

Supplementary Materials

Analogues of muraymycin nucleoside antibiotics with epimeric uridine-derived core structures

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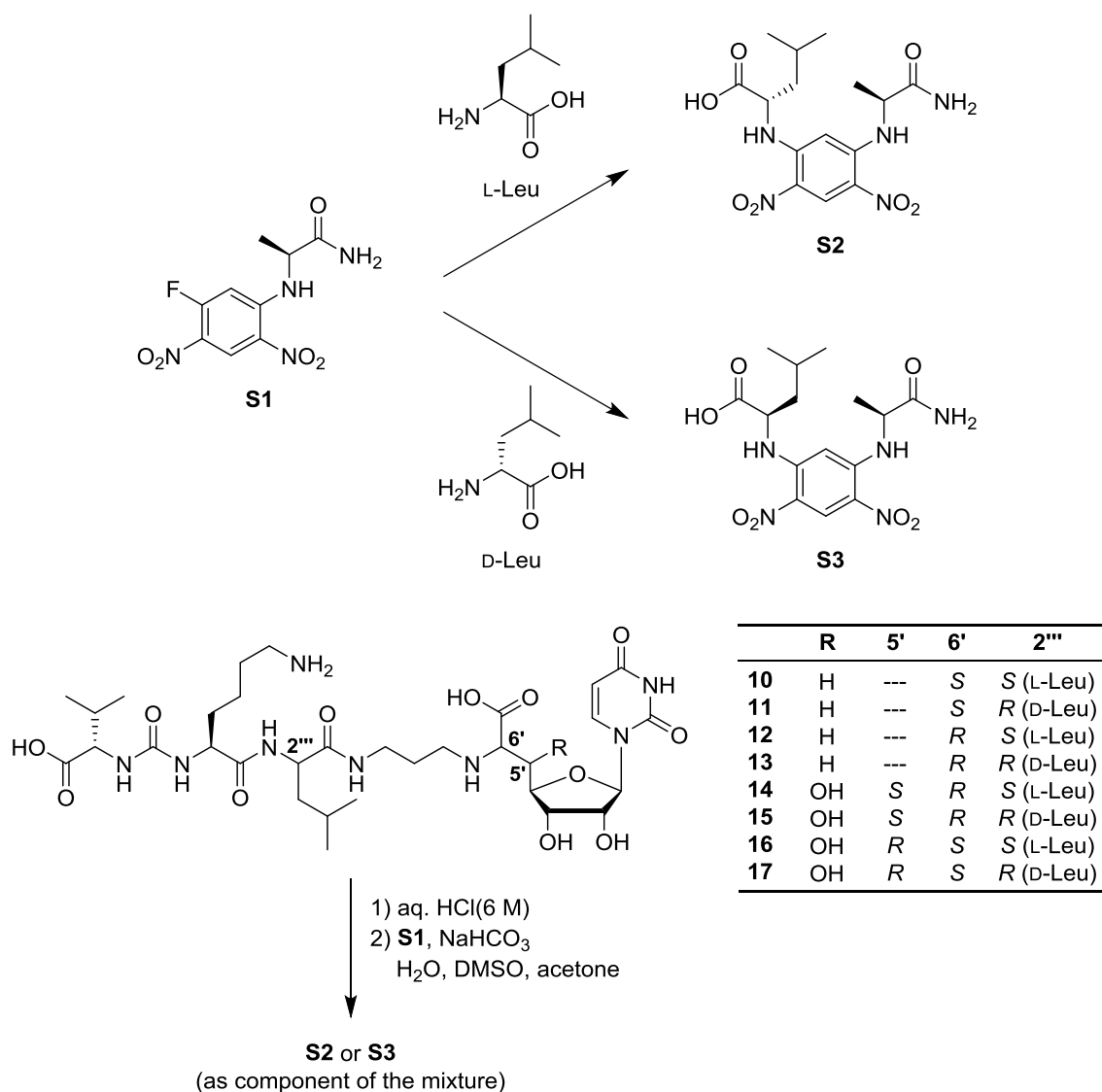
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Analysis of peptide units using Marfey's reagent

Peptide coupling of uridine-derived building blocks **19-22** with the urea tripeptide **18** and subsequent global deprotection gave two compounds in each of these two-step transformations. Each such pair of compounds showed identical masses and could be separated by preparative HPLC, thus identifying them as diastereomers. We assumed that epimerization in the 2'''-position (*i.e.*, at leucine-C- α) had occurred during the peptide coupling, giving rise to the formation of epimers (cf. Scheme 5 and Table 1). To confirm this assumption and assign the correct stereoconfiguration to each epimer, we used a method based on derivatization with Marfey's reagent **S1** [1], similar to an approach reported by Ichikawa, Matsuda and co-workers [2]. First, we synthesized two reference compounds **S2** and **S3** from L-configured Marfey's reagent **S1** with L-leucine and D-leucine, respectively, in the presence of base (Scheme S1). The L-L-isomer **S2** (obtained from L-leucine) showed a shorter retention time in analytical RP-HPLC relative to the D-L-isomer **S3** (obtained from D-leucine). This behaviour results from the enhanced tendency to form intramolecular H-bond interactions within **S3**, making it less polar than **S2** [1-3].

We then performed an acidic hydrolysis of the isolated muraymycin analogues **10-17** with aqueous HCl (6 M), followed by a transformation of the resultant mixture with Marfey's reagent **S1** in the presence of base (Scheme S1). This procedure should either give **S2** or **S3** as a component of the mixture, depending on the configuration of **10-17** in the 2'''-position (*i.e.*, at leucine-C- α). We therefore analyzed the mixtures by RP-HPLC and additionally performed co-injection experiments. For these, we doped the samples with authentic L-leucine or D-leucine derivatives **S2** or **S3**, respectively, and demonstrated an increased peak area at the respective retention time. According RP-HPLC chromatograms are shown in Figures S1-S19.



Scheme S1. Synthesis of authentic standards **S2** and **S3** and reaction of Marfey's reagent with hydrolyzed muraymycin analogues **10-17** for stereochemical analysis of the peptide units.

Method for analytical HPLC. eluent A water, eluent B MeOH; 0-15 min gradient of B (80-90%), 15-25 min 100% B, 25-30 min 80% B; flow 1 mL/min.

Synthesis of standards S2 and S3. In two separate reactions, to a solution of either L-leucine or D-leucine (25 mM in water, 80 μ L, 2.0 μ mol) in DMSO (15 μ L), Marfey's reagent **S1** (20 mM in acetone, 50 μ L, 1.0 μ mol) and NaHCO₃ (1 M in water, 5 μ L, 5 μ mol) were added

and the reaction mixture was stirred at 40 °C for 15 h. Then, HCl (1 M, 10 μ L, 10 μ mol) was added. The solvent was removed by lyophilization, and the resultant residue was dissolved in DMSO (1 mL) and analyzed by RP-HPLC (t_R = 34.6 min (**S2**), t_R = 42.3 min (**S3**)). The eluents of the analytical HPLC runs were collected and analyzed by MS to confirm the identity of the respective compounds. **S2**: MS (ESI⁺): m/z = 406.2 [M+Na]⁺. HRMS (ESI⁺): calcd.: 406.1333 [M+Na]⁺, found: 406.1316. **S3**: MS (ESI⁺): m/z = 406.2 [M+Na]⁺. HRMS (ESI⁺): calcd.: 406.1333 [M+Na]⁺, found: 406.1330.

Analysis of the peptide units in 10-17. A solution of the respective muraymycin analogue **10-17** (0.1 mg, 0.1 μ mol) in HCl (6 M, 200 μ L) was heated to 120 °C for 20 h. The solvent was then removed under reduced pressure, and the resultant residue was dissolved in water (80 μ L) and DMSO (15 μ L). Marfey's reagent **S1** (20 mM in acetone, 50 μ L, 1.0 μ mol) and NaHCO₃ (1 M in water, 5 μ L, 5 μ mol) were added and the reaction mixture was stirred at 40 °C for 16 h. Then, HCl (1 M, 10 μ L, 10 μ mol) was added. The solvent was removed by lyophilization, and the resultant residue was dissolved in DMSO (1 mL) and analyzed by RP-HPLC. The according RP-HPLC chromatograms are shown in Figures S1-S19.

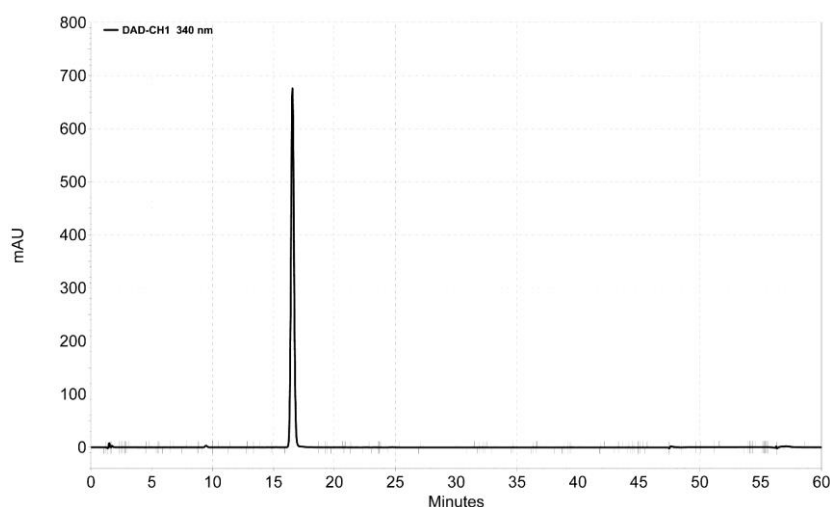


Figure S1. HPLC chromatogram of Marfey's reagent **S1**.

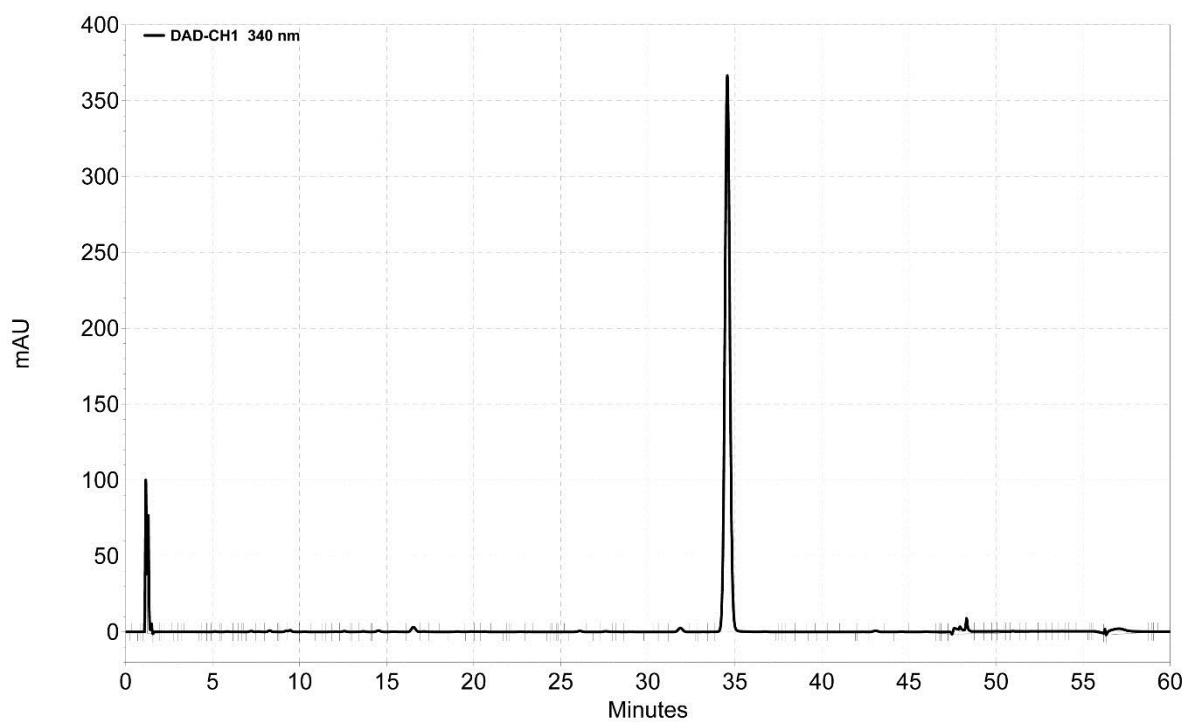


Figure S2. HPLC chromatogram of the L-L-configured authentic standard **S2**.

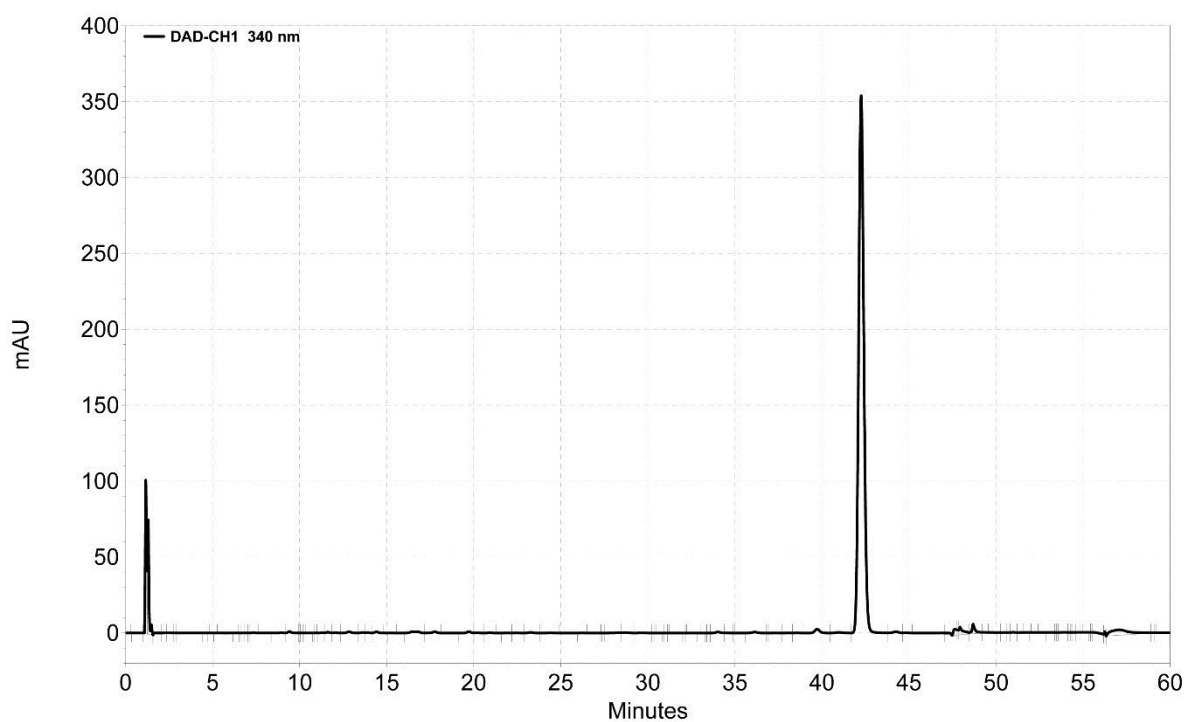


Figure S3. HPLC chromatogram of the D-L-configured authentic standard **S3**.

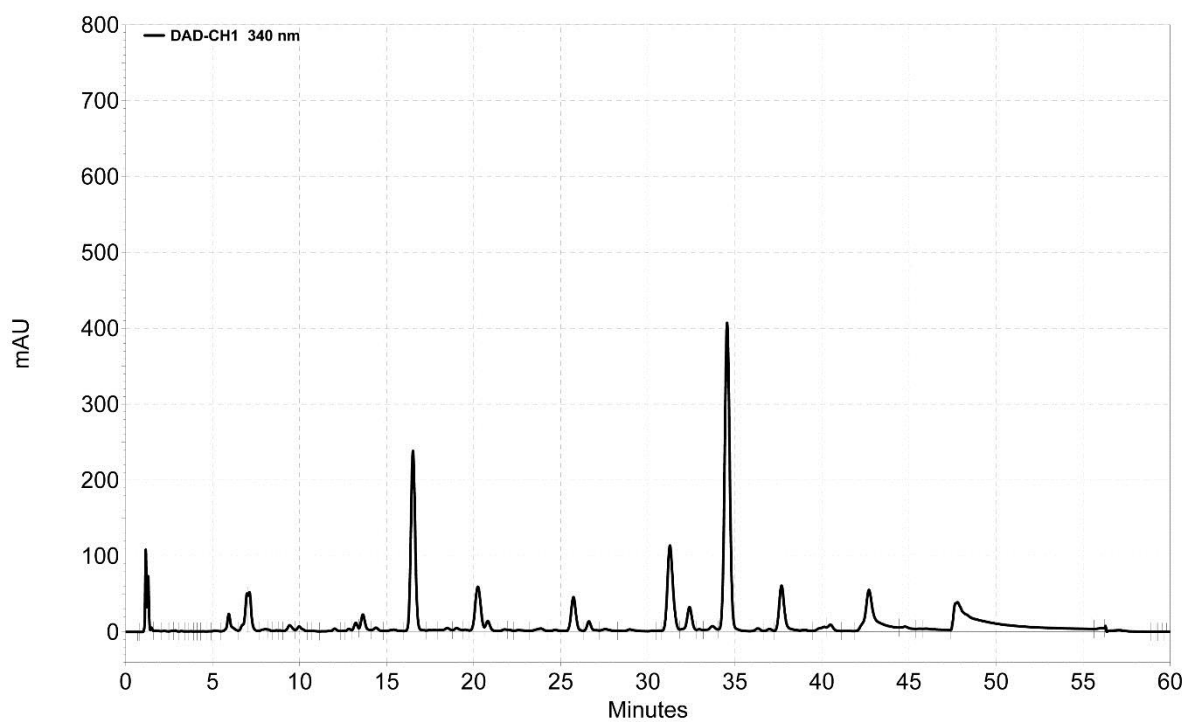


Figure S4. HPLC chromatogram of the mixture obtained from **10**.

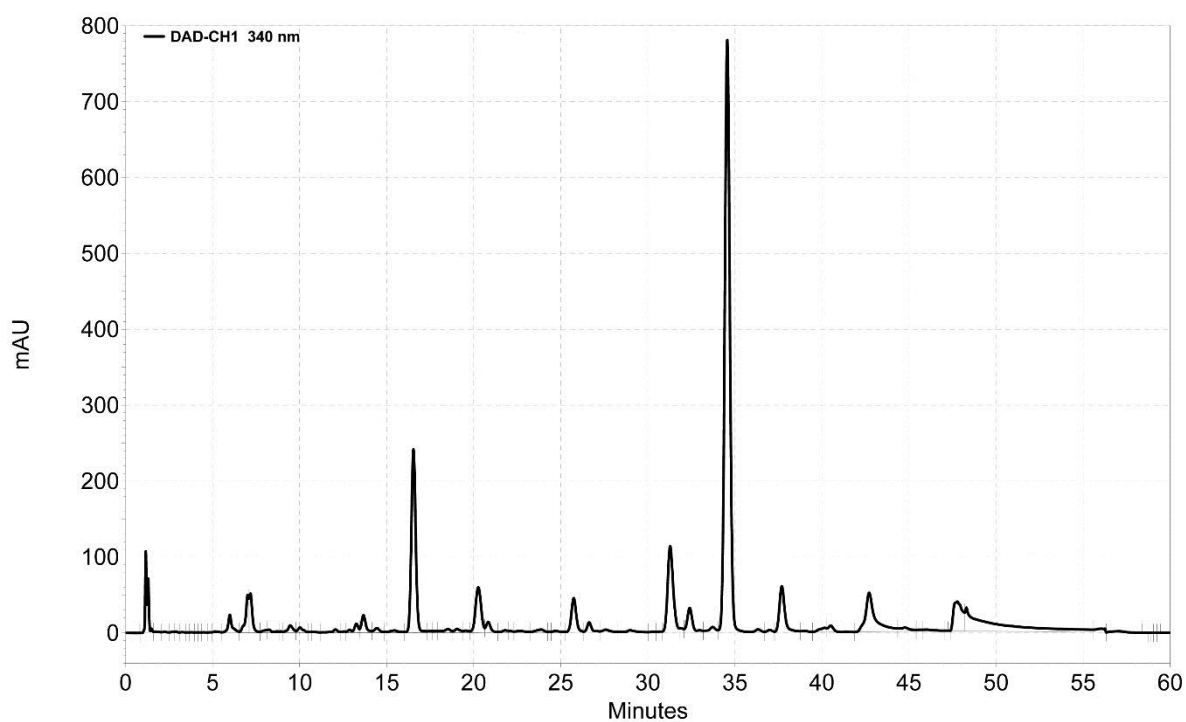


Figure S5. Co-injection of the mixture obtained from **10** with standard **S2**.

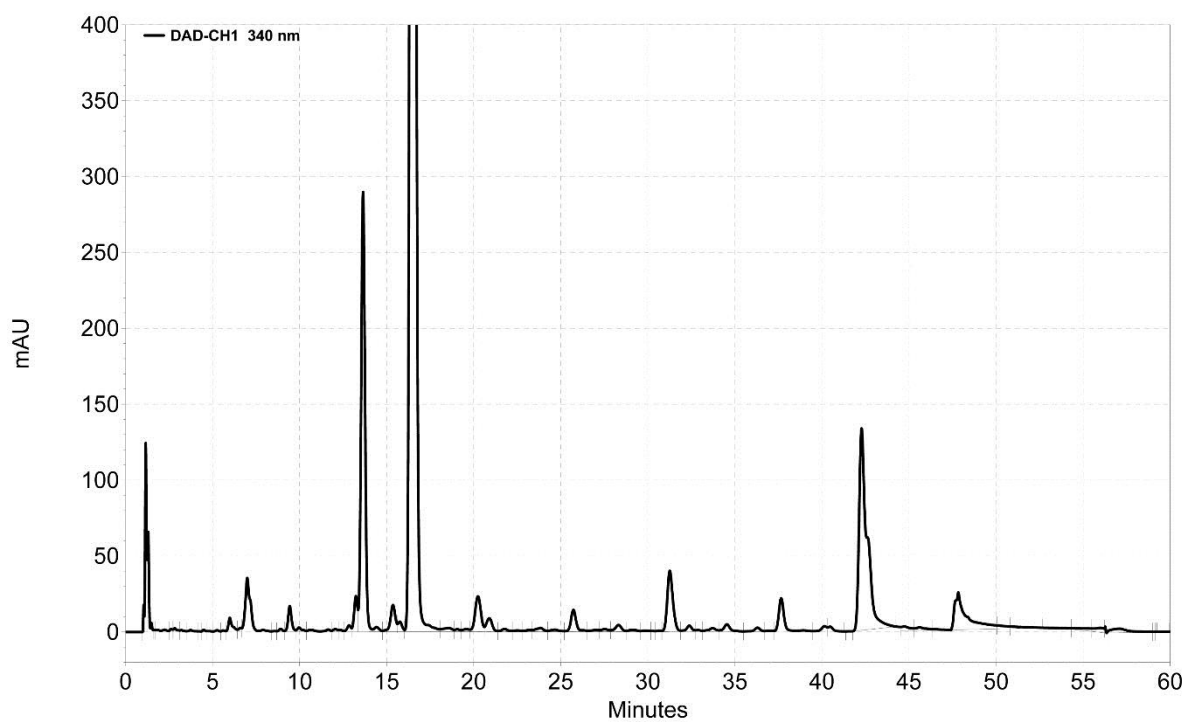


Figure S6. HPLC chromatogram of the mixture obtained from **11**.

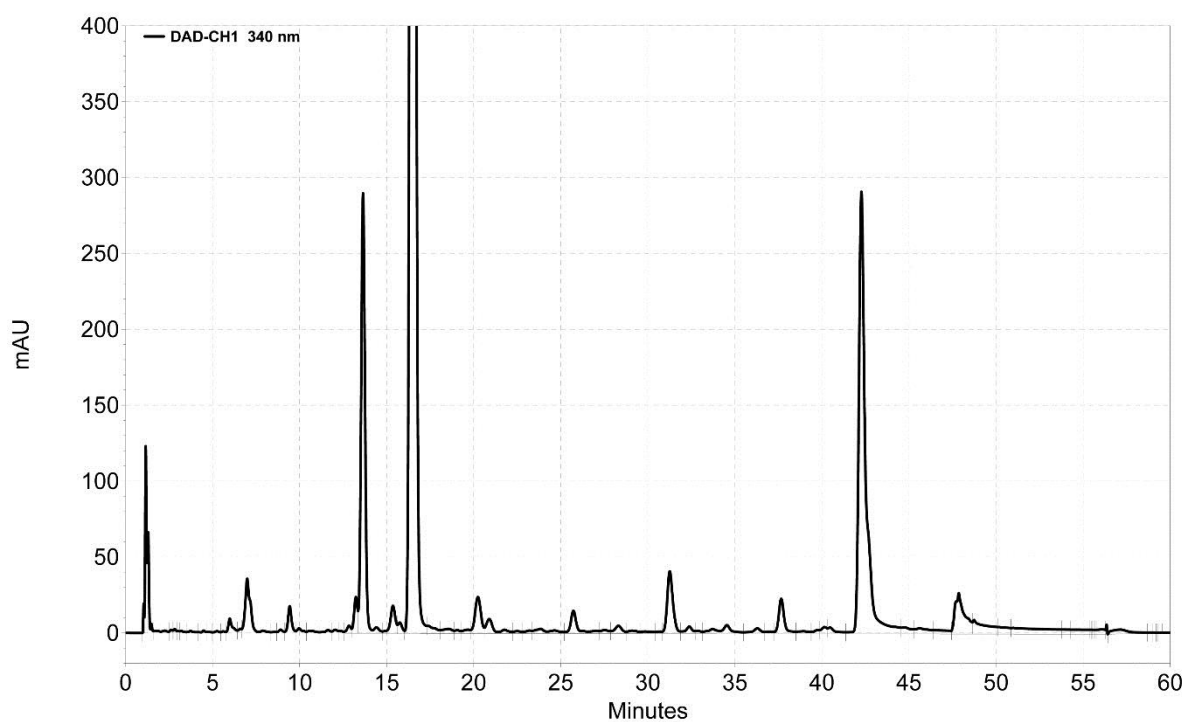


Figure S7. Co-injection of the mixture obtained from **11** with standard **S3**.

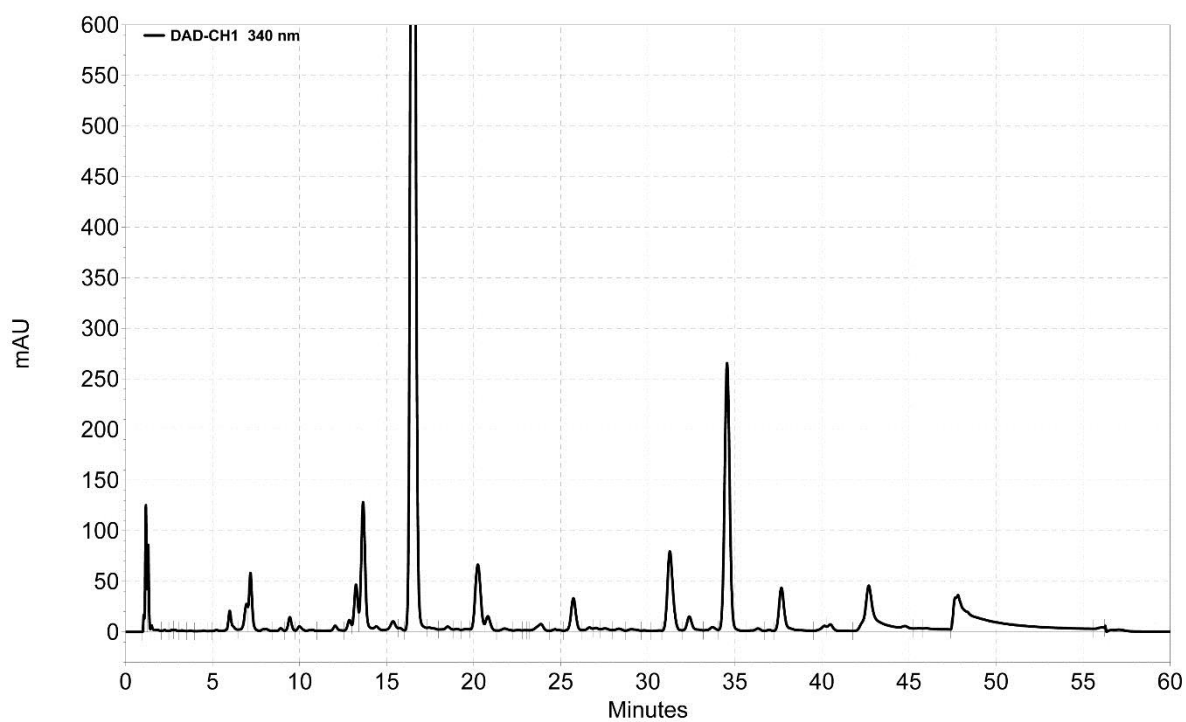


Figure S8. HPLC chromatogram of the mixture obtained from **12**.

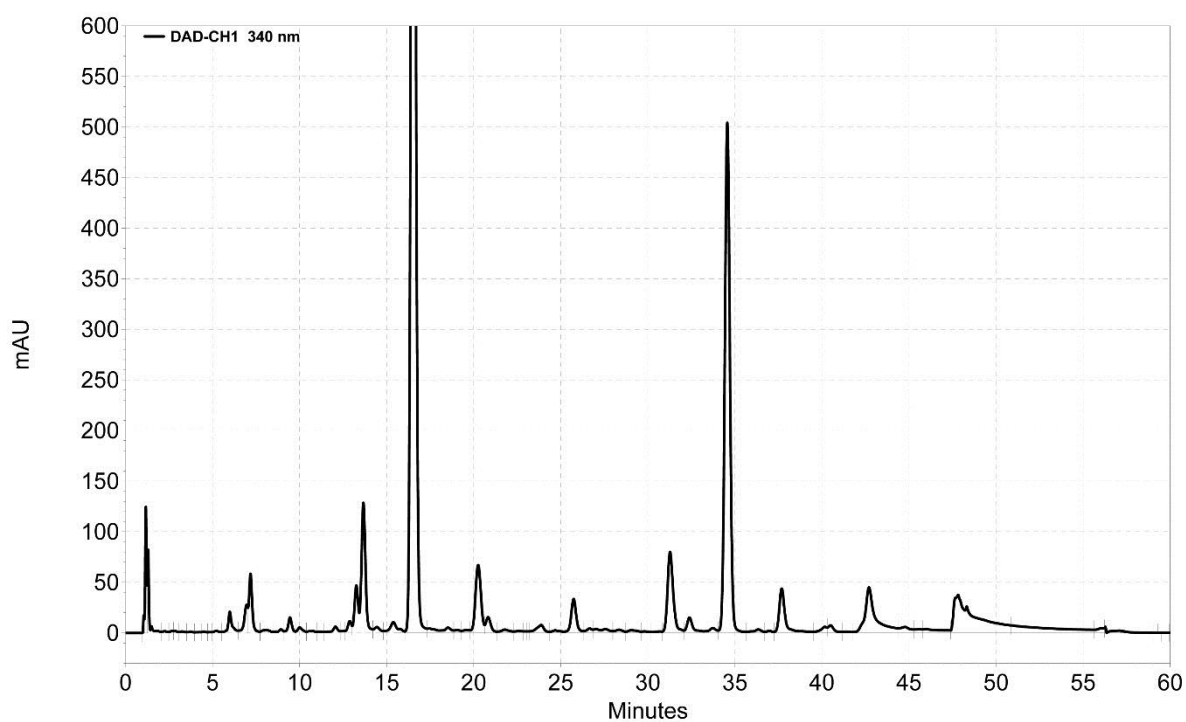


Figure S9. Co-injection of the mixture obtained from **12** with standard **S2**.

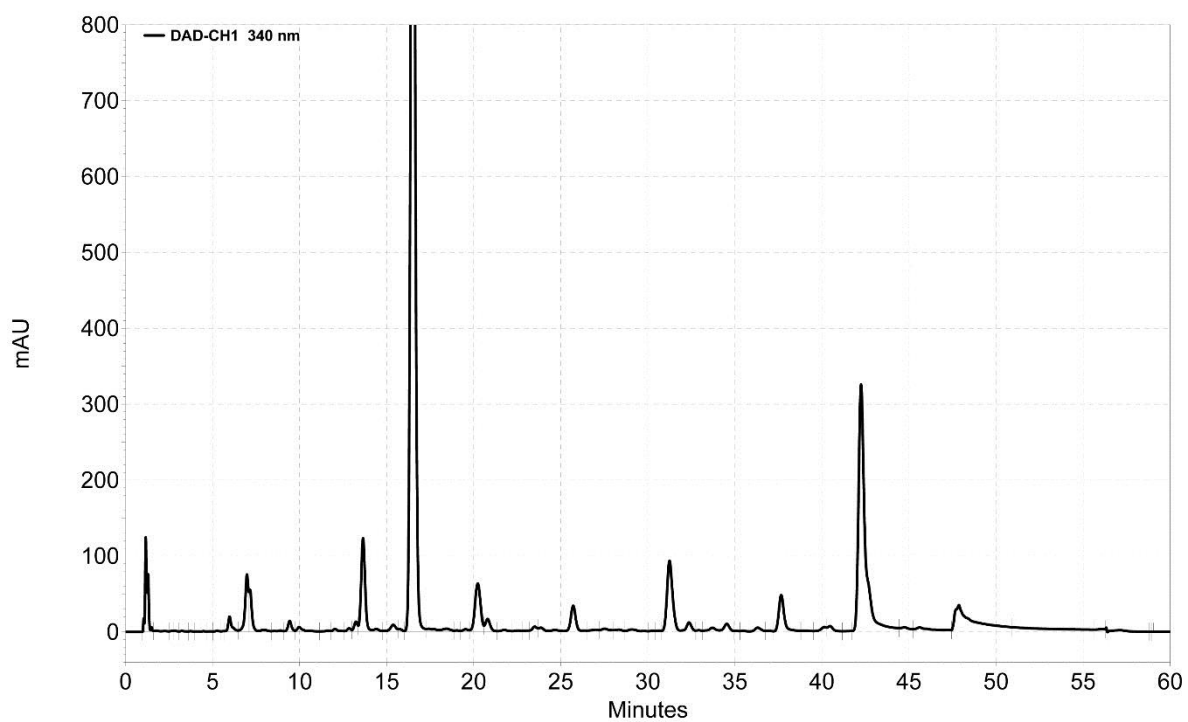


Figure S10. HPLC chromatogram of the mixture obtained from **13**.

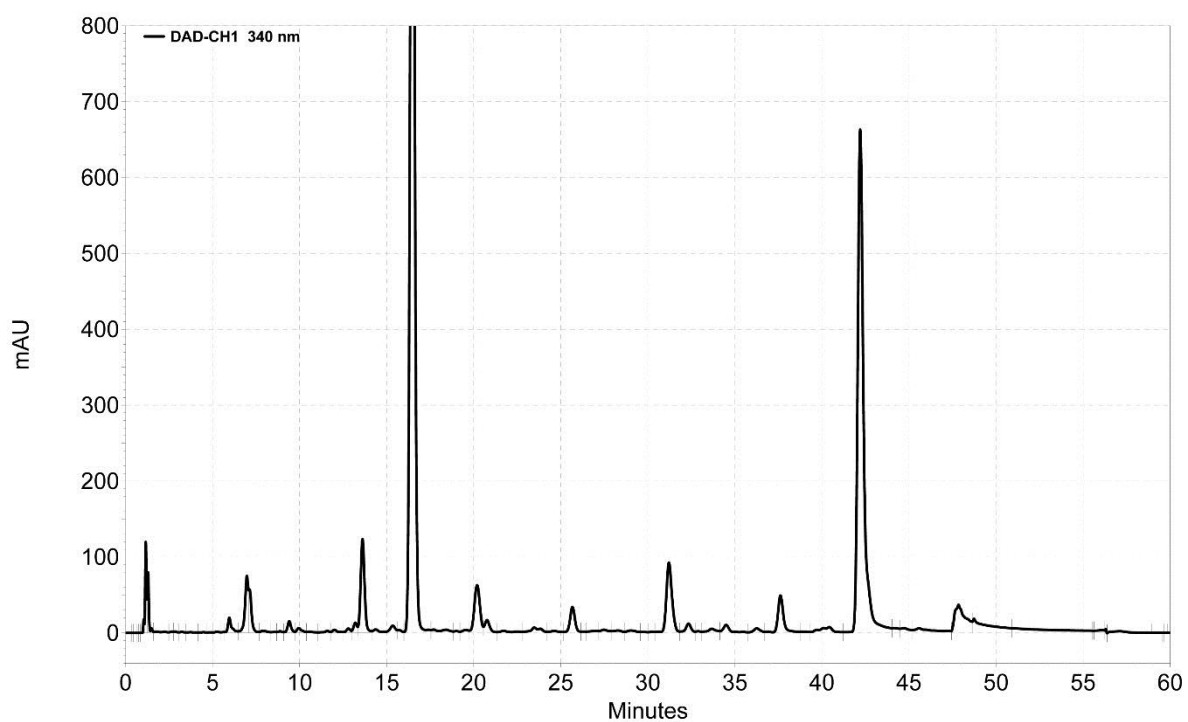


Figure S11. Co-injection of the mixture obtained from **13** with standard **S3**.

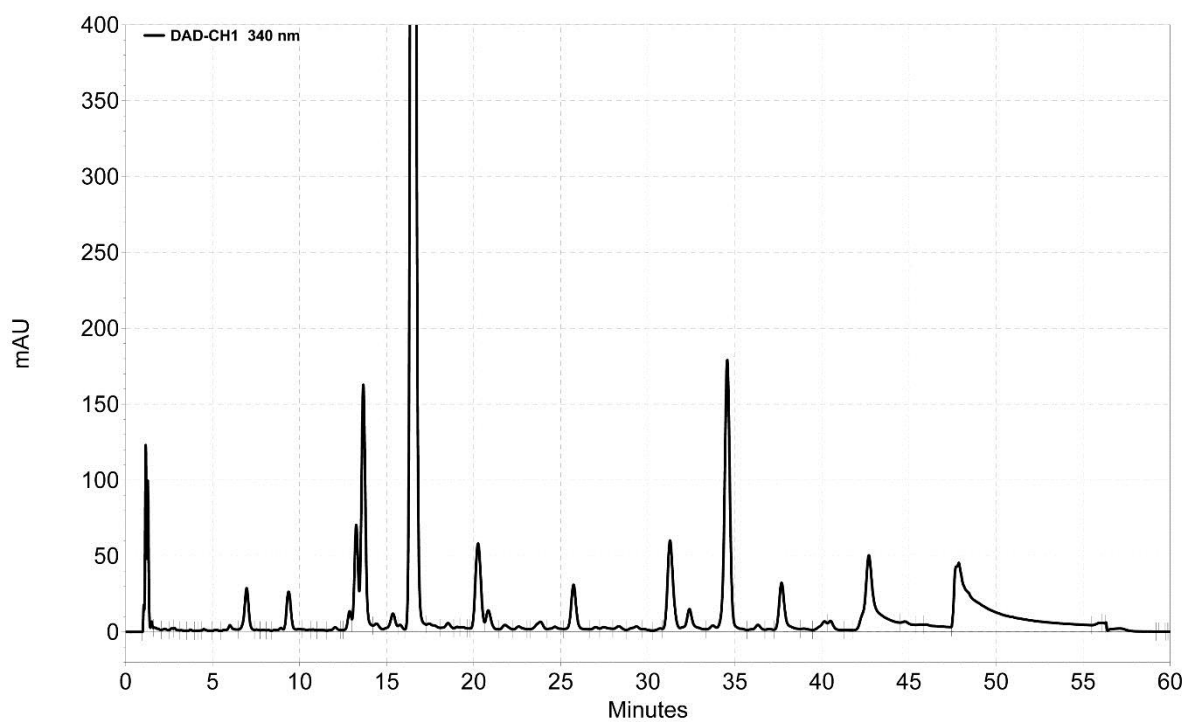


Figure S12. HPLC chromatogram of the mixture obtained from **14**.

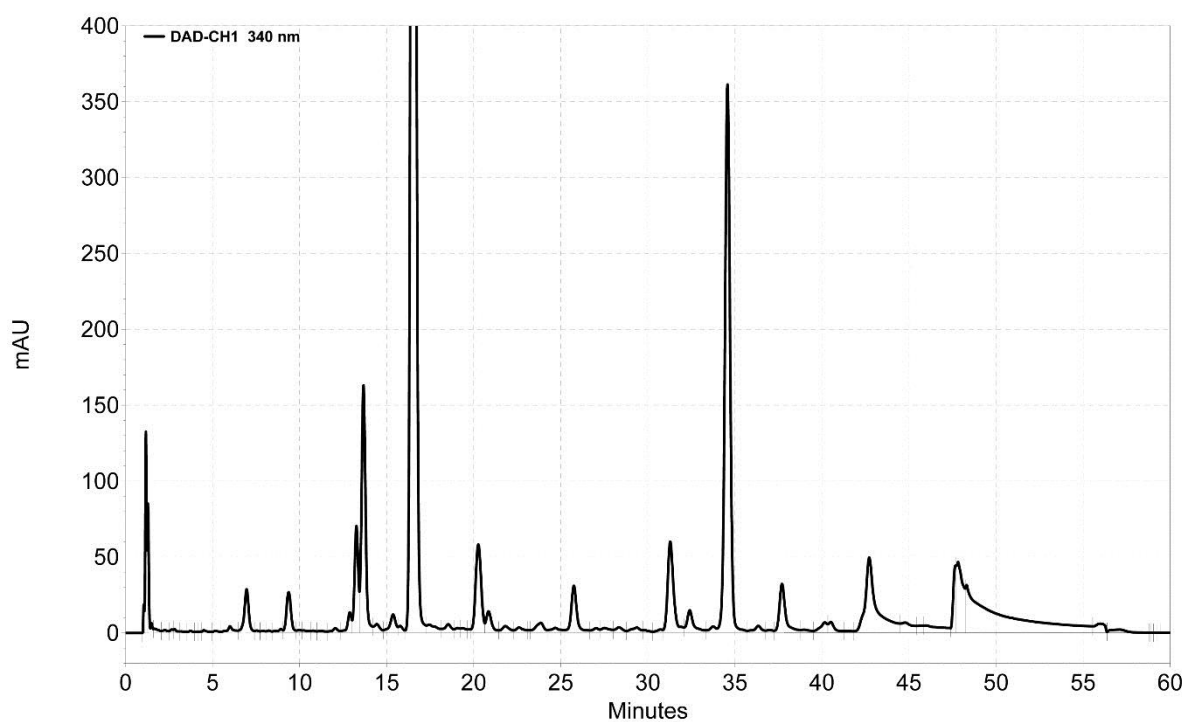


Figure S13. Co-injection of the mixture obtained from **14** with standard **S2**.

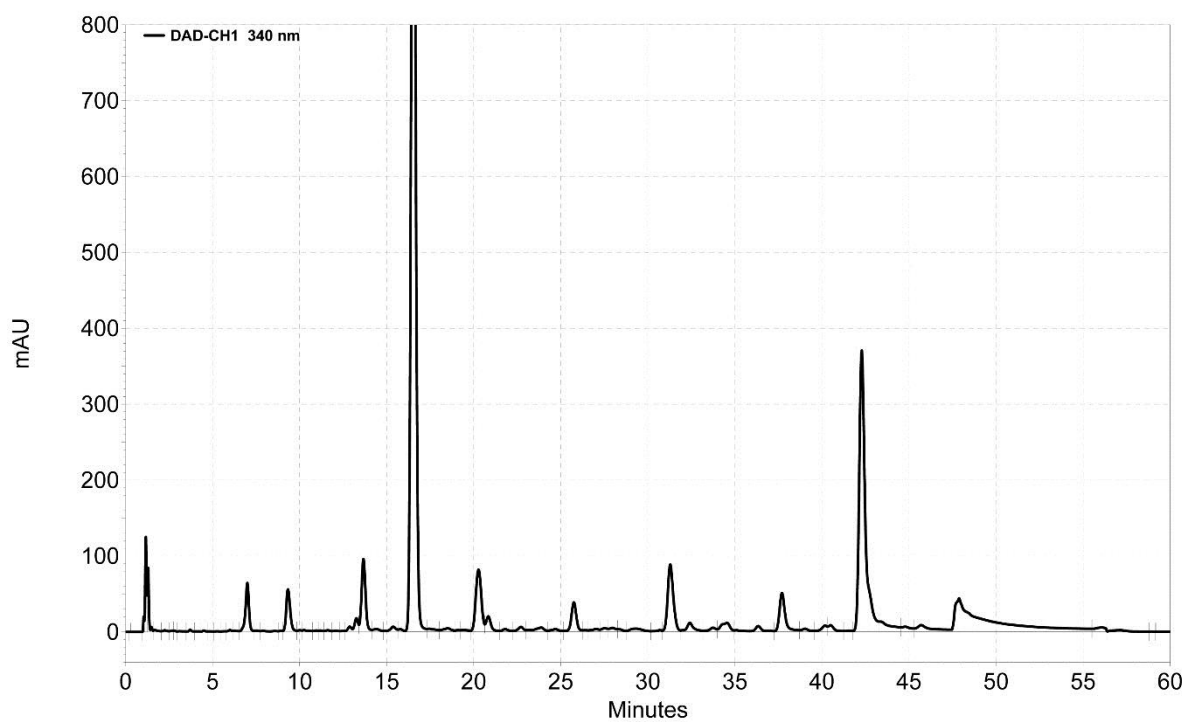


Figure S14. HPLC chromatogram of the mixture obtained from **15**.

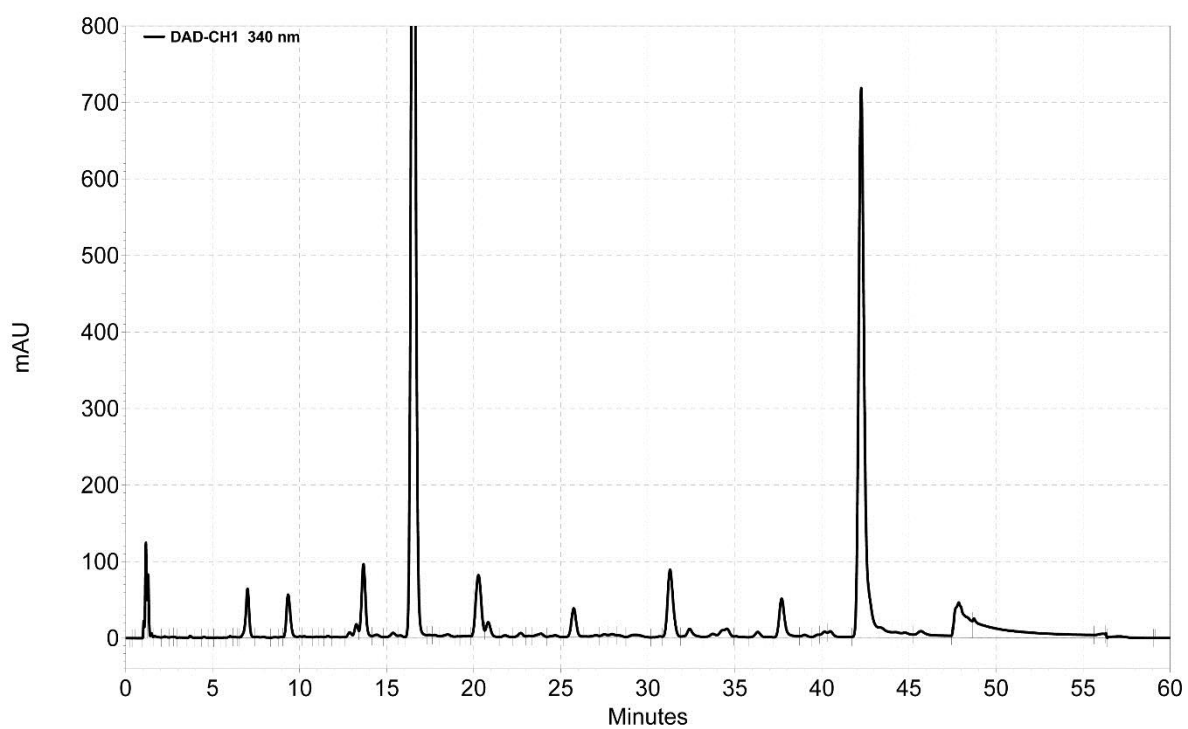


Figure S15. Co-injection of the mixture obtained from **15** with **S3**.

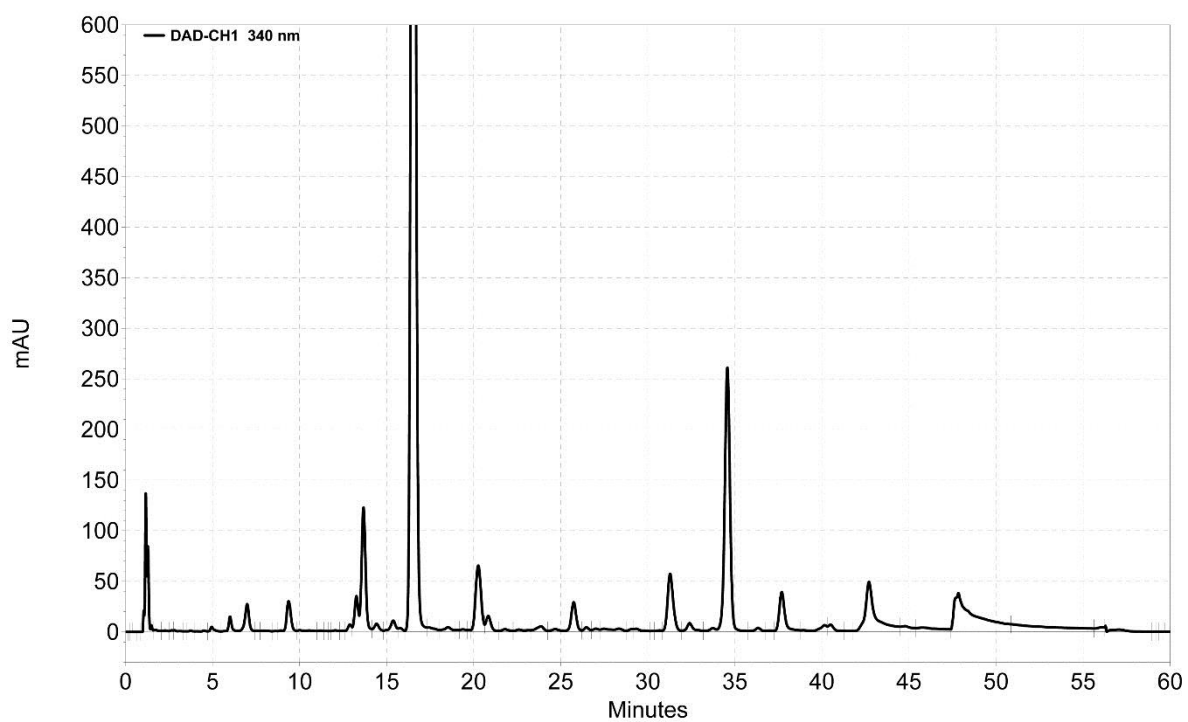


Figure S16. HPLC chromatogram of the mixture obtained from **16**.

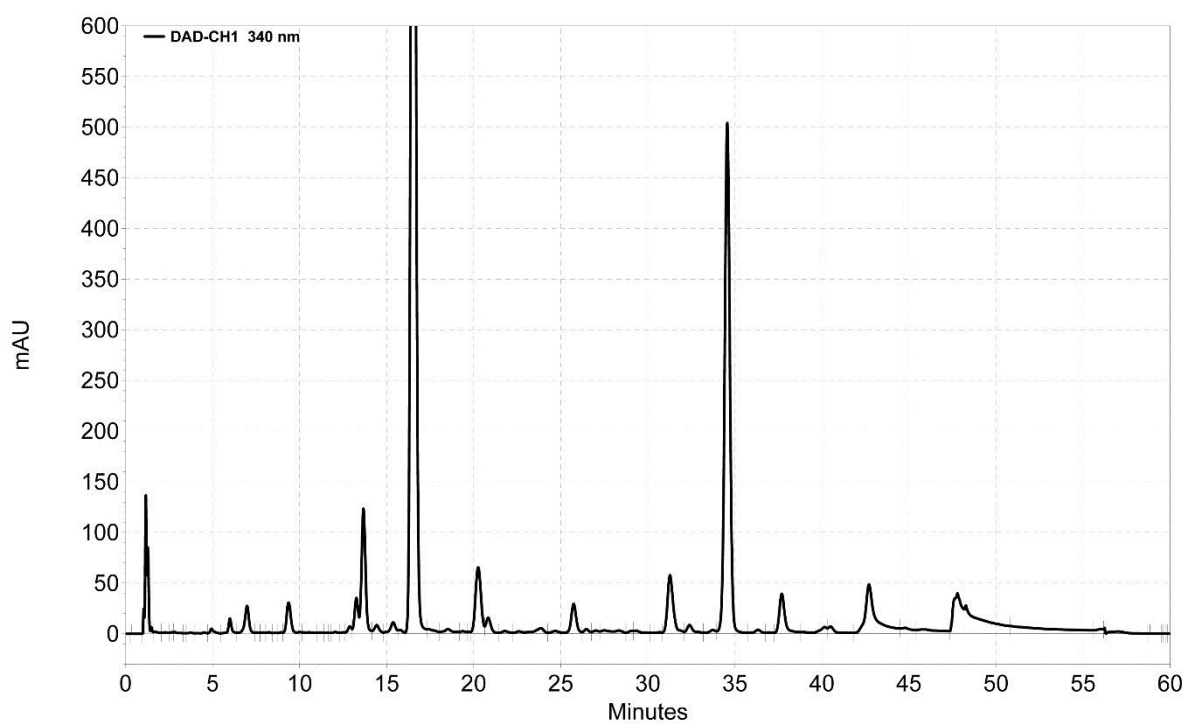


Figure S17. Co-injection of the mixture obtained from **16** with standard **S2**.

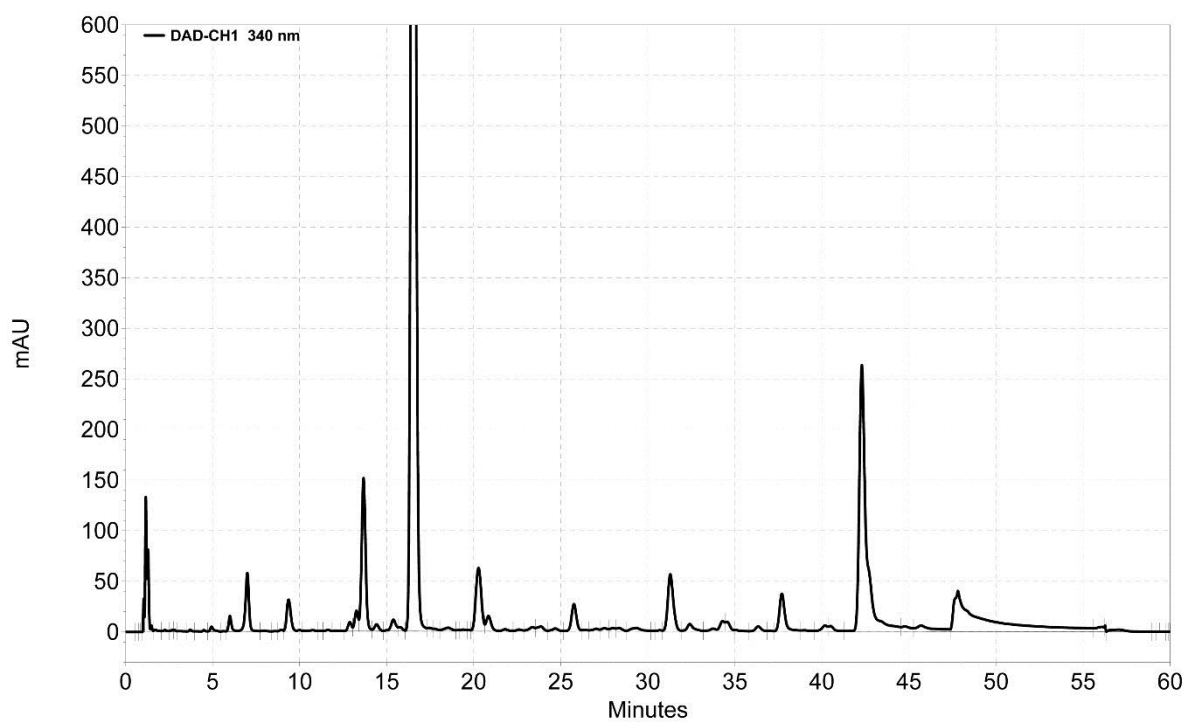


Figure S18. HPLC chromatogram of the mixture obtained from **17**.

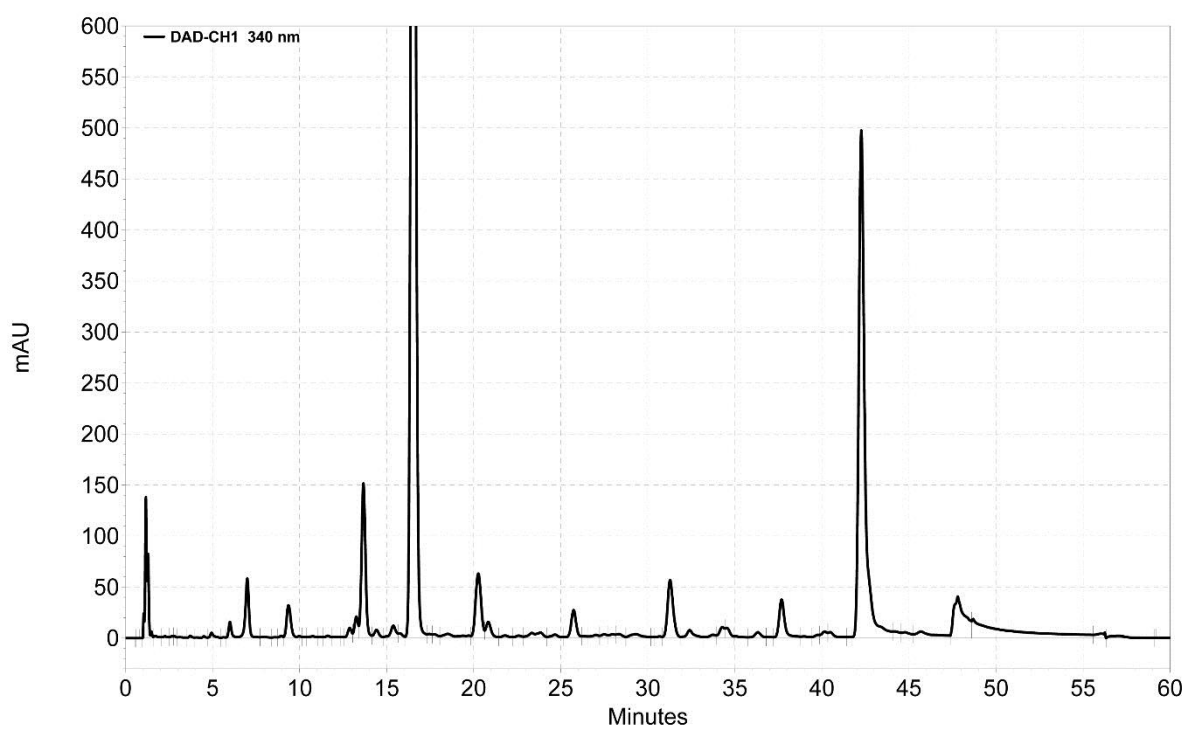


Figure S19. Co-injection of the mixture obtained from **17** with standard **S3**.

^1H , ^{13}C and ^{19}F NMR spectra of synthesized compounds

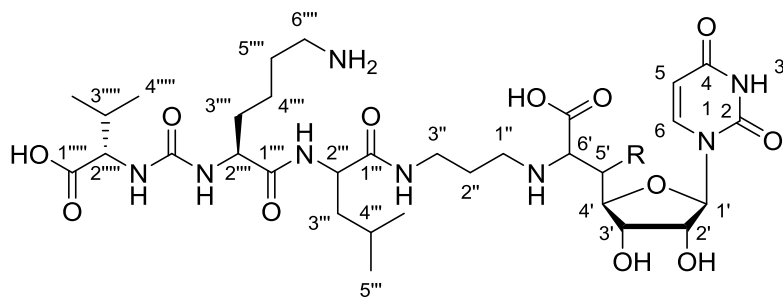


Figure S20. Numbering of atoms of muraymycin target structures for the assignment of NMR signals.

DCAS0606b.dbo
Spork / bucho

Feb 25 2012

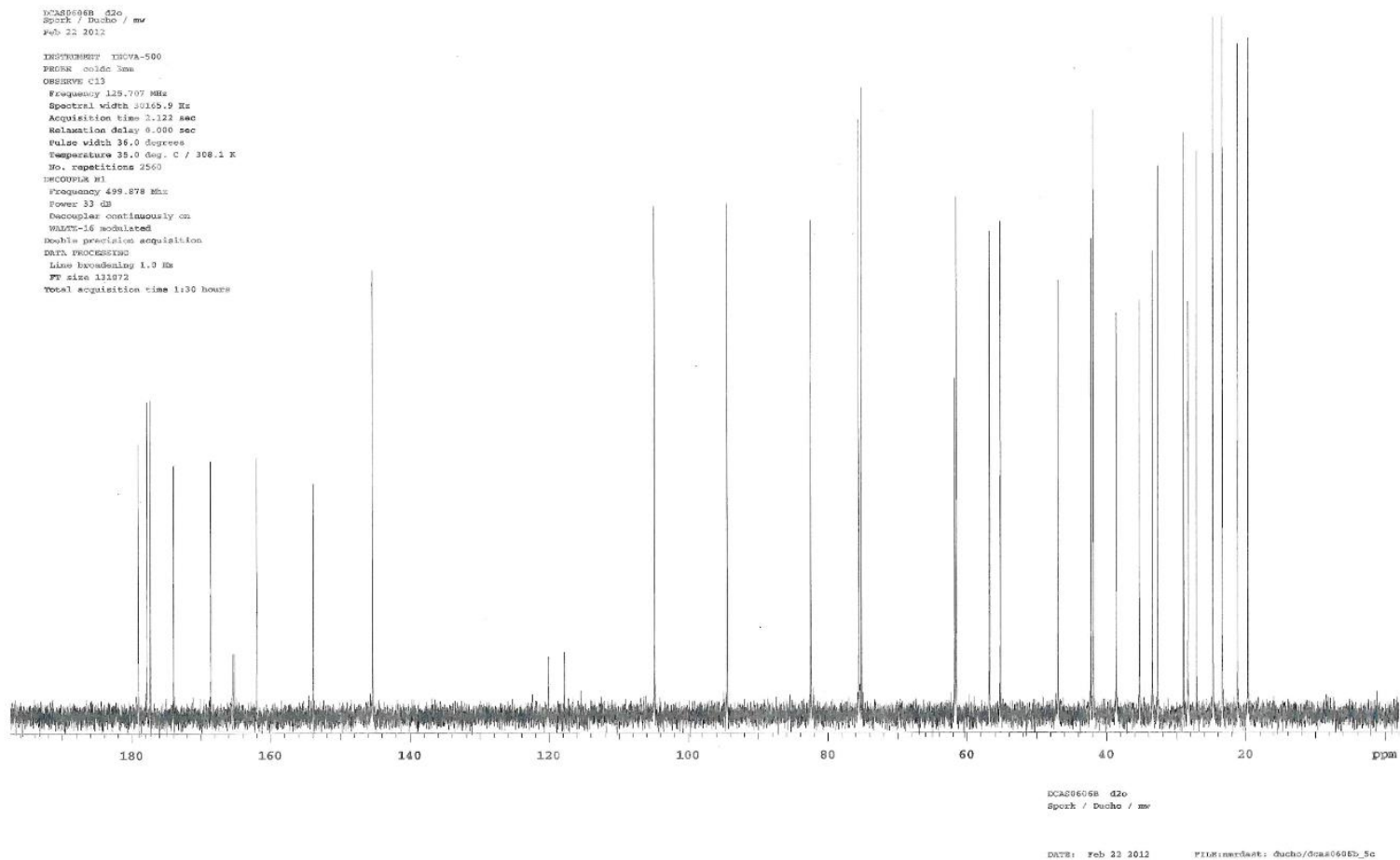
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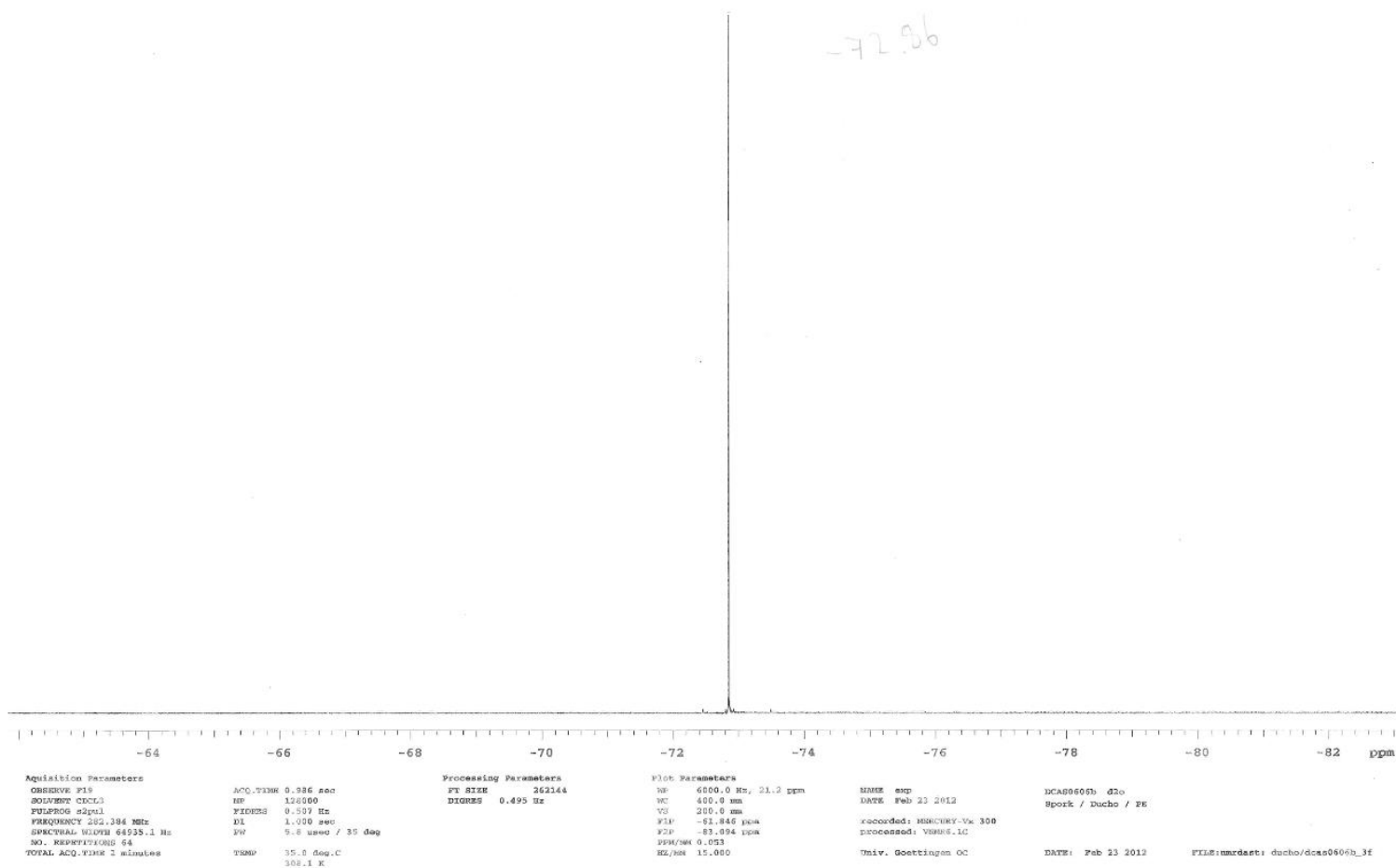
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5.87 (4')
4.56 (6') d 5.40 d 8.70 (2'')
4.38 (3') d 6.70 d 6.48 (3'')
4.24 (2') d 6.70 d 6.48 (3'')
3.44 (4'') d 7.42 d 7.42 (6'')
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2.28 (3'') d 6.90 d 6.90 (3'')
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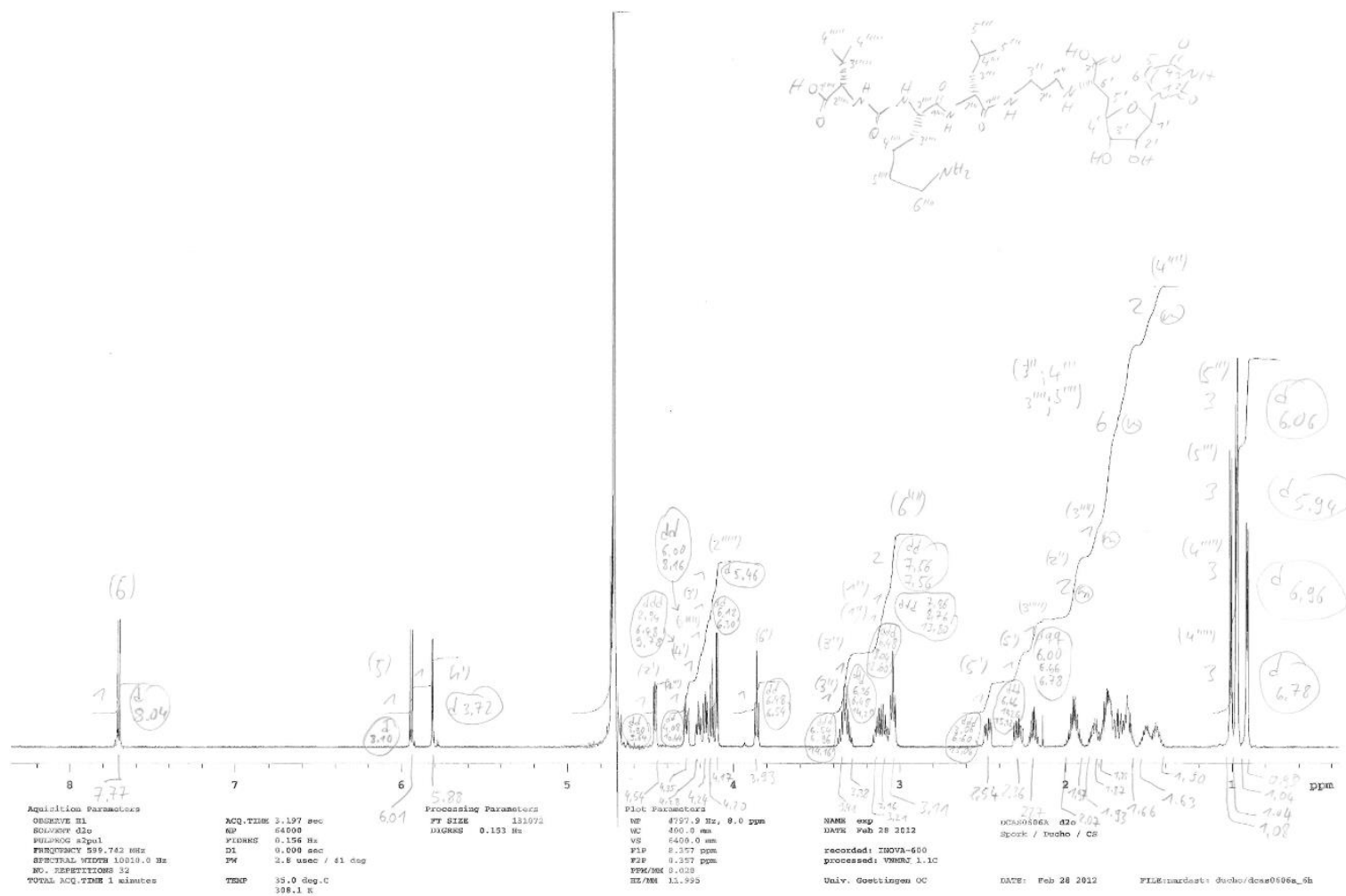
DATE: Feb 25 2012
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^{13}C NMR spectrum of **10** (126 MHz, D_2O , 35 °C)



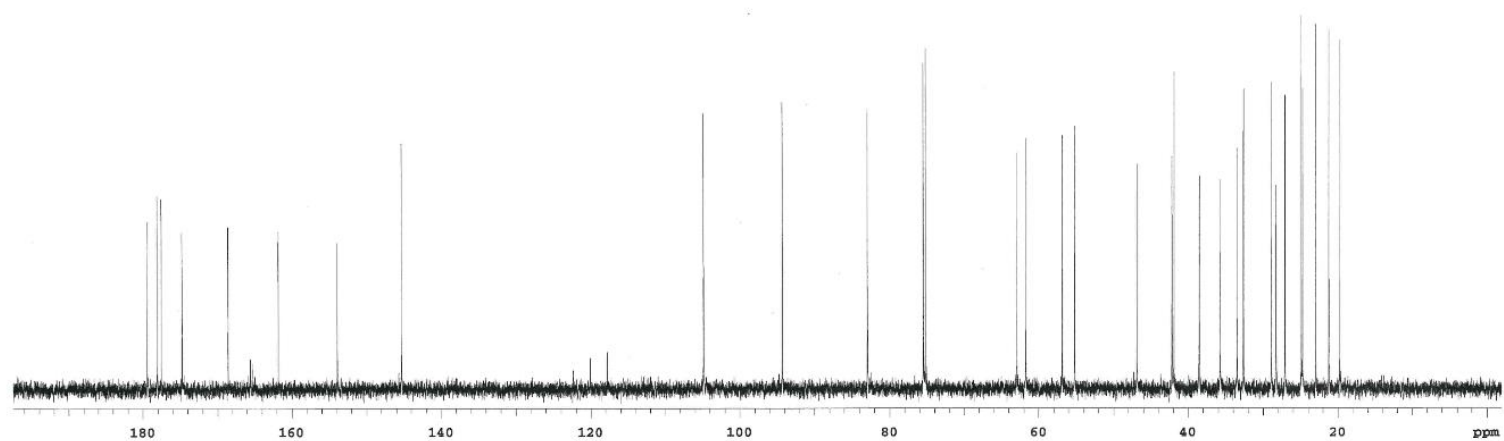
^{19}F NMR spectrum of **10** (282 MHz, D_2O , 35 °C)



¹H NMR spectrum of **11** (600 MHz, D₂O, 35 °C)

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 Spark / Ducho
 Feb 25 2012

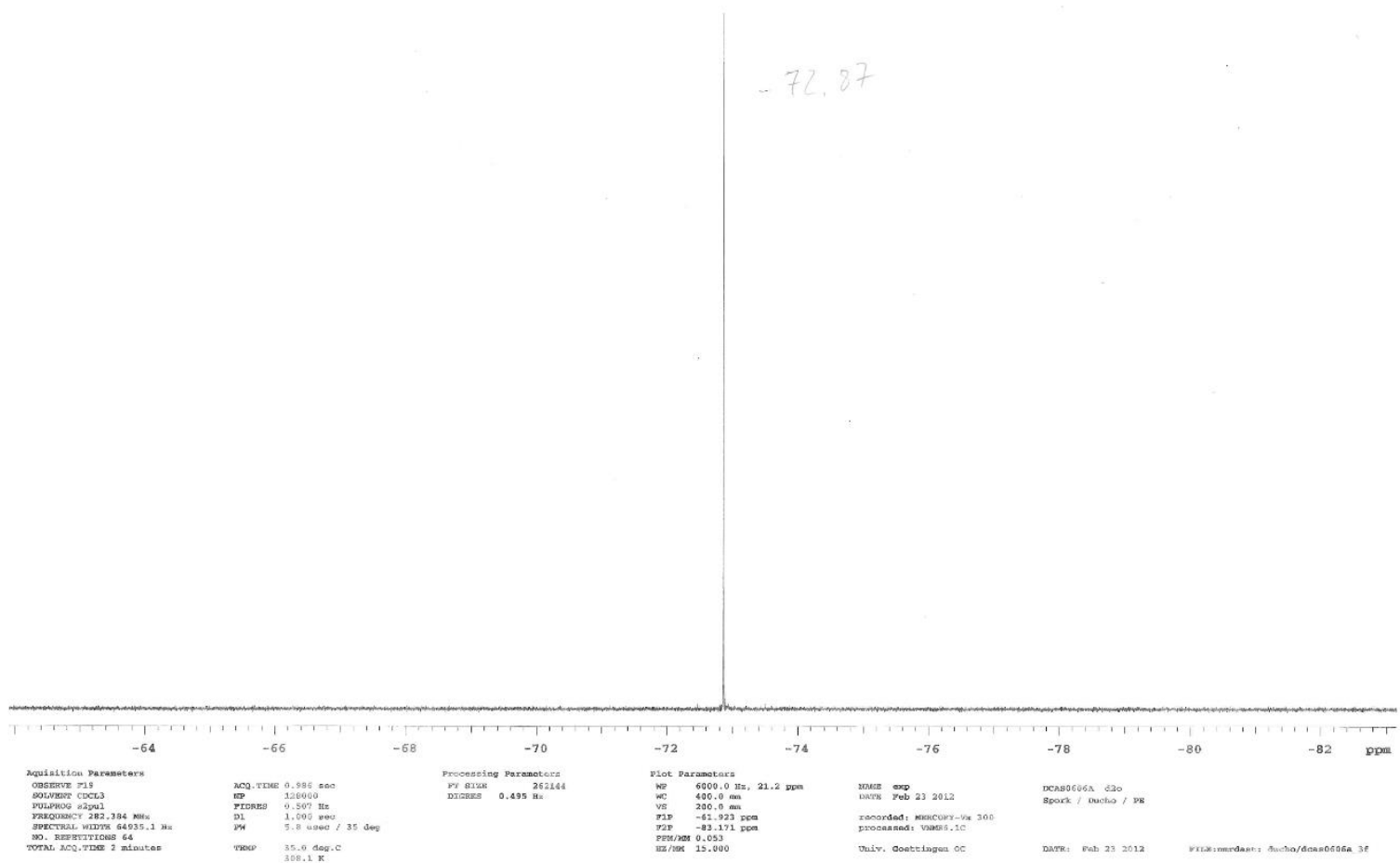
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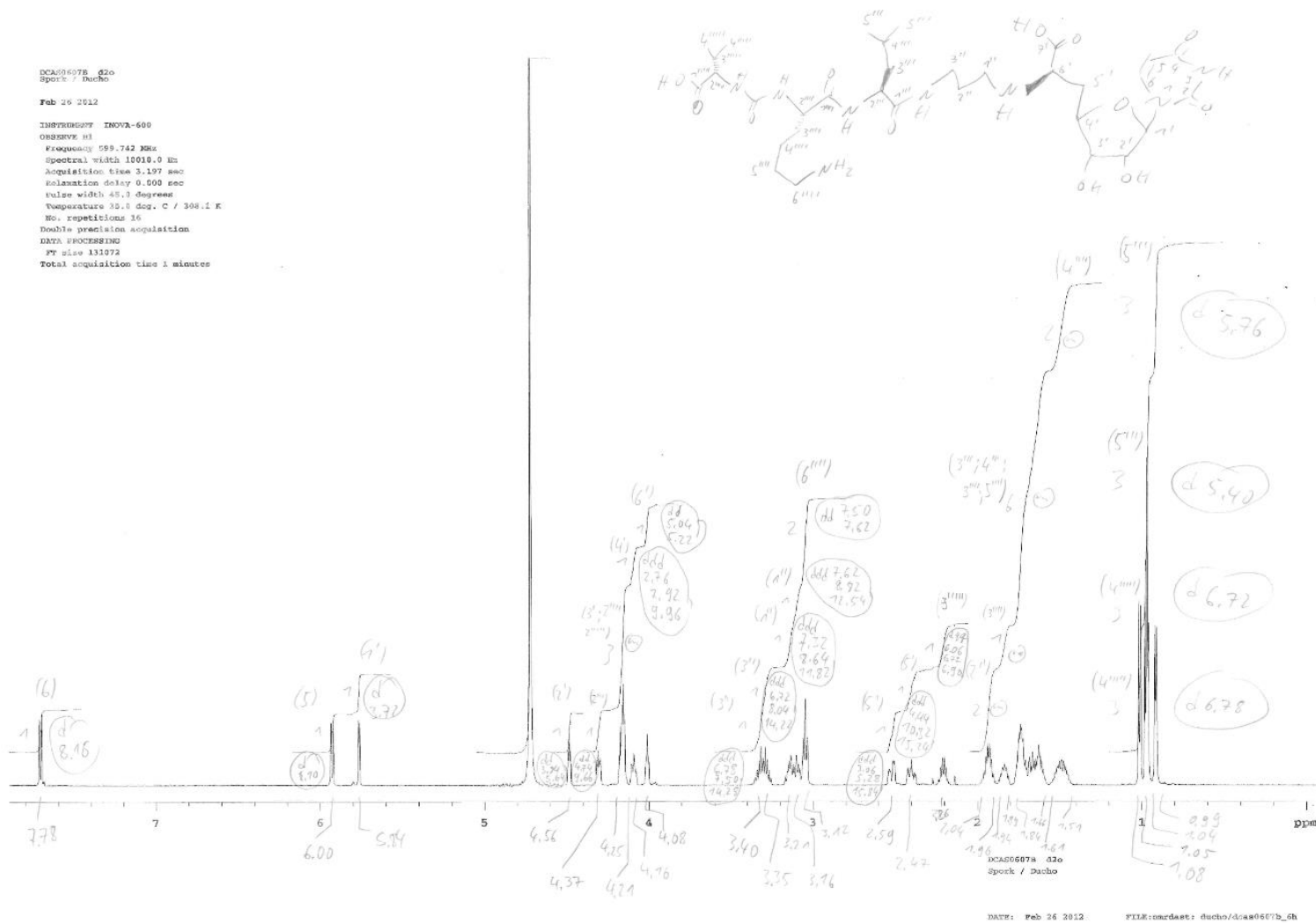
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 Spark / Ducho

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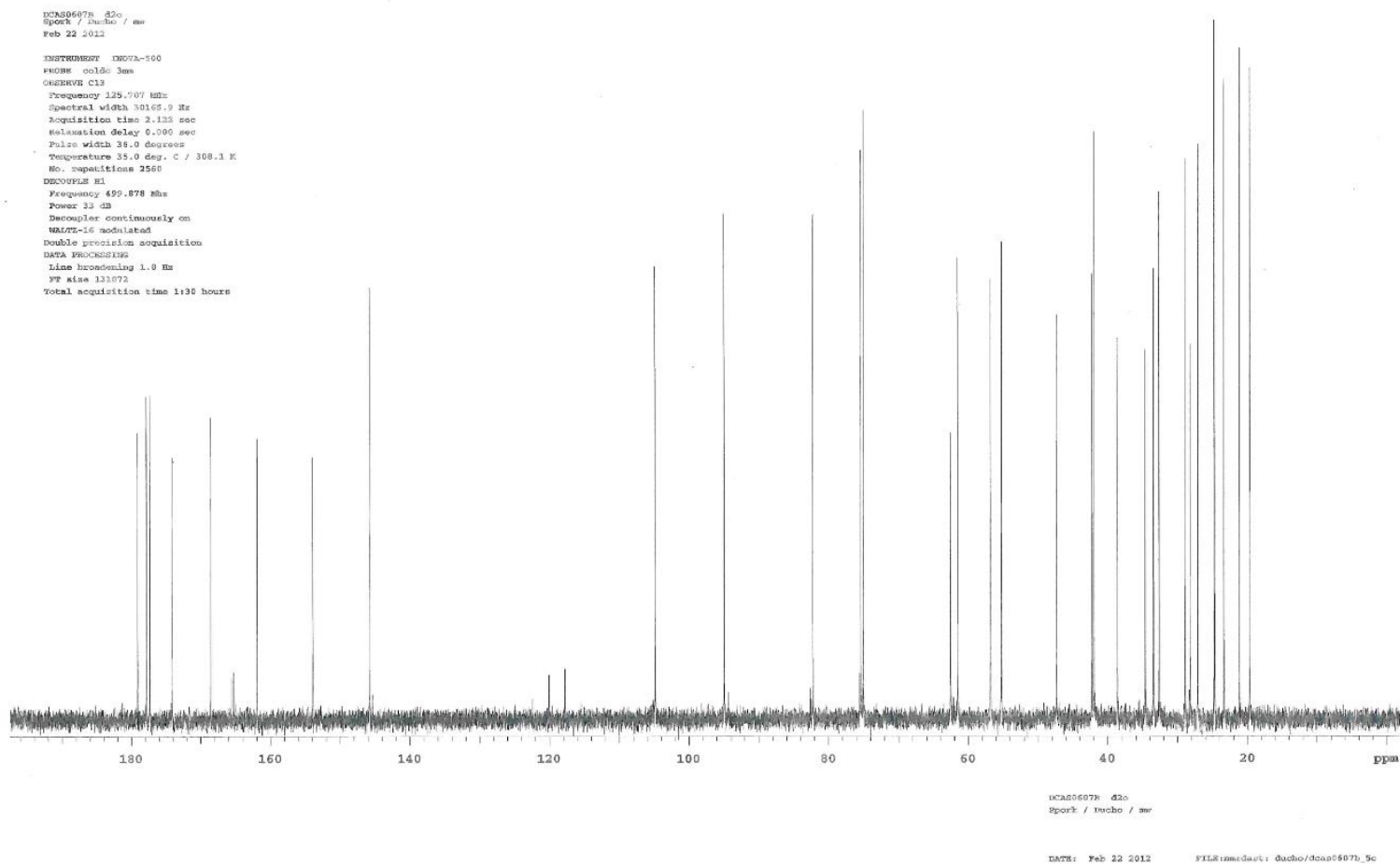
^{13}C NMR spectrum of **11** (126 MHz, D_2O , 35 °C)



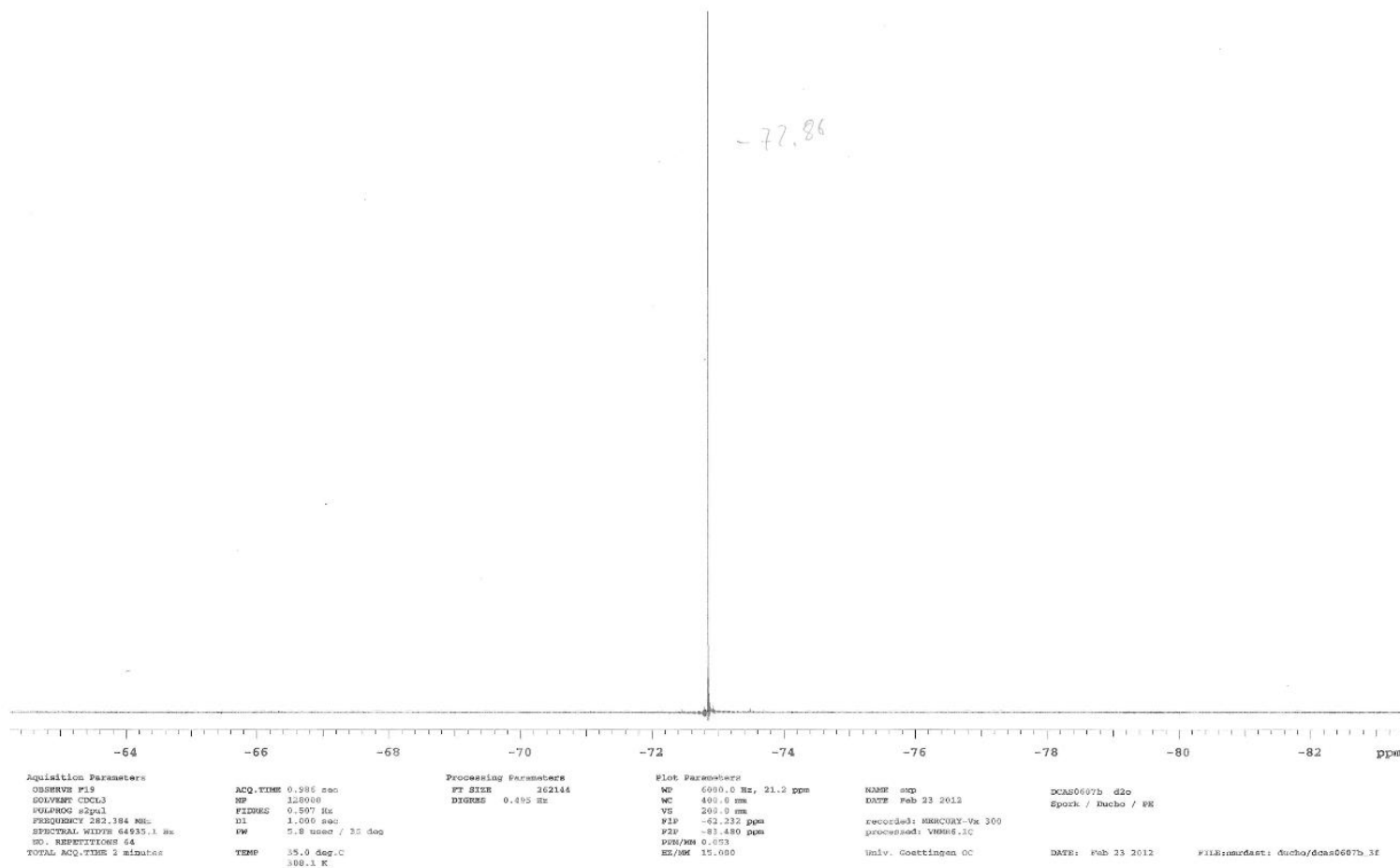
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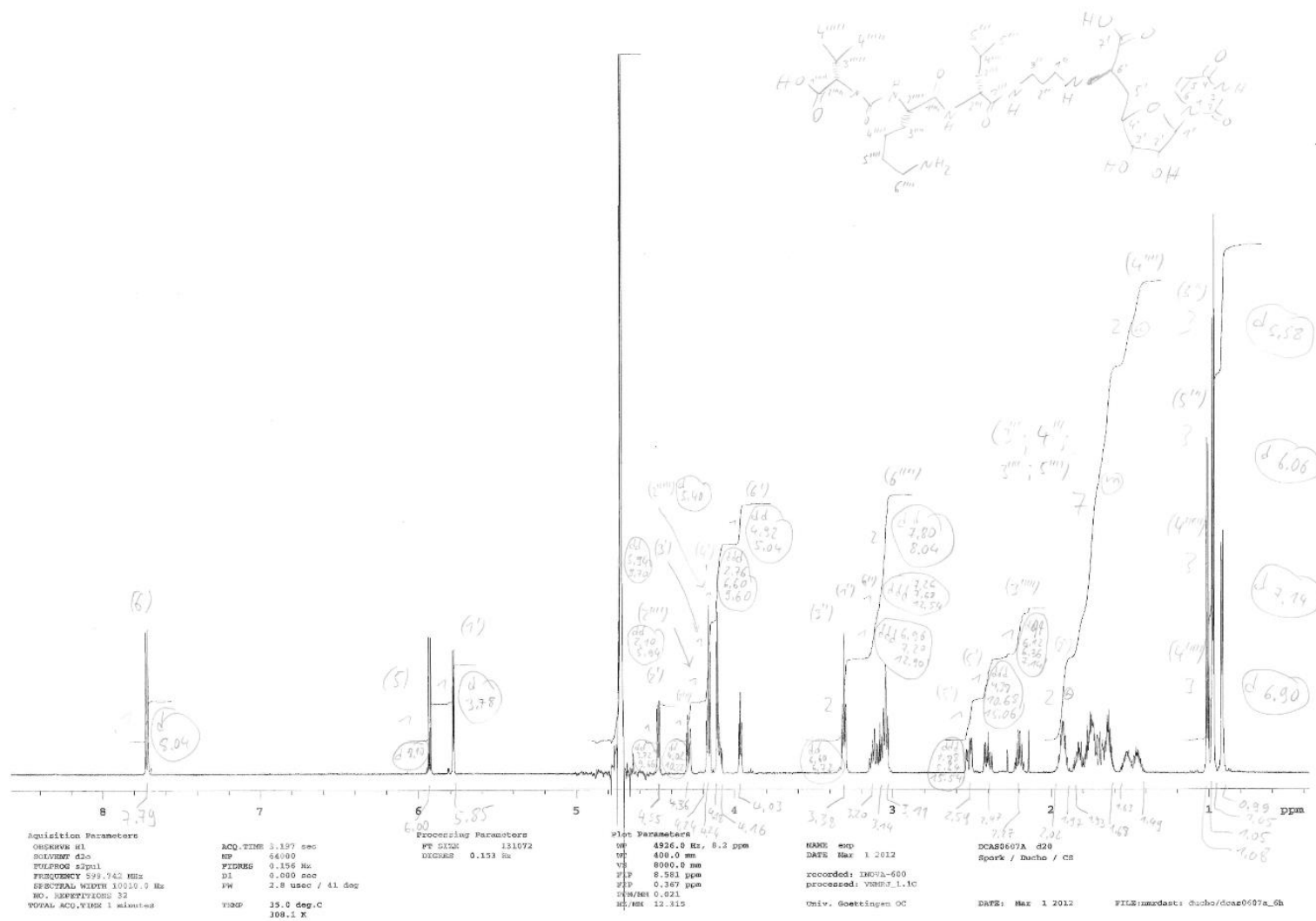
^1H NMR spectrum of **12** (600 MHz, D_2O , 35 °C)



^{13}C NMR spectrum of **12** (126 MHz, D_2O , 35 $^\circ\text{C}$)



^{19}F NMR spectrum of **12** (282 MHz, D_2O , 35 °C)

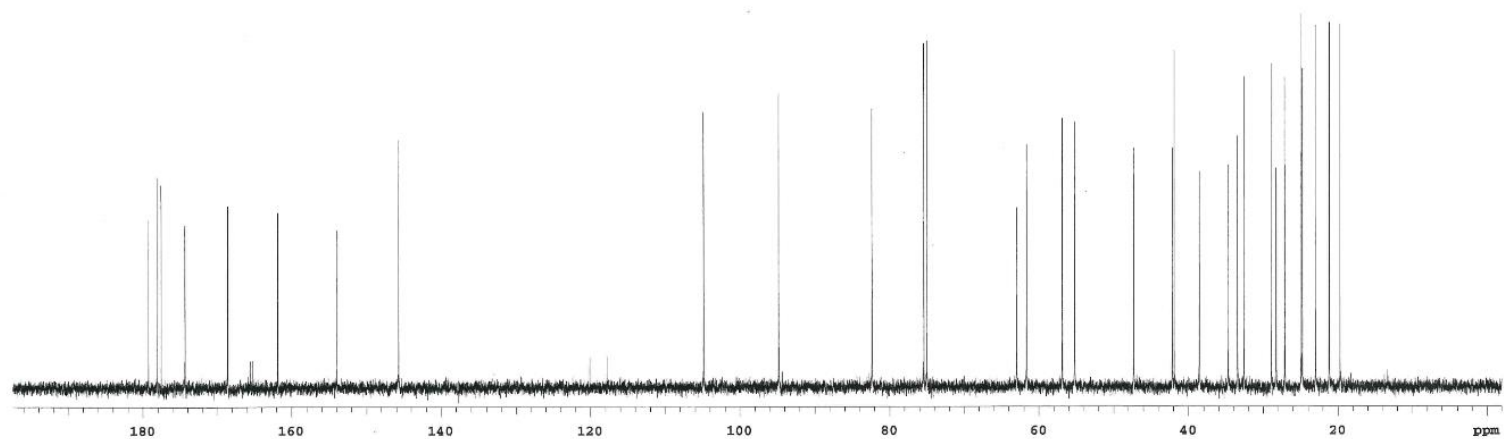


DCMS0607A 62o
 Spork / Ducho
 Feb 24 2012

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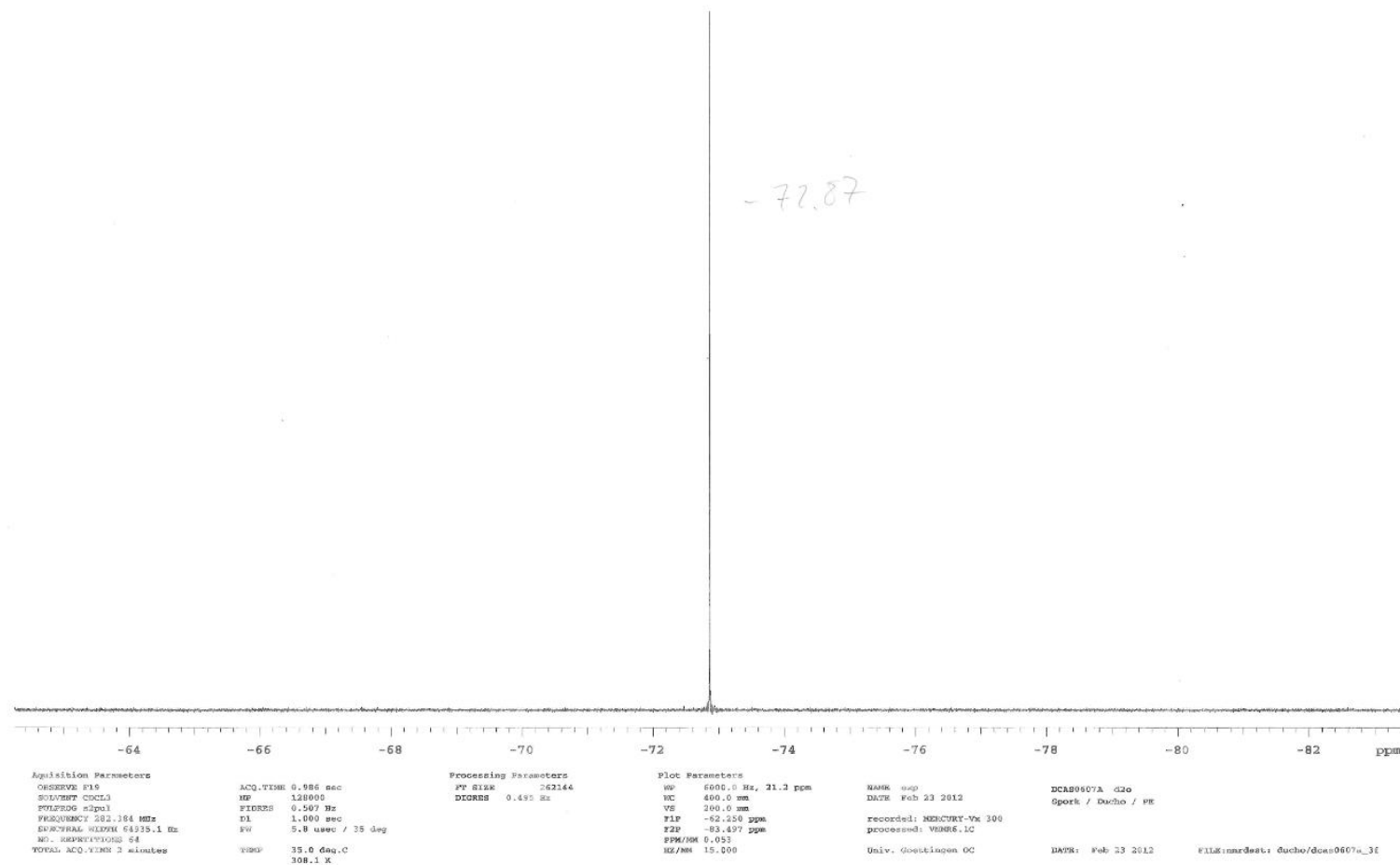
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DCMS0607A 62o
 Spork / Ducho

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^{13}C NMR spectrum of **13** (126 MHz, D_2O , 35 °C)



^{19}F NMR spectrum of **13** (282 MHz, D_2O , 35 °C)

DJL8608B_02o
Spec# / Ducho / wv

Mar 3 2013

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(G)

(H)

(I)

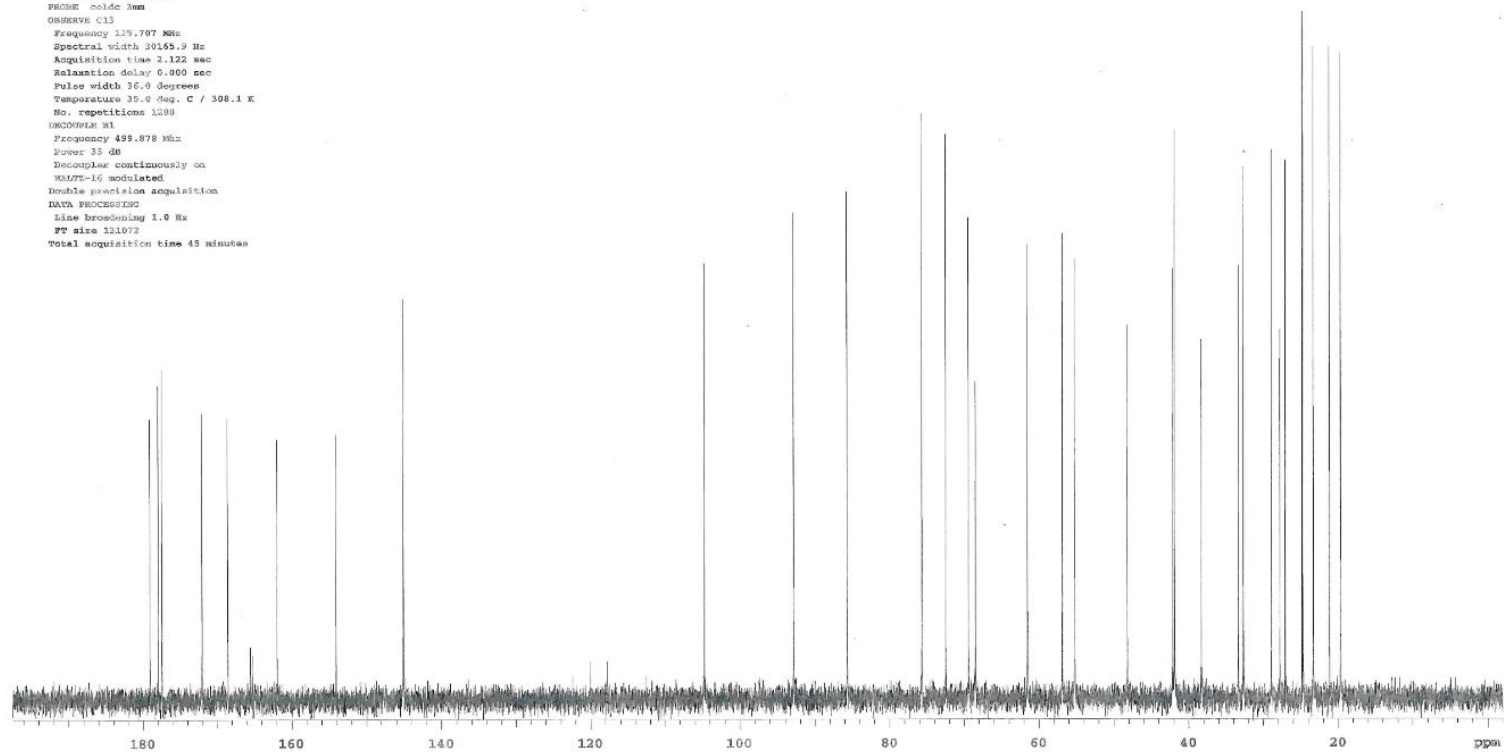
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PAGE: Mar 3 2013 FILE:\nardest\ducho\doss609db_gh

S27

DCAS605B d2o
 Spork / Ducho / mw
 Feb 22 2012

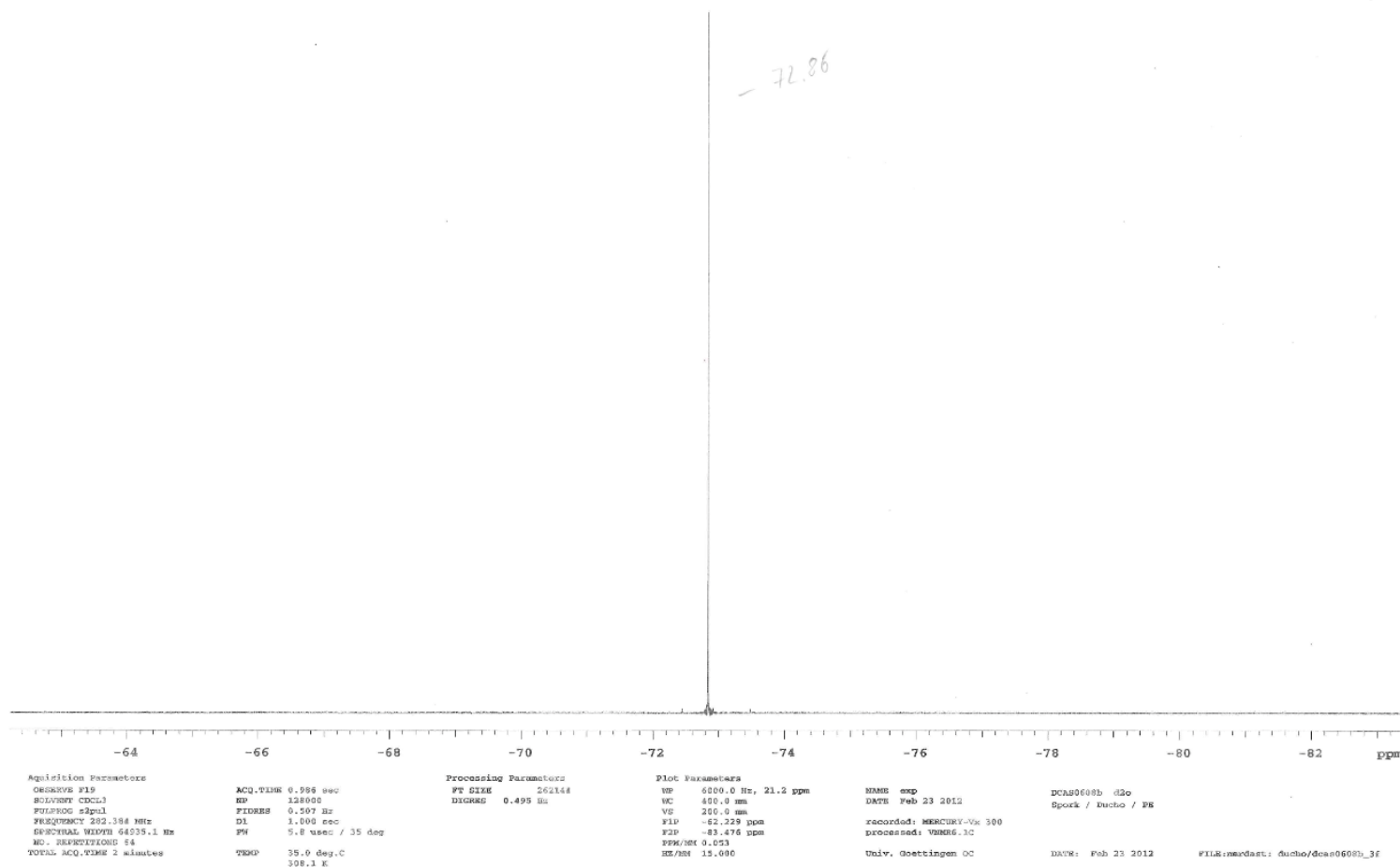
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DCAS605B d2o
 Spork / Ducho / mw

DATE: Feb 23 2013 FILE:merdest: ducho/dcas605Bb_0c

^{13}C NMR spectrum of **14** (126 MHz, D_2O , 35 °C)



^{19}F NMR spectrum of **14** (282 MHz, D_2O , 35 °C)

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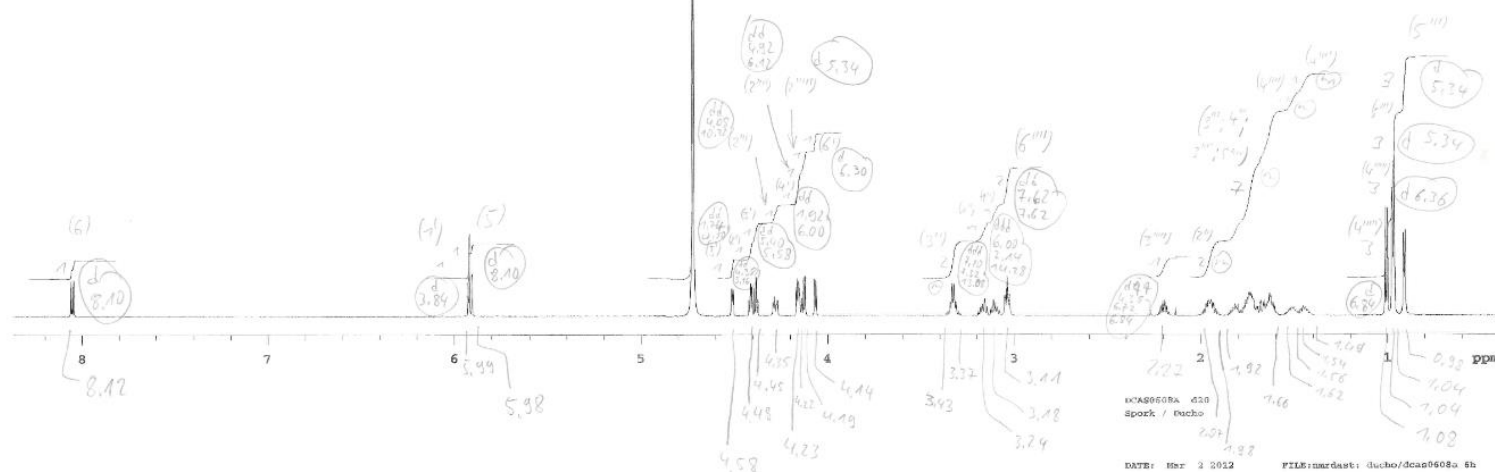
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Spock6 / Buchto

Mar. 2 2012

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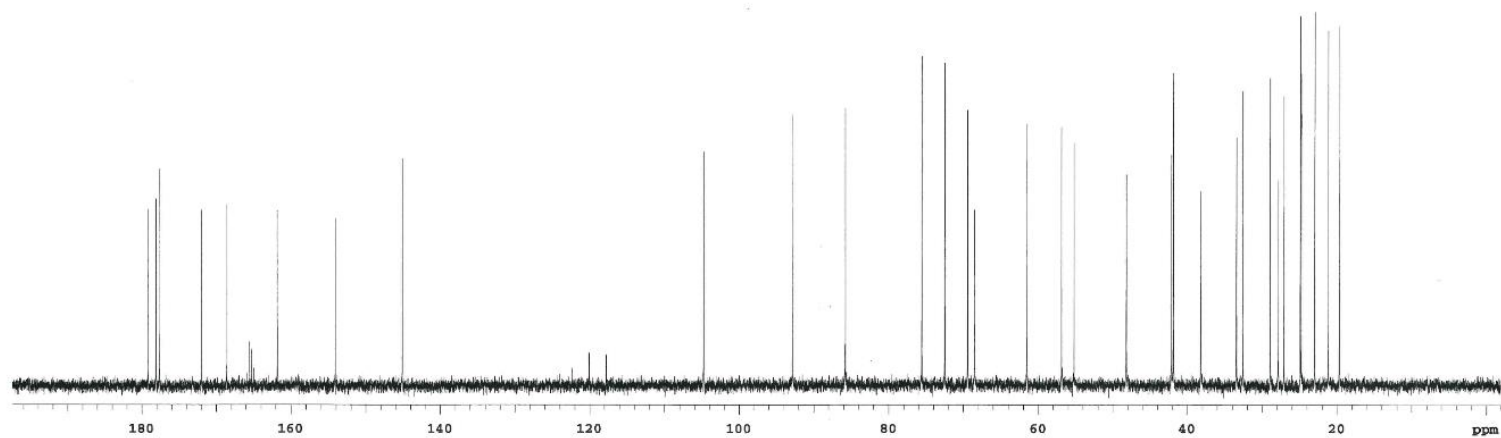
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¹H NMR spectrum of **15** (600 MHz, D₂O, 35 °C)

DCA00608A.d2o
 Spork / Ducho
 Feb 22 2012

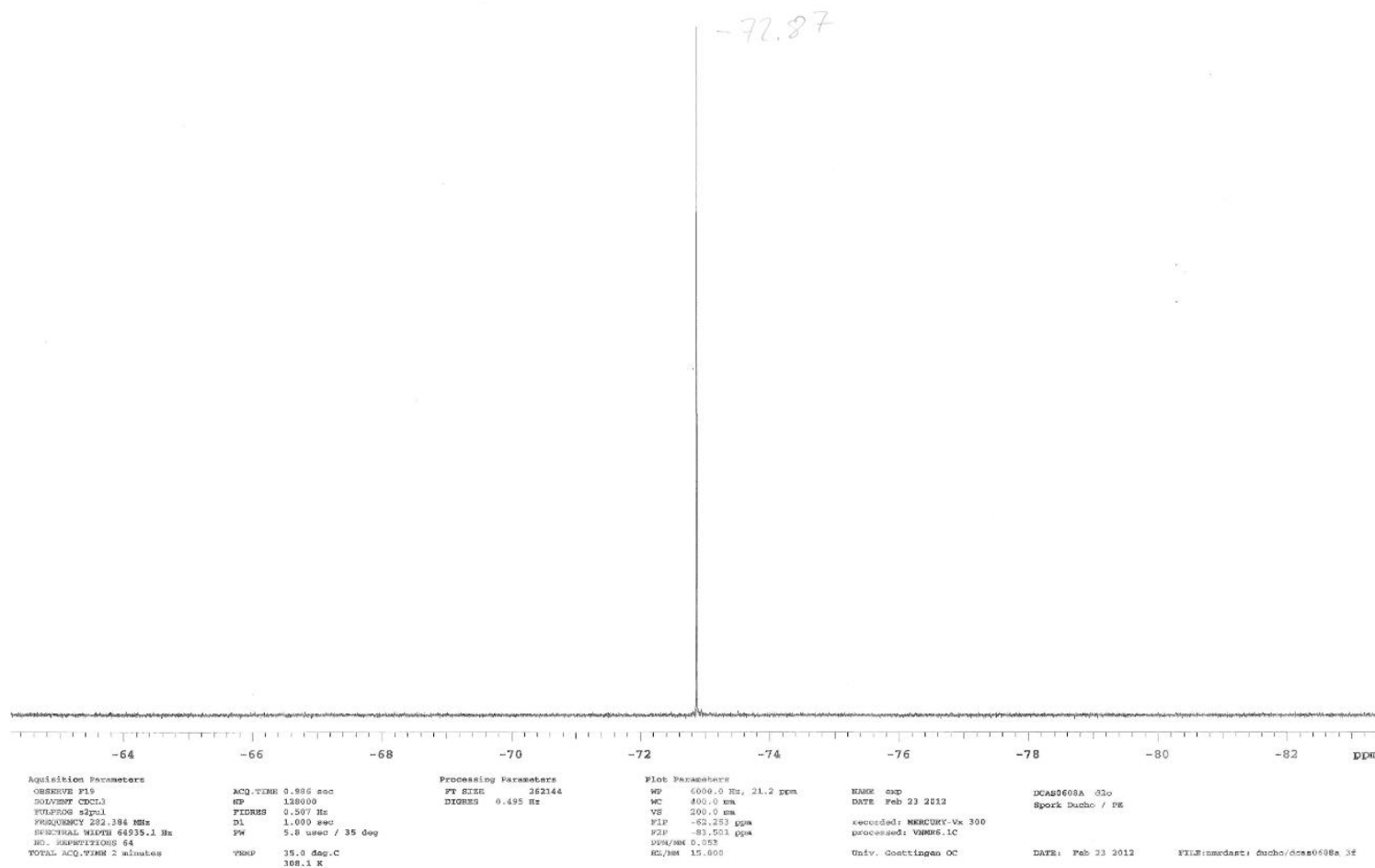
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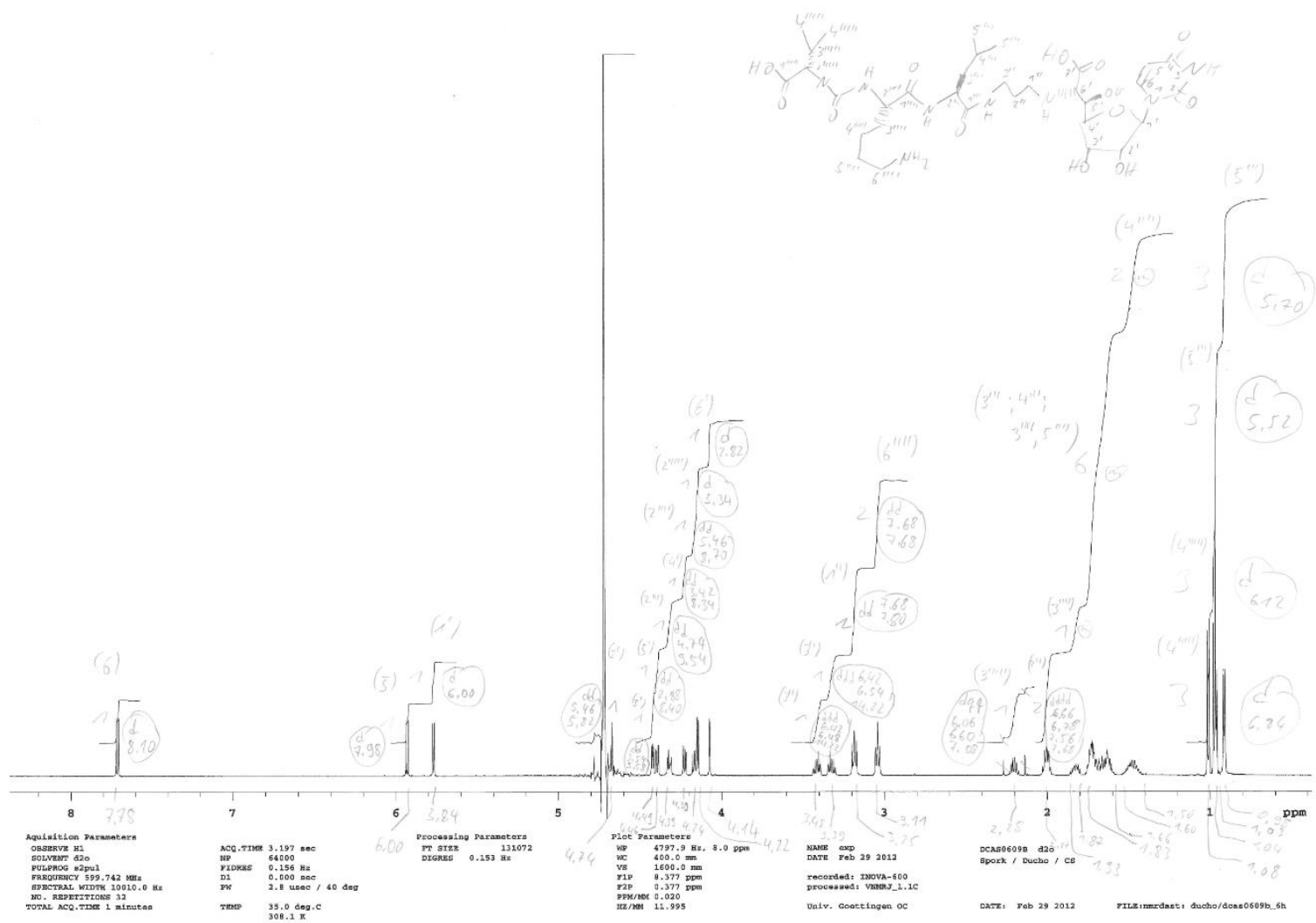
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 Spork / Ducho

DATE: Feb 22 2012 FILE:mardest: ducho/dca00608a_5c

^{13}C NMR spectrum of **15** (126 MHz, D_2O , 35 °C)



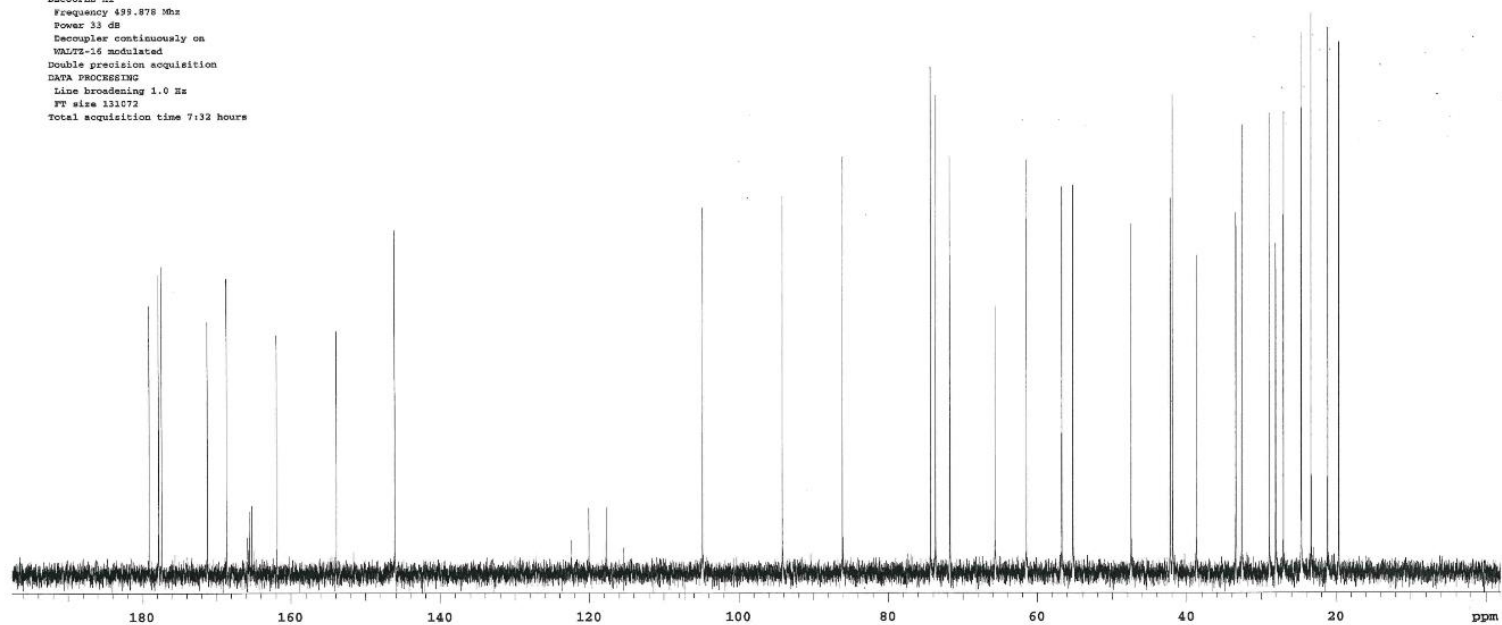
^{19}F NMR spectrum of **15** (282 MHz, D_2O , 35 °C)



¹H NMR spectrum of **16** (600 MHz, D₂O, 35 °C)

DCM80609B d2o
 Spork / Ducho
 Feb 23 2012

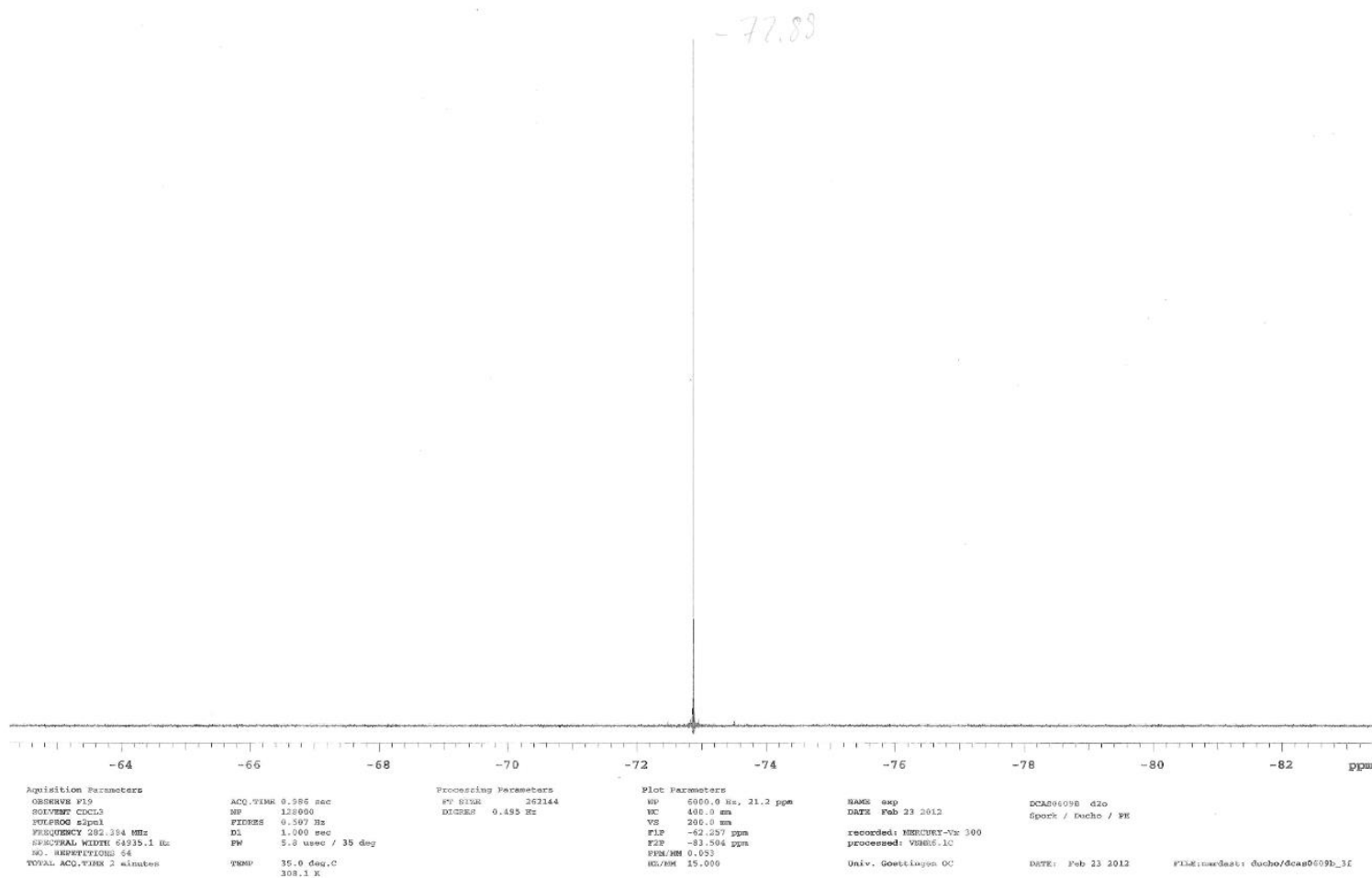
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 DATA PROCESSING
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DCM80609B d2o
 Spork / Ducho

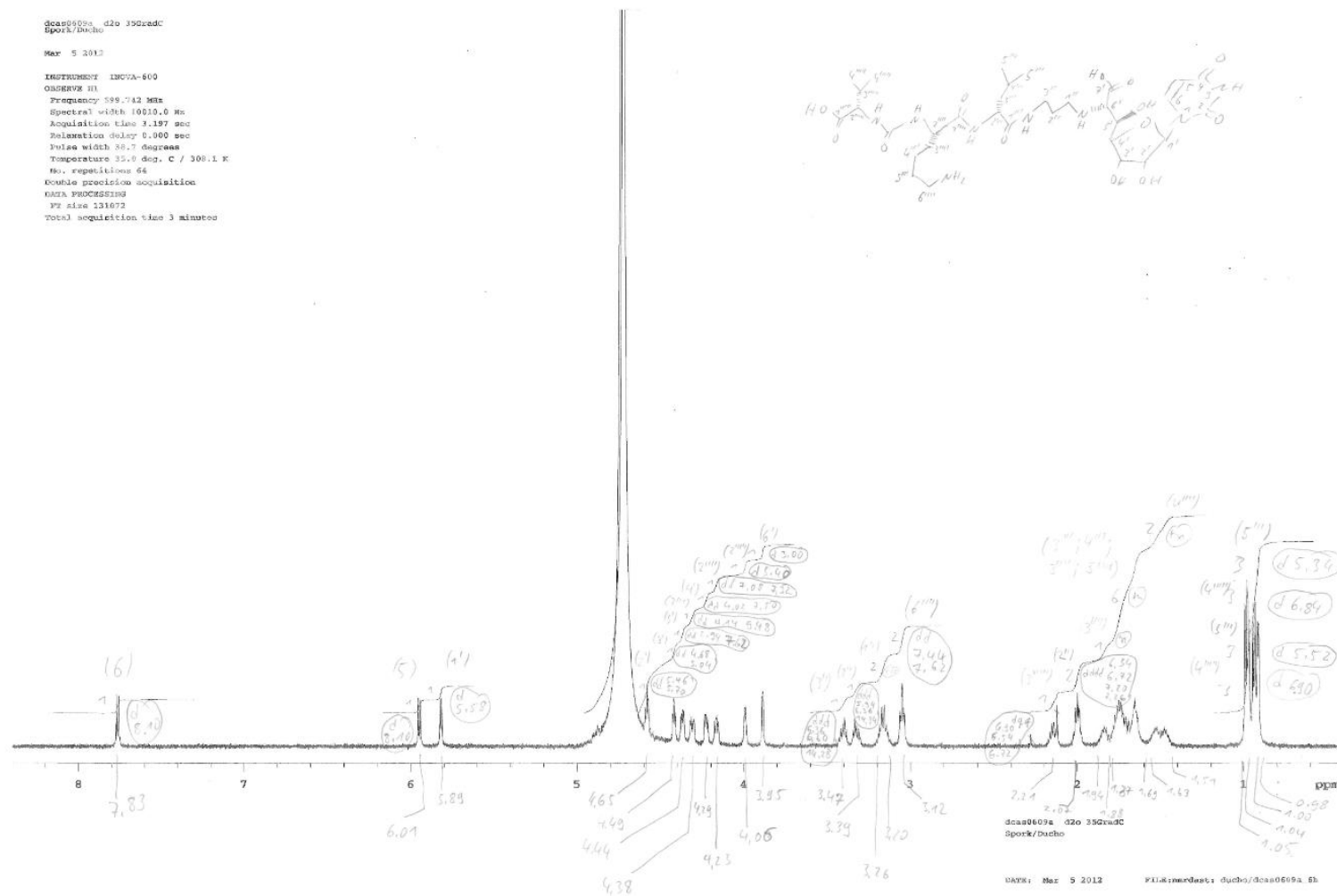
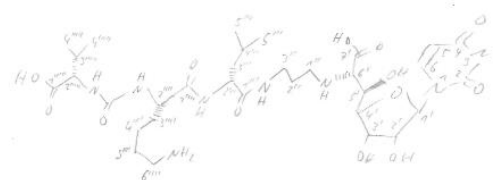
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^{13}C NMR spectrum of **16** (126 MHz, D_2O , 35 °C)



^{19}F NMR spectrum of **16** (282 MHz, D_2O , 35 °C)

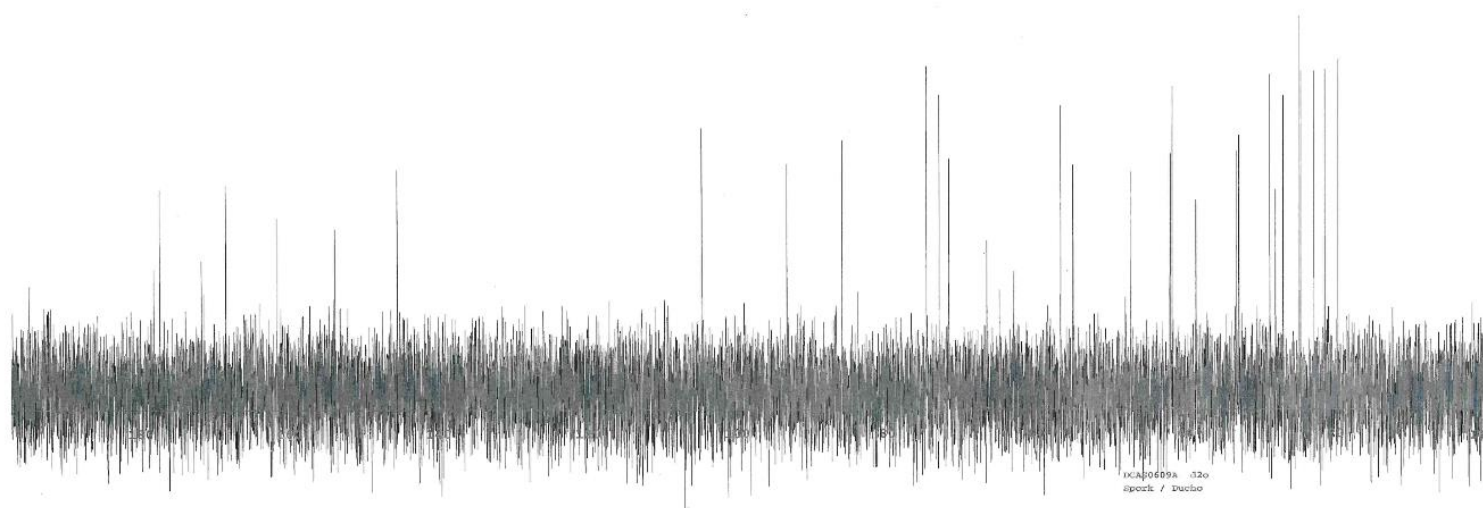
dca0609a.d2o 350radc
 Spork/Ducho
 Mar 5 2012
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¹H NMR spectrum of **17** (600 MHz, D₂O, 35 °C)

DCA0509A.d2o
 Spork / Ducho
 Feb 26 2012

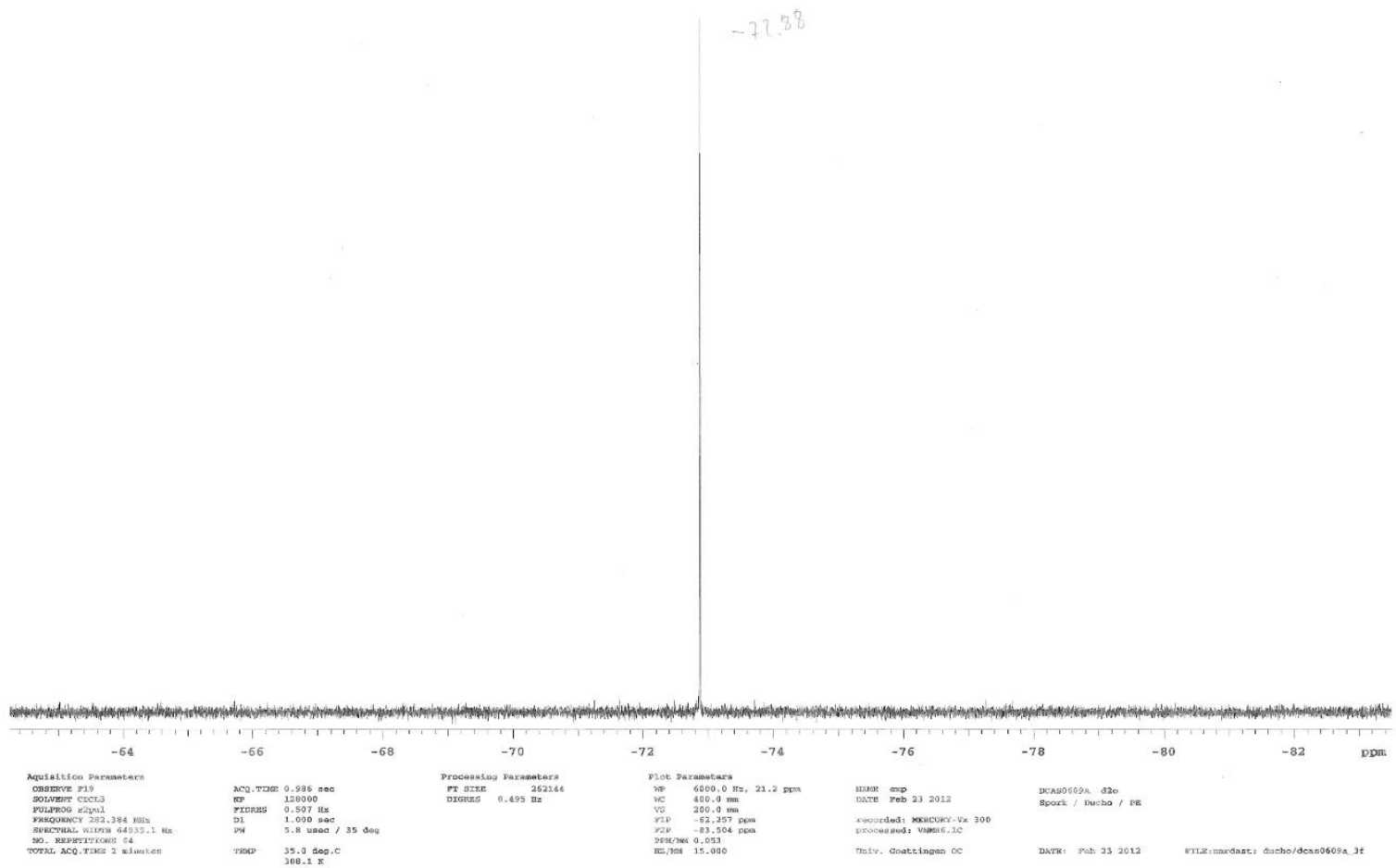
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 Power 25 dB
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 Double precision acquisition
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 262144
 Total acquisition time 9:25 hours



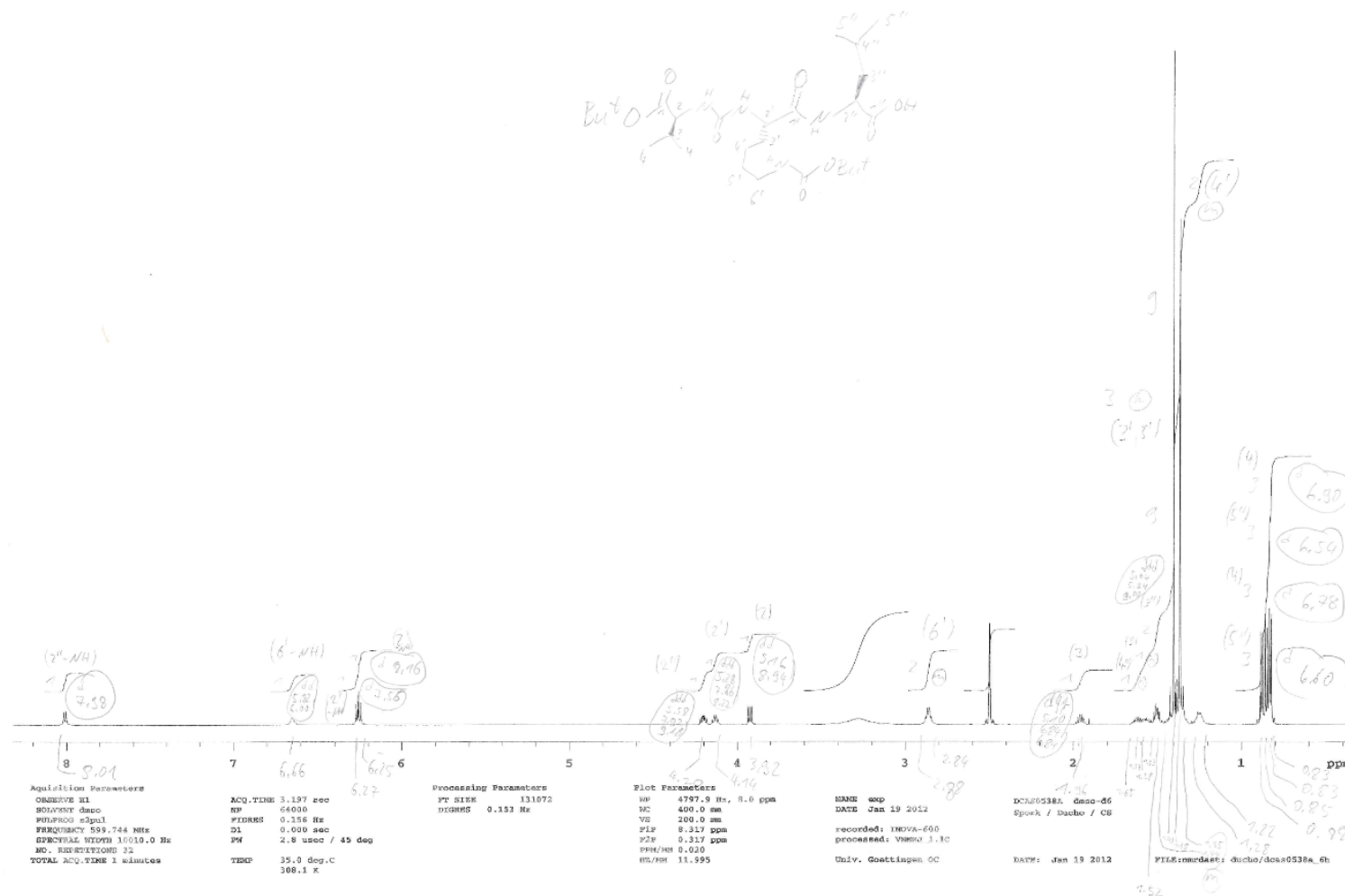
DCA0509A.d2o
 Spork / Ducho

DATE: Feb 26 2012 FILENAME: ducho/dca0509a_sc

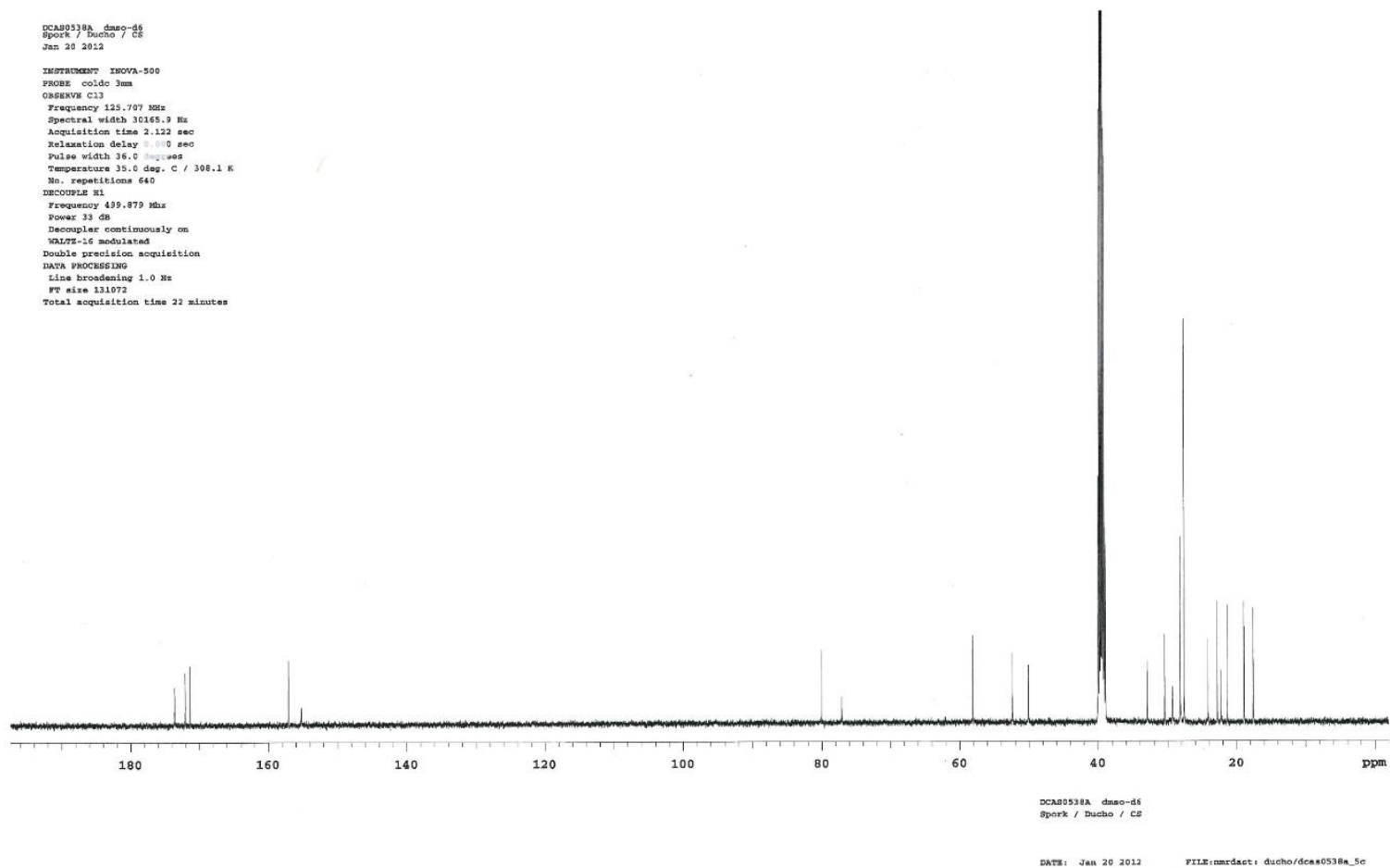
^{13}C NMR spectrum of **17** (126 MHz, D_2O , 35 °C)



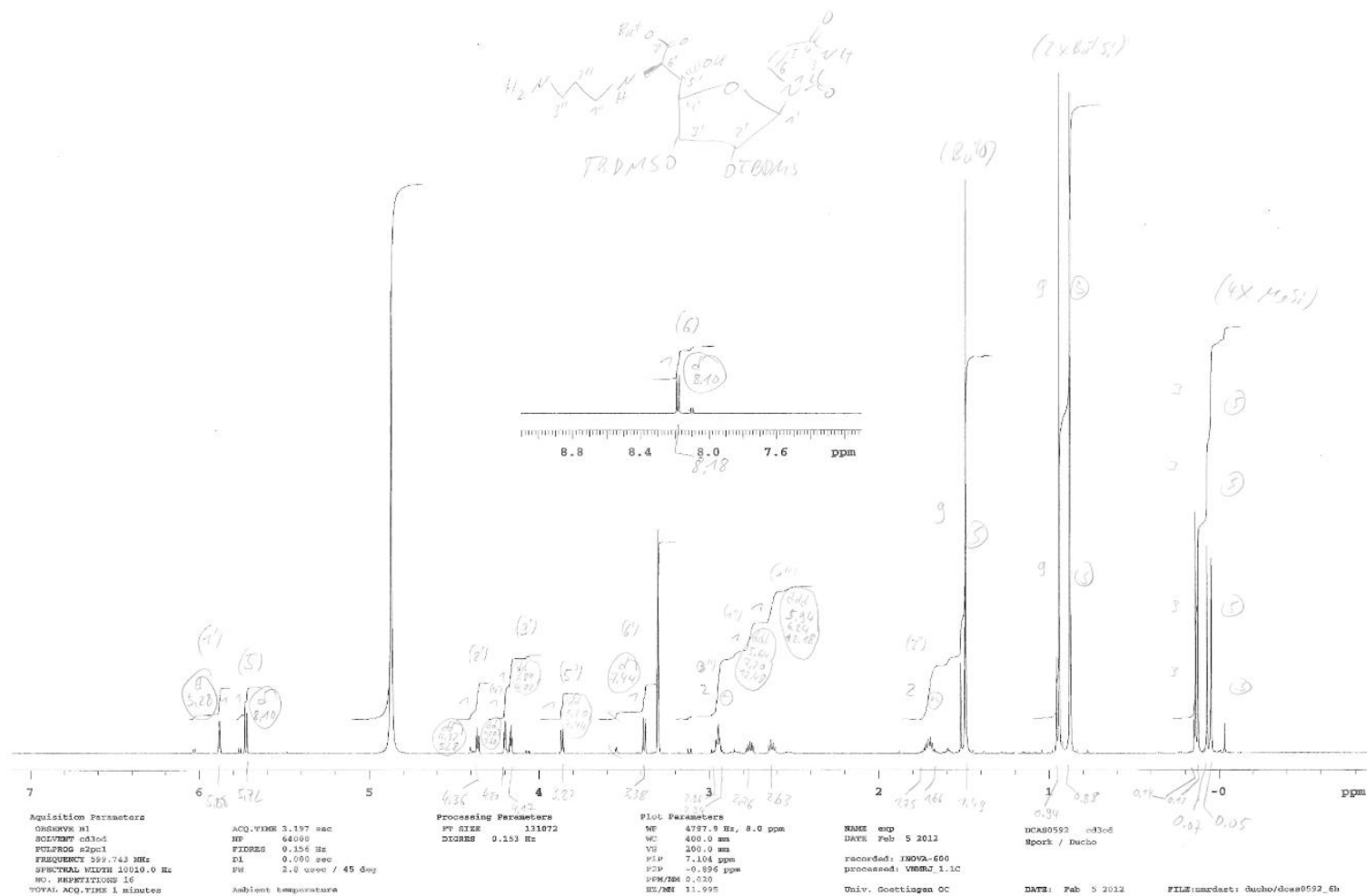
^{19}F NMR spectrum of **17** (282 MHz, D_2O , 35 °C)



¹H NMR spectrum of **18** (600 MHz, DMSO-d₆, 35 °C)



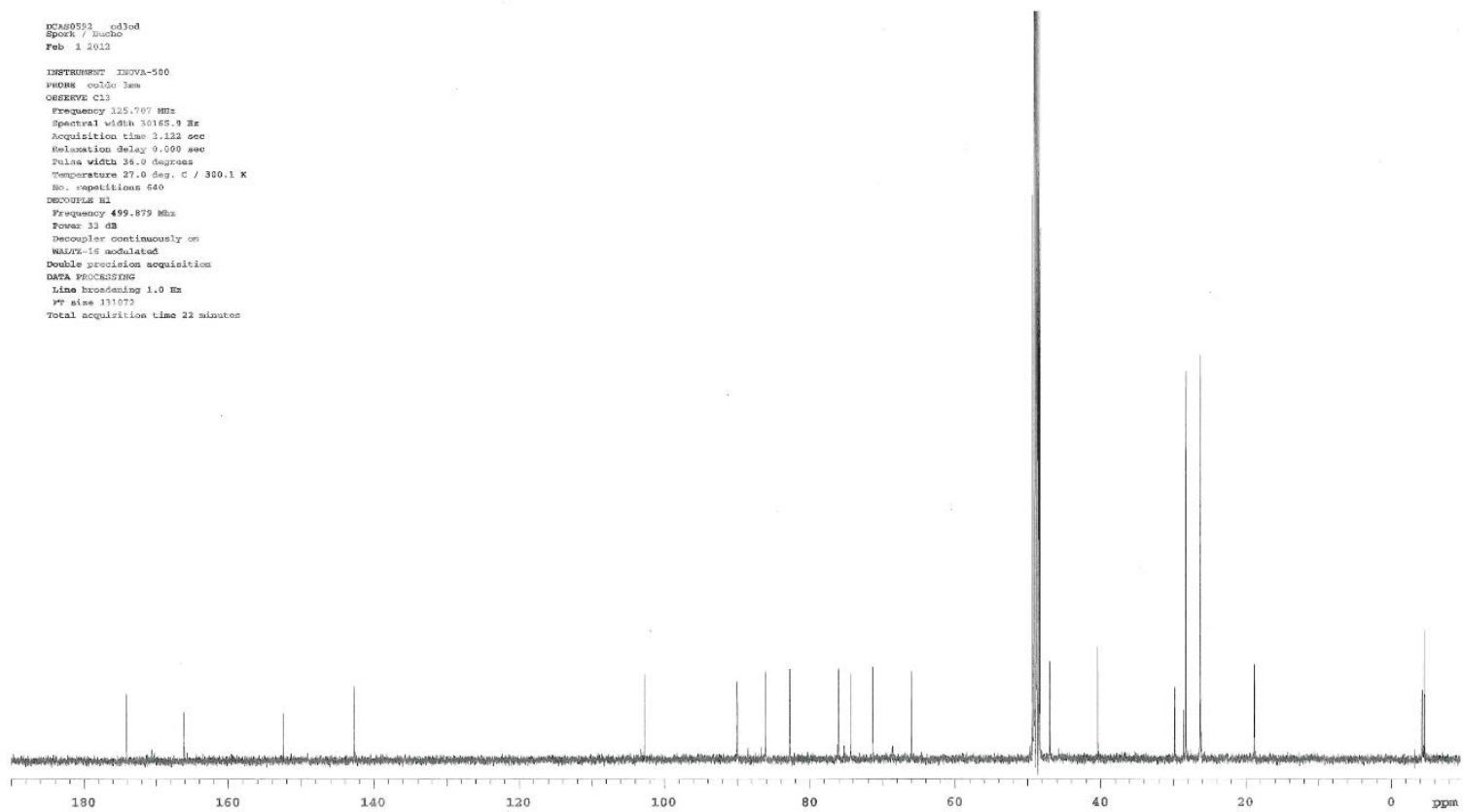
^{13}C NMR spectrum of **18** (126 MHz, DMSO- d_6 , 35 °C)



^1H NMR spectrum of **21** (600 MHz, CD_3OD)

DCA0592 edited
 Spork / Ducho
 Feb 1 2012

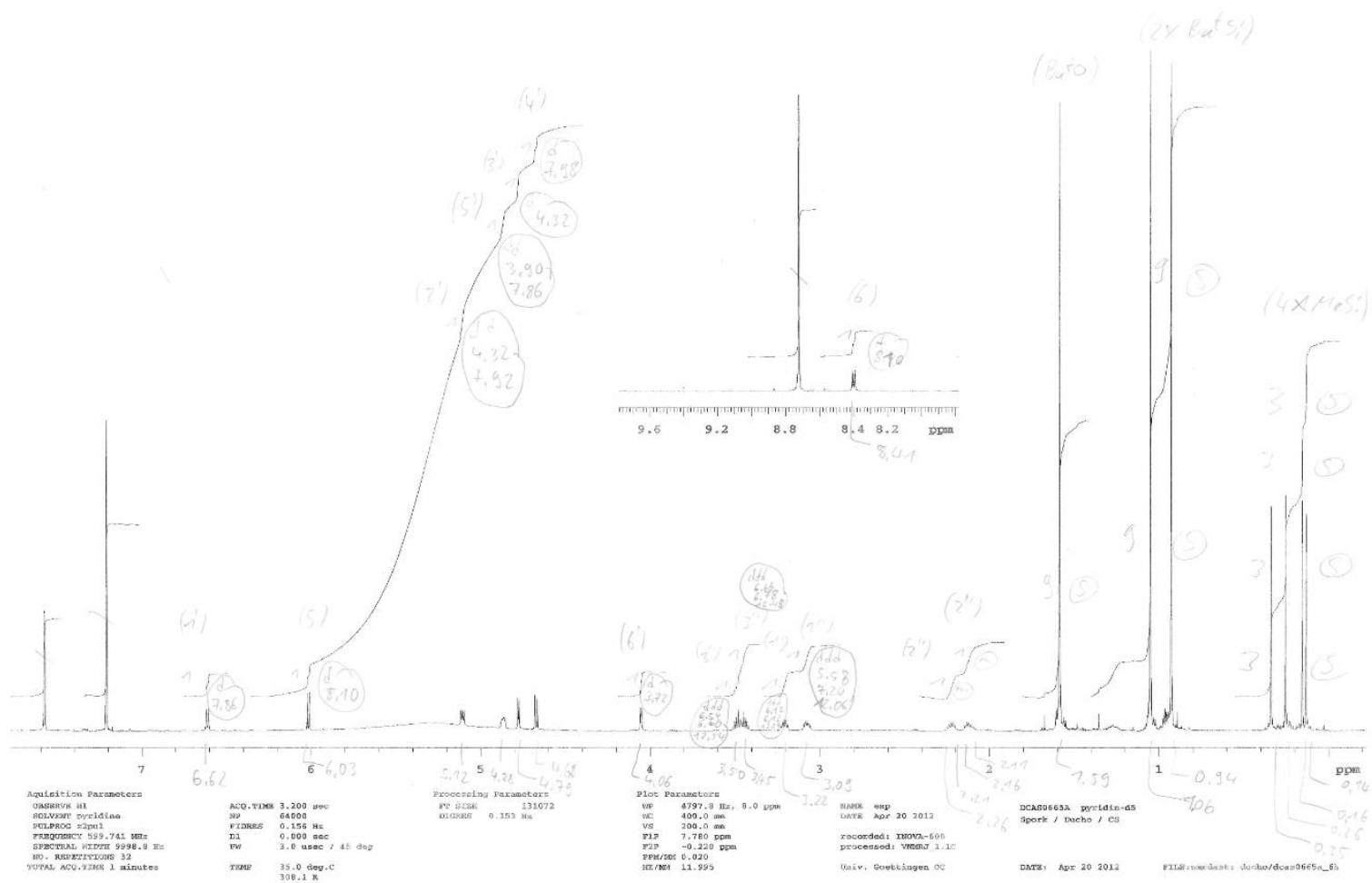
 INSTRUMENT INOVA-500
 P100K 400.150 MHz
 OBSERVE C13
 Frequency 125.767 MHz
 Spectral width 50165.9 Hz
 Acquisition time 3.112 sec
 Relaxation Delay 9.000 sec
 Pulse width 36.0 degrees
 Temperature 27.0 deg. C / 300.1 K
 No. repetitions 640
 DECOUPLE H1
 Frequency 499.879 MHz
 Power 33 dB
 Decoupler continuously on
 WALTZ-16 modulated
 Double precision acquisition
 DATA PROCESSING
 F2 file processing 1.0 Hz
 F2 size 131072
 Total acquisition time 22 minutes



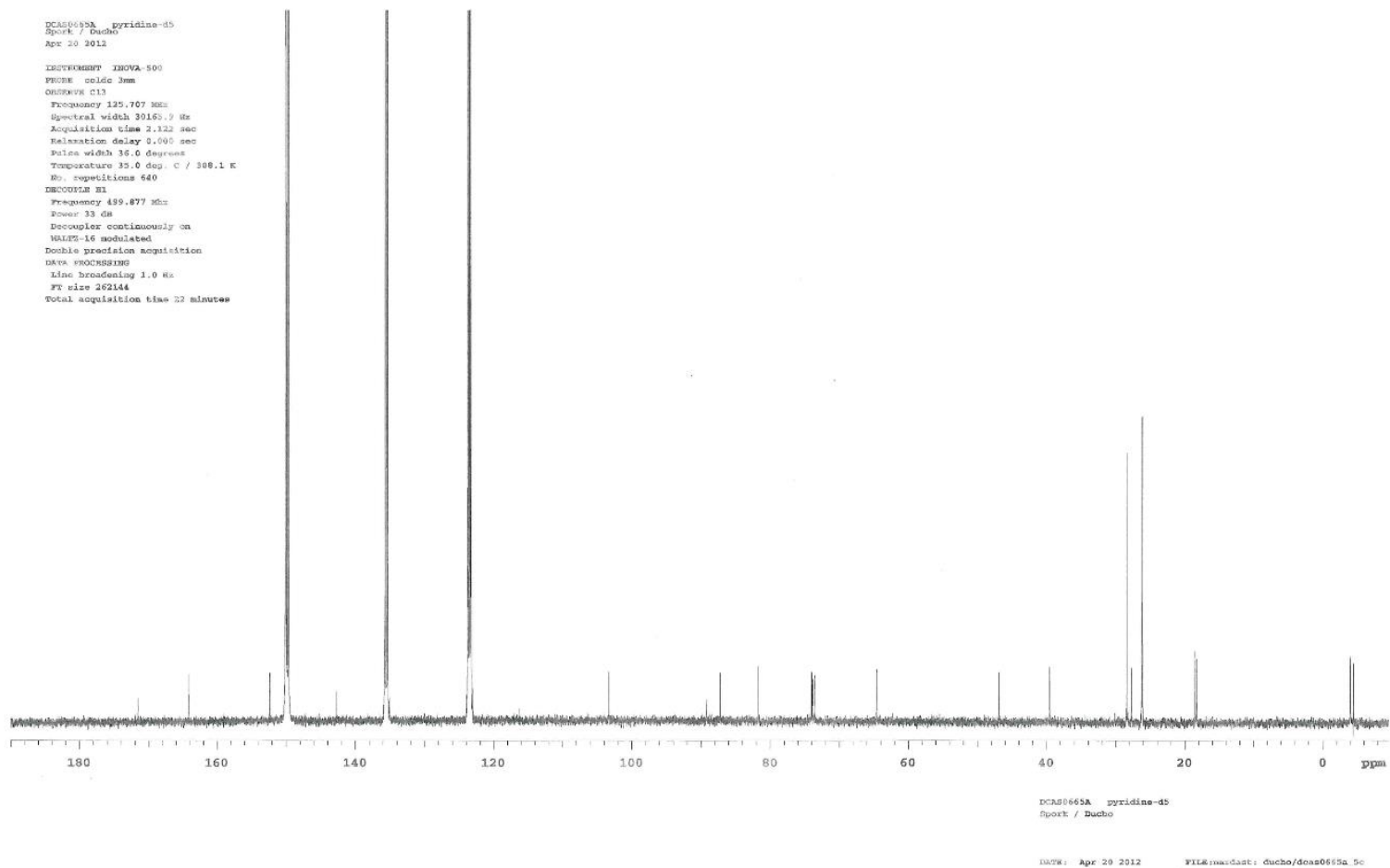
DCA0592 edited
 Spork / Ducho

DATE: Feb 1 2012 FILENAME: ducho/dca0592_5c

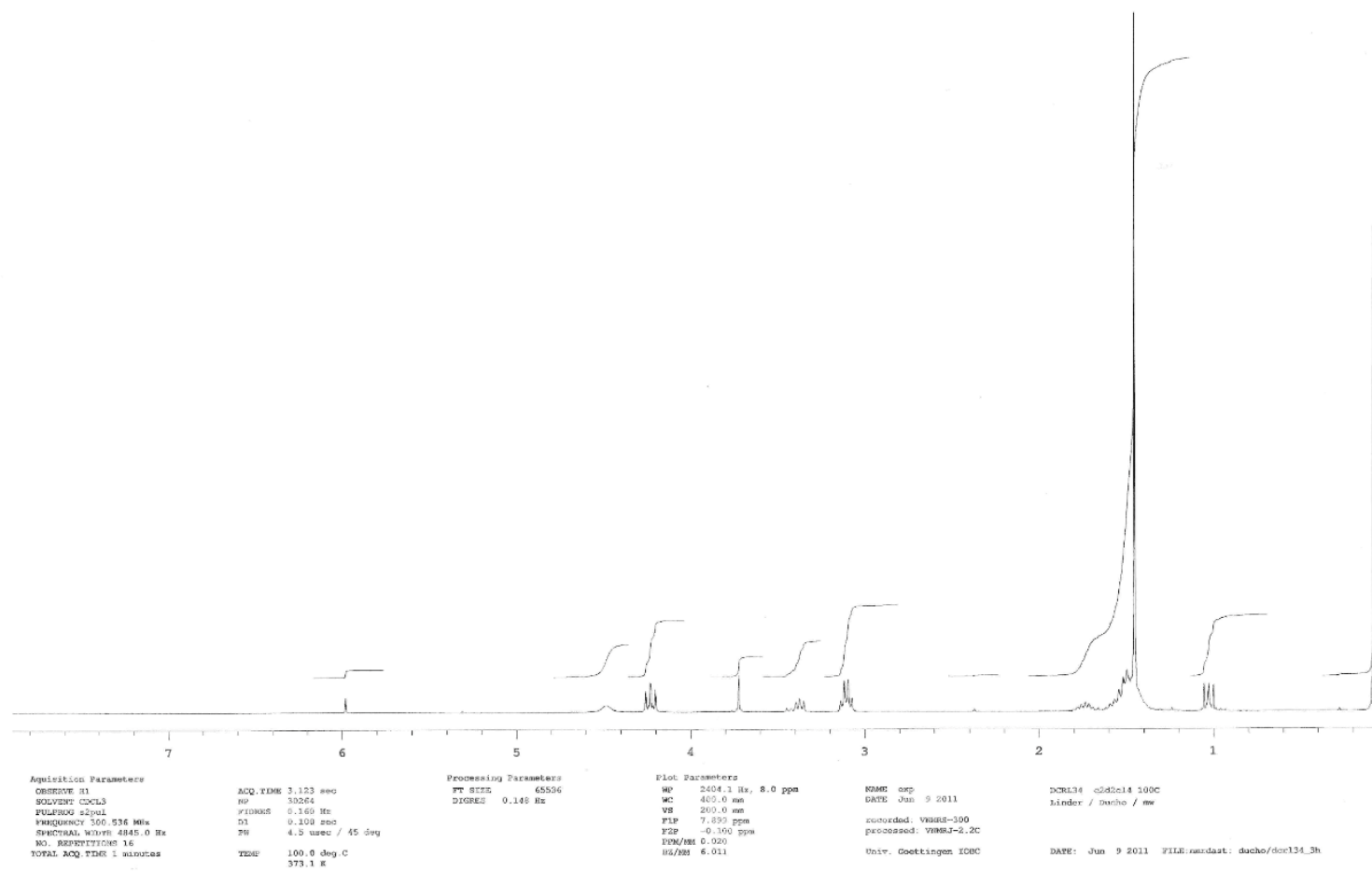
^{13}C NMR spectrum of **21** (126 MHz, CD_3OD)



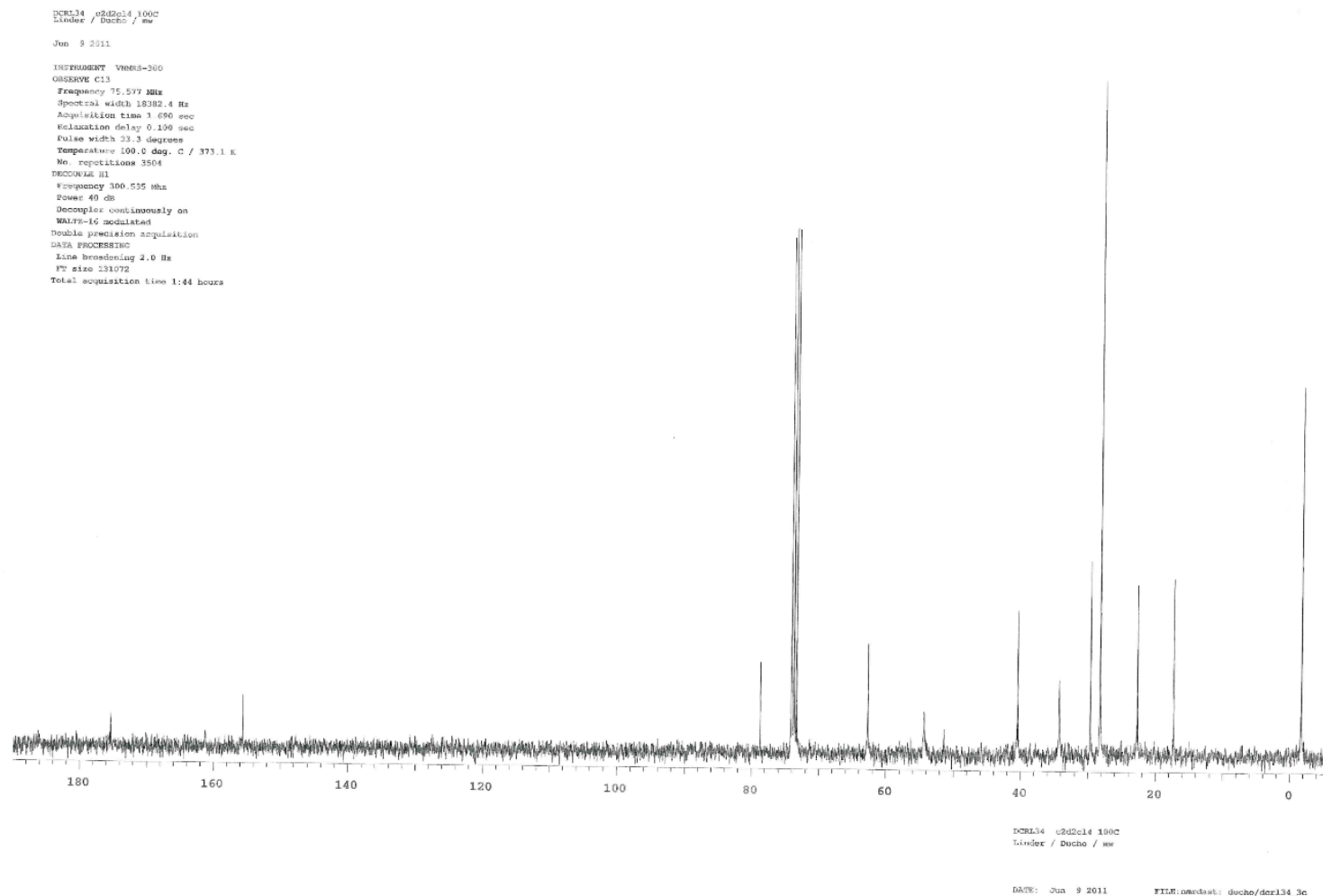
^1H NMR spectrum of **22** (600 MHz, pyridine- d_5 , 35 °C)



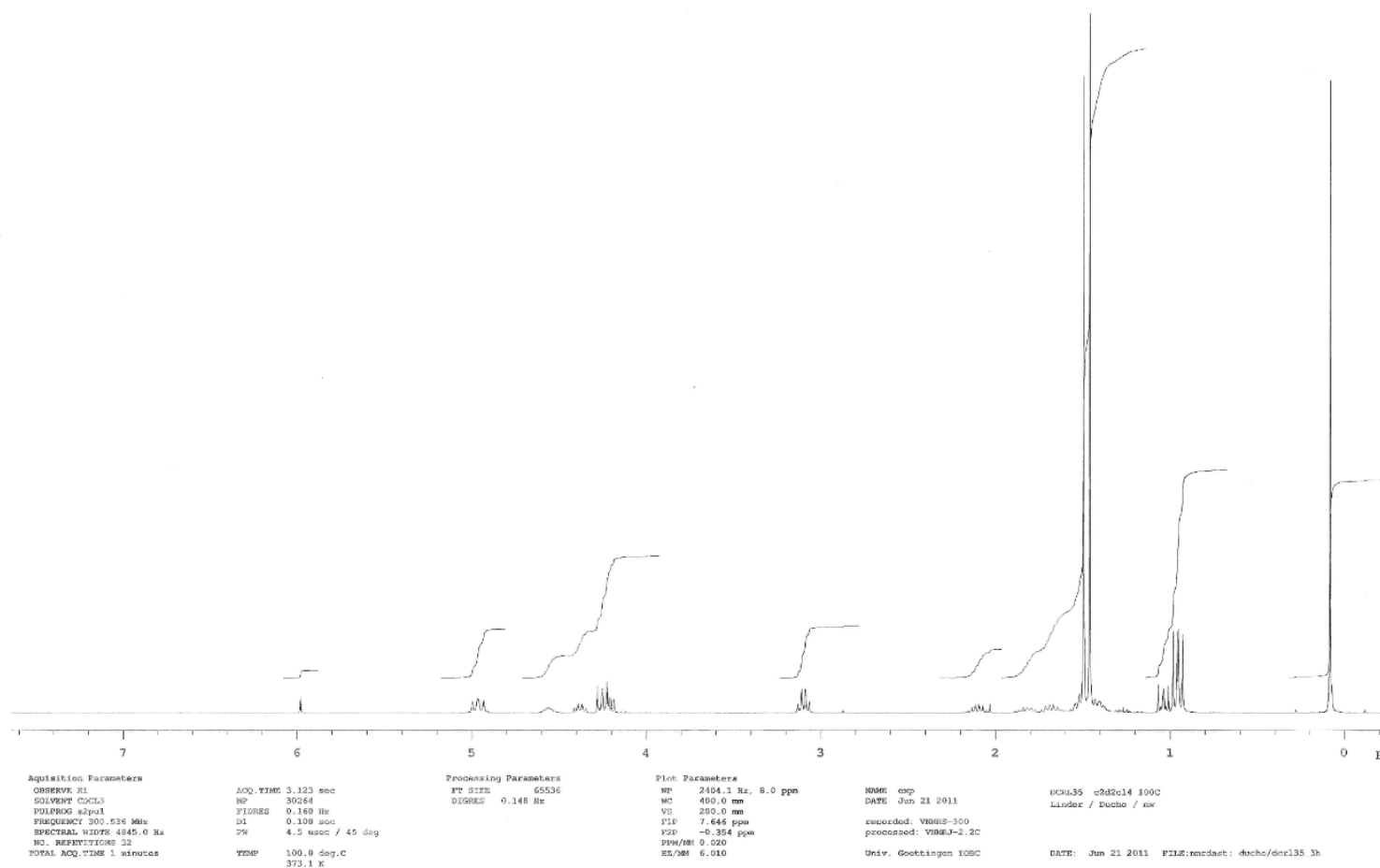
^{13}C NMR spectrum of **22** (126 MHz, pyridine- d_5 , 35 °C)



^1H -NMR of **24** (300 MHz, CD_2Cl_4 , 100 °C)



^{13}C NMR spectrum of **24** (75 MHz, CD_2Cl_4 , 100 $^\circ\text{C}$)

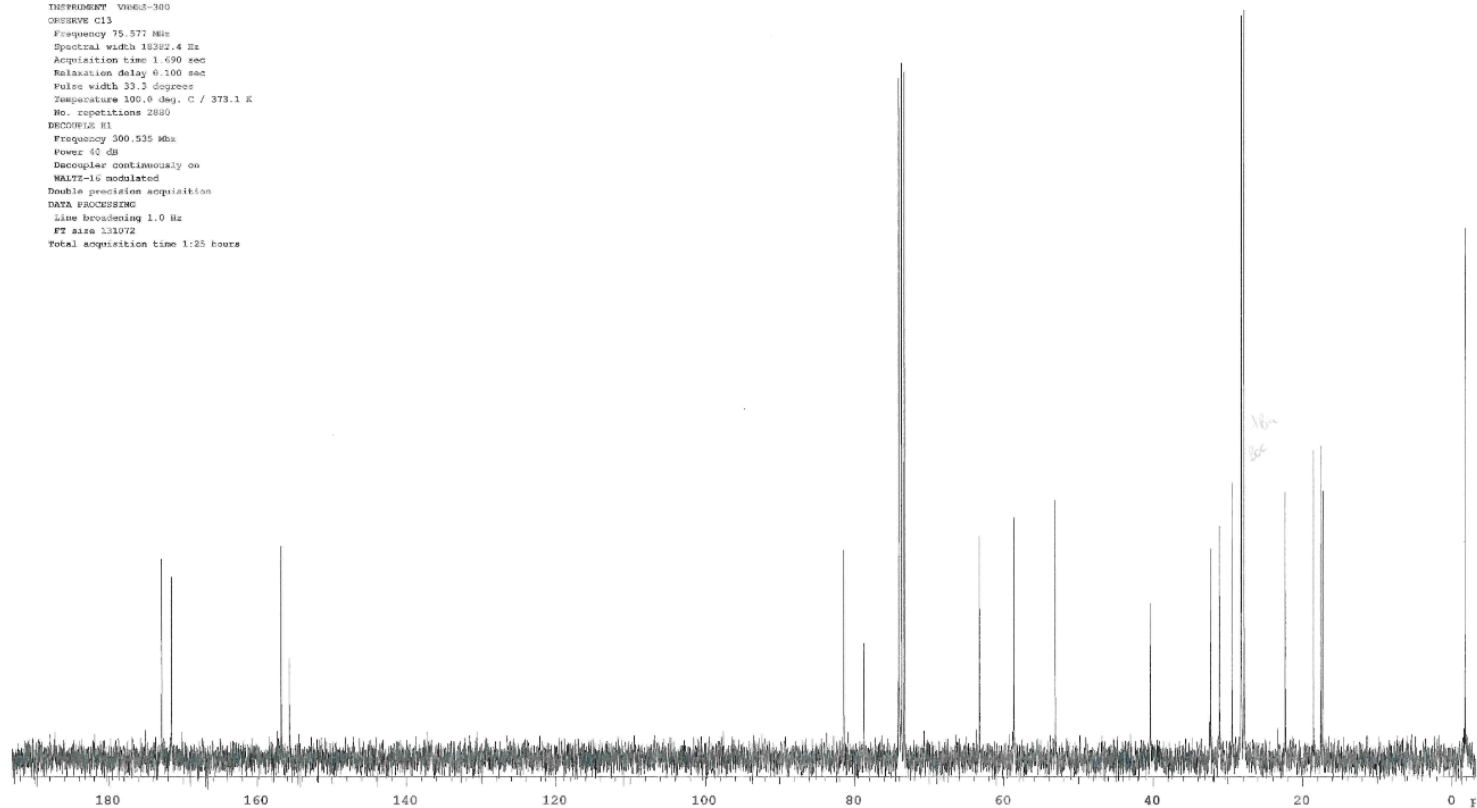


^1H NMR spectrum of **26** (300 MHz, CD_2Cl_4 , 100 °C)

DCL135 c2d2c14 100C
 linder / Ducho / mw

 Jun 21 2011

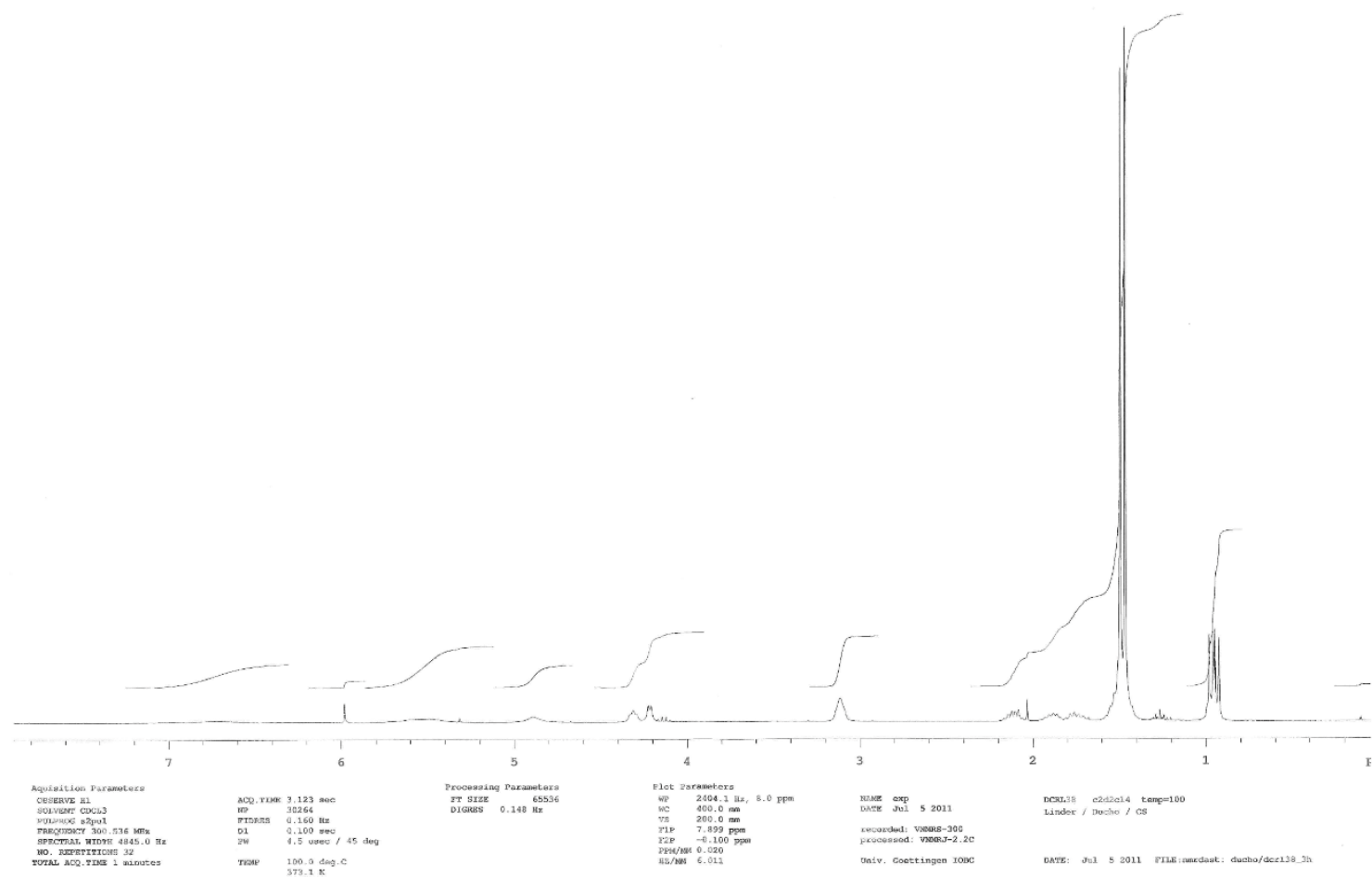
 INCREMENT 00005-300
 OBSERVE C13
 Frequency 75.577 MHz
 Spectral width 18392.4 Hz
 Acquisition time 1.490 sec
 Relaxation delay 6.100 sec
 Pulse width 33.3 degrees
 Temperature 100.0 deg. C / 373.1 K
 No. repetitions 2880
 DECOUPLER H1
 Frequency 500.535 MHz
 Power 40 dB
 Decoupler continuously on
 WALTZ-16 modulated
 Double precision acquisition
 DATA PROCESSING
 Line broadening 1.0 Hz
 F2 size 331072
 Total acquisition time 1:25 hours



DCL135 c2d2c14 100C
 linder / Ducho / mw

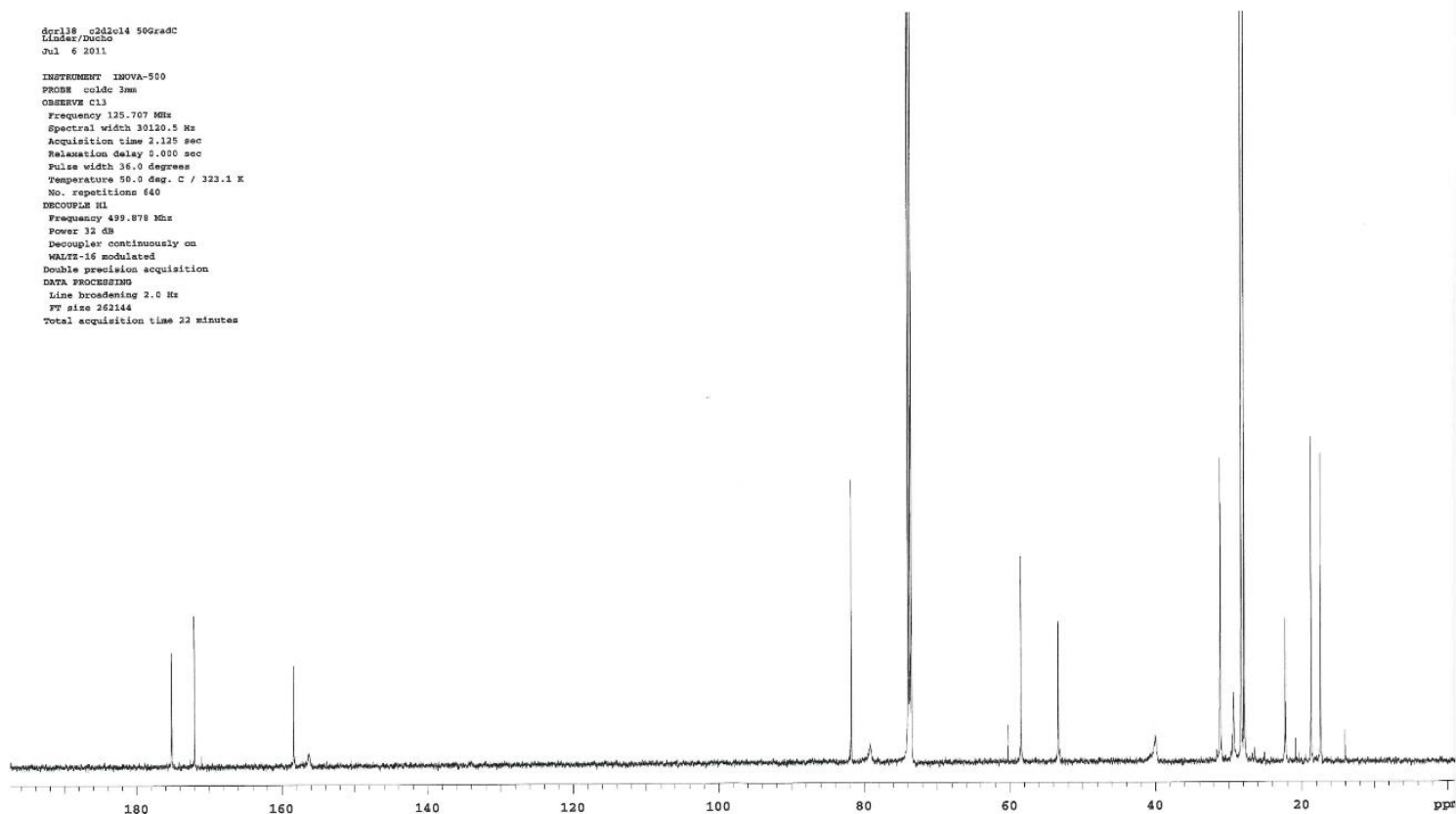
DATE: Jun 21 2011 FILE:ncrdest: ducho/dcl135 3c

^{13}C NMR spectrum of **26** (75 MHz, CD_2Cl_4 , 100 °C)



^1H NMR spectrum of **27** (300 MHz, CD_2Cl_4 , 100 °C)

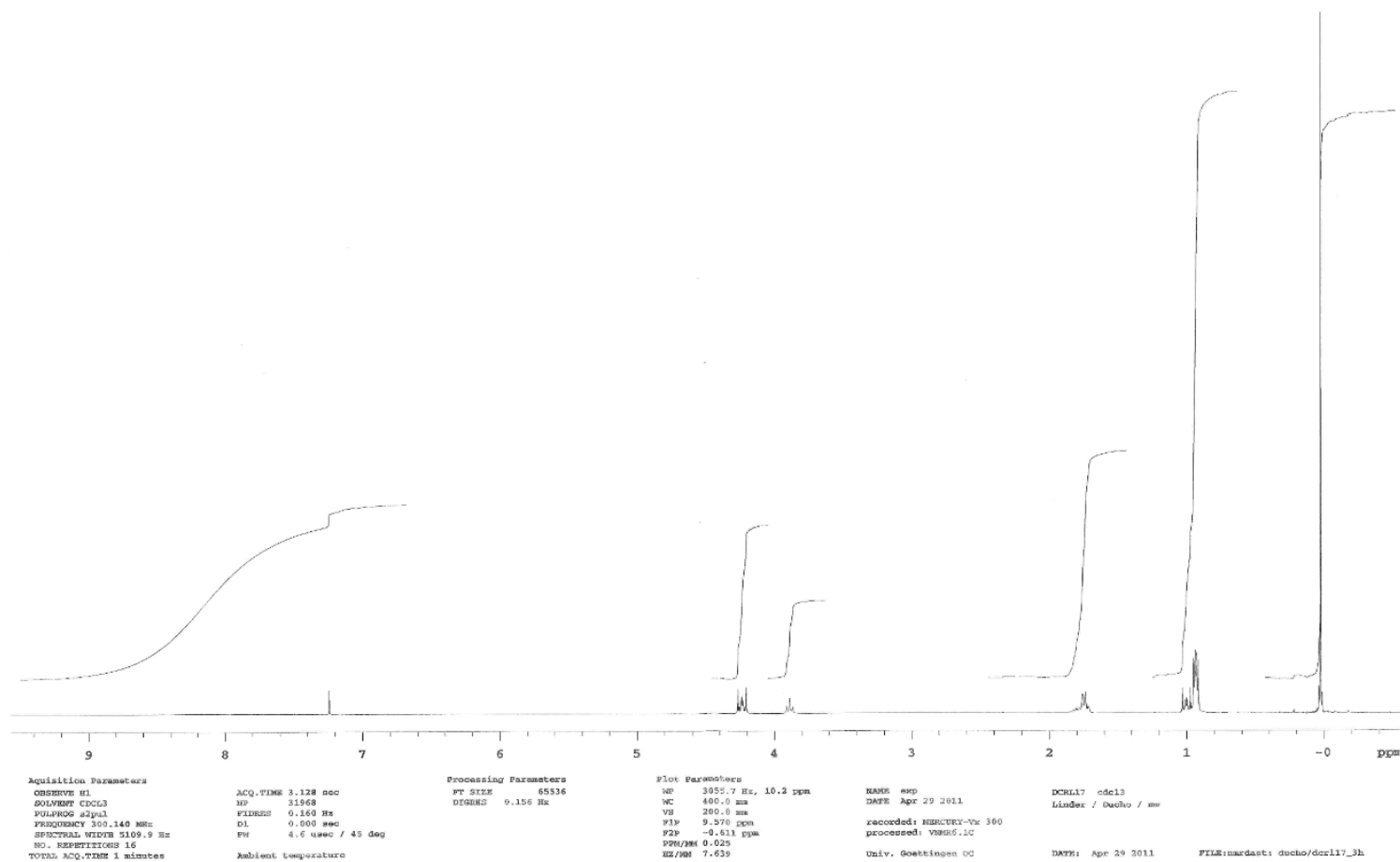
dcr138 c2d2c14 50gradC
 Lindar/Ducho
 Jul 6 2011
 INSTRUMENT INOVA-500
 PROBE cddc 3mm
 OBSERVE CL3
 Frequency 125.707 MHz
 Spectral width 30120.5 Hz
 Acquisition time 2.125 sec
 Relaxation delay 5.000 sec
 Pulse width 16.0 degrees
 Temperature 50.0 deg. C / 323.1 K
 No. repetitions 640
 DECOUPLE H1
 Frequency 499.878 MHz
 Power 12 dB
 Decoupler continuously on
 WALTZ-16 modulated
 Double precision acquisition
 DATA PROCESSING
 Line broadening 2.0 Hz
 FT size 262144
 Total acquisition time 22 minutes



dcr138 c2d2c14 50GradC
 Lindar/Ducho

DATE: Jul 6 2011 FILE:mrdata: ducho/dcr138_5c

^{13}C NMR spectrum of **27** (75 MHz, CD_2Cl_4 , 100 °C)

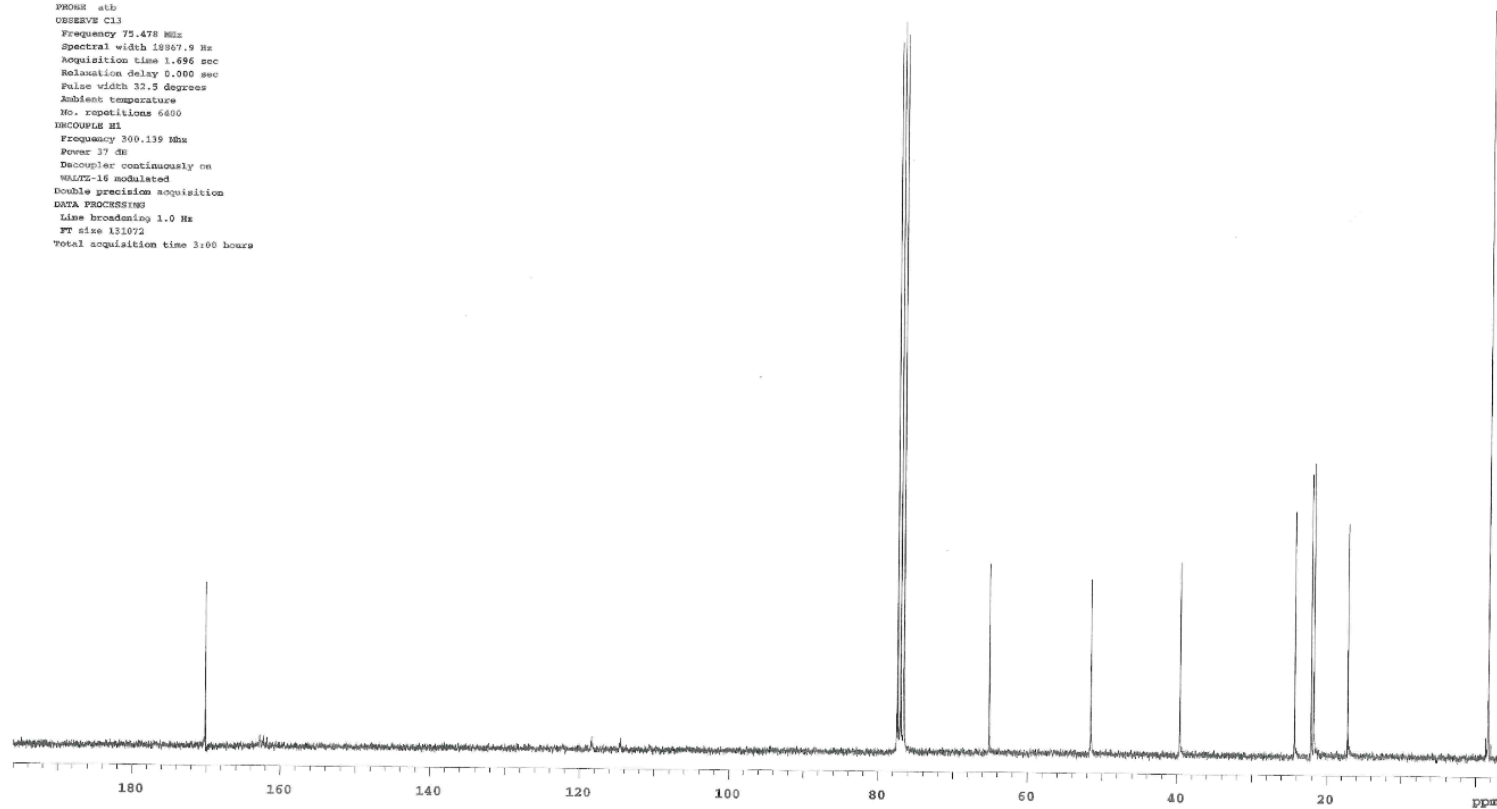


^1H NMR spectrum of **28** (300 MHz, CDCl_3)

DCE117 cd13
 kinder / Ducho / mw

 Apr 29 2011

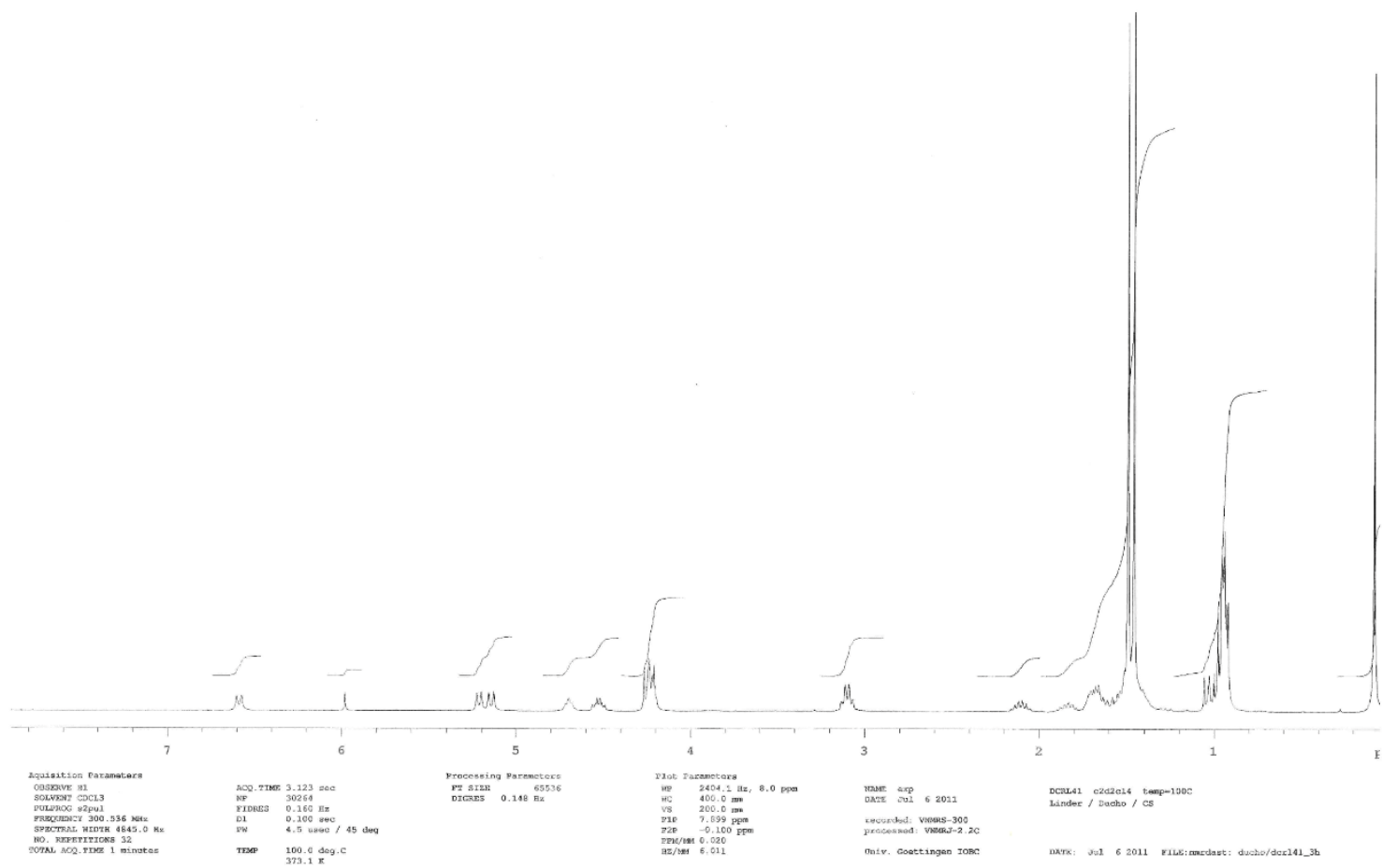
 INSTRUMENT MERCURY-300
 PROBE atb
 OBSERVE C13
 Frequency 75.478 MHz
 Spectral width 19867.9 Hz
 Acquisition time 1.696 sec
 Relaxation delay 0.000 sec
 Pulse width 32.5 degrees
 Ambient temperature
 No. repetitions 6400
 DECOUPLE H1
 Frequency 300.139 MHz
 Power 37 dB
 Decoupler continuously on
 WALTZ-16 modulated
 Double precision acquisition
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 131072
 Total acquisition time 3:00 hours



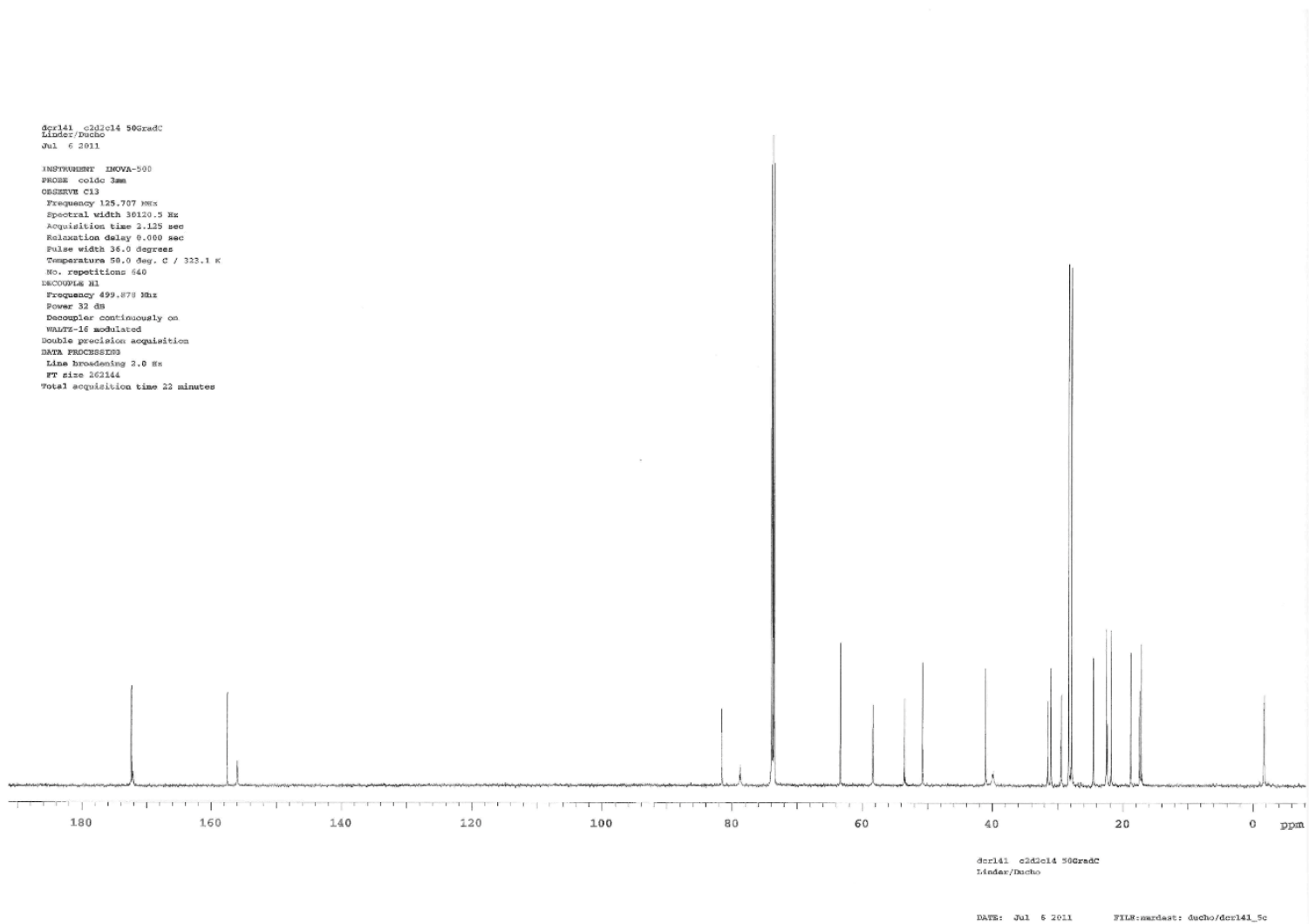
DCE117 cd13
 kinder / Ducho / mw

DATE: Apr 29 2011 FILE:mrdest: ducho/dor117_3c

^{13}C NMR spectrum of **28** (75 MHz, CDCl_3)



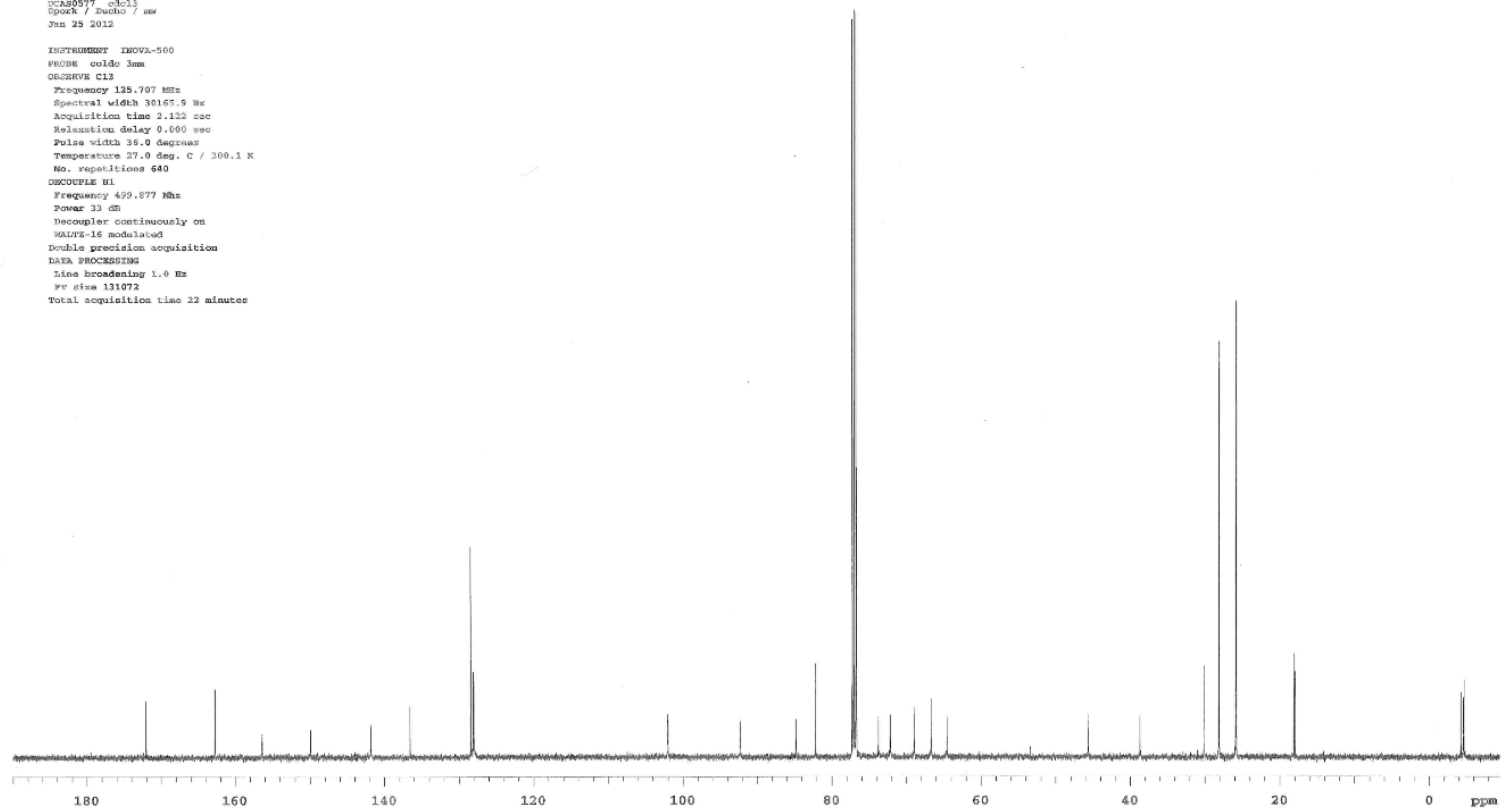
^1H NMR spectrum of **29** (300 MHz, CD_2Cl_4 , 100 °C)



^{13}C NMR spectrum of **29** (75 MHz, CD_2Cl_4 , 100 $^\circ\text{C}$)

DCAS0577 cd13
 Sport / Ducho / mw
 Jan 25 2012

 INSTRUMENT INOVA-500
 PROBE cpdco 3mm
 OBSERVE C13
 Frequency 125.767 MHz
 Spectral width 30165.5 Hz
 Acquisition time 2.122 sec
 Relaxation delay 0.000 sec
 Pulse width 36.0 degrees
 Temperature 27.0 deg. C / 300.1 K
 No. repetitions 640
 DECOUPLE H1
 Frequency 499.877 MHz
 Power 15 dB
 Decoupler continuously on
 SALT-16 modulated
 Double precision acquisition
 DATA PROCESSING
 Line broadening 1.0 Hz
 vt size 131072
 Total acquisition time 22 minutes



DCAS0577 cd13
 Sport / Ducho / mw

DATE: Jan 25 2012 FILE:mardest: ducho/dcas0577_5c

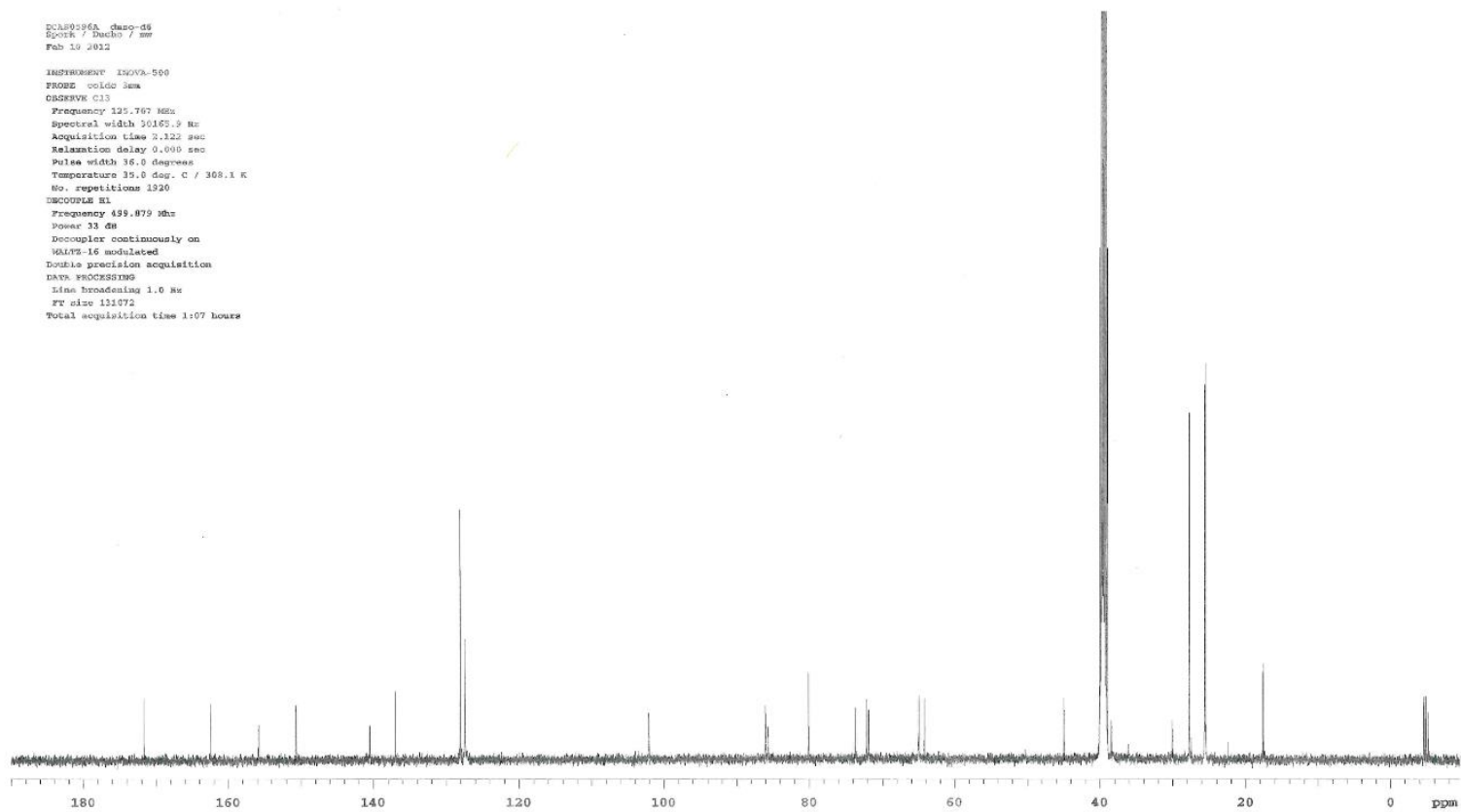
^{13}C NMR of **33** (126 MHz, CDCl_3)

DCA60596A dmsd-05
Spork / Duchs / av
Feb 10 2012

INSTRUMENT INOVA-500
PROBE cp100 5mm
CROSSPOLE C13
Frequency 125.767 MHz
Spectral width 20165.9 Hz
Acquisition time 2.122 sec
Relaxation delay 0.000 sec
Pulse width 16.0 degrees
Temperature 35.0 deg. C / 308.1 K
No. repetitions 1920

DECOUPLE H1
Frequency 499.879 MHz
Power 32 dB
Decoupler continuously on
WALTZ-16 modulated
Double precision acquisition

DATA PROCESSING
Time broadening 1.0 Hz
FT size 131072
Total acquisition time 1:07 hours



DCA60596A dmsd-05
Spork / Duchs / av

DATE: Feb 10 2012 FILE:msdata: duchs/dcas6596a_5c

^{13}C NMR spectrum of **35** (126 MHz, DMSO- d_6 , 35 °C)

References

1. Marfey, P. Determination of D-amino acids. II. Use of a bifunctional reagent, 1,5-difluoro-2,4-dinitrobenzene. *Carlsberg Res. Commun.* **1984**, 49, 591, doi:10.1007/BF02908688.
2. Tanino, T.; Ichikawa, S.; Shiro, M.; Matsuda, A. Total Synthesis of (–)-Muraymycin D2 and Its Epimer. *J. Org. Chem.* **2010**, 75, 1366-1377, doi:10.1021/jo9027193.
3. Bhushan, R.; Brückner, H. Marfey's reagent for chiral amino acid analysis: A review. *Amino Acids* **2004**, 27, 231-247, doi:10.1007/s00726-004-0118-0.