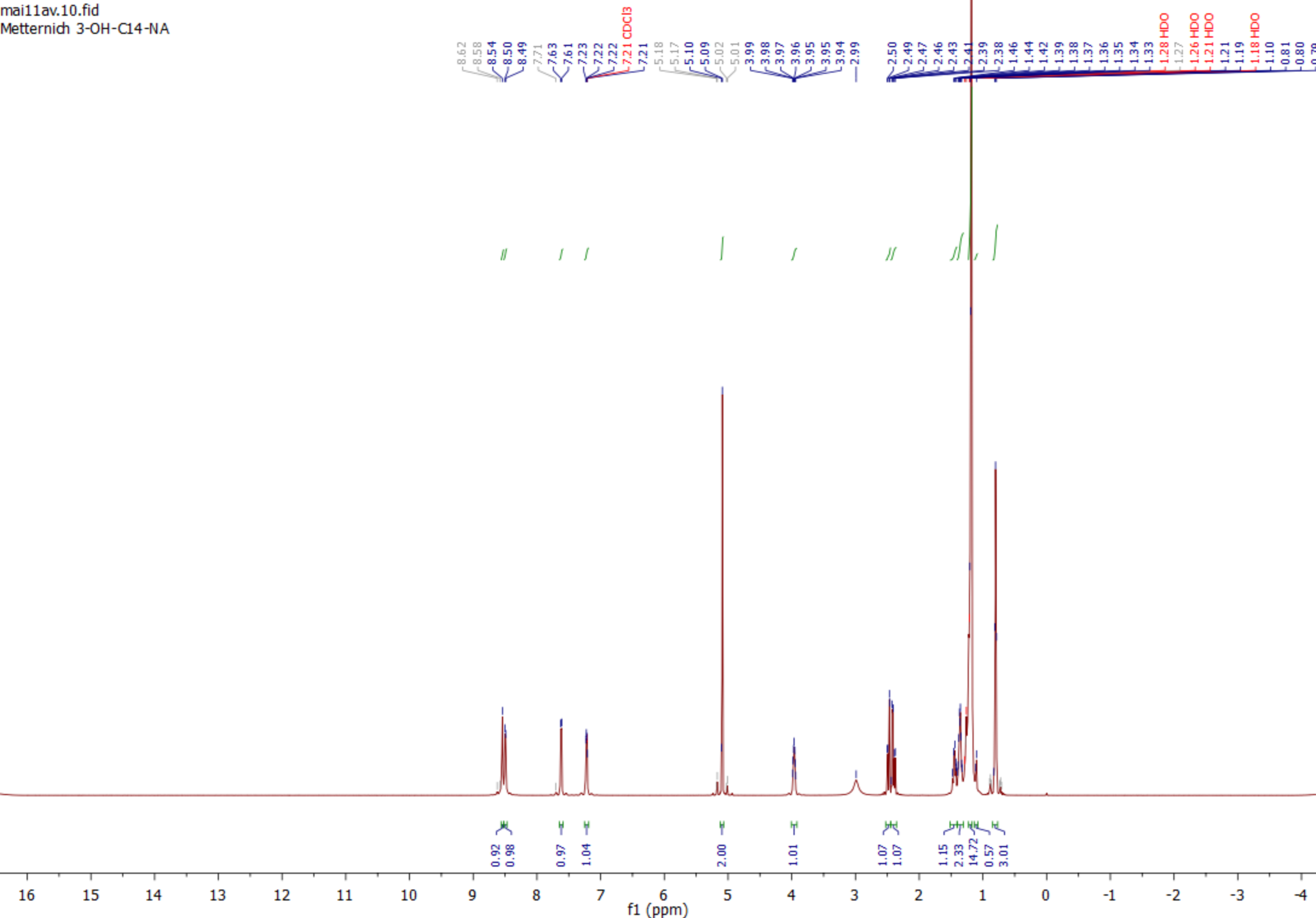
[Detailed Information]
Creation date 26.09.2017 13:25



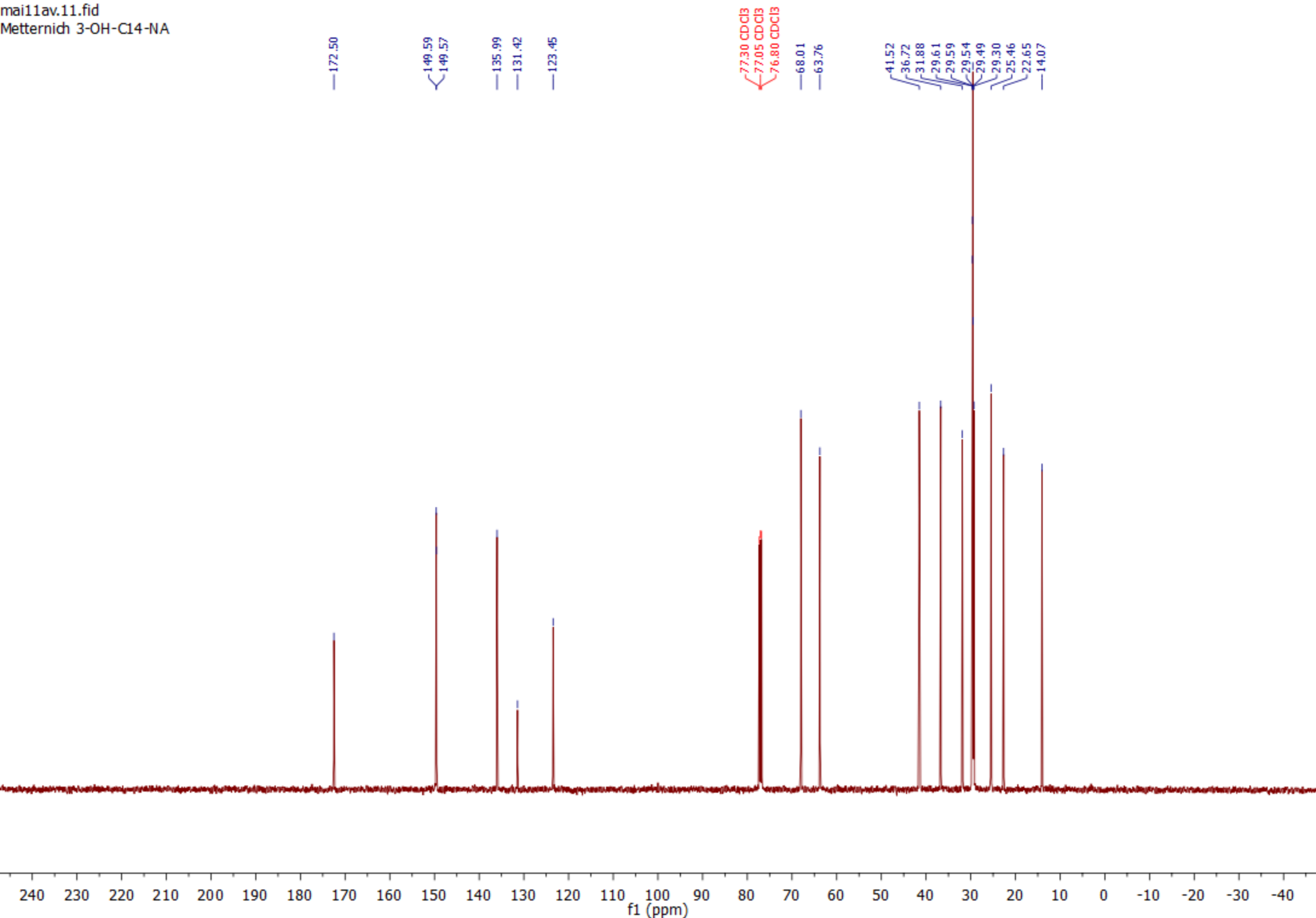
ak Find

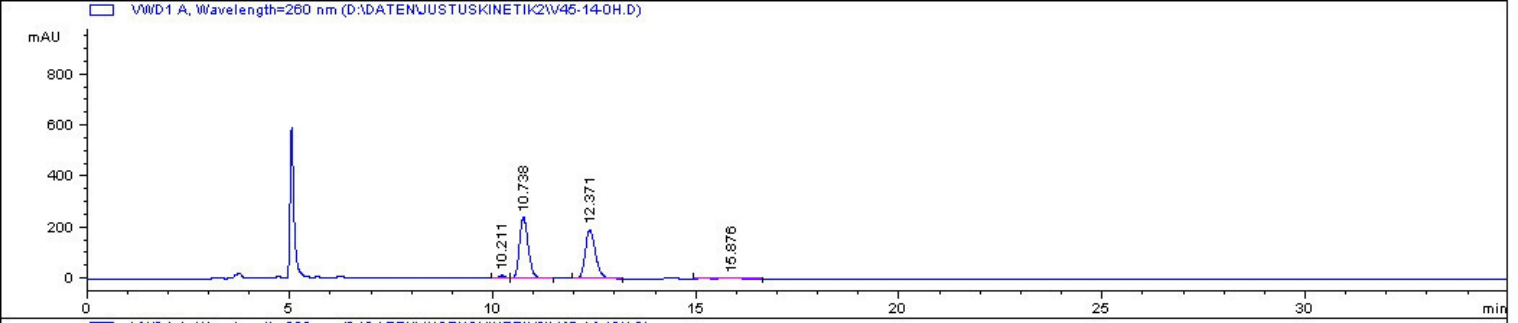
	Intensity
5	91.4443
9	94.8579
3	85.5279
3	46.9171
3	57.3818
9	80.5808
9	94.7698
4	95.1606
3	70.9798
	72.7120
9	73.9944
7	73.6427
3	68.6305
4	75.7904
4	85.0970
4	71.5977
7	72.1257
3	72.6951
5	81.0370
5	83.1705
7	84.1262
5	83.8484
5	56.8071
5	67.9475

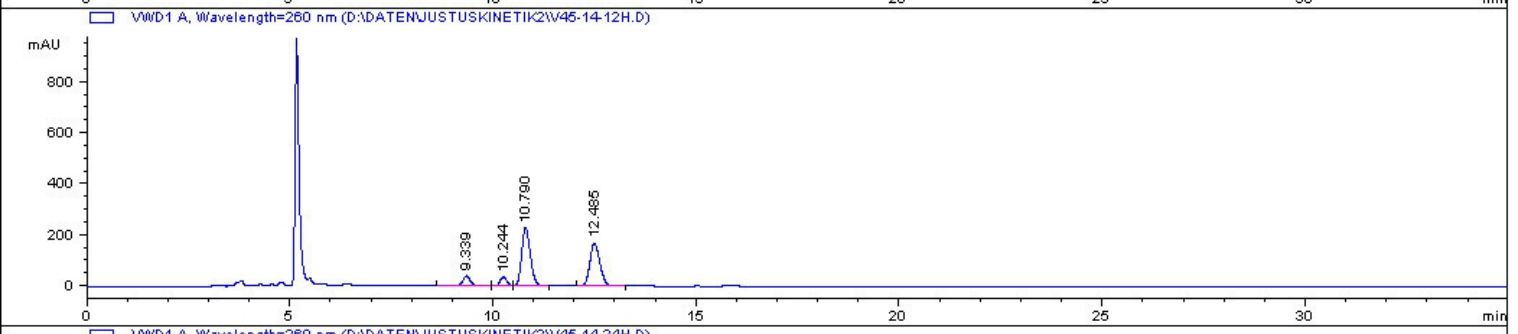
mai11av.10.fid
Metternich 3-OH-C14-NA

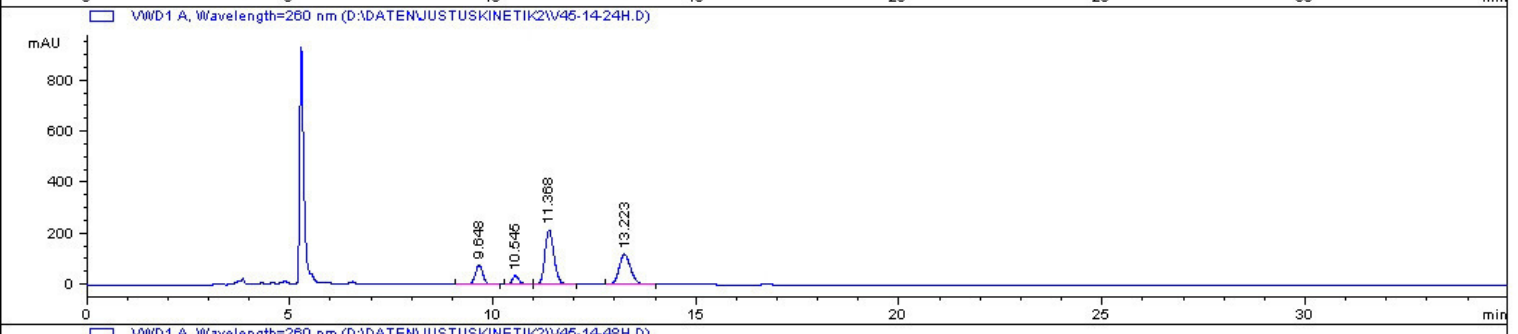


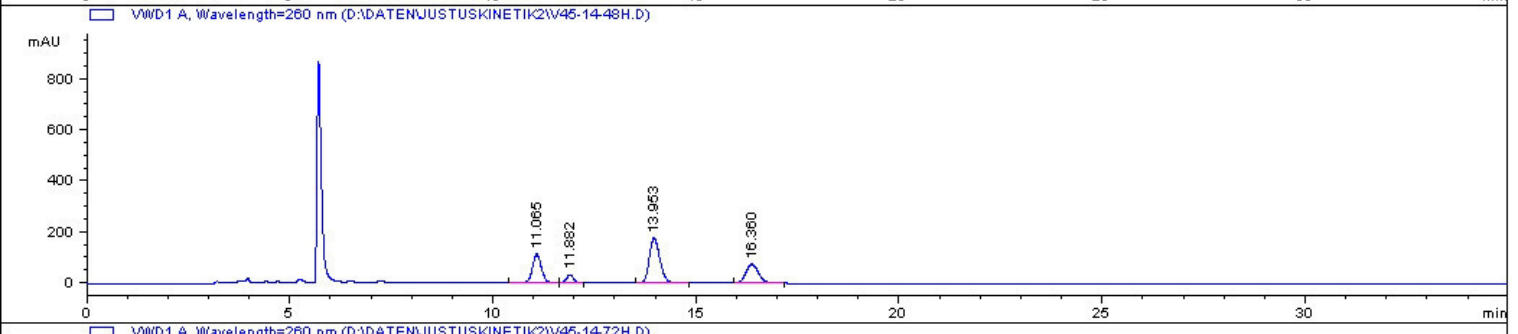
mai11av.11.fid
Metternich 3-OH-C14-NA

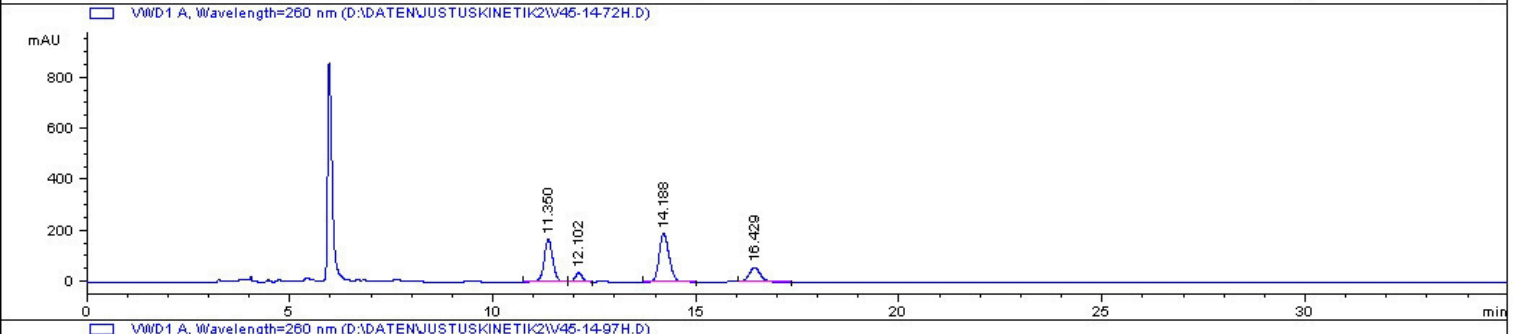


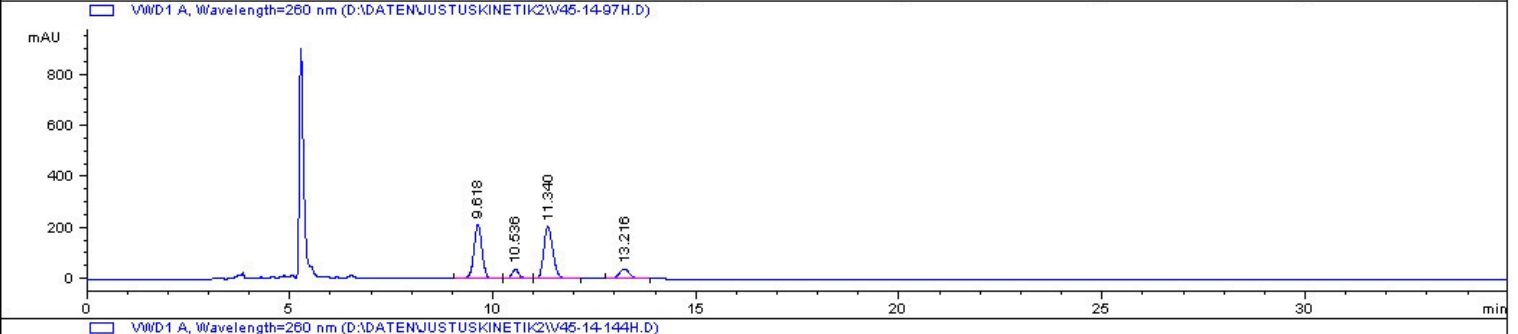

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-0H.D)
mAU
10.211
10.738
12.371
15.876
min

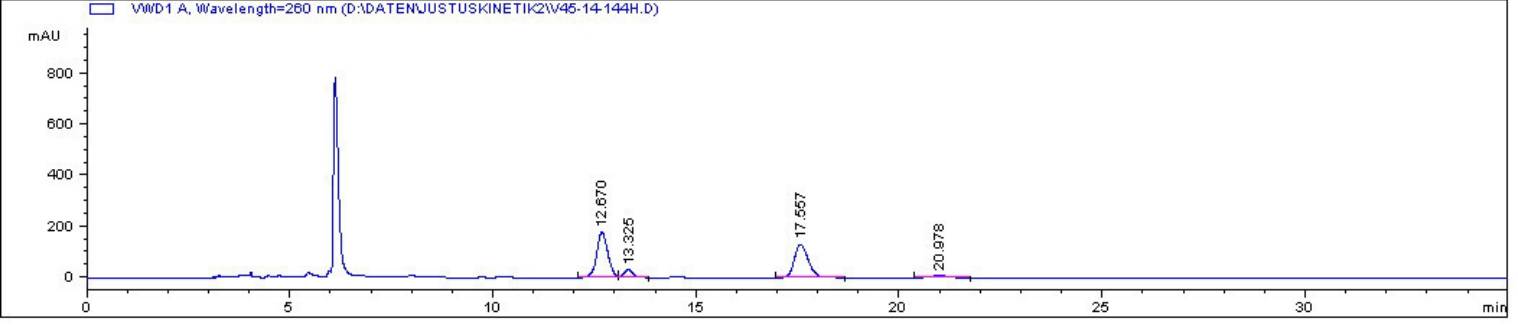

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-12H.D)
mAU
9.339
10.244
10.790
12.485
min

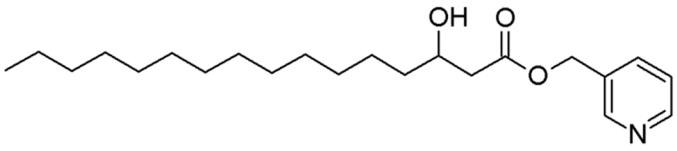

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-24H.D)
mAU
9.648
10.545
11.368
13.223
min


VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-48H.D)
mAU
11.065
11.882
13.953
16.360
min

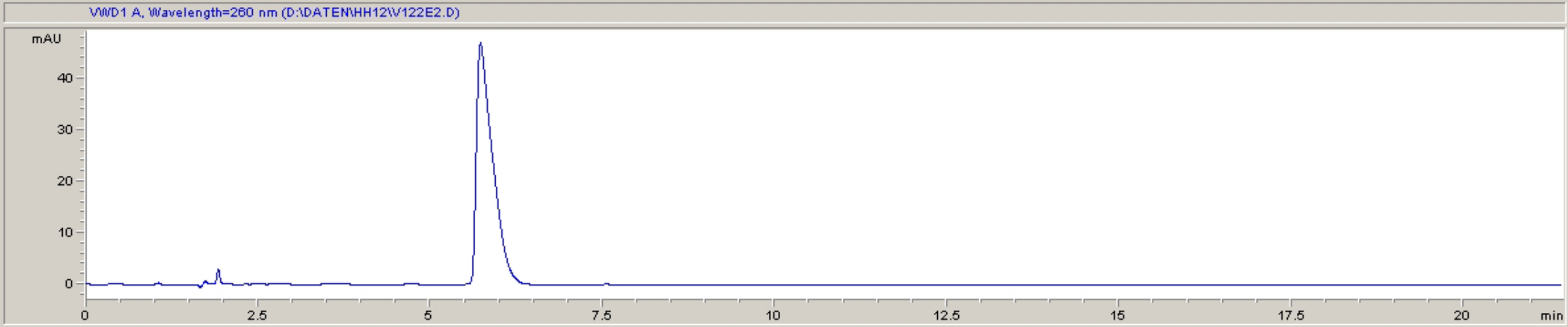

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-72H.D)
mAU
11.350
12.102
14.188
16.429
min

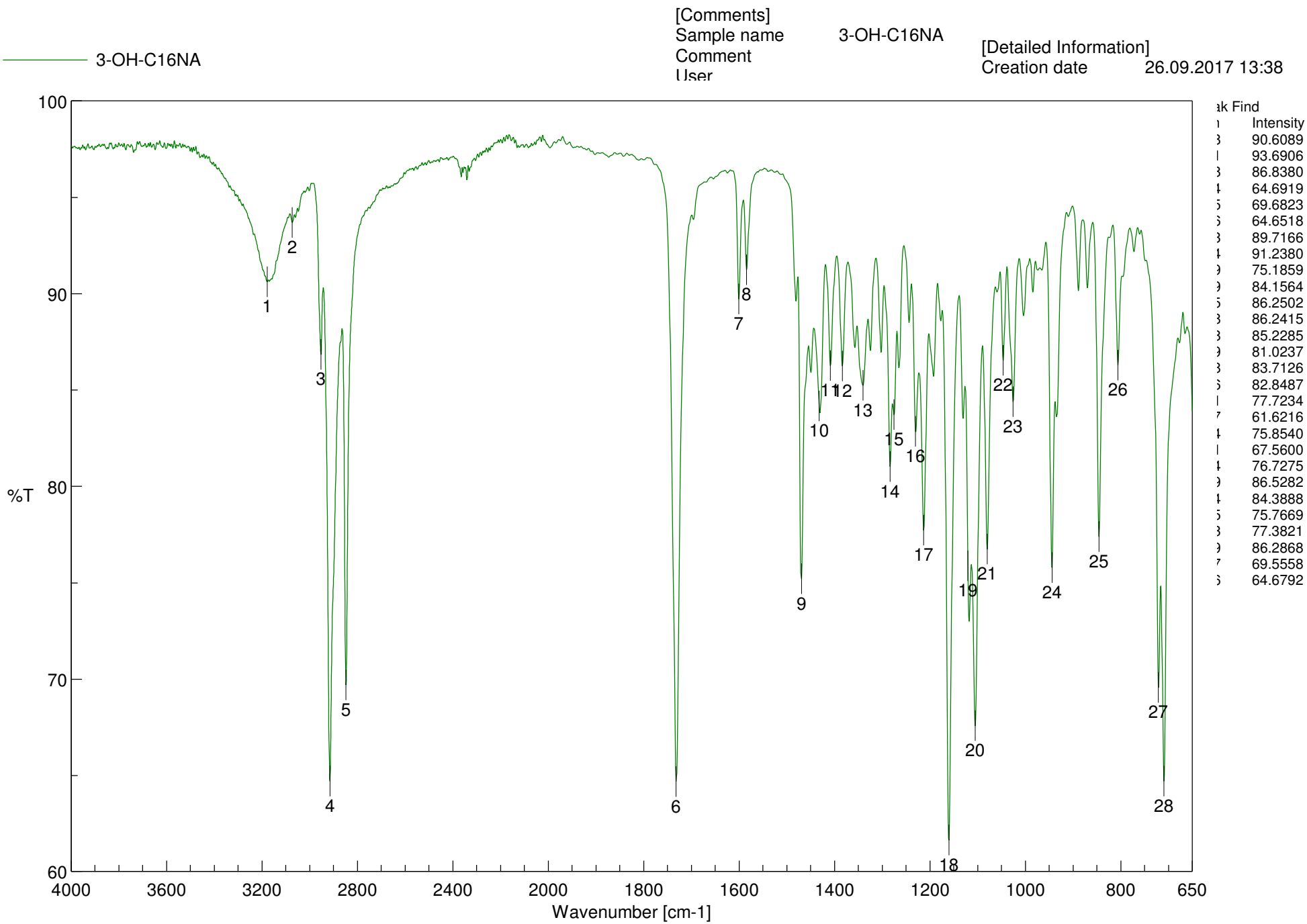

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-97H.D)
mAU
9.618
10.536
11.340
13.216
min

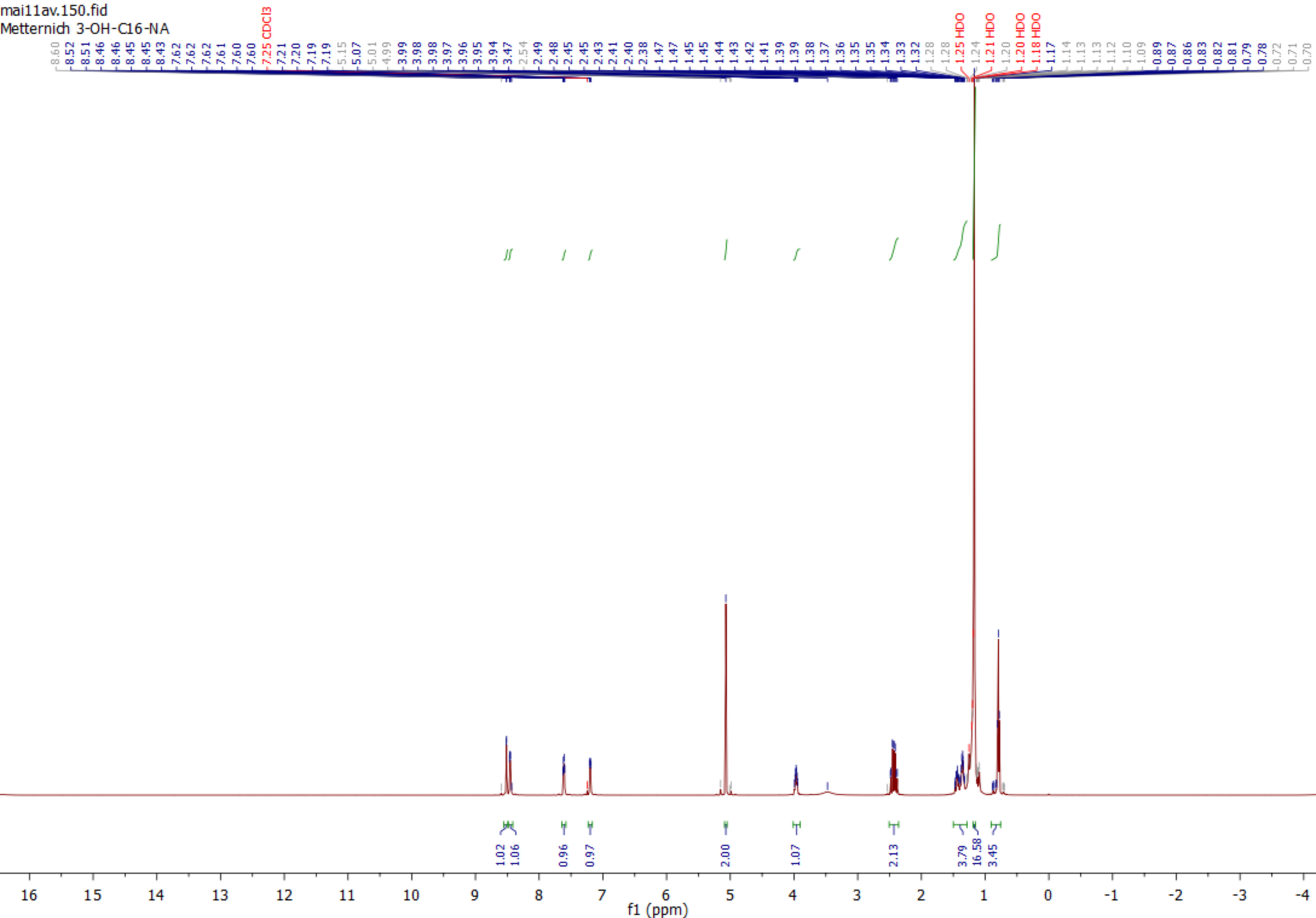

VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUS\KINETIK2\V45-14-144H.D)
mAU
12.670
13.325
17.557
20.978
min

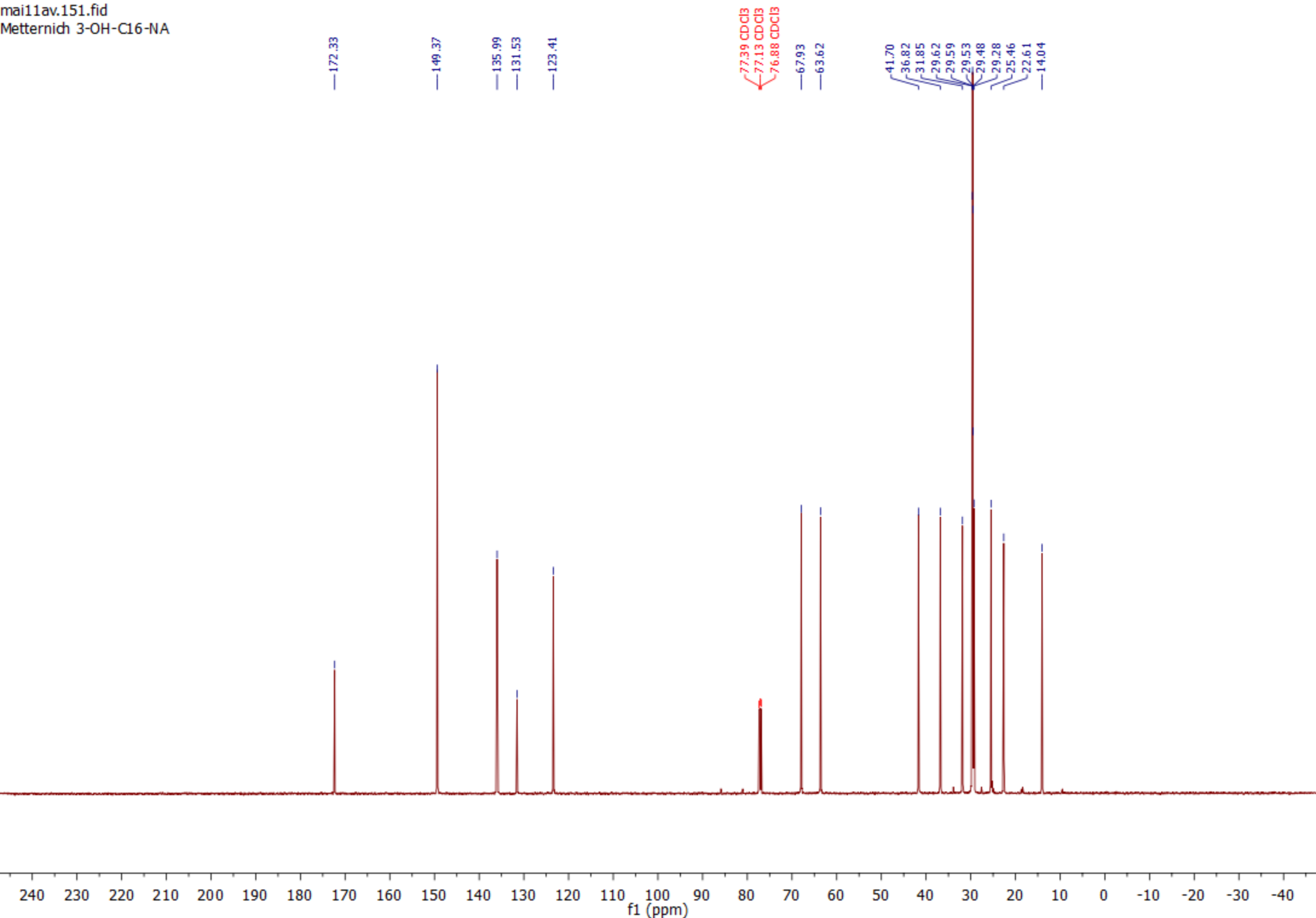


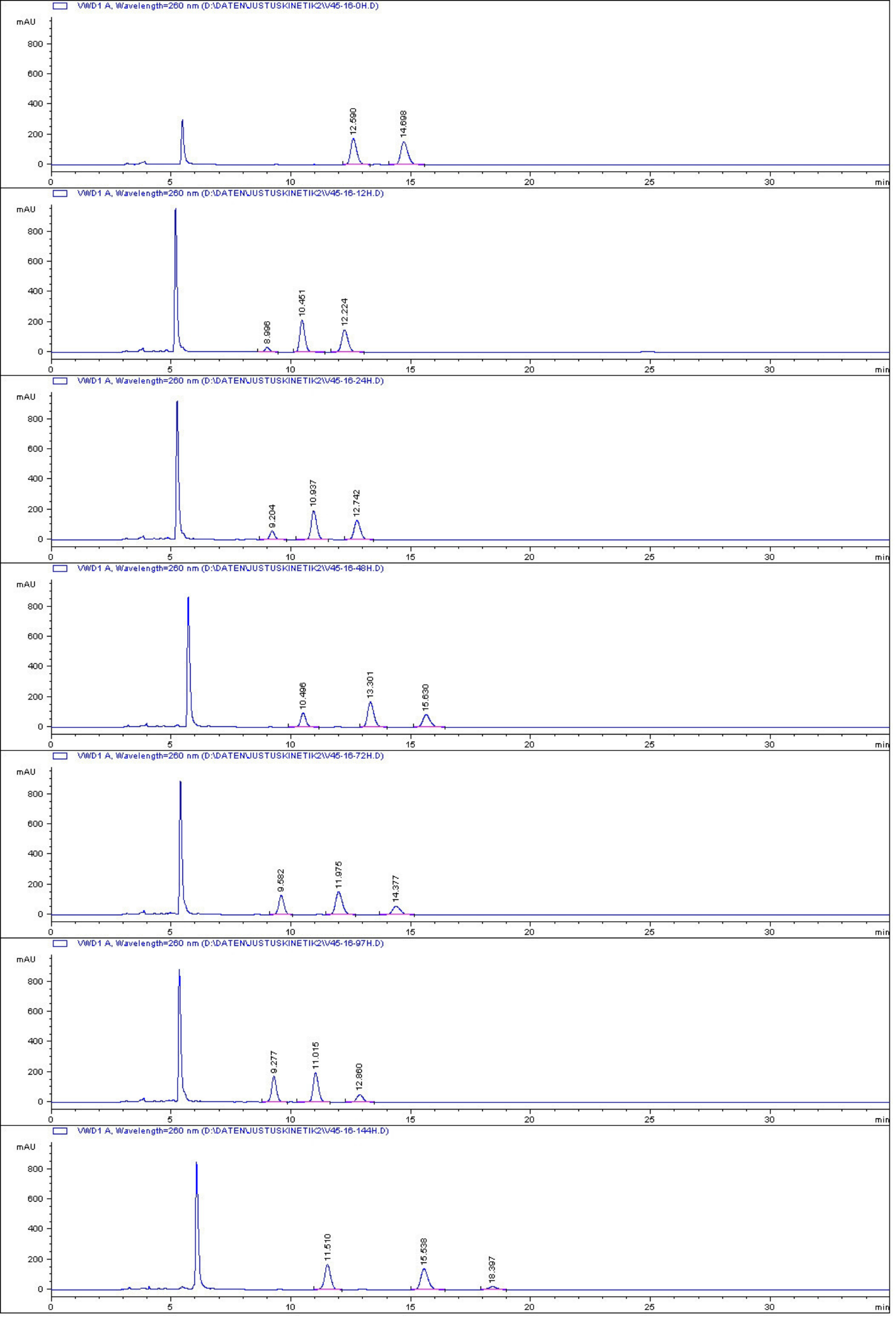
3-hydroxy hexadecanoic acid pyridin-3-ylmethylester **(4f).** Following the general procedure (A) from the substrate **3f** (361.5 mg, 0.1 mmol) the title molecule was obtained as a yellow solid (316 mg, 86% yield). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ (ppm): 8.51 (d, J = 2.2 Hz, 1H), 8.46 (dd, J = 4.9, 1.7 Hz, 1H), 7.61 (dt, J = 7.9, 2.0 Hz, 1H), 7.20 (dd, J = 7.8, 4.8 Hz, 1H), 5.07 (s, 2H), 3.97 (tt, J = 8.2, 4.0 Hz, 1H), 2.51 - 2.36 (m, 2H), 1.50 - 1.29 (m, 4H), 1.17 (s, 17H), 0.79 (t, J = 6.9 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 126 MHz) δ (ppm): 172.3, 149.4, 136.0, 131.5, 123.4, 67.9, 63.6, 41.7, 36.8, 31.9, 29.6, 29.6, 29.5, 29.5, 29.3, 25.5, 22.6, 14.0; IR v: 3179; 3073; 2952; 2916; 2848; 1732; 1160; 1026; 710 cm^{-1}; HPLC: Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ; ACN:H_2O 60:40 1,5ml/min (t_r =5.73 min); Anal. Calcd for $C_{22}H_{37}NO_3$: C, 72.69; H, 10.26; N, 3.85. Found: C, 72.48; H, 10.33; N, 3.710.

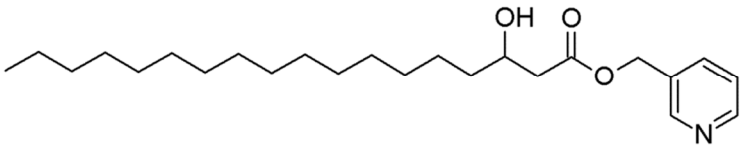

VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V122E2.D)
mAU
40
30
20
10
0
0
2.5
5
7.5
10
12.5
15
17.5
20
min


3-OH-C16NA
[Comments]
Sample name 3-OH-C16NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 13:38
ak Find
Intensity
90.6089
93.6906
86.8380
64.6919
69.6823
64.6518
89.7166
91.2380
75.1859
84.1564
86.2502
86.2415
85.2285
81.0237
83.7126
82.8487
77.7234
61.6216
75.8540
67.5600
76.7275
86.5282
84.3888
75.7669
77.3821
86.2868
69.5558
64.6792
%T
100
90
80
70
60
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28

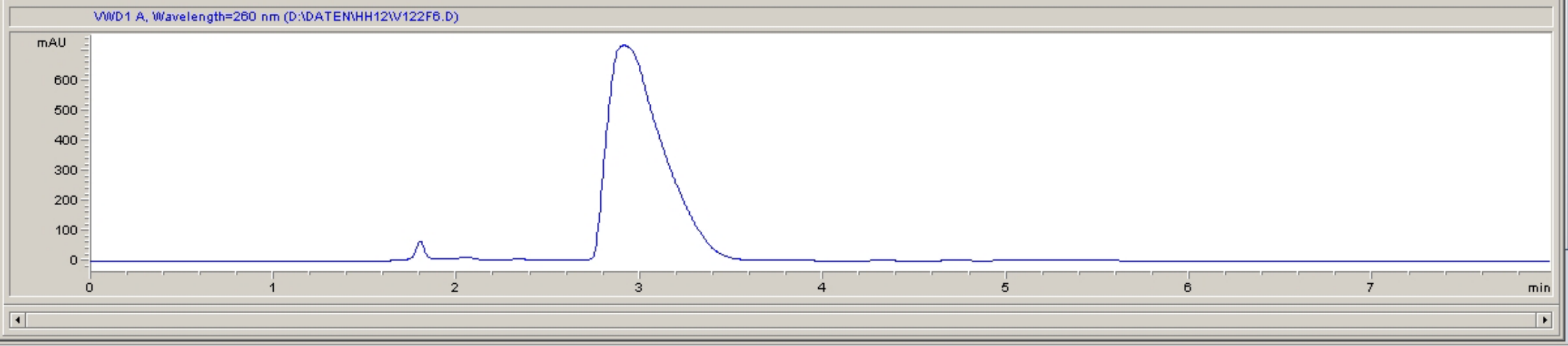

mai11av.150.fid
Metternich 3-OH-C16-NA
7.25 CDCl3
1.25 HDO
1.21 HDO
1.20 HDO
1.18 HDO
1.02
1.06
0.96
0.97
2.00
1.07
2.13
3.79
16.58
3.45
f1 (ppm)

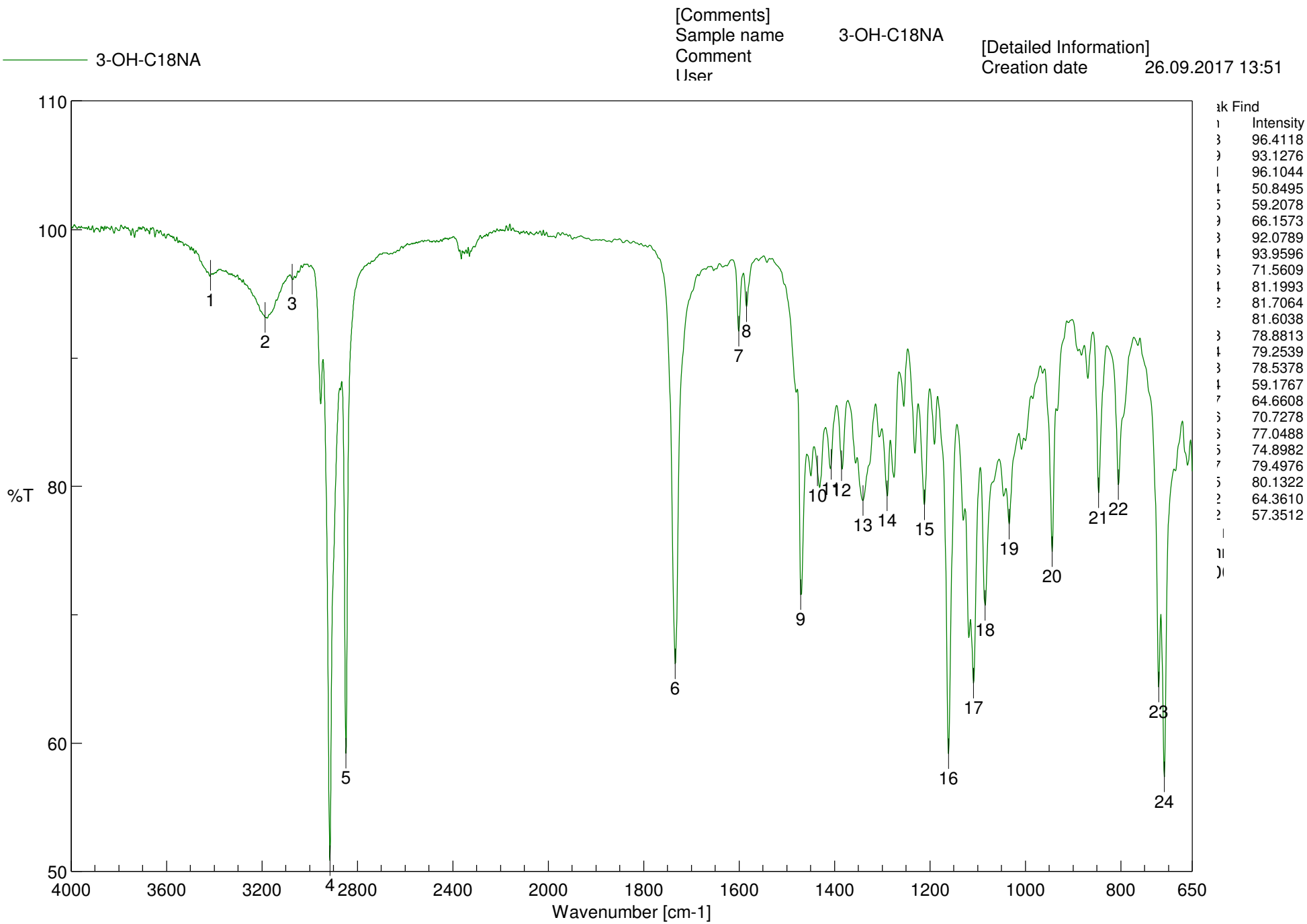

mai11av.151.fid
Metternich 3-OH-C16-NA
172.33
149.37
135.99
131.53
123.41
77.39 CDCl3
77.13 CDCl3
76.88 CDCl3
67.93
63.62
41.70
36.82
31.85
29.62
29.59
29.53
29.48
29.28
25.46
22.61
14.04
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40
f1 (ppm)


VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-0H.D)
mAU
12.590
14.698
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-12H.D)
mAU
8.996
10.451
12.224
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-24H.D)
mAU
9.204
10.937
12.742
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-48H.D)
mAU
10.496
13.301
15.630
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-72H.D)
mAU
9.582
11.975
14.377
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-97H.D)
mAU
9.277
11.015
12.860
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-16-144H.D)
mAU
11.510
15.538
18.397
min

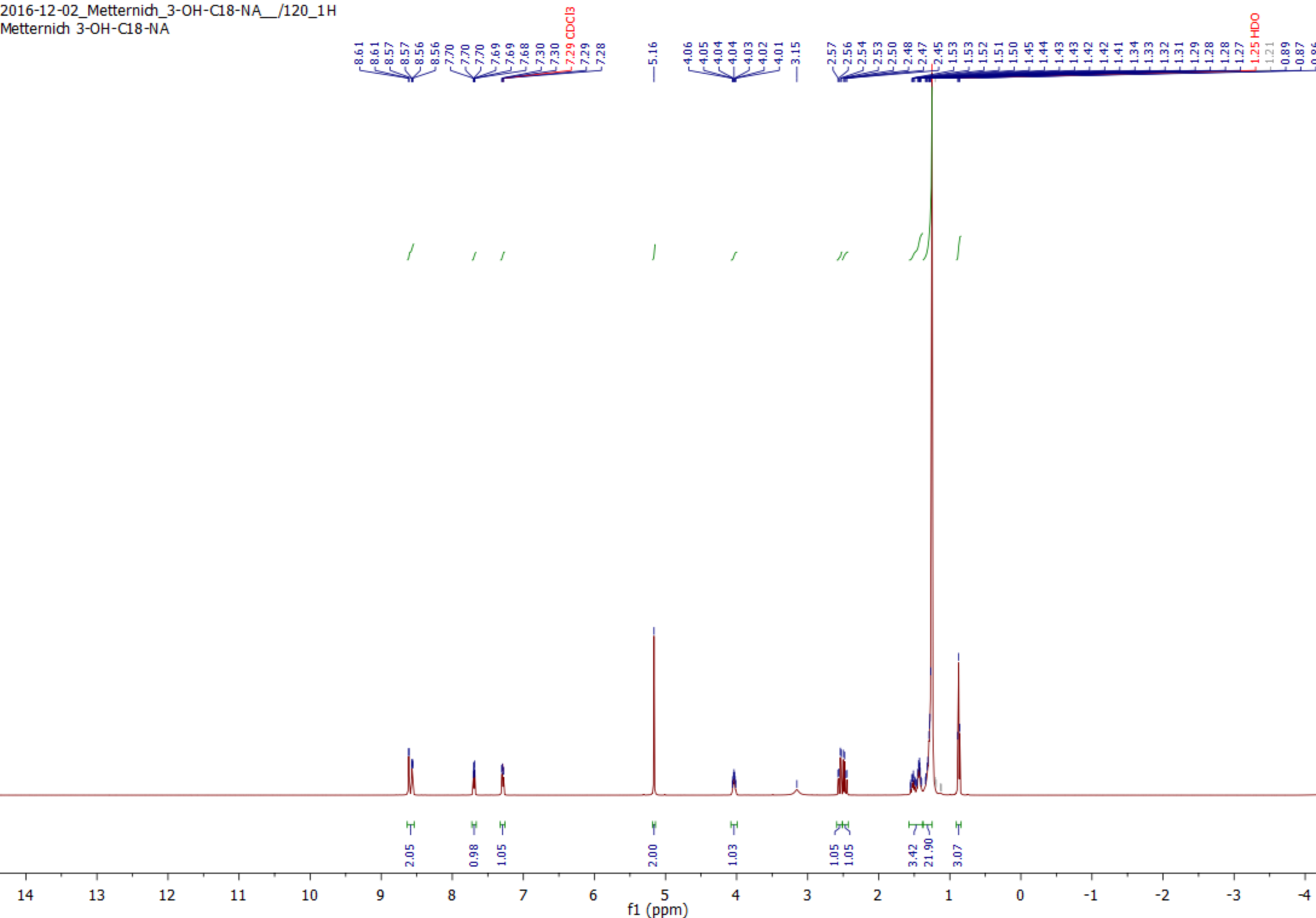


3-hydroxy octadecanoic acid pyridin-3-ylmethylester **(4g).** Following the general procedure (A) from the substrate **3g** (389.5 mg, 0.1 mmol) the title molecule was obtained as a yellow solid (365.5 mg, 93% yield). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ (ppm): 8.63 - 8.54 (m, 2H), 7.69 (dt, J = 7.8, 1.9 Hz, 1H), 7.29 (dd, J = 8.5, 4.1 Hz, 1H), 5.16 (s, 2H), 4.04 (tt, J = 7.9, 3.8 Hz, 1H), 2.55 (dd, J = 16.1, 3.4 Hz, 1H), 2.47 (dd, J = 16.2, 8.8 Hz, 1H), 1.57 - 1.38 (m, 3H), 1.38 - 1.25 (m, 22H), 0.87 (t, J = 6.9 Hz, 3H); ^{13}C-NMR ($CDCl_3$, 126 MHz) δ (ppm): 172.5, 149.5, 136.0, 131.5, 123.5, 68.0, 63.7, 41.5, 36.7, 31.9, 29.66, 29.62, 29.55, 29.49, 29.3, 25.5, 22.7, 14.1; IR v: 3416; 3187; 3073; 2916; 2848; 1734; 1161; 709 cm^{-1}; HPLC: Macherey-Nagel EC 250/4 Nucleodur 300-5-C4ec ; ACN:H_2O 70:30; 1,5ml/min; t_r = 2.91 min; Anal. Calcd for $C_{24}H_{41}NO_3$: C, 73.61; H, 10.55; N, 3.48. Found: C, 73.32; H, 10.59; N, 3.517.

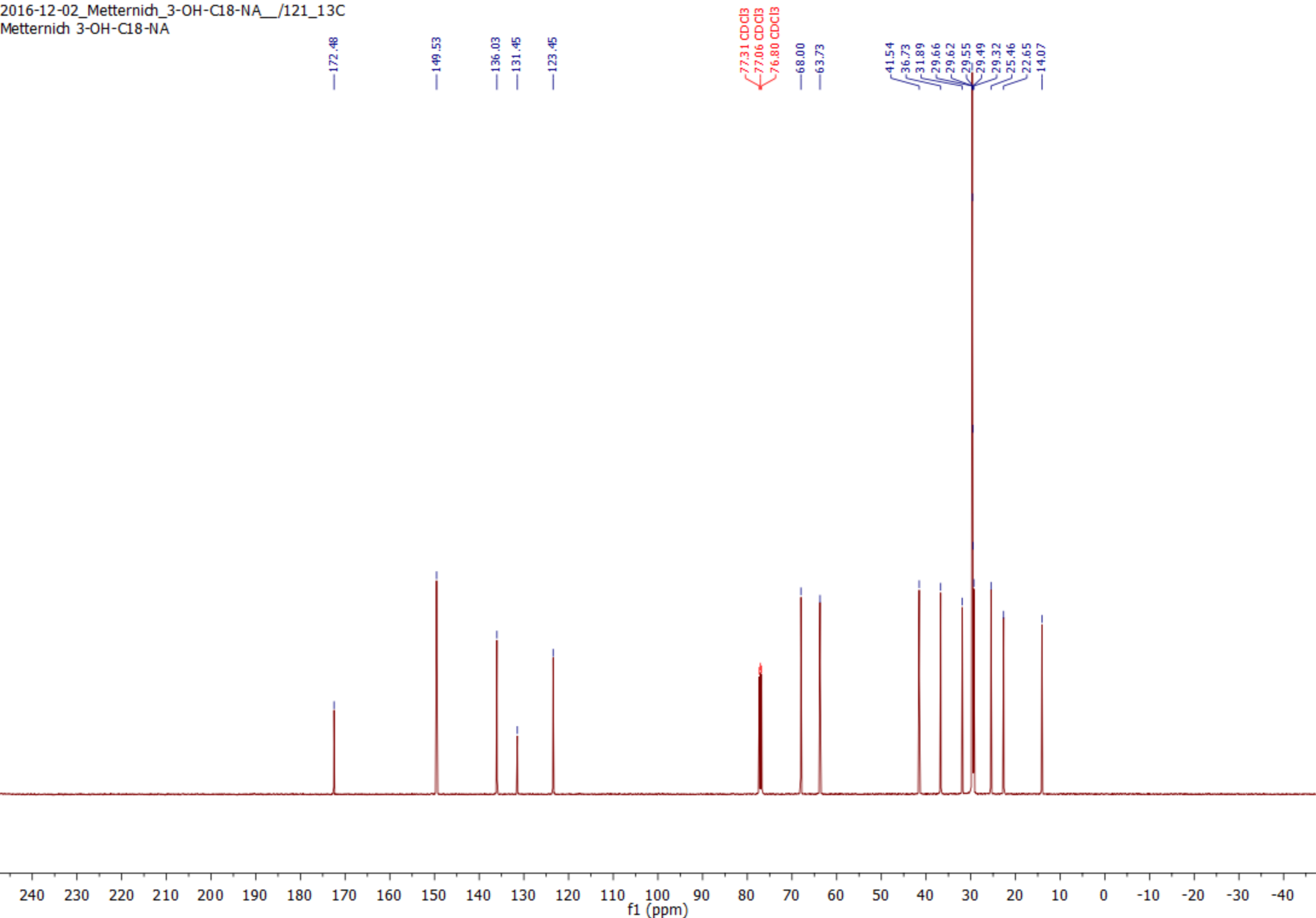

VWD1 A, Wavelength=260 nm (D:\DATEN\HH12\V122F6.D)
mAU
600
500
400
300
200
100
0
0
1
2
3
4
5
6
7
min

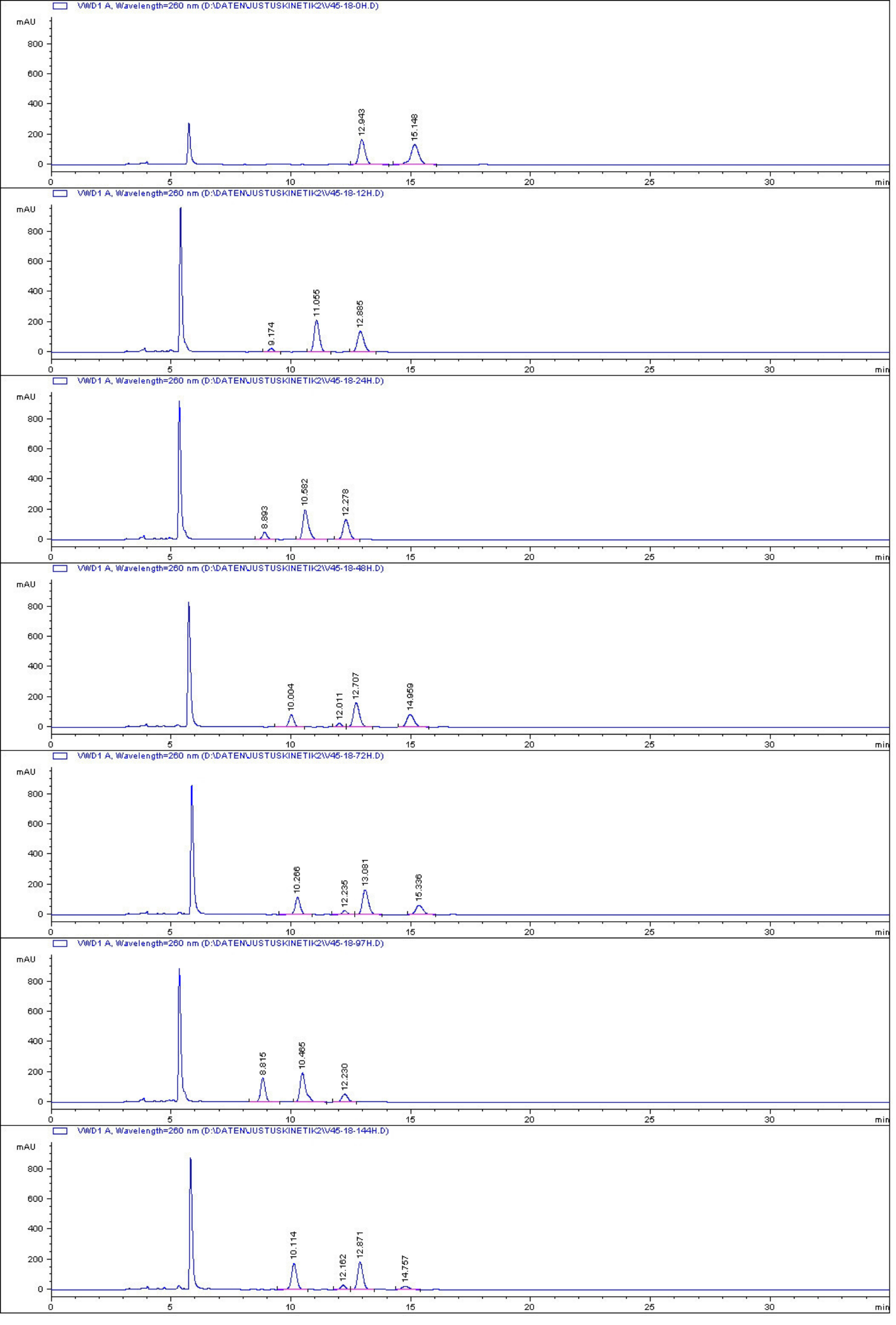

3-OH-C18NA
[Comments]
Sample name 3-OH-C18NA
Comment
User
[Detailed Information]
Creation date 26.09.2017 13:51
ak Find
Intensity
96.4118
93.1276
96.1044
50.8495
59.2078
66.1573
92.0789
93.9596
71.5609
81.1993
81.7064
81.6038
78.8813
79.2539
78.5378
59.1767
64.6608
70.7278
77.0488
74.8982
79.4976
80.1322
64.3610
57.3512
%T
110
100
80
60
50
4000
3600
3200
2800
2400
2000
1800
1600
1400
1200
1000
800
650
Wavenumber [cm-1]
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24

2016-12-02_Metternich_3-OH-C18-NA__/120_1H
Metternich 3-OH-C18-NA



2016-12-02_Metternich_3-OH-C18-NA__/121_13C
Metternich 3-OH-C18-NA




VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-0H.D)
mAU
12.943
15.148
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-12H.D)
mAU
9.174
11.055
12.885
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-24H.D)
mAU
8.893
10.582
12.278
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-48H.D)
mAU
10.004
12.011
12.707
14.959
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-72H.D)
mAU
10.266
12.235
13.081
15.336
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-97H.D)
mAU
8.815
10.485
12.230
min
VWD1 A, Wavelength=260 nm (D:\DATEN\JUSTUSKINETIK2\W45-18-144H.D)
mAU
10.114
12.162
12.871
14.757
min

compound	time/h	conversion/%	ee(substrate)
C8	0	0	0,48775975
	12	13,1036505	12,2563652
	24	23,5322924	26,2923791
	48	37,0257578	49,0122342
	72	45,6062995	71,6257667
	97	53,3081539	83,4906109
	144	59,2113841	100
C10	0	0	0,16878631
	12	13,3785541	12,5742075
	24	23,6792185	26,0767485
	48	37,7904013	51,1665796
	72	46,7764998	71,515587
	97	52,3465227	84,9455114
	144	59,3567498	100
C12	0	0	0,4891566
	12	8,99733775	9,9822057
	24	17,4370113	20,0840409
	48	29,8400852	37,6725487
	72	38,7754567	54,3719693
	97	44,9881796	68,5769393
	144	51,0765437	86,372871
C14	0	0	4,43084115
	12	13,6044147	7,71215167
	24	20,8161847	17,2376899
	48	31,5027276	33,0093643
	72	40,1376061	49,1572846
	97	46,8973236	64,46957
	144	53,7953878	85,5190466
C16	0	0	0,11464255
	12	6,76334647	6,44454397
	24	13,029233	13,2566357
	48	23,0331748	26,5290582
	72	31,5562474	40,0820132
	97	37,8878831	53,2378273
	144	46,0646044	72,7634282
C18	0	0	4,38234675
	12	5,21683726	10,0502232
	24	10,5208012	16,3051279
	48	24,5895801	23,4789484
	72	32,6107811	36,6096782
	97	34,1890702	55,0057389
	144	47,5619418	71,3493331