Supporting information

Synthesis of Thiophene-Based TAK-779 Analogues by C–H Arylation

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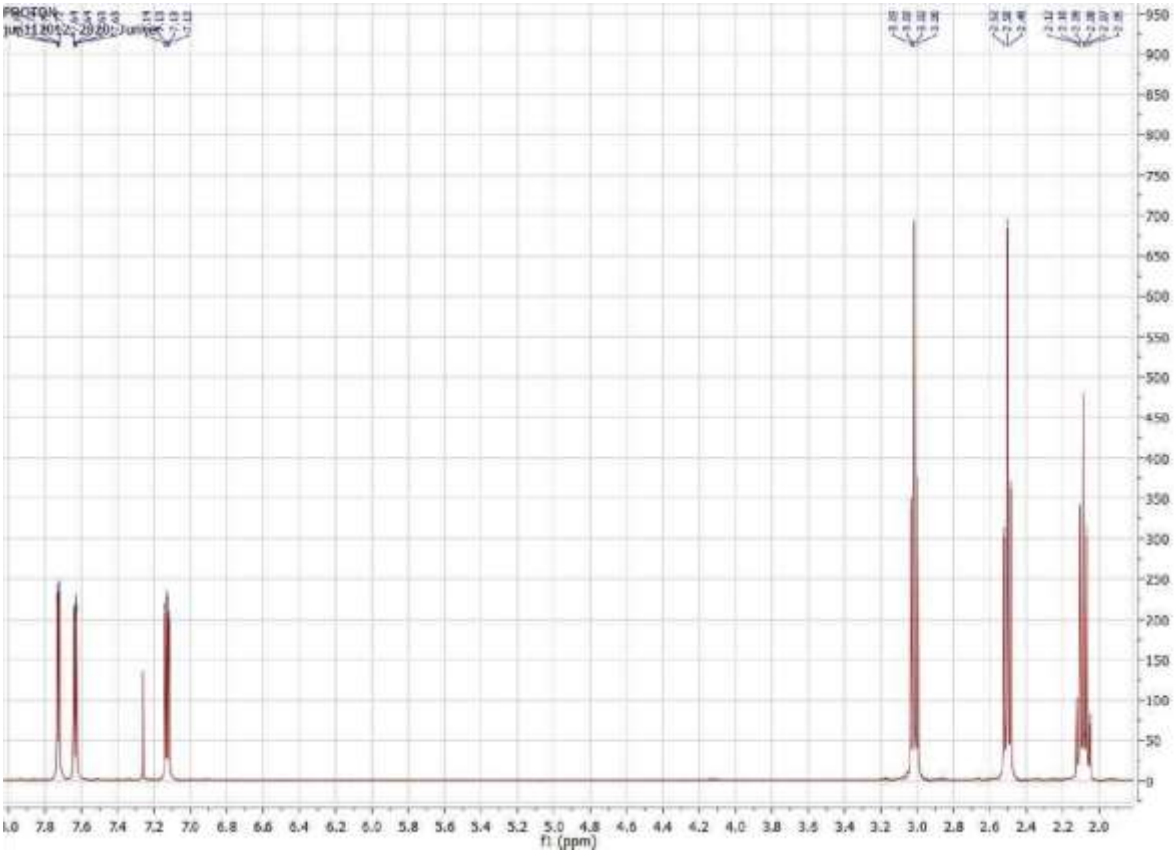
Content

5-Oxo-5-(thiophen-2-yl)pentanoic acid



HPLC

Analyzed: 08.09.10 12:00

Reported: 08.09.10 15:17
Processed: 08.09.10 15:17

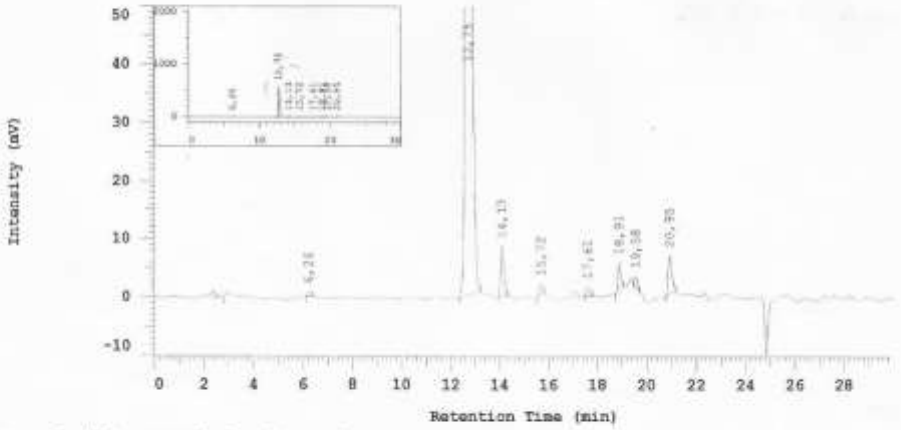
Data Path: D:\WIN32APP\HSM\Chromni\DATA\2100\
Application: Chromni

Series:2100
Vial Number: 23
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ2011

Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

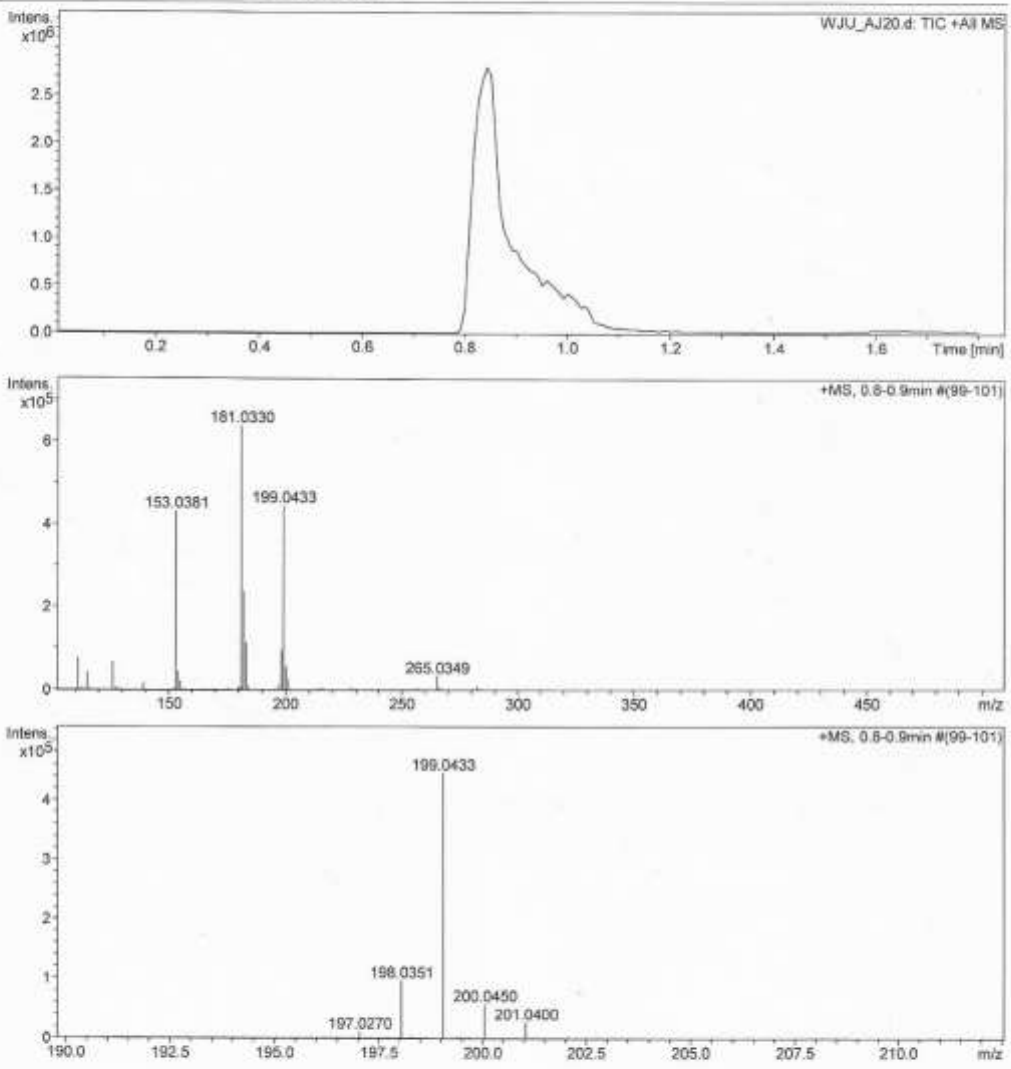
No.	RT	Area	Conc 1	BC
1	6,26	6646	0,103	MC
2	12,75	6227288	96,385	MC
3	14,13	77962	1,207	MC
4	15,72	16399	0,254	MC
5	17,61	13842	0,214	MC
6	18,91	39523	0,612	MC
7	19,58	12894	0,200	MC
8	20,95	66270	1,026	MC
		6460824	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ20.d	Acquisition Date	8/30/2012 1:22:36 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ20	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



Bruker Compass DataAnalysis 4.0 printed: 8/30/2012 1:25:48 PM Page 1 of 1

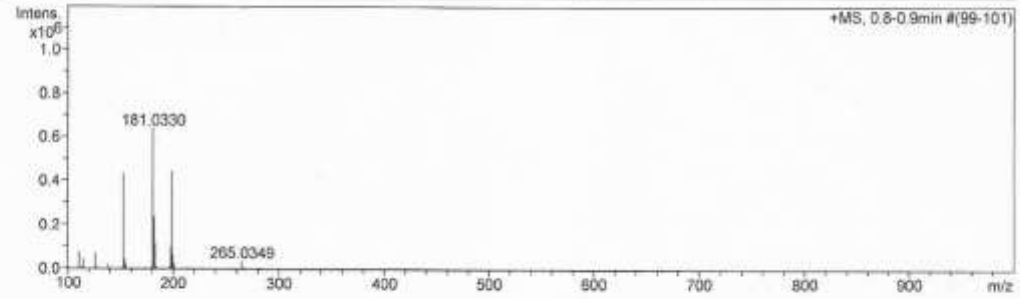
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ20.d	Acquisition Date	8/30/2012 1:22:36 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ20	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

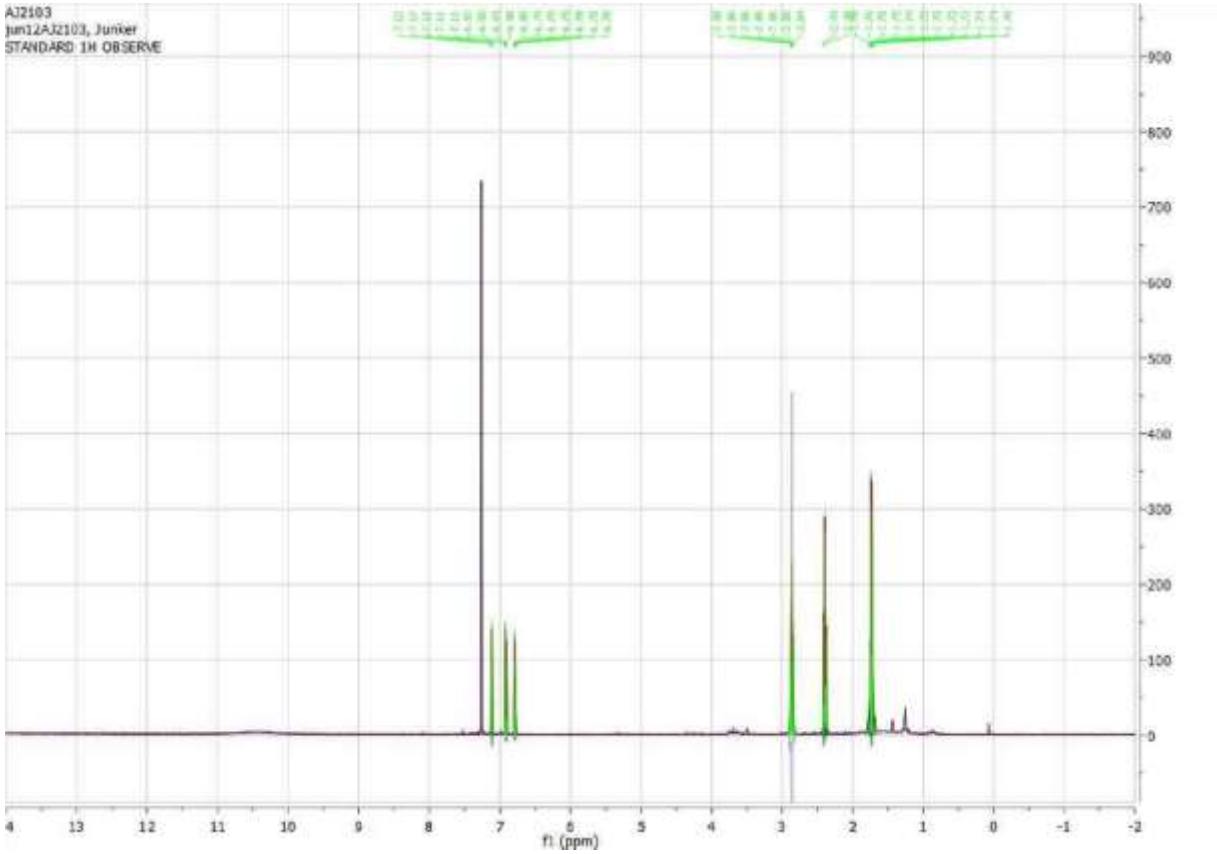
Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
199.0433	1	C 9 H 11 O 3 S	100.00	199.0423	-0.9	-4.6	11.5	4.5	even	ok
	2	C 6 H 15 O 3 S 2	22.63	199.0457	2.4	12.3	30.6	-0.5	even	ok

5-(Thiophene-2-yl)pentanoic acid (5)



HPLC

Analyzed: 11.02.10 07:23

Reported: 11.02.10 15:13
Processed: 11.02.10 15:13

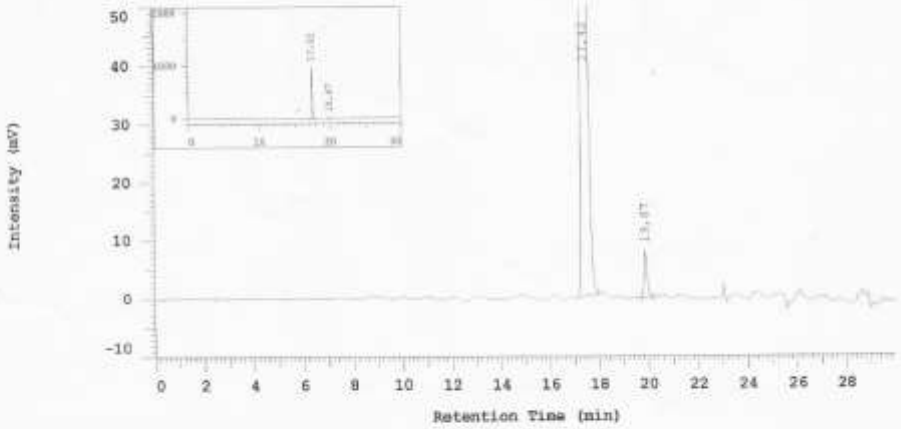
Data Path: D:\WIN32APP\HSM\Chromni\DATA\1174\
Application: Chromni

Series:1174
Vial Number: 17
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ2104

Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

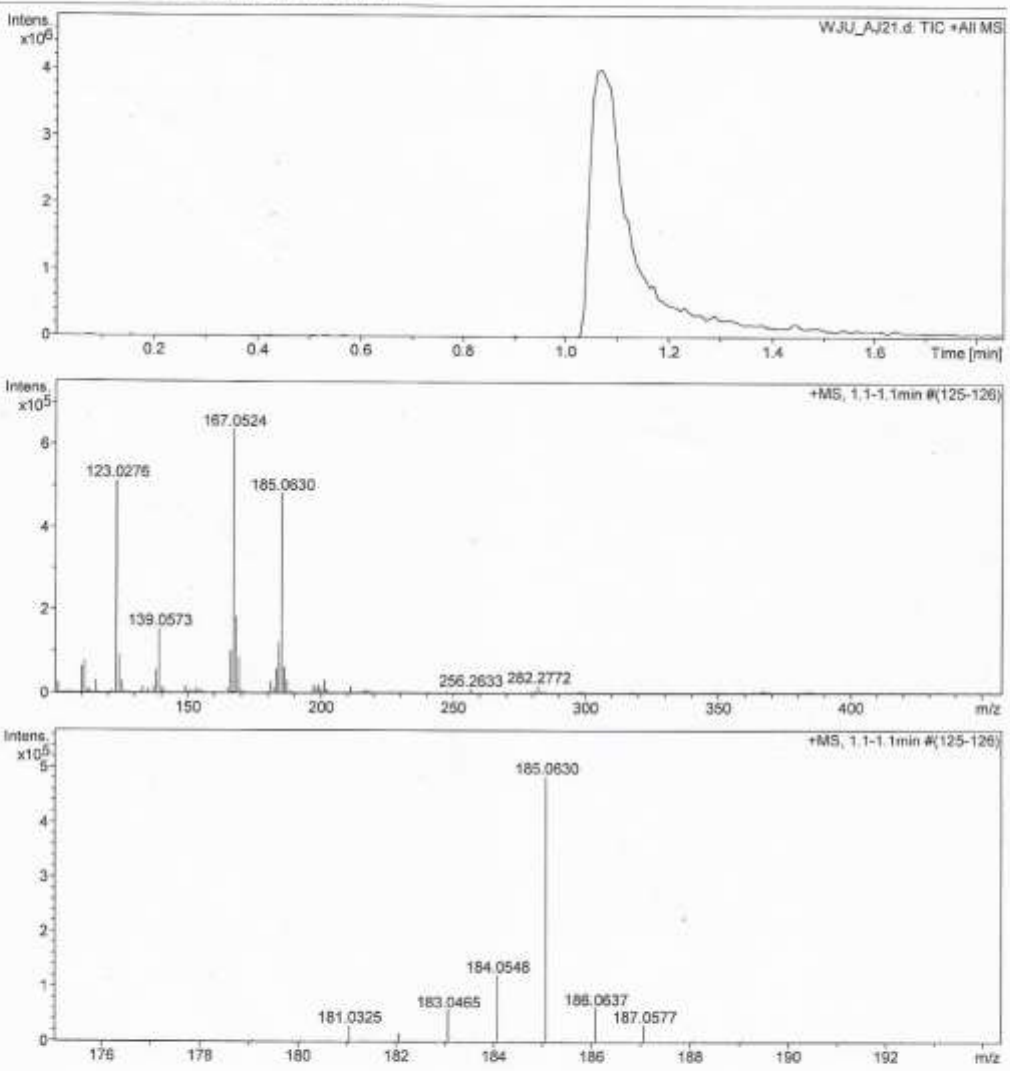
No.	RT	Area	Conc 1	BC
1	17,42	8616516	99,242	BB
2	19,87	65815	0,758	MC
		8682331	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ21.d	Acquisition Date	8/30/2012 1:35:22 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ21	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



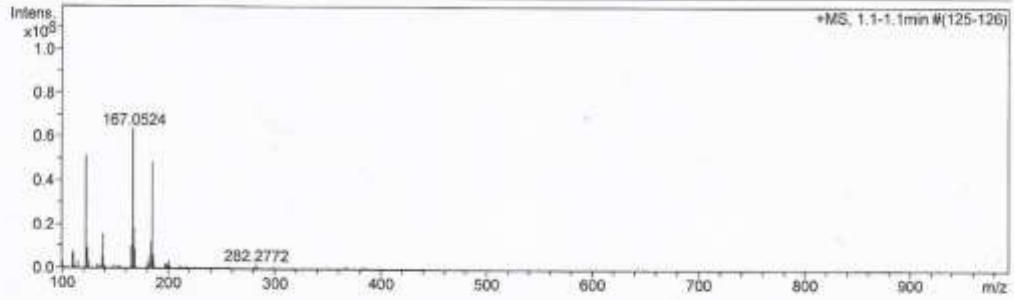
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ21.d	Acquisition Date	8/30/2012 1:35:22 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ21	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

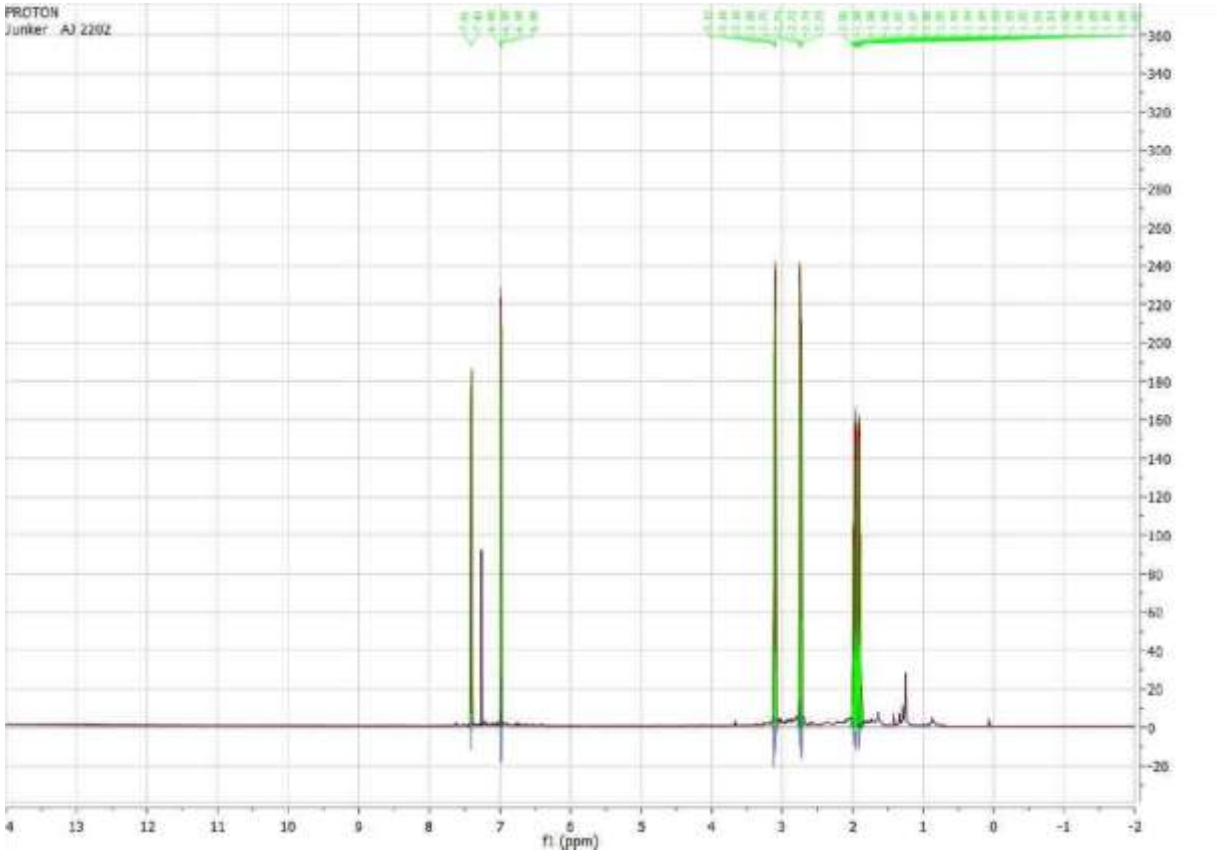
Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
185.0630	1	C 9 H 13 O 2 S	100.00	185.0631	0.1	0.6	11.8	3.5	even	ok

5,6,7,8-Tetrahydro[7]annuleno[*b*]thiophen-4-one (6)



$C_9H_{10}OS$ (166.24)

HPLC

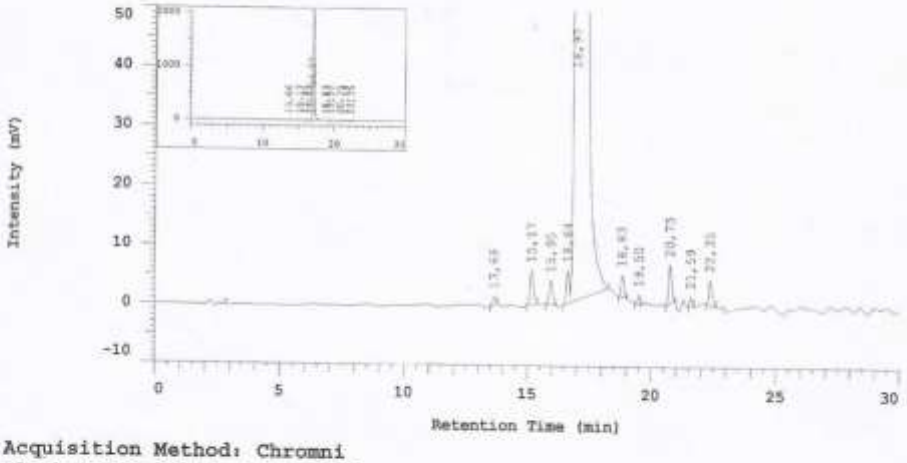
Analyzed: 20.04.11 05:59

Reported: 20.04.11 14:19
Processed: 20.04.11 14:19

Data Path: D:\WIN32APP\HSM\Chromni\DATA\2996\
Application: Chromni
Sample Name: AJ22
Injection from this vial: 1 of 1

Series:2996
Vial Number: 15
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

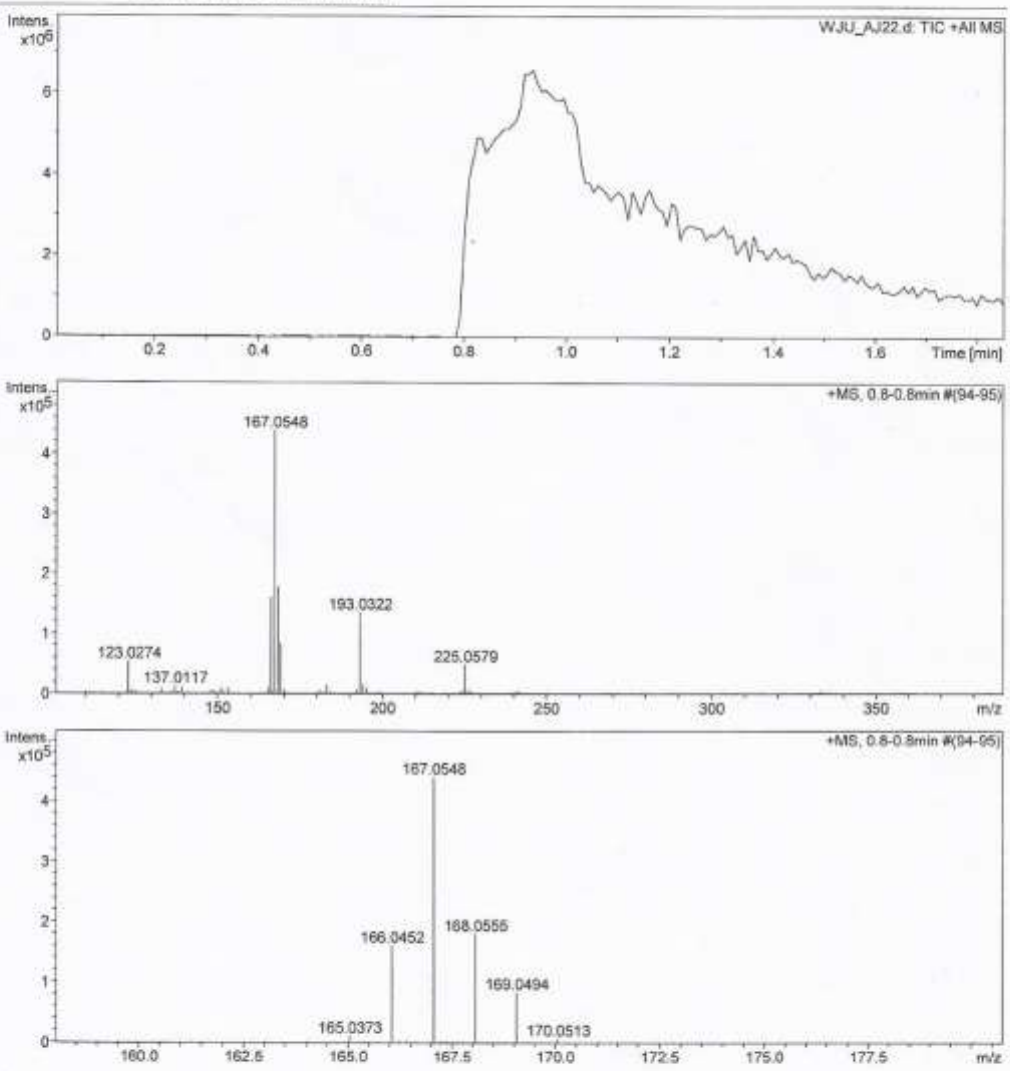
No.	RT	Area	Conc 1	BC
1	13,66	11199	0,028	BB
2	15,17	55553	0,139	MC
3	15,95	37094	0,093	MC
4	16,64	46136	0,116	MC
5	16,97	39585865	99,260	MC
6	18,83	31554	0,079	MC
7	19,50	9574	0,024	MC
8	20,75	56151	0,141	MC
9	21,59	9951	0,025	MC
10	22,35	37907	0,095	MC
		39880984	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\PMC\PharmChemie\Routine\APCI\12_08\WJU_AJ22.d	Acquisition Date	8/30/2012 1:38:03 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ22	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



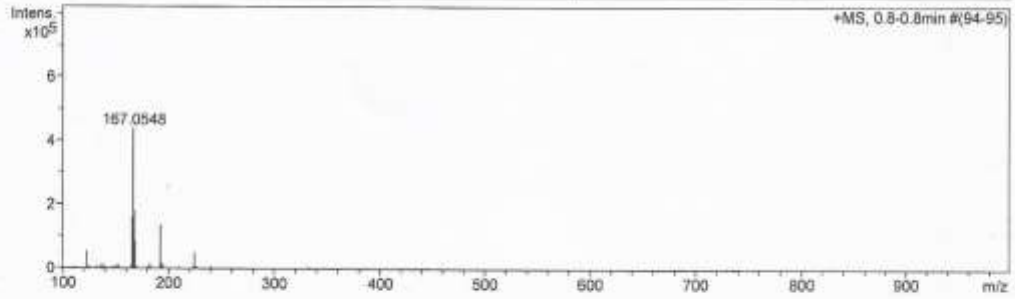
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ22.d	Acquisition Date	8/30/2012 1:38:03 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ22	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

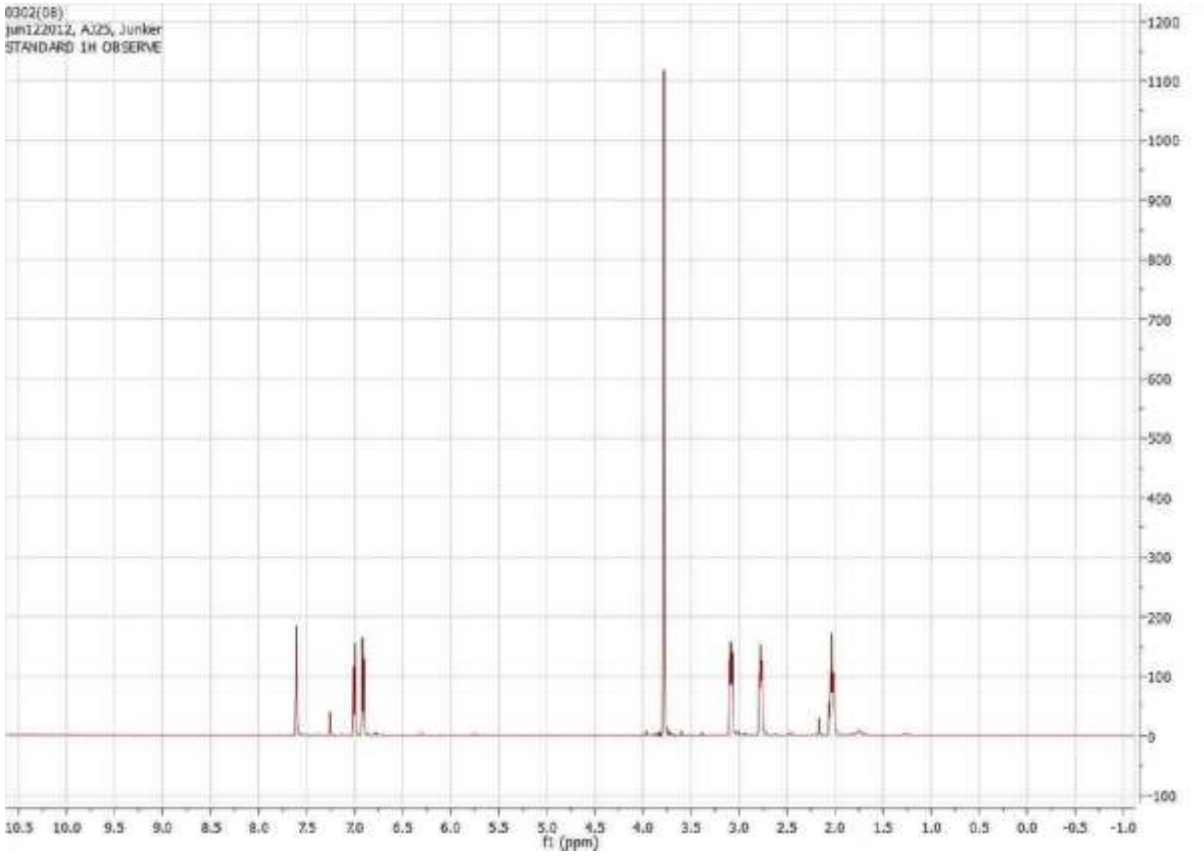
Acquisition Parameter

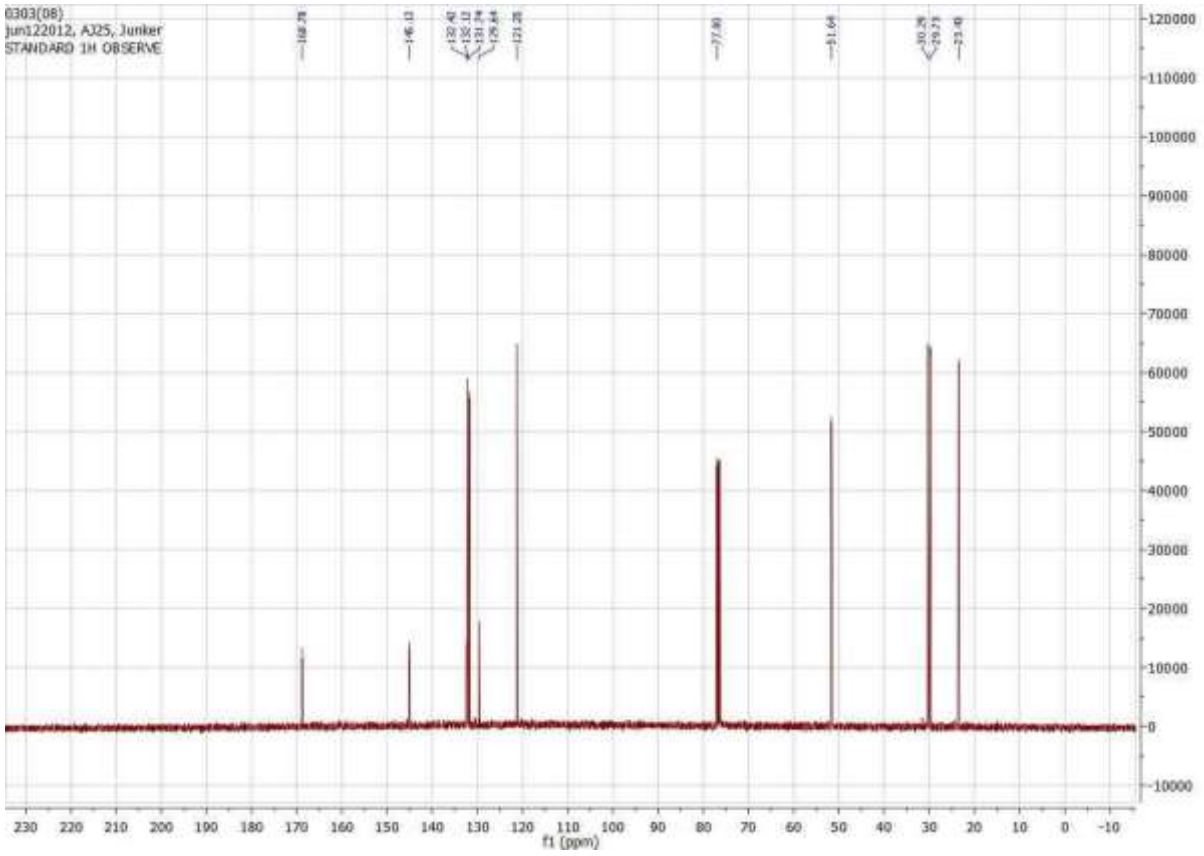
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

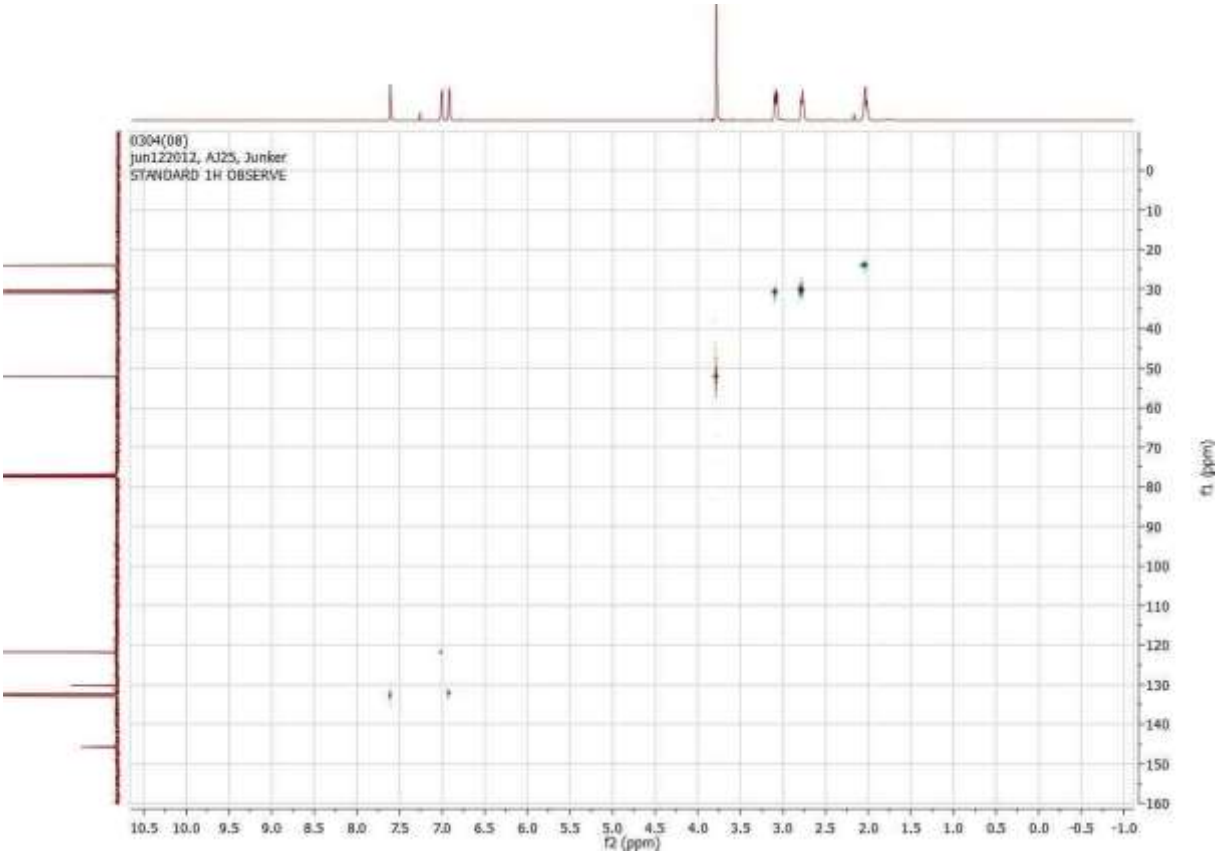


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
167.0548	1	C 9 H 11 O S	59.04	167.0525	-2.3	-13.6	166.2	4.5	even	ok
	2	C 6 H 15 O S 2	100.00	167.0559	1.1	6.5	170.4	-0.5	even	ok

Methyl 7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (2)






0304(08)
jun122012, AJ25, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 20.12.10 21:05

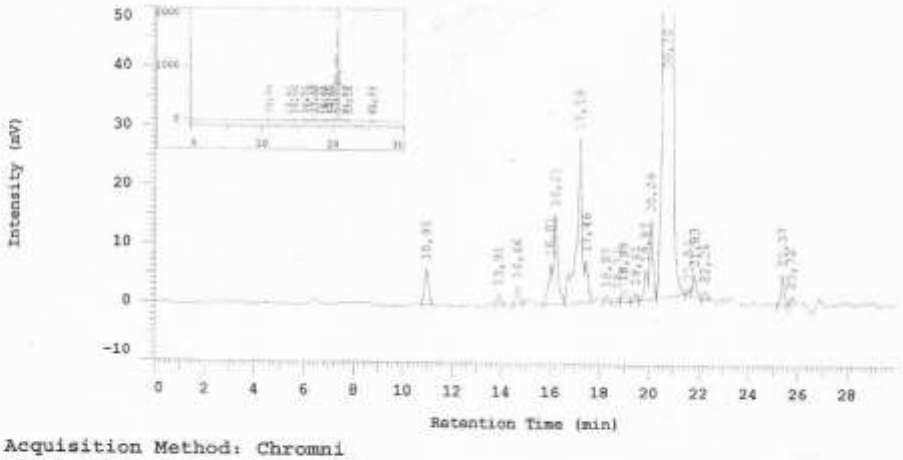
Reported: 21.12.10 14:28
Processed: 21.12.10 14:28

Data Path: D:\WIN32APP\HSM\Chromni\DATA\2457\
Application: Chromni

Series:2457
Vial Number: 4
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ2503
Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

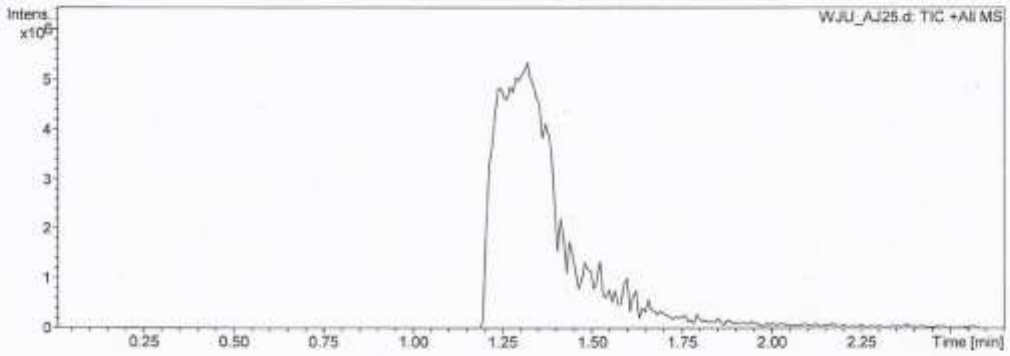
No.	RT	Area	Conc 1	BC
1	10,95	68957	0,287	BB
2	13,91	16483	0,069	BB
3	14,66	30314	0,126	BB
4	16,01	69611	0,290	BV
5	16,21	174185	0,725	VB
6	17,19	430031	1,790	BV
7	17,46	63900	0,266	MC
8	18,27	9847	0,041	BB
9	18,73	6584	0,027	MC
10	18,98	22891	0,095	MC
11	19,45	10042	0,042	BB
12	19,87	39682	0,165	BV
13	20,09	127291	0,530	VB
14	20,70	22846184	95,087	BB
15	21,59	7562	0,031	MC
16	21,83	35863	0,149	MC
17	22,26	12121	0,050	BB
18	25,37	46720	0,194	BB
19	25,72	8457	0,035	BB
		24026725	100,000	

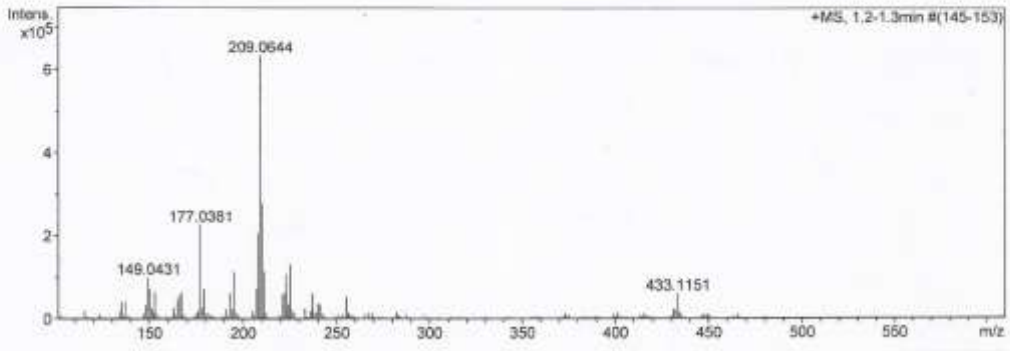
Peak rejection level: 0

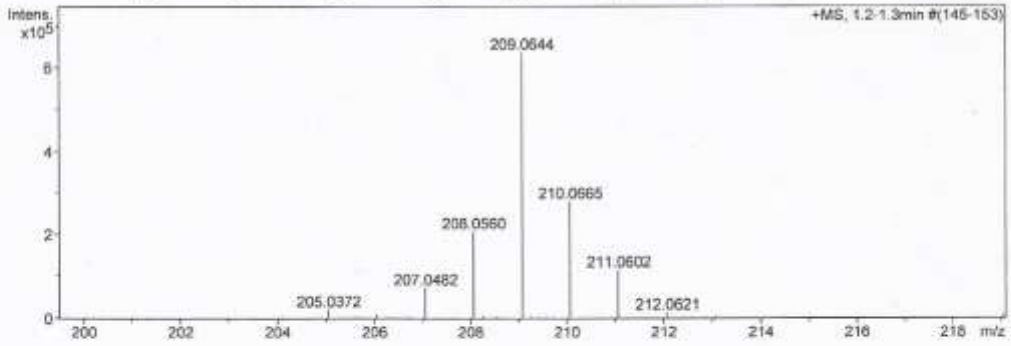
Generic Display Report

Analysis Info

Acquisition Date	12/17/2012 8:38:57 AM		
Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_12\WJU_AJ25.d		
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ25	Instrument	micrOTOF-Q II
Comment	~~Hilbig~~ APCI-Direkt Kalibration mit Fettsaeureestern		







Bruker Compass DataAnalysis 4.0 printed: 12/18/2012 10:58:19 AM Page 1 of 1

Mass Spectrum SmartFormula Report

Analysis Info

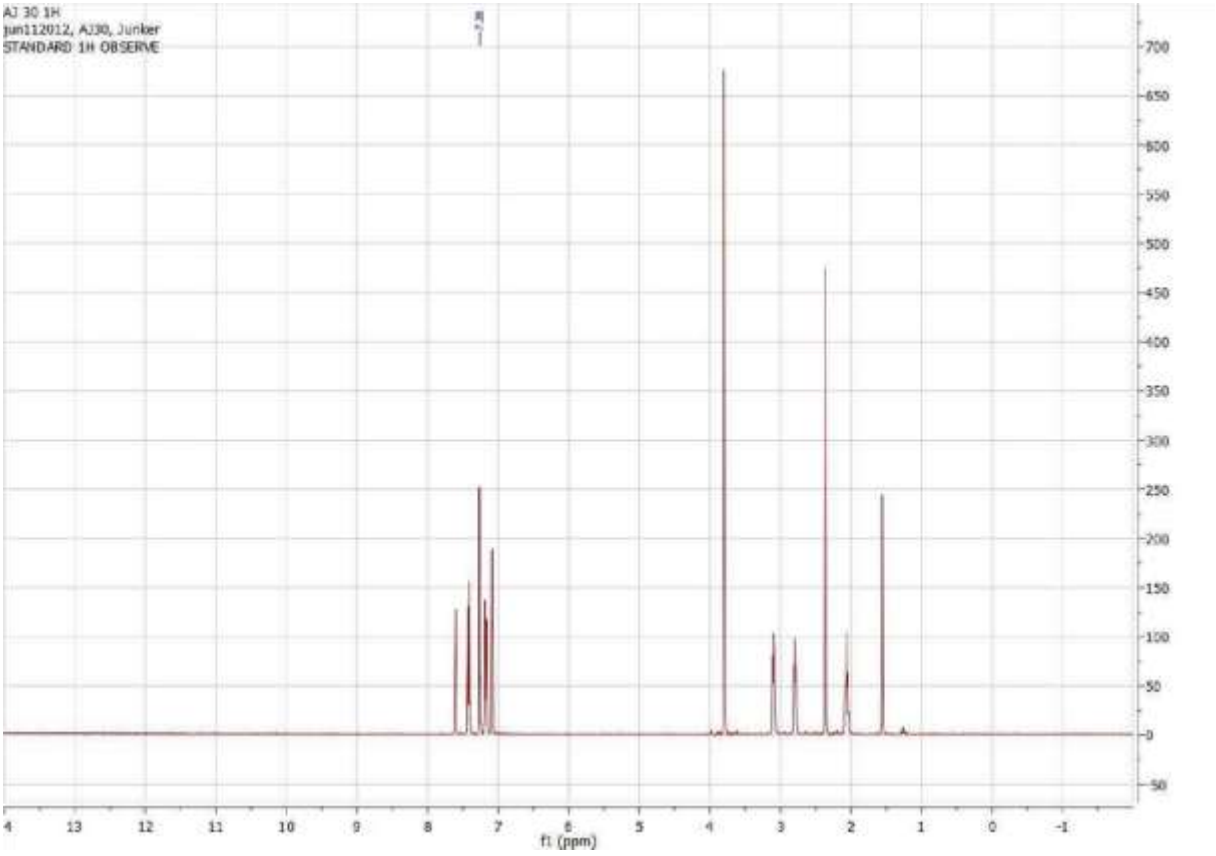
Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_12\WJU_AJ25.d	Acquisition Date	12/17/2012 8:38:57 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ25	Instrument / Ser#	micrOTOF-Q II 10252
Comment	~~Hilbig~~ Brüder APCI-Direkt Kalibration mit Fettsaeureestern		

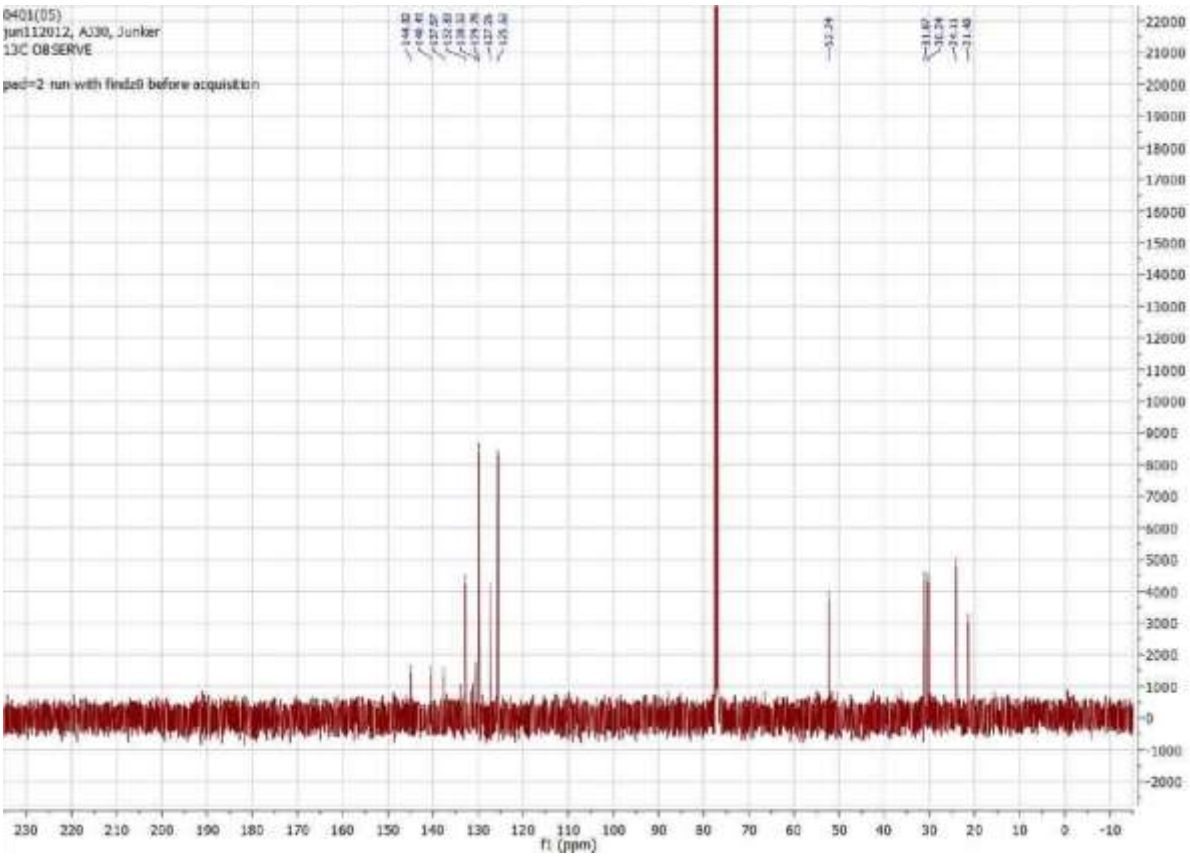
Acquisition Parameter

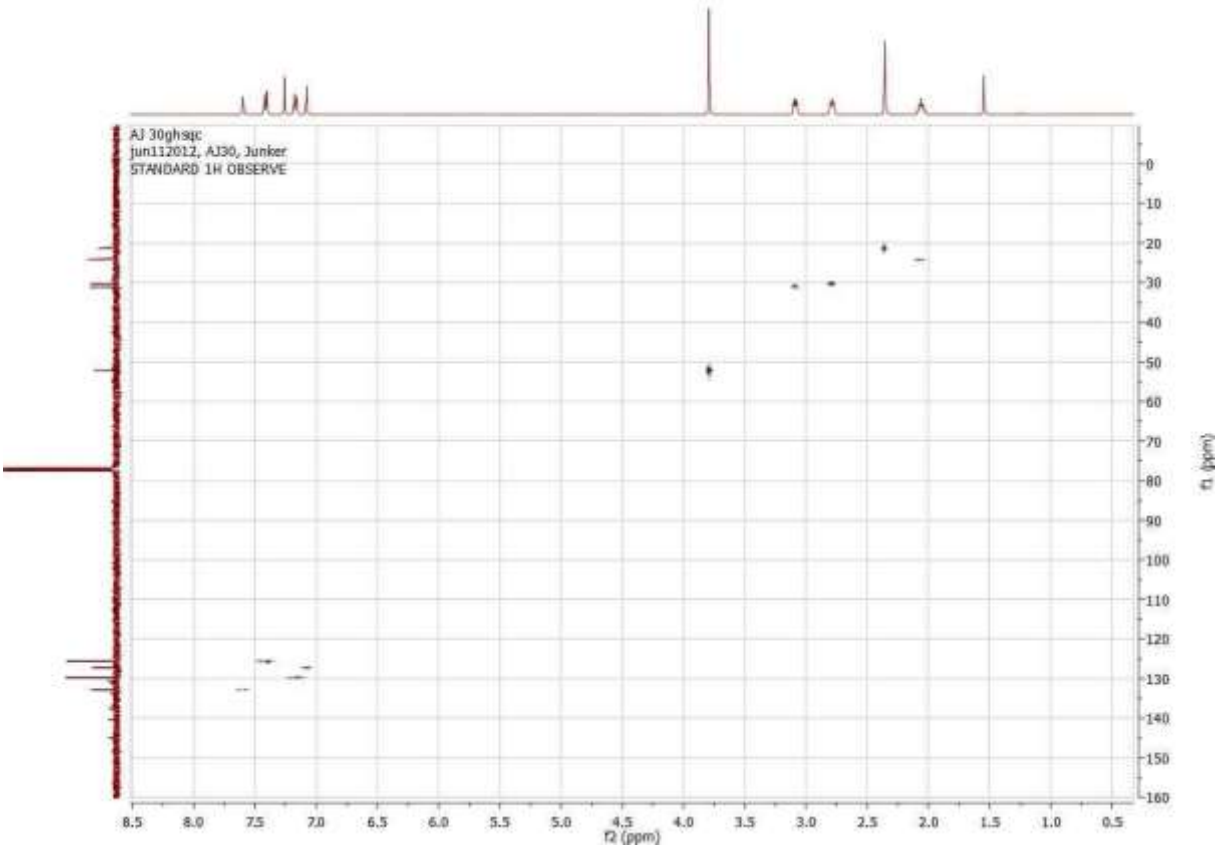
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
209.0644	1	C 11 H 13 O 2 S	100.00	209.0631	-1.3	-6.4	166.3	5.5	even	ok
	2	C 8 H 17 O 2 S 2	40.52	209.0684	2.0	9.7	172.0	0.5	even	ok
	3	C 7 H 9 N 6 S	0.41	209.0604	-4.0	-19.2	205.2	6.5	even	ok

Methyl 2-(4-methylphenyl)-7,8-dihydro-6*H*-[7]annuleno[b]thiophene-5-carboxylate (9a)






AJ 30ghsqc
jun112012, AJ30, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

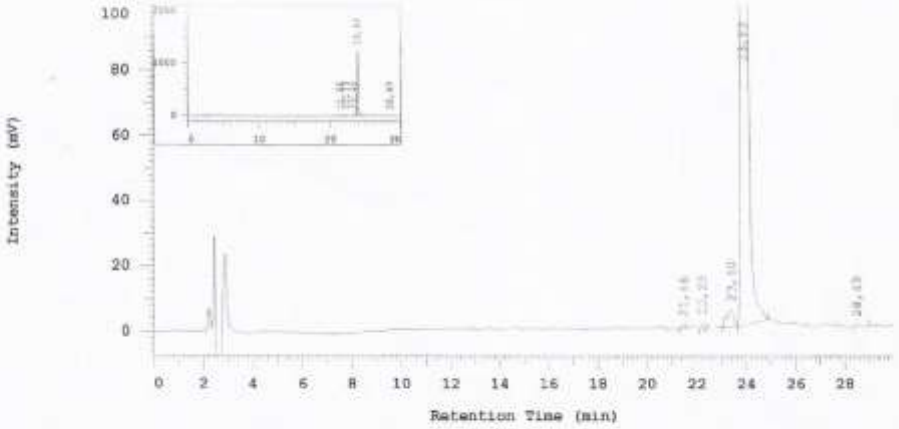
HPLC

Analyzed: 23.08.12 01:13
Reported: 24.08.12 11:17
Processed: 24.08.12 11:17

Data Path: D:\WIN32APP\HSM\Chromni\DATA\5119\
Application: Chromni
Series:5119

Sample Name: AJ30
Vial Number: 11
Vial Type: UNK
Injection from this vial: 1 of 1
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

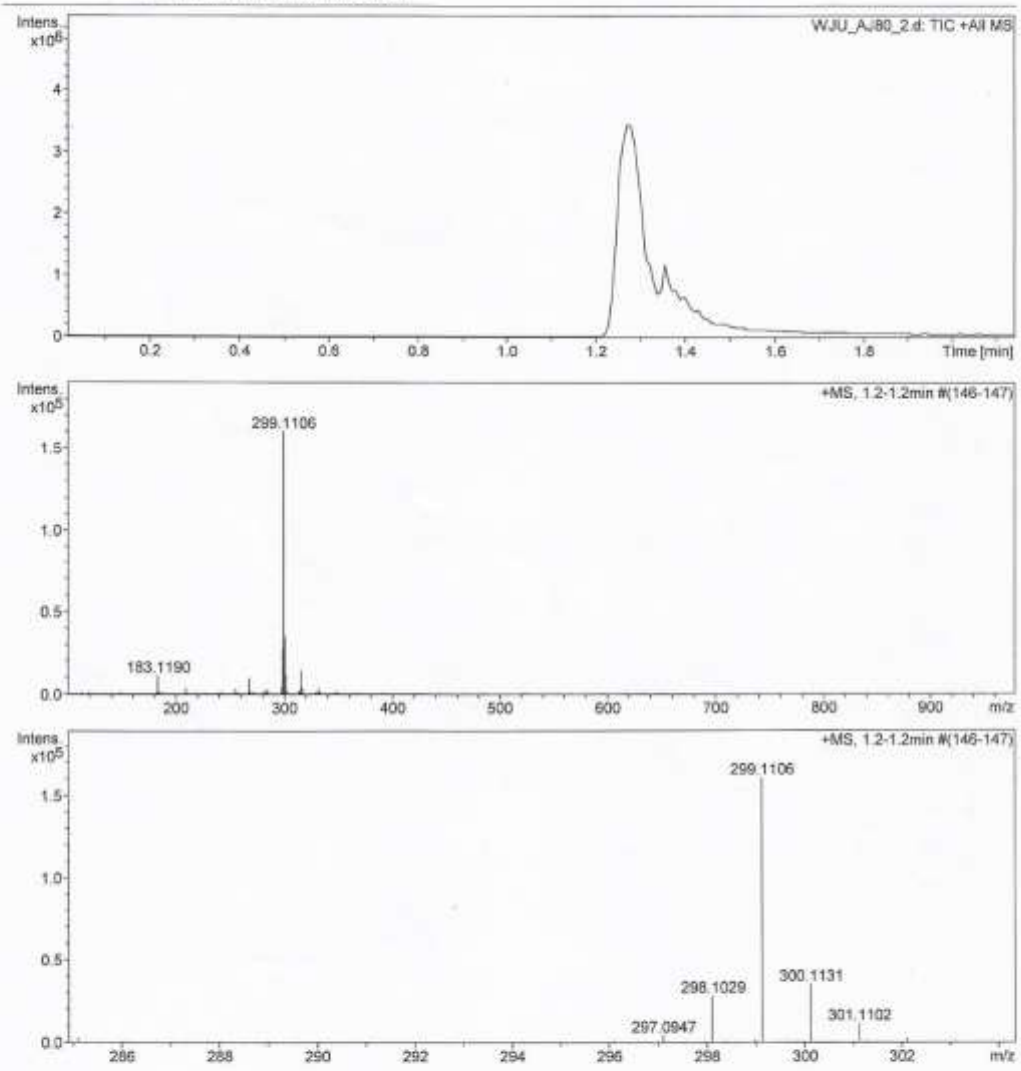
No.	RT	Area	Conc 1	BC
1	21,46	7331	0,062	BB
2	22,23	12023	0,102	BB
3	23,40	100362	0,849	BB
4	23,89	11697155	98,987	BB
5	28,49	0	0,000	
		11816871	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ80_2.d	Acquisition Date	8/28/2012 1:23:25 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	~~AJ80~~ AJ30	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



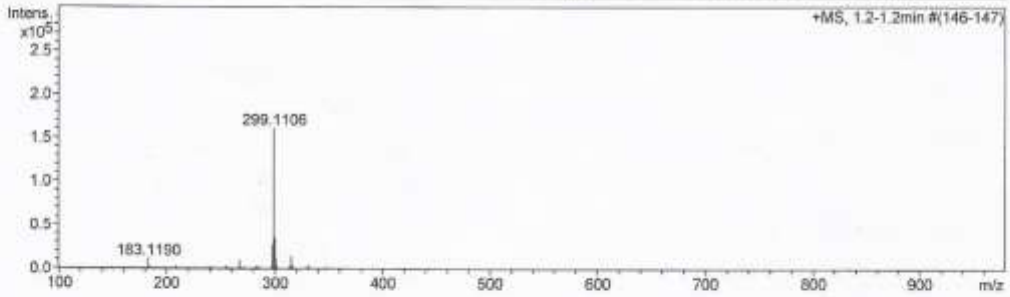
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\PMC\PharmChemie\Routine\APCI\12_08\WJU_AJ80_2.d	Acquisition Date	8/28/2012 1:23:25 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ80 AJ30	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

Acquisition Parameter

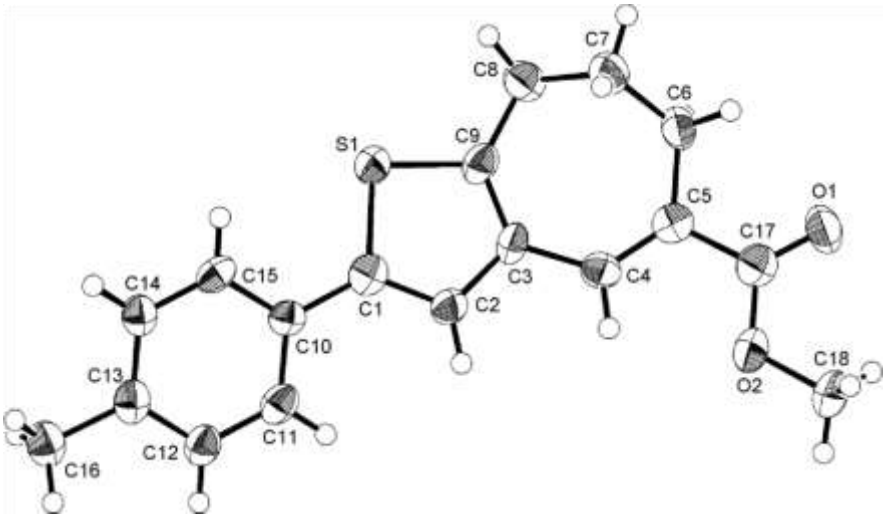
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
299.1106	1	C 18 H 19 O 2 S	100.00	299.1100	-0.6	-1.9	7.3	9.5	even	ok
	2	C 14 H 15 N 6 S	10.74	299.1073	-3.3	-10.9	18.4	10.5	even	ok
	3	C 15 H 23 O 2 S 2	13.72	299.1134	2.8	9.3	27.9	4.5	even	ok

9a was recrystallized from $CHCl_3$/pentane to give crystals, which were suitable for X-ray crystal structure analysis.

X-ray crystal structure of **9a**



Intensity data were collected at 103 K. Total 15111 reflections were corrected, of which 5638 were independent reflections (R_{int} = 0.1087). The crystal data are as follows: $C_{18}H_{18}O_2S$, *FW* = 298.38, crystal size 0.05 × 0.03 × 0.02 mm^3, monoclinic, space group *P*-1. *a* = 9.917(18) Å, *b* = 10.332(18) Å, *c* = 17.37(4) Å, *α* = 89.13(7)°, β = 88.64(7)°, *γ* = 67.30(5)°, *V* = 1642(5) $Å^3$, *Z* = 4, D_{calc} = 1.207 g/cm^3. The refinement converged to R_1 = 0.1153, wR_2 = 0.2623 (I > 2□(*I*)), R_1 = 0.1788, wR_2 = 0.3115 (for all data), *GOF* = 1.115.

Table 1. Crystal data and structure refinement for **qq** (**9a**).

Identification code	qq	
Empirical formula	C18 H18 O2 S	
Formula weight	298.38	
Temperature	103(2) K	
Wavelength	0.71070 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.917(18) Å	α= 89.13(7)°.
	b = 10.332(18) Å	β= 88.64(7)°.
	c = 17.37(4) Å	γ = 67.30(5)°.
Volume	1642(5) $Å^3$	
Z	4	

Density (calculated)	1.207 Mg/m^3
Absorption coefficient	0.199 mm^{-1}
F(000)	632
Crystal size	0.05 x 0.03 x 0.02 mm^3
Theta range for data collection	3.16 to 25.00°.
Index ranges	-11<=h<=11, -12<=k<=12, -20<=l<=20
Reflections collected	15111
Independent reflections	5638 [R(int) = 0.1087]
Completeness to theta = 25.00°	97.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9960 and 0.9901
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5638 / 0 / 383
Goodness-of-fit on F^2	1.115
Final R indices [I>2sigma(I)]	R1 = 0.1153, wR2 = 0.2623
R indices (all data)	R1 = 0.1788, wR2 = 0.3115
Largest diff. peak and hole	0.678 and -0.478 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for qq. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	9407(2)	2041(2)	8167(1)	43(1)
C(1)	7994(6)	3225(6)	8782(4)	41(2)
C(2)	7376(6)	4549(6)	8400(3)	39(2)
C(3)	8014(6)	4578(6)	7626(4)	40(2)
C(4)	7336(6)	5894(6)	7144(4)	43(2)
C(5)	7596(7)	6153(6)	6368(4)	44(2)
C(6)	8783(7)	5133(6)	5813(4)	48(2)
C(7)	9277(7)	3514(7)	5927(4)	50(2)
C(8)	10120(7)	2899(7)	6679(3)	48(2)
C(9)	9157(6)	3306(6)	7423(4)	42(2)
C(10)	7669(6)	2761(6)	9578(3)	34(1)
C(11)	6922(6)	3789(6)	10155(4)	42(2)
C(12)	6663(6)	3372(6)	10916(4)	43(2)
C(13)	7154(6)	1931(6)	11121(4)	42(2)
C(14)	7886(6)	890(7)	10543(3)	40(2)
C(15)	8142(6)	1309(6)	9784(4)	40(2)

C(16)	6987(7)	1448(7)	11966(4)	50(2)
C(17)	6695(7)	7568(7)	5992(4)	48(2)
O(1)	6858(5)	7905(5)	5315(3)	57(1)
O(2)	5633(5)	8472(5)	6492(3)	58(1)
C(18)	4617(7)	9817(7)	6134(4)	62(2)
S(2)	4693(2)	2282(2)	8336(1)	43(1)
C(19)	2929(6)	3153(6)	8790(3)	35(1)
C(20)	2029(6)	4214(6)	8304(3)	41(2)
C(21)	2758(6)	4369(6)	7570(3)	40(2)
C(22)	1954(6)	5609(6)	7034(3)	43(2)
C(23)	2456(6)	6034(6)	6364(4)	42(2)
C(24)	3940(7)	5261(7)	5934(4)	60(2)
C(25)	4668(7)	3623(7)	6037(4)	49(2)
C(26)	5371(6)	3128(7)	6841(4)	48(2)
C(27)	4204(6)	3373(7)	7498(4)	43(2)
C(28)	2641(6)	2723(6)	9595(3)	38(2)
C(29)	1154(6)	3069(6)	9892(4)	41(2)
C(30)	901(7)	2633(6)	10633(4)	42(2)
C(31)	2076(6)	1814(6)	11129(3)	39(2)
C(32)	3539(6)	1514(6)	10840(4)	44(2)
C(33)	3824(6)	1943(6)	10102(3)	41(2)
C(34)	1769(7)	1313(7)	11940(4)	49(2)
C(35)	1527(7)	7462(7)	6002(4)	46(2)
O(3)	2016(5)	8072(5)	5509(3)	60(1)
O(4)	104(4)	8047(4)	6303(2)	54(1)
C(36)	-821(7)	9465(6)	5989(4)	56(2)

Table 3. Bond lengths [Å] and angles [°] for qq.

S(1)-C(9)	1.777(7)
S(1)-C(1)	1.803(7)
C(1)-C(2)	1.426(8)
C(1)-C(10)	1.524(9)
C(2)-C(3)	1.477(8)
C(2)-H(1)	0.9500
C(3)-C(9)	1.408(8)
C(3)-C(4)	1.513(8)
C(4)-C(5)	1.408(8)
C(4)-H(2)	0.9500
C(5)-C(17)	1.533(9)
C(5)-C(6)	1.565(9)
C(6)-C(7)	1.562(9)
C(6)-H(3)	0.9900
C(6)-H(4)	0.9900
C(7)-C(8)	1.558(9)
C(7)-H(5)	0.9900
C(7)-H(6)	0.9900
C(8)-C(9)	1.551(9)
C(8)-H(7)	0.9900
C(8)-H(8)	0.9900
C(10)-C(15)	1.431(8)
C(10)-C(11)	1.440(8)
C(11)-C(12)	1.432(9)
C(11)-H(9)	0.9500
C(12)-C(13)	1.419(8)
C(12)-H(10)	0.9500
C(13)-C(14)	1.444(9)
C(13)-C(16)	1.569(9)
C(14)-C(15)	1.429(8)
C(14)-H(11)	0.9500
C(15)-H(12)	0.9500
C(16)-H(13)	0.9800
C(16)-H(14)	0.9800
C(16)-H(15)	0.9800
C(17)-O(1)	1.247(8)
C(17)-O(2)	1.399(8)

O(2)-C(18)	1.503(7)
C(18)-H(16)	0.9800
C(18)-H(17)	0.9800
C(18)-H(18)	0.9800
S(2)-C(27)	1.785(7)
S(2)-C(19)	1.798(6)
C(19)-C(20)	1.400(8)
C(19)-C(28)	1.515(8)
C(20)-C(21)	1.488(8)
C(20)-H(19)	0.9500
C(21)-C(27)	1.410(8)
C(21)-C(22)	1.535(8)
C(22)-C(23)	1.385(8)
C(22)-H(20)	0.9500
C(23)-C(35)	1.539(9)
C(23)-C(24)	1.556(9)
C(24)-C(25)	1.572(9)
C(24)-H(21)	0.9900
C(24)-H(22)	0.9900
C(25)-C(26)	1.564(9)
C(25)-H(23)	0.9900
C(25)-H(24)	0.9900
C(26)-C(27)	1.558(8)
C(26)-H(25)	0.9900
C(26)-H(26)	0.9900
C(28)-C(33)	1.448(8)
C(28)-C(29)	1.460(8)
C(29)-C(30)	1.405(8)
C(29)-H(27)	0.9500
C(30)-C(31)	1.443(8)
C(30)-H(28)	0.9500
C(31)-C(32)	1.441(8)
C(31)-C(34)	1.557(9)
C(32)-C(33)	1.408(8)
C(32)-H(29)	0.9500
C(33)-H(30)	0.9500
C(34)-H(31)	0.9800
C(34)-H(32)	0.9800
C(34)-H(33)	0.9800

C(35)-O(3)	1.252(7)
C(35)-O(4)	1.395(7)
O(4)-C(36)	1.499(7)
C(36)-H(34)	0.9800
C(36)-H(35)	0.9800
C(36)-H(36)	0.9800

C(9)-S(1)-C(1)	94.0(3)
C(2)-C(1)-C(10)	130.6(6)
C(2)-C(1)-S(1)	108.4(5)
C(10)-C(1)-S(1)	121.0(4)
C(1)-C(2)-C(3)	113.9(5)
C(1)-C(2)-H(1)	123.0
C(3)-C(2)-H(1)	123.0
C(9)-C(3)-C(2)	113.5(5)
C(9)-C(3)-C(4)	128.5(6)
C(2)-C(3)-C(4)	117.9(5)
C(5)-C(4)-C(3)	130.0(6)
C(5)-C(4)-H(2)	115.0
C(3)-C(4)-H(2)	115.0
C(4)-C(5)-C(17)	120.2(6)
C(4)-C(5)-C(6)	126.6(6)
C(17)-C(5)-C(6)	113.2(6)
C(7)-C(6)-C(5)	119.6(5)
C(7)-C(6)-H(3)	107.4
C(5)-C(6)-H(3)	107.4
C(7)-C(6)-H(4)	107.4
C(5)-C(6)-H(4)	107.4
H(3)-C(6)-H(4)	107.0
C(8)-C(7)-C(6)	115.7(6)
C(8)-C(7)-H(5)	108.4
C(6)-C(7)-H(5)	108.4
C(8)-C(7)-H(6)	108.4
C(6)-C(7)-H(6)	108.4
H(5)-C(7)-H(6)	107.4
C(9)-C(8)-C(7)	114.0(5)
C(9)-C(8)-H(7)	108.7
C(7)-C(8)-H(7)	108.7
C(9)-C(8)-H(8)	108.7

C(7)-C(8)-H(8)	108.7
H(7)-C(8)-H(8)	107.6
C(3)-C(9)-C(8)	130.2(6)
C(3)-C(9)-S(1)	110.1(5)
C(8)-C(9)-S(1)	119.7(5)
C(15)-C(10)-C(11)	118.2(6)
C(15)-C(10)-C(1)	121.6(5)
C(11)-C(10)-C(1)	120.2(5)
C(12)-C(11)-C(10)	121.0(6)
C(12)-C(11)-H(9)	119.5
C(10)-C(11)-H(9)	119.5
C(13)-C(12)-C(11)	120.6(6)
C(13)-C(12)-H(10)	119.7
C(11)-C(12)-H(10)	119.7
C(12)-C(13)-C(14)	118.9(6)
C(12)-C(13)-C(16)	121.6(6)
C(14)-C(13)-C(16)	119.4(6)
C(15)-C(14)-C(13)	120.4(6)
C(15)-C(14)-H(11)	119.8
C(13)-C(14)-H(11)	119.8
C(14)-C(15)-C(10)	120.9(6)
C(14)-C(15)-H(12)	119.5
C(10)-C(15)-H(12)	119.5
C(13)-C(16)-H(13)	109.5
C(13)-C(16)-H(14)	109.5
H(13)-C(16)-H(14)	109.5
C(13)-C(16)-H(15)	109.5
H(13)-C(16)-H(15)	109.5
H(14)-C(16)-H(15)	109.5
O(1)-C(17)-O(2)	122.2(6)
O(1)-C(17)-C(5)	125.0(6)
O(2)-C(17)-C(5)	112.8(6)
C(17)-O(2)-C(18)	115.2(5)
O(2)-C(18)-H(16)	109.5
O(2)-C(18)-H(17)	109.5
H(16)-C(18)-H(17)	109.5
O(2)-C(18)-H(18)	109.5
H(16)-C(18)-H(18)	109.5
H(17)-C(18)-H(18)	109.5

C(27)-S(2)-C(19)	93.0(3)
C(20)-C(19)-C(28)	130.4(5)
C(20)-C(19)-S(2)	109.8(4)
C(28)-C(19)-S(2)	119.8(4)
C(19)-C(20)-C(21)	114.0(5)
C(19)-C(20)-H(19)	123.0
C(21)-C(20)-H(19)	123.0
C(27)-C(21)-C(20)	112.6(5)
C(27)-C(21)-C(22)	128.2(5)
C(20)-C(21)-C(22)	119.0(5)
C(23)-C(22)-C(21)	129.1(6)
C(23)-C(22)-H(20)	115.4
C(21)-C(22)-H(20)	115.4
C(22)-C(23)-C(35)	119.0(5)
C(22)-C(23)-C(24)	127.3(5)
C(35)-C(23)-C(24)	113.6(5)
C(23)-C(24)-C(25)	116.9(5)
C(23)-C(24)-H(21)	108.1
C(25)-C(24)-H(21)	108.1
C(23)-C(24)-H(22)	108.1
C(25)-C(24)-H(22)	108.1
H(21)-C(24)-H(22)	107.3
C(26)-C(25)-C(24)	114.2(6)
C(26)-C(25)-H(23)	108.7
C(24)-C(25)-H(23)	108.7
C(26)-C(25)-H(24)	108.7
C(24)-C(25)-H(24)	108.7
H(23)-C(25)-H(24)	107.6
C(27)-C(26)-C(25)	112.5(5)
C(27)-C(26)-H(25)	109.1
C(25)-C(26)-H(25)	109.1
C(27)-C(26)-H(26)	109.1
C(25)-C(26)-H(26)	109.1
H(25)-C(26)-H(26)	107.8
C(21)-C(27)-C(26)	130.9(6)
C(21)-C(27)-S(2)	110.6(5)
C(26)-C(27)-S(2)	118.5(5)
C(33)-C(28)-C(29)	117.0(5)
C(33)-C(28)-C(19)	121.7(5)

C(29)-C(28)-C(19)	121.3(5)
C(30)-C(29)-C(28)	120.8(6)
C(30)-C(29)-H(27)	119.6
C(28)-C(29)-H(27)	119.6
C(29)-C(30)-C(31)	122.3(5)
C(29)-C(30)-H(28)	118.8
C(31)-C(30)-H(28)	118.8
C(32)-C(31)-C(30)	116.4(6)
C(32)-C(31)-C(34)	122.1(5)
C(30)-C(31)-C(34)	121.5(5)
C(33)-C(32)-C(31)	122.3(6)
C(33)-C(32)-H(29)	118.8
C(31)-C(32)-H(29)	118.8
C(32)-C(33)-C(28)	121.0(5)
C(32)-C(33)-H(30)	119.5
C(28)-C(33)-H(30)	119.5
C(31)-C(34)-H(31)	109.5
C(31)-C(34)-H(32)	109.5
H(31)-C(34)-H(32)	109.5
C(31)-C(34)-H(33)	109.5
H(31)-C(34)-H(33)	109.5
H(32)-C(34)-H(33)	109.5
O(3)-C(35)-O(4)	122.9(6)
O(3)-C(35)-C(23)	123.4(6)
O(4)-C(35)-C(23)	113.7(5)
C(35)-O(4)-C(36)	115.4(5)
O(4)-C(36)-H(34)	109.5
O(4)-C(36)-H(35)	109.5
H(34)-C(36)-H(35)	109.5
O(4)-C(36)-H(36)	109.5
H(34)-C(36)-H(36)	109.5
H(35)-C(36)-H(36)	109.5

Symmetry transformations used to generate equivalent atoms.

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for qq. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S(1)	39(1)	40(1)	43(1)	5(1)	0(1)	-9(1)
C(1)	31(3)	45(4)	48(4)	2(3)	-8(3)	-17(3)
C(2)	33(3)	36(3)	42(4)	2(3)	1(3)	-8(3)
C(3)	37(3)	34(3)	45(4)	12(3)	-8(3)	-11(3)
C(4)	35(3)	46(4)	45(4)	-1(3)	7(3)	-13(3)
C(5)	41(4)	43(4)	49(4)	-2(3)	-1(3)	-17(3)
C(6)	51(4)	52(4)	38(4)	11(3)	-2(3)	-17(3)
C(7)	45(4)	55(4)	47(4)	5(3)	3(3)	-18(3)
C(8)	47(4)	55(4)	42(4)	-3(3)	1(3)	-18(3)
C(9)	32(3)	38(3)	51(4)	8(3)	-6(3)	-8(3)
C(10)	24(3)	40(3)	37(4)	-2(3)	-4(3)	-11(3)
C(11)	31(3)	38(3)	55(4)	5(3)	-5(3)	-11(3)
C(12)	38(3)	41(4)	47(4)	6(3)	-5(3)	-12(3)
C(13)	30(3)	51(4)	48(4)	12(3)	-7(3)	-17(3)
C(14)	33(3)	46(4)	42(4)	9(3)	-3(3)	-16(3)
C(15)	29(3)	34(3)	51(4)	-1(3)	-6(3)	-6(3)
C(16)	40(4)	62(4)	47(4)	11(3)	-3(3)	-19(3)
C(17)	42(4)	49(4)	52(5)	1(4)	-4(3)	-17(3)
O(1)	70(3)	64(3)	40(3)	12(2)	-1(2)	-27(3)
O(2)	57(3)	48(3)	53(3)	16(2)	-2(2)	-4(2)
C(18)	62(5)	51(4)	55(5)	15(4)	-14(4)	-3(4)
S(2)	31(1)	46(1)	48(1)	9(1)	-2(1)	-11(1)
C(19)	27(3)	44(4)	37(4)	5(3)	-3(3)	-17(3)
C(20)	29(3)	42(4)	45(4)	-2(3)	4(3)	-5(3)
C(21)	37(3)	44(4)	40(4)	0(3)	-1(3)	-19(3)
C(22)	33(3)	47(4)	42(4)	6(3)	-9(3)	-8(3)
C(23)	42(4)	36(3)	45(4)	4(3)	-2(3)	-12(3)
C(24)	46(4)	73(5)	53(5)	20(4)	4(3)	-15(4)
C(25)	44(4)	52(4)	45(4)	11(3)	-2(3)	-15(3)
C(26)	38(4)	50(4)	57(5)	7(3)	1(3)	-18(3)
C(27)	32(3)	57(4)	40(4)	1(3)	1(3)	-17(3)
C(28)	34(3)	32(3)	46(4)	6(3)	-4(3)	-12(3)
C(29)	32(3)	40(3)	52(4)	6(3)	-6(3)	-14(3)
C(30)	37(3)	42(4)	46(4)	-5(3)	4(3)	-13(3)

C(31)	39(4)	35(3)	39(4)	1(3)	4(3)	-11(3)
C(32)	35(3)	37(4)	53(4)	5(3)	0(3)	-6(3)
C(33)	30(3)	47(4)	44(4)	9(3)	0(3)	-13(3)
C(34)	49(4)	47(4)	54(4)	1(3)	3(3)	-22(3)
C(35)	47(4)	47(4)	43(4)	0(3)	5(3)	-17(3)
O(3)	51(3)	67(3)	65(3)	28(3)	-1(2)	-27(3)
O(4)	44(3)	50(3)	52(3)	7(2)	10(2)	-2(2)
C(36)	48(4)	41(4)	66(5)	13(3)	-1(4)	-2(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for qq.

	x	y	z	U(eq)
H(1)	6622	5341	8623	46
H(2)	6624	6666	7406	51
H(3)	8418	5360	5282	58
H(4)	9666	5356	5842	58
H(5)	8399	3280	5920	60
H(6)	9908	3041	5482	60
H(7)	10558	1863	6641	58
H(8)	10929	3229	6721	58
H(9)	6595	4758	10029	51
H(10)	6159	4067	11287	51
H(11)	8198	-78	10669	48
H(12)	8634	613	9412	48
H(13)	6813	577	11954	75
H(14)	6159	2178	12225	75
H(15)	7883	1287	12247	75
H(16)	3901	9630	5823	93
H(17)	5180	10212	5804	93
H(18)	4106	10487	6540	93
H(19)	1043	4784	8433	49
H(20)	973	6164	7181	51
H(21)	4641	5661	6108	72
H(22)	3791	5471	5378	72
H(23)	3917	3228	5960	58

H(24)	5433	3236	5632	58
H(25)	6000	2117	6818	58
H(26)	5999	3644	6961	58
H(27)	348	3595	9582	50
H(28)	-79	2887	10813	51
H(29)	4340	1008	11159	53
H(30)	4806	1718	9934	49
H(31)	1787	1982	12329	73
H(32)	2520	386	12052	73
H(33)	805	1254	11948	73
H(34)	-347	10122	6081	84
H(35)	-1783	9803	6246	84
H(36)	-936	9397	5434	84

Table 6. Torsion angles [°] for qq.

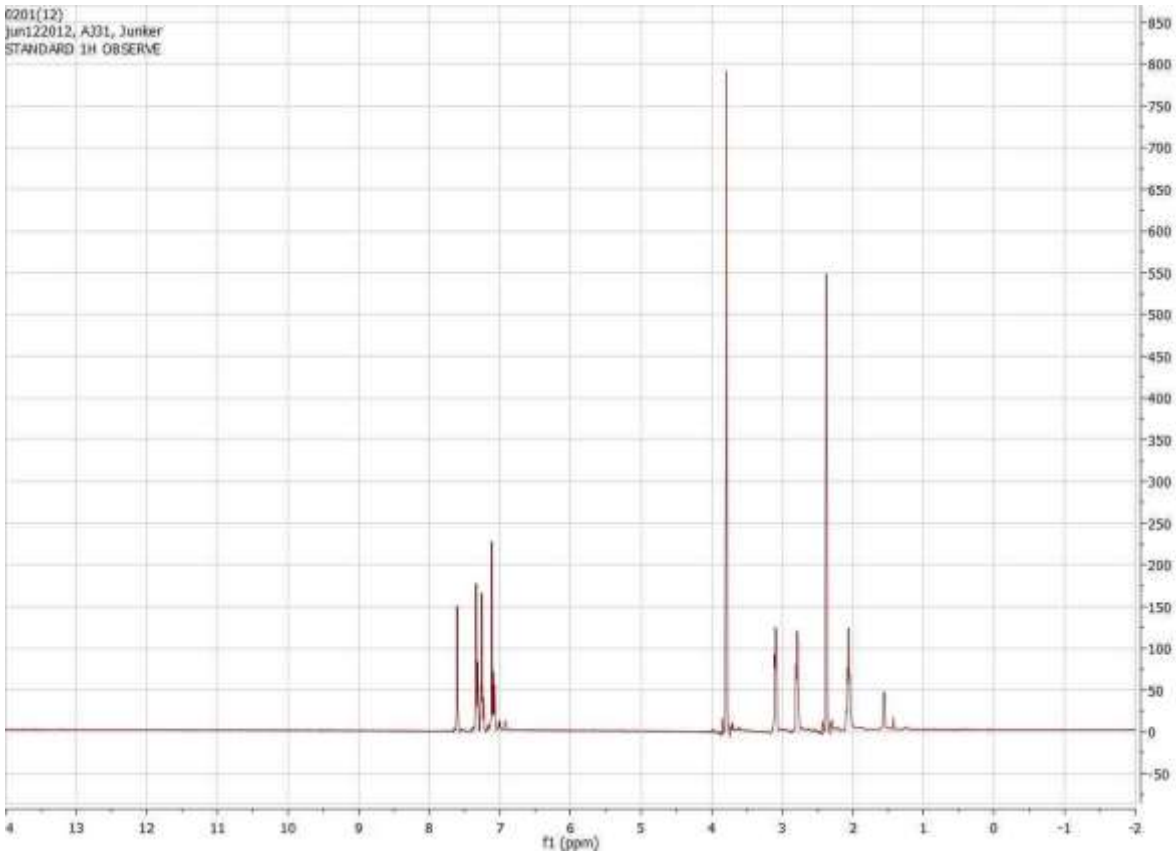
C(9)-S(1)-C(1)-C(2)	0.0(4)
C(9)-S(1)-C(1)-C(10)	-178.0(4)
C(10)-C(1)-C(2)-C(3)	179.1(5)
S(1)-C(1)-C(2)-C(3)	1.3(6)
C(1)-C(2)-C(3)-C(9)	-2.5(7)
C(1)-C(2)-C(3)-C(4)	174.4(5)
C(9)-C(3)-C(4)-C(5)	4.8(11)
C(2)-C(3)-C(4)-C(5)	-171.5(6)
C(3)-C(4)-C(5)-C(17)	176.5(5)
C(3)-C(4)-C(5)-C(6)	-5.2(10)
C(4)-C(5)-C(6)-C(7)	30.0(9)
C(17)-C(5)-C(6)-C(7)	-151.6(5)
C(5)-C(6)-C(7)-C(8)	-66.8(8)
C(6)-C(7)-C(8)-C(9)	70.4(7)
C(2)-C(3)-C(9)-C(8)	-179.3(6)
C(4)-C(3)-C(9)-C(8)	4.3(11)
C(2)-C(3)-C(9)-S(1)	2.4(6)
C(4)-C(3)-C(9)-S(1)	-174.1(5)
C(7)-C(8)-C(9)-C(3)	-38.3(9)
C(7)-C(8)-C(9)-S(1)	140.0(5)
C(1)-S(1)-C(9)-C(3)	-1.4(5)
C(1)-S(1)-C(9)-C(8)	-180.0(5)
C(2)-C(1)-C(10)-C(15)	164.3(6)
S(1)-C(1)-C(10)-C(15)	-18.2(7)

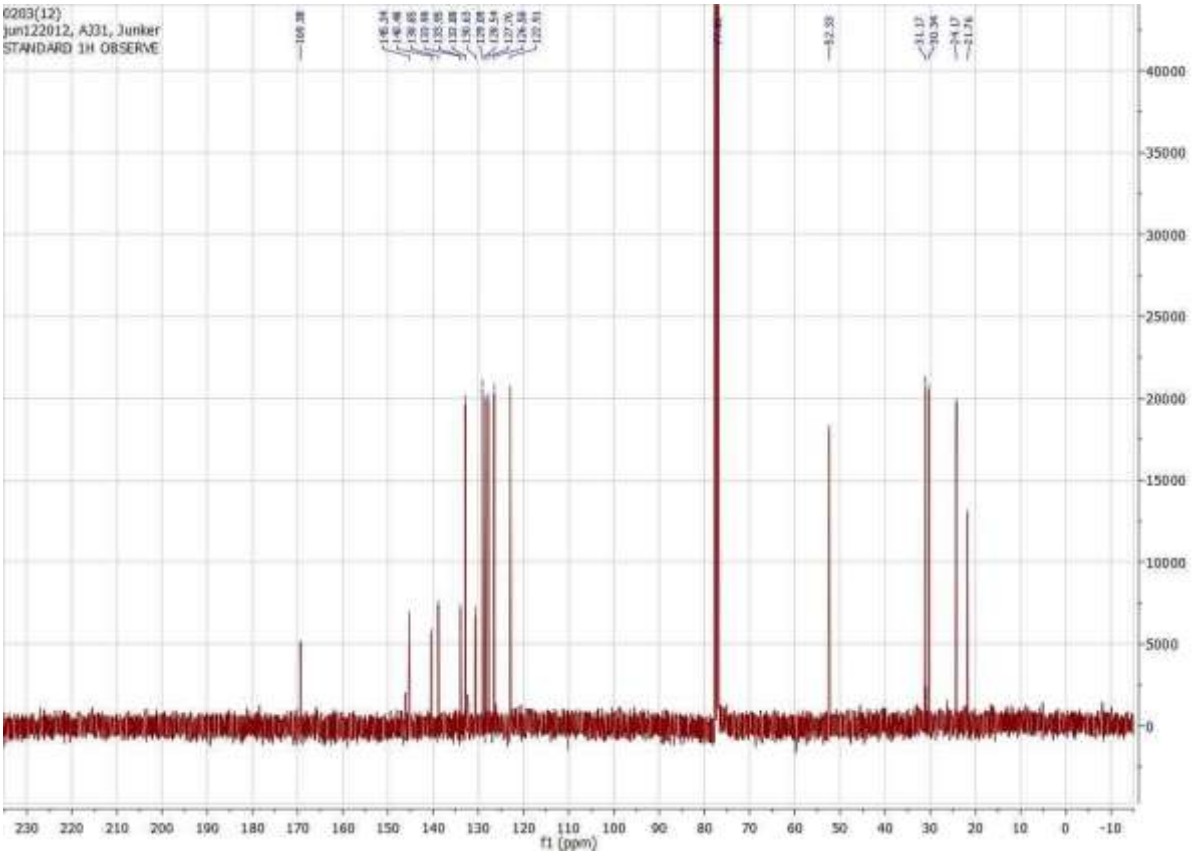
C(2)-C(1)-C(10)-C(11)	-18.3(9)
S(1)-C(1)-C(10)-C(11)	159.2(4)
C(15)-C(10)-C(11)-C(12)	0.2(8)
C(1)-C(10)-C(11)-C(12)	-177.2(5)
C(10)-C(11)-C(12)-C(13)	0.7(8)
C(11)-C(12)-C(13)-C(14)	-1.5(8)
C(11)-C(12)-C(13)-C(16)	175.5(5)
C(12)-C(13)-C(14)-C(15)	1.5(8)
C(16)-C(13)-C(14)-C(15)	-175.5(5)
C(13)-C(14)-C(15)-C(10)	-0.7(8)
C(11)-C(10)-C(15)-C(14)	-0.2(8)
C(1)-C(10)-C(15)-C(14)	177.2(5)
C(4)-C(5)-C(17)-O(1)	178.7(6)
C(6)-C(5)-C(17)-O(1)	0.2(9)
C(4)-C(5)-C(17)-O(2)	-1.6(8)
C(6)-C(5)-C(17)-O(2)	179.8(5)
O(1)-C(17)-O(2)-C(18)	5.5(9)
C(5)-C(17)-O(2)-C(18)	-174.1(5)
C(27)-S(2)-C(19)-C(20)	0.6(5)
C(27)-S(2)-C(19)-C(28)	-177.2(4)
C(28)-C(19)-C(20)-C(21)	175.9(5)
S(2)-C(19)-C(20)-C(21)	-1.6(6)
C(19)-C(20)-C(21)-C(27)	2.2(7)
C(19)-C(20)-C(21)-C(22)	-172.5(5)
C(27)-C(21)-C(22)-C(23)	-0.2(10)
C(20)-C(21)-C(22)-C(23)	173.6(6)
C(21)-C(22)-C(23)-C(35)	-169.6(6)
C(21)-C(22)-C(23)-C(24)	6.8(10)
C(22)-C(23)-C(24)-C(25)	26.7(10)
C(35)-C(23)-C(24)-C(25)	-156.7(6)
C(23)-C(24)-C(25)-C(26)	-74.2(7)
C(24)-C(25)-C(26)-C(27)	72.2(7)
C(20)-C(21)-C(27)-C(26)	179.8(6)
C(22)-C(21)-C(27)-C(26)	-6.1(11)
C(20)-C(21)-C(27)-S(2)	-1.7(6)
C(22)-C(21)-C(27)-S(2)	172.5(5)
C(25)-C(26)-C(27)-C(21)	-27.2(9)
C(25)-C(26)-C(27)-S(2)	154.3(5)
C(19)-S(2)-C(27)-C(21)	0.6(5)

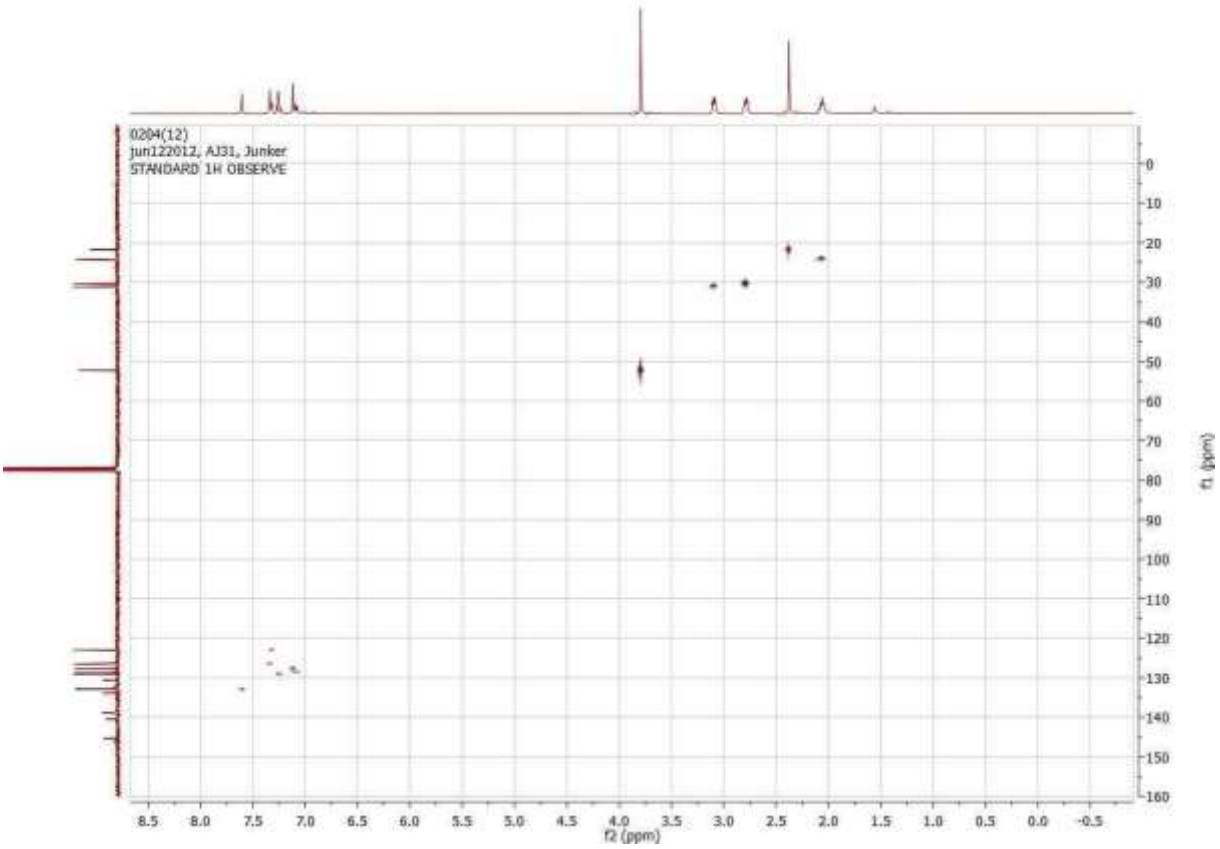
C(19)-S(2)-C(27)-C(26)	179.4(5)
C(20)-C(19)-C(28)-C(33)	-158.7(6)
S(2)-C(19)-C(28)-C(33)	18.6(7)
C(20)-C(19)-C(28)-C(29)	21.7(9)
S(2)-C(19)-C(28)-C(29)	-161.0(4)
C(33)-C(28)-C(29)-C(30)	-1.3(8)
C(19)-C(28)-C(29)-C(30)	178.3(5)
C(28)-C(29)-C(30)-C(31)	-0.9(9)
C(29)-C(30)-C(31)-C(32)	2.7(8)
C(29)-C(30)-C(31)-C(34)	-178.5(6)
C(30)-C(31)-C(32)-C(33)	-2.4(8)
C(34)-C(31)-C(32)-C(33)	178.9(5)
C(31)-C(32)-C(33)-C(28)	0.3(9)
C(29)-C(28)-C(33)-C(32)	1.5(8)
C(19)-C(28)-C(33)-C(32)	-178.0(5)
C(22)-C(23)-C(35)-O(3)	163.7(6)
C(24)-C(23)-C(35)-O(3)	-13.1(9)
C(22)-C(23)-C(35)-O(4)	-13.3(8)
C(24)-C(23)-C(35)-O(4)	169.8(5)
O(3)-C(35)-O(4)-C(36)	0.6(9)
C(23)-C(35)-O(4)-C(36)	177.7(5)

Symmetry transformations used to generate equivalent atoms.

Methyl 2-(3-methylphenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (9b)




0203(12)
jun122012, AJ31, Junker
STANDARD 1H OBSERVE
f1 (ppm)


0204(12)
jun122012, AJ31, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 09.01.13 00:29

Reported: 09.01.13 15:59
Processed: 09.01.13 15:59

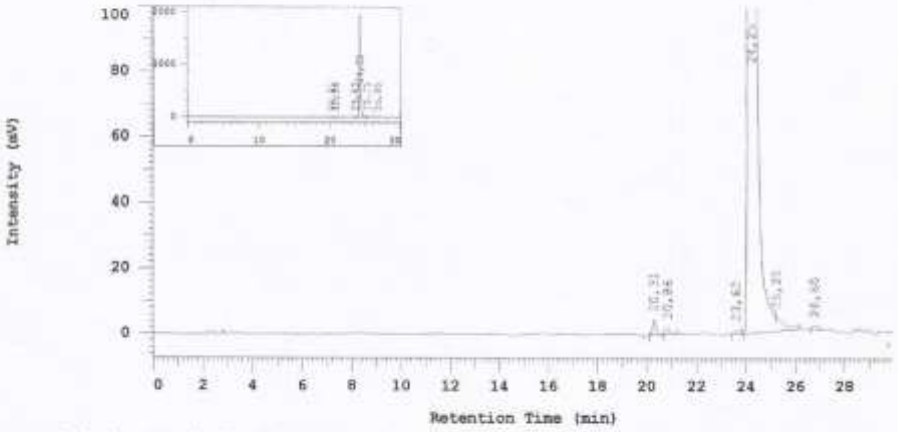
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5764\
Application: Chromni

Sample Name: AJ31

Injection from this vial: 1 of 1

Series:5764
Vial Number: 16
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

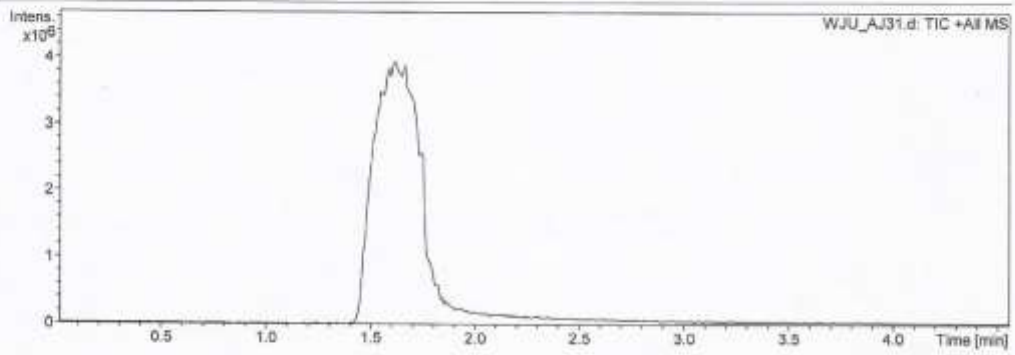
No.	RT	Area	Conc 1	BC
1	20,31	46749	0,217	BB
2	20,86	24970	0,116	MC
3	23,62	15607	0,072	MC
4	24,25	21354243	99,009	MC
5	25,25	113173	0,525	MC
6	26,80	13336	0,062	BB
		21568078	100,000	

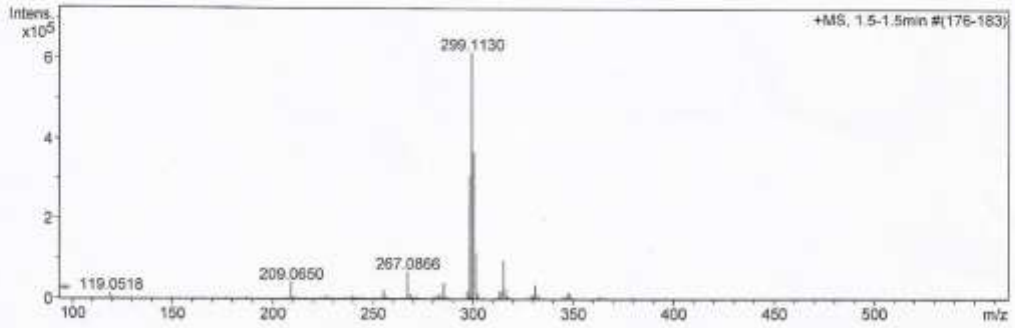
Peak rejection level: 0

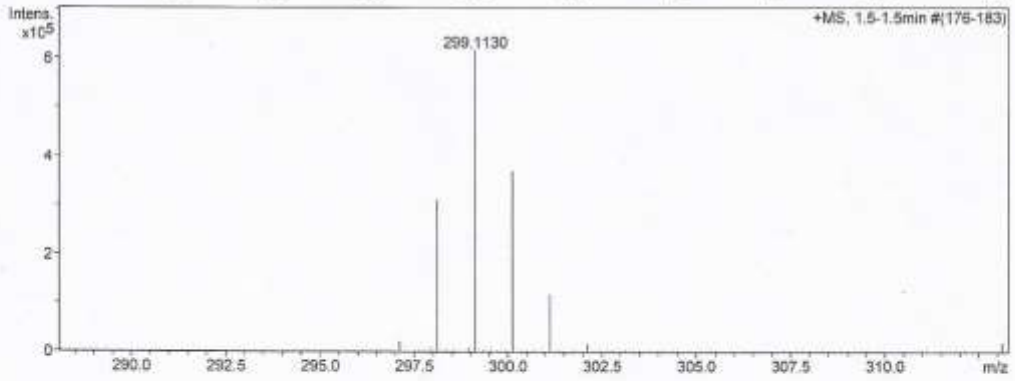
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\13_01\WJU_AJ31.d	Acquisition Date	1/7/2013 7:57:37 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ31	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		







Bruker Compass DataAnalysis 4.0 printed: 1/7/2013 2:01:37 PM Page 1 of 1

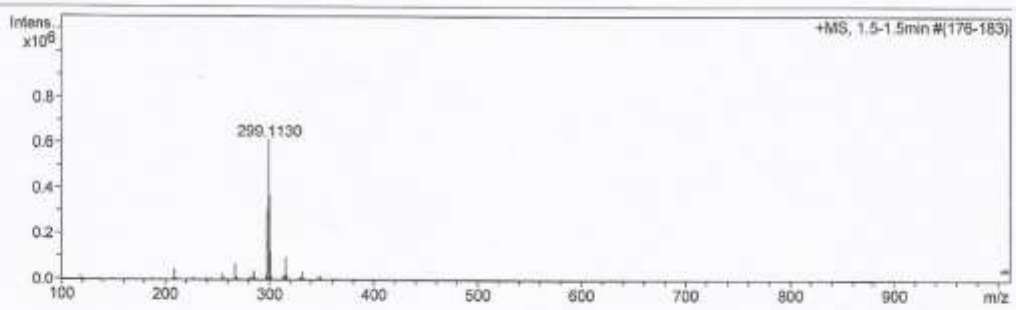
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\13_01\WJU_AJ31.d	Acquisition Date	1/7/2013 7:57:37 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ31	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

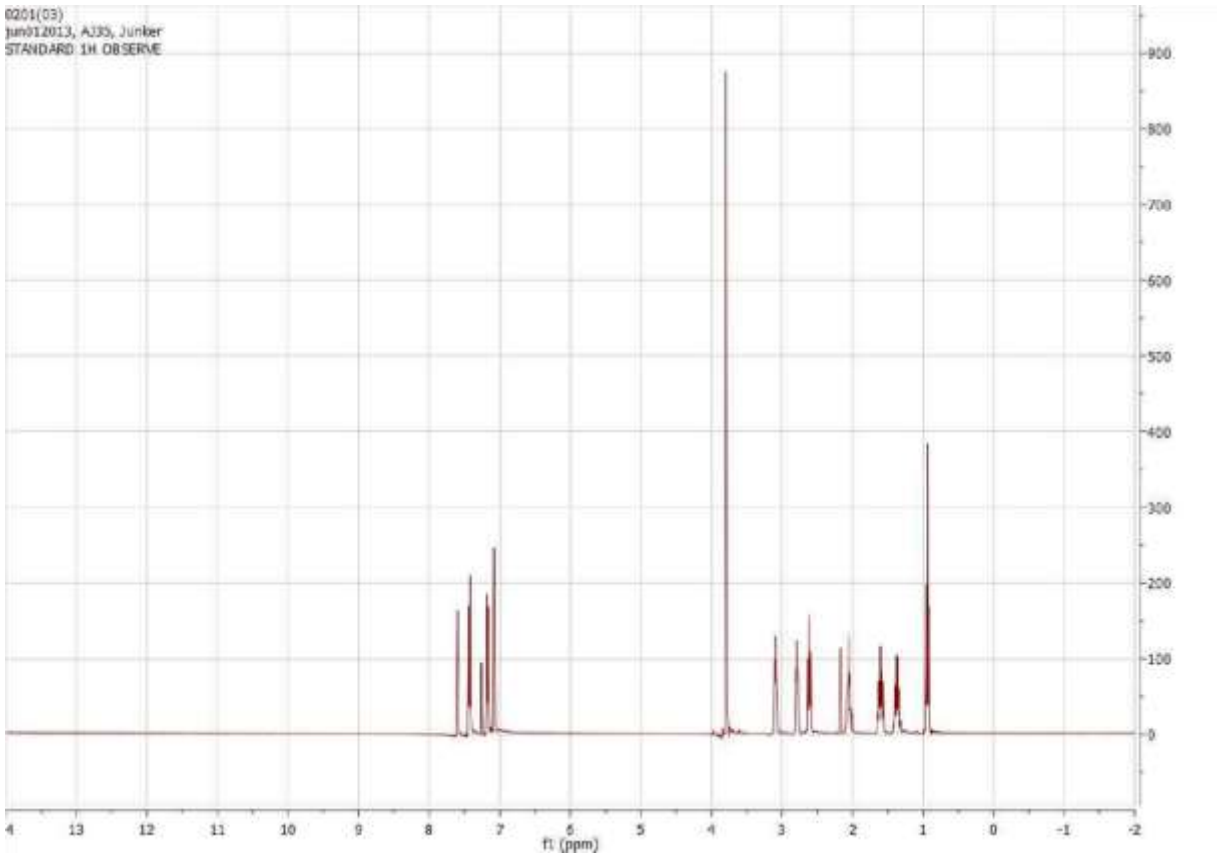
Acquisition Parameter

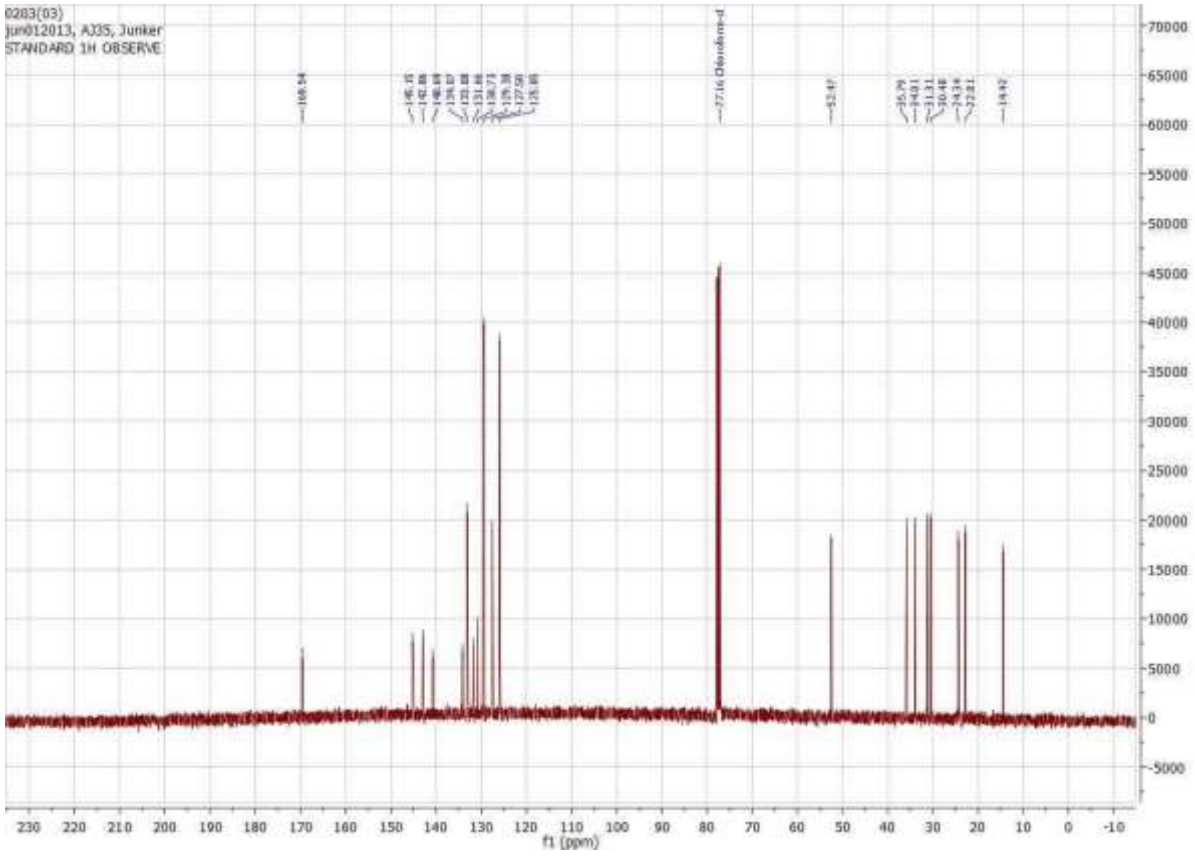
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

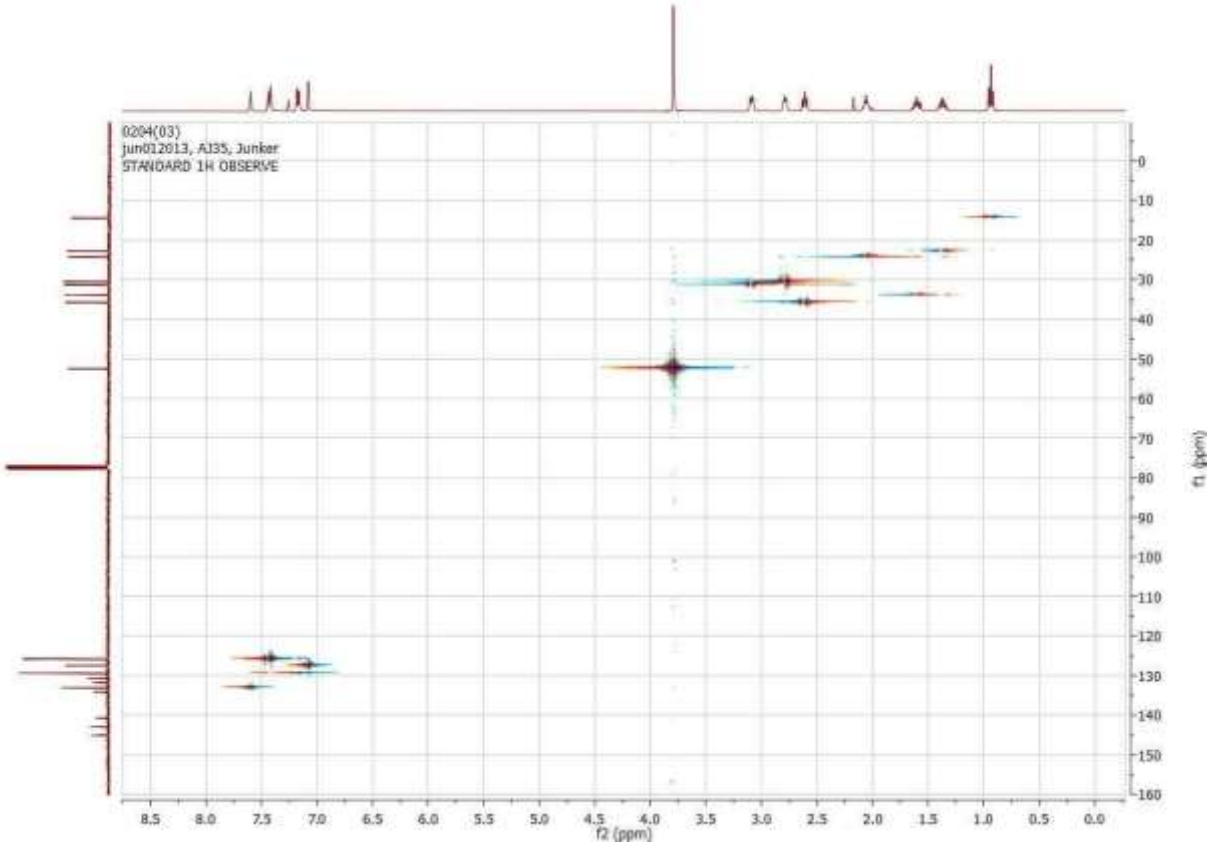


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
299.1130	1	C 12 H 27 O 2 S 3	23.11	299.1168	3.8	12.7	199.3	-0.5	even	ok
	2	C 18 H 19 O 2 S	31.51	299.1100	-2.9	-9.8	206.4	9.5	even	ok
	3	C 8 H 23 N 6 S 3	100.00	299.1141	1.1	3.8	209.1	0.5	even	ok
	4	C 15 H 23 O 2 S 2	98.29	299.1134	0.4	1.5	213.4	4.5	even	ok
	5	C 14 H 15 N 6 S	0.40	299.1073	-5.6	-18.8	217.7	10.5	even	ok
	6	C 11 H 19 N 6 S 2	11.20	299.1107	-2.2	-7.5	224.5	5.5	even	ok
	7	C 13 H 19 N 2 O 4 S	0.01	299.1060	-7.0	-23.3	229.4	5.5	even	ok
	8	C 12 H 19 N 4 O 3 S	0.72	299.1172	4.3	14.3	231.4	5.5	even	ok
	9	C 10 H 23 N 2 O 4 S 2	0.97	299.1094	-3.6	-12.0	236.7	0.5	even	ok
	10	C 9 H 23 N 4 O 3 S 2	0.00	299.1206	7.6	25.6	238.6	0.5	even	ok
	11	C 11 H 23 O 7 S	0.99	299.1159	2.9	9.8	243.1	0.5	even	ok
	12	C 8 H 15 N 10 O S	2.92	299.1146	1.6	5.3	243.3	6.5	even	ok
	13	C 7 H 19 N 6 O 5 S	0.02	299.1132	0.3	0.9	293.7	1.5	even	ok

Methyl 2-(4-butylphenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (9c)






0204(03)
jun012013, AI35, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 09.01.13 01:10

Reported: 09.01.13 16:00
Processed: 09.01.13 15:59

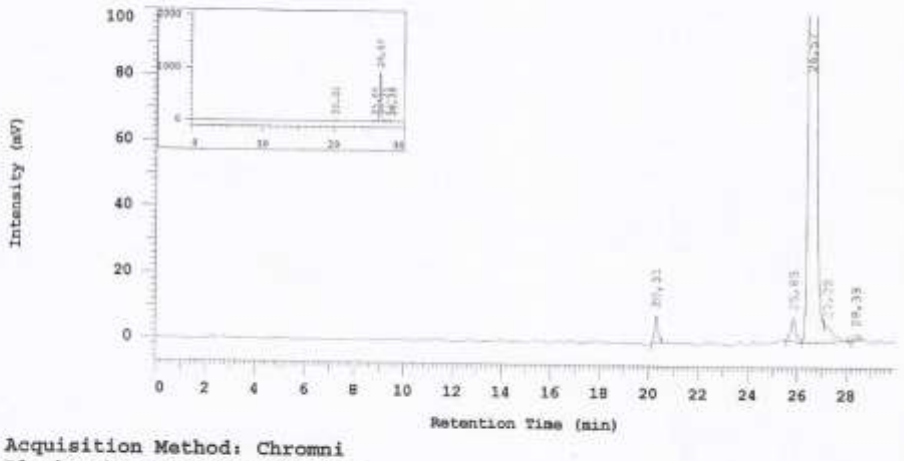
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5765\
Application: Chromni

Sample Name: AJ35

Injection from this vial: 1 of 1

Series:5765
Vial Number: 17
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Solvent A: Wasser + 0,05%TFA

Developed by: Jens
Solvent B: ACN + 0,05%TFA

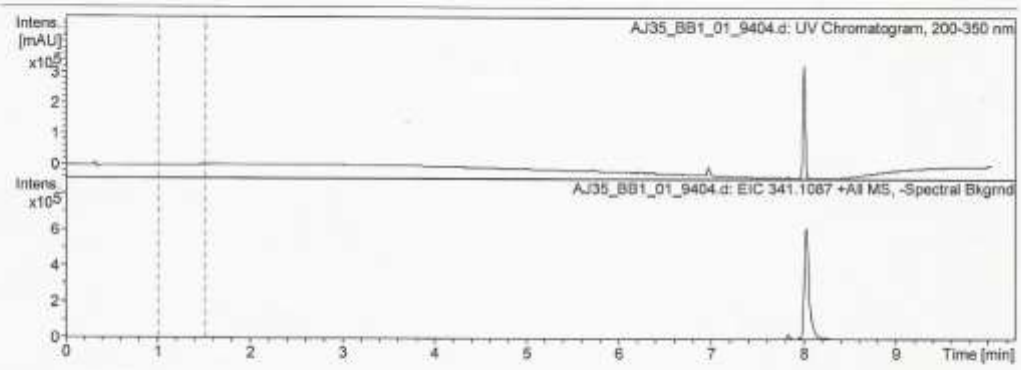
No.	RT	Area	Conc 1	BC
1	20,31	75454	0,716	BB
2	25,85	101297	0,961	BB
3	26,57	10212264	96,888	MC
4	27,25	134052	1,272	MC
5	28,39	17220	0,163	MC
		10540287	100,000	

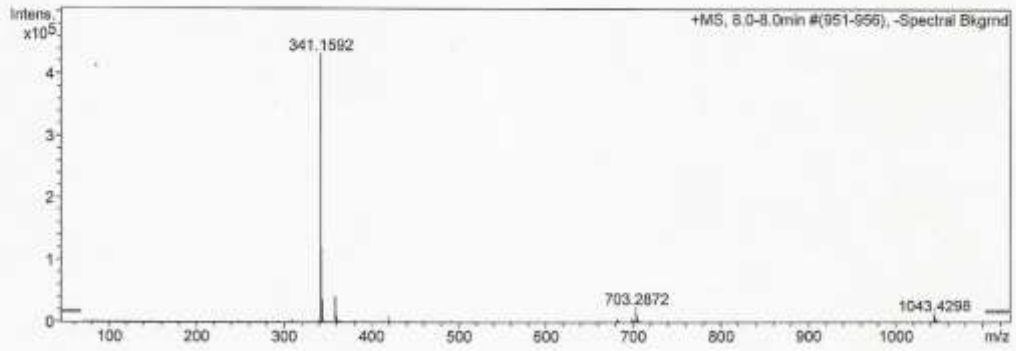
Peak rejection level: 0

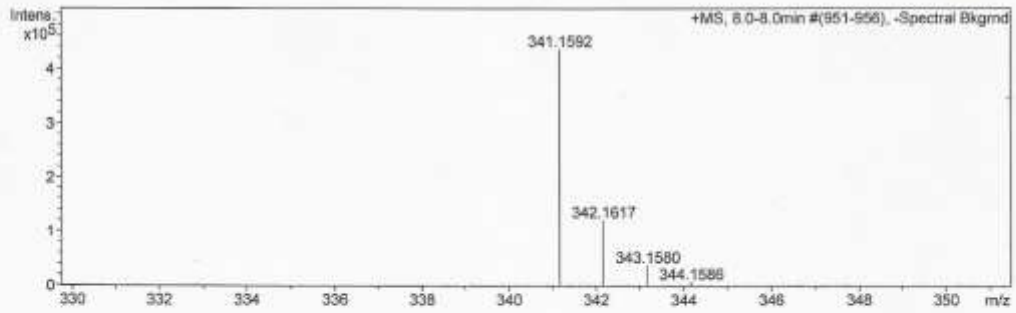
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_28\AJ35_BB1_01_9404.d	Acquisition Date	1/28/2013 3:39:14 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ35	Instrument	micrOTOF-Q II
Comment	Junker LCMS-ESI+ Kalibration mit Li-Formate		







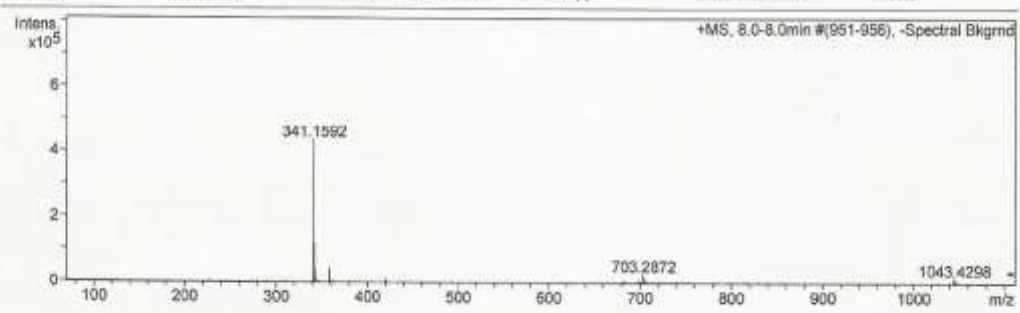
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_28\AJ35_BB1_01_9404.d	Acquisition Date	1/28/2013 3:39:14 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ35	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker LCMS-ESI+ Kalibration mit Li-Formate		

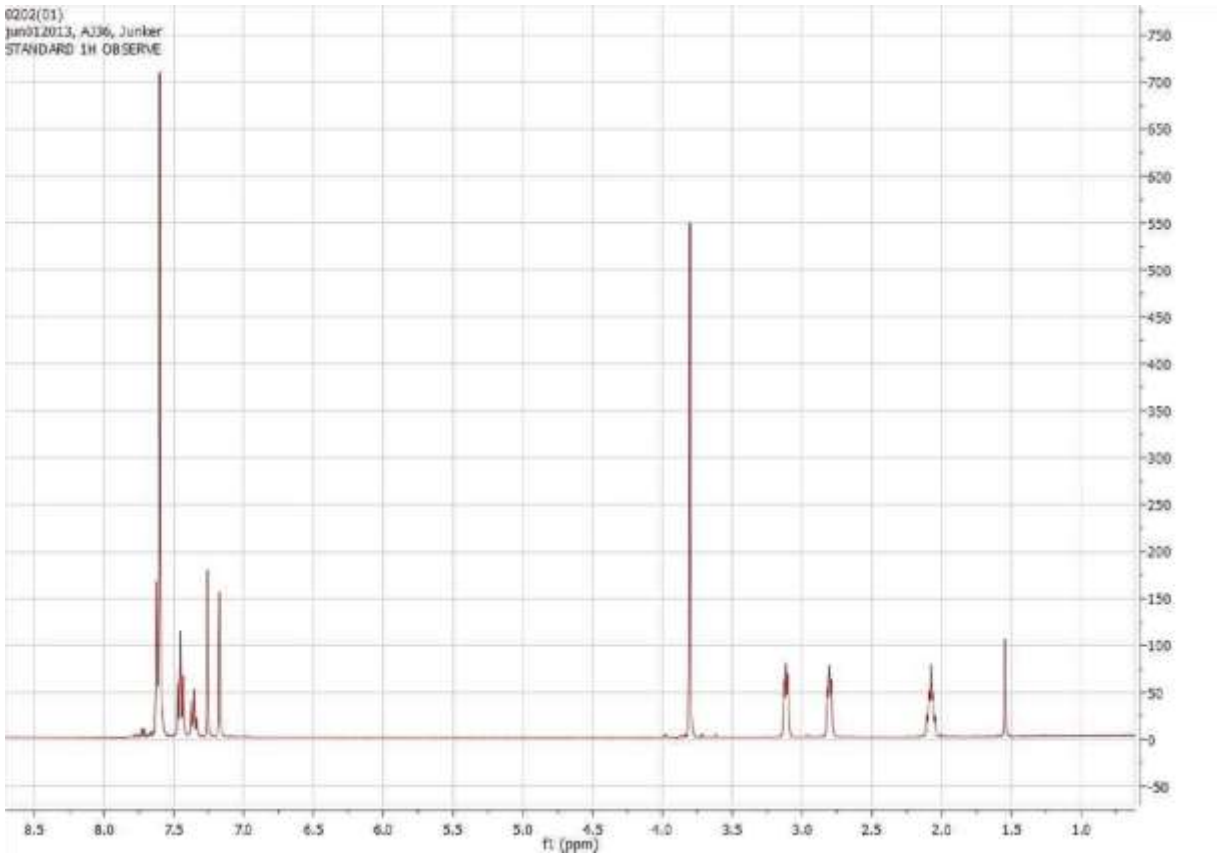
Acquisition Parameter

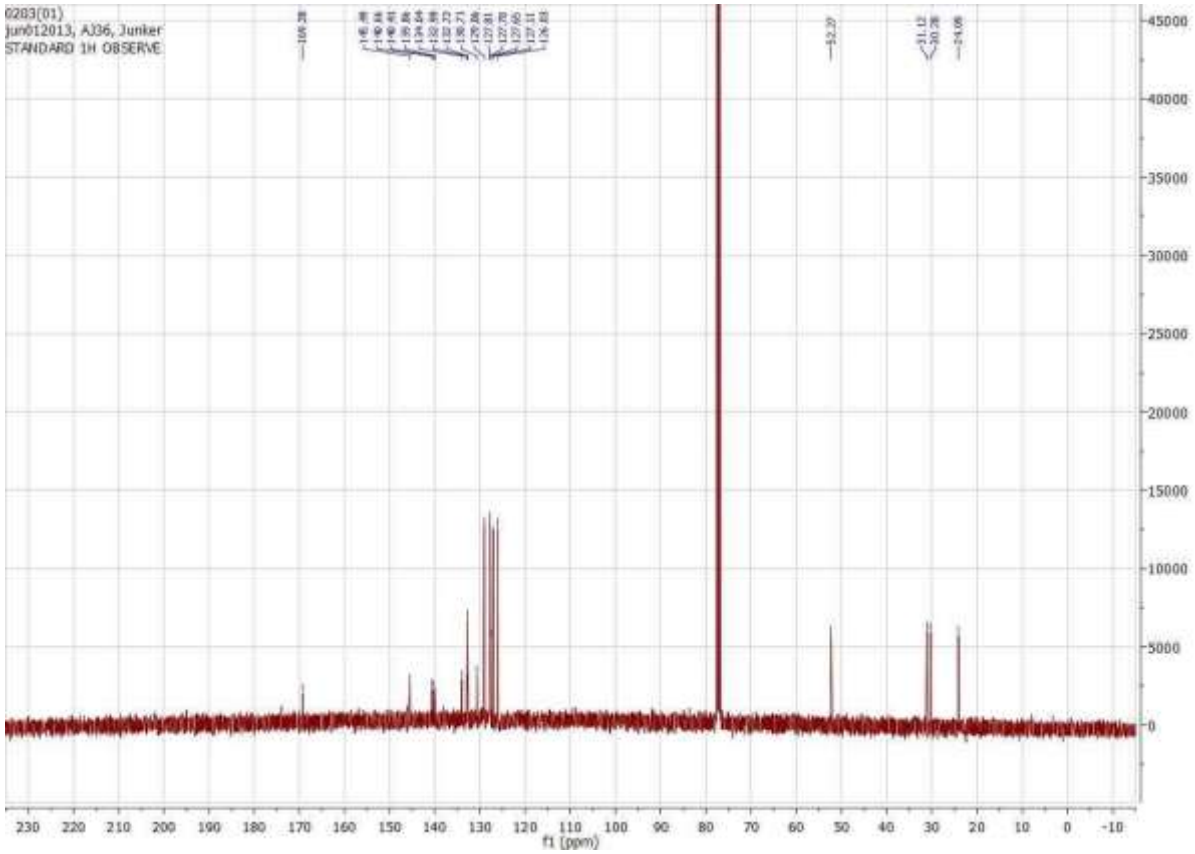
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

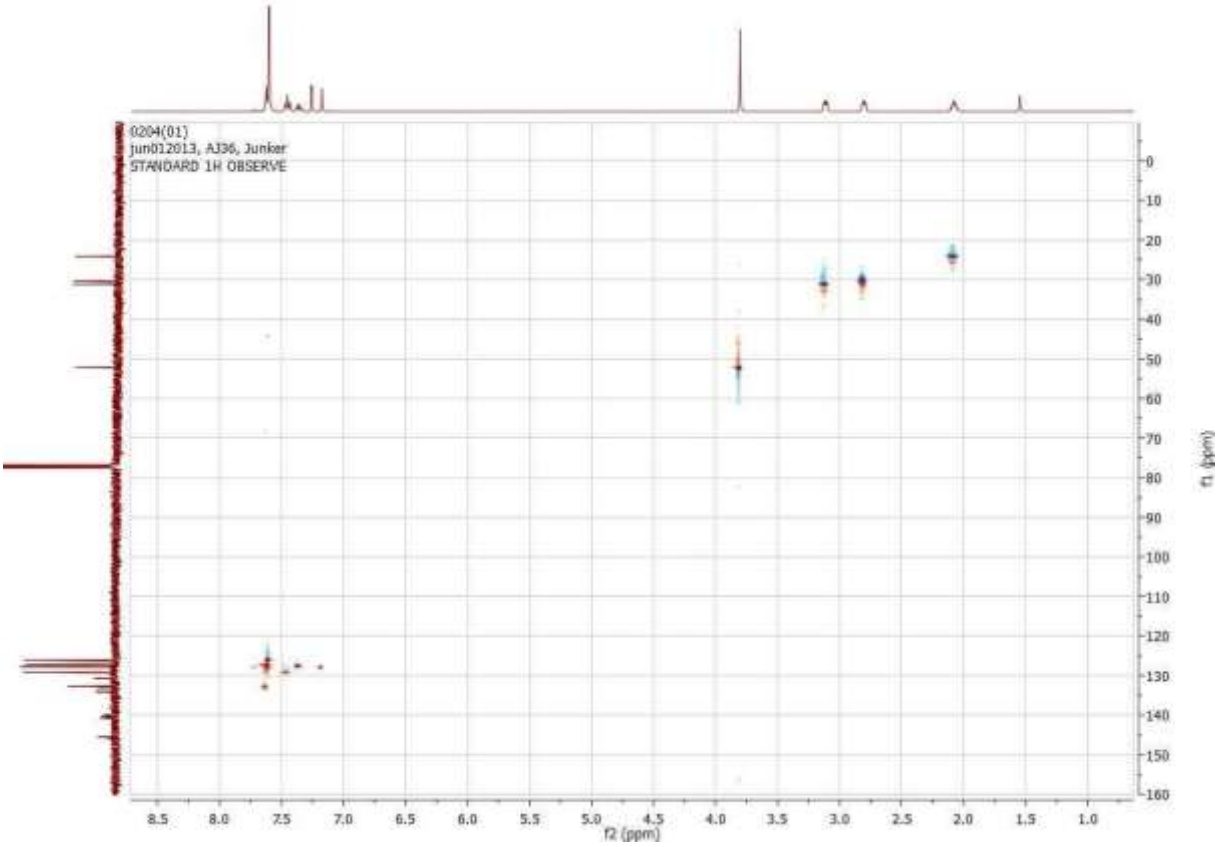


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
341.1592	1	C 21 H 25 O 2 S	66.42	341.1570	-2.2	-6.6	21.0	9.5	even	ok
	2	C 18 H 29 O 2 S 2	100.00	341.1603	1.1	3.3	36.7	4.5	even	ok

Methyl 2-([1,1'-biphenyl]-4-yl)-7,8-dihydro-6*H*-[7]annulene[*b*]thiophene-5-carboxylate (9d)






0204(01)
jun012013, AJ36, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

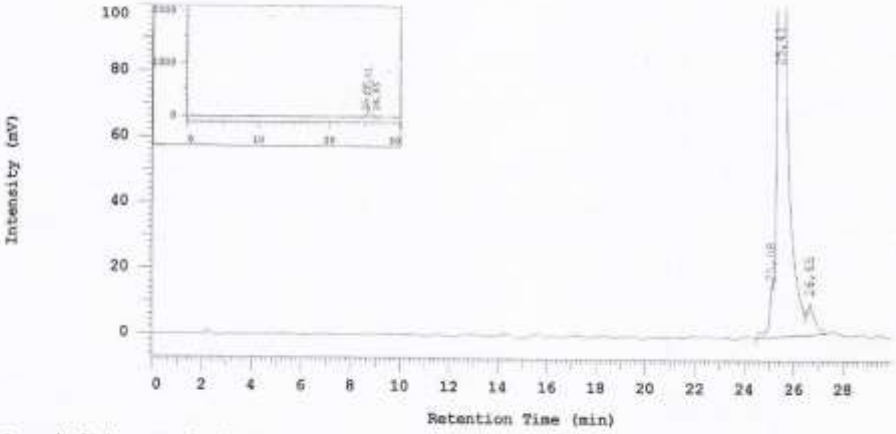
Analyzed: 09.01.13 02:33

Reported: 09.01.13 16:02
Processed: 09.01.13 16:02

Data Path: D:\WIN32APP\HSM\Chromni\DATA\5767\
Application: Chromni
Sample Name: AJ36
Injection from this vial: 1 of 1

Series:5767
Vial Number: 19
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

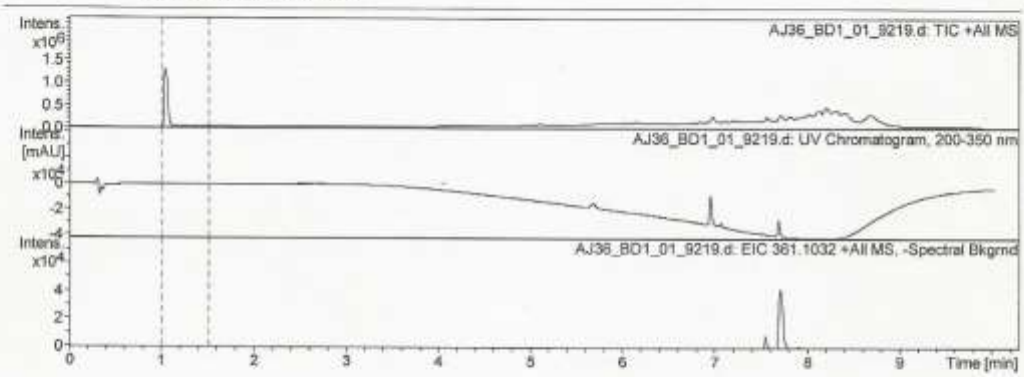
No.	RT	Area	Conc 1	BC
1	25,08	170084	2,860	MC
2	25,41	5580168	93,842	MC
3	26,65	196062	3,297	MC
		5946314	100,000	

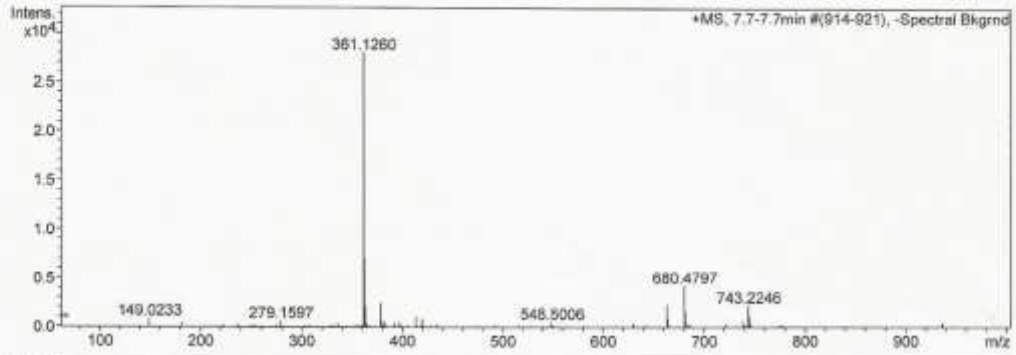
Peak rejection level: 0

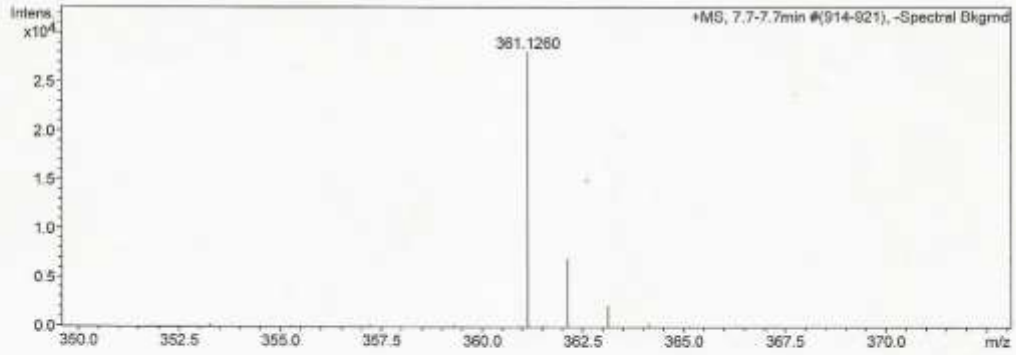
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ36_BD1_01_9219.d	Acquisition Date	1/14/2013 6:56:50 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ36	Instrument	micrOTOF-Q II
Comment	Junker Kalibration mit Li-Formate		







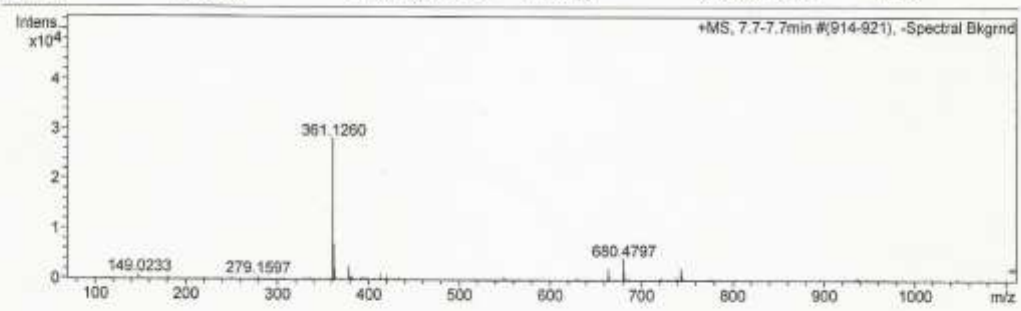
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ36_BD1_01_9219.d	Acquisition Date	1/14/2013 6:56:50 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ36	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker Kalibration mit Li-Formate		

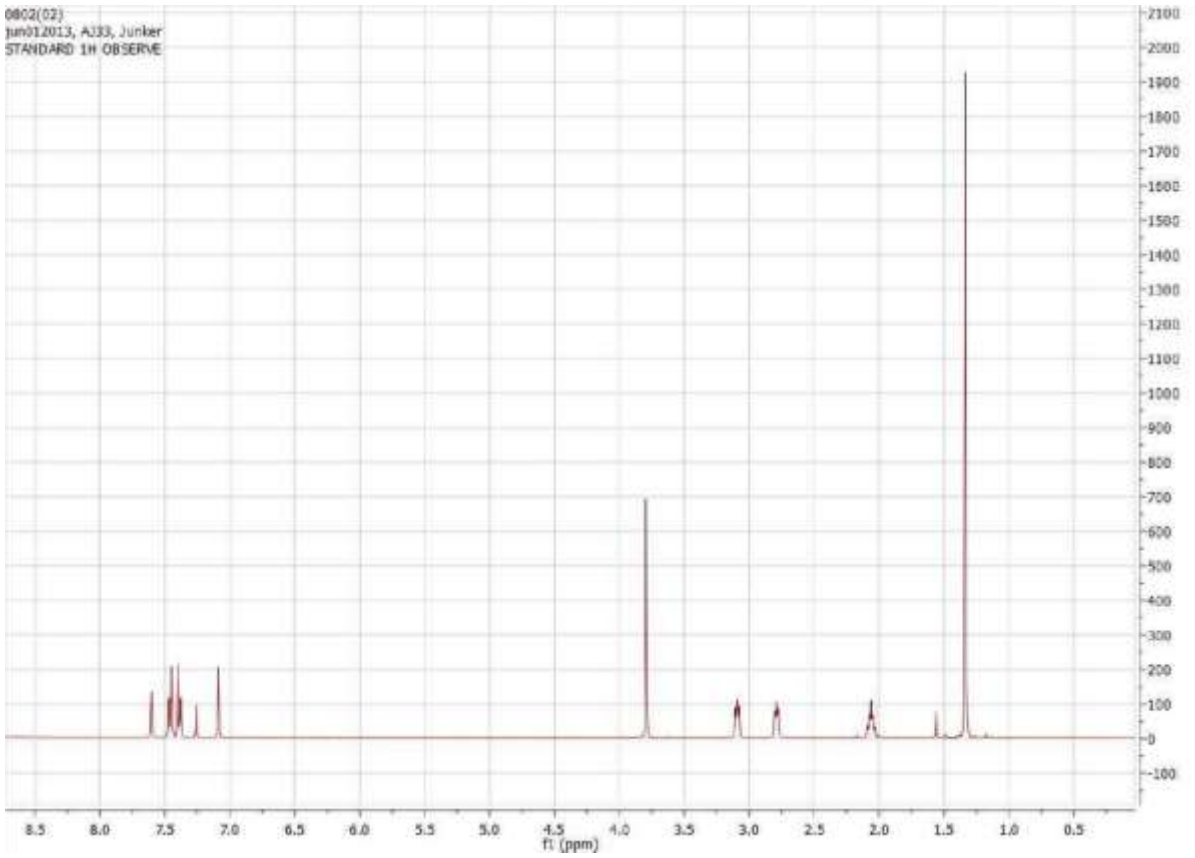
Acquisition Parameter

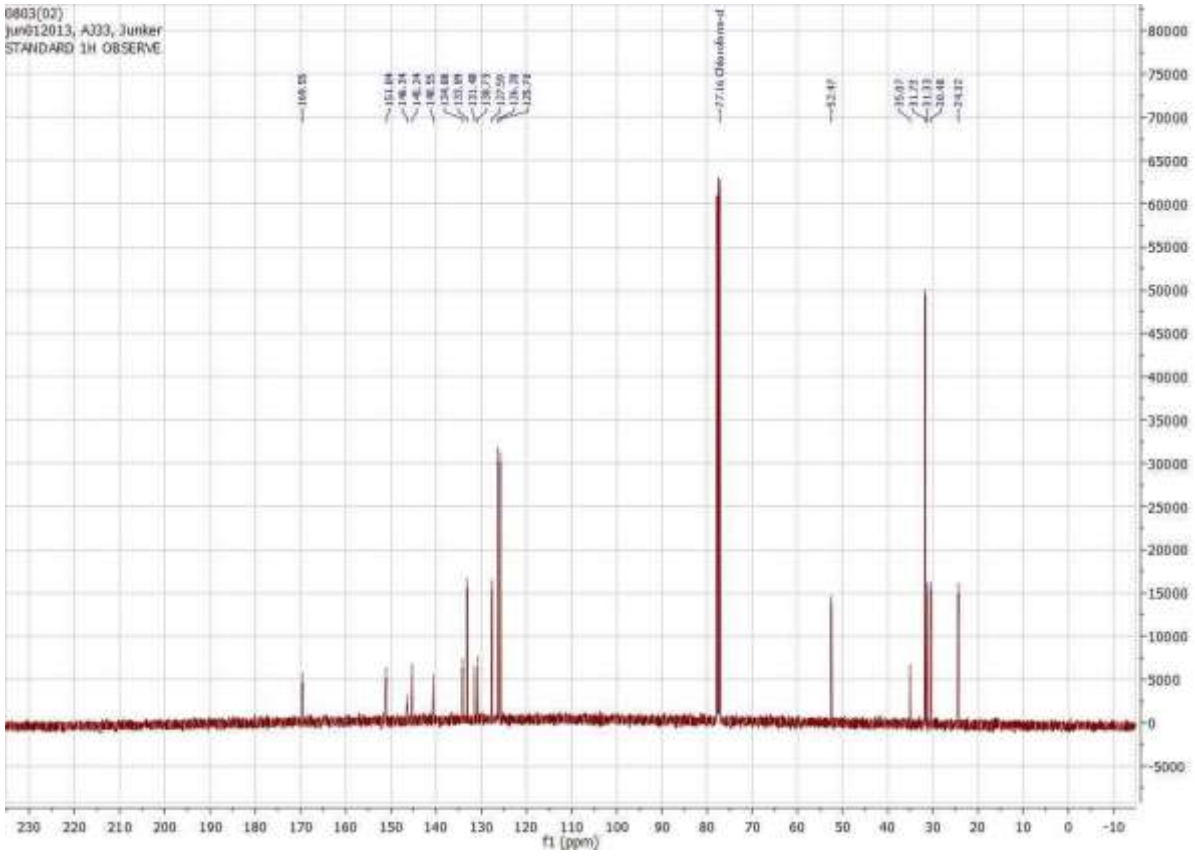
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

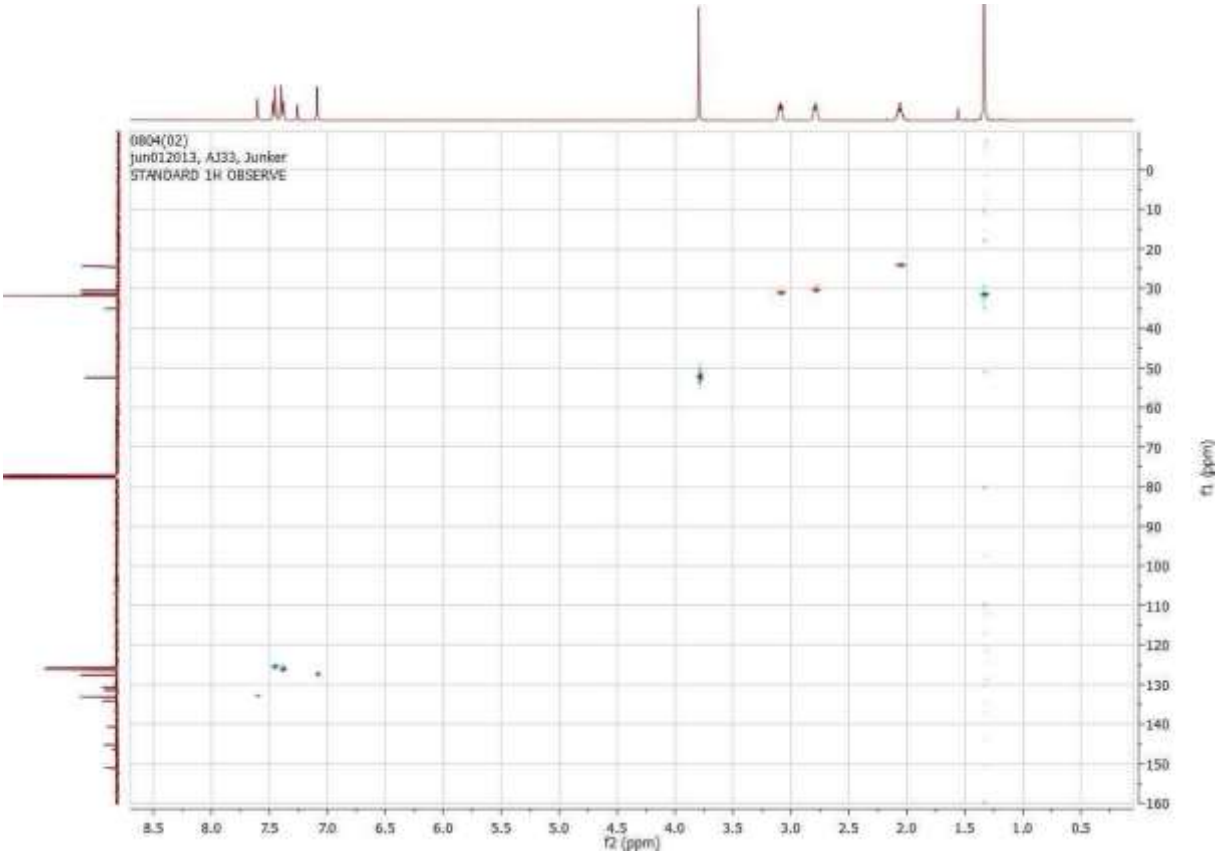


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
361.1260	1	C 23 H 21 O 2 S	100.00	361.1257	-0.3	-1.0	5.3	13.5	even	ok
	2	C 19 H 17 N 6 S	14.73	361.1230	-3.0	-8.4	6.3	14.5	even	ok
	3	C 20 H 25 O 2 S 2	10.54	361.1290	3.0	8.4	23.7	8.5	even	ok
	4	C 16 H 21 N 6 S 2	67.10	361.1264	0.3	0.9	25.5	9.5	even	ok

Methyl 2-(4-*tert*-butylphenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (9e)






0804(02)
jun012013, AJ33, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 23.08.12 01:55

Reported: 24.08.12 11:18
Processed: 24.08.12 11:18

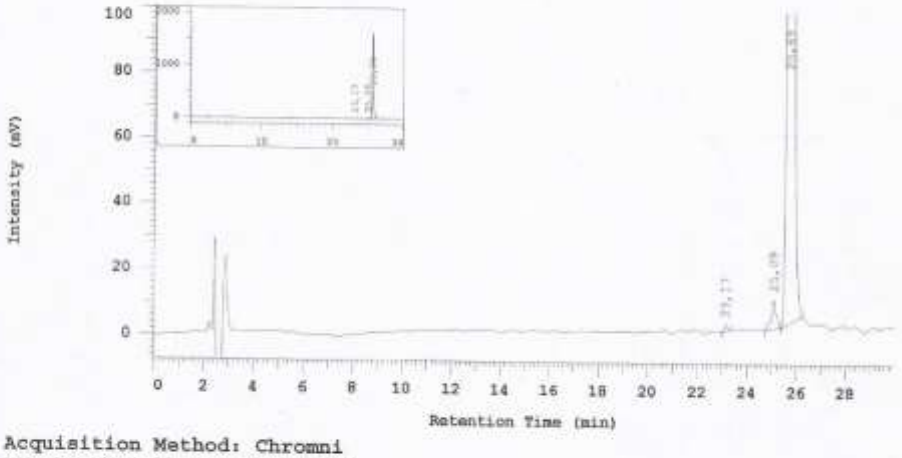
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5120\
Application: Chromni

Sample Name: AJ33

Injection from this vial: 1 of 1

Series:5120
Vial Number: 12
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

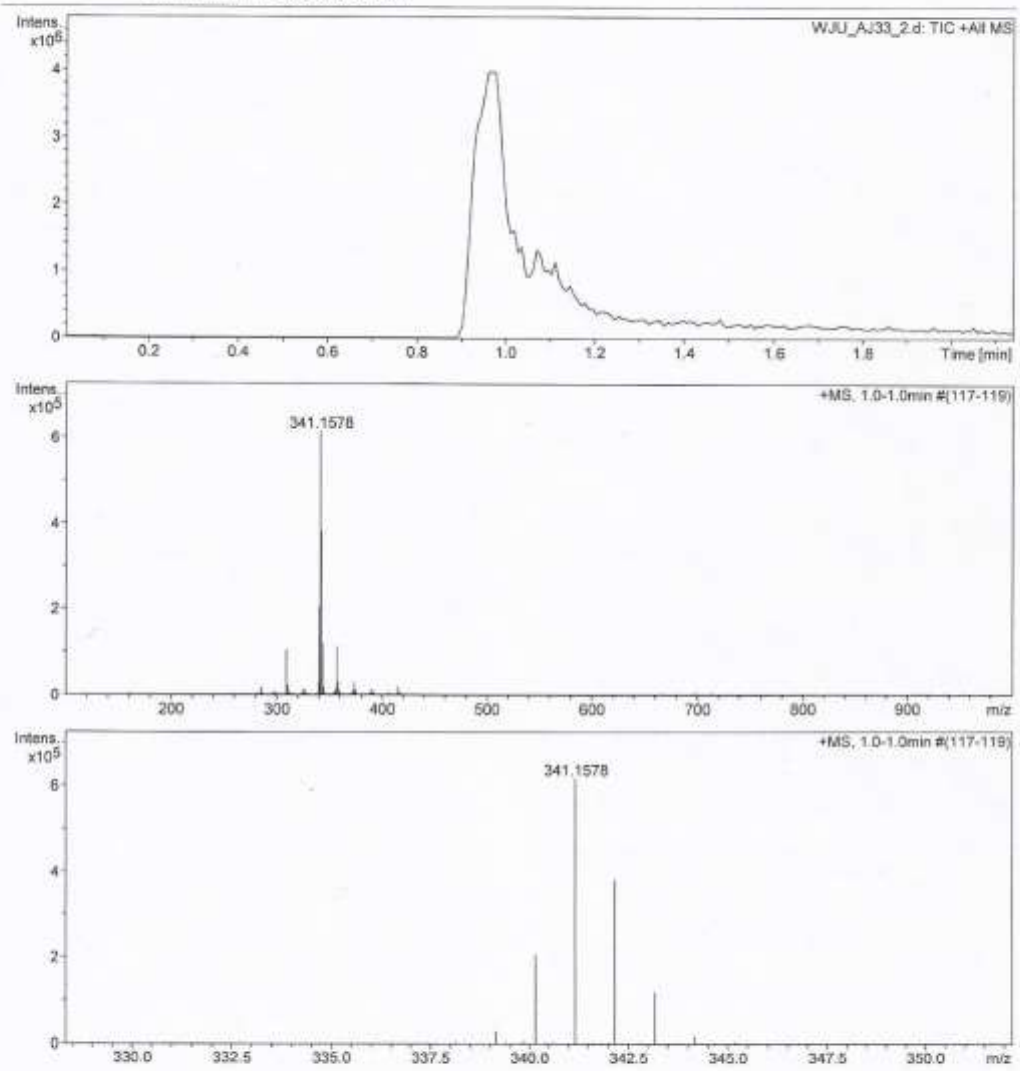
No.	RT	Area	Conc 1	BC
1	23,17	16870	0,106	BB
2	25,09	121161	0,758	BB
3	25,69	15838412	99,136	BB
		15976443	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ33_2.d	Acquisition Date	8/28/2012 1:48:12 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ33	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



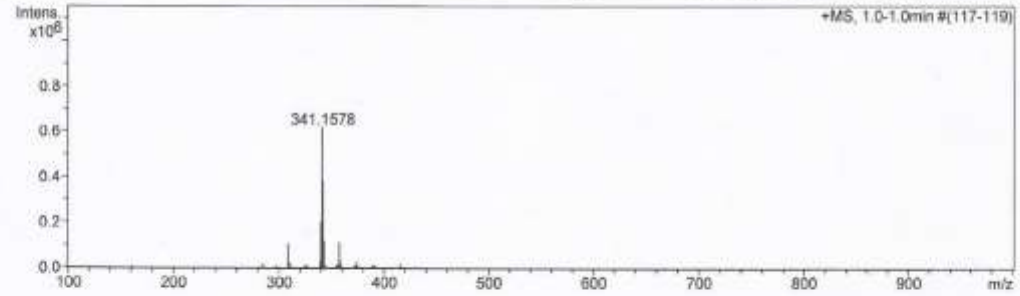
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ33_2.d	Acquisition Date	8/28/2012 1:48:12 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ33	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

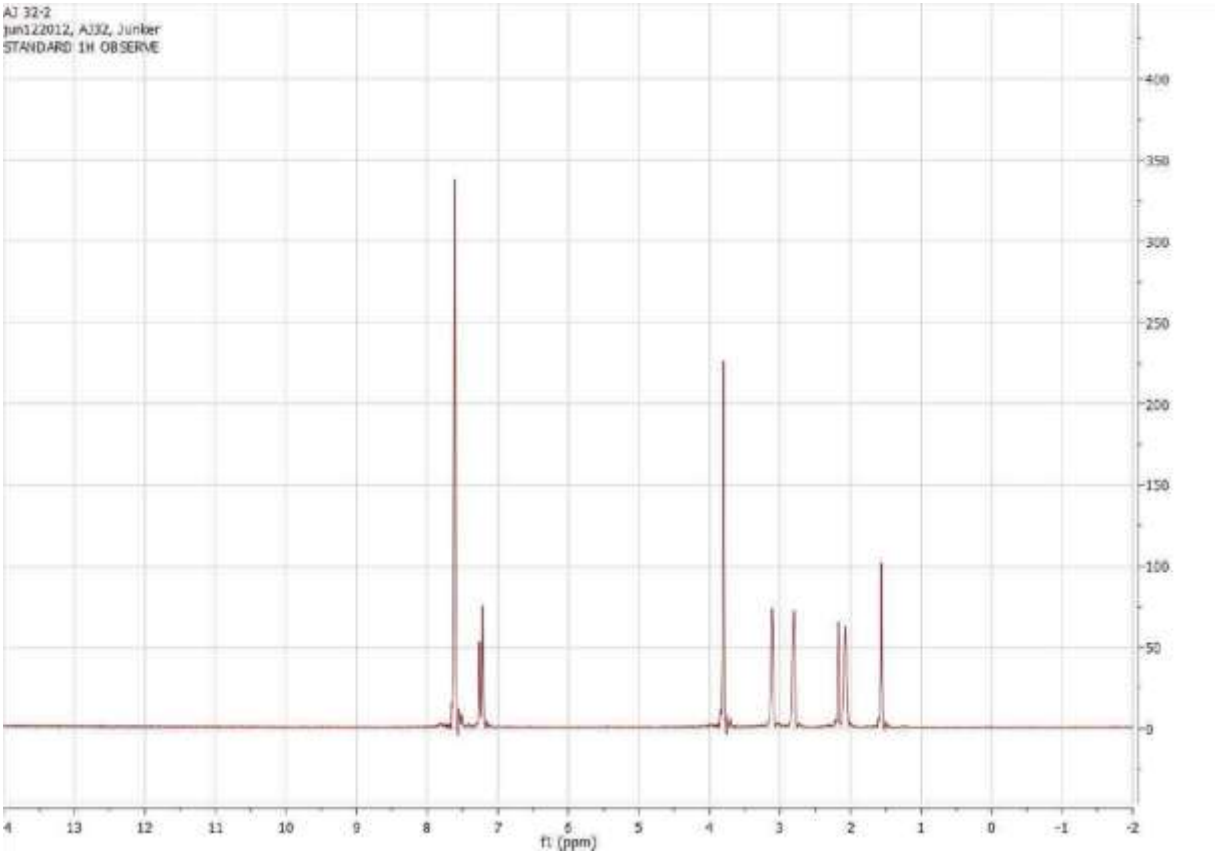
Acquisition Parameter

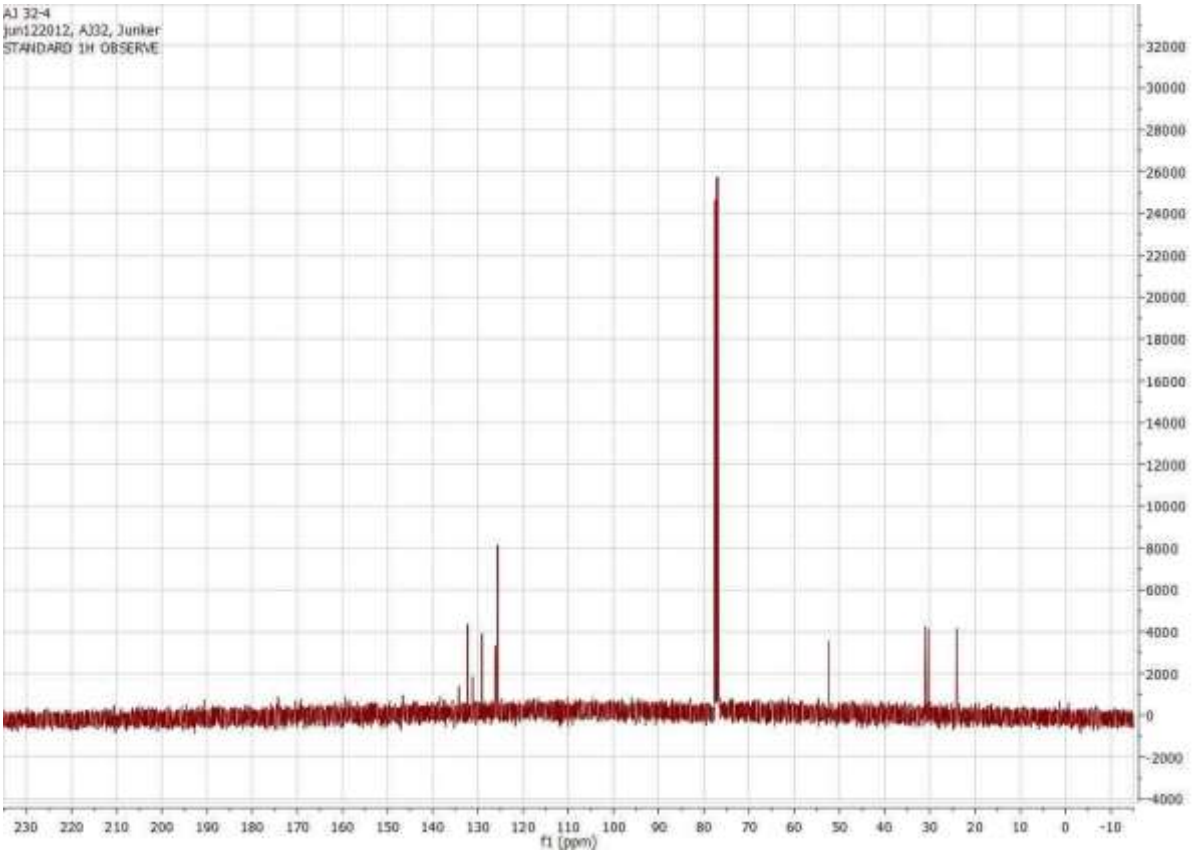
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

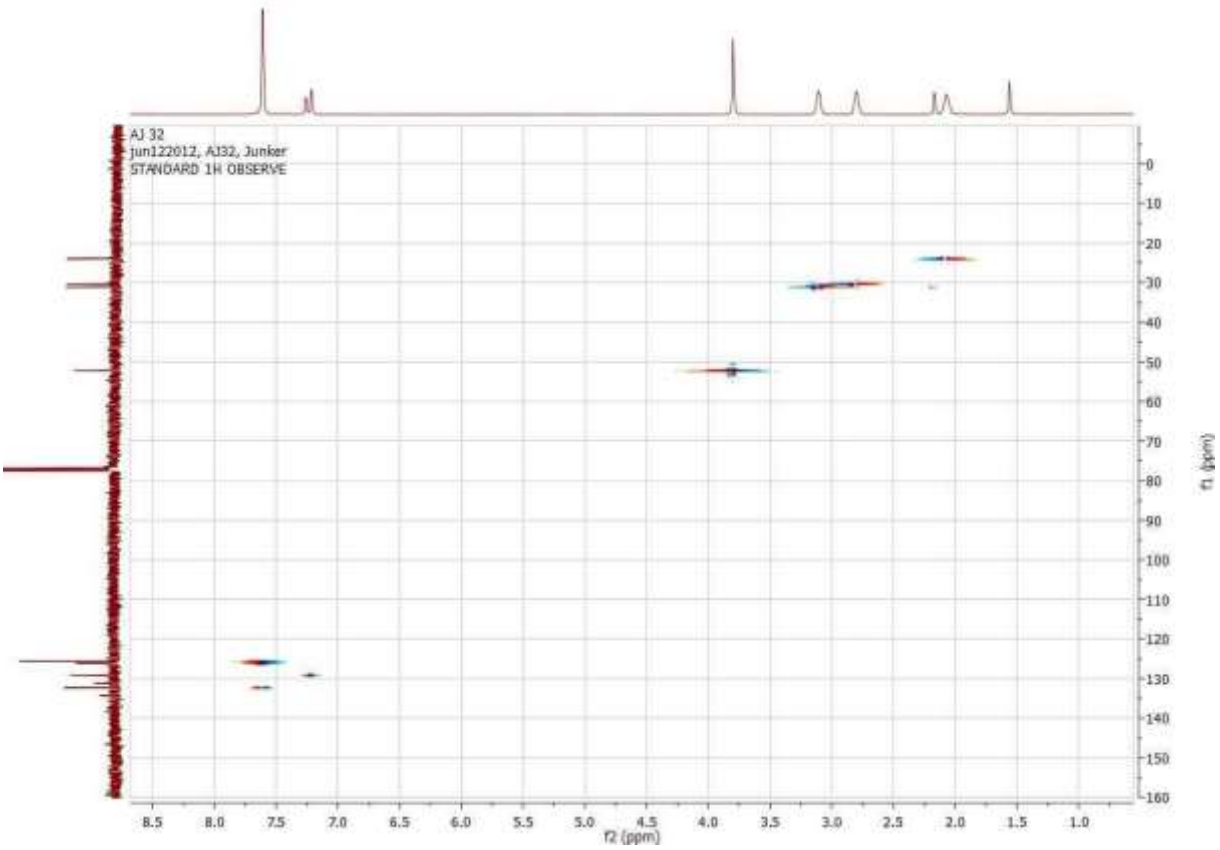


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
341.1578	1	C 21 H 25 O 2 S	100.00	341.1570	-0.8	-2.4	200.5	9.5	even	ok
	2	C 11 H 29 N 6 S 3	12.07	341.1610	3.2	9.5	203.6	0.5	even	ok
	3	C 18 H 29 O 2 S 2	16.55	341.1603	2.5	7.5	207.4	4.5	even	ok
	4	C 17 H 21 N 6 S	4.31	341.1543	-3.5	-10.3	211.9	10.5	even	ok
	5	C 14 H 25 N 6 S 2	28.15	341.1577	-0.1	-0.4	218.6	5.5	even	ok
	6	C 16 H 25 N 2 O 4 S	0.29	341.1530	-4.9	-14.2	223.7	5.5	even	ok
	7	C 13 H 29 N 2 O 4 S 2	4.35	341.1563	-1.5	-4.3	230.8	0.5	even	ok
	8	C 11 H 21 N 10 O S	0.33	341.1615	3.7	10.8	237.3	6.5	even	ok
	9	C 9 H 25 N 8 O 2 S 2	0.12	341.1536	-4.2	-12.2	241.9	1.5	even	ok
	10	C 10 H 25 N 6 O 5 S	0.38	341.1602	2.4	6.9	246.6	1.5	even	ok

Methyl-2-(4-(trifluoromethyl)phenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (9f)






AJ 32
jun122012, AJ32, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

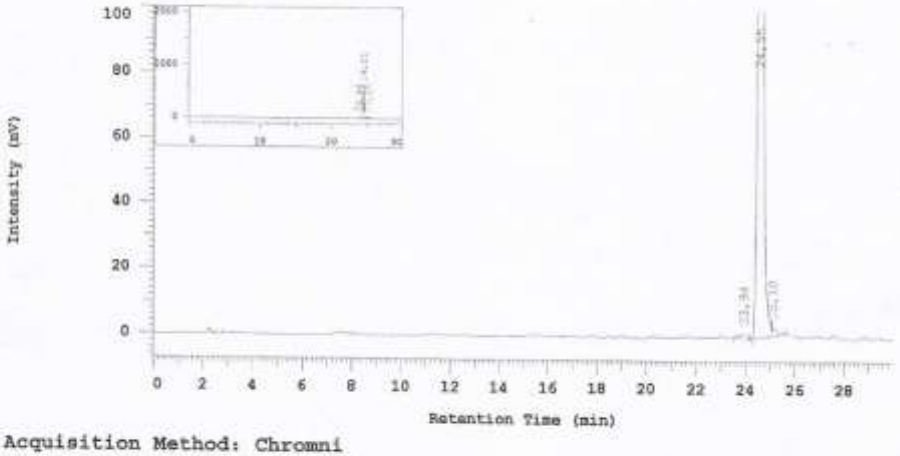
Analyzed: 08.01.13 21:43

Reported: 09.01.13 15:56
Processed: 09.01.13 15:56

Data Path: D:\WIN32APP\HSM\Chromni\DATA\5760\
Application: Chromni
Sample Name: AJ32
Injection from this vial: 1 of 1

Series:5760
Vial Number: 13
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

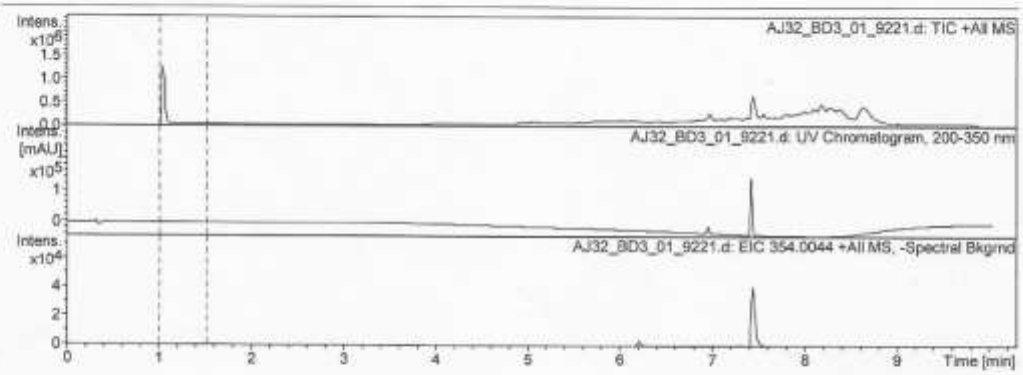
No.	RT	Area	Conc 1	BC
1	23,94	31091	0,499	BB
2	24,55	6163019	98,850	MC
3	25,10	40585	0,651	MC
		6234695	100,000	

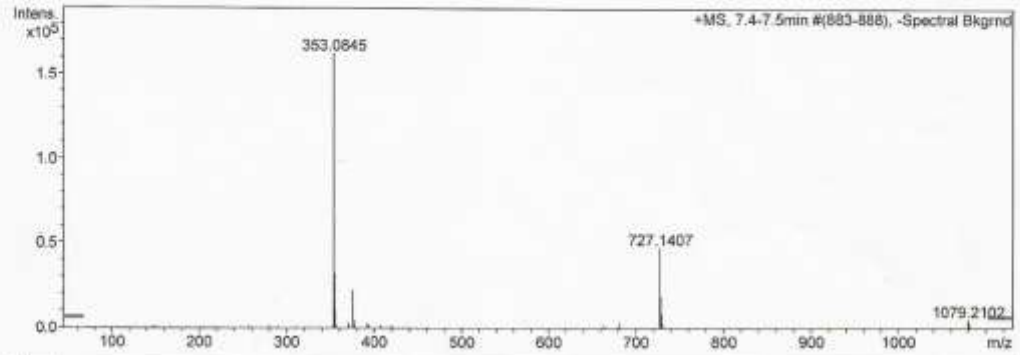
Peak rejection level: 0

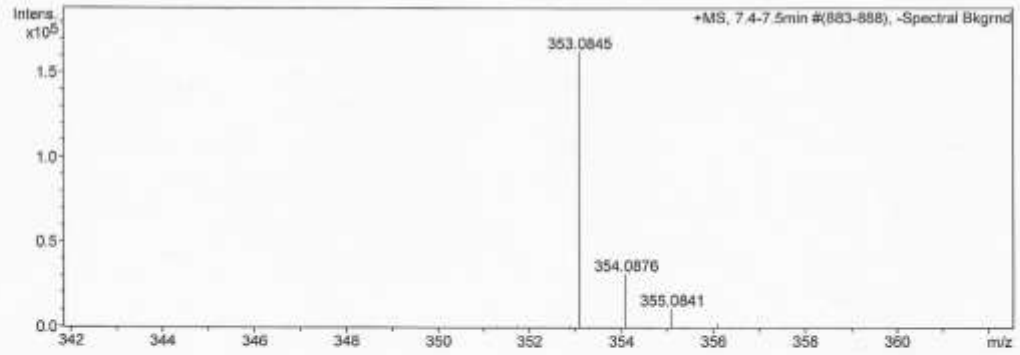
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ32_BD3_01_9221.d	Acquisition Date	1/14/2013 7:19:43 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ32	Instrument	micrOTOF-Q II
Comment	Junker Kalibration mit Li-Formate		







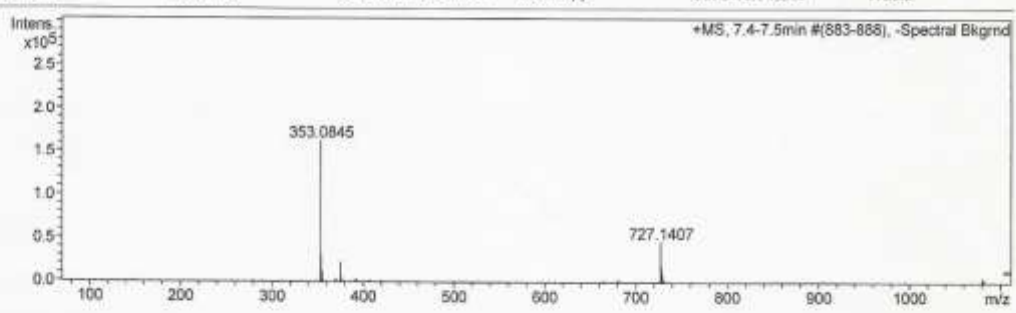
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ32_BD3_01_9221.d	Acquisition Date	1/14/2013 7:19:43 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ32	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker Kalibration mit Li-Formate		

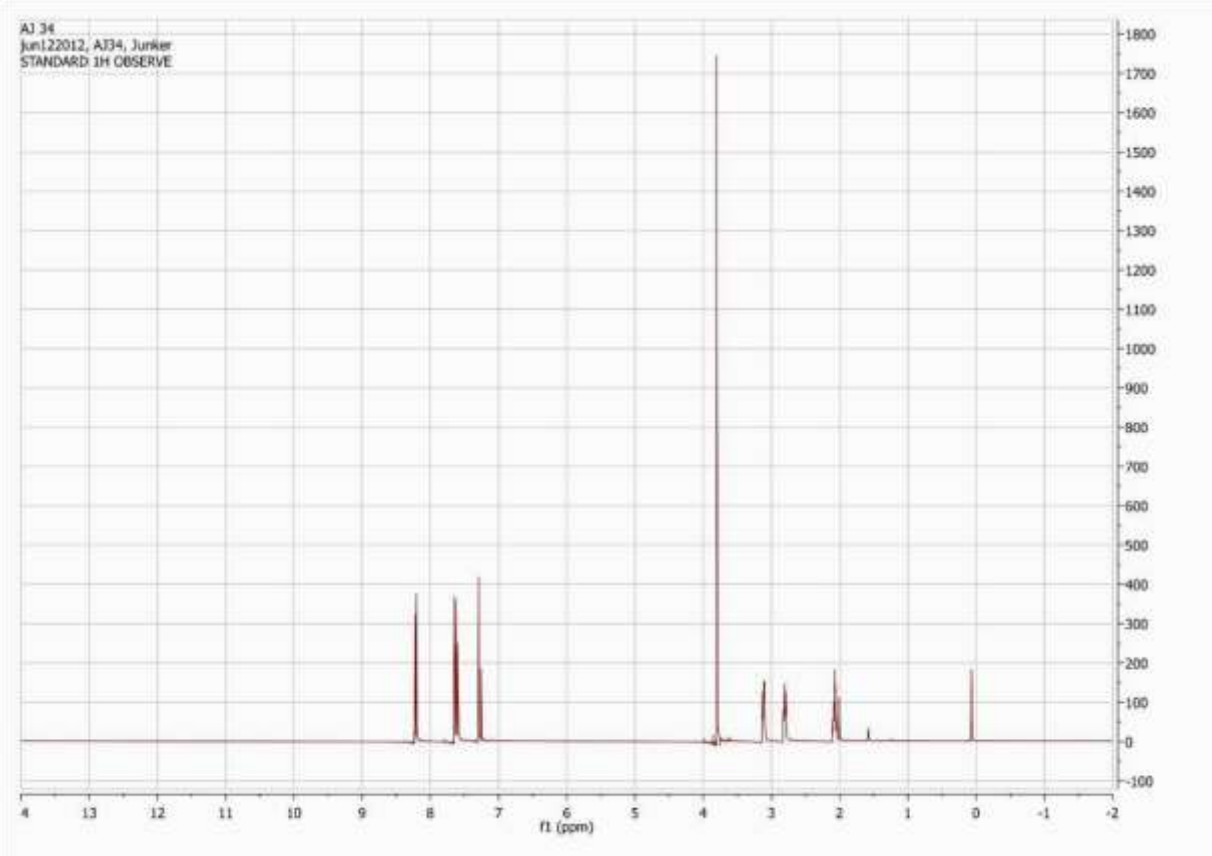
Acquisition Parameter

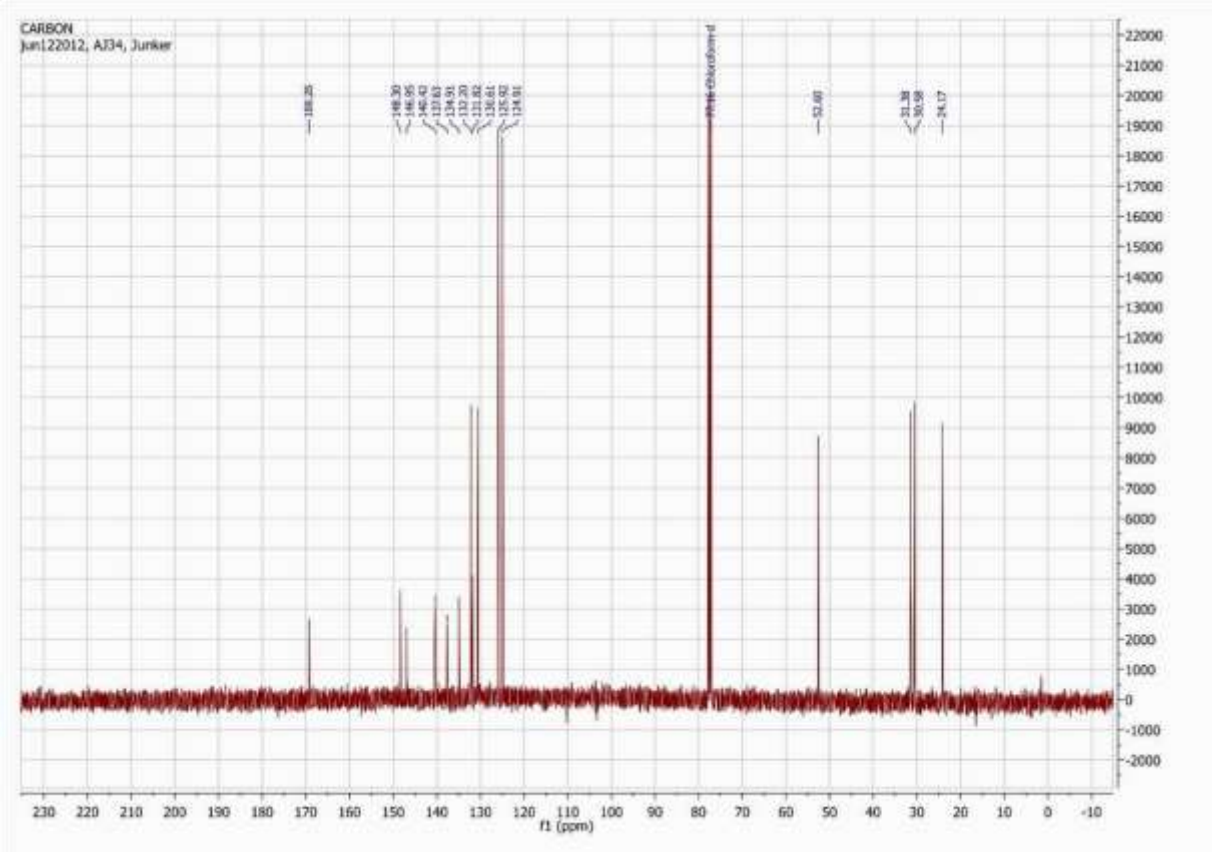
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

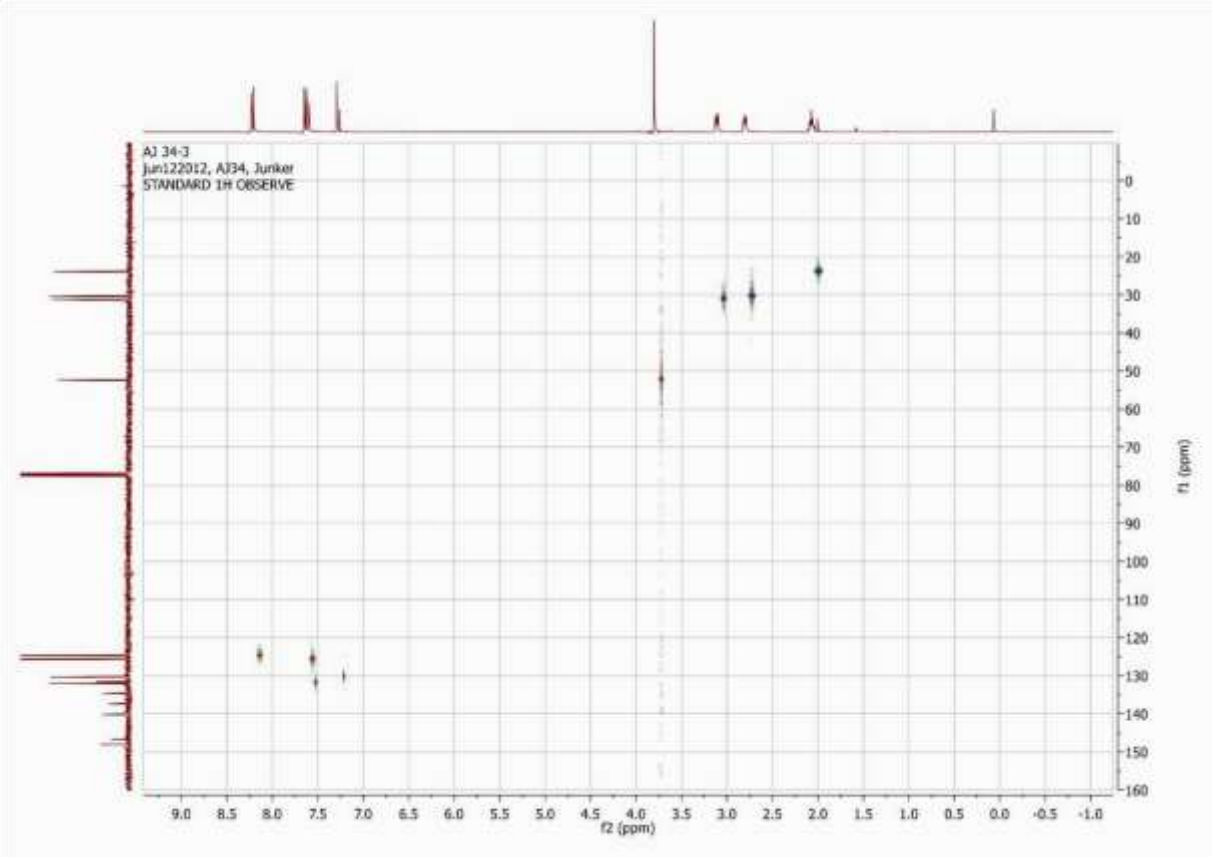


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
353.0845	1	C 18 H 16 F 3 O 2 S	31.22	353.0818	-2.8	-7.8	4.7	9.5	even	ok
	2	C 15 H 17 F 4 O 3 S	68.42	353.0829	-1.6	-4.6	11.8	5.5	even	ok
	3	C 13 H 14 F 5 N 4 S	100.00	353.0854	0.9	2.5	15.9	6.5	even	ok
	4	C 15 H 20 F 3 O 2 S 2	96.08	353.0851	0.6	1.7	23.0	4.5	even	ok
	5	C 13 H 19 F 6 S 2	43.84	353.0827	-1.8	-5.2	25.7	1.5	even	ok
	6	C 12 H 18 F 5 O 4 S	93.64	353.0840	-0.5	-1.3	28.1	1.5	even	ok
	7	C 12 H 21 F 4 O 3 S 2	40.45	353.0863	1.8	5.0	31.3	0.5	even	ok
	8	C 12 H 24 F 3 O 2 S 3	3.72	353.0885	4.0	11.3	42.0	-0.5	even	ok

Methyl 2-(4-nitrophenyl)-7,8-dihydro-6*H*-[7]annulene[*b*]thiophene-5-carboxylate (9g)






AJ 34-3
jun122012, AJ34, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 31.01.13 02:27

Reported: 01.02.13 09:38
Processed: 01.02.13 09:38

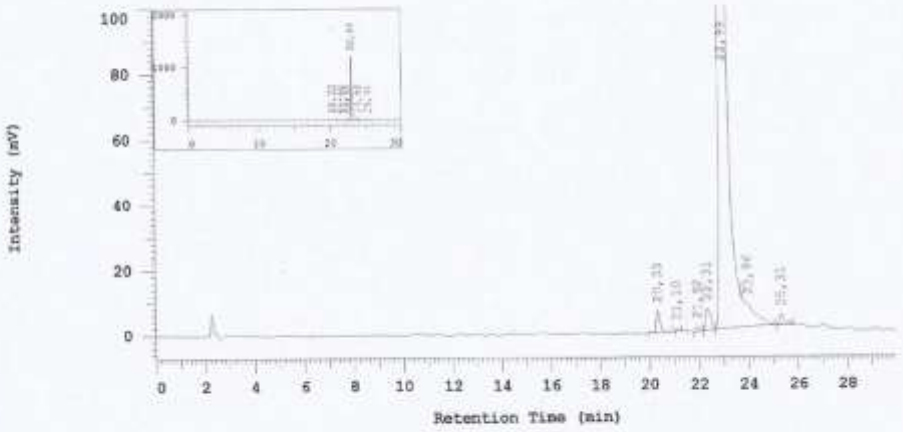
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5883\
Application: Chromni

Sample Name: AJ34

Injection from this vial: 1 of 1

Series:5883
Vial Number: 11
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

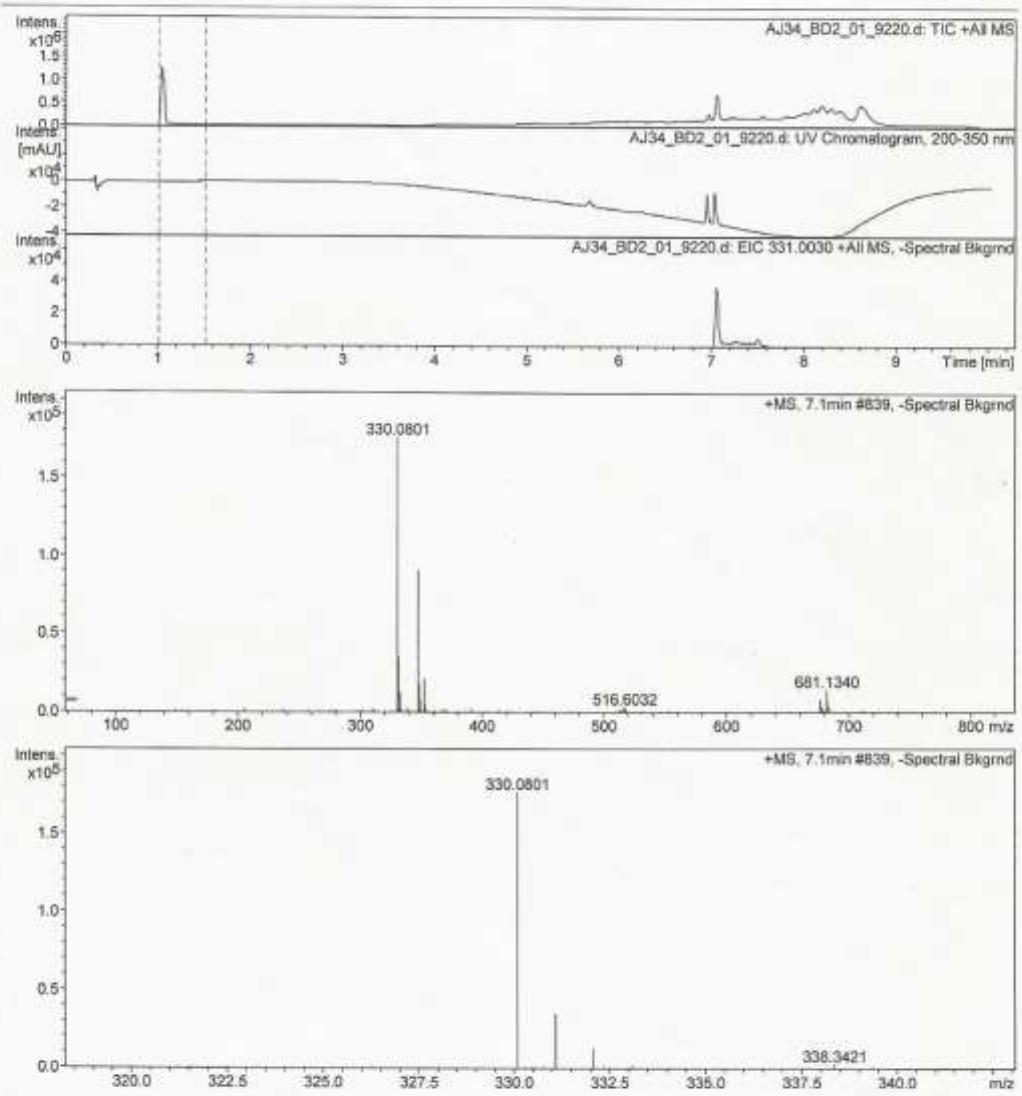
No.	RT	Area	Conc 1	BC
1	20,33	77359	0,589	BB
2	21,10	3944	0,030	BB
3	21,92	13765	0,105	BB
4	22,31	93427	0,711	BB
5	22,99	12596748	95,862	MC
6	23,96	310962	2,366	MC
7	25,31	44325	0,337	BB
		13140530	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ34_BD2_01_9220.d	Acquisition Date	1/14/2013 7:08:16 PM
Method	tune_low_lcms_routine_positiv_10min.m		
Sample Name	AJ34	Operator	Meiners
Comment	Junker Kalibration mit Li-Formate	Instrument	micrOTOF-Q II



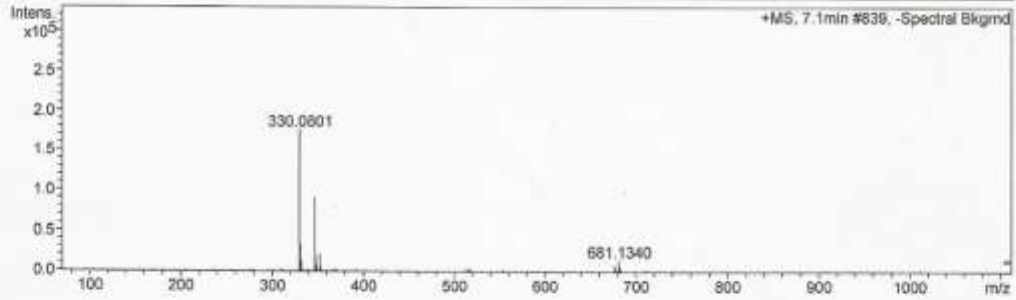
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ34_BD2_01_9220.d	Acquisition Date	1/14/2013 7:08:16 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ34	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker Kalibration mit Li-Formate		

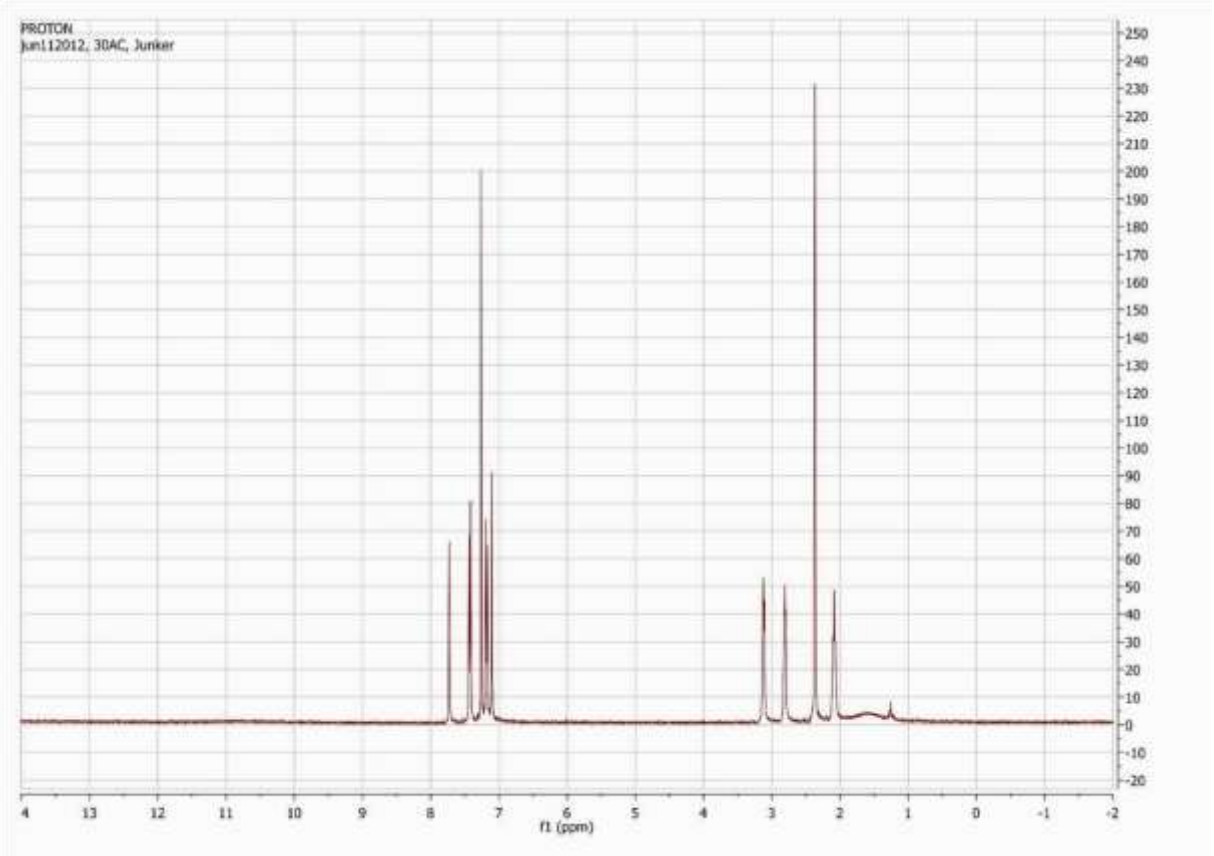
Acquisition Parameter

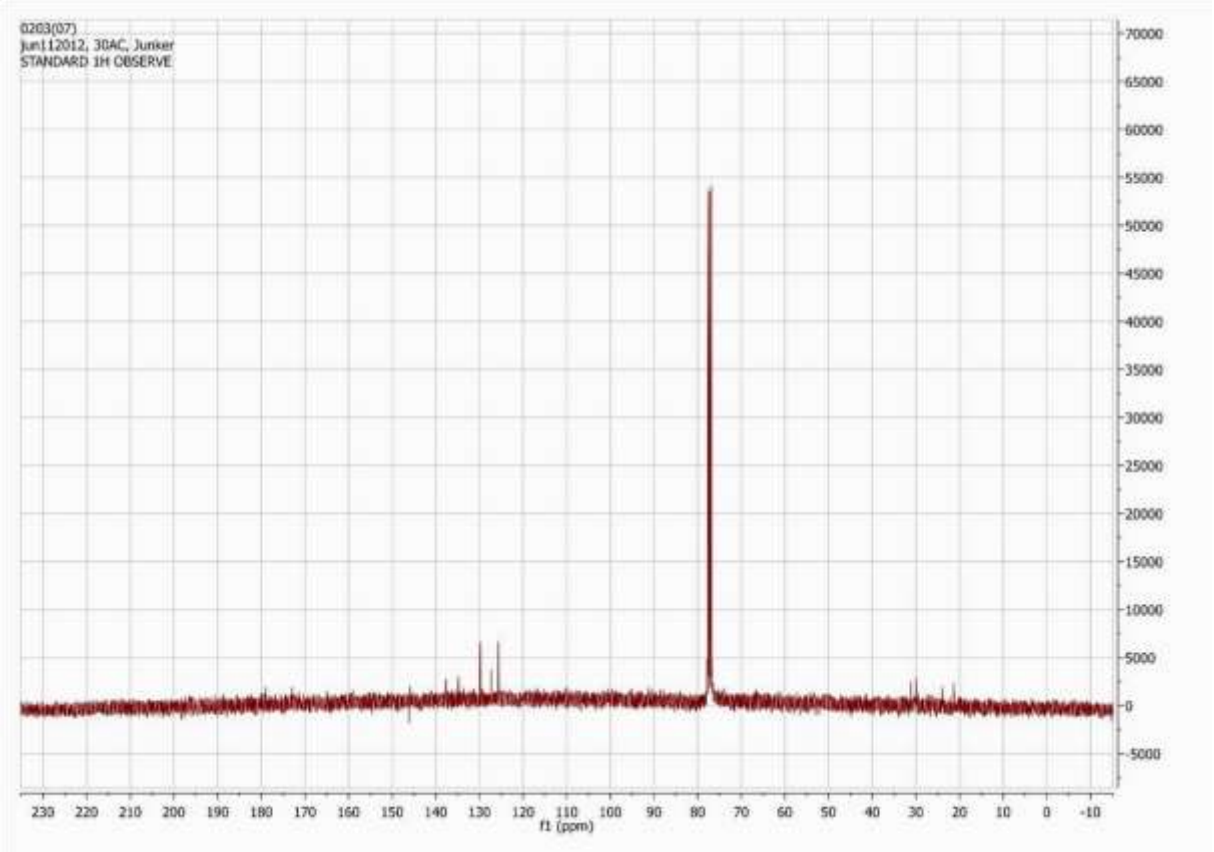
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

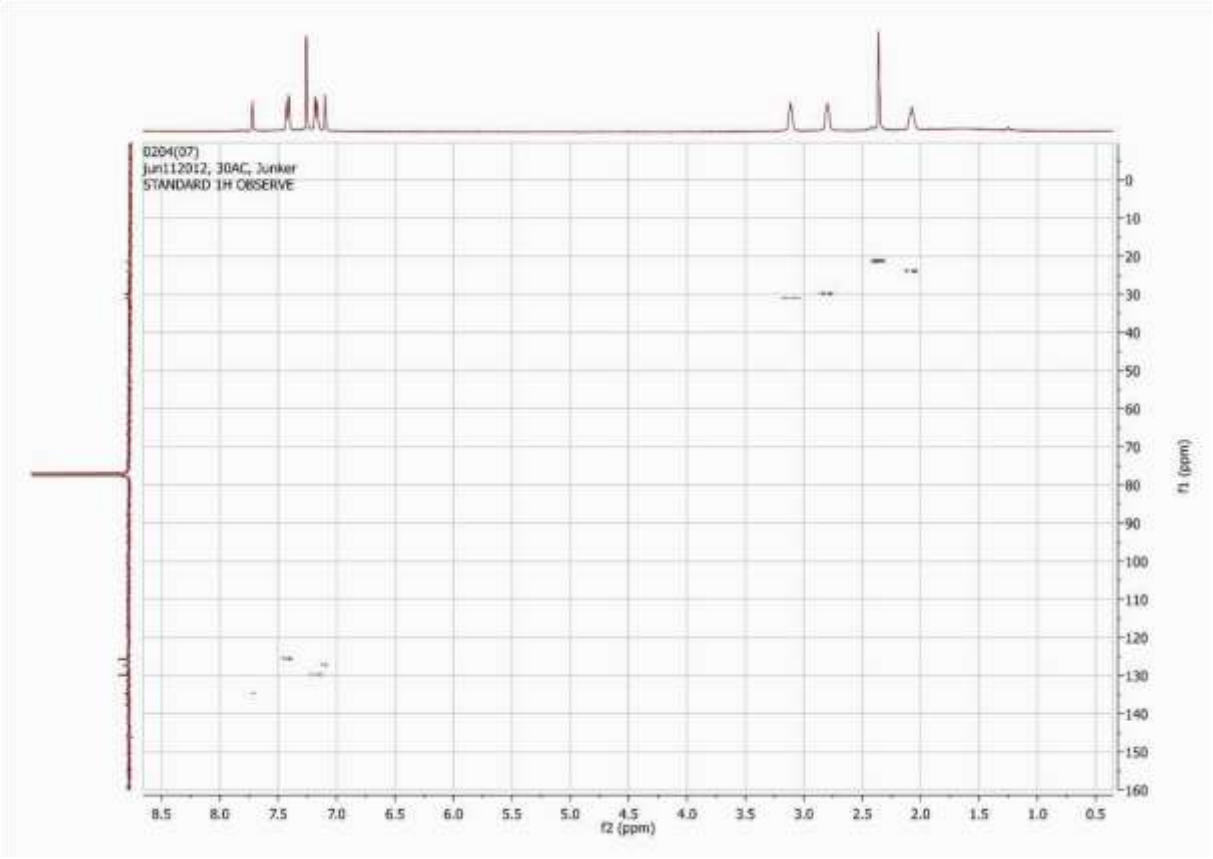


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
330.0801	1	C 17 H 16 N O 4 S	100.00	330.0795	-0.6	-1.9	1.8	10.5	even	ok
	2	C 13 H 12 N 7 O 2 S	11.07	330.0768	-3.3	-10.0	11.0	11.5	even	ok
	3	C 18 H 12 N 5 S	77.47	330.0808	0.7	2.2	12.7	15.5	even	ok
	4	C 14 H 20 N O 4 S 2	13.88	330.0828	2.8	8.4	25.4	5.5	even	ok
	5	C 13 H 20 N 3 O S 3	3.60	330.0763	-3.8	-11.4	38.1	5.5	even	ok

2-(4-Methylphenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylic acid (10a)



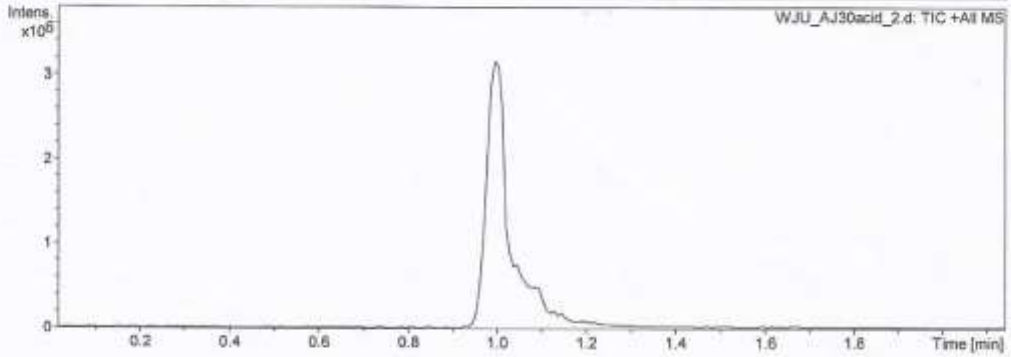


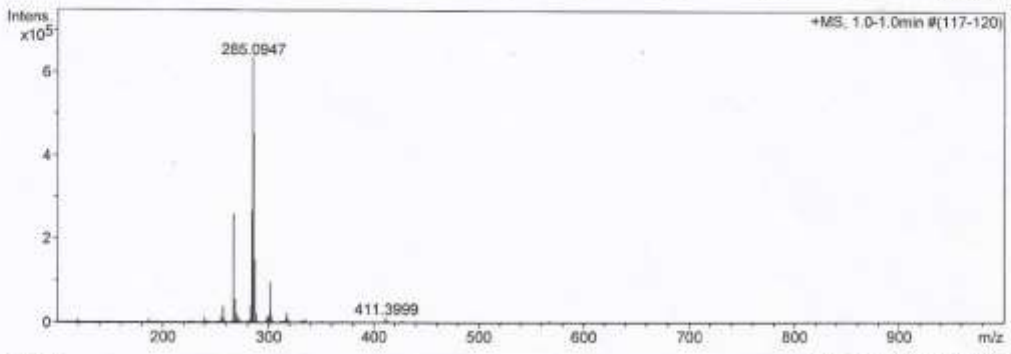

0204(07)
jun112012, 30AC, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

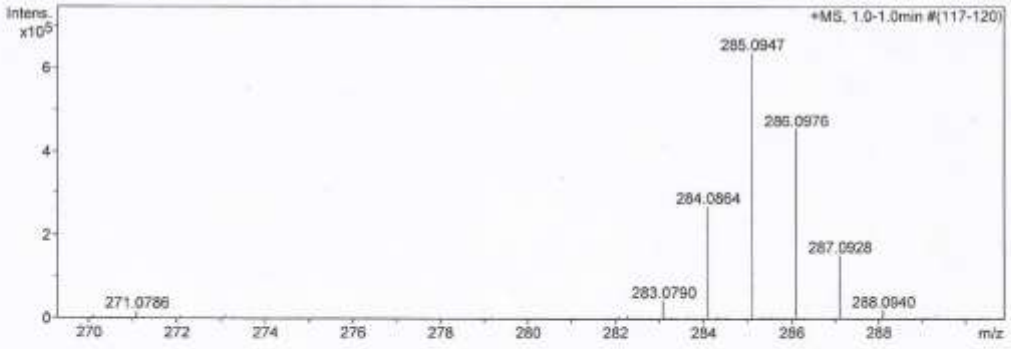
Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ30acid_2.d	Acquisition Date	8/28/2012 1:42:35 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ30acid	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		







Bruker Compass DataAnalysis 4.0 printed: 8/28/2012 1:46:47 PM Page 1 of 1

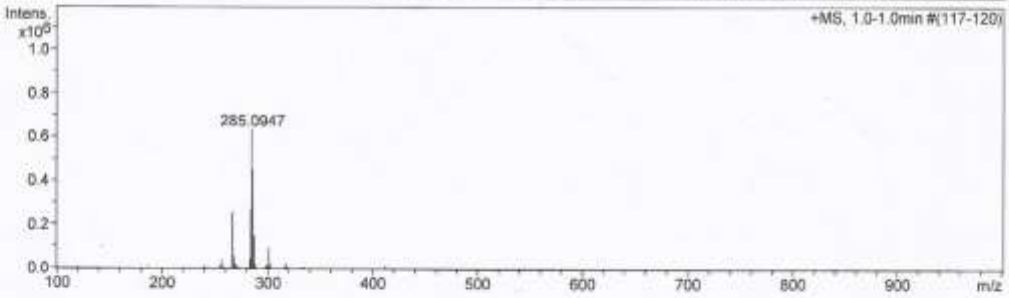
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ30acid_2.d	Acquisition Date	8/28/2012 1:42:35 PM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ30acid	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
285.0947	1	C 7 H 21 N 6 S 3	15.16	285.0984	3.7	13.0	267.8	0.5	even	ok
	2	C 17 H 17 O 2 S	100.00	285.0944	-0.4	-1.3	274.0	9.5	even	ok
	3	C 14 H 21 O 2 S 2	7.66	285.0977	3.0	10.5	260.1	4.5	even	ok
	4	C 13 H 13 N 6 S	4.06	285.0917	-3.0	-10.7	285.2	10.5	even	ok
	5	C 10 H 17 N 6 S 2	13.56	285.0951	0.3	1.1	291.3	5.5	even	ok
	6	C 12 H 17 N 2 O 4 S	0.23	285.0904	-4.4	-15.4	296.8	5.5	even	ok
	7	C 9 H 21 N 2 O 4 S 2	2.22	285.0937	-1.0	-3.6	303.2	0.5	even	ok
	8	C 7 H 13 N 10 O S	0.00	285.0989	4.2	14.6	358.4	6.5	even	ok
	9	C 6 H 17 N 6 O 5 S	0.00	285.0976	2.8	9.9	371.1	1.5	even	ok

HPLC

Analyzed: 23.08.12 03:18

Reported: 24.08.12 11:20
Processed: 24.08.12 11:20

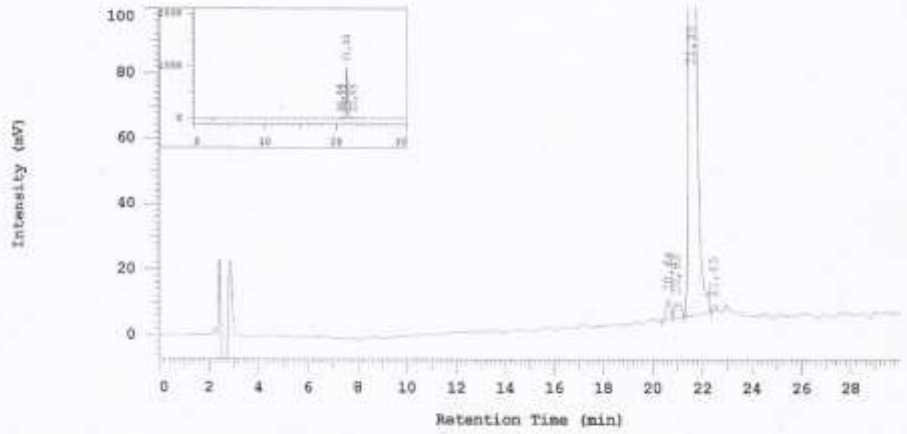
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5122\
Application: Chromni

Series:5122
Vial Number: 14
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ30acid

Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1

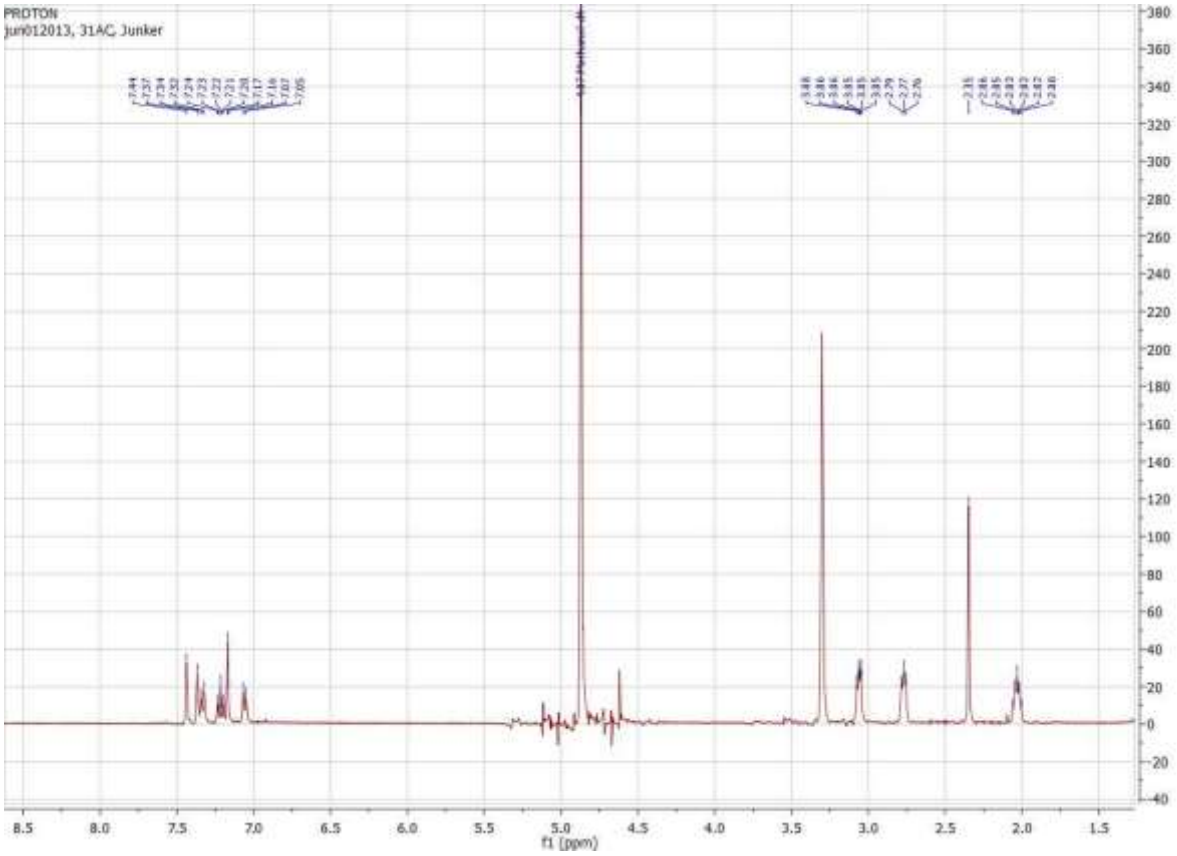


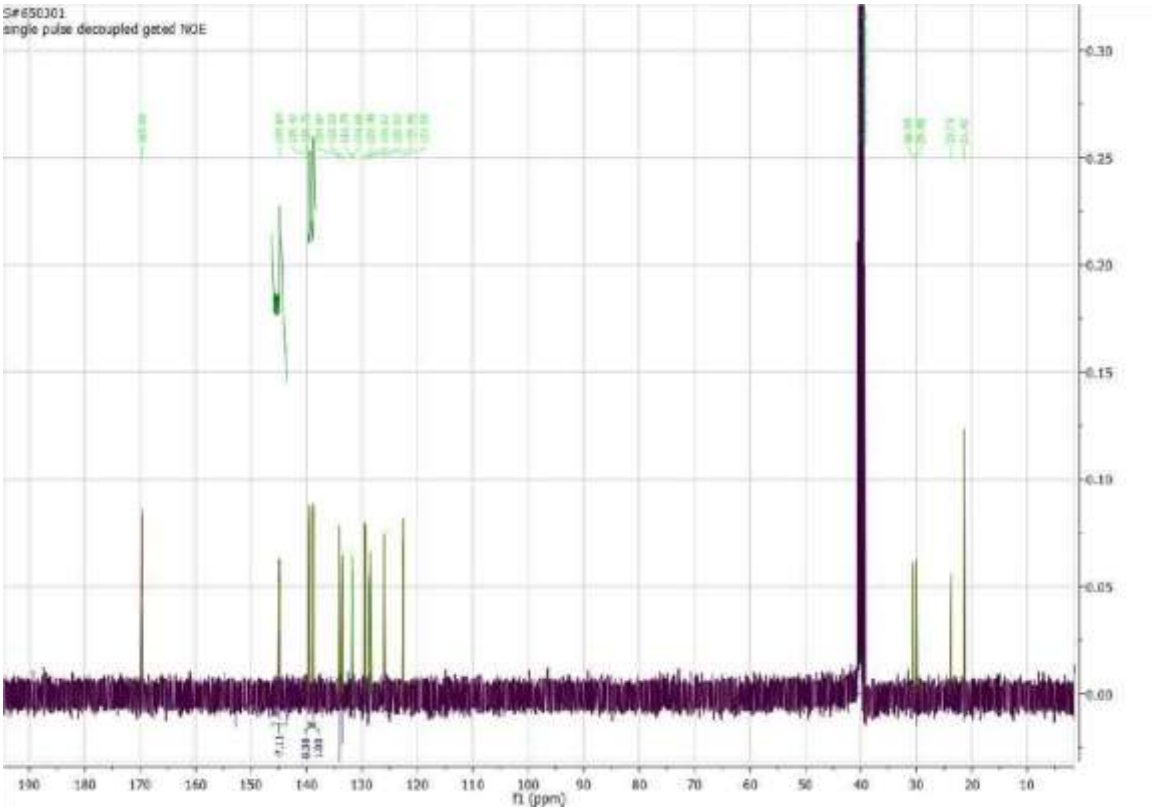
Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

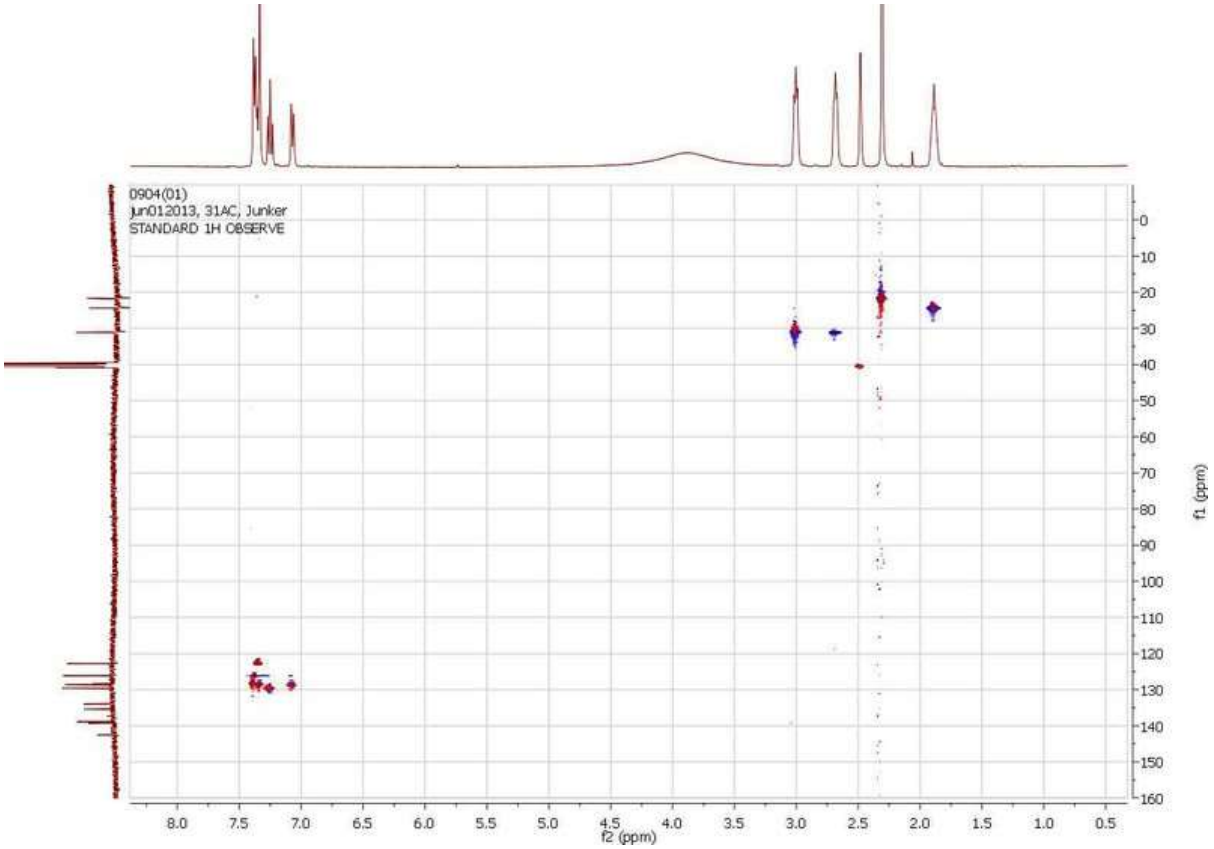
No.	RT	Area	Conc 1	BC
1	20,64	76136	0,706	BV
2	20,93	79988	0,741	VB
3	21,55	10621110	98,443	BB
4	22,45	11808	0,109	BB
		10789042	100,000	

Peak rejection level: 0

2-(3-Methylphenyl)-7,8-dihydro-6*H*-[7]annuleno[b]thiophene-5-carboxylic acid (10b)




S#650301
single pulse decoupled gated NOE
f1 (ppm)


0904(01)
jun012013, 31AC, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

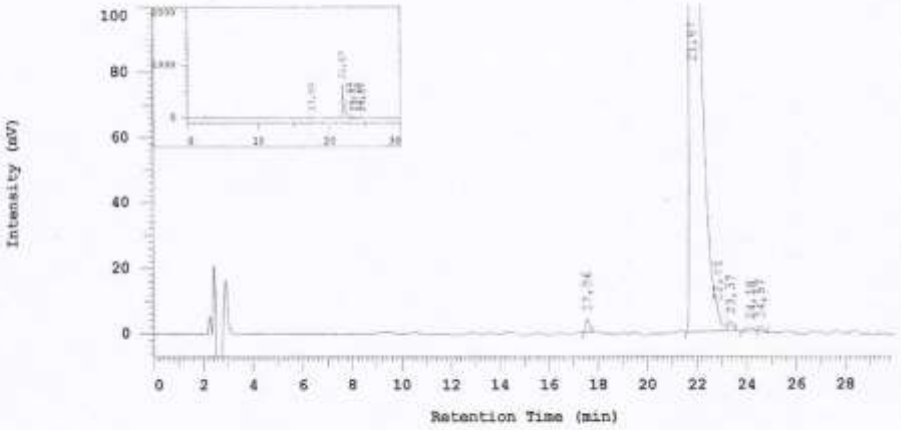
Analyzed: 09.01.13 01:52

Data Path: D:\WIN32APP\HSM\Chromni\DATA\5766\
Application: Chromni
Sample Name: AJ31AC
Injection from this vial: 1 of 1

Reported: 09.01.13 16:01
Processed: 09.01.13 16:01

Series:5766
Vial Number: 18
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010 Developed by: Jens
Solvent A: Wasser + 0,05%TFA Solvent B: ACN + 0,05%TFA

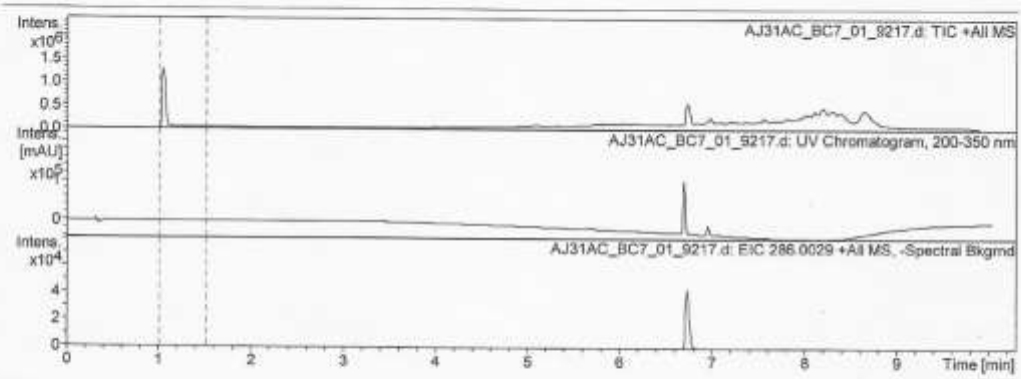
No.	RT	Area	Conc 1	BC
1	17,56	36436	0,337	BB
2	21,87	10565120	97,702	MC
3	22,84	140020	1,295	MC
4	23,37	31931	0,295	MC
5	24,18	23044	0,213	MC
6	24,57	17069	0,158	MC
		10813620	100,000	

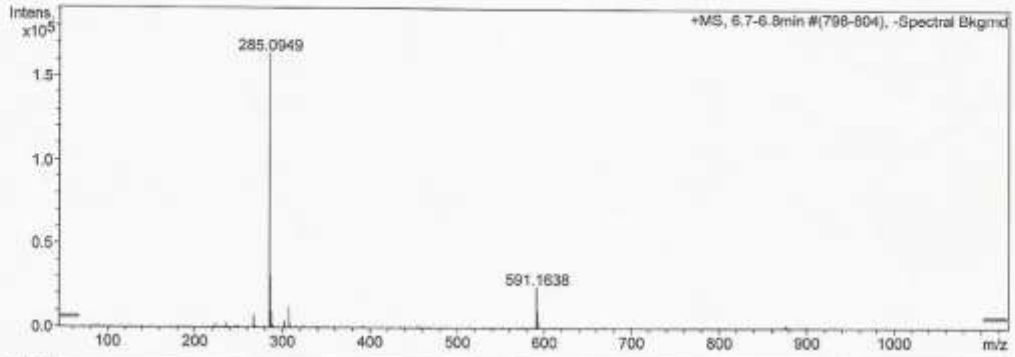
Peak rejection level: 0

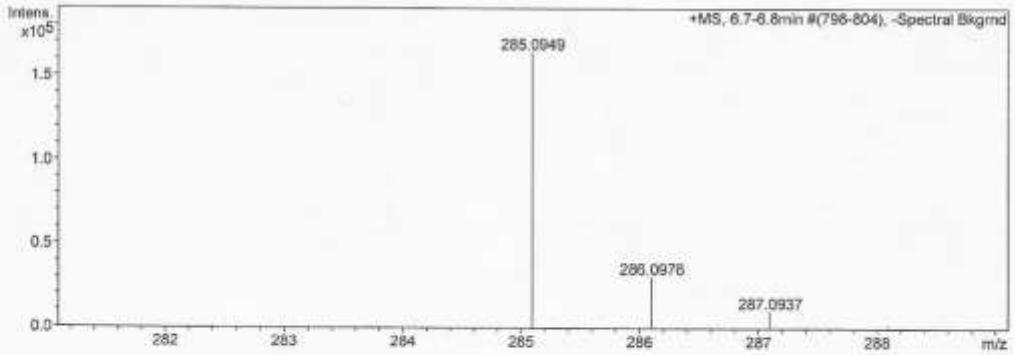
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ31AC_BC7_01_9217.d	Acquisition Date	1/14/2013 6:33:57 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ31AC	Instrument	micrOTOF-Q II
Comment	Junker Kalibration mit Li-Formate		







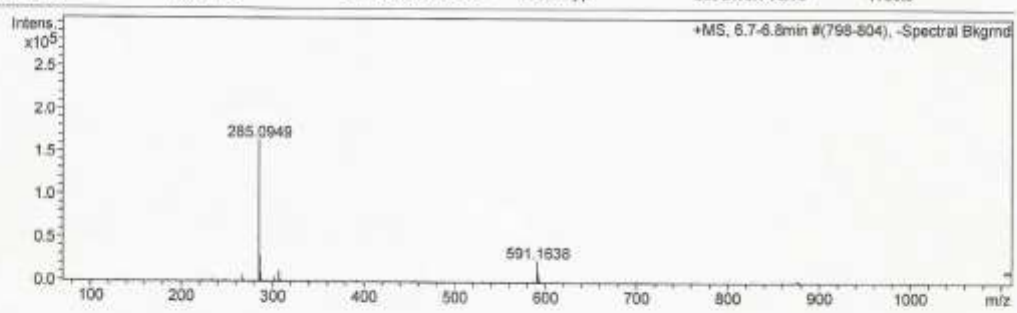
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ31AC_BC7_01_9217.d	Acquisition Date	1/14/2013 6:33:57 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ31AC	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker Kalibration mit Li-Formate		

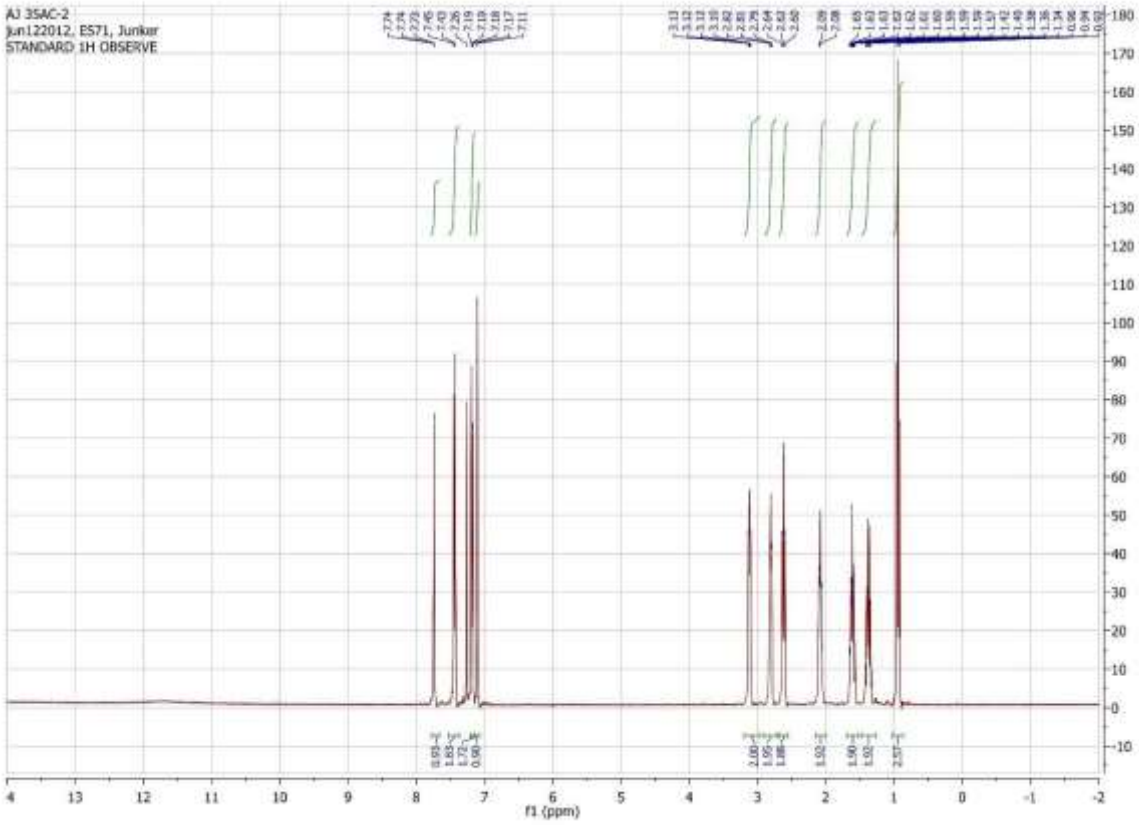
Acquisition Parameter

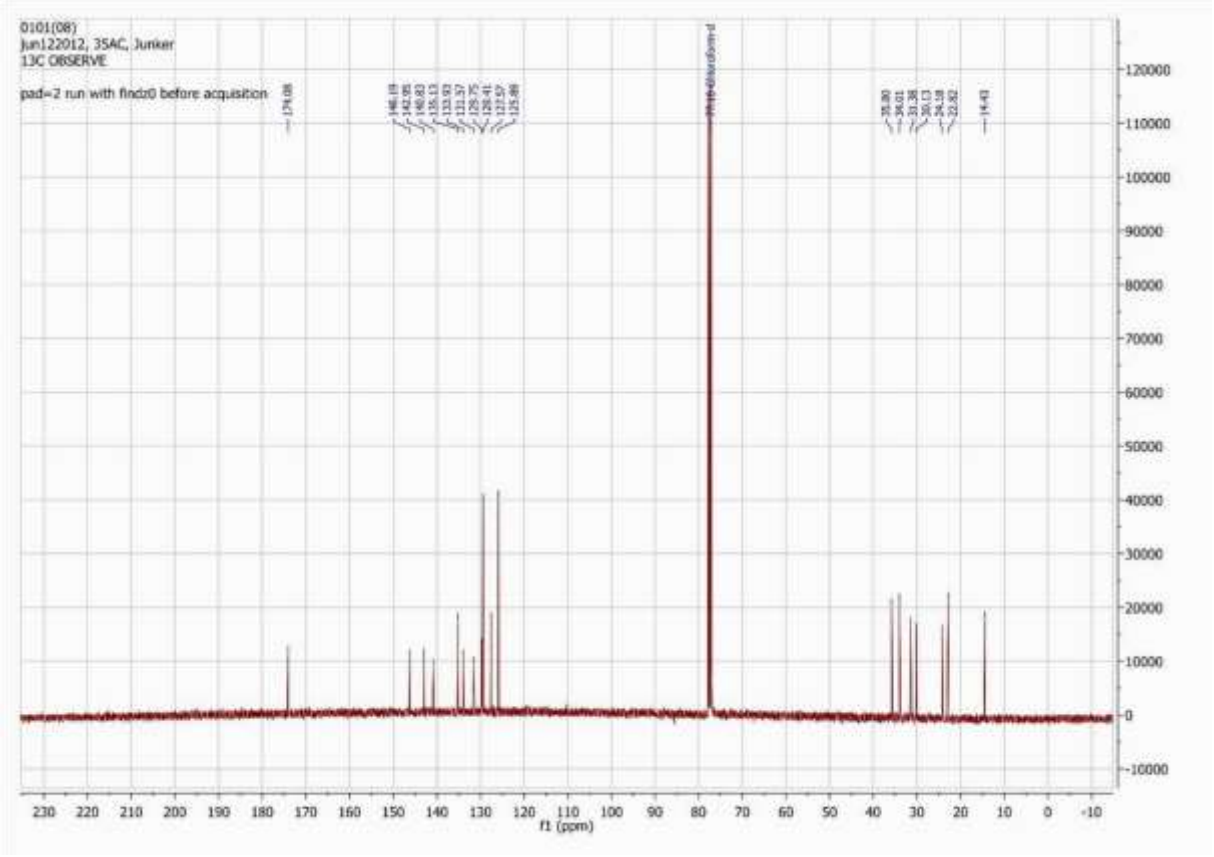
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste

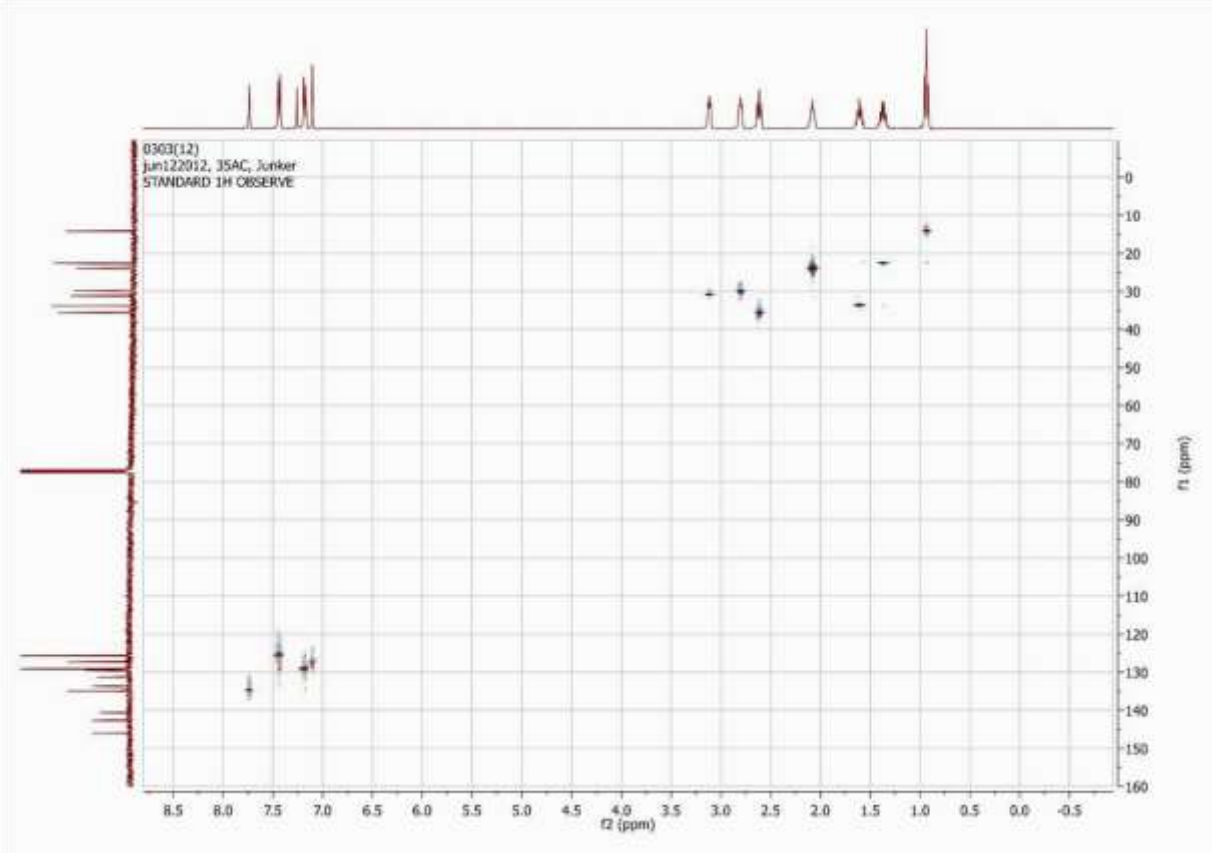


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
285.0949	1	C 17 H 17 O 2 S	100.00	285.0944	-0.5	-1.8	4.9	9.5	even	ok
	2	C 13 H 13 N 6 S	13.15	285.0917	-3.2	-11.2	7.3	10.5	even	ok
	3	C 14 H 21 O 2 S 2	12.71	285.0977	2.9	10.0	25.9	4.5	even	ok

2-(4-*n*-Butylphenyl)-7,8-dihydro-6*H*-[7]annulene[*b*]thiophene-5-carboxylic acid (10c)




0101(08)
Jun122012, 3SAC, Junker
13C OBSERVE
pad=2 run with findz0 before acquisition
f1 (ppm)


0303(12)
Jun122012, 3SAC, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 09.01.13 04:38

Reported: 09.01.13 16:04
Processed: 09.01.13 16:04

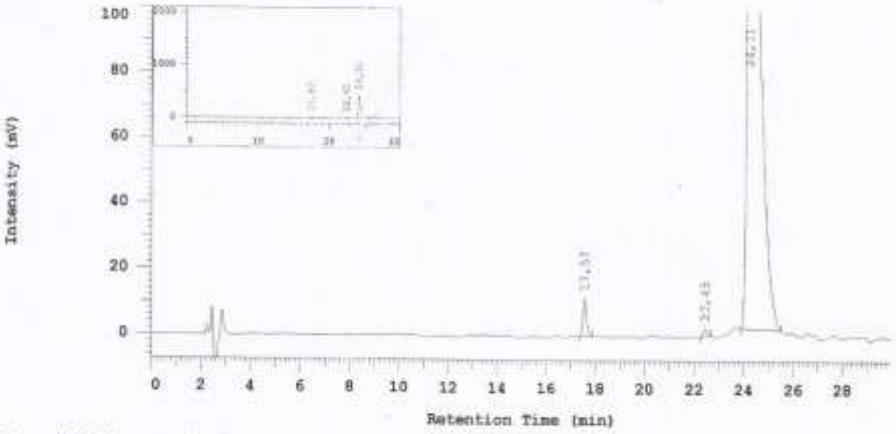
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5770\
Application: Chromni

Series:5770
Vial Number: 21
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ35AC

Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

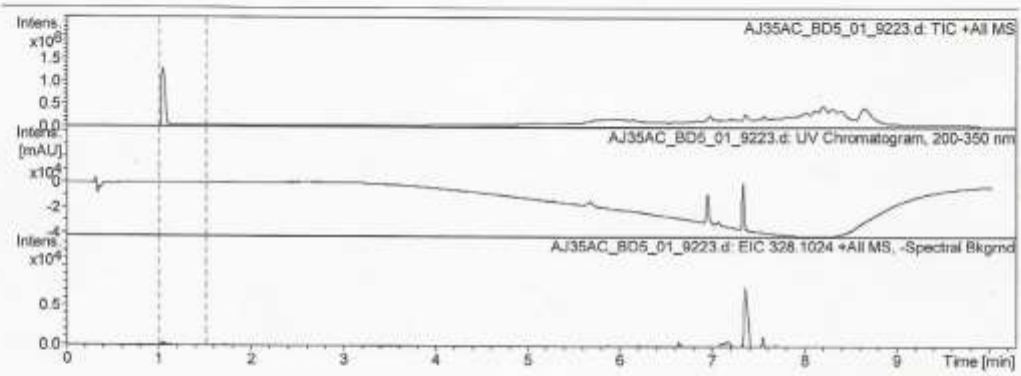
No.	RT	Area	Conc 1	BC
1	17,57	112246	1,205	BB
2	22,45	26603	0,286	BB
3	24,21	9172728	98,509	BB
		9311577	100,000	

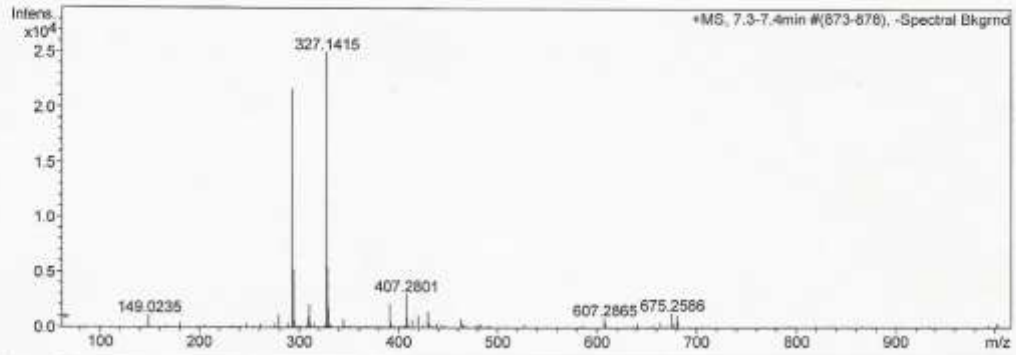
Peak rejection level: 0

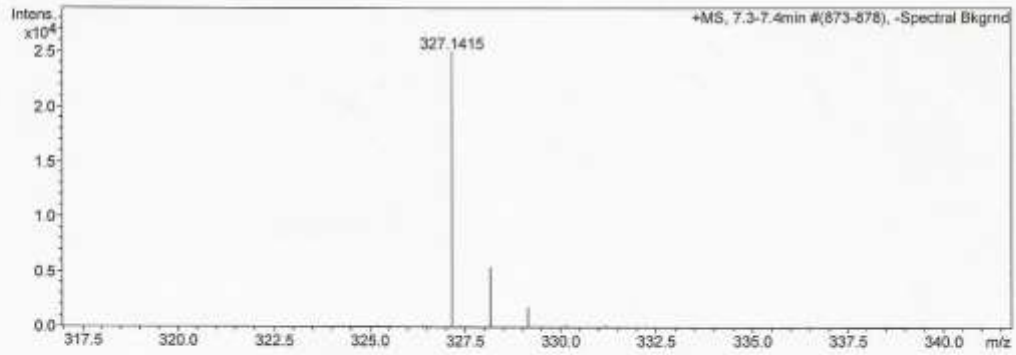
Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ35AC_BD5_01_9223.d	Acquisition Date	1/14/2013 7:42:37 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ35AC	Instrument	micrOTOF-Q II
Comment	Junker Kalibration mit Li-Formate		







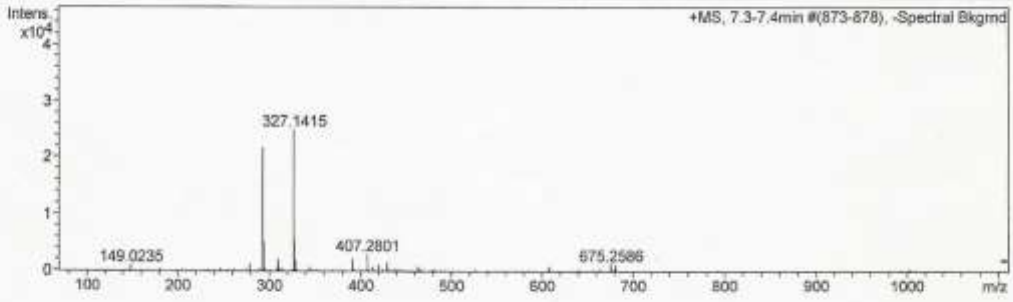
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\2013_01_14\AJ35AC_BD5_01_9223.d	Acquisition Date	1/14/2013 7:42:37 PM
Method	tune_low_lcms_routine_positiv_10min.m	Operator	Meiners
Sample Name	AJ35AC	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker Kalibration mit Li-Formate		

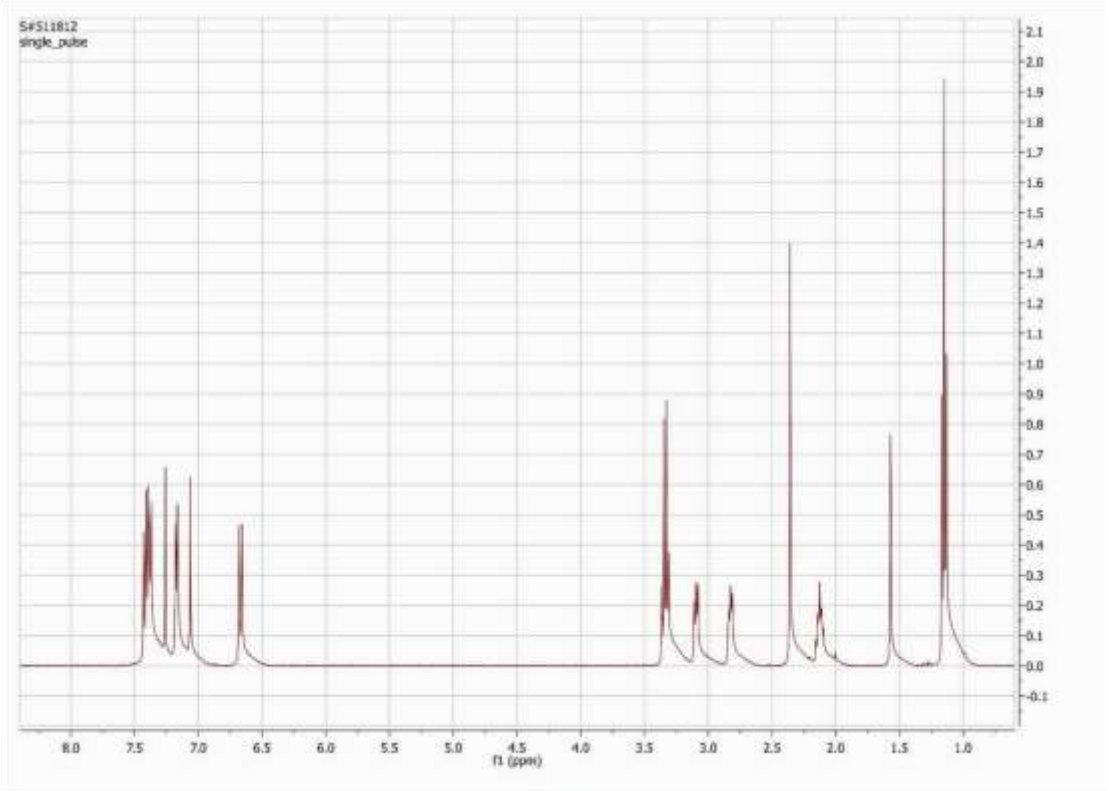
Acquisition Parameter

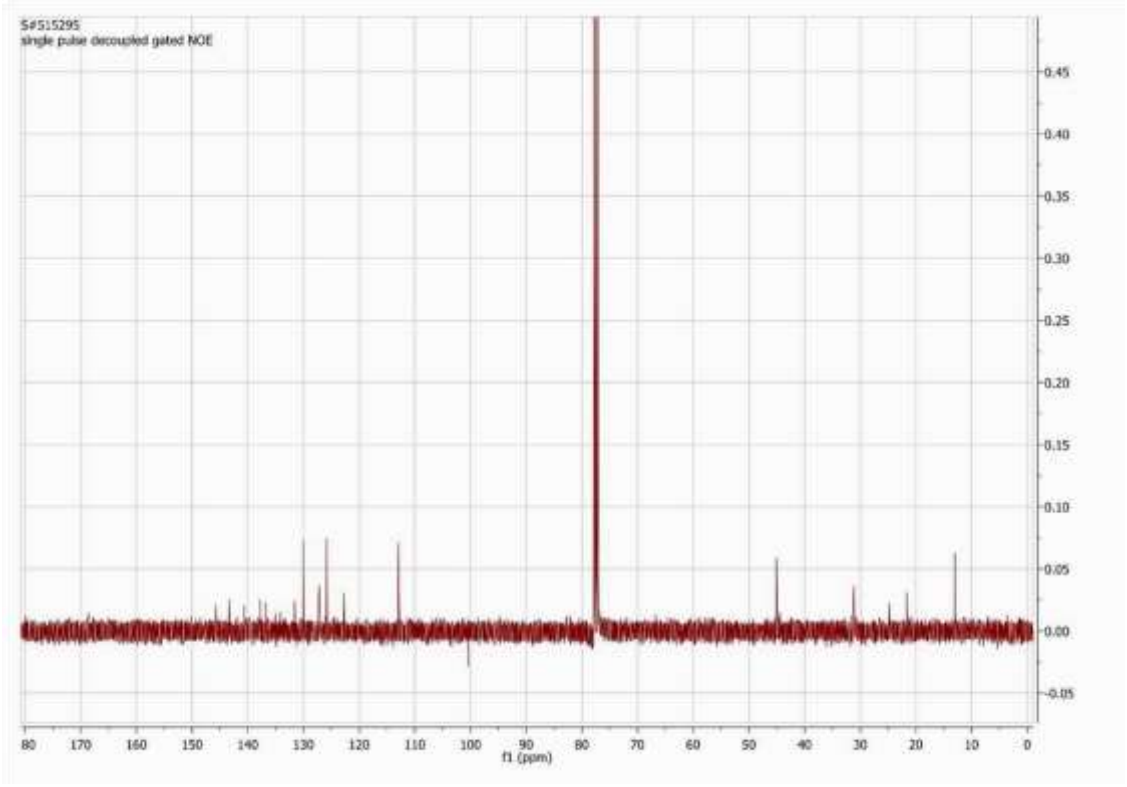
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	2.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	9.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	300.0 Vpp	Set Divert Valve	Waste



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
327.1415	1	C 20 H 23 O 2 S	100.00	327.1413	-0.2	-0.7	4.2	9.5	even	ok
	2	C 16 H 19 N 6 S	15.16	327.1386	-2.9	-8.9	7.4	10.5	even	ok
	3	C 17 H 27 O 2 S 2	8.71	327.1447	3.2	9.6	23.2	4.5	even	ok

N-[4-(Diethylamino)phenyl]-2-(4-methylphenyl)-7,8-dihydro-6*H*-[7]annuleno[*b*]thiophe-ne-5carboxamide (1aA)





HPLC

Analyzed: 18.07.12 23:27

Reported: 19.07.12 13:31
Processed: 19.07.12 13:31

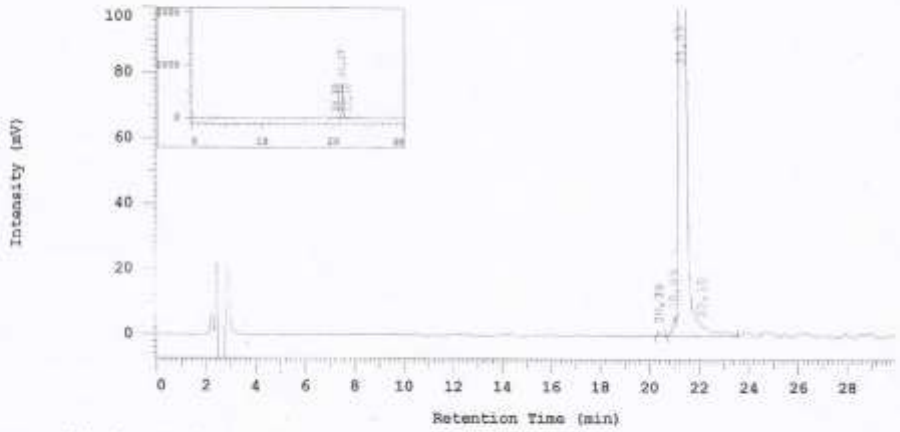
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5002\
Application: Chromni

Sample Name: AJ4601

Injection from this vial: 1 of 1

Series:5002
Vial Number: 6
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

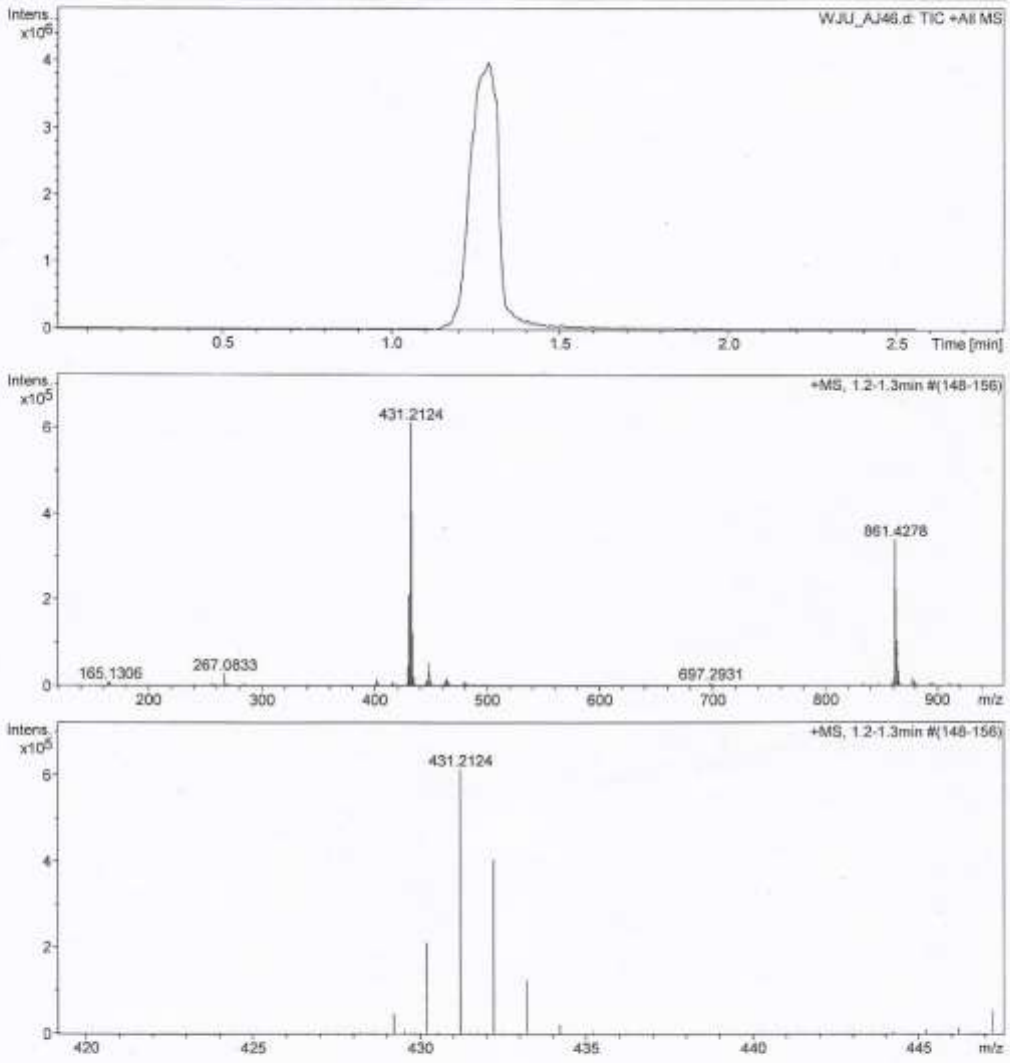
No.	RT	Area	Conc 1	BC
1	20,39	13812	0,168	MC
2	20,99	51741	0,628	MC
3	21,27	7941576	96,333	MC
4	22,10	236717	2,871	MC
		8243846	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ46.d	Acquisition Date	8/29/2012 8:47:10 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ46	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		



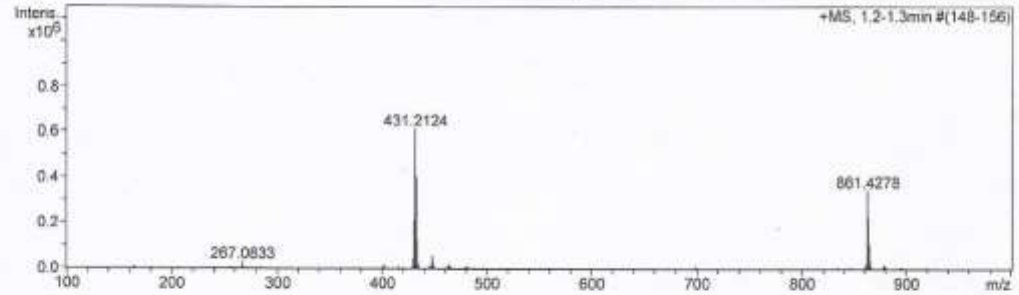
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\iPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ46.d	Acquisition Date	8/29/2012 8:47:10 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ46	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

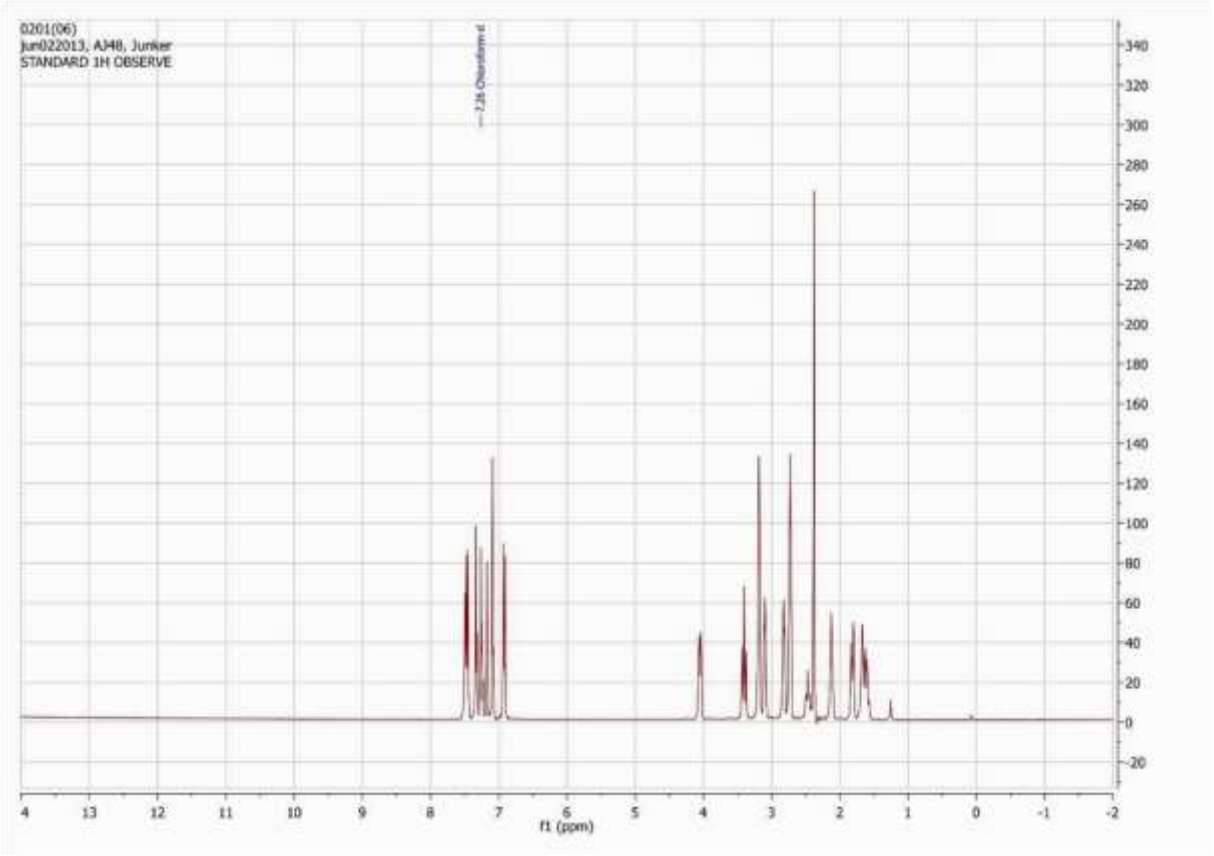
Acquisition Parameter

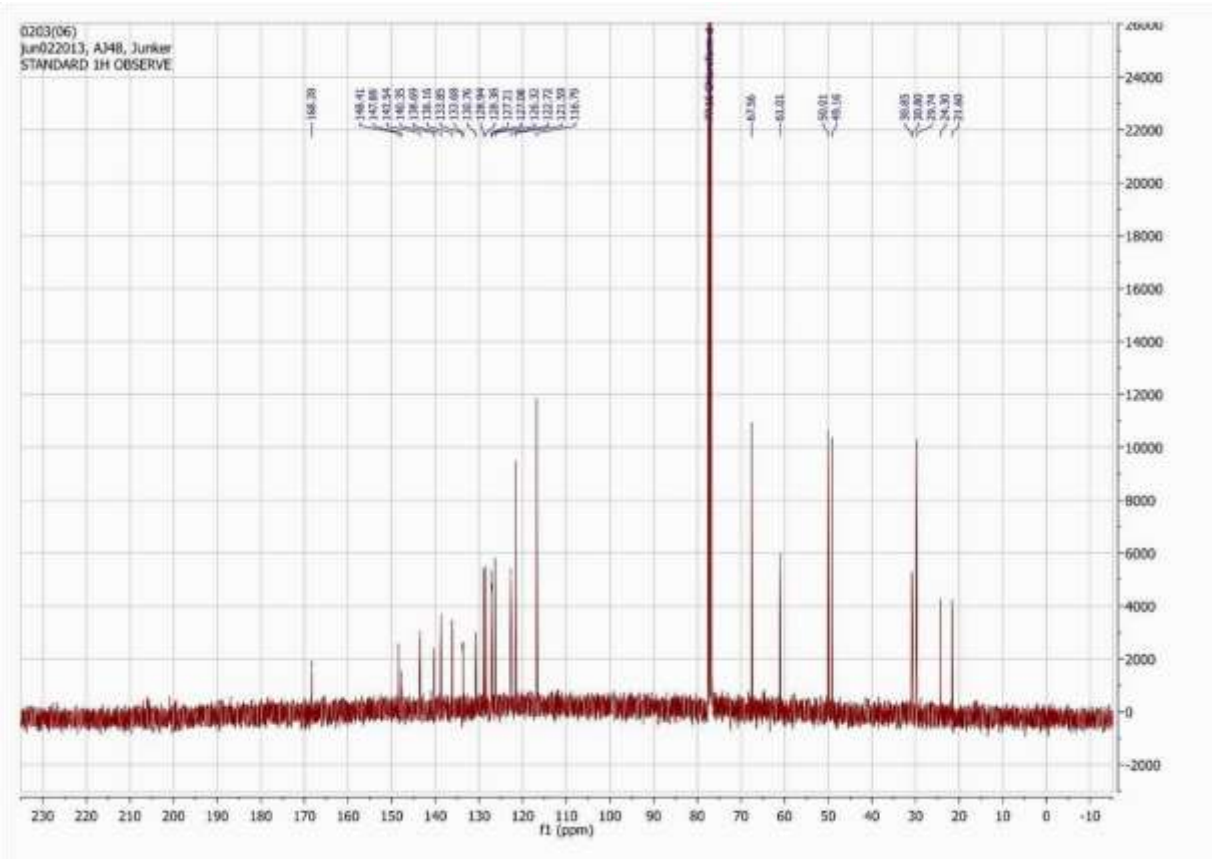
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

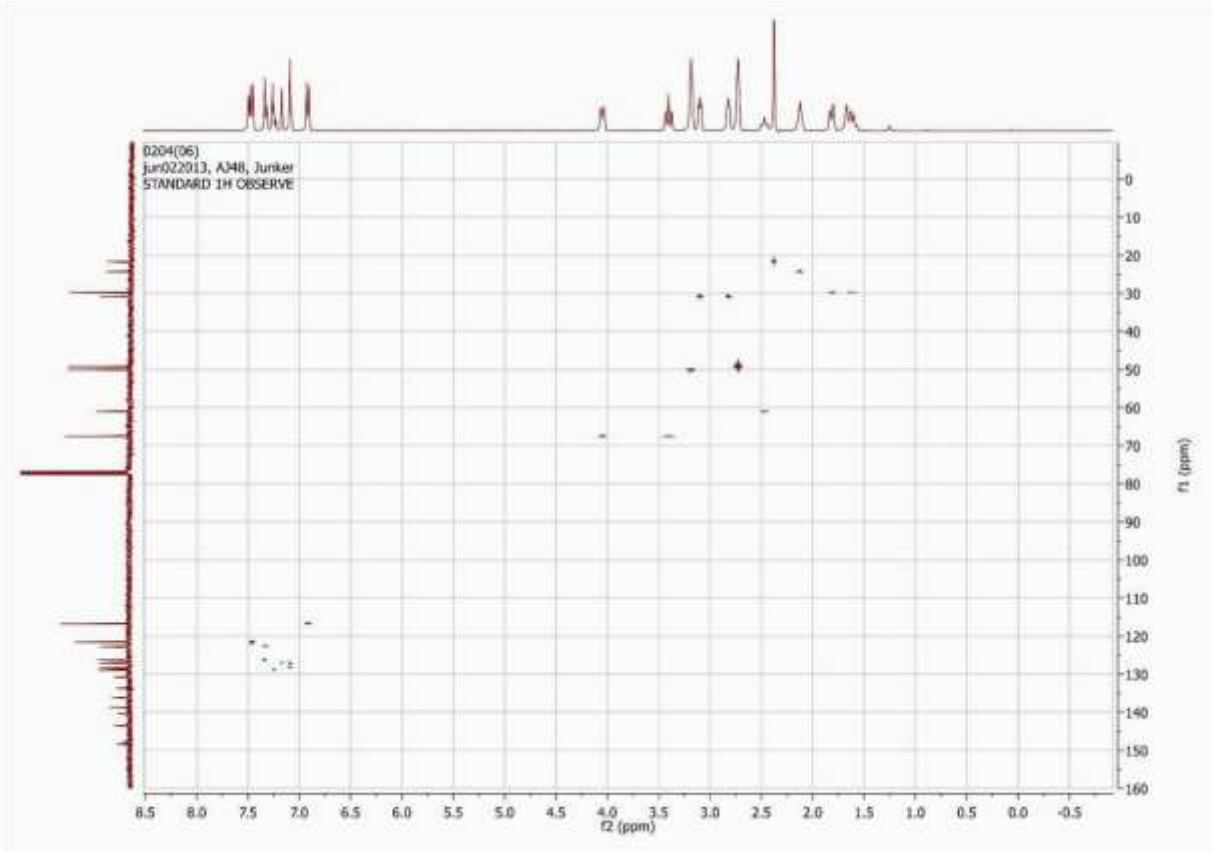


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
431.2124	1	C 22 H 39 O 2 S 3	100.00	431.2107	-1.7	-3.9	174.3	3.5	even	ok
	2	C 27 H 31 N 2 O S	25.27	431.2152	2.8	6.5	180.6	13.5	even	ok
	3	C 18 H 35 N 6 S 3	3.36	431.2080	-4.4	-10.2	184.2	4.5	even	ok
	4	C 15 H 39 N 6 S 4	32.03	431.2114	-1.0	-2.3	194.3	-0.5	even	ok
	5	C 22 H 31 N 4 O 3 S	12.47	431.2111	-1.2	-2.8	204.0	9.5	even	ok
	6	C 12 H 35 N 10 O S 3	2.78	431.2152	2.8	6.6	207.1	0.5	even	ok
	7	C 19 H 35 N 4 O 3 S 2	3.56	431.2145	2.1	5.0	211.0	4.5	even	ok
	8	C 21 H 35 O 7 S	1.63	431.2098	-2.6	-5.9	215.8	4.5	even	ok
	9	C 18 H 27 N 10 O S	0.35	431.2085	-3.9	-9.1	217.7	10.5	even	ok
	10	C 15 H 31 N 10 O S 2	3.41	431.2118	-0.5	-1.3	222.7	5.5	even	ok
	11	C 18 H 39 O 7 S 2	2.85	431.2132	0.8	1.9	223.1	-0.5	even	ok
	12	C 14 H 35 N 6 O 5 S 2	0.49	431.2105	-1.9	-4.4	234.5	0.5	even	ok
	13	C 15 H 35 N 4 O 8 S	0.01	431.2170	4.6	10.8	241.1	0.5	even	ok
	14	C 12 H 27 N 14 O 2 S	0.06	431.2157	3.3	7.7	242.7	6.5	even	ok
	15	C 11 H 31 N 10 O 6 S	0.07	431.2143	2.0	4.6	252.7	1.5	even	ok
	16	C 8 H 23 N 20 S	0.14	431.2130	0.6	1.4	254.6	7.5	even	ok

2-(3-Methylphenyl)-N-{4-[4-(tetrahydro-2*H*-pyran-4-yl)piperazin-1-yl]phenyl}-7,8-dihydro-6*H*[7]annuleno[*b*]thiophene-5-carboxamide (1bB)




0203(06)
jun022013, AJ48, Junker
STANDARD 1H OBSERVE
f1 (ppm)


0204(06)
jun022013, AJ48, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

HPLC

Analyzed: 02.08.12 03:28

Reported: 02.08.12 15:01
Processed: 02.08.12 15:01

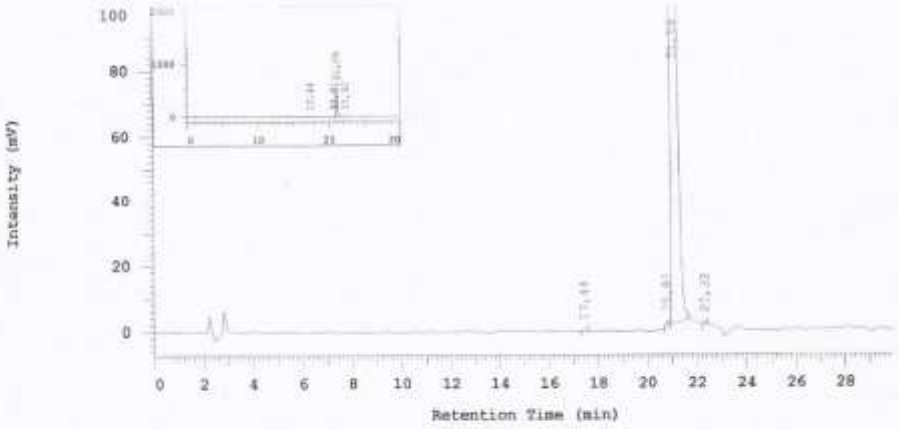
Data Path: D:\WIN32APP\HSM\Chromni\DATA\5050\
Application: Chromni

Series:5050
Vial Number: 12
Vial Type: UNK
Volume: 5,0 ul

Sample Name: AJ4801

Injection from this vial: 1 of 1

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

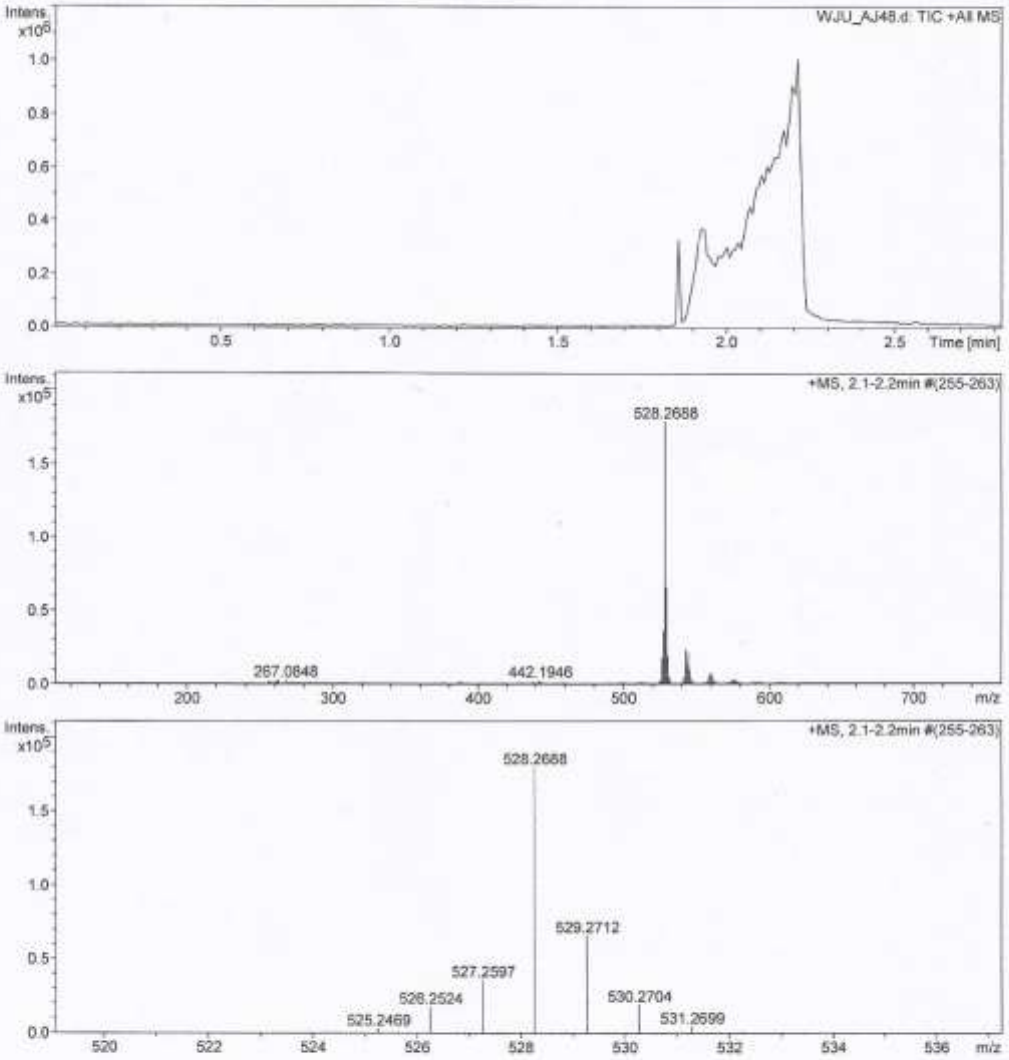
No.	RT	Area	Conc 1	BC
1	17,44	4975	0,074	BB
2	20,81	10864	0,162	BB
3	21,09	6698628	99,663	BB
4	22,32	6808	0,101	BB
		6721275	100,000	

Peak rejection level: 0

Generic Display Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ48.d	Acquisition Date	8/29/2012 8:27:34 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ48	Instrument	micrOTOF-Q II
Comment	Junker APCI-Direkt Kalibration mit Feltsaeureestern		



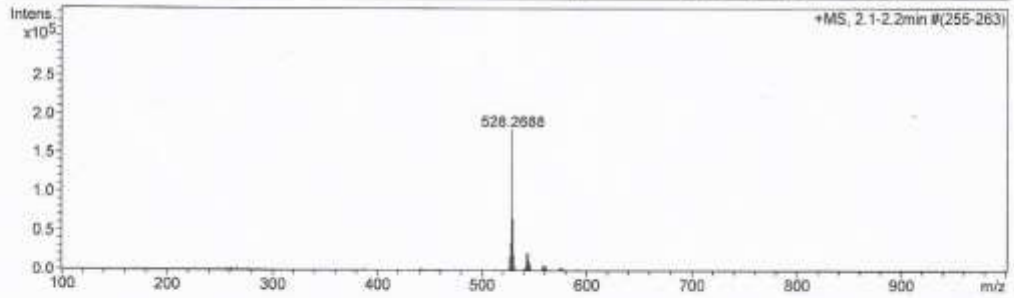
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	D:\Data\IPMC\PharmChemie\Routine\APCI\12_08\WJU_AJ48.d	Acquisition Date	8/29/2012 8:27:34 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ48	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

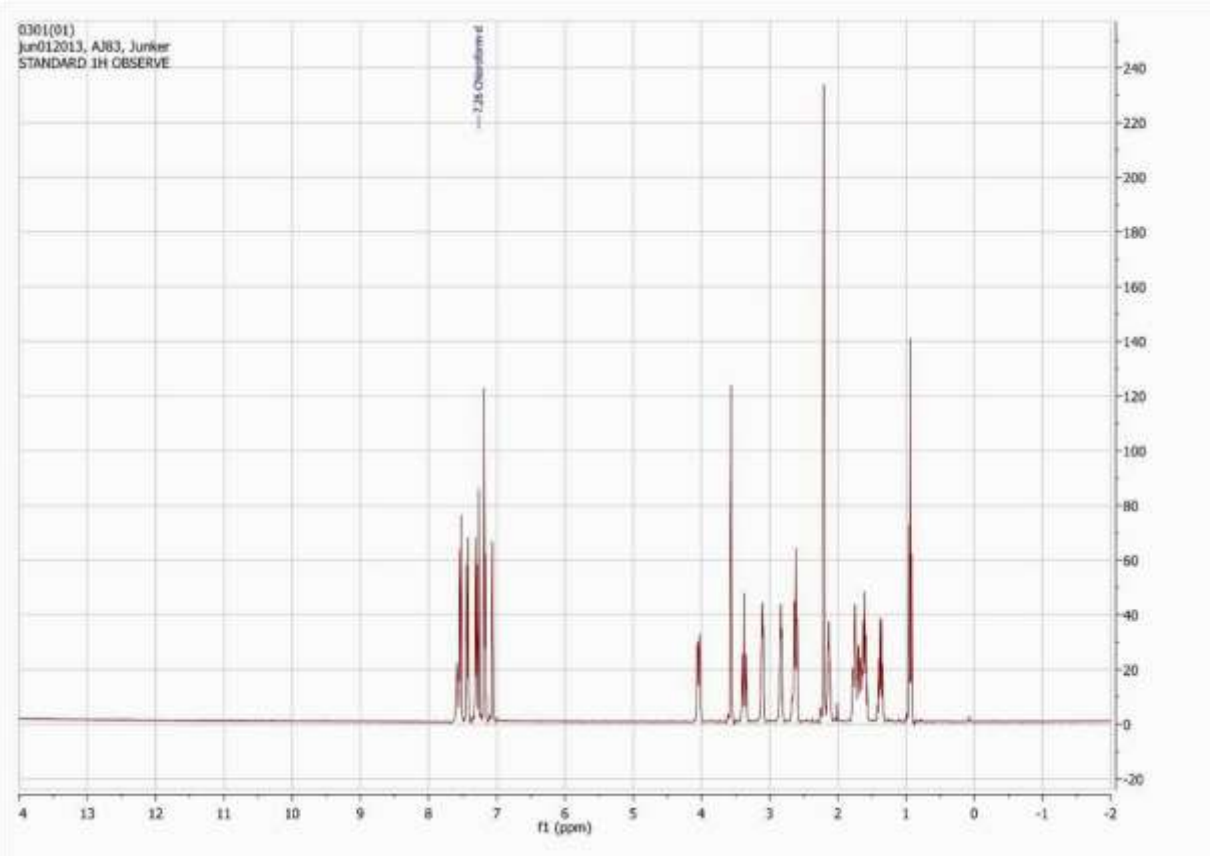
Acquisition Parameter

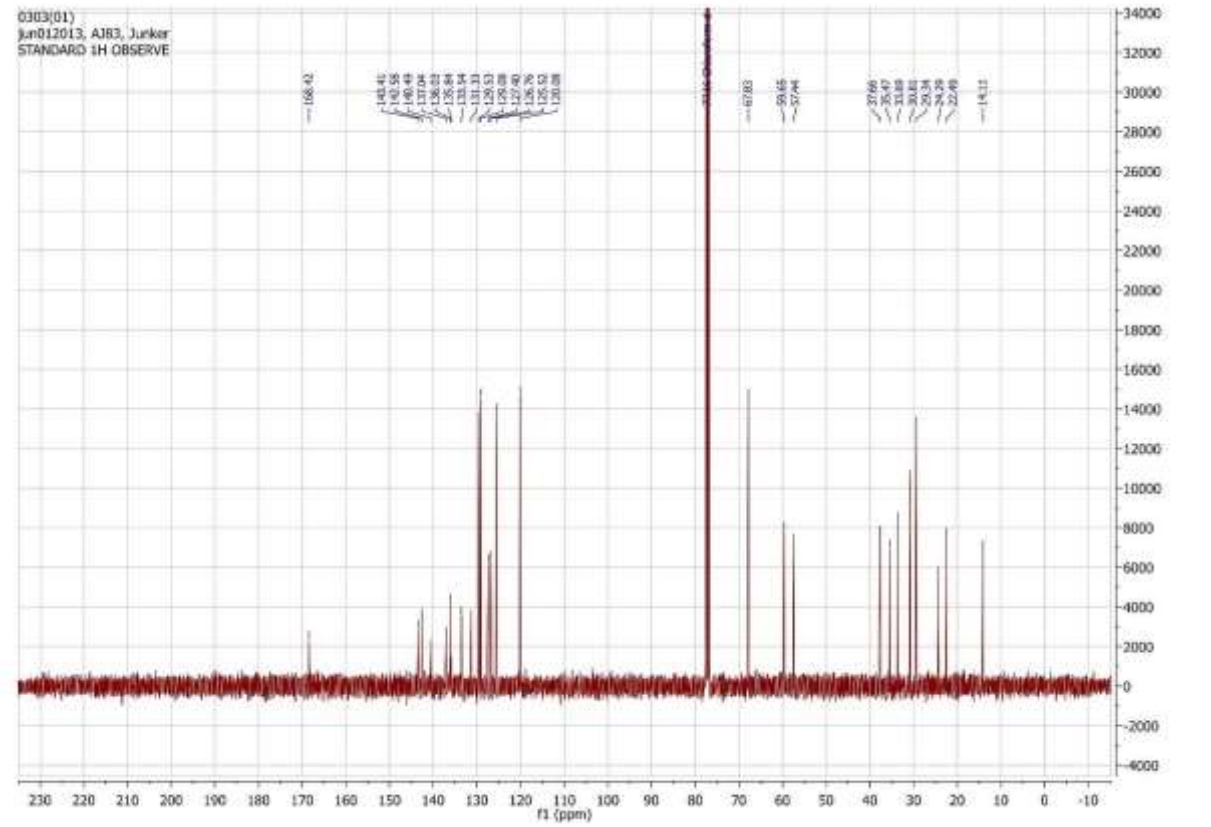
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

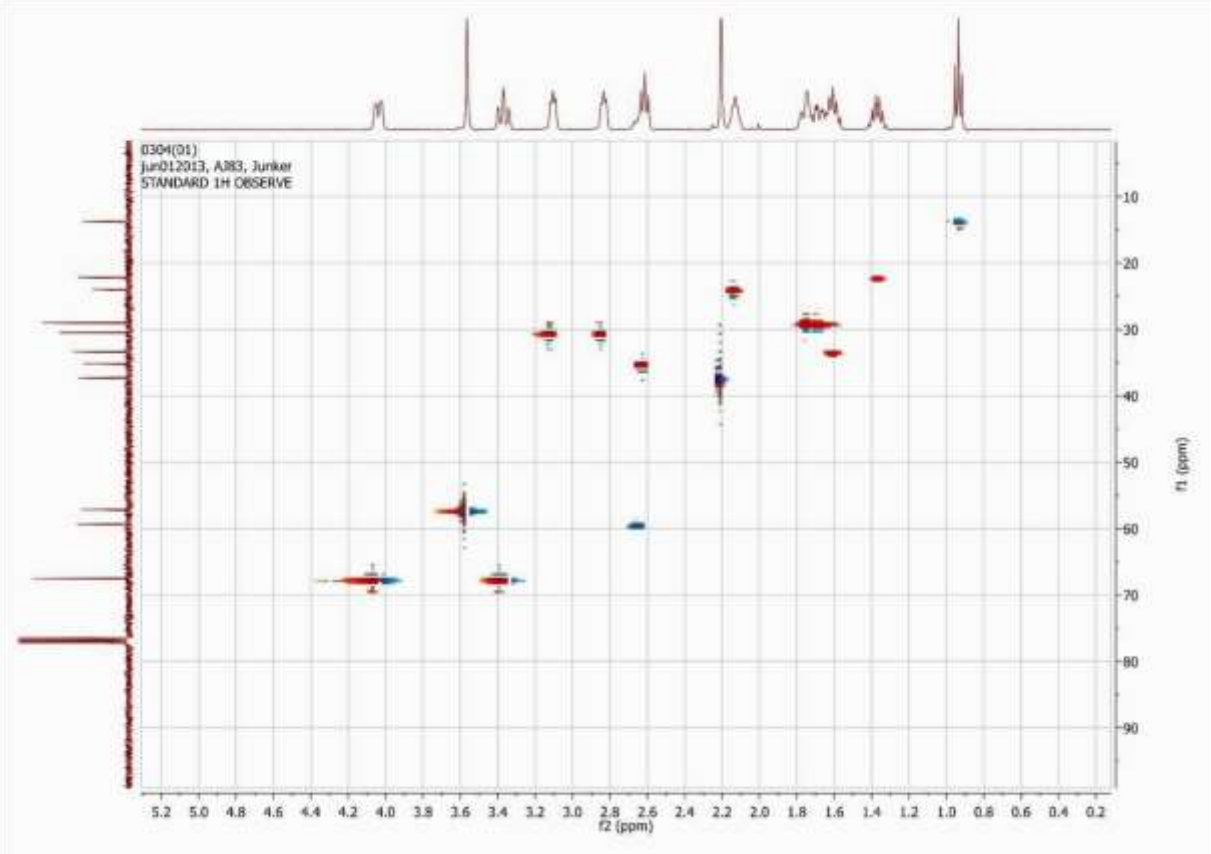


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
528.2688	1	C 32 H 38 N 3 O 2 S	100.00	528.2679	-0.9	-1.7	6.1	15.5	even	ok
	2	C 28 H 34 N 9 S	10.26	528.2652	-3.6	-6.8	9.5	16.5	even	ok
	3	C 27 H 38 N 5 O 4 S	1.51	528.2639	-4.9	-9.3	21.9	11.5	even	ok
	4	C 29 H 42 N 3 O 2 S 2	23.55	528.2713	2.5	4.6	23.6	10.5	even	ok
	5	C 37 H 38 N S	12.57	528.2719	3.1	5.9	24.7	19.5	even	ok

2-(4-Butylphenyl)-N-4-[N-methyl-N-(tetrahydro-2*H*-pyran-4-yl)aminomethyl]phenyl}—7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxamide (1cC)






0304(01)
jun012013, A383, Junker
STANDARD 1H OBSERVE
f2 (ppm)
f1 (ppm)

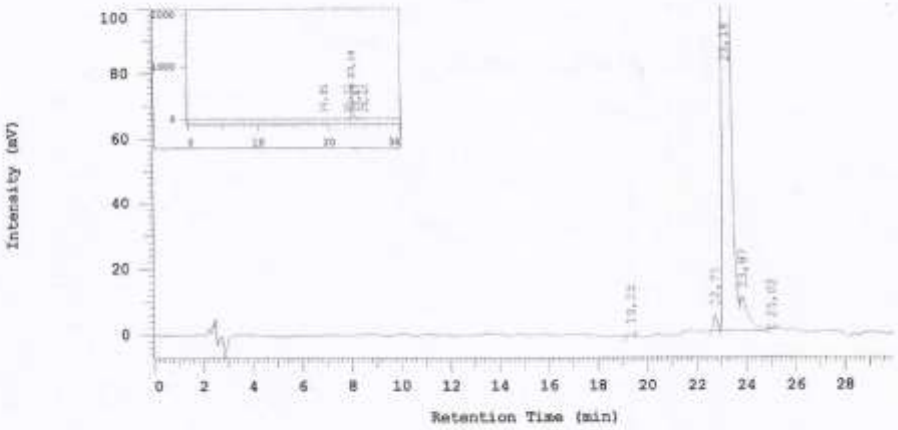
HPLC

Analyzed: 10.01.13 04:03
Reported: 10.01.13 10:20
Processed: 10.01.13 10:20

Data Path: D:\WIN32APP\HSM\Chromni\DATA\5800\
Application: Chromni
Sample Name: AJ 83
Injection from this vial: 1 of 1

Series:5800
Vial Number: 9
Vial Type: UNK
Volume: 5,0 ul

Chrom Type: HPLC Channel : 1



Acquisition Method: Chromni
Blank Subtr Sample Name: ACN
Column Type: 010
Developed by: Jens
Solvent A: Wasser + 0,05%TFA
Solvent B: ACN + 0,05%TFA

No.	RT	Area	Conc 1	BC
1	19,35	9633	0,102	BB
2	22,75	44467	0,469	BB
3	23,18	9200306	97,003	MC
4	23,87	224861	2,371	MC
5	25,02	5251	0,055	BB
		9484518	100,000	

Peak rejection level: 0

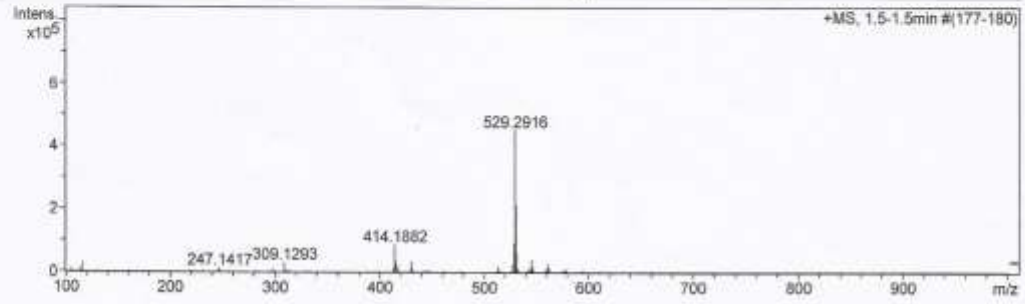
Mass Spectrum SmartFormula Report

Analysis Info

Analysis Name	E:\Meiners\13_01\WJU_AJ83.d	Acquisition Date	1/7/2013 11:47:25 AM
Method	APCI_directprobe_positiv.m	Operator	Meiners
Sample Name	AJ83	Instrument / Ser#	micrOTOF-Q II 10252
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern		

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	0.7 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	3.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

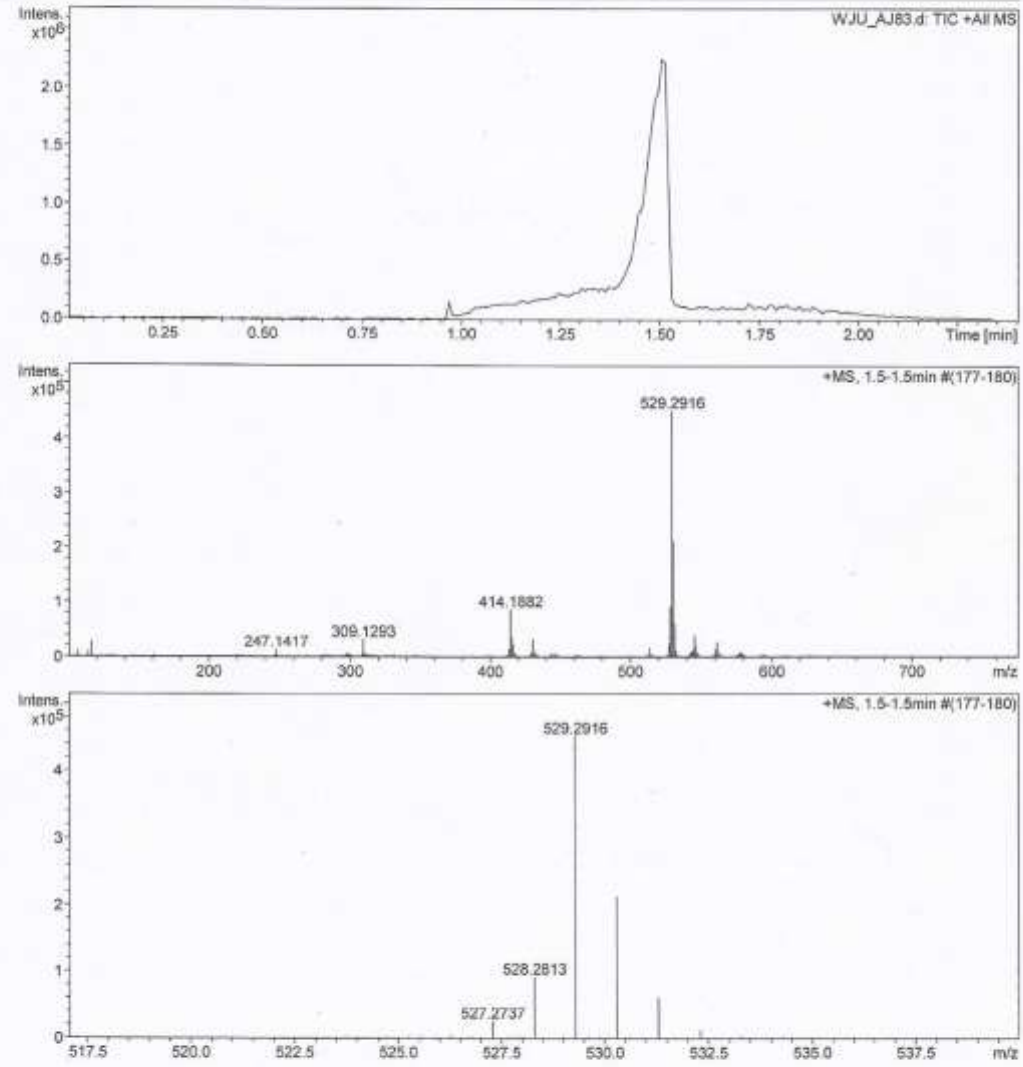


Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
529.2916	1	C 38 H 41 S	100.00	529.2923	0.8	1.4	20.6	18.5	even	ok
	2	C 35 H 45 S 2	3.82	529.2957	4.1	7.8	35.9	13.5	even	ok
	3	C 33 H 41 N 2 O 2 S	7.56	529.2883	-3.3	-6.2	46.8	14.5	even	ok
	4	C 30 H 45 N 2 O 2 S 2	57.31	529.2917	0.1	0.2	53.3	9.5	even	ok

Generic Display Report

Analysis Info

Analysis Name	E:\Meiners\13_01\WJU_AJ83.d	Acquisition Date	1/7/2013 11:47:25 AM
Method	APCI_directprobe_positiv.m		
Sample Name	AJ83	Operator	Meiners
Comment	Junker APCI-Direkt Kalibration mit Fettsaeureestern	Instrument	micrOTOF-Q II



Representative LC-MS spectrum of $PdBr_2$/bipy-catalysed coupling of methyl 7,8-dihydro-6*H*-[7]annuleno[*b*]thiophene-5-carboxylate (**2**) with 4-iodotoluene (**3a**)