Fluoride Protects Boronic Acids in Copper (I)-mediated Click Reaction

Shan Jin,[a] Gaurav Choudhary,[a] Yunfeng Cheng,[a] Chaofeng Dai,[a] Minyong Li,[a] and Binghe Wang*[a]

Experimental Section

General methods and materials

^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz NMR spectrometer in deuterated choloroform ($CDCl_3$), methanol-d_4 (CD_3OD), and DMSO-d_6 ($(CD_3)_2SO$) with either tetramethylsilane (TMS) (0.00 ppm) or the NMR solvent as the internal reference unless otherwise specified. HPLC purification was carried out using a Shimadzu LC-10AT VP system, a Pinnacle C8 reverse phase column (10 mm × 250 mm, No. 9413571, for **B-iii** to **I-iii**) and a YMC-Pack SIL normal phase column (10 mm × 250 mm, No. 102505809, for purification and analysis of **A-iii**). HPLC analytical studies (stability and model reaction yield studies except for **A-iii**) were carried out using a Shimadzu LC-10AT VP system and a Pinnacle C8 reverse phase column (4.6 mm × 250 mm, No. 9413575). Boronic acids were obtained from Frontier Scientific and Combi-Blocks, Inc. Chemicals and solvents were bought from Aldrich or Acros and were used as received. Water used for HPLC studies was doubly distilled and further purified with a Milli-Q filtration system.

HPLC conditions for analytical studies (except A-iii, see synthesis section)

The stabilities and click reaction yields of different boronic acids were studied by HPLC (C8 RP analytical column, 260 nm). 20 μL of 50 μM boronic acid solution (diluted by methanol) was used for each injection. Elution condition: CH_3CN-H_2O, flow rate = 1.0 mL/min), 0-2 min (CH_3CN 5 %), 2-18 min (CH_3CN 5-95 %), 18-25 min (CH_3CN 95-5 %), 25-30 min (CH_3CN 5 %).

Stability studies

0.05 mmol boronic acid (1.0 equiv), 0.01 mmol TBTA ligand (0.2 equiv), and 0.02 mmol CuBr (0.4 equiv) were dissolved in a mixture of H_2O/DMF/*t*-BuOH (0.2 mL/0.6 mL/0.2 mL) in a 3 mL vial [0.10 mmol CsF (2.0 equiv) was added only for the fluoride protected reactions]. The mixture was stirred at room temperature for 5 h. The solution was diluted by methanol (5 mL) and precipitate was filtered out. 5 μL of the reaction mixture (50 mM for each boronic acid) was diluted to 5 mL by methanol. 20 μL of the diluted reaction mixture was used for each injection to the analytical HPLC column. 20 μL of 50 μM pure boronic acid was used in HPLC studies for comparison. The ratio of the peak areas between the reaction mixture and pure boronic acid gives the degradation percentage.

Standard curve

After HPLC purification of triazole product (**iii**), 5-6 vials (20 mL) with different

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concentrations (10-60 μM in methanol) of pure product (**iii**) were prepared. 20 μL product solution was used for each injection to HPLC. Standard curves were generated by plotting peak areas against concentrations. 5-6 data points were generated for each curve.

Reaction yield studies

5 μL of click reaction mixture (50 mM for each boronic acid starting material) was diluted to 5 mL by methanol. 20 μL of the diluted reaction mixture was used for each injection into HPLC. The peak area of the product (**iii**) was used for yield calculations using standard curves.

Synthesis

General procedure for the model click reaction

0.05 mmol azide (1.0 equiv), 0.05 mmol alkyne (1.0 equiv), 0.01 mmol TBTA ligand (0.2 equiv), and 0.02 mmol CuBr (0.4 equiv) were dissolved in a mixture of H_2O/DMF/*t*-BuOH (0.2 mL/0.6 mL/0.2 mL) in a 3 mL vial. [0.10 mmol CsF (2.0 equiv) was added only for the fluoride protected click reactions] The mixture was stirred at room temperature for 5 h. The solution was diluted by methanol (5 mL) and precipitate was filtered out. The product was purified by HPLC (260 nm, Sil NP semi-preparation column for **A-iii**, C8 RP semi-preparation column for **B-iii** to **I-iii**).

***Tert*-butyl 2-(4-(4-bromophenyl)-1H-1,2,3-triazol-1-yl)acetate (A-iii).** HPLC elution condition: ethyl acetate-hexane, 0-2 min (hexane 90 %), 2-13 min (hexane 90-0 %), 13-13.5 min (hexane 0 %), 13.5-16 min (hexane 0-90 %), flow rate = 2 mL/min, RT = 11.5 min. This gave **A-iii** (79% without fluoride protection, 81% with fluoride protection) as a white solid. The same HPLC condition was used for the analytical studies of **A-iii**. ^{1}H NMR ($CDCl_3$) δ 7.93 (s, 1H), 7.75 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.4 Hz, 1H), 5.13 (s, 2H), 1.53 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 165.2, 147.2, 132.0, 129.5, 127.4, 122.1, 121.0, 84.1, 51.6, 28.0; MS (ESI+), m/z 340.1 $(M+H)^+$.

4-(1-(2-Tert-butoxy-2-oxoethyl)-1H-1,2,3-triazol-4-yl)phenylboronic acid (B-iii). HPLC elution condition: isocratic flow of 30 % ACN in H_2O, flow rate = 2 mL/min, RT = 9 min. This gave **B-iii** (57% without fluoride protection, 96% with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.37 (s, 1H), 7.84 (m, 3H), 7.73 (s, 1H), 5.28 (s, 2H), 1.53 (s, 9H); ^{13}C NMR (CD_3OD) δ 167.5, 135.2, 128.5, 128.2, 128.1, 127.3, 125.6, 123.7, 116.8, 115.4, 84.5, 52.6, 28.2; MS (ESI-), m/z 302.2 $(M-H)^-$, 316.1 $(M-2H+CH_3)^-$.

4-((2-(4-(4-Bromophenyl)-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (C-iii). HPLC elution condition: isocratic flow of 50 % ACN in H_2O, flow rate = 3 mL/min, RT = 8 min. This gave **C-iii** (49 % without fluoride protection, 89% with fluoride protection) as a white solid. ^{1}H NMR (DMSO-d_6) δ 8.62 (d, J = 7.2 Hz, 1H),

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8.01 (t, J = 7.2 Hz, 2H), 7.83 (d, J = 7.6 Hz, 2H), 7.75 (d, J = 7.2 Hz, 2H), 7.67 (t, J = 8.8 Hz, 2H), 7.26 (s, 2H), 5.24 (d, J = 7.6 Hz, 2H), 4.36 (s, 2H); ^{13}C NMR (DMSO-d_6) δ 165.6, 145.0, 134.1, 131.8, 129.9, 127.0, 126.3, 123.2, 120.7, 51.7; MS (ESI-), m/z 415.6 $(M-H)^-$.

4-((4-(4-Bromophenyl)-1H-1,2,3-triazol-1-yl)methyl)phenylboronic acid (D-iii). HPLC elution condition: isocratic flow of 55 % ACN in H_2O, flow rate = 3 mL/min, RT = 8 min. This gave **D-iii** (60 % without fluoride protection, 71 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.37 (s, 1H), 7.75 (m, 3H), 7.59 (m, 3H), 7.36 (m, 2H), 5.67 (s, 2H); ^{13}C NMR (CD_3OD) δ 148.2, 133.2, 130.9, 128.5, 123.1, 122.6, 55.1; MS (ESI+), m/z 359.5 $(M+H)^+$.

4-((2-(4-(4-Ethylphenyl)-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (E-iii). HPLC elution condition: isocratic flow of 50 % ACN in H_2O, flow rate = 3 mL/min, RT = 7.5 min. This gave **E-iii** (79 % without fluoride protection, 94 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.28 (s, 1H), 7.73 (m, 3H), 7.60 (s, 1H), 7.29 (m, 4H), 5.23 (s, 2H), 4.44 (s, 2H), 2.68 (m, J = 6.8 Hz, 2H), 1.27 (m, 3H); ^{13}C NMR (CD_3OD) δ 167.8, 146.0, 129.5, 129.1, 127.8, 126.8, 123.6, 53.3, 44.5, 29.7, 16.11; MS (ESI+), m/z 365.3 $(M+H)^+$.

4-((2-(4-p-Tolyl-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (F-iii). HPLC elution condition: isocratic flow of 50 % ACN in H_2O, flow rate = 3 mL/min, RT = 7 min. This gave **F-iii** (43% without fluoride protection, 77 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.29 (s, 1H), 7.70 (d, J = 8.0 Hz, 3H), 7.58 (s, 1H), 7.26 (m, 4H), 5.23 (s, 2H), 4.58 (s, 1H), 4.43 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (CD_3OD) δ 137.8, 129.2, 126.4, 125.3, 122.1, 51.8, 19.9; MS (ESI-), m/z 349.3 $(M-H)^-$.

4-((2-(4-(4-Fluorophenyl)-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (G-iii). HPLC elution condition: isocratic flow of 40 % ACN in H_2O, flow rate = 3 mL/min, RT = 8.5 min. This gave **G-iii** (47 % without fluoride protection, 83 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.32 (s, 1H), 7.86 (t, J = 8.8 Hz, 2H), 7.72 (s, 1H), 7.59 (s, 1H), 7.29 (s, 2H), 7.18 (t, J = 8.8 Hz, 2H), 5.24 (s, 2H), 4.57 (s, 1H), 4.44 (s, 2H); ^{13}C NMR (CD_3OD) δ 127.4, 127.3, 126.4, 122.4, 115.5, 115.3, 51.8, 29.3; MS (ESI-), m/z 353.3 $(M-H)^-$.

4-((2-(4-(4-Aminophenyl)-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (H-iii). HPLC elution condition: isocratic flow of 25 % ACN in H_2O, flow rate = 3 mL/min, RT = 7.5 min. This gave **H-iii** (57 % without fluoride protection, 86 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.16 (s, 1H), 7.60 (m, 4H), 7.30 (s, 2H), 6.77 (s, 2H), 5.23 (s, 2H), 4.45 (s, 2H); ^{13}C NMR (CD_3OD) δ 163.5, 133.5, 131.3, 129.3, 126.4, 121.0, 115.1, 51.8, 35.6; MS (ESI+), m/z 366.3

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$(M+CH_3)^+$.

4-((2-(4-(4-Boroxophenyl)-1H-1,2,3-triazol-1-yl)acetamido)methyl)phenylboronic acid (I-iii). HPLC elution condition: isocratic flow of 25 % ACN in H_2O, flow rate = 3 mL/min, RT = 7.5 min. This gave **I-iii** (43 % without fluoride protection, 86 % with fluoride protection) as a white solid. ^{1}H NMR (CD_3OD) δ 8.38 (s, 1H), 7.84 (m, 3H), 7.73 (m, 2H), 7.62 (s, 1H), 7.33 (m, 2H), 5.27 (s, 2H), 4.60 (s, 1H), 4.47 (s, 2H); ^{13}C NMR (CD_3OD) δ 167.8, 137.1, 135.6, 127.9, 125.8, 124.1, 53.3, 30.4; MS (ESI+), m/z 395.3 $(M+CH_3)^+$, 409.4 $(M-H+2CH_3)^+$, 409.4 $(M-2H+2CH_3+Na)^+$.

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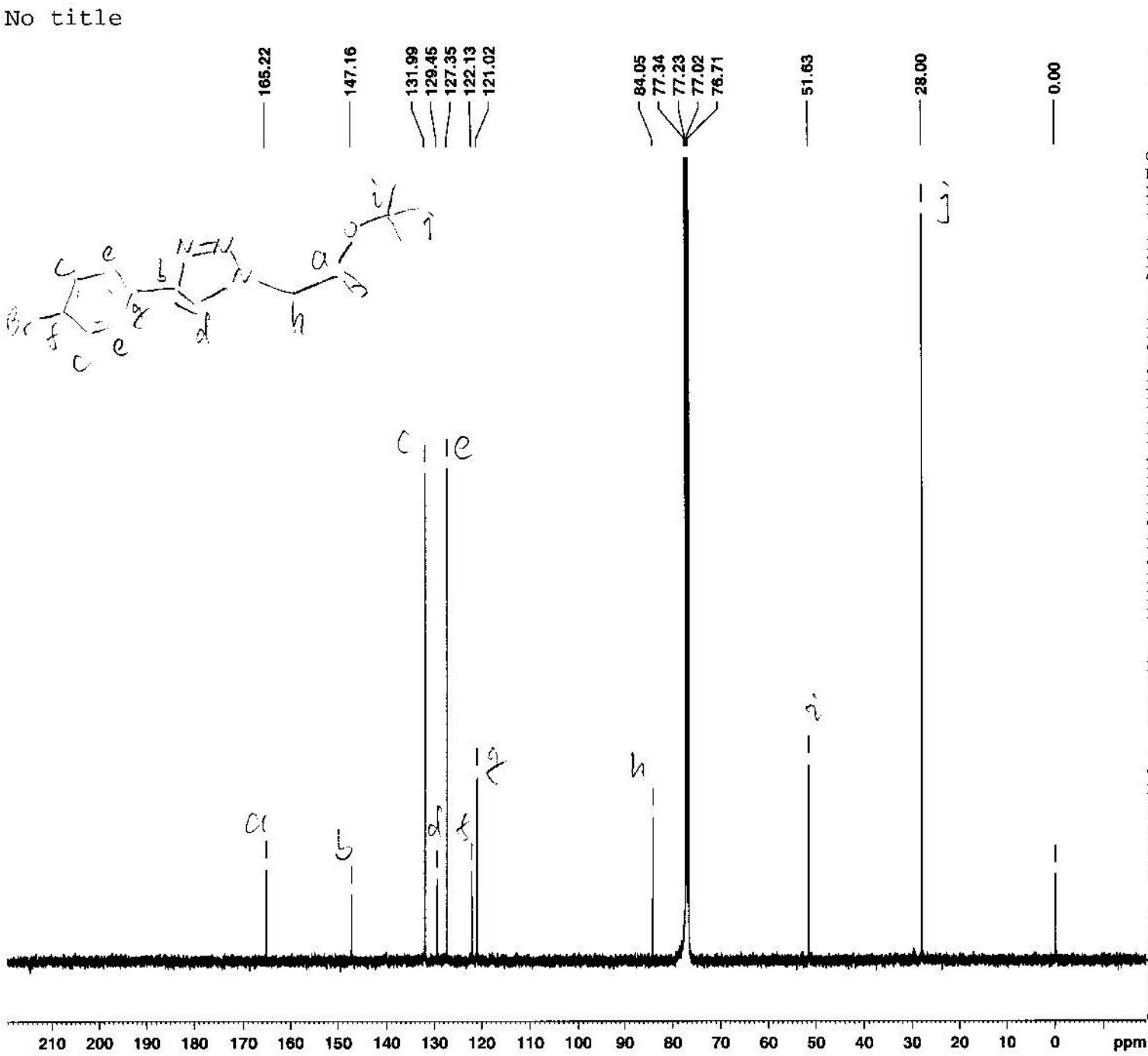

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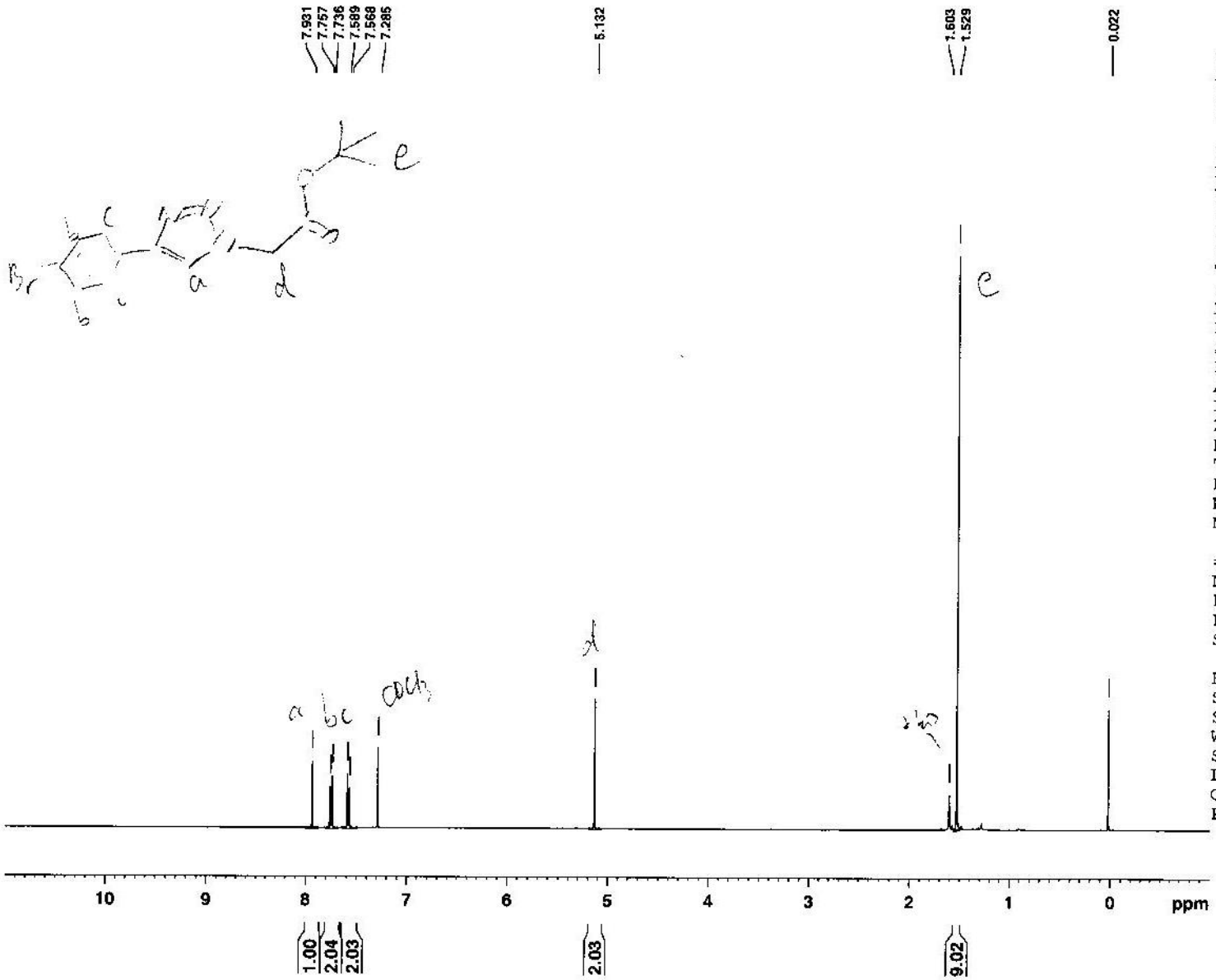

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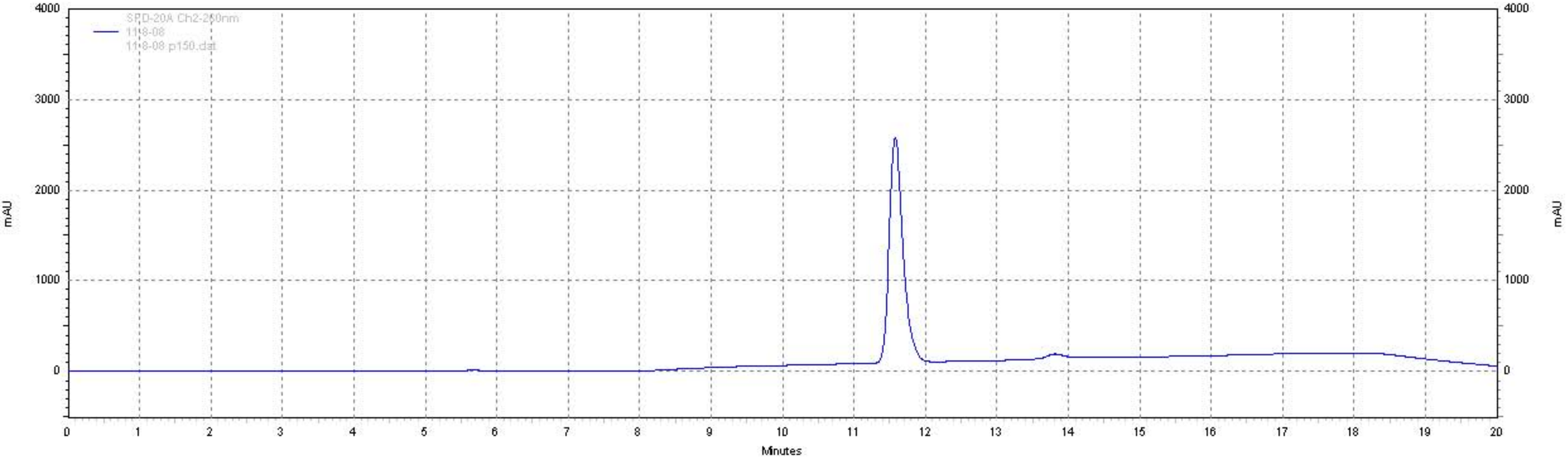

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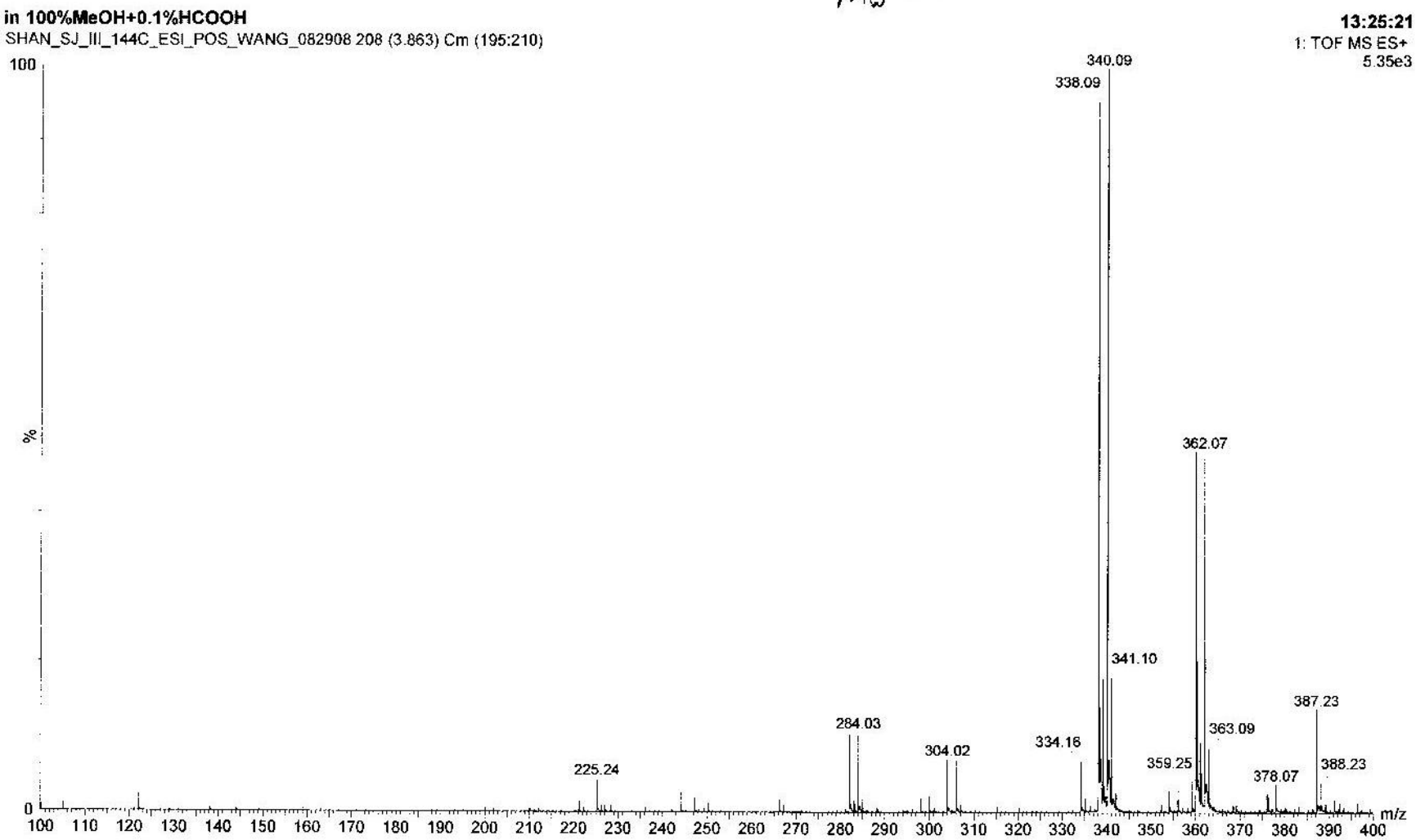

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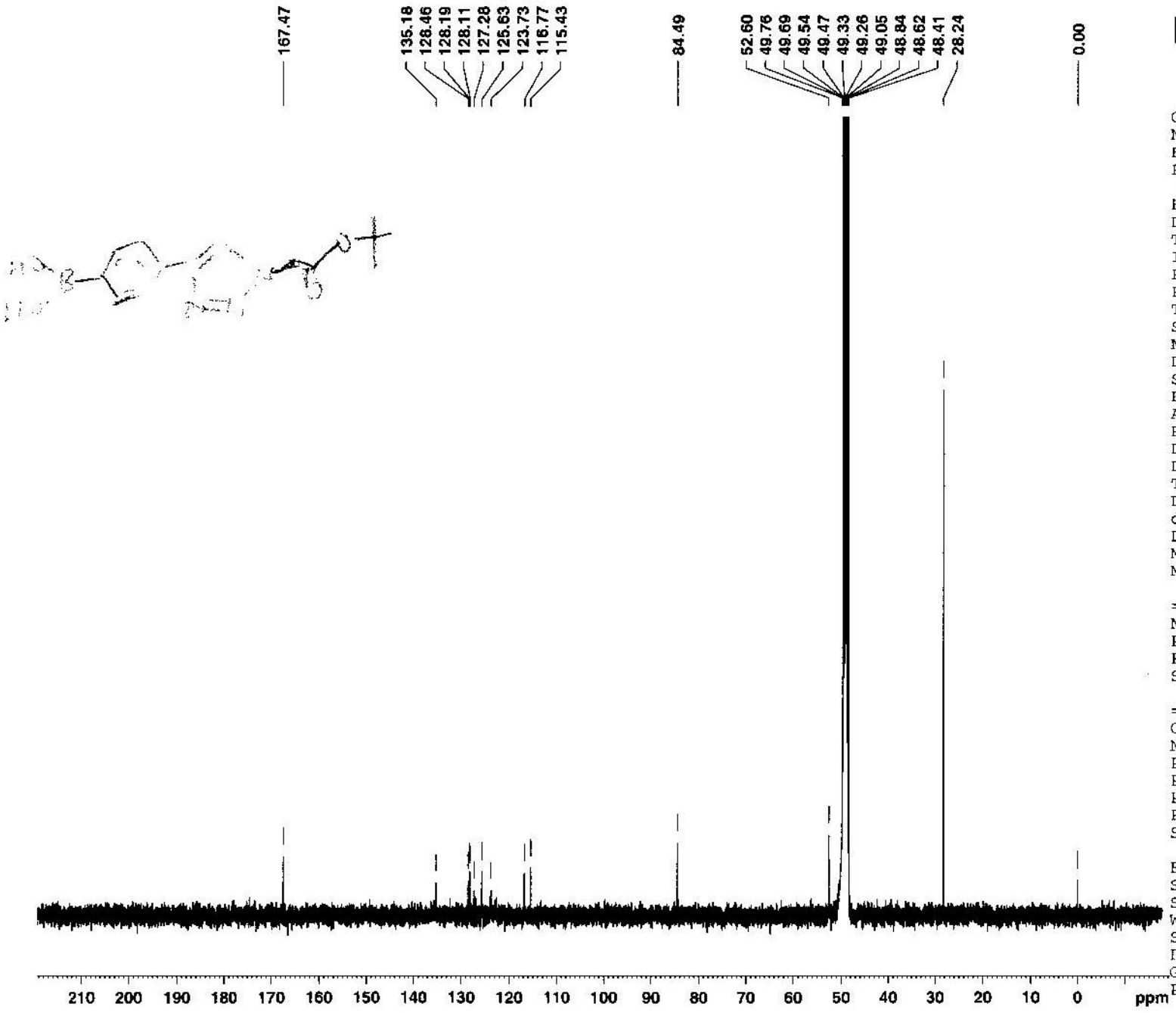

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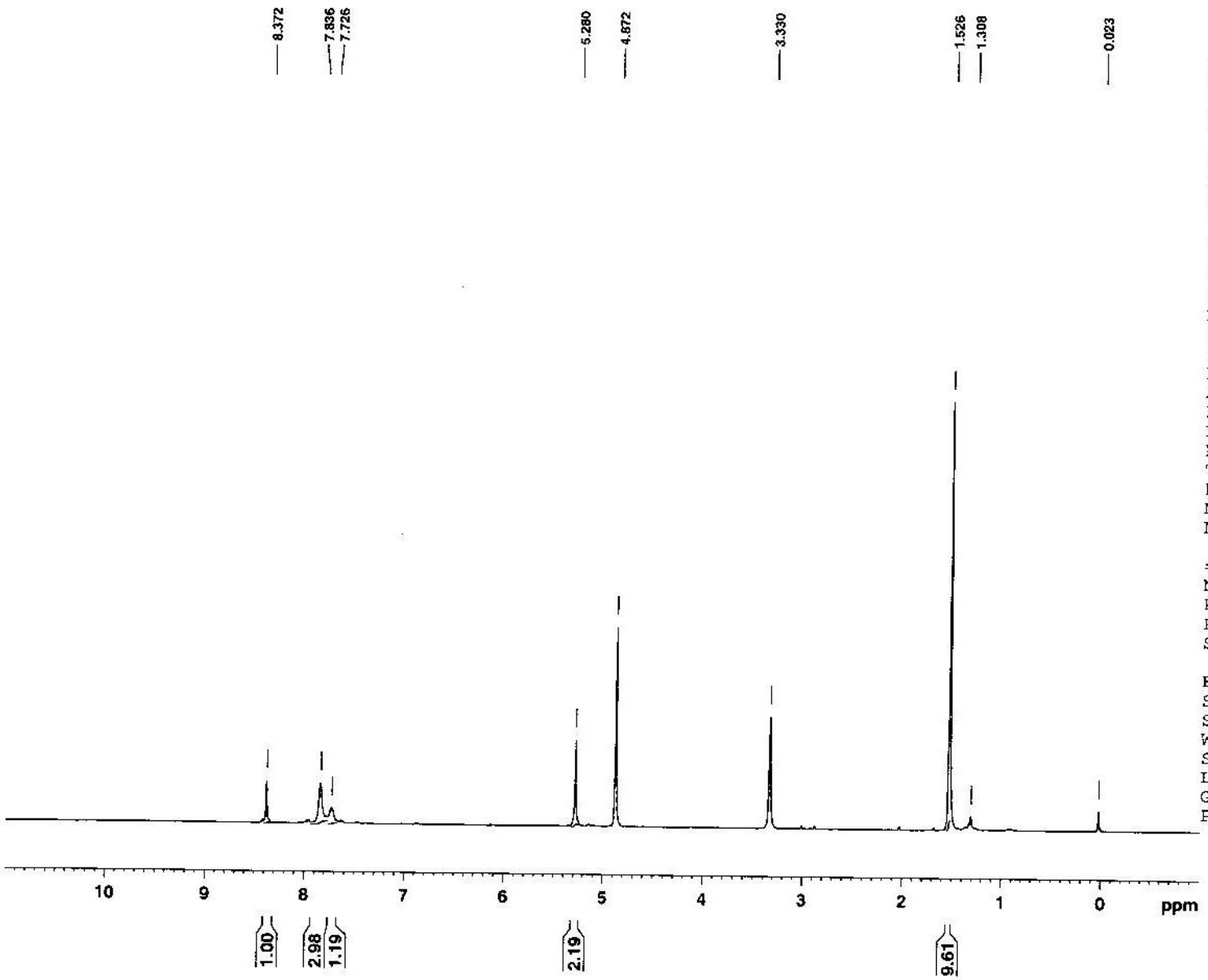

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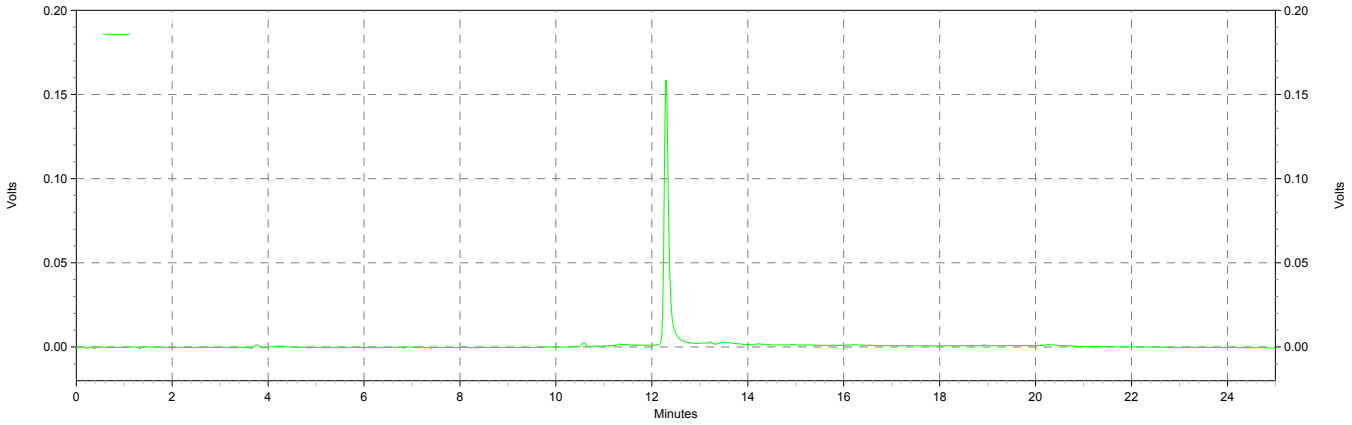

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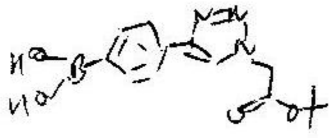

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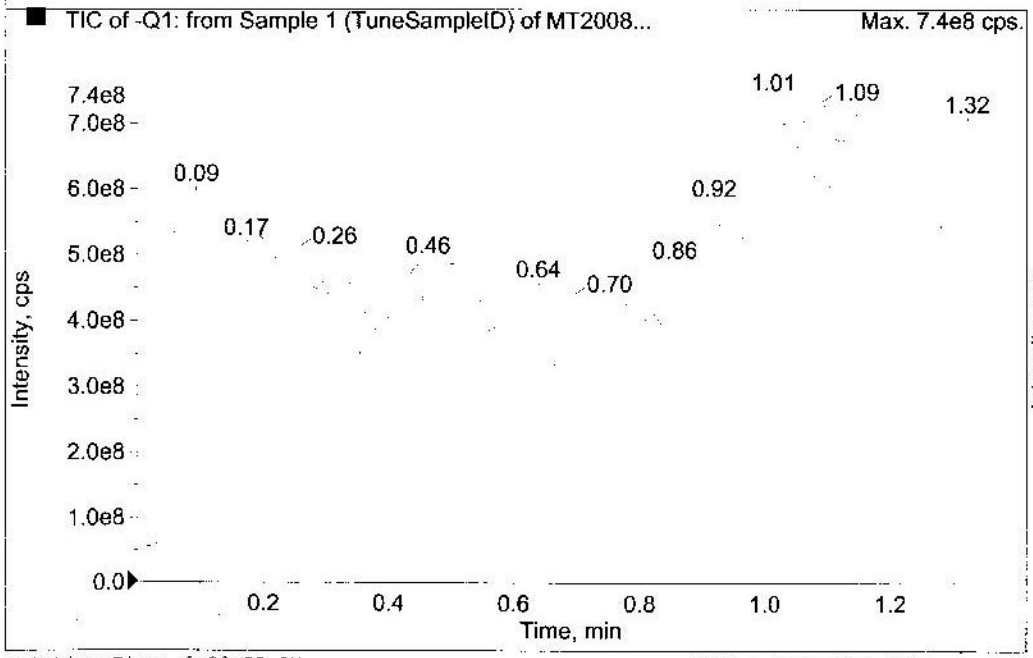

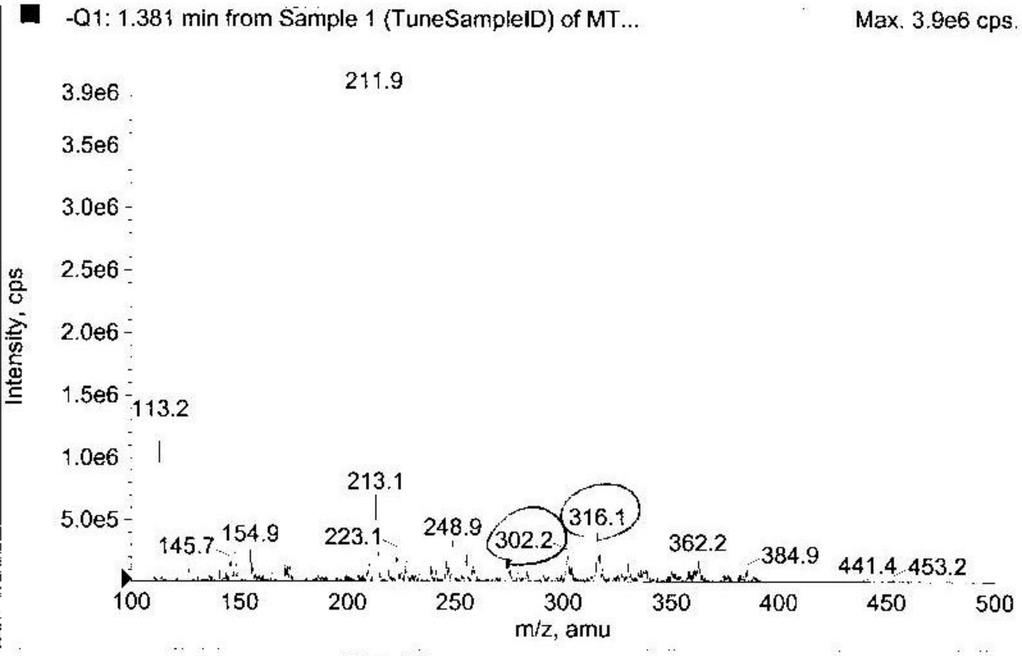

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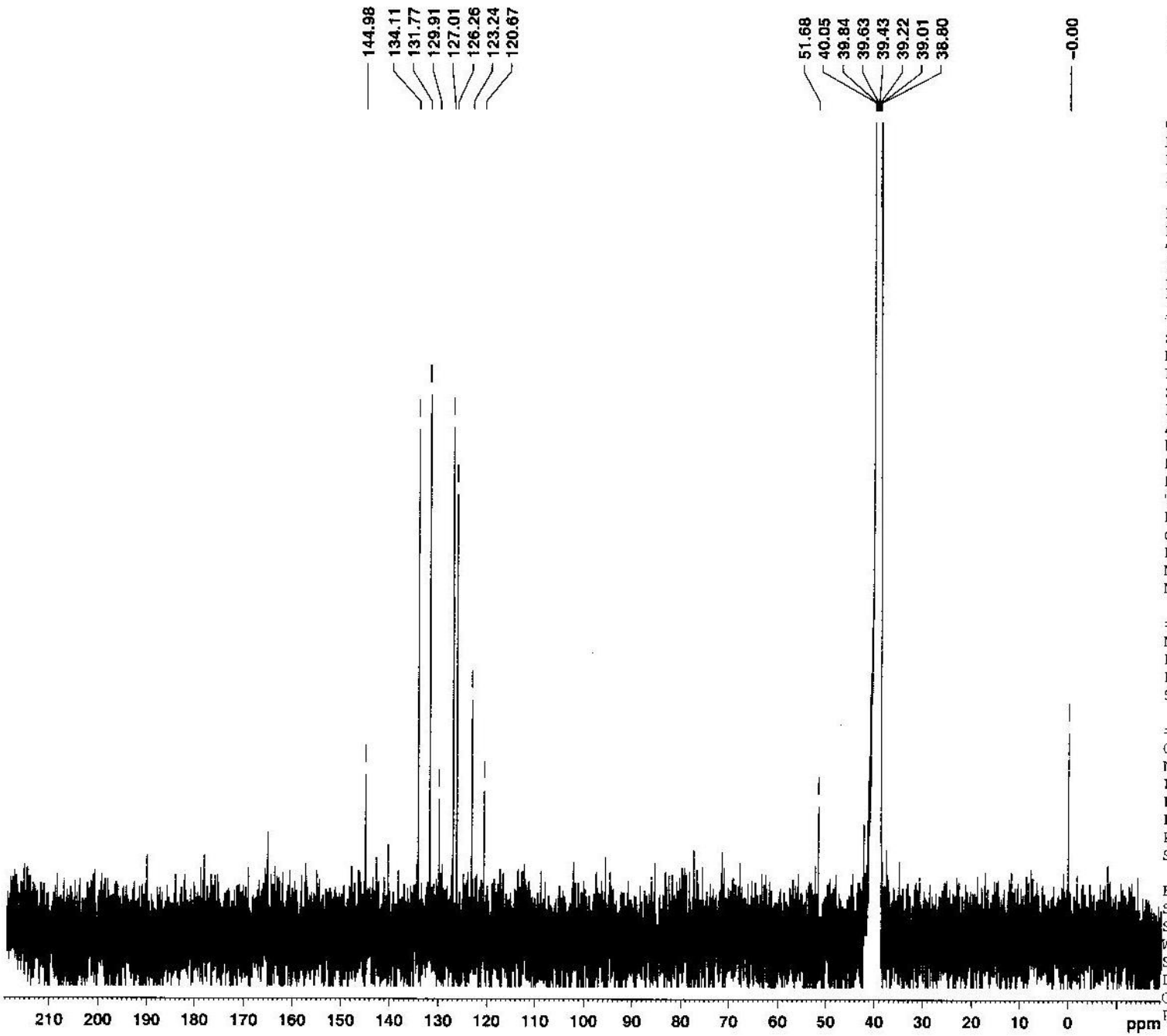

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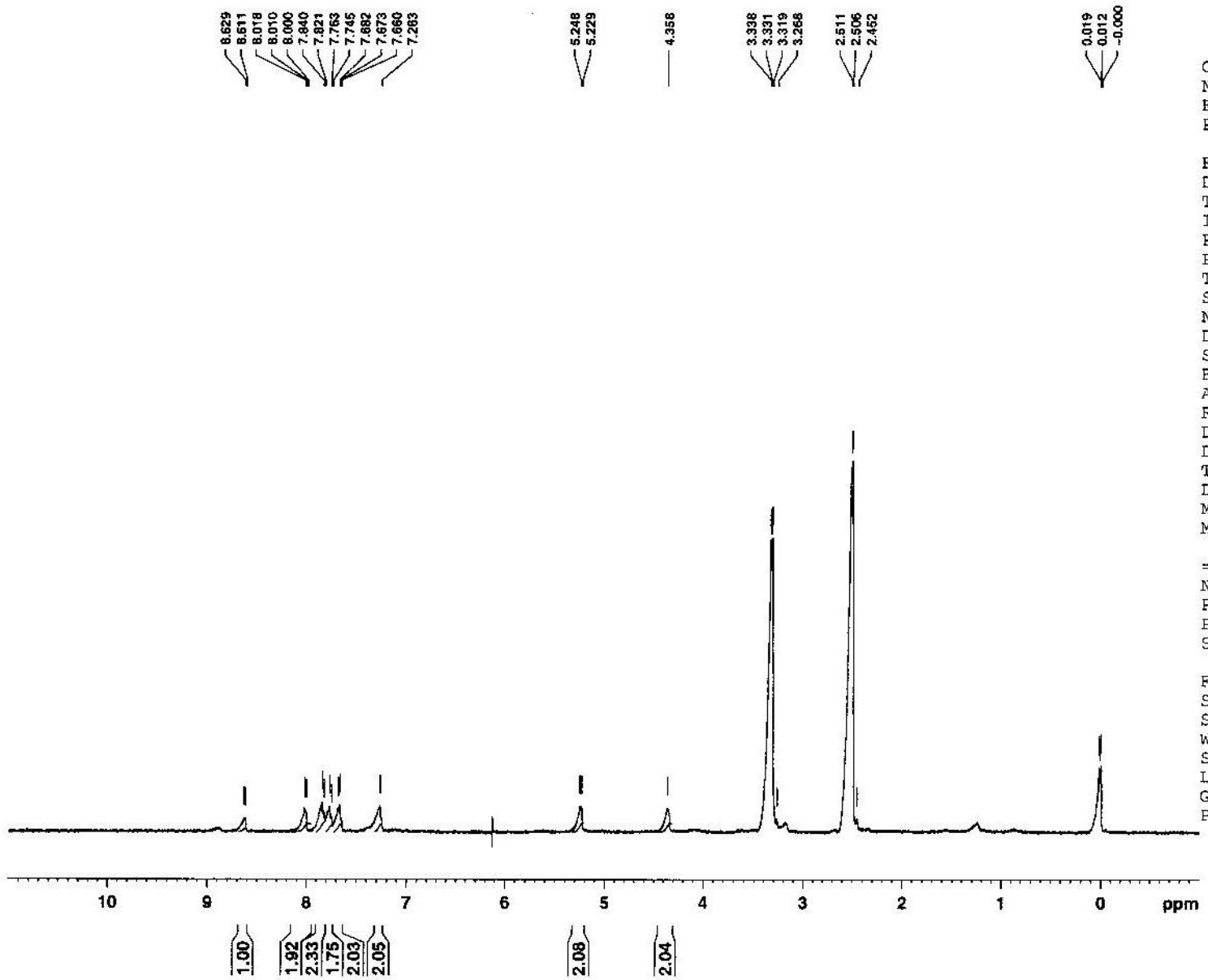

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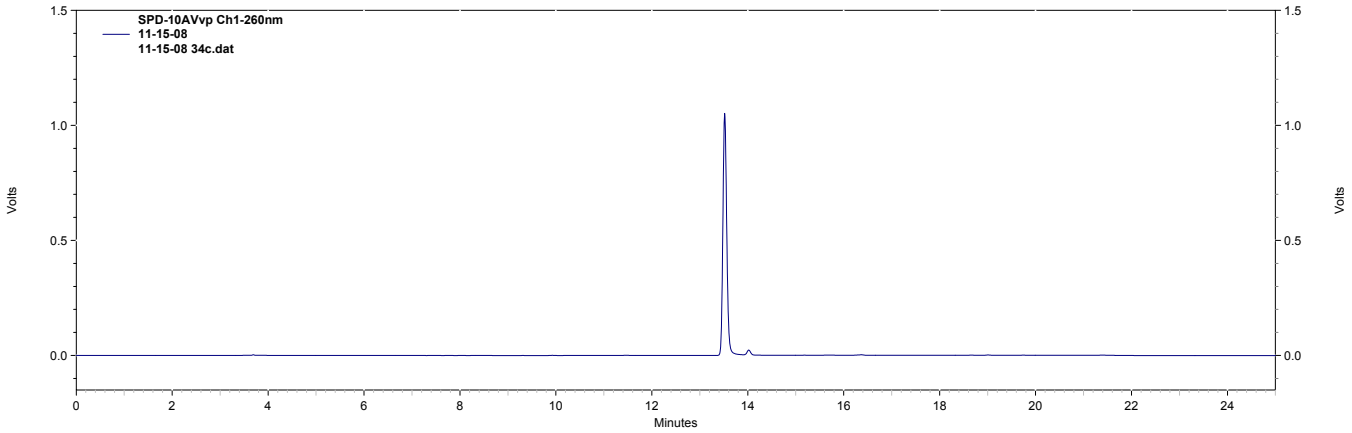

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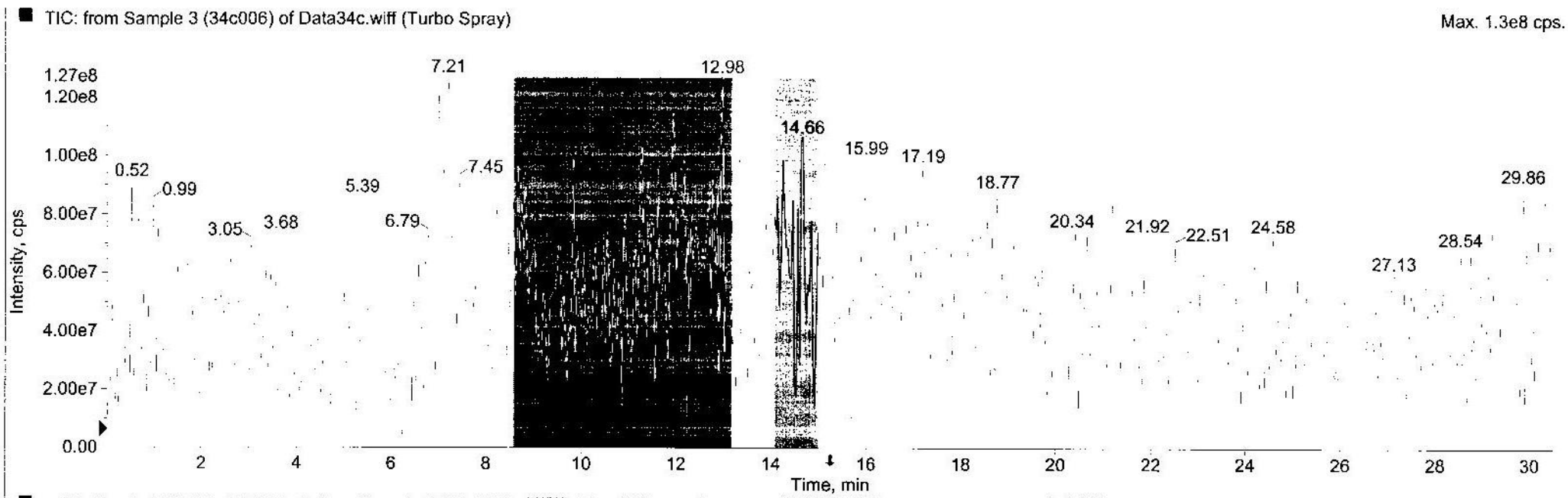

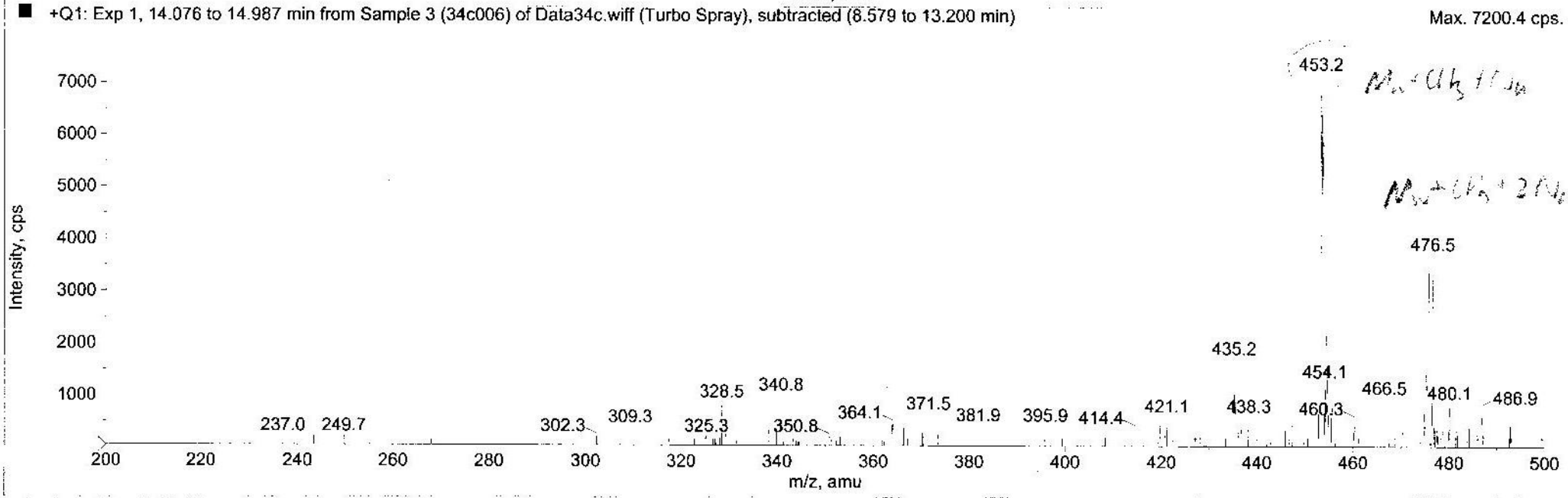

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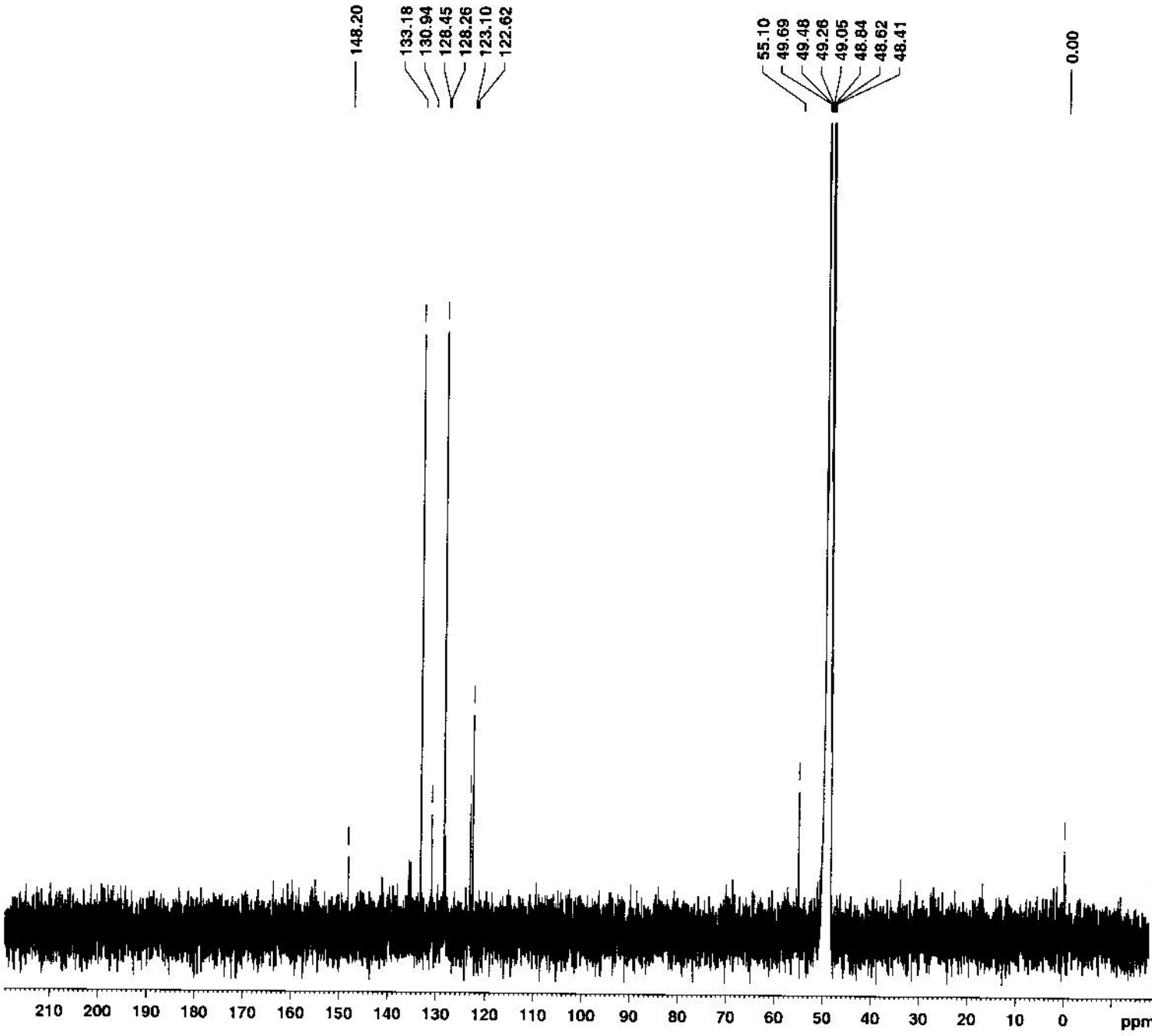


BRUKER

Current Data Parameters

NAME	JS-35c
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20081205
Time	10.24
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	12439
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	20642.5
DW	20.850 usec
DE	7.00 usec
TE	299.5 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	-3.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

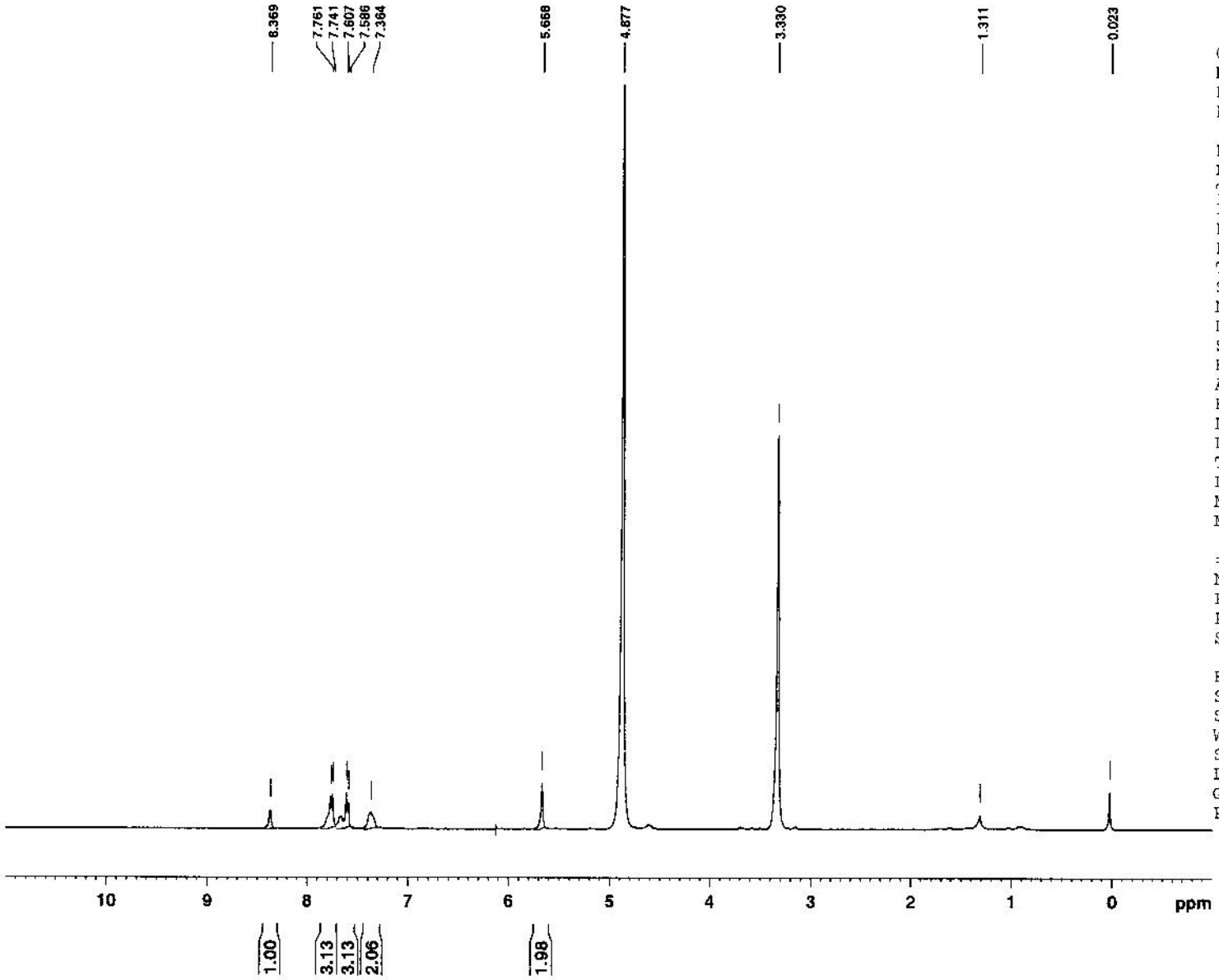
CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-1.00 dB
PL12	14.00 dB
PL13	14.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6126221 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

No title

(a)



Current Data Parameters

NAME	sj-iv-35c
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20081125
Time	17.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	406.4
DW	60.400 usec
DE	7.00 usec
TE	298.8 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	12.80 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

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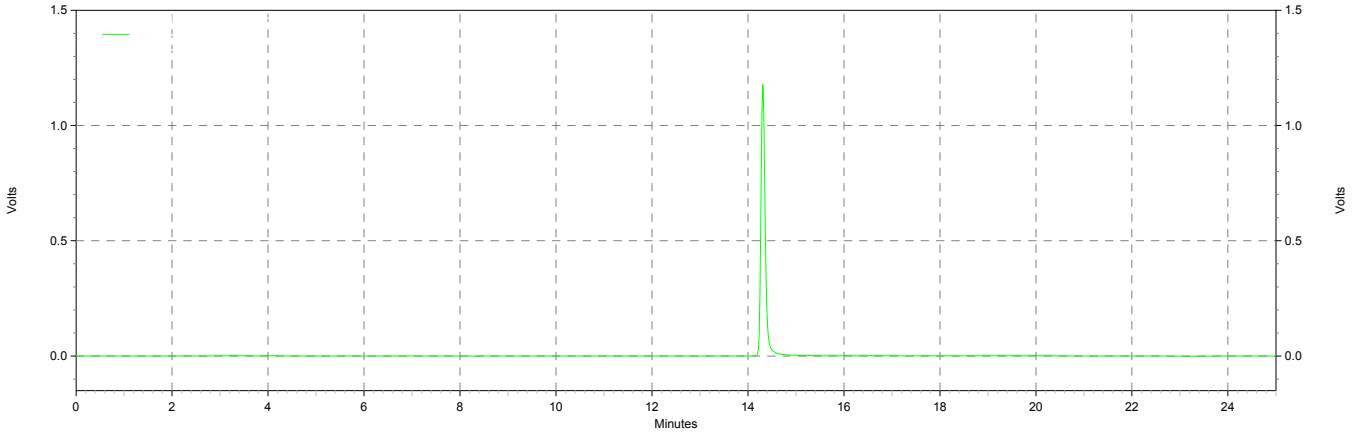

C:\shan\11-19-08 35c 0000.dat, SPD-10AVvp Ch1-260nm

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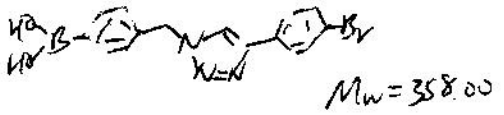


State Parameter Editor

Ion Source:	Turbo Spray
Temperature Reached	
Curtain Gas (CUR):	10.0
IonSpray Voltage (IS):	4500.0
Temperature (TEM):	400.0
Ion Source Gas 1 (GS1):	10.0
Ion Source Gas 2 (GS2):	0.0
Interface Heater (ihe):	On
Declustering Potential (DP):	45.0
Entrance Potential (EP):	10.0
Q1 Resolution:	Unit
Ion Energy 1 (IE1):	0.5
Reflector (RE):	-150.0
CEM (CEM):	2400.0

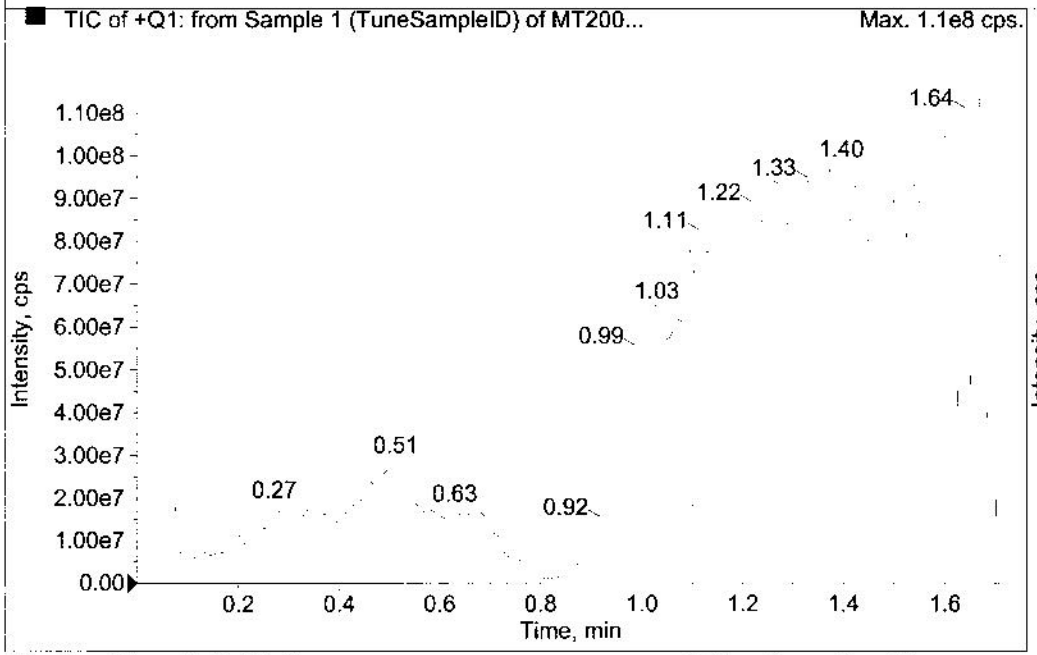
Mass Spectrometer Method Properties

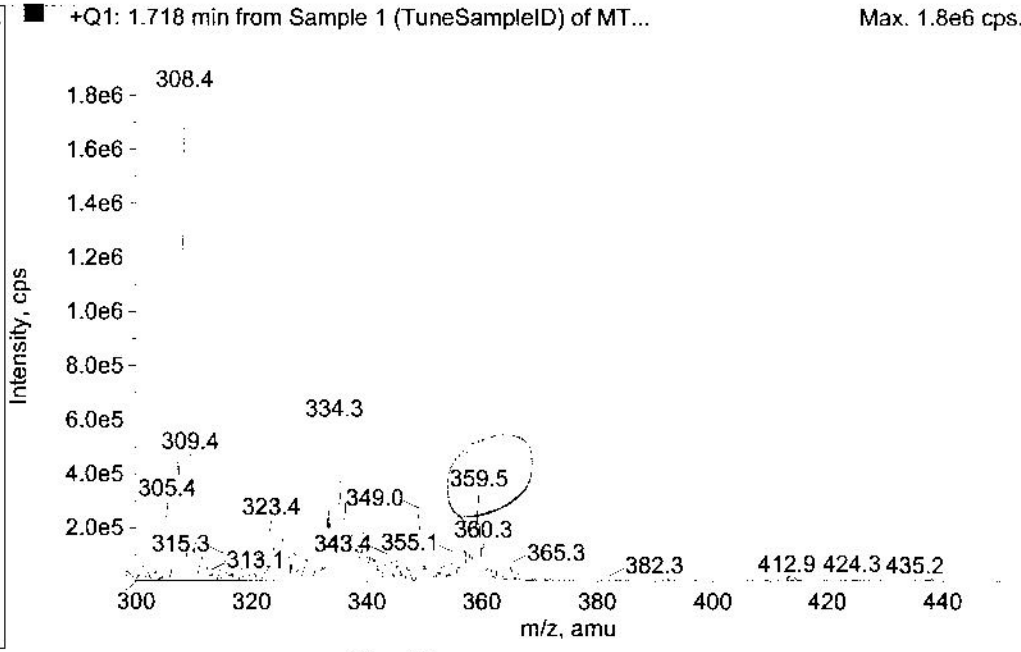
Period 1:

Scans in Period:	594
Relative Start Time:	0.00 msec
Experiments in Period:	1

Period 1 Experiment 1:

Scan Type:	Q1 MS (Q1)
Polarity:	Positive
Scan Mode:	Profile
Ion Source:	Turbo Spray
Resolution Q1:	Unit
Intensity Thres.:	0.00 cps
Settling Time:	0.0000 msec
MR Pause:	5.0070 msec
MCA:	No
Center/Width:	No
Step Size:	0.10 amu





Printing Time: 5:56:17 PM
Printing Date: Saturday, November 22, 2008

*FSE : Tyrus Lavallee

*Pre PM

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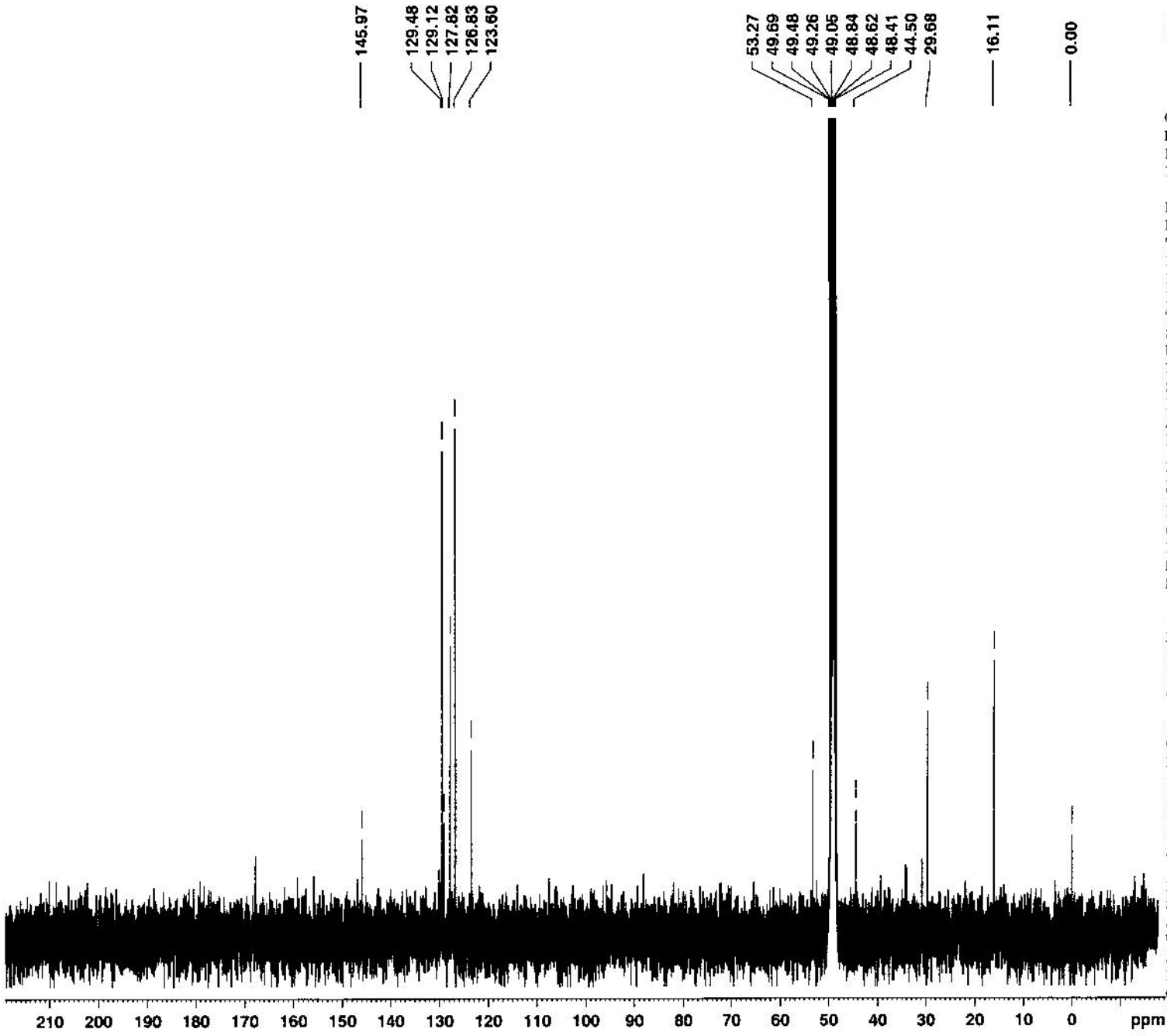
No title



BRUKER

Current Data Parameters

NAME	JS-35c
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20081212
Time	12.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	1577
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	32768
DW	20.850 usec
DE	7.00 usec
TE	299.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	-3.00 dB
SFO1	100.6228298 MHz

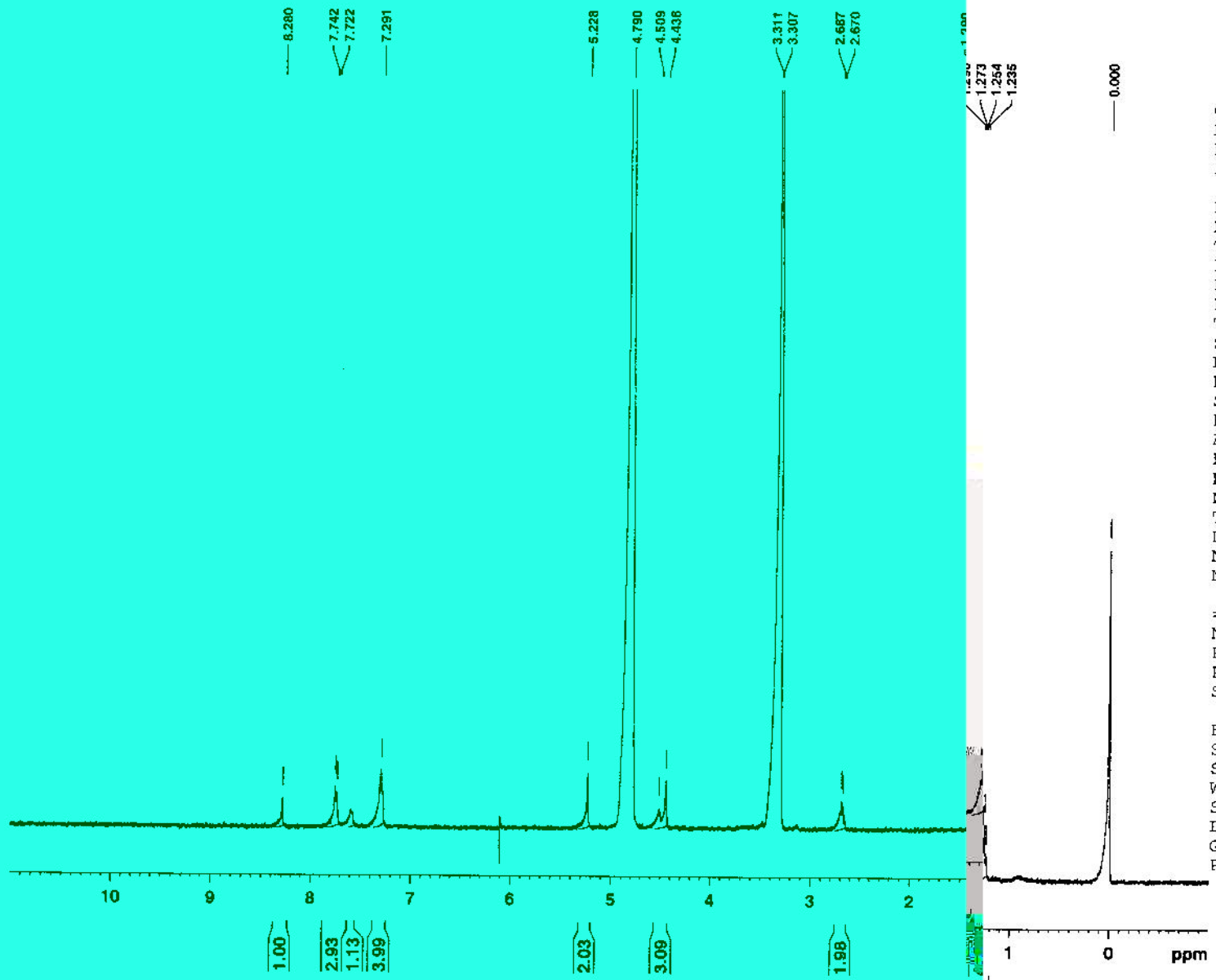
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-1.00 dB
PL12	14.00 dB
PL13	14.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6126229 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

No title



BRUKER

Current Data Parameters
NAME sj-iv-45 (1)c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_	20081208
Time	11.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	512
DW	60.400 usec
DE	7.00 usec
TE	306.8 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	12.80 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300086 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.40

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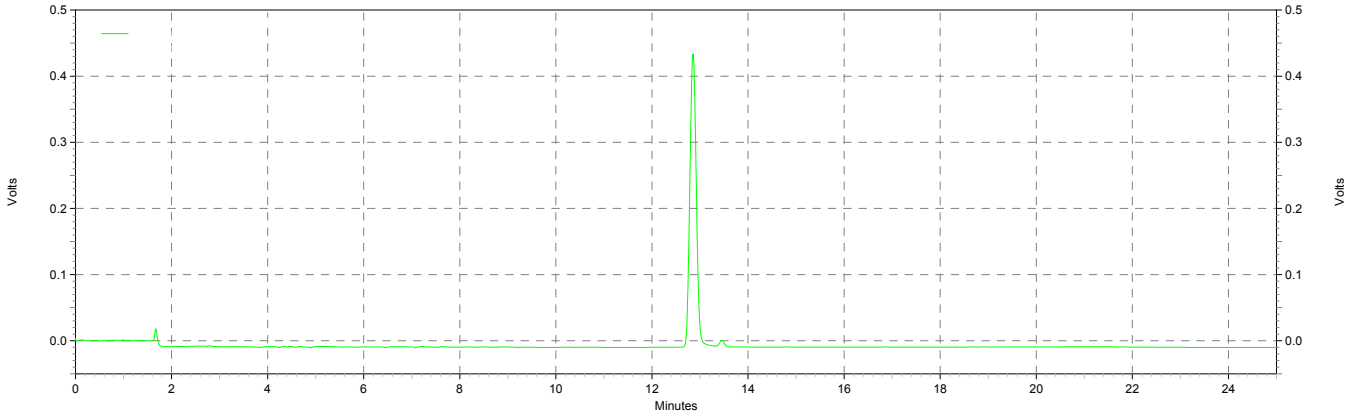

C:\shan\12-5-09 45(2) 000.dat, SPD-10AVvp Ch1-260nm

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M.w = 364.2

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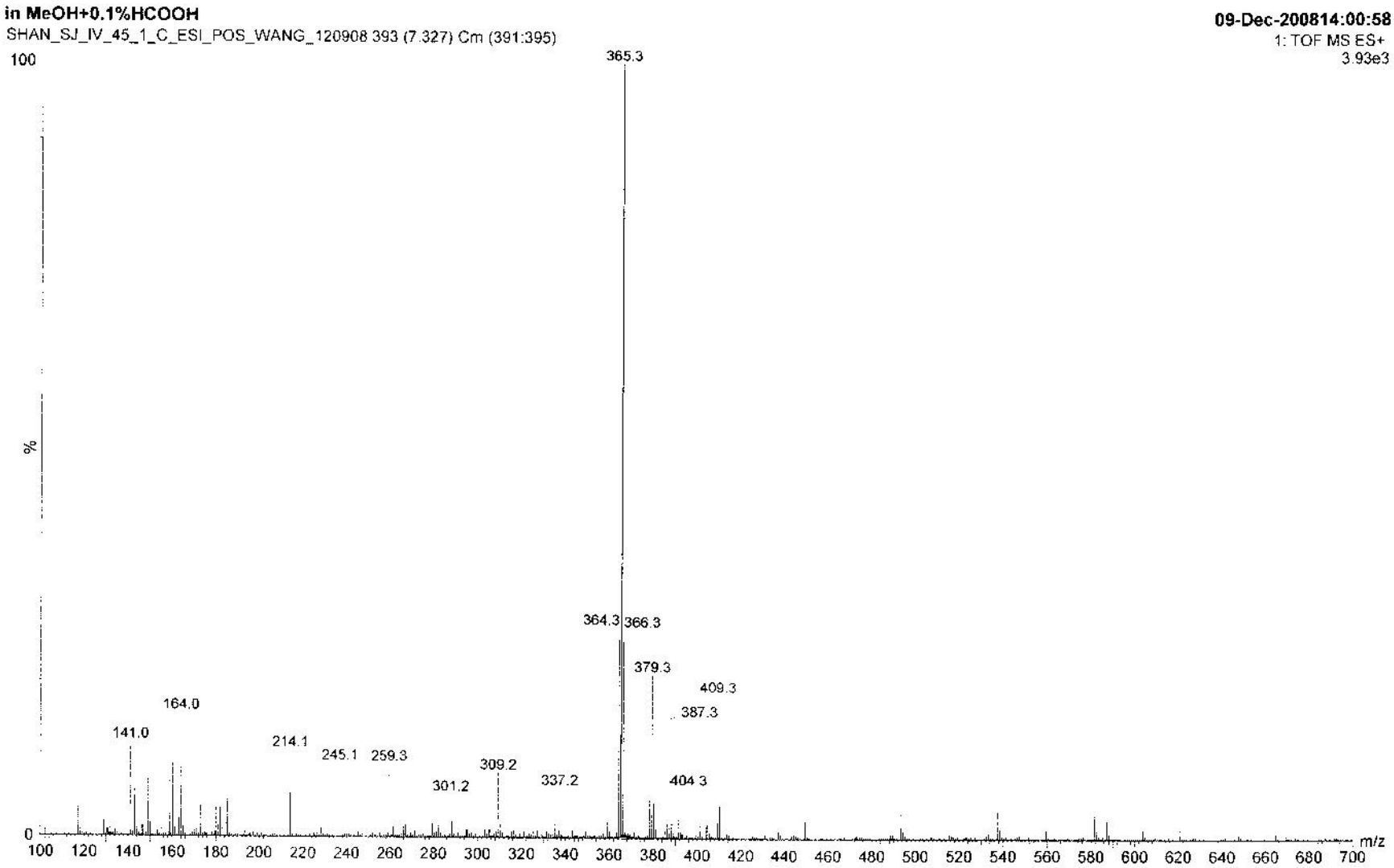

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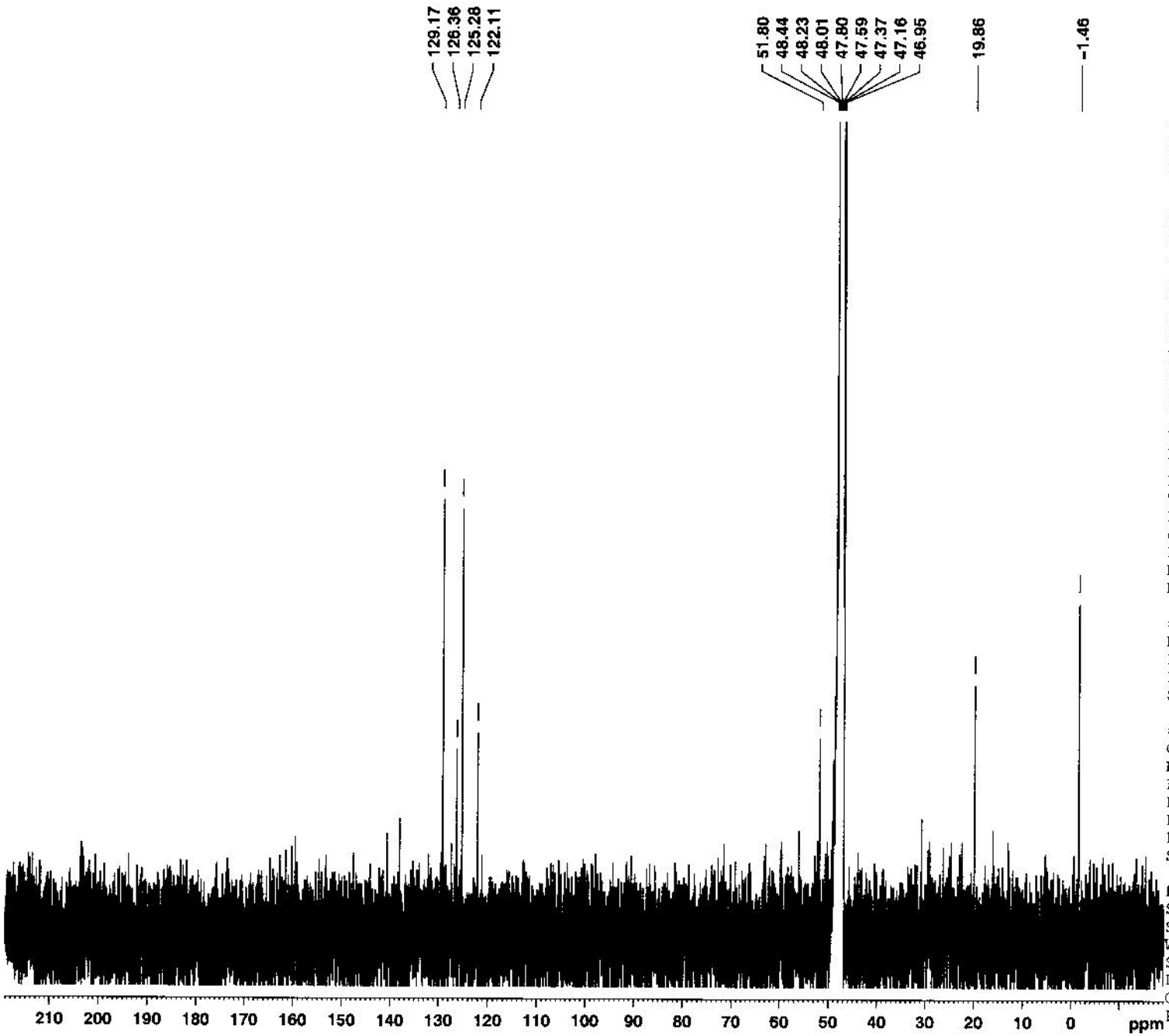

BRUKER

Current Data Parameters
NAME sj-iv-45 (3) c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_	20081212
Time	19.32
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	16126
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	32768
DW	20.850 usec
DE	7.00 usec
TE	299.3 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	-3.00 dB
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-1.00 dB
PL12	14.00 dB
PL13	14.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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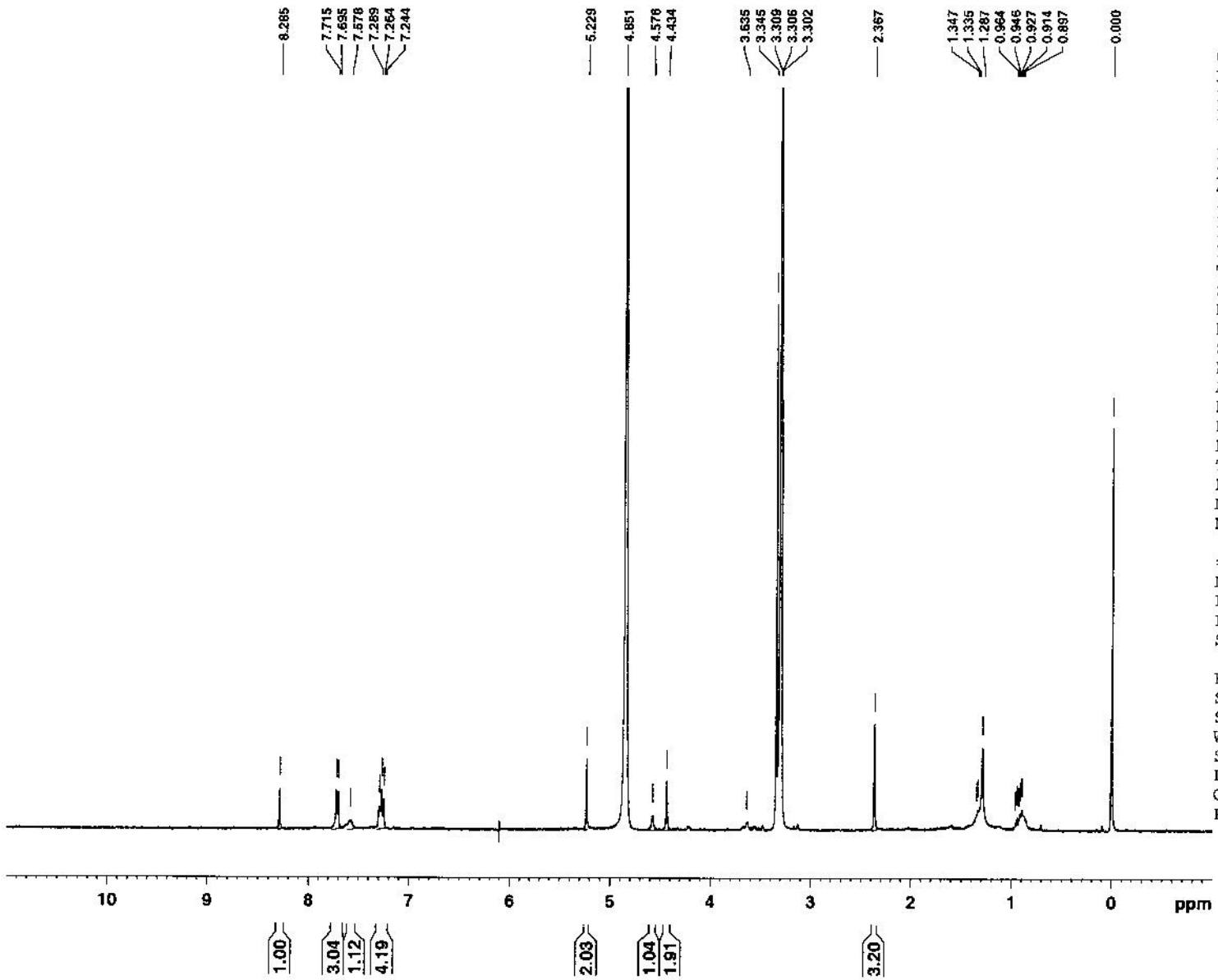
No title



BRUKER

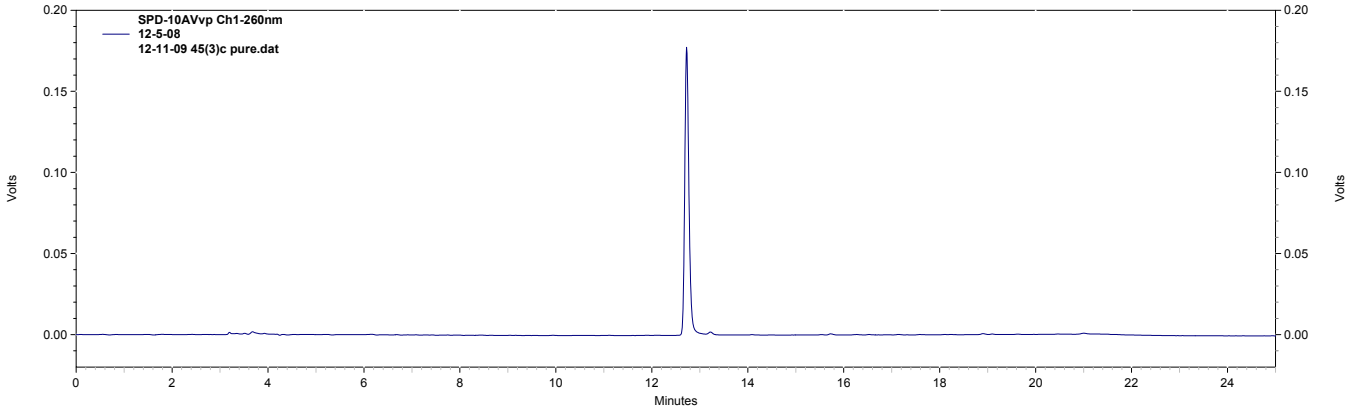
(6)

Current Data Parameters
NAME sj-iv-45 (3)
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081212
Time 19.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 406.4
DW 60.400 usec
DE 7.00 usec
TE 298.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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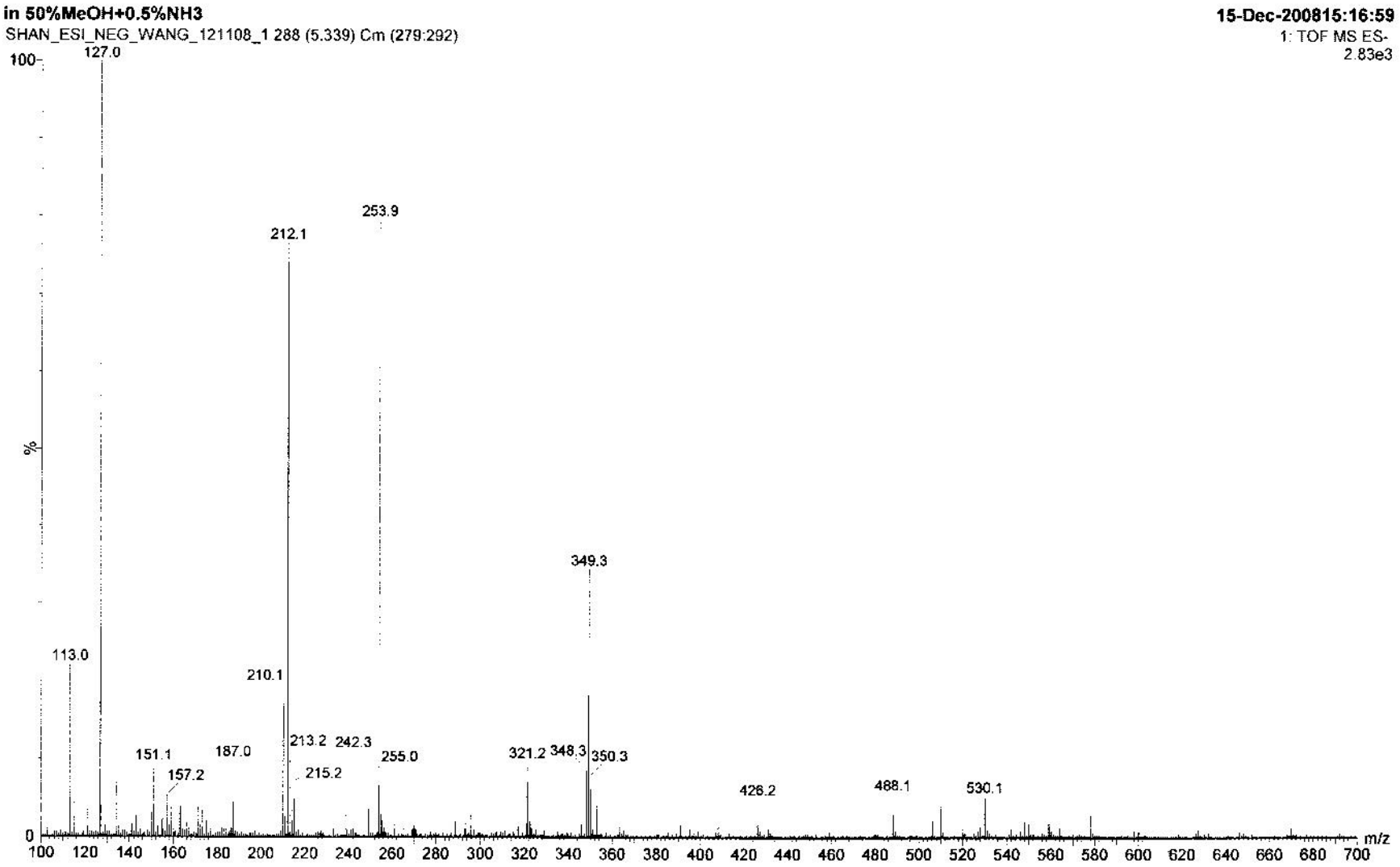

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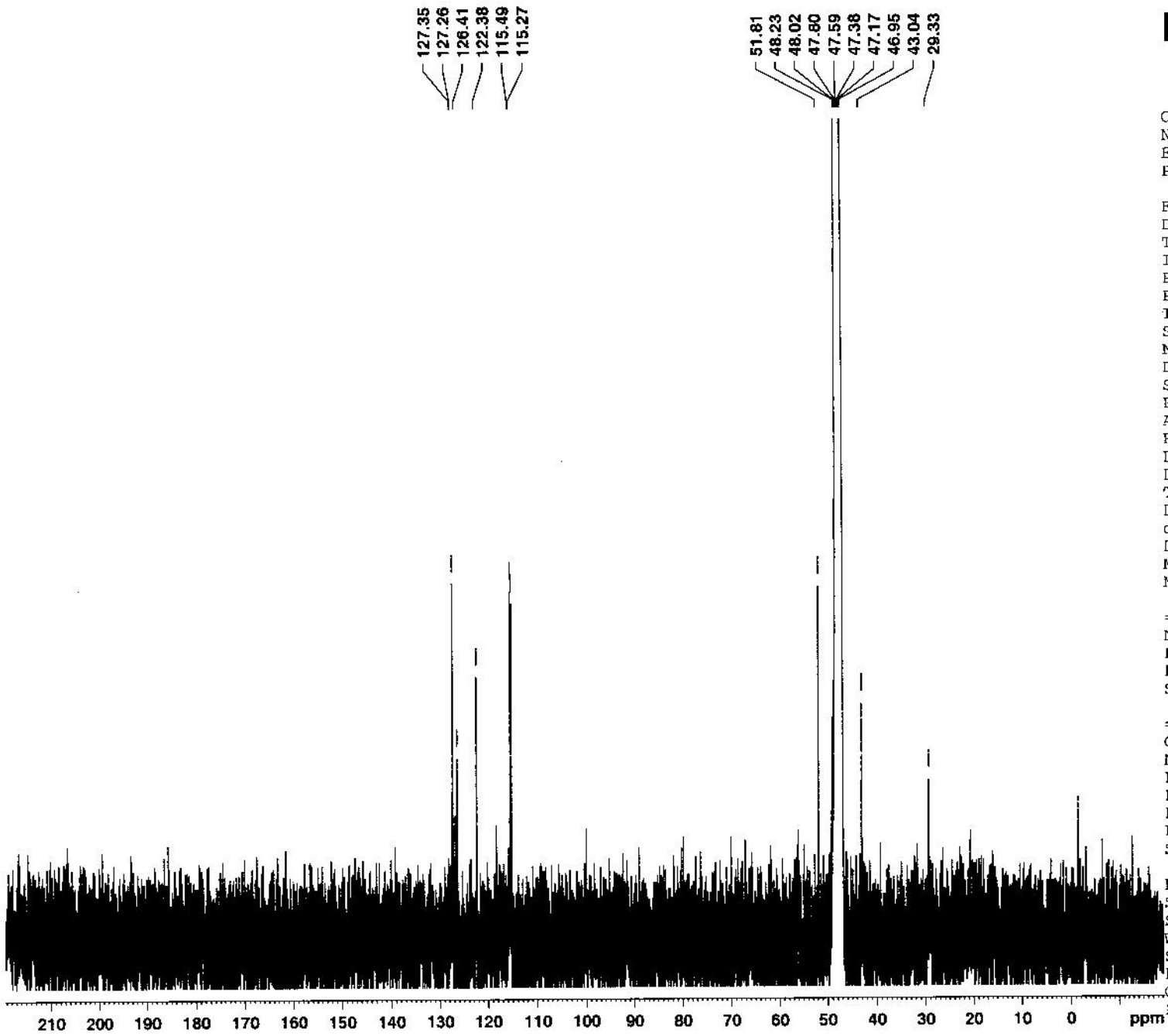
No title



BRUKER

Current Data Parameters
NAME sj-iv-45 (5) c
EXPNO 1
PROCNO 1

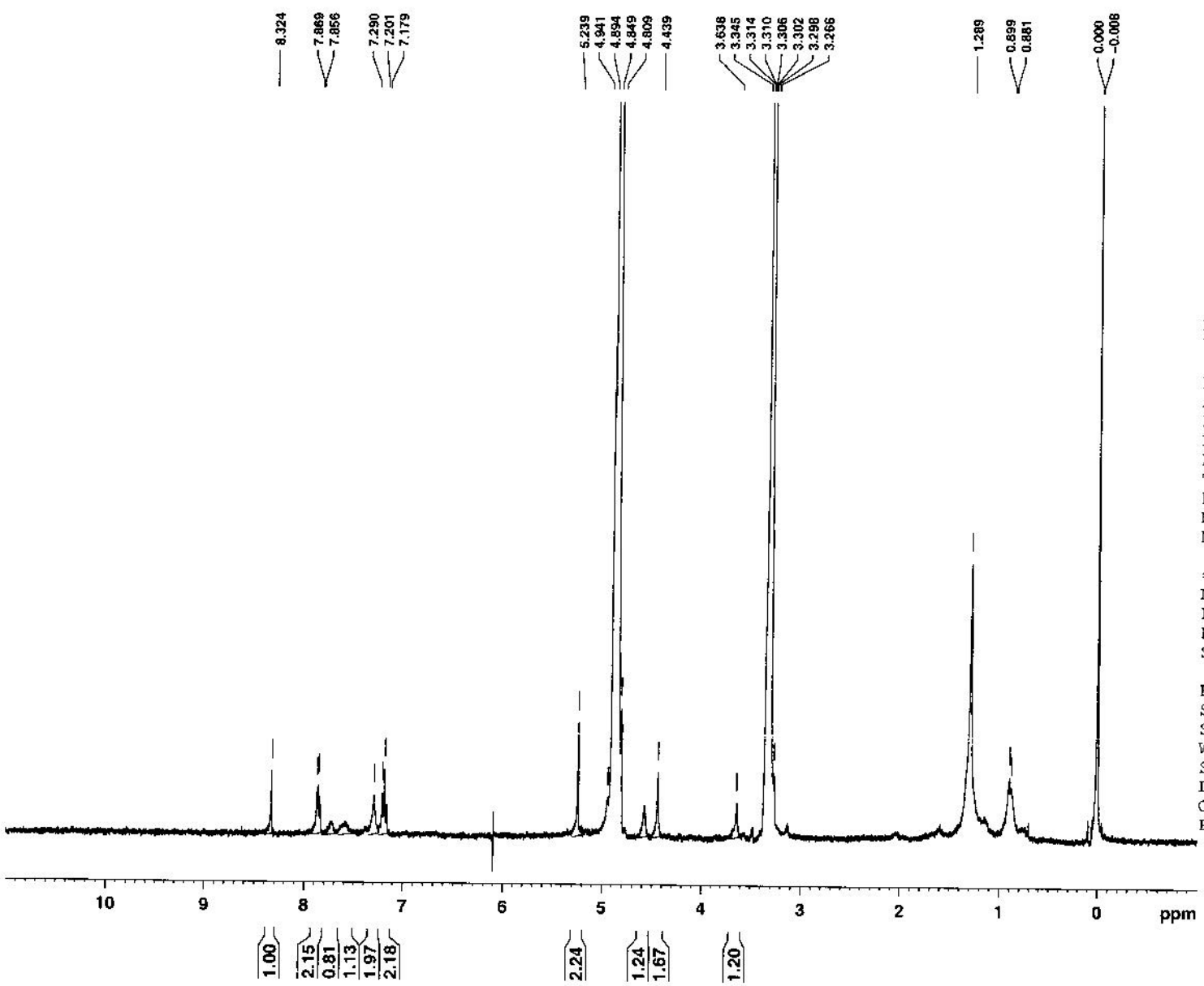
F2 - Acquisition Parameters
Date_ 20090117
Time 20.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 17884
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 7.00 usec
TE 299.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

No title



BRUKER

Current Data Parameters

NAME	sj-iv-45 (5)
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20081222
Time	14.03
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	406.4
DW	60.400 usec
DE	7.00 usec
TE	299.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	12.80 usec
PL1	0.00 dB
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

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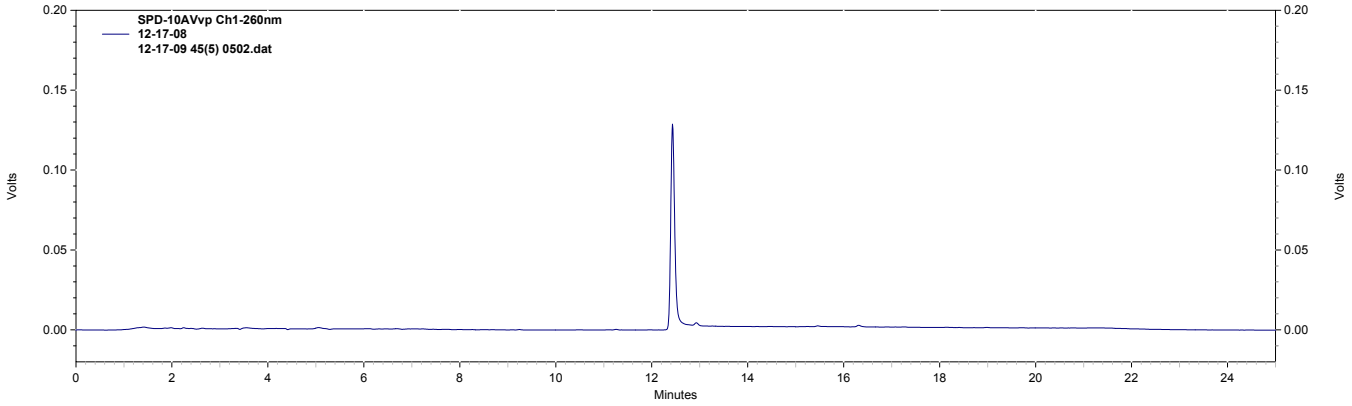

C:\shan\12-17-09 45(5) 0502.dat, SPD-10AVvp Ch1-260nm

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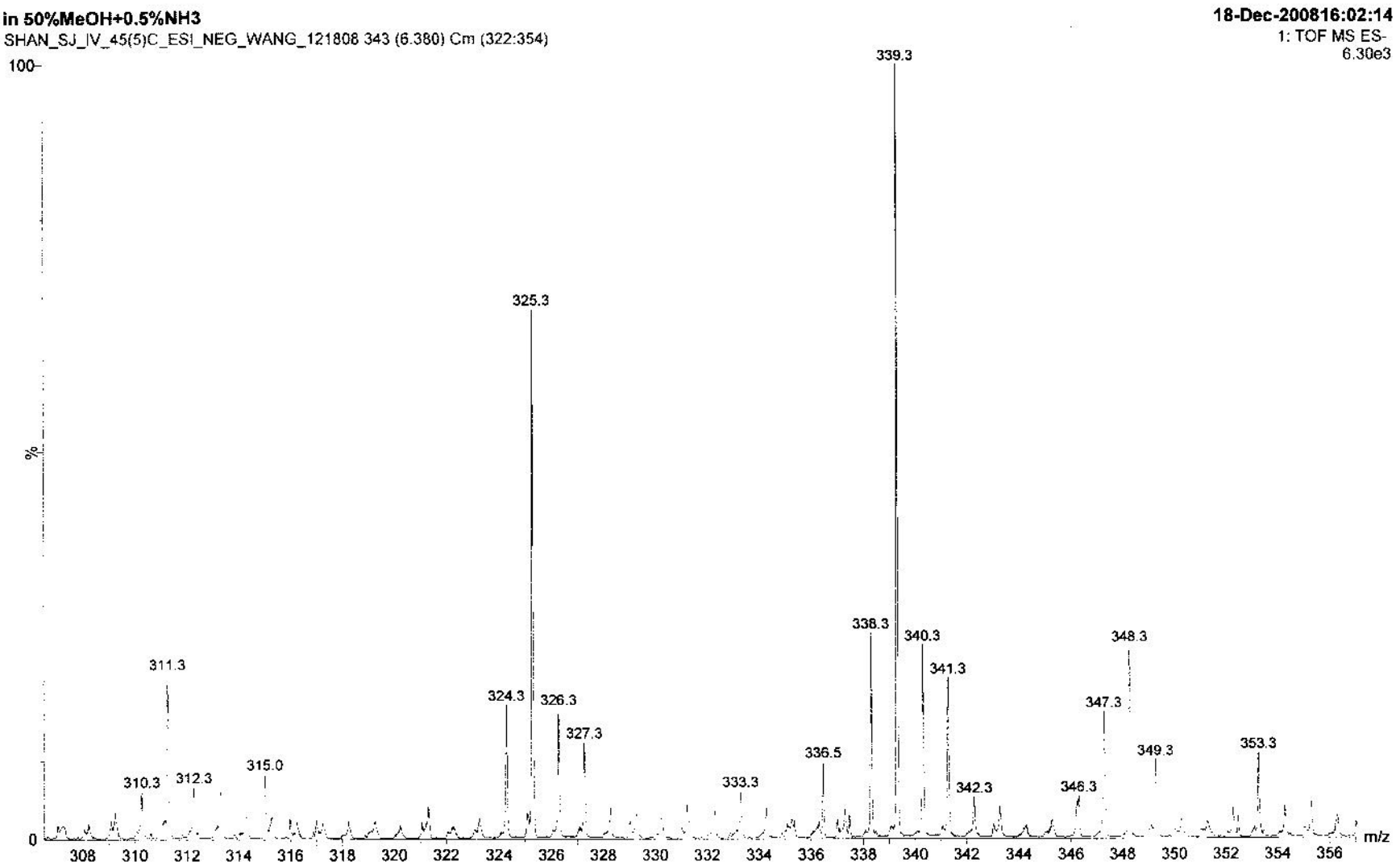

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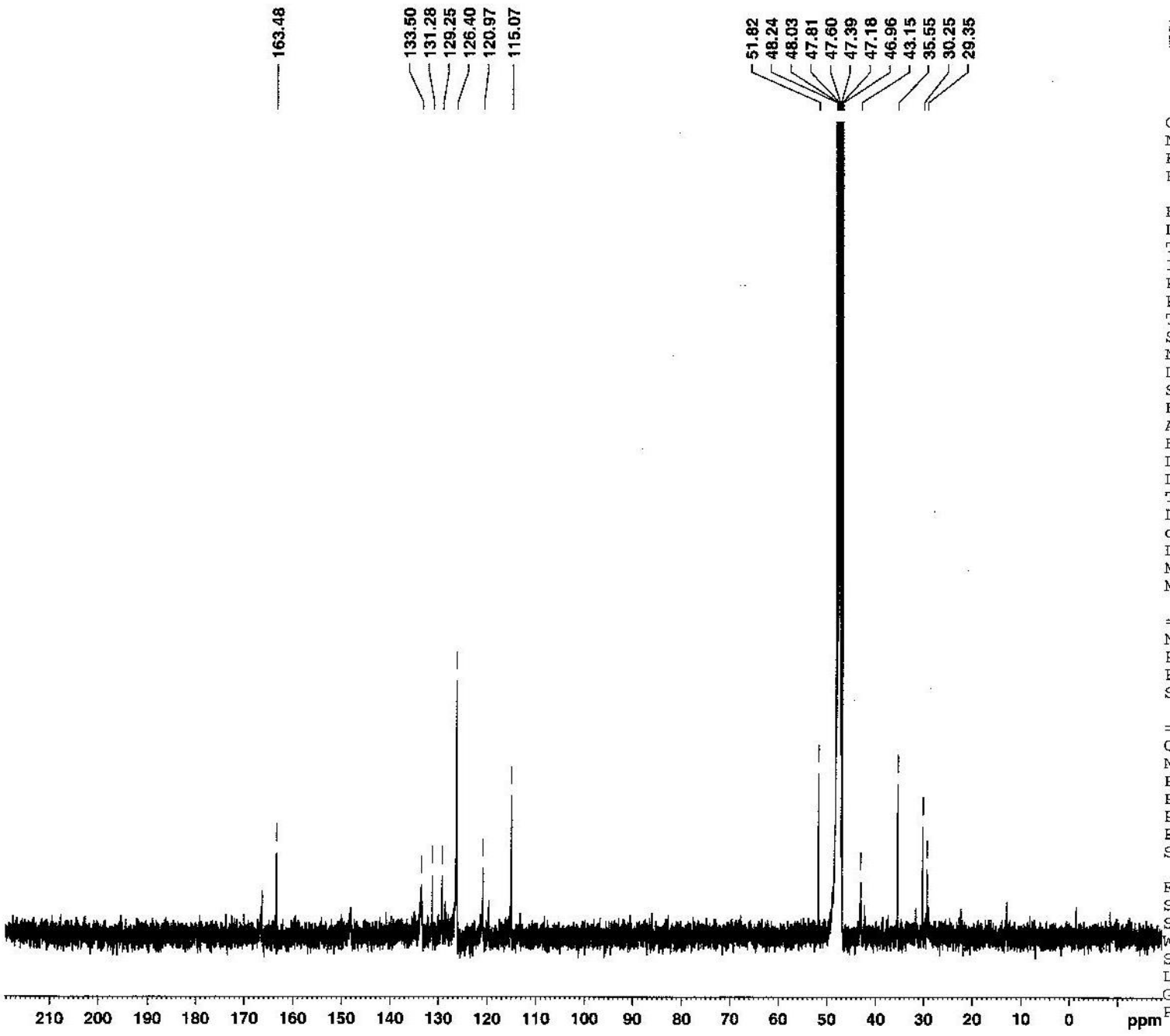

BRUKER

Current Data Parameters
NAME SJ-III-45(7) c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090507
Time 20.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 14926
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 7.00 usec
TE 299.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 8.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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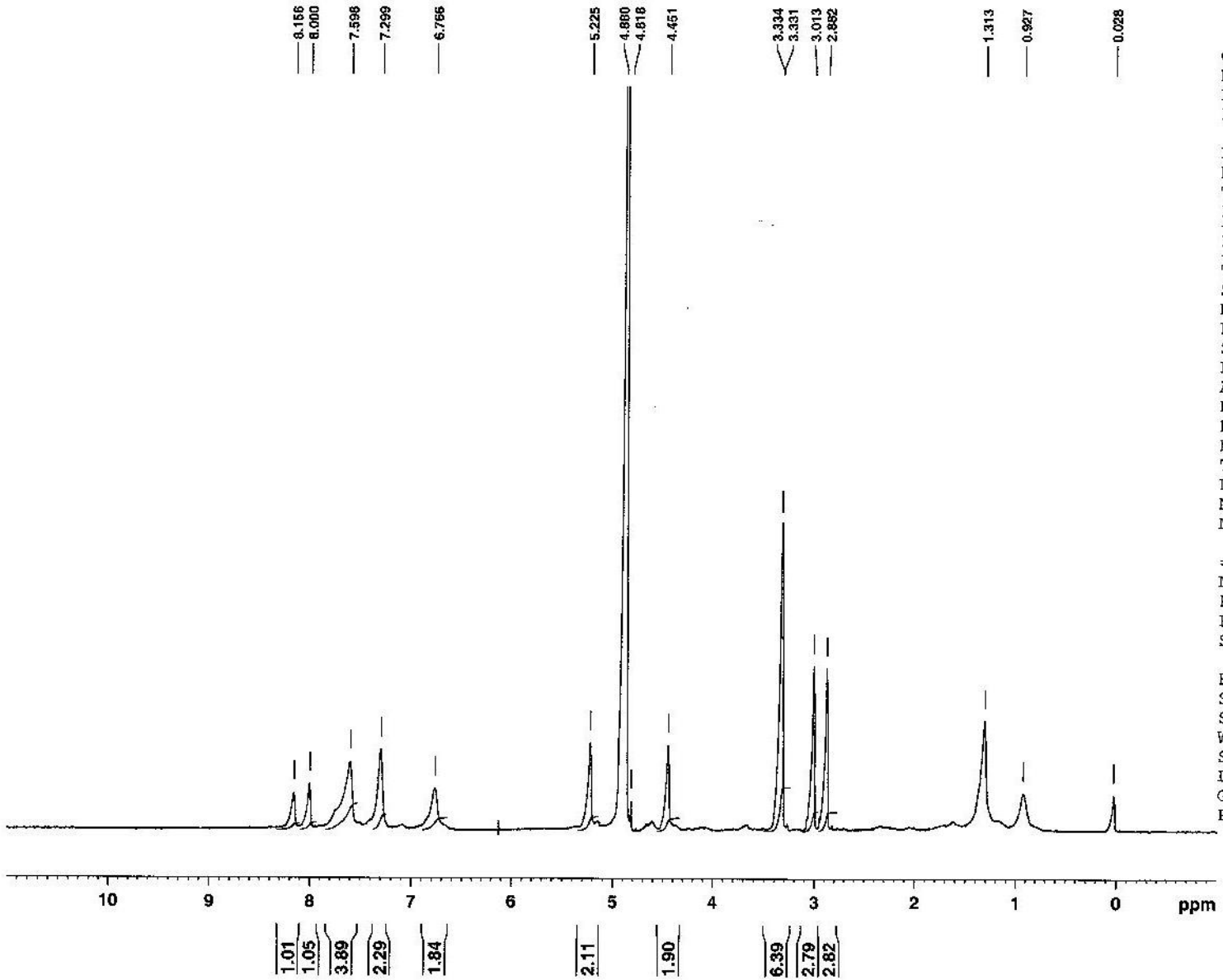
No title



Current Data Parameters
NAME SJ-III-45(7)
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090507
Time 20.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 7.00 usec
TE 298.8 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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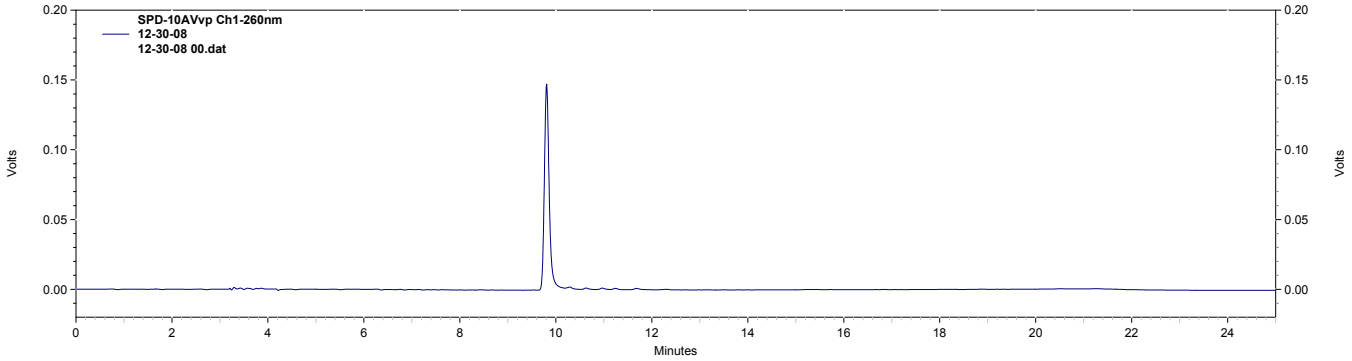

C:\nnt\12-30-08 00.dat, SPD-10AVvp Ch1-260nm

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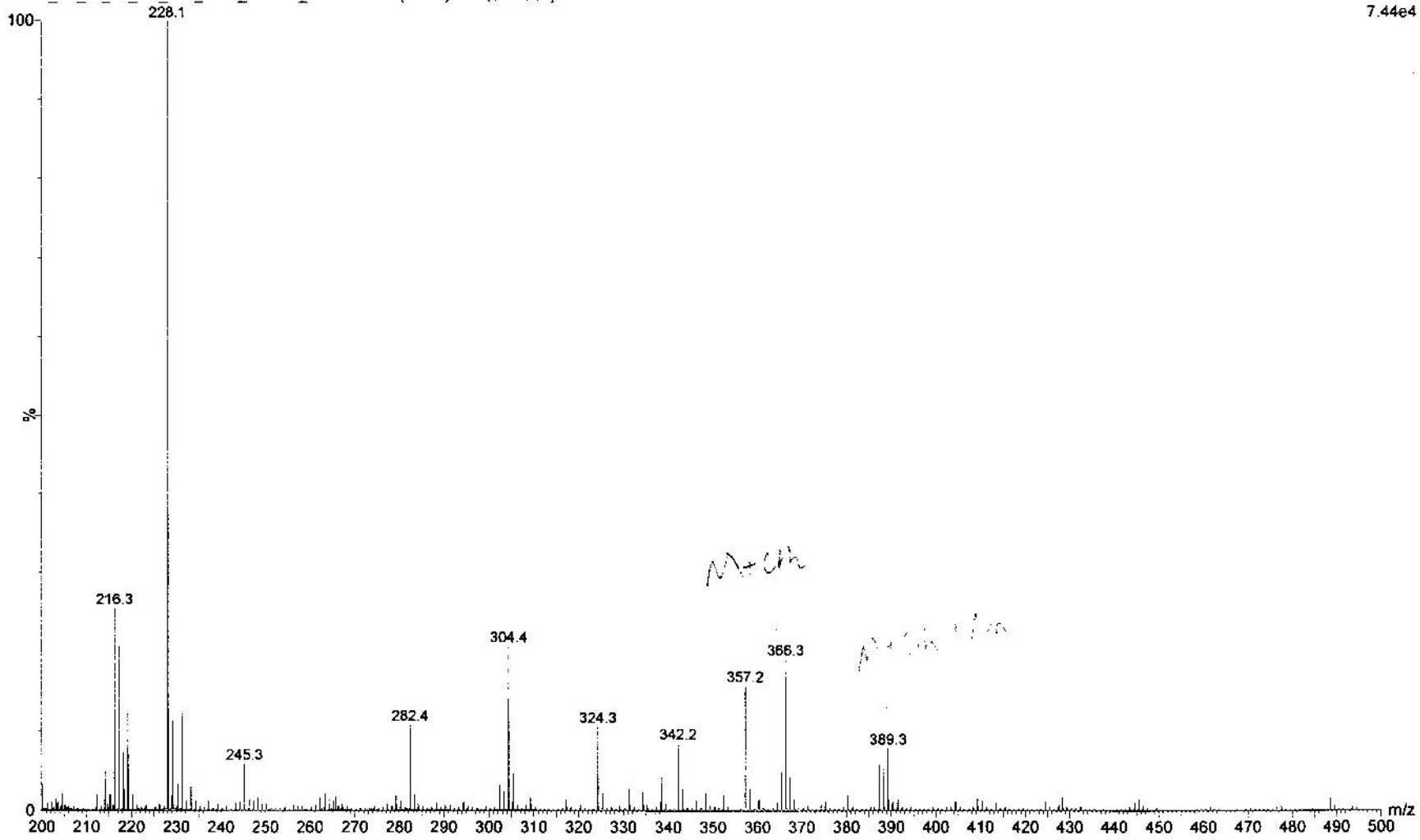

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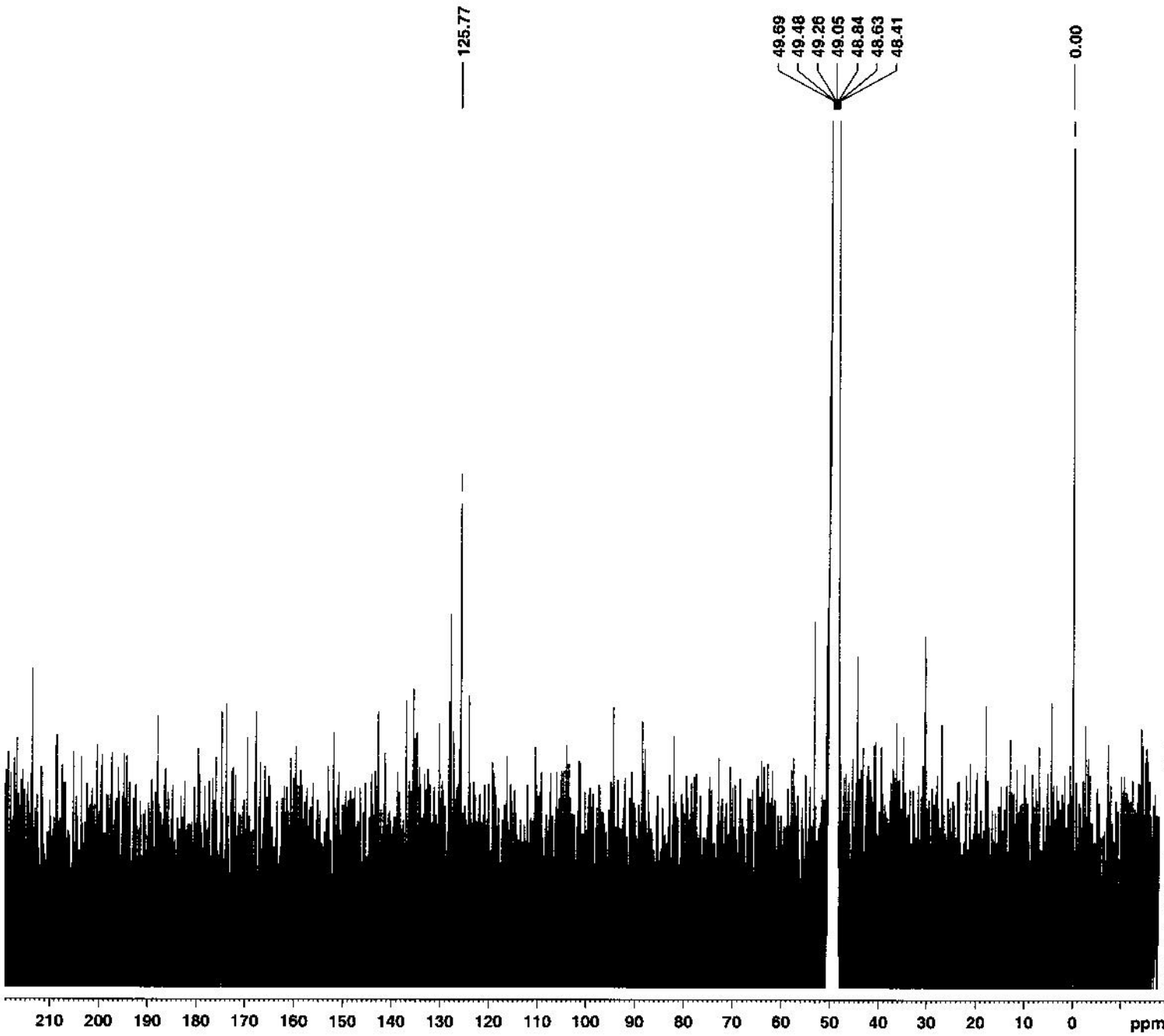
No title



BRUKER

Current Data Parameters

NAME	sj-iv-45-(9) c
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20090123
Time	20.23
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	19778
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	32768
DW	20.850 usec
DE	7.00 usec
TE	299.6 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	-3.00 dB
SFO1	100.6228298 MHz

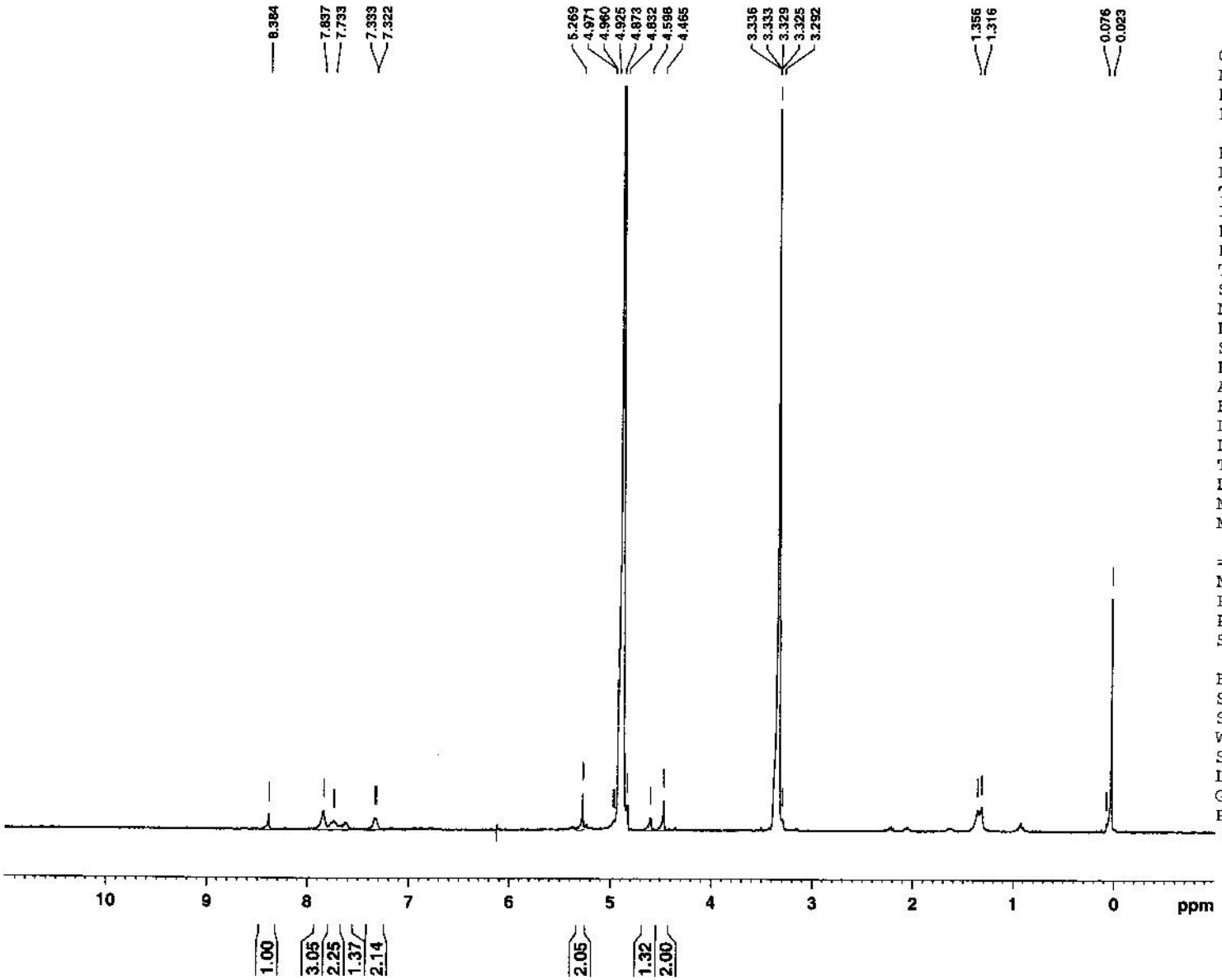
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	70.00 usec
PL2	-1.00 dB
PL12	14.00 dB
PL13	14.00 dB
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6126221 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

No title



BRUKER

(9)

Current Data Parameters
NAME sj-iv-45-(9)
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090123
Time 20.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 7.00 usec
TE 299.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.80 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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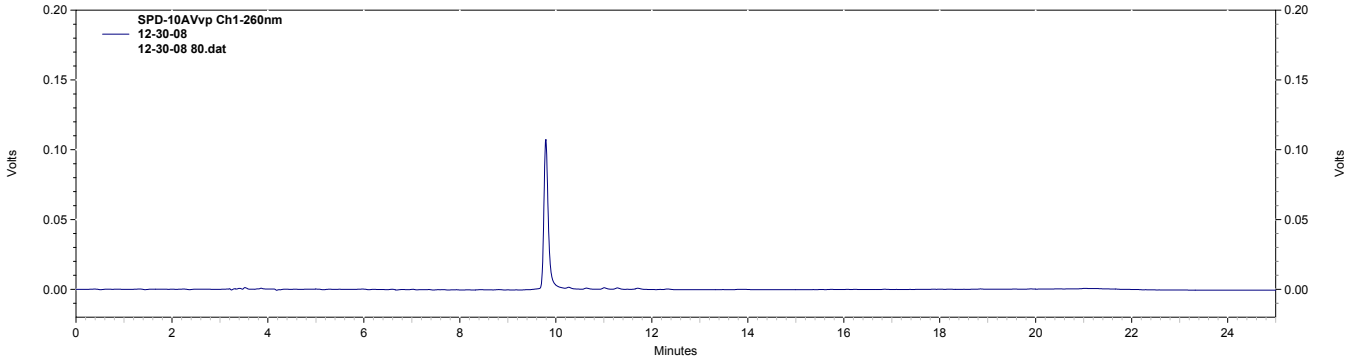

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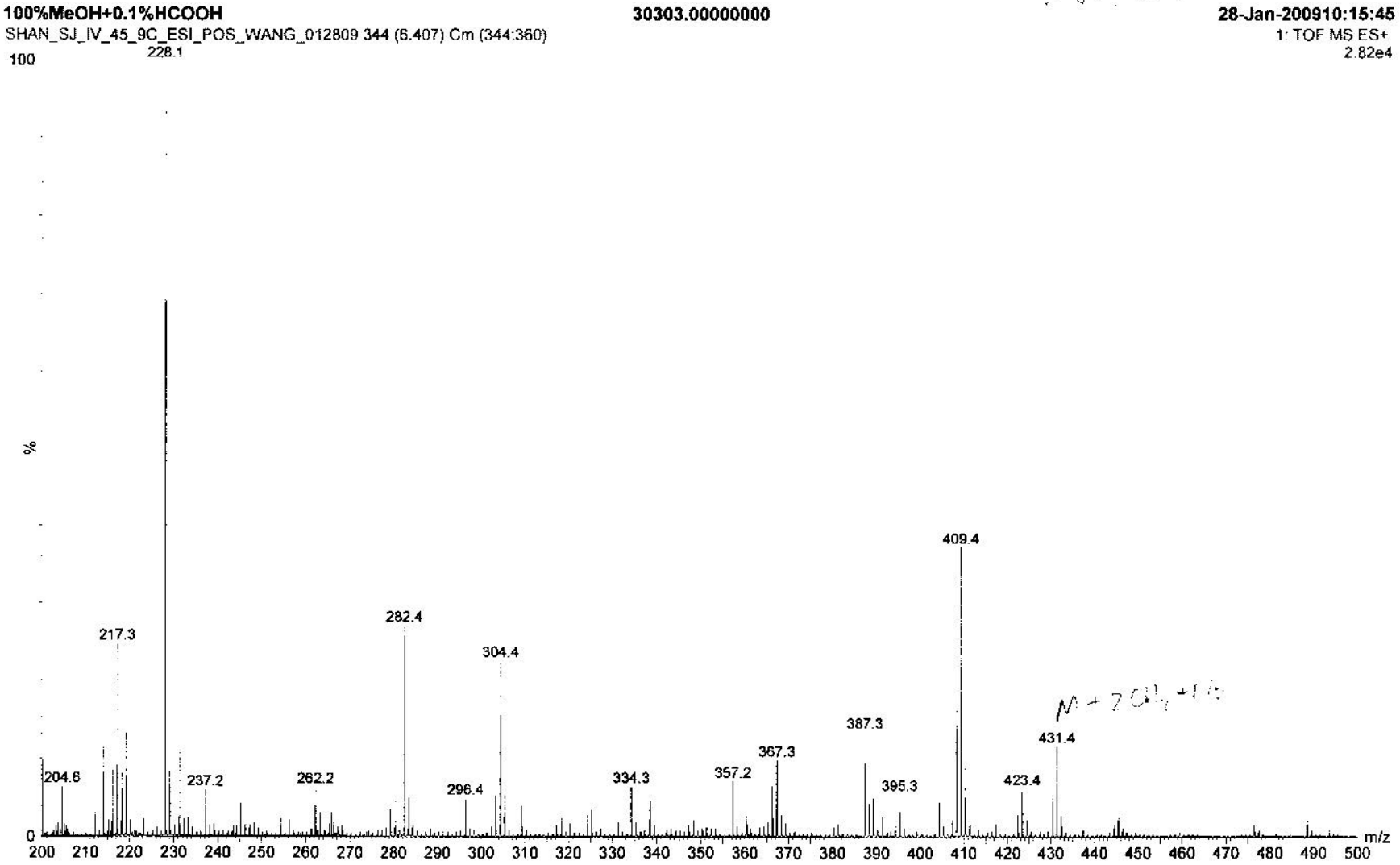

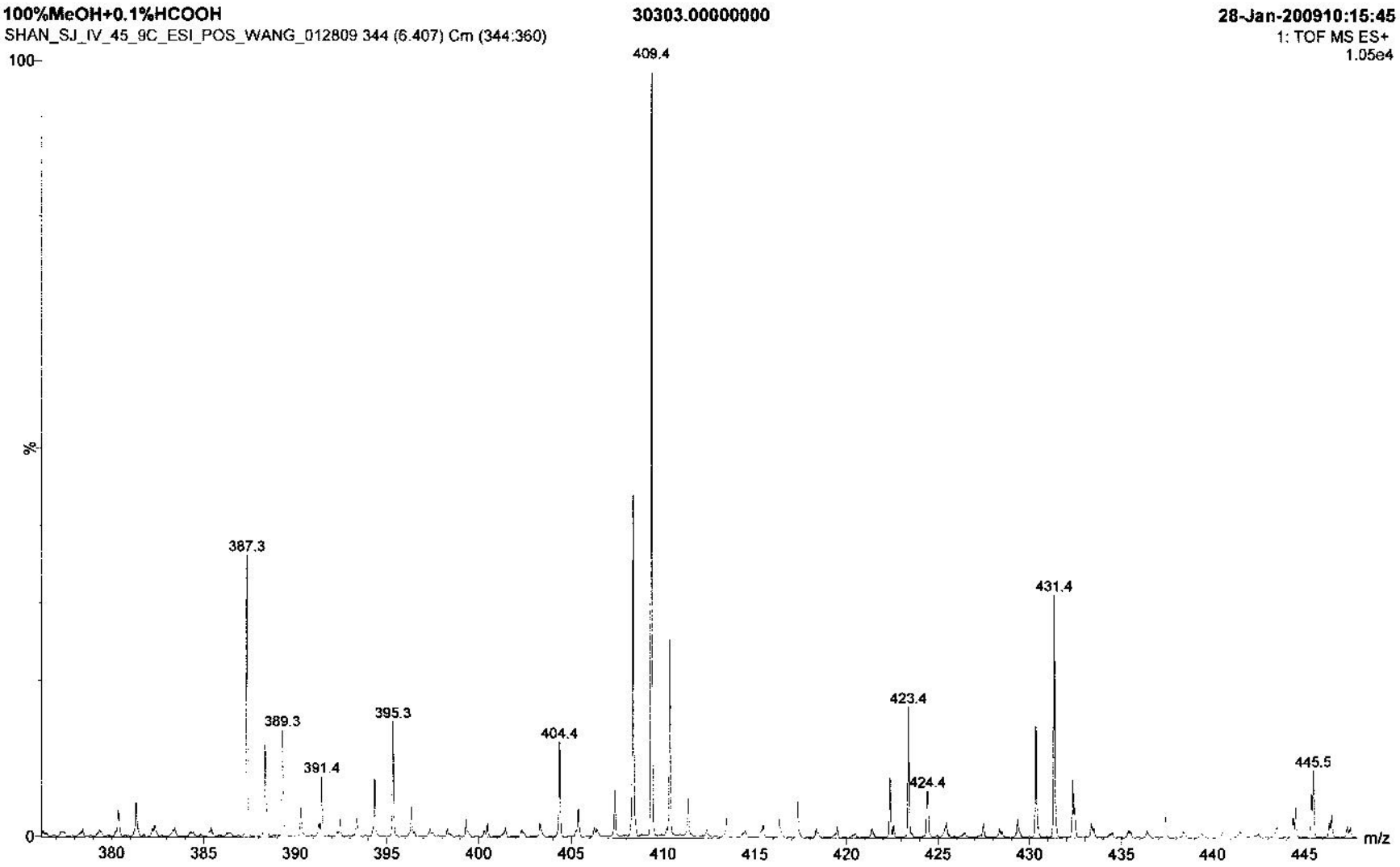

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