Supporting Information

Sulfonated N-heterocyclic Carbenes for Suzuki Coupling in Water

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Experimental

General Experimental:

All chemicals were purchased as reagent grade from commercial suppliers and used without further purification, unless otherwise noted. All solvents were used as technical grade purity. DMSO (crowncap) was purchased from *Fluka*.

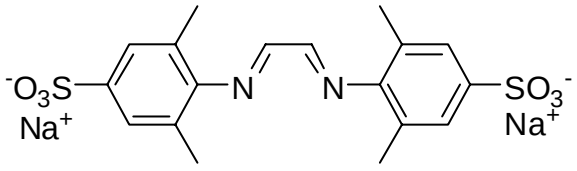
Proton (^{1}H NMR), carbon (^{13}C NMR) and phosphorus (^{31}P NMR) nuclear magnetic resonance spectra were recorded on Bruker DRX 500 at 500 MHz, 125.75 MHz and 202.46, respectively or on Bruker DRX 300 at 300 MHz and 75.07 MHz respectively at 293 K. The chemical shifts are given in parts per million (ppm) on the delta scale (δ) and are referenced to residual non deuterated solvent signals. Abbreviations for NMR data: s = singlet; d = doublet; t = triplet; q = quartet; qi = quintet; sept = septet; dd = doublet of doublets; dt = doublet of triplets; dq = doublet of quartets; tt = triplet of triplets; m = multiplet. Mass spectra were recorded on a Finnigan MAT 95 magnetic sector spectrometer. Thin layer chromatography (TLC) was performed using TLC plates RP_{18} F 254 (0.2 mm) on glass plates. Reversed phase columns for chromatography were prepared with HP-20 polymer.

GC experiments were run on a *Clarus* 500 GC with autosampler and FID detector. Column: Varian CP-Sil 8 CB (l = 15 m, d_i = 0.25 mm, d_F = 1.0 µm), N_2 (flow: 17 cm/sec; split 1:50); Injector-temperature: 270 °C, detector temperature: 350 °C. Temperature program: isotherm 150°C for 5 min, heating to 300°C with 25 °C/min, isotherm for 15 min.

4-sodiumsulfonato-2,6-dimethyl-aniline: In a 500 mL 2-necked round bottomed flask 2,6-dimethylaniline (36.4 g, 300 mmol) was dropped to a mixture of conc. sulfuric acid (30.6 g, 300 mmol) and water (55 mL) within 20 min using a metal bath, the resulting clear solution was treated at 160 °C at 4 mbar without stirring to remove most of the water (30 min), a white

solid was formed. Then the temperature was raised to 260 °C for 3.5 h (baking process). The resulting grey solid was allowed to cool to 80 °C, NaOH (300 mL, 1.7 M aqueous solution) was added and the reaction mixture refluxed for additional 1.5 h. The hot mixture was filtered over a paper filter, then acidified with conc. HCl to pH 1.5. The precipitated grey solid was separated via suction filtration, suspended in water (350 mL), activated charcoal (2.0 g) was added, the solution was adjusted to pH 8 adding by Na_2CO_3, and the solution was refluxed for 20 min. After hot filtration of the solution some NaCl (50 g) was added and the solution allowed standing in a fridge overnight. The crystallized product was separated via suction filtration and the water removed in vacuo affording 4-sodiumsulfonato-2,6-diisopropyl-aniline as off-white crystals (34 g, 51 %).
The NMR data are identical to those in the literature (ref. 32).

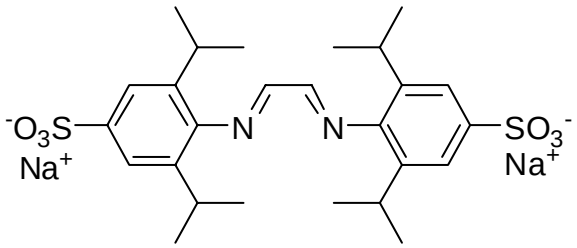
4-sodiumsulfonato-2,6-diisopropyl-aniline: In a 500 mL two-necked round bottomed flask 2,6-diisopropylaniline (44.3 g, 250 mmol) was dropped to a mixture of conc. sulfuric acid (25.5 g, 250 mmol) and water (46 mL) within 20 min. The resulting white solid was afterwards crushed several times with a glass rod. Using a metal bath, the reaction mixture was treated at 160 °C at 4 mbar without stirring to remove most of the water. Then the temperature was raised to 260 °C for 3.5 h (baking process). The resulting grey solid was allowed to cool to 80 °C, NaOH (240 mL, 1.7 M aqueous solution) was added and the reaction mixture refluxed for additional 1.5 h. The hot mixture was filtered over a paper filter, then acidified with conc. HCl to pH 1.5. The precipitated grey solid was separated via suction filtration, suspended in water (350 mL), activated charcoal (2.0 g) was added, the pH was adjusted to 8 adding Na_2CO_3, and the solution was refluxed for 20 min. After hot filtration of the solution some NaCl (50 g) was added and the solution allowed to stand in a fridge overnight. The crystallized product was separated via suction filtration and the water removed in vacuo affording 4-sodiumsulfonato-2,6-diisopropyl-aniline as dark grey crystals (46 g, 66 %). The NMR data are identical to those in the literature. G. J. P. Brotovsek, G. Y. Y. Woo, N. Assavathorn, *J. Organomet. Chem*. **2003**, *679*, 110.



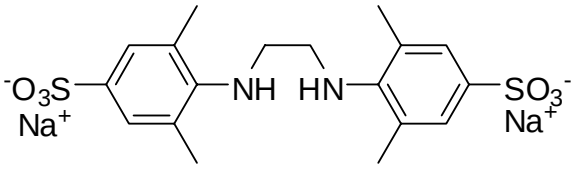
In a 2 L round-bottomed flask 4-sodiumsulfonato-2,6-dimethyl-aniline (5.0 g, 22.4 mmol) were dissolved in EtOH (150 mL, dried over CaH_2). 2,3-dihydroxy-1,4-dioxane (1.22 g, 10.2

mmol) and formic acid (15 drops) were added to the clear solution. Within 30 min a yellow solid started to precipitate, the suspension was stirred for additional 48 h at room temperature. Then the yellow solid was separated via suction filtration, digerated in boiling EtOH (3 times, 200 mL) to remove traces of starting material (aniline) and the volatiles removed in vacuo to afford the product as a yellow solid (1.8 g, 38 %)

^{1}H NMR (500 MHz, $DMSO_{d6}$) δ [ppm] 8.15 (s, 2 H, *CH*, C*H*=N), 7.37 (s, 4 H, *CH*, ar), 2.12 (s, 12 H, C*H*$_3$); ^{13}C{^{1}H} NMR (125.8 MHz, $DMSO_{d6}$) δ [ppm] 164.1, 150.0, 144.5, 125.8, 125.5, 18.2; MS (70 eV) m/z 445 $[M-Na]^-$ (neg.), 491 $[M+Na]^+$ (pos.).



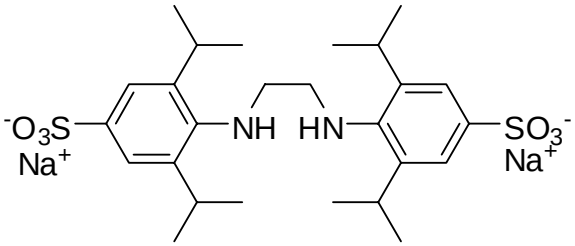
In a 250 mL round bottomed flask 4-sodiumsulfonato-2,6-diisopropyl-aniline (13.0 g, 46.5 mmol) were dissolved in EtOH (150 mL, dried over $MgSO_4$). 2,3-dihydroxy-1,4-dioxane (2.54 g, 21.2 mmol) and formic acid (7 drops) were added to the clear solution. Within 30 min a yellow solid precipitated, the suspension was stirred for additional 48 h at room temperature. Then the yellow solid was separated via suction filtration, washed with ice-cold EtOH and the volatiles removed in vacuo to afford the product as a yellow solid (12.3 g, 76 %). ^{1}H NMR (500 MHz, $DMSO_{d6}$) δ [ppm] 8.15 (s, 2 H, *CH*, C*H*=N), 7.45 (s, 4 H, *CH*, ar), 2.86 (sept, ^{3}J = 6.9 Hz, 4 H, C*H*CH_3), 1.15 (d, ^{3}J = 6.9 Hz, 24 H, *CH*$_3$); ^{13}C{^{1}H} NMR (125.8 MHz, DMSO-d_6) δ [ppm] 163.8, 148.3, 144.9, 135.6, 120.8, 28.0, 23.4; MS (70 eV) m/z 557 $[M-Na]^-$ (neg.), 603 $[M+Na]^+$ (pos.).



(1.01 g, 2.16 mmol) of the tetramethyldiimine were suspended in MeOH (220 mL, dried over CaH_2). Molecular sieves (3 g, 4 Å) and Pd/C (150 mg, Pd: 10 % (w/w)) were added and the mixture hydrogenated for 2 h at 7 bar. The reaction mixture was filtered over a pad of celite to remove the catalyst and the molecular sieves and the pad was washed with MeOH (2 x 20

mL). Removal of the MeOH from the clear colourless filtrate afforded the product (0.99 g, 98 %) as a white solid

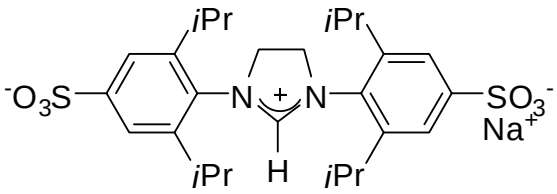
^{1}H NMR (500 MHz, DMSO$_{d6}$) δ [ppm] 7.18 (s, 4 H, *CH*, ar), 4.06 (s (br), 2 H, N*H*), 3.07 (s, 4 H, *CH*), 2.20 (s, 12 H, C*H*$_3$); ^{13}C{^{1}H} NMR (125.8 MHz, DMSO-d$_6$) δ [ppm] 147.7, 142.1, 128.9, 127.3, 49.3, 19.9; MS (70 eV) m/z 449 [M-Na]$^-$ (neg.), 495 [M+Na]$^+$ (pos.).



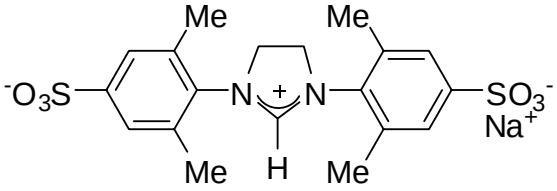
(10.1 g, 2.16 mmol) of the tetraisopropyldiimine was suspended in MeOH (220 mL, technical grade) and Pd/C (1.50 g, Pd: 10 %(w/w)) were added and the mixture hydrogenated for 6 h at 7 bar. The reaction mixture was filtered over a pad of celite to remove the catalyst and the pad was washed with MeOH (2 x 20 mL). Removal of the MeOH from the clear colourless filtrate afforded the product (10.0 g, 99 %) as a white solid. ^{1}H NMR (500 MHz, DMSO-d$_6$) δ [ppm] 7.33 (s, 4 H, *CH*, ar), 3.92 (s (br), 2 H, N*H*), 3.46-3.33 (m, H, C*H*CH$_3$), 1.17 (d, ^{3}J = 6.9 Hz, 24 H, *CH*$_3$); ^{13}C{^{1}H} NMR (125.8 MHz, DMSO$_{d6}$) δ [ppm] 144.4, 143.1, 141.6, 121.1, 52.1, 27.3, 24.6; MS (70 eV) m/z 561 [M-Na]$^-$ (neg.), 607 [M+Na]$^+$ (pos.).

General procedure for the synthesis of sulfonated Imidazolinium salts

The respective sulfonated diamine (1 eq) and NH_4Cl (1 eq) were weight in a round-bottom flask equipped with a reflux condenser. The solids were suspended in a mixture of EtOH (10 mL/mmol) and $HC(OEt)_3$ (10 eq) containing one drop of HCOOH. The reaction mixture was stirred at 120 °C overnight. Afterwards the reflux condenser was replaced by a distillation head and the solvent was distilled off. The residue was again suspended in a minimum amount of EtOH and filtered off. Purification was achieved by column chromatography (HP-20) using water-methanol mixtures as eluent.

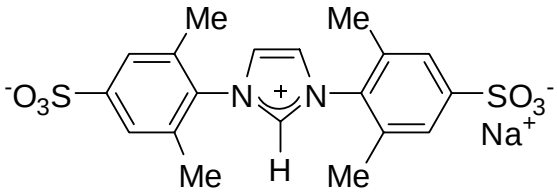


This residue (1.1 g) was purified by reversed phase column-chromatography (stationary phase: HP 20, column: 30 x 5 cm, eluent: pure water, then switched to water:MeOH (10:1)) to afford the product as an off white solid (760 mg, 76 %), R_f = 0.53 (H_2O:MeOH (10:1), RP-18 silica), ^{1}H NMR (500 MHz, DMSO-d_6) δ [ppm] 9.49 (s, 1 H, N-CH-N), 7.58 (s, 4 H, CH, ar), 4.53 (s, 4 H, NCH_2-CH_2), 3.12-3.04 (m, 4 H, CH, iPr), 1.34 (d, 3J = 6.7 Hz, 12 H, CH_3), 1.19 (d, ^{3}J = 6.8 Hz, 12 H, CH_3); ^{13}C{^{1}H} NMR (125.8 MHz, DMSO-d_6) δ [ppm] 159.7, 150.2, 145.2, 129.2, 121.2, 53.1, 27.7, 24.4, 22.8.



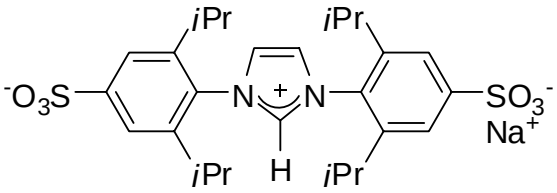
This residue (900 mg) was purified by reversed phase column-chromatography (stationary phase: HP 20, column: 30 x 5 cm, eluent : pure water, then switched to water : MeOH (10:1)) to afford the product as an off white solid (500 mg, 63 %), R_f = 0.50 (H_2O, RP_{18} silica), ^{1}H NMR (500 MHz, DMSO-d_6) δ [ppm] 9.05 (s, 1 H, N-C*H*-N), 7.47 (s, 4 H, C*H*, ar), 4.48 (s, 4 H, N-CH_2-CH_2), 2.40 (s, 12 H, CH_3); ^{13}C{^{1}H} NMR (125.8 MHz, DMSO-d_6) δ [ppm] 160.6, 150.0, 135.7, 133.5, 126.4, 51.2, 17.7; MS (70 eV) m/z 437 [M-Na]$^-$ (neg.).

Procedures for the synthesis of the Imidazolium salts



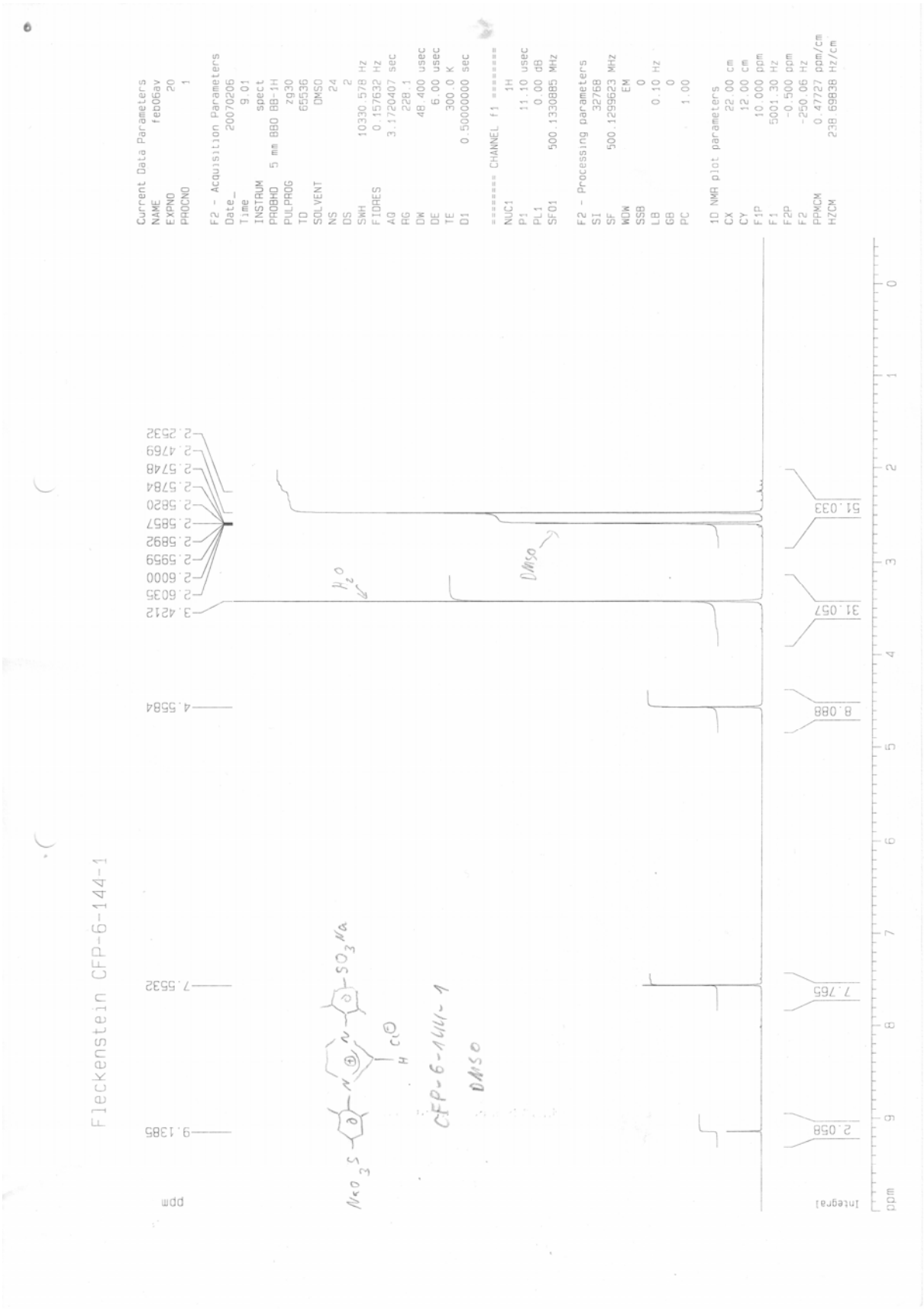
In a 25 mL Schlenk tube the tetramethyldiimine (1.22 g, 2.6 mmol) was suspended in DMSO (13 mL) and chloromethylpivalate (577 mg, 3.8 mmol) was added. The reaction mixture was stirred at 45 °C for 3 days. Then the DMSO was removed in vacuo to leave a brownish solid. This residue was purified by reversed phase column-chromatography (stationary phase: HP 20, column: 30 x 5 cm, eluent: pure water, then switched to water : MeOH (40:1)) to afford

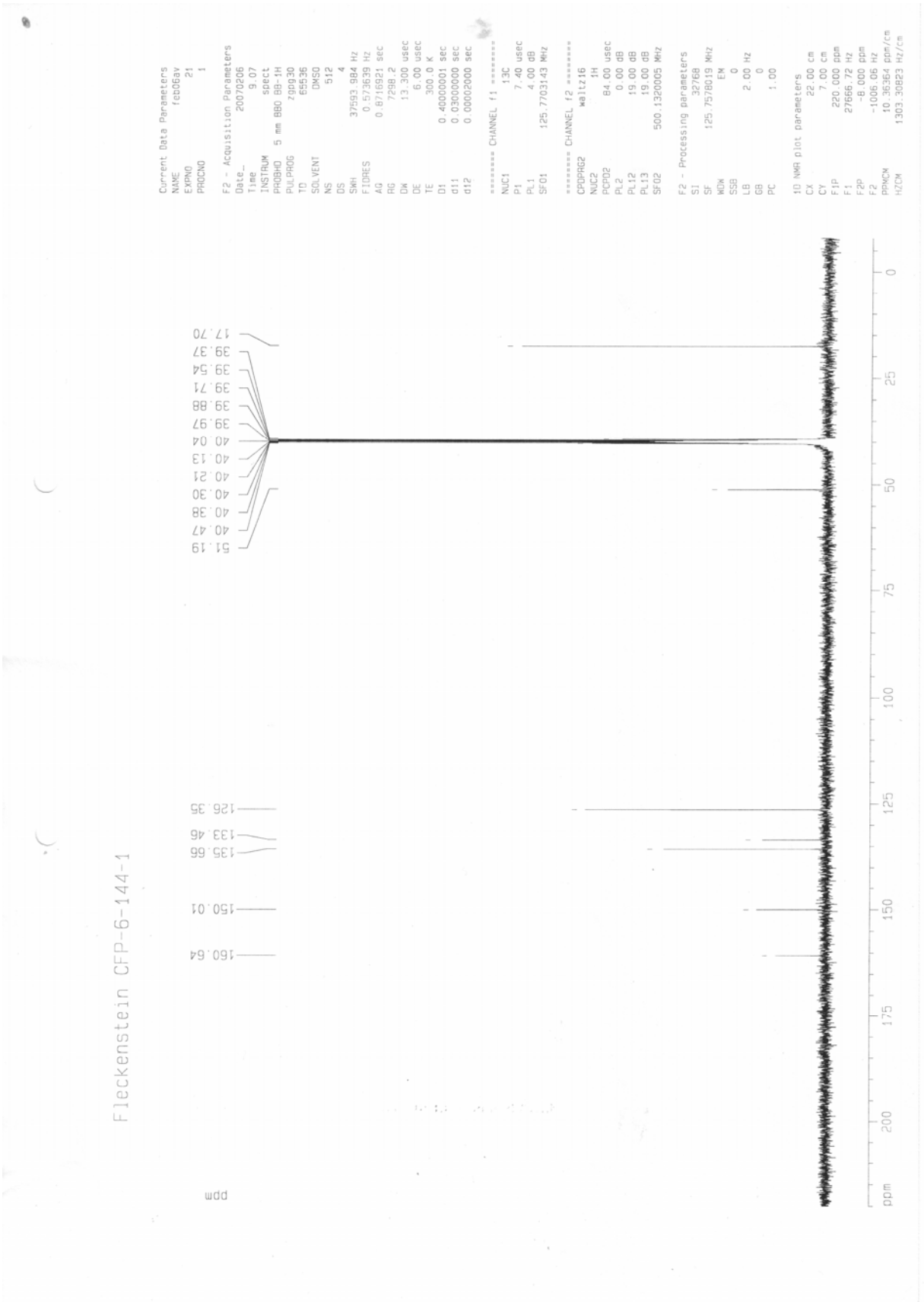
the product as an off white solid (1.10 g, 92 %), R_f = 0.55 (H_2O, RP_{18} silica), ^{1}H NMR (500 MHz, $DMSO_{d6}$) δ [ppm] 9.73 (t, ^4J = 1.5 Hz, 1 H, N-C*H*-N), 8.32 (d, ^4J = 1.5 Hz, 2 H, N-C*H*-C*H*), 7.57 (s, 4 H, C*H*, ar), 2.17 (s, 12 H, CH_3); ^{13}C{^{1}H} NMR (125.8 MHz, $DMSO_{d6}$) δ [ppm] 150.8, 138.9, 134.6, 133.4, 126.2, 125.0, 17.4; MS (70 eV) m/z. 511.2 $[M+Na]^+$ (pos.).



In a 25 mL Schlenk tube the tetraisopropyl-diimine (2.03 g, 3.5 mmol) was suspended in DMSO (13 mL) and chloromethylpivalate (774 mg, 5.14 mmol) was added. The reaction mixture was stirred at 45 °C for 3 days. Then the DMSO was removed in vacuo to leave a brownish solid. This residue was purified by reversed phase column-chromatography (stationary phase: HP 20, column: 30 x 5 cm, eluent: pure water, then switched to water:MeOH (10:1)) to afford the product as an off white solid (1.9 g, 95 %), R_f = 0.50 (H_2O:MeOH (10:1), RP_{18} silica), ^{1}H NMR (500 MHz, $DMSO_{d6}$) δ [ppm] 10.22 (t, ^4J = 1.5 Hz, 1 H, N-C*H*-N), 8.57 (d, ^4J = 1.5 Hz, 2 H, N-C*H*-C*H*), 7.67 (s, 4 H, C*H*, ar), 2.40-2.34 (m, 4 H, C*H*, iPr), 1.26 (d, ^3J = 7.0 Hz, 12 H, CH_3), 1.16 (d, ^3J = 8.5 Hz, 12 H, CH_3); ^{13}C{^{1}H} NMR (125.8 MHz, $DMSO_{d6}$) δ [ppm] 152.6, 145.5, 140.5, 130.9, 127.3, 122.5, 29.7, 25.1, 24.1; MS (70 eV) m/z 593 $[M+Na]^+$ (pos.), 547 $[M-Na]^-$ (neg.).

Scanned NMR of spectra of the newly synthesized compounds

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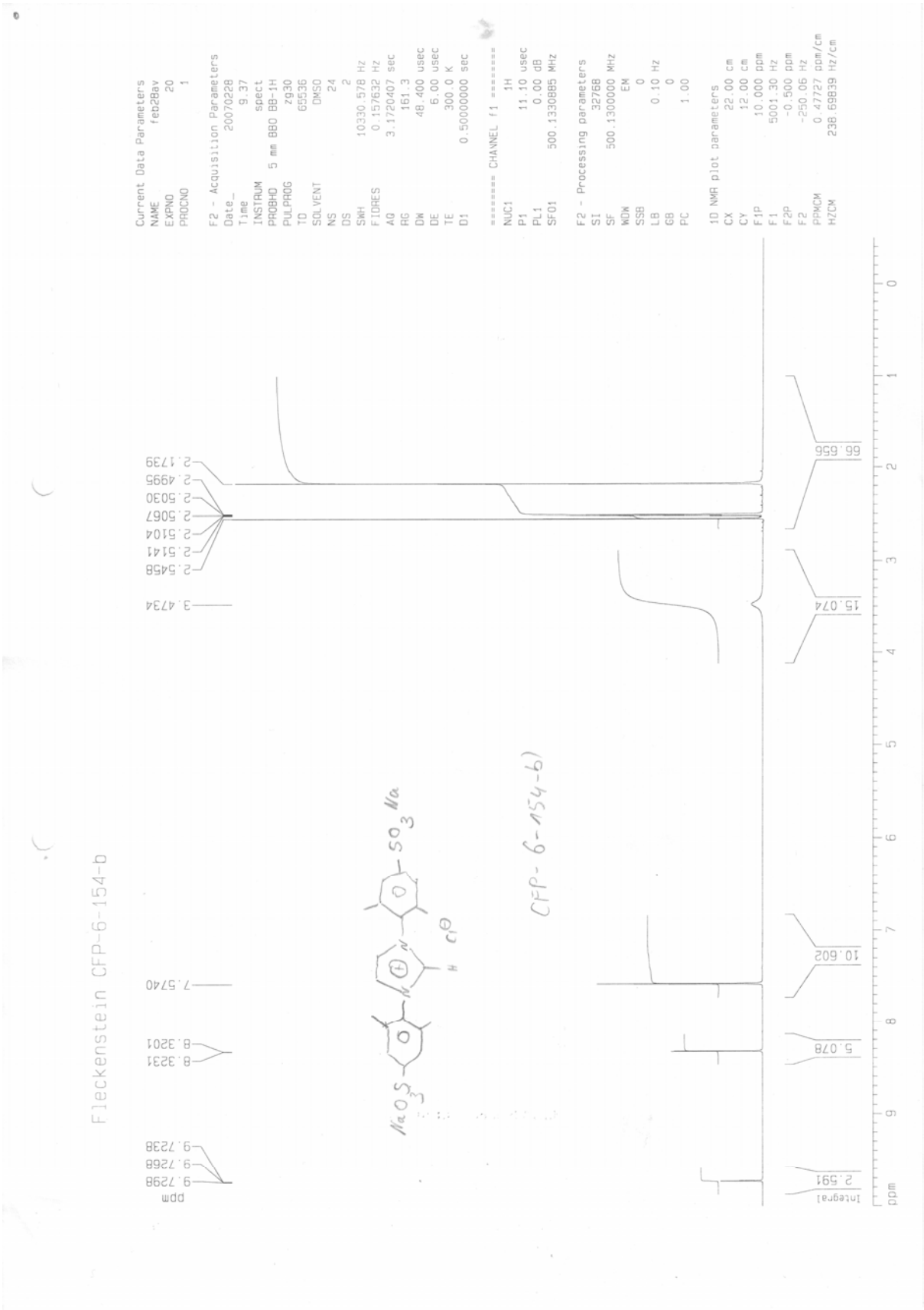
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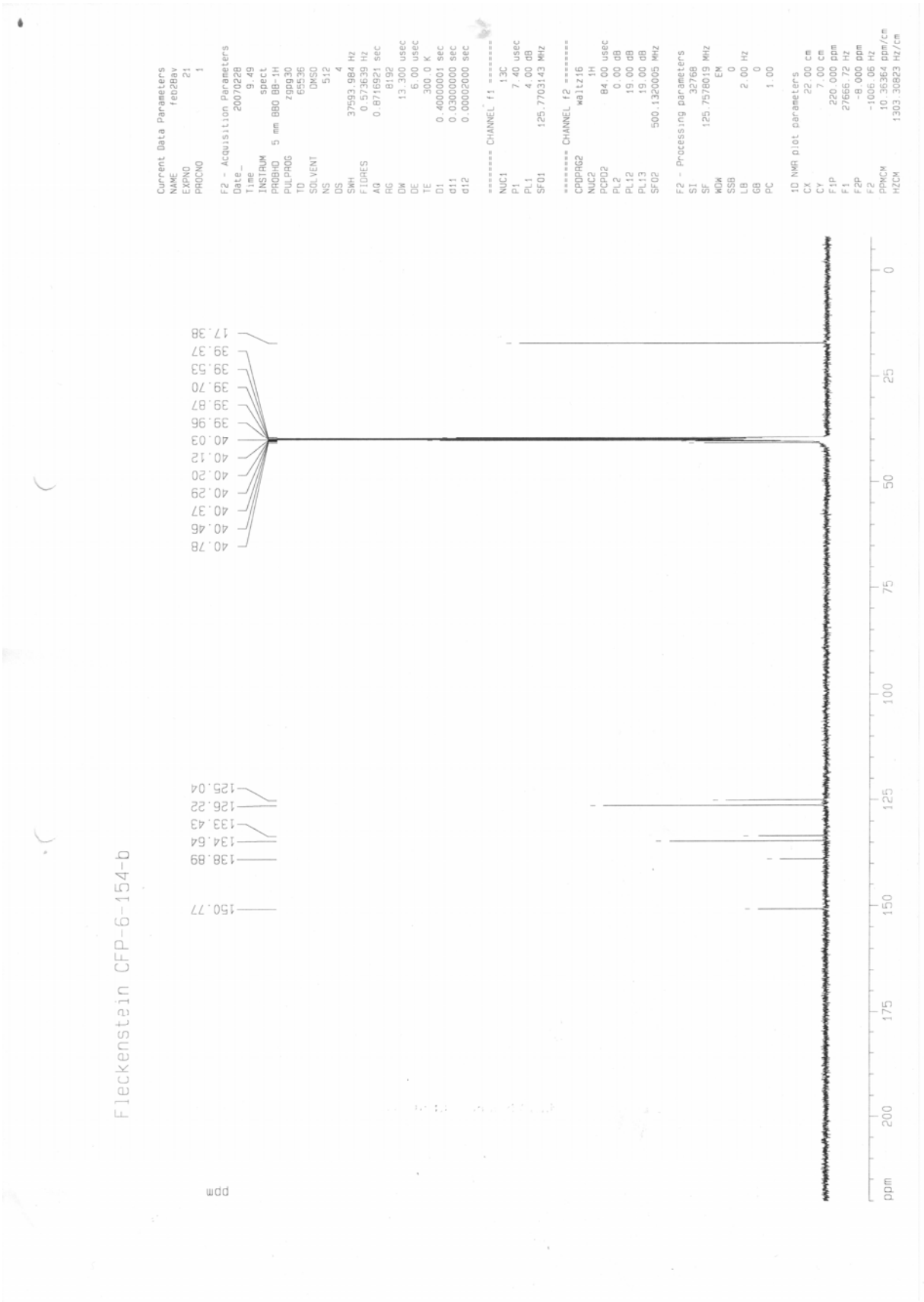
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SSB 0
LB 2.00 Hz
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1D NMR plot parameters
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CY 7.00 cm
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F2P -8.000 ppm
F2 -1006.06 Hz
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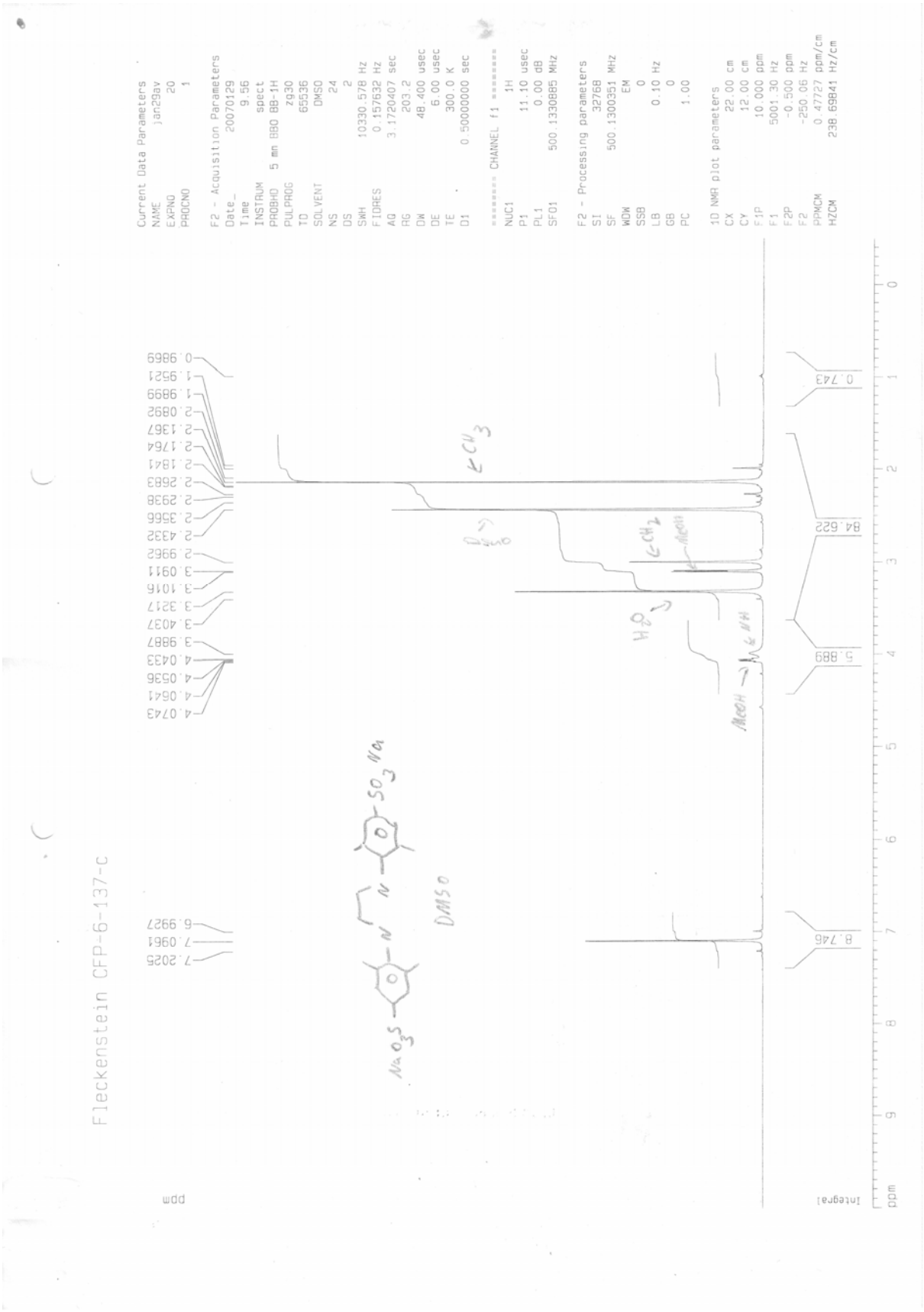
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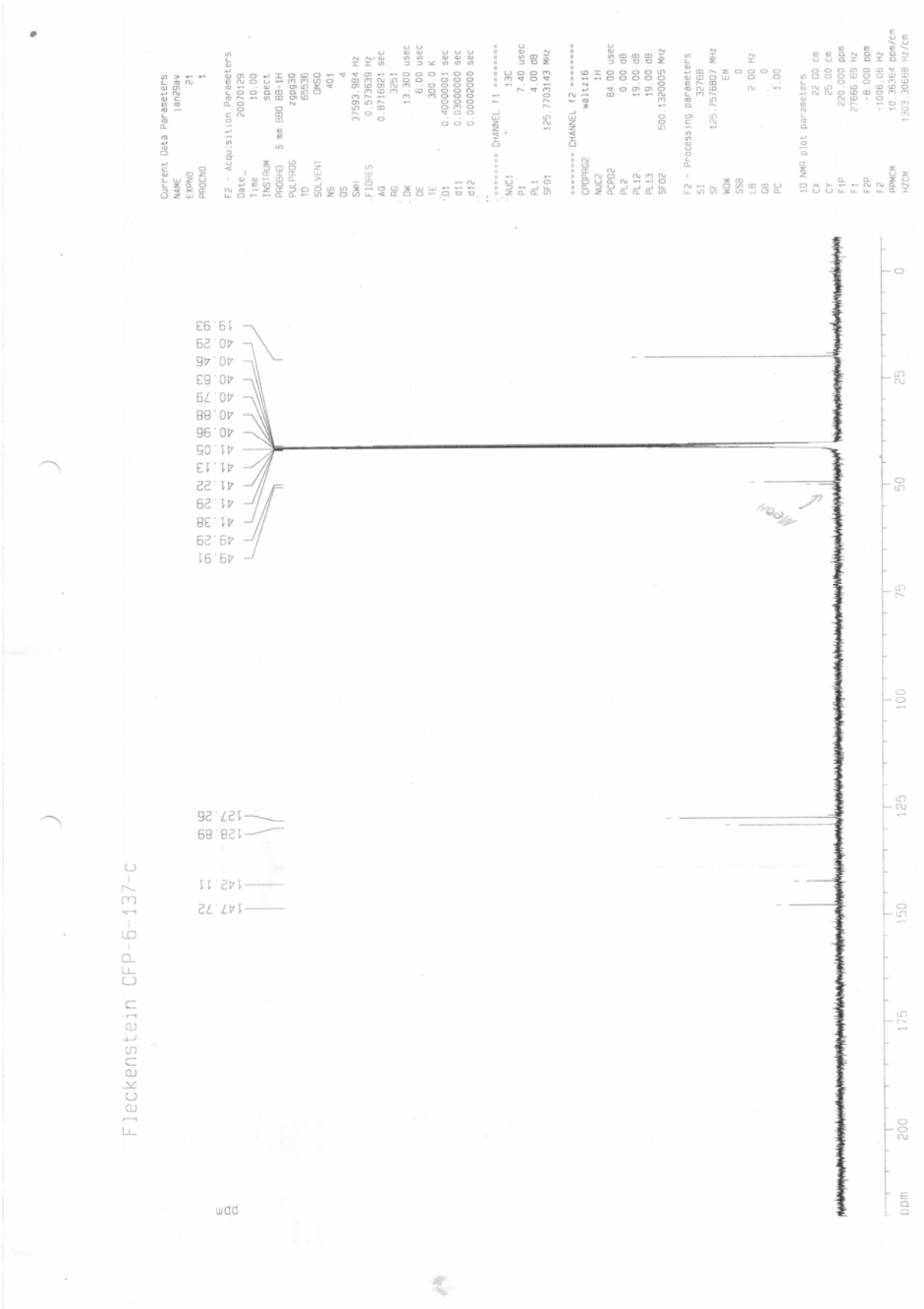
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PL13 19.00 dB
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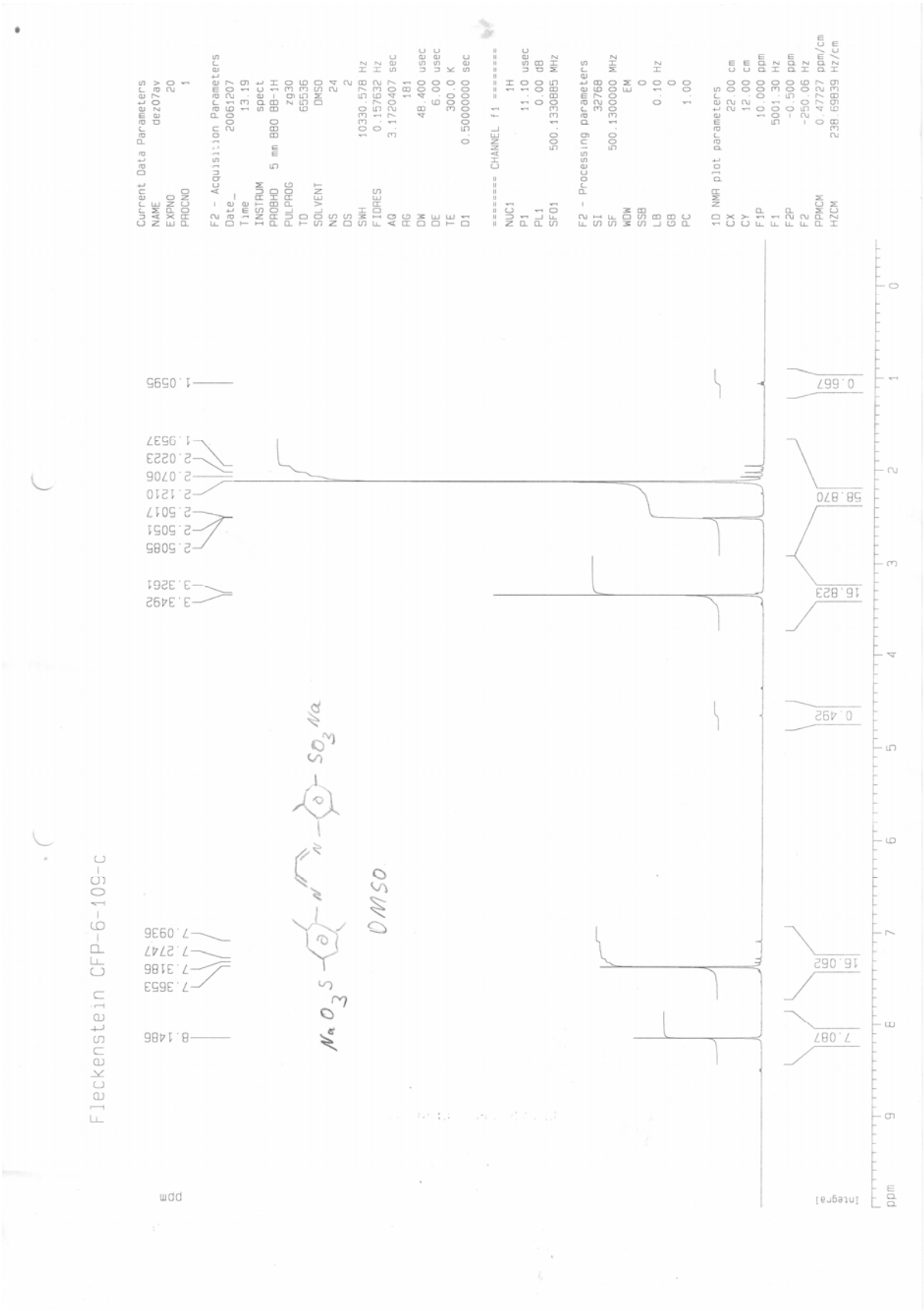
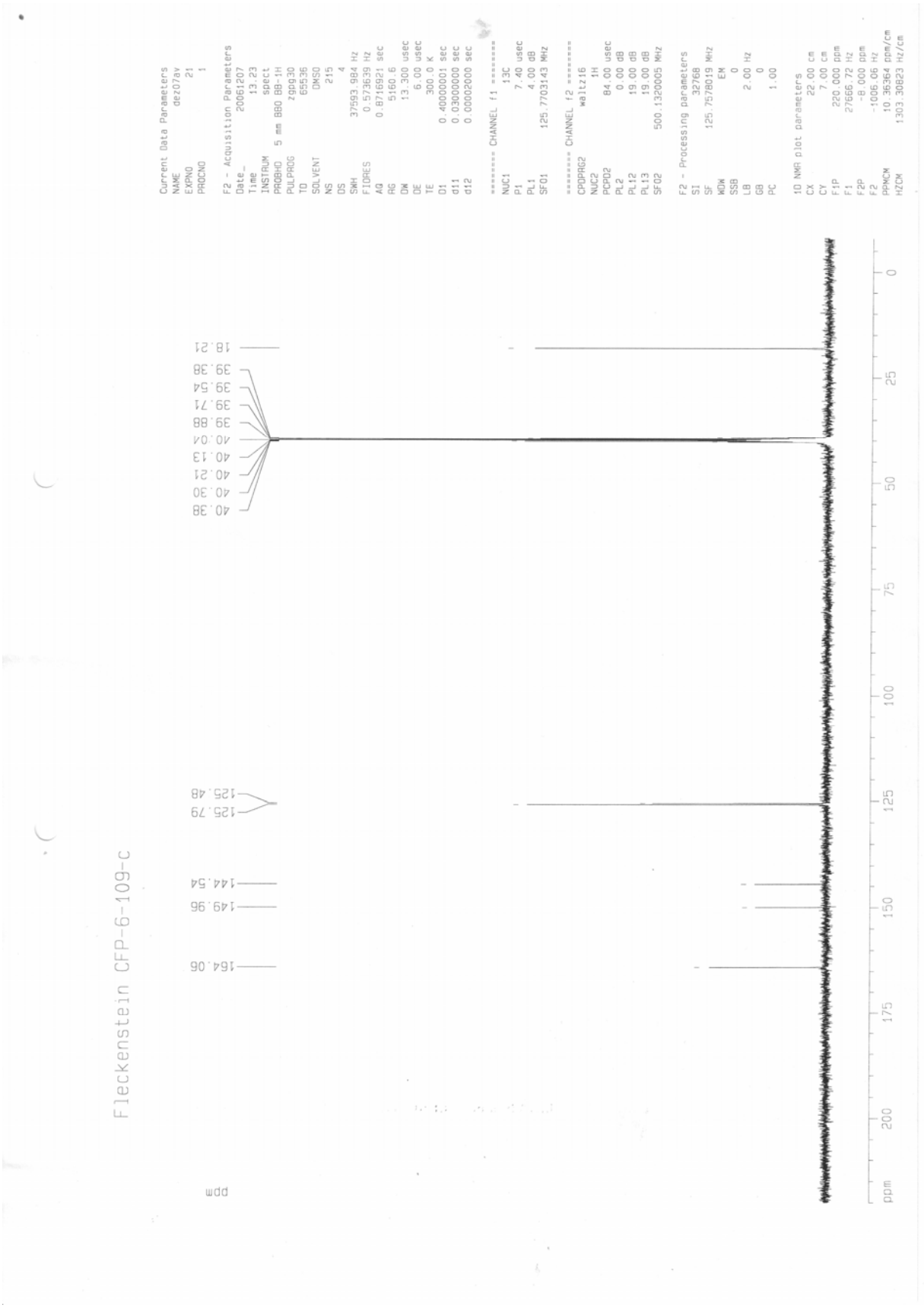
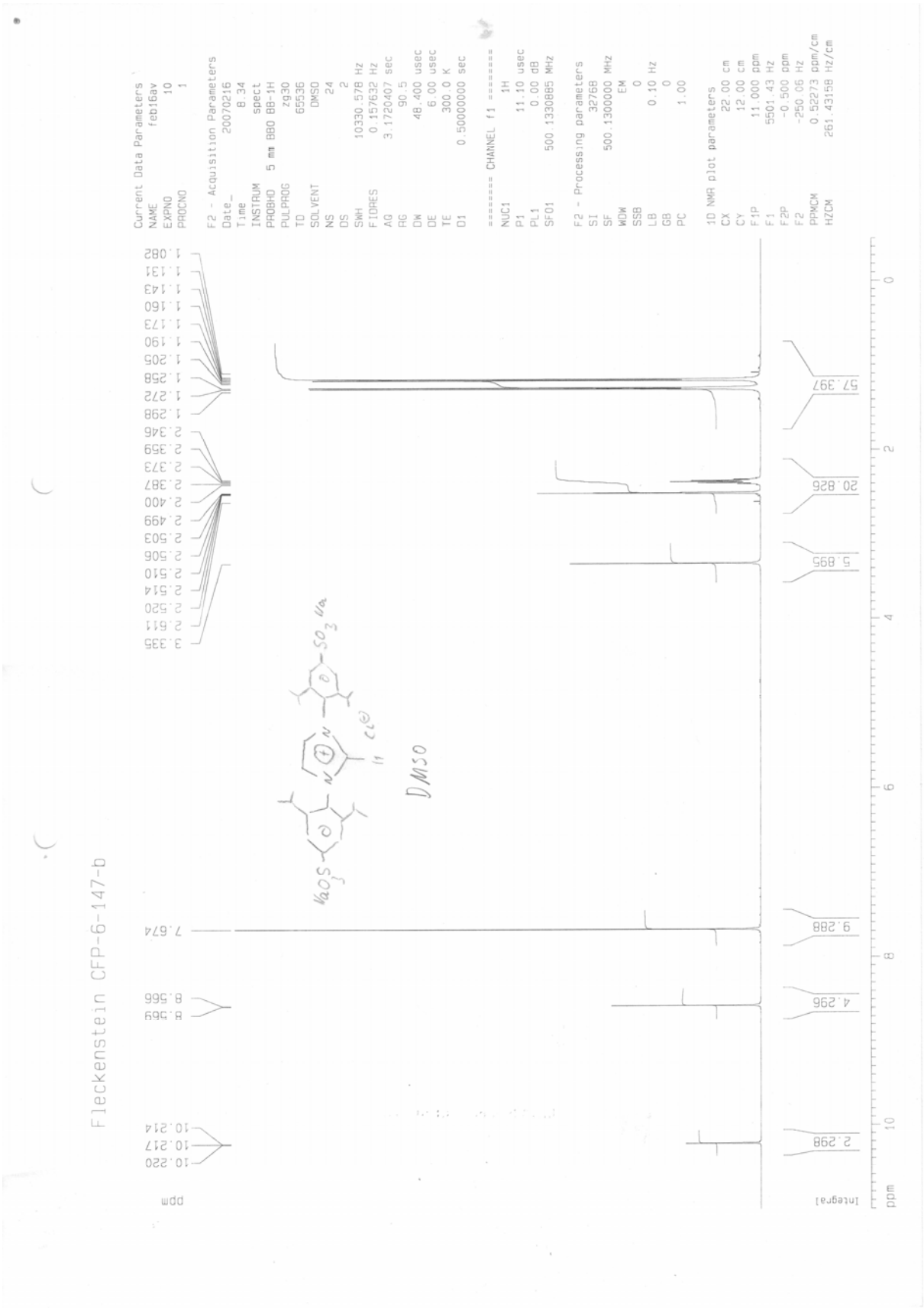
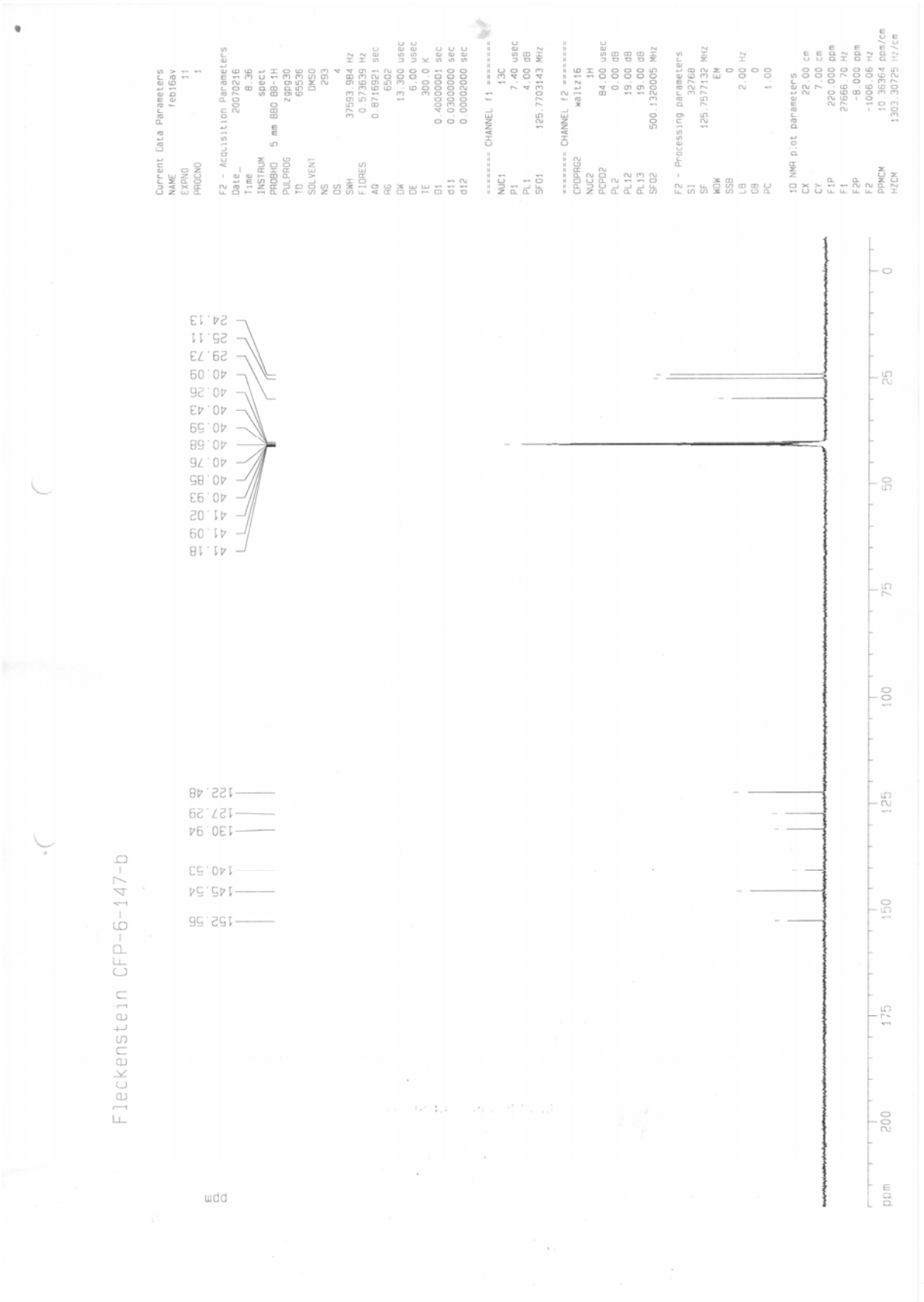
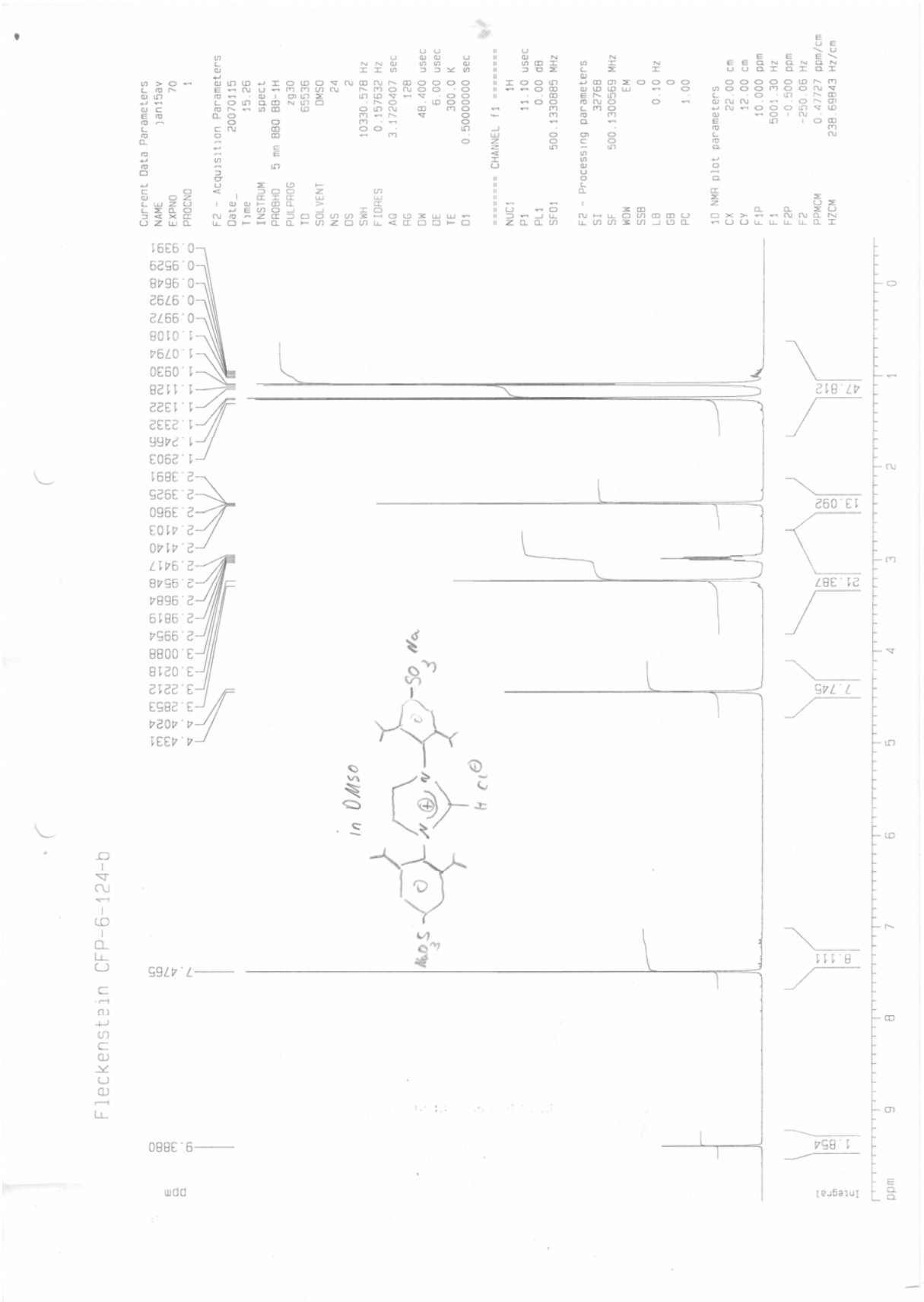
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30688 Hz/cm

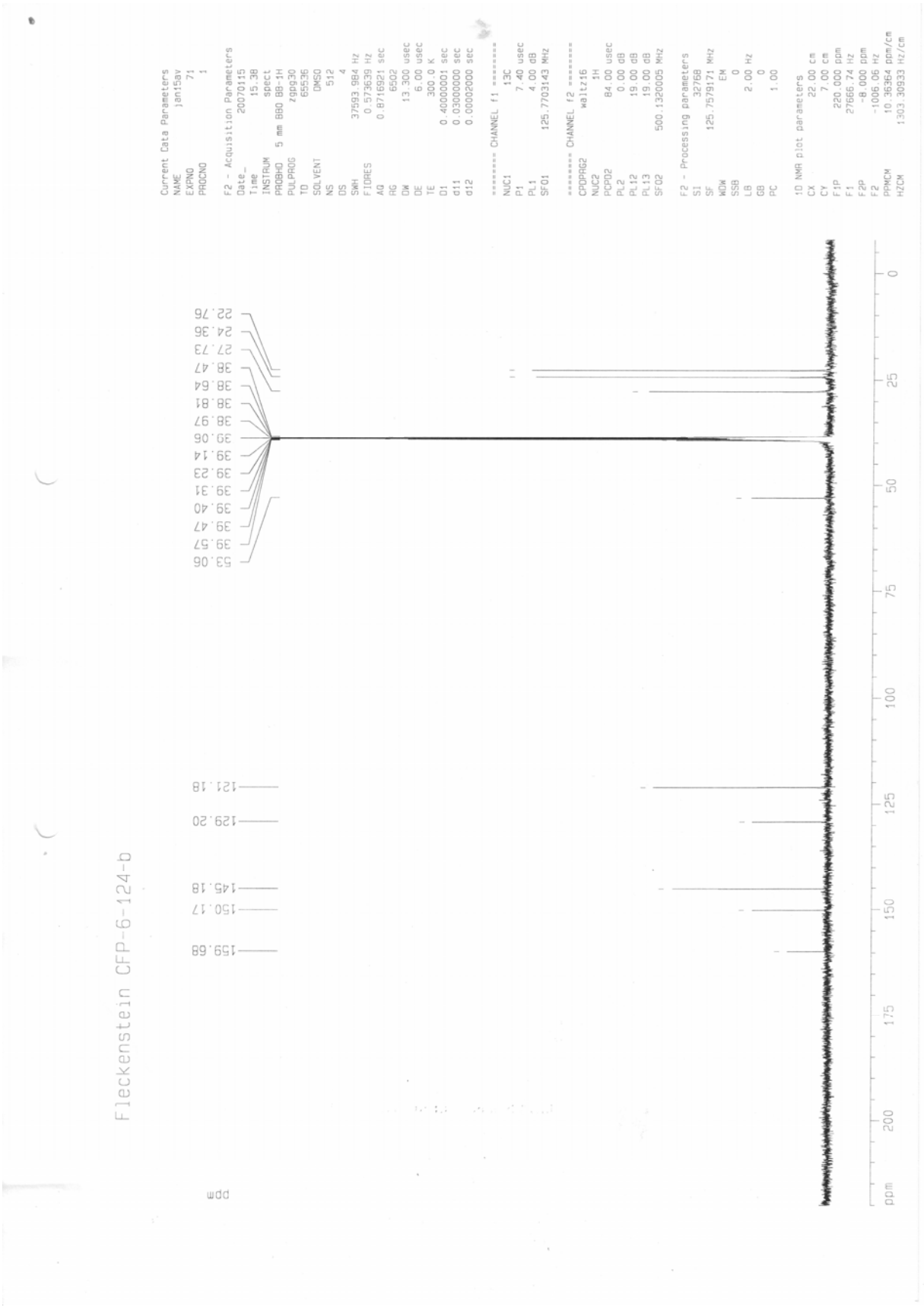

Fleckenstein CFP-6-109-c
Current Data Parameters
NAME dez07av
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061207
Time 13.19
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 238.69839 Hz/cm
1.0595
1.9537
2.0223
2.0706
2.1210
2.5017
2.5051
2.5085
3.3261
3.3492
7.0936
7.2747
7.3186
7.3653
8.1486
0.667
58.870
16.823
0.492
16.062
7.087
DMSO
ppm
Integral

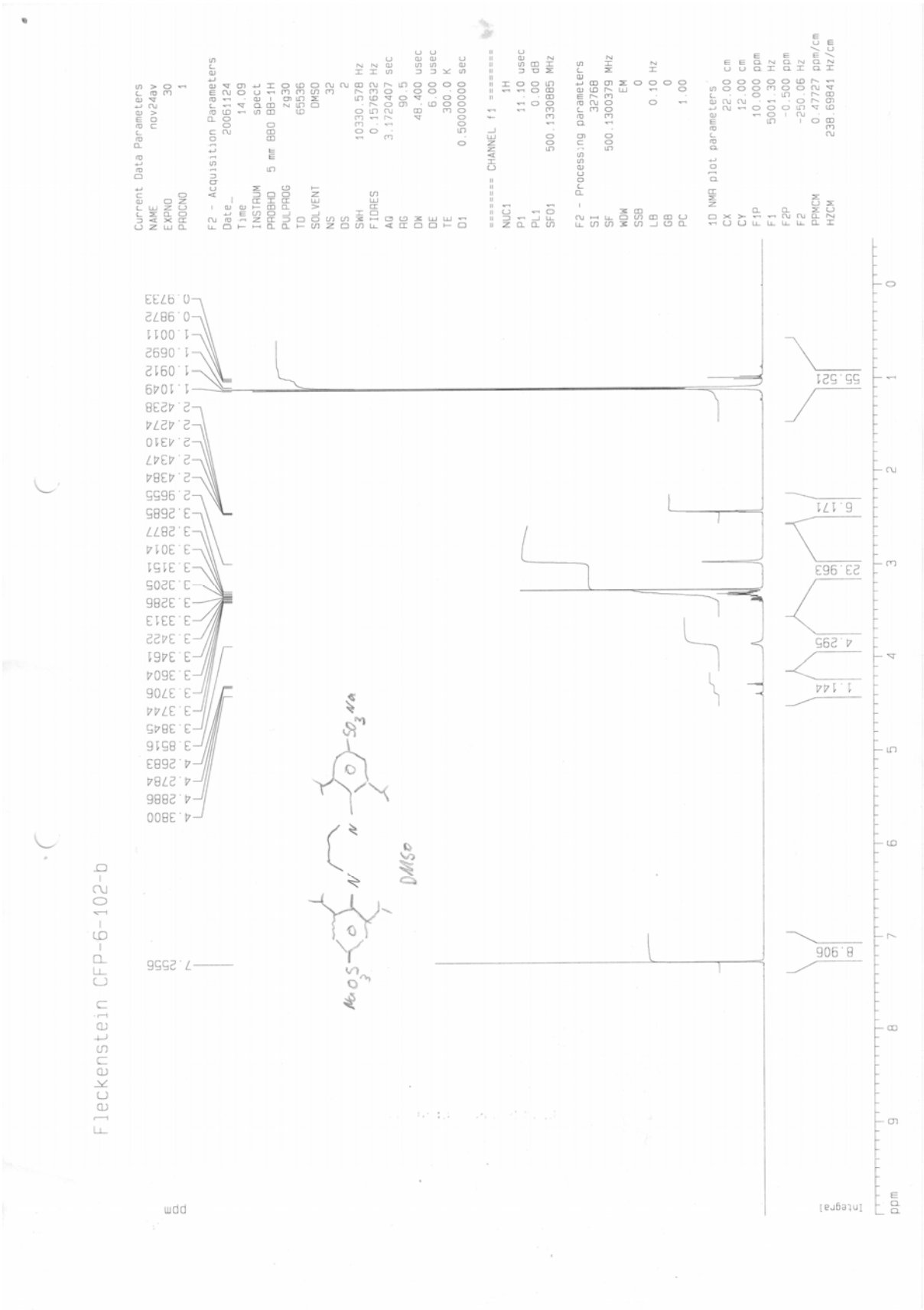

Fleckenstein CFP-6-109-c
Current Data Parameters
NAME dez07av
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061207
Time 13.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 215
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 5160.6
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SF01 125.7703143 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7578019 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.00 cm
F1P 220.000 ppm
F1 27666.72 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30823 Hz/cm
18.21
39.38
39.54
39.71
39.88
40.04
40.13
40.21
40.30
40.38
125.48
125.79
144.54
149.96
164.06
ppm
0
25
50
75
100
125
150
175
200
ppm

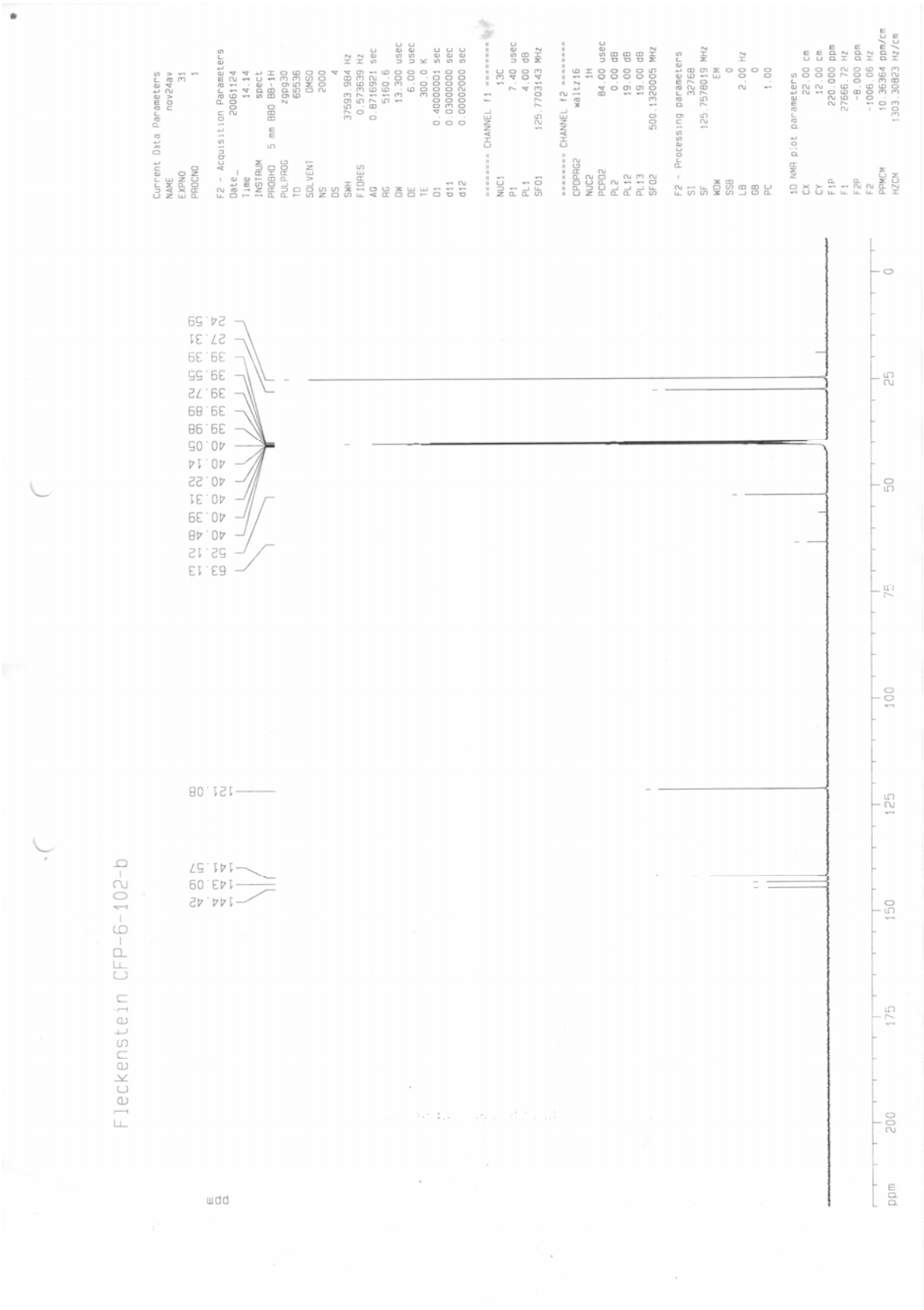

Fleckenstein CFP-6-147-b
DMSO
Current Data Parameters
NAME feb16av
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070216
Time 8.34
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SF01 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 11.000 ppm
F1 5501.43 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.52273 ppm/cm
HZCM 261.43158 Hz/cm
ppm
Integral

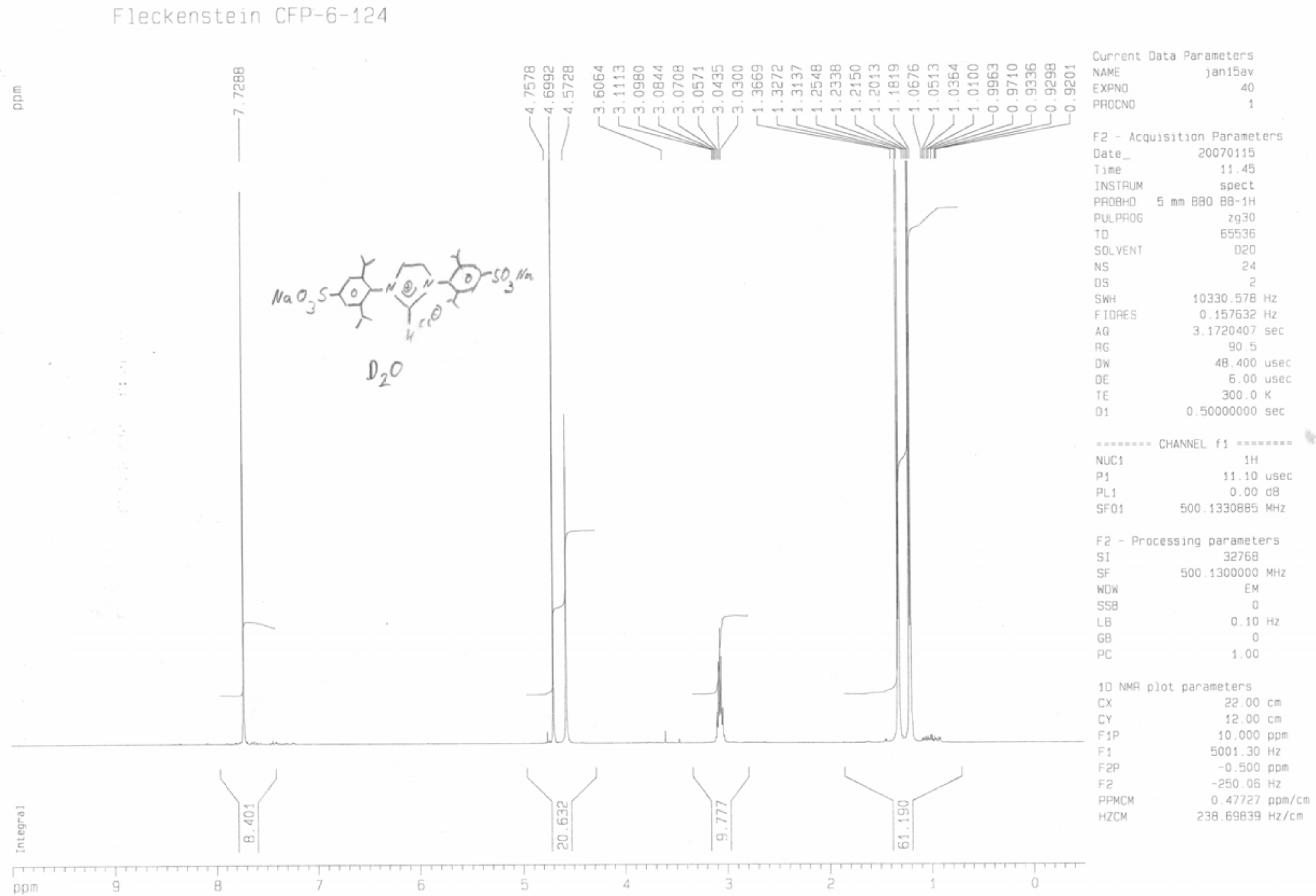

Fleckenstein CFP-6-147-b
Current Data Parameters
NAME feb16av
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070216
Time 8.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 293
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 6502
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======= CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SFO1 125.7703143 MHz
======= CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577132 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.00 cm
F1P 220.000 ppm
F1 27666.70 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30725 Hz/cm
41.18
41.09
41.02
40.93
40.85
40.76
40.68
40.59
40.43
40.26
40.09
29.73
25.11
24.13
152.56
145.54
140.53
130.94
127.29
122.48
ppm
200
175
150
125
100
75
50
25
0

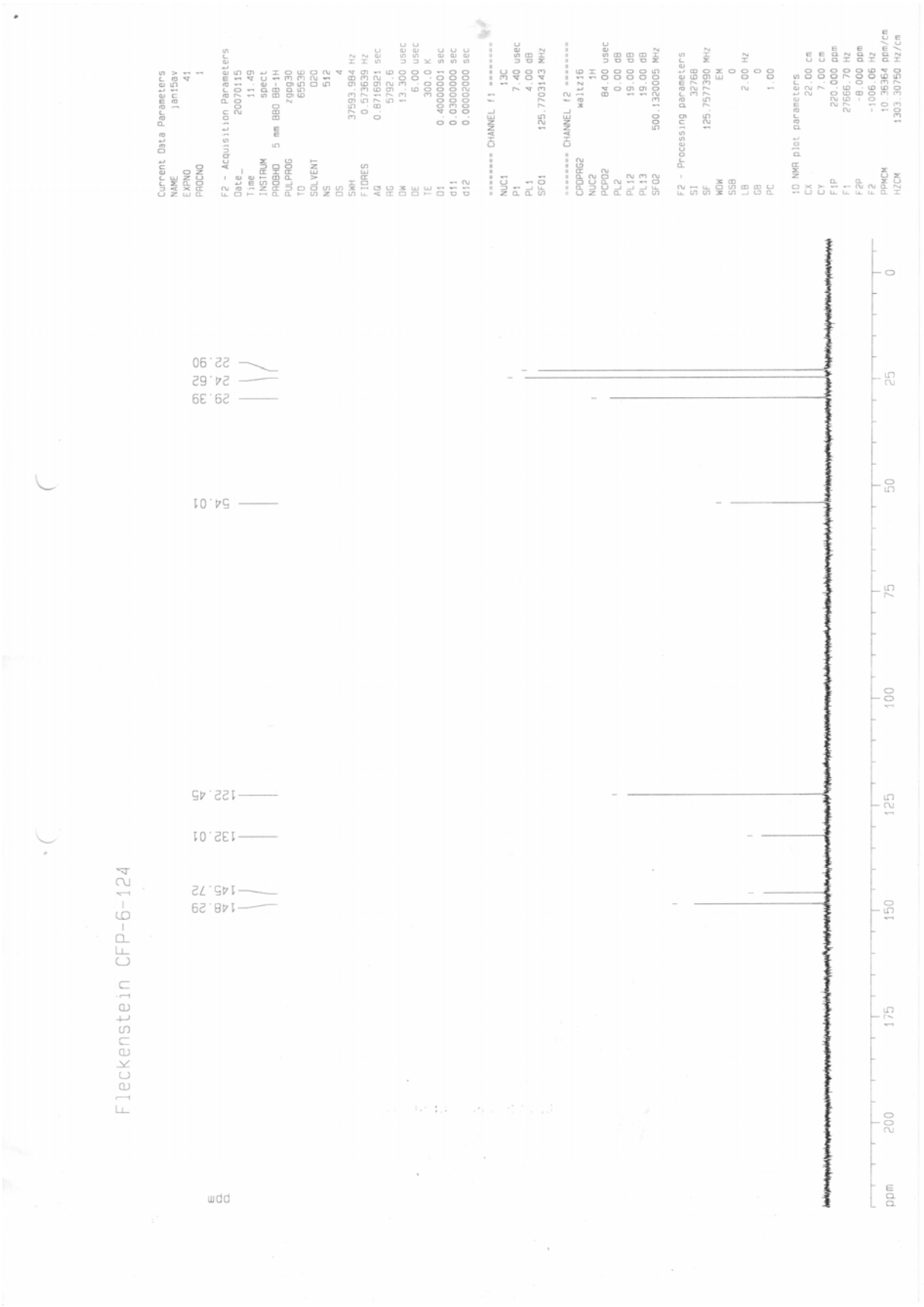

Fleckenstein CFP-6-124-b
in DMSO
Current Data Parameters
NAME jan15av
EXPNO 70
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070115
Time 15.26
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 24
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300569 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 238.69843 Hz/cm
ppm
Integral

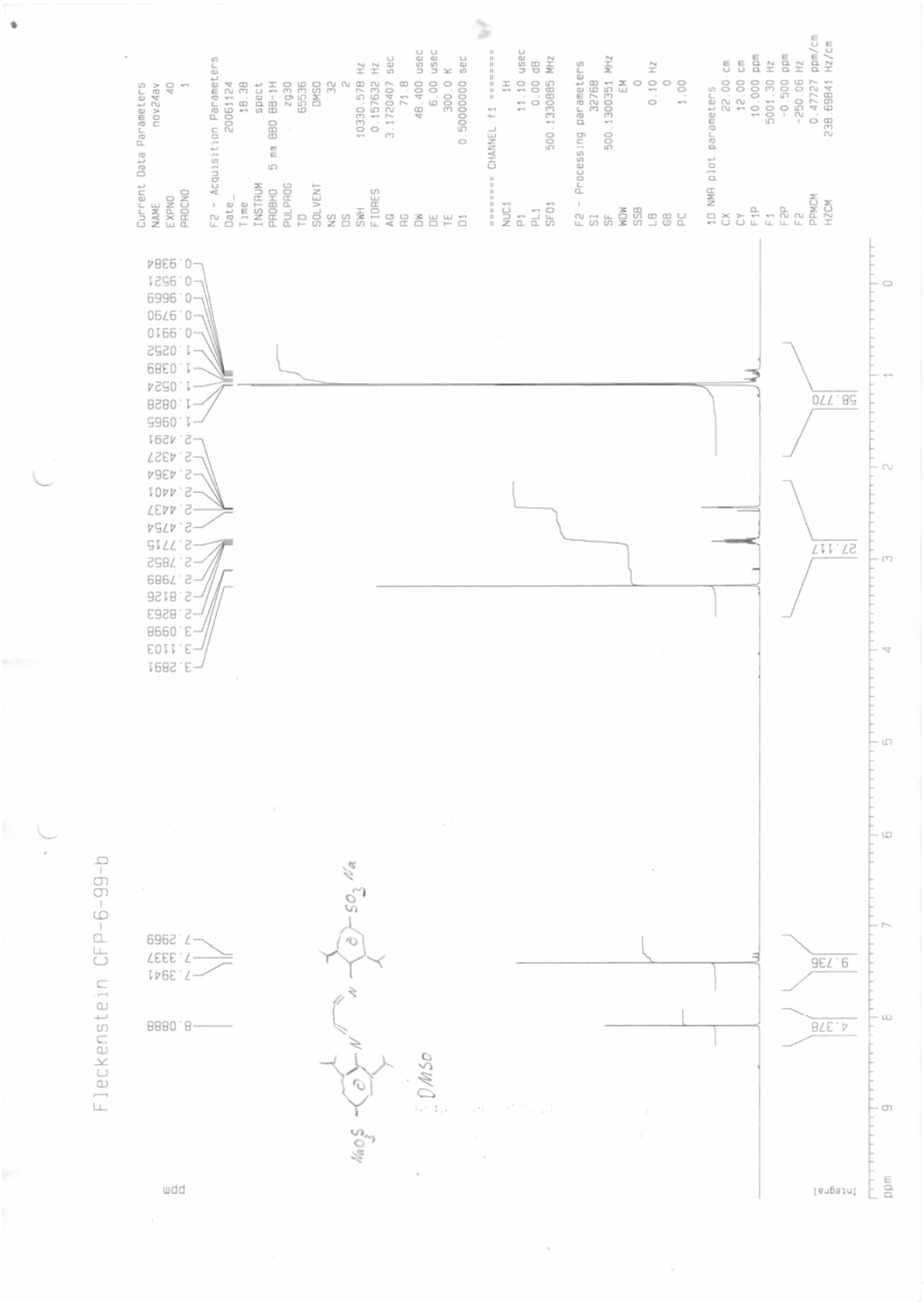

Fleckenstein CFP-6-124-b
ppm
Current Data Parameters
NAME jan15ay
EXPNO 71
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070115
Time 15.38
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 6502
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SFO1 125.7703143 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7579171 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.00 cm
F1P 220.000 ppm
F1 27666.74 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30933 Hz/cm
53.06
39.57
39.47
39.40
39.31
39.23
39.14
39.06
38.97
38.81
38.64
38.47
27.73
24.36
22.76
159.68
150.17
145.18
129.20
121.18
ppm 200 175 150 125 100 75 50 25 0

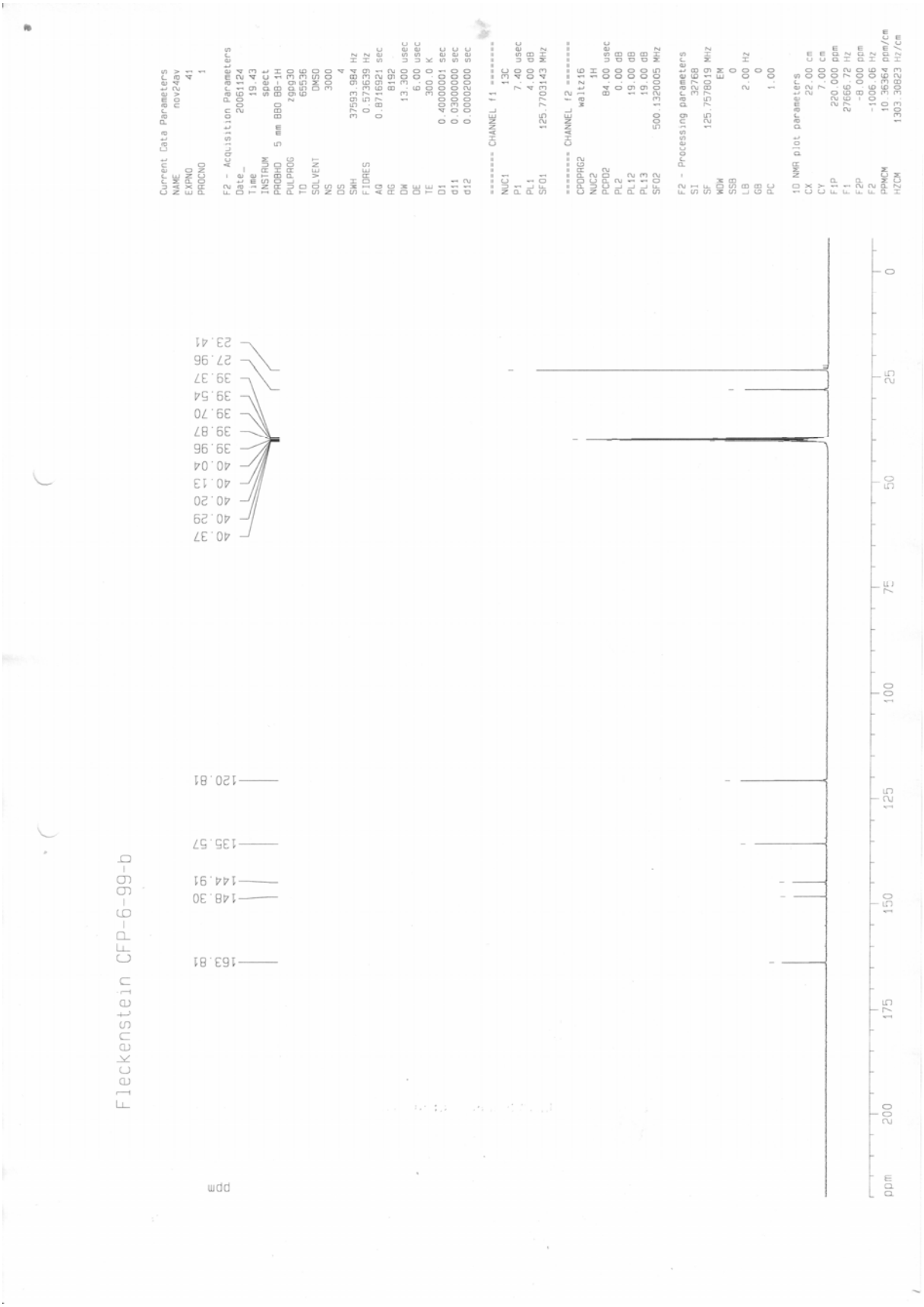

Fleckenstein CFP-6-102-b
Current Data Parameters
NAME nov24av
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061124
Time 14.09
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300379 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 238.69841 Hz/cm
DMSO
ppm
Integral


Fleckenstein CFP-6-102-b
Current Data Parameters
NAME nov24av
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061124
Time 14.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2000
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 5160.6
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SF01 125.7703143 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7578019 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 220.000 ppm
F1 27666.72 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30823 Hz/cm
24.59
27.31
39.39
39.55
39.72
39.89
39.98
40.05
40.14
40.22
40.31
40.39
40.48
52.12
63.13
121.08
141.57
143.09
144.42
ppm
200
175
150
125
100
75
50
25
0


Fleckenstein CFP-6-124
7.7288
4.7578
4.6992
4.5728
3.6064
3.1113
3.0980
3.0844
3.0708
3.0571
3.0435
3.0300
1.3669
1.3272
1.3137
1.2548
1.2338
1.2150
1.2013
1.1819
1.0676
1.0513
1.0364
1.0100
0.9963
0.9710
0.9336
0.9298
0.9201
D2O
Integral
8.401
20.632
9.777
61.190
ppm 9 8 7 6 5 4 3 2 1 0
Current Data Parameters
NAME jan15av
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070115
Time 11.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT D2O
NS 24
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 238.69839 Hz/cm


Fleckenstein CFP-6-124
Current Data Parameters
NAME jan15av
EXPNO 41
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070115
Time 11.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 512
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 5792.6
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SFO1 125.7703143 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577390 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.00 cm
F1P 220.000 ppm
F1 27666.70 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30750 Hz/cm
22.90
24.62
29.39
54.01
122.45
132.01
145.72
148.29
ppm
200
175
150
125
100
75
50
25
0


Fleckenstein CFP-6-99-b
Current Data Parameters
NAME nov24av
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061124
Time 18.38
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.10 usec
PL1 0.00 dB
SF01 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300351 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.06 Hz
PPMCM 0.47727 ppm/cm
HZCM 238.69841 Hz/cm
0.9384
0.9521
0.9669
0.9790
0.9910
1.0252
1.0389
1.0524
1.0828
1.0965
2.4291
2.4327
2.4364
2.4401
2.4437
2.4754
2.7715
2.7852
2.7989
2.8126
2.8263
3.0998
3.1103
3.2891
7.2969
7.3337
7.3941
8.0888
58.770
27.117
9.736
4.378
DMSO
ppm
Integral


Fleckenstein CFP-6-99-b
Current Data Parameters
NAME nov24ay
EXPNO 41
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061124
Time 19.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3000
DS 4
SWH 37593.984 Hz
FIDRES 0.573639 Hz
AQ 0.8716921 sec
RG 8192
DW 13.300 usec
DE 6.00 usec
TE 300.0 K
D1 0.40000001 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.40 usec
PL1 4.00 dB
SF01 125.7703143 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 84.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 19.00 dB
SF02 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7578019 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 22.00 cm
CY 7.00 cm
F1P 220.000 ppm
F1 27666.72 Hz
F2P -8.000 ppm
F2 -1006.06 Hz
PPMCM 10.36364 ppm/cm
HZCM 1303.30823 Hz/cm
23.41
27.96
39.37
39.54
39.70
39.87
39.96
40.04
40.13
40.20
40.29
40.37
120.81
135.57
144.91
148.30
163.81
ppm
200
175
150
125
100
75
50
25
0