Screening method QTOF

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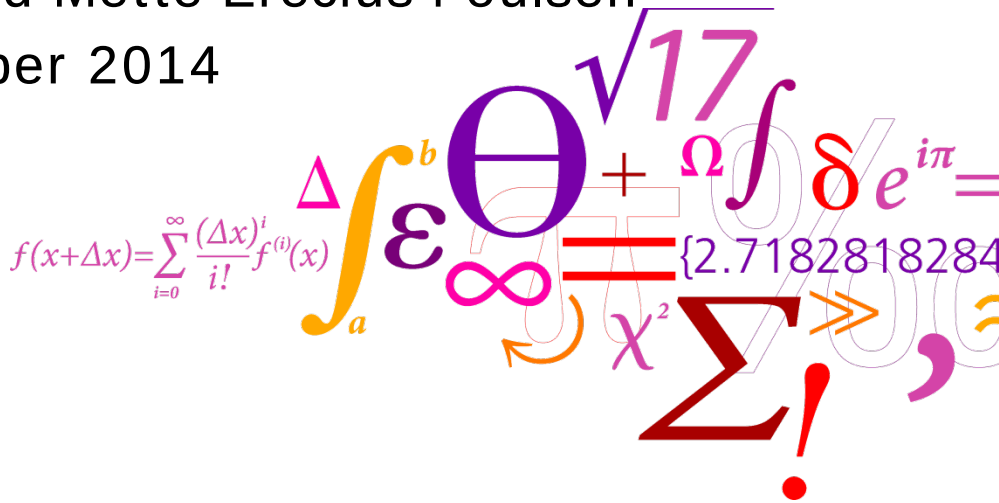

DTU Food
National Food Institute

Disposition

- LC-QTOF screening - MassHunter Qualitative
- LC-QTOF - Validation
- GC-QTOF screening - MassHunter Quantitative
- GC-QTOF - Validation

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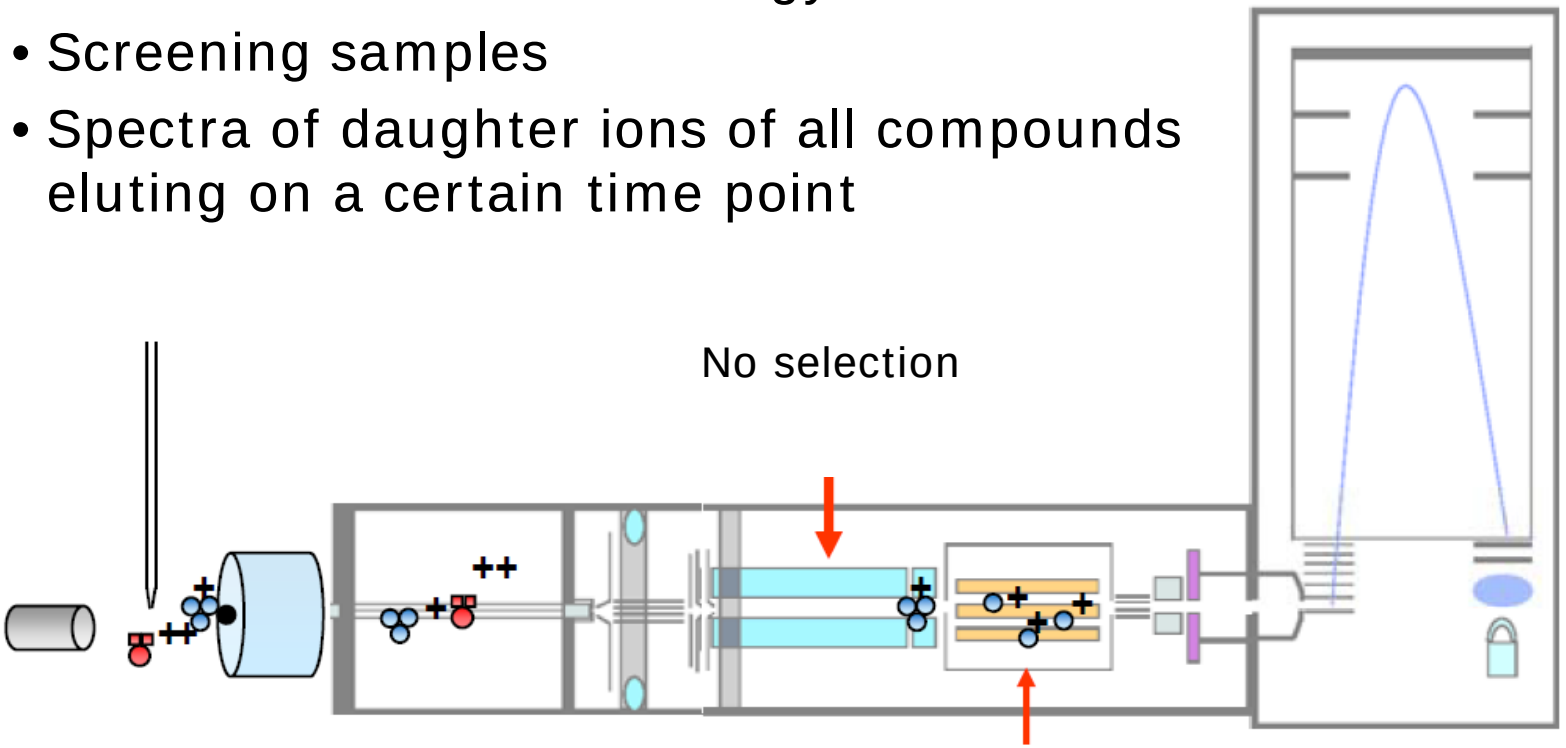
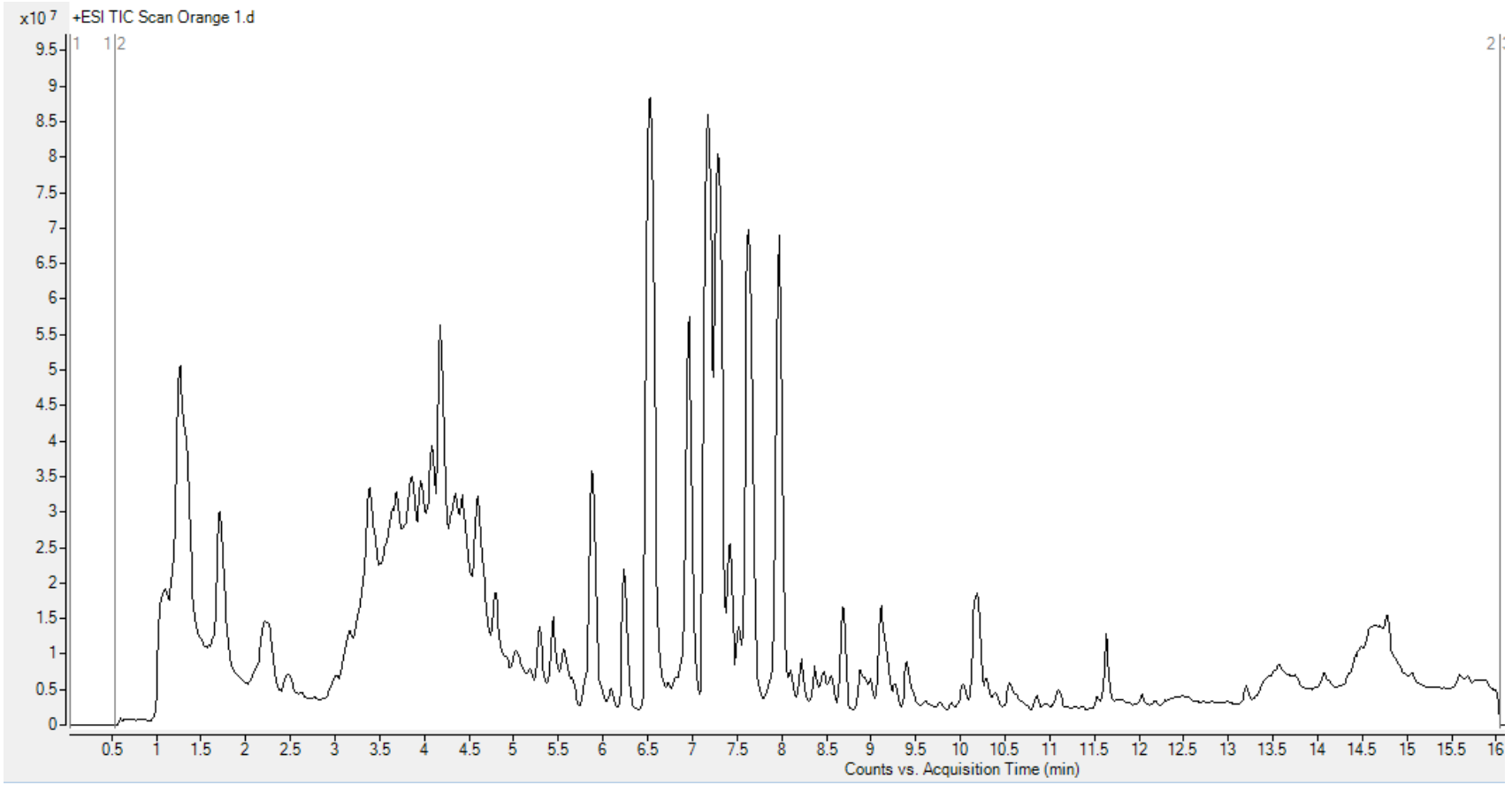
LC-QTOF instruments

QTOF-analysis – All ion MS

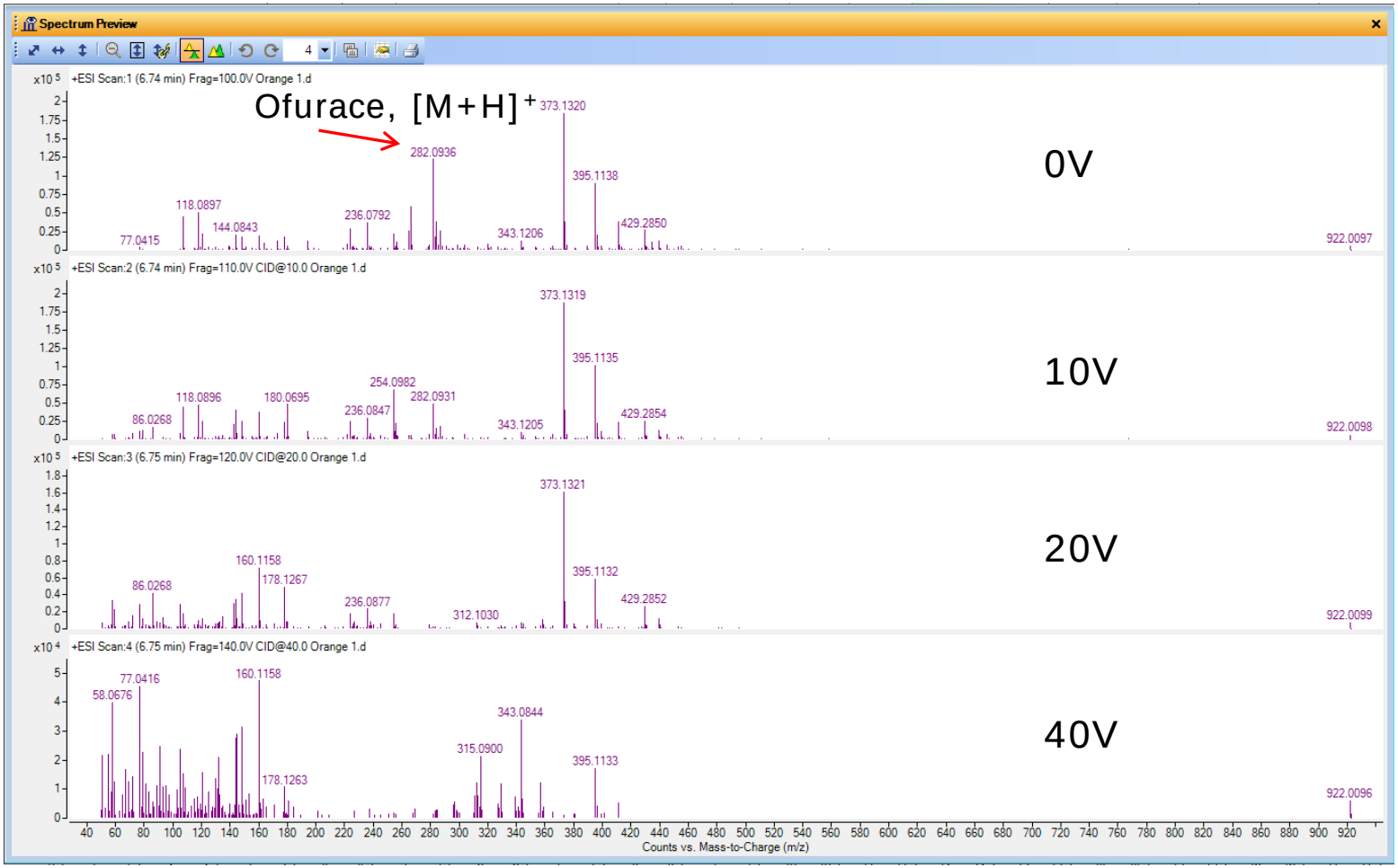
- MS scan with collision energy
- Screening samples
- Spectra of daughter ions of all compounds eluting on a certain time point



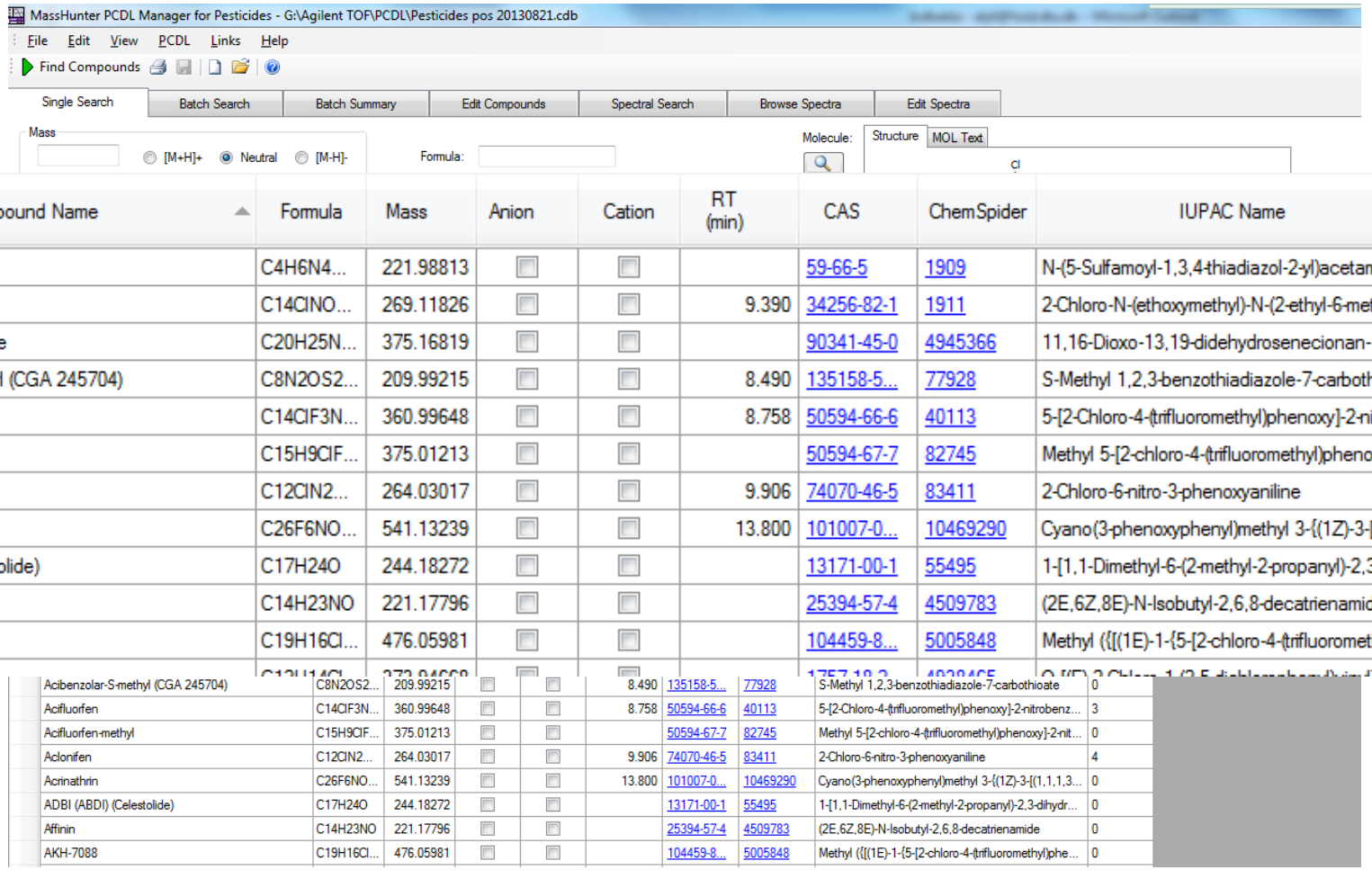
Chromatogram - TIC



Spectra – MSscan incl. collision

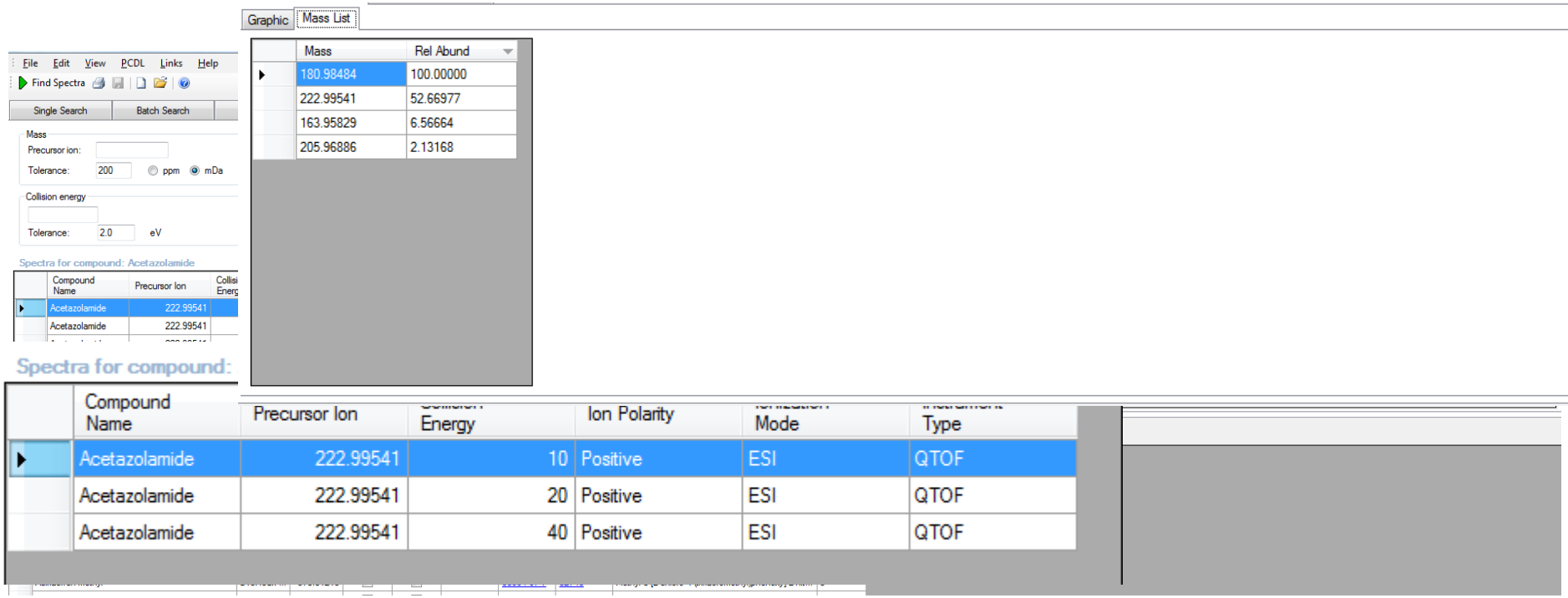


PCDL - library

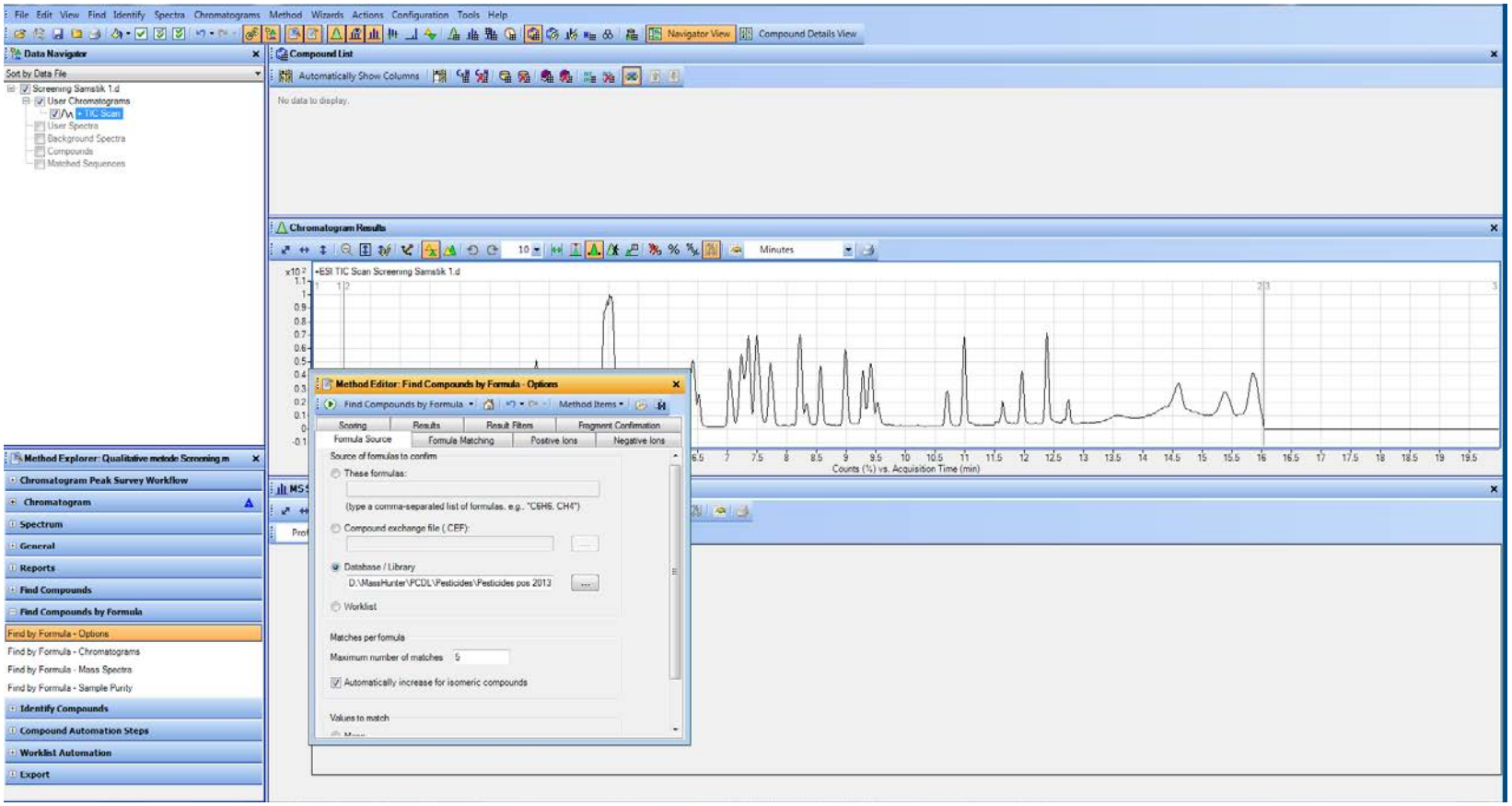


Compound Name	Formula	Mass	Anion	Cation	RT (min)	CAS	ChemSpider	IUPAC Name	Num Spectra
Acetazolamide	C4H6N4...	221.98813				59-66-5	1909	N-(5-Sulfamoyl-1,3,4-thiadiazol-2-yl)acetamide	3
Acetochlor	C14ClNO...	269.11826			9.390	34256-82-1	1911	2-Chloro-N-(ethoxymethyl)-N-(2-ethyl-6-methylphe...	3
Acetyl-Seneciphylline	C20H25N...	375.16819				90341-45-0	4945366	11,16-Dioxo-13,19-didehydrosenecionan-12-yl ac...	0
Acibenzolar-S-methyl (CGA 245704)	C8N2OS2...	209.99215			8.490	135158-5...	77928	S-Methyl 1,2,3-benzothiadiazole-7-carbothioate	0
Acifluorfen	C14ClF3N...	360.99648			8.758	50594-66-6	40113	5-[2-Chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenz...	3
Acifluorfen-methyl	C15H9ClF...	375.01213				50594-67-7	82745	Methyl 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nit...	0
Aclonifen	C12ClN2...	264.03017			9.906	74070-46-5	83411	2-Chloro-6-nitro-3-phenoxyaniline	4
Acrinathrin	C26F6NO...	541.13239			13.800	101007-0...	10469290	Cyano(3-phenoxyphenyl)methyl 3-{(1Z)-3-[(1,1,1,3...	0
ADBI (ABDI) (Celestolide)	C17H24O	244.18272				13171-00-1	55495	1-[1,1-Dimethyl-6-(2-methyl-2-propanyl)-2,3-dihydr...	0
Affinin	C14H23NO	221.17796				25394-57-4	4509783	(2E,6Z,8E)-N-Isobutyl-2,6,8-decatrienamide	0
AKH-7088	C19H16Cl...	476.05981				104459-8...	5005848	Methyl ({[(1E)-1-{5-[2-chloro-4-(trifluoromethyl)phe...	0

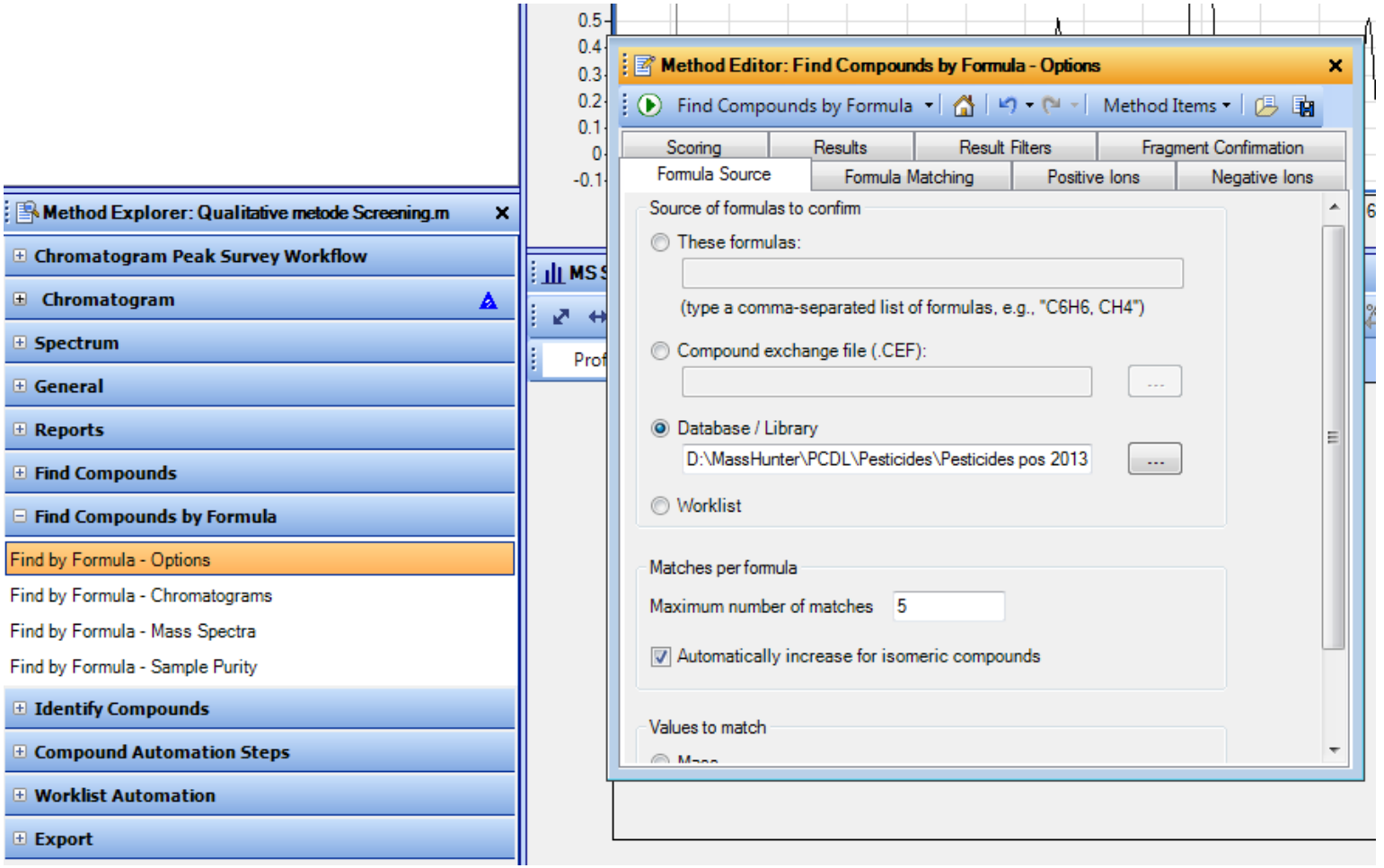
PCDL – library - spectra



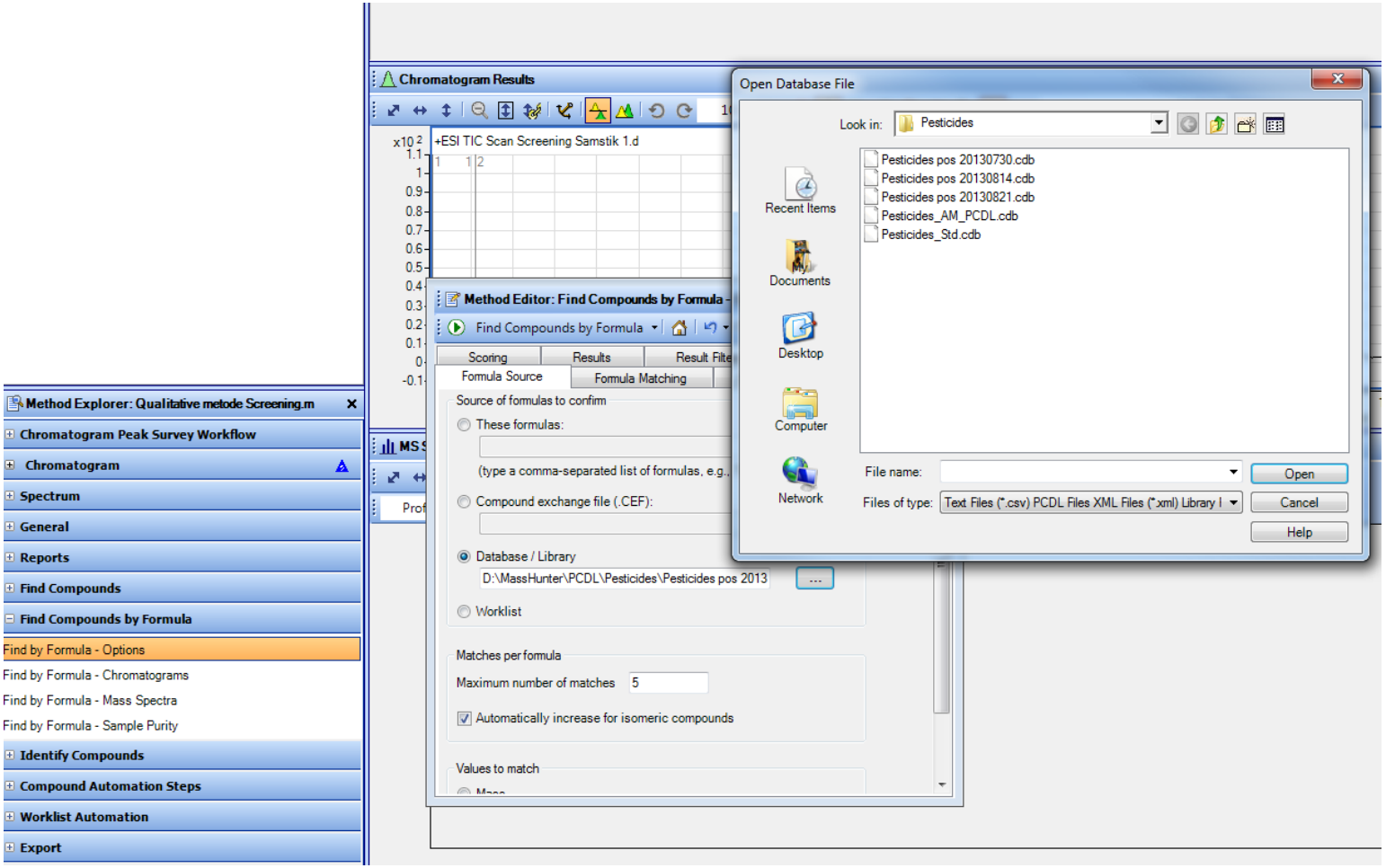
MassHunter Qualitative - Library Search



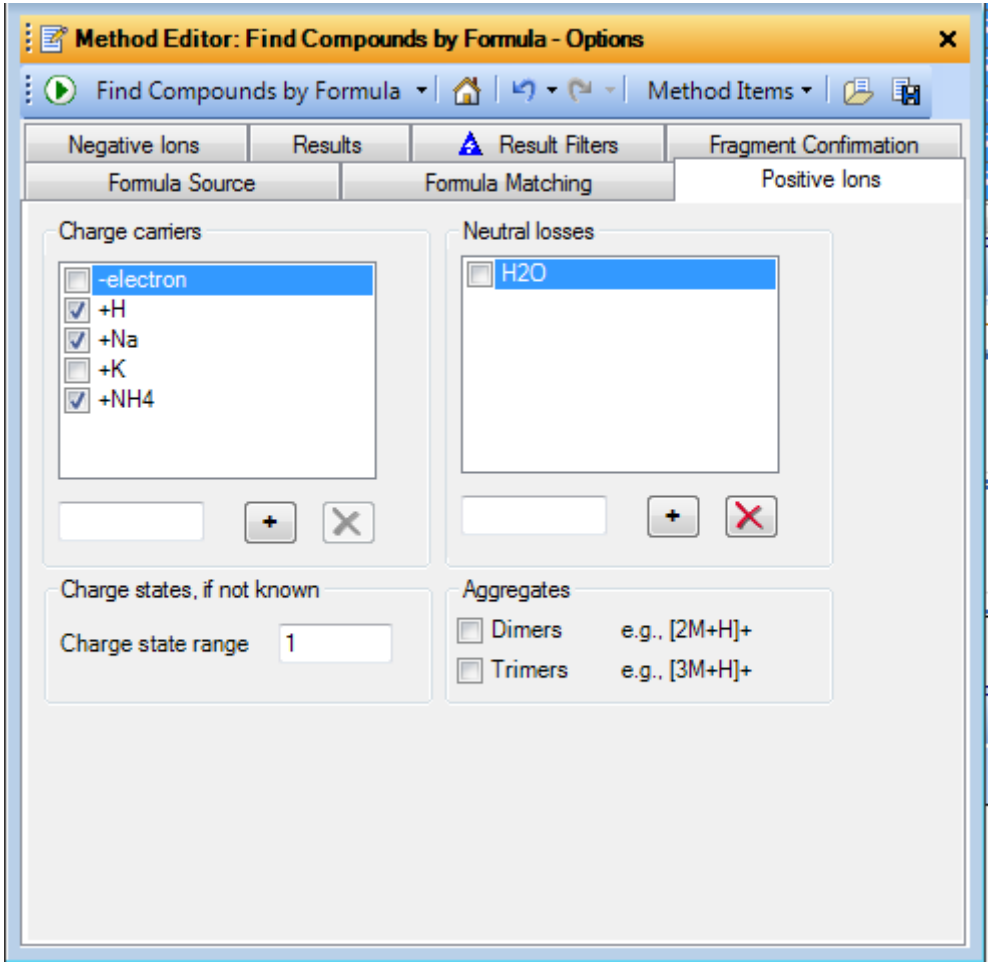
Library Search



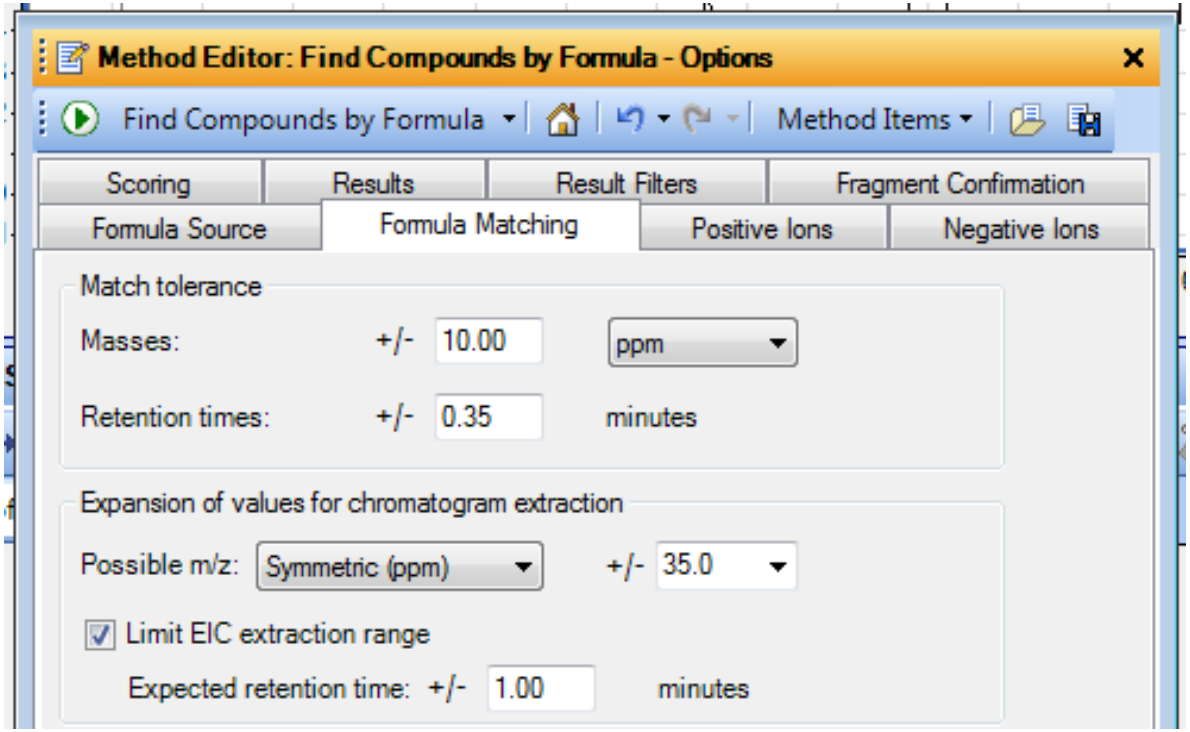
Library Search

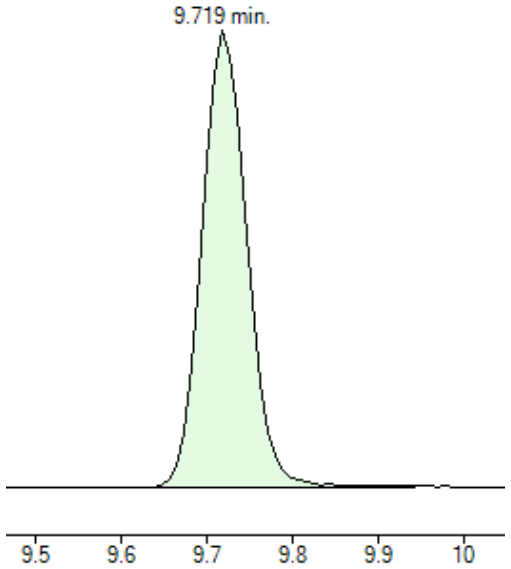


Library Search - adducts

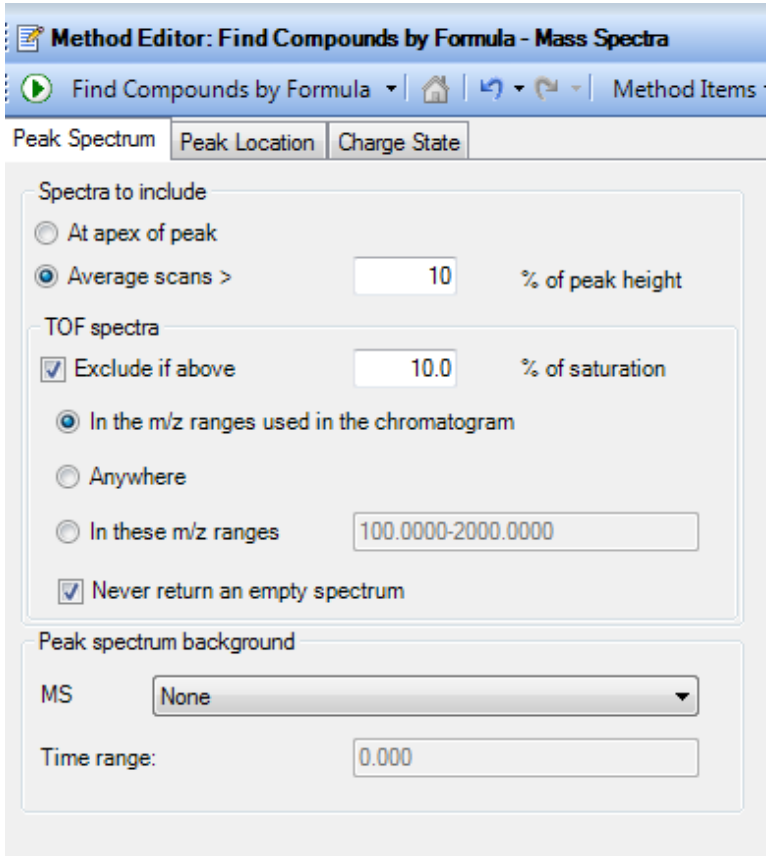


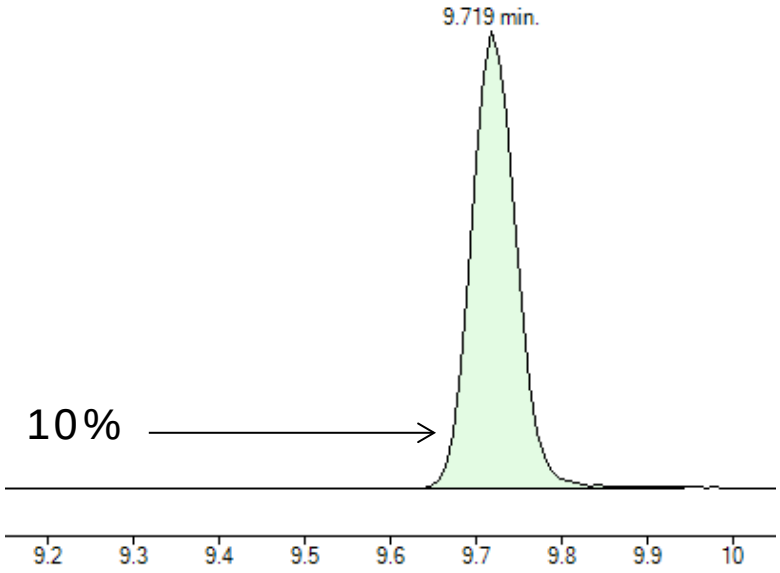
Screening criterea – Match tolerance



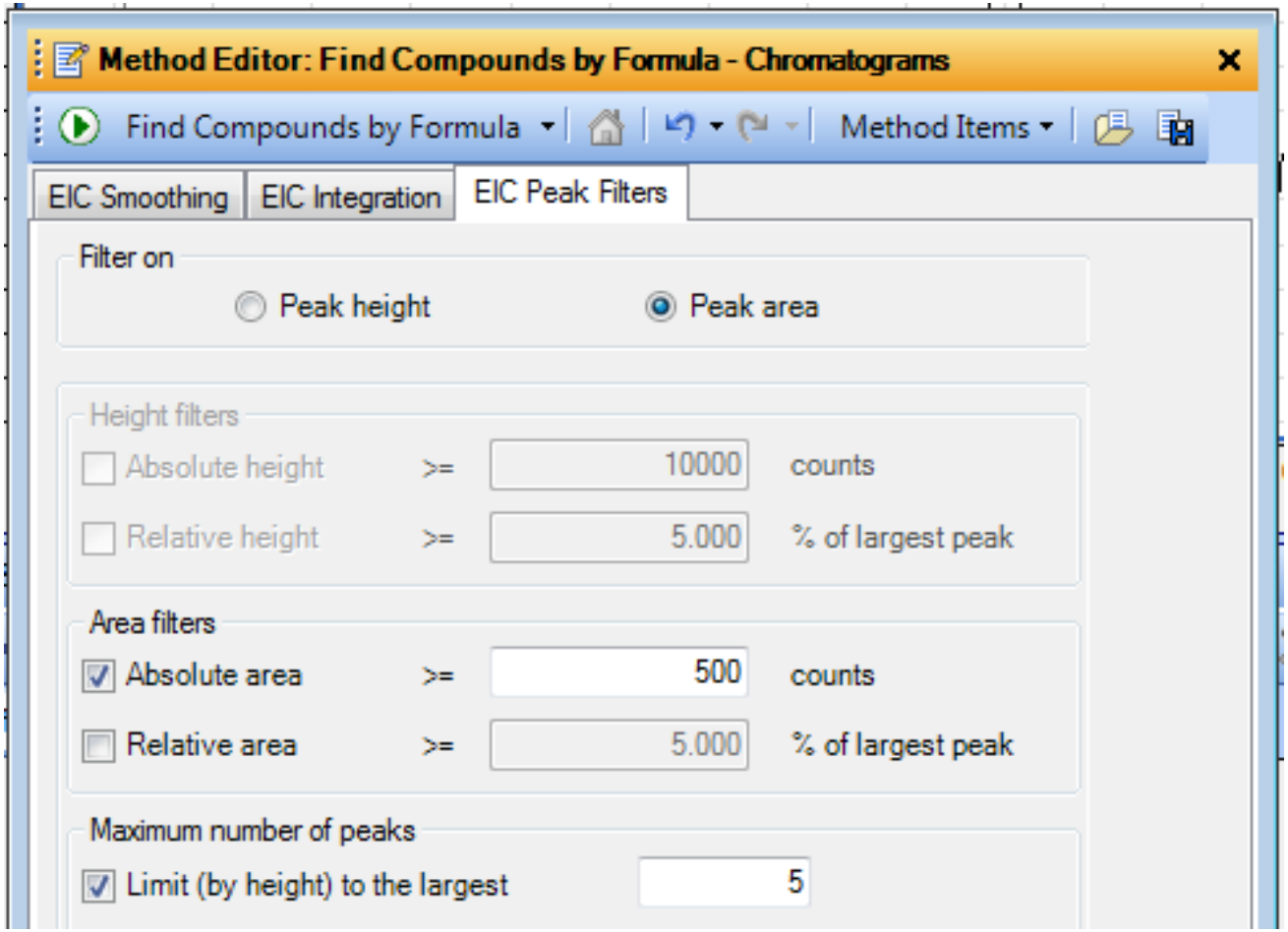


Screening criterea– Average scans

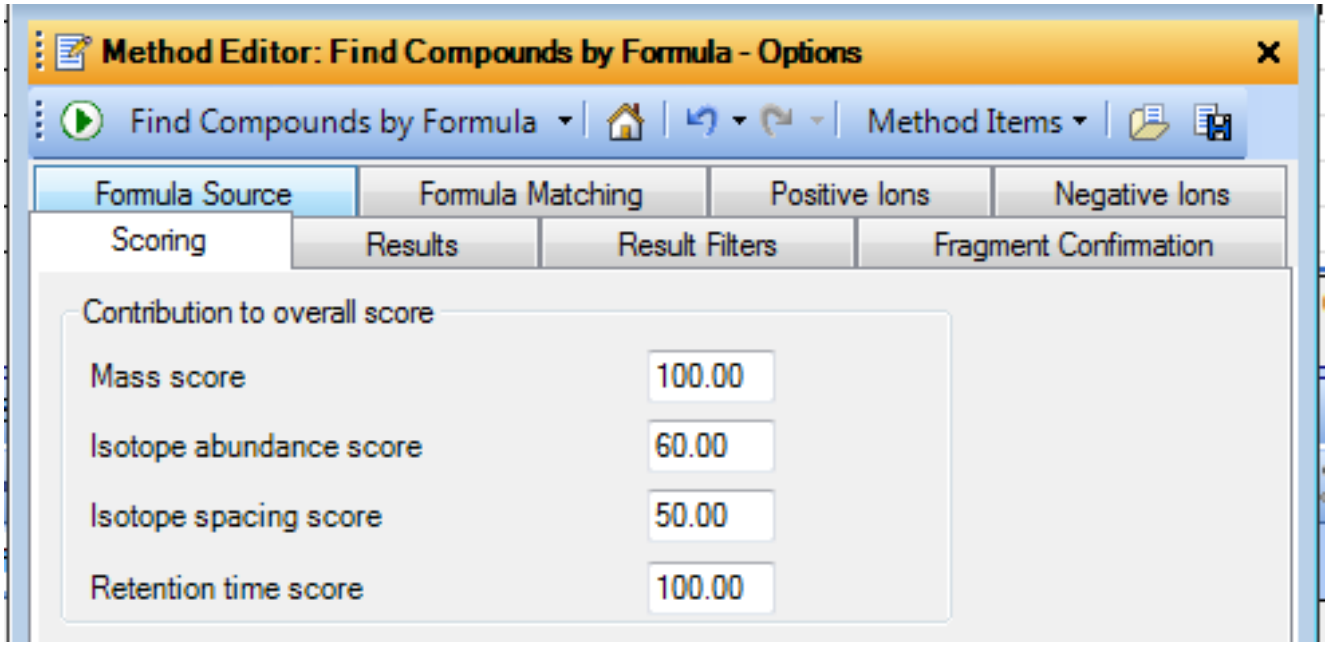




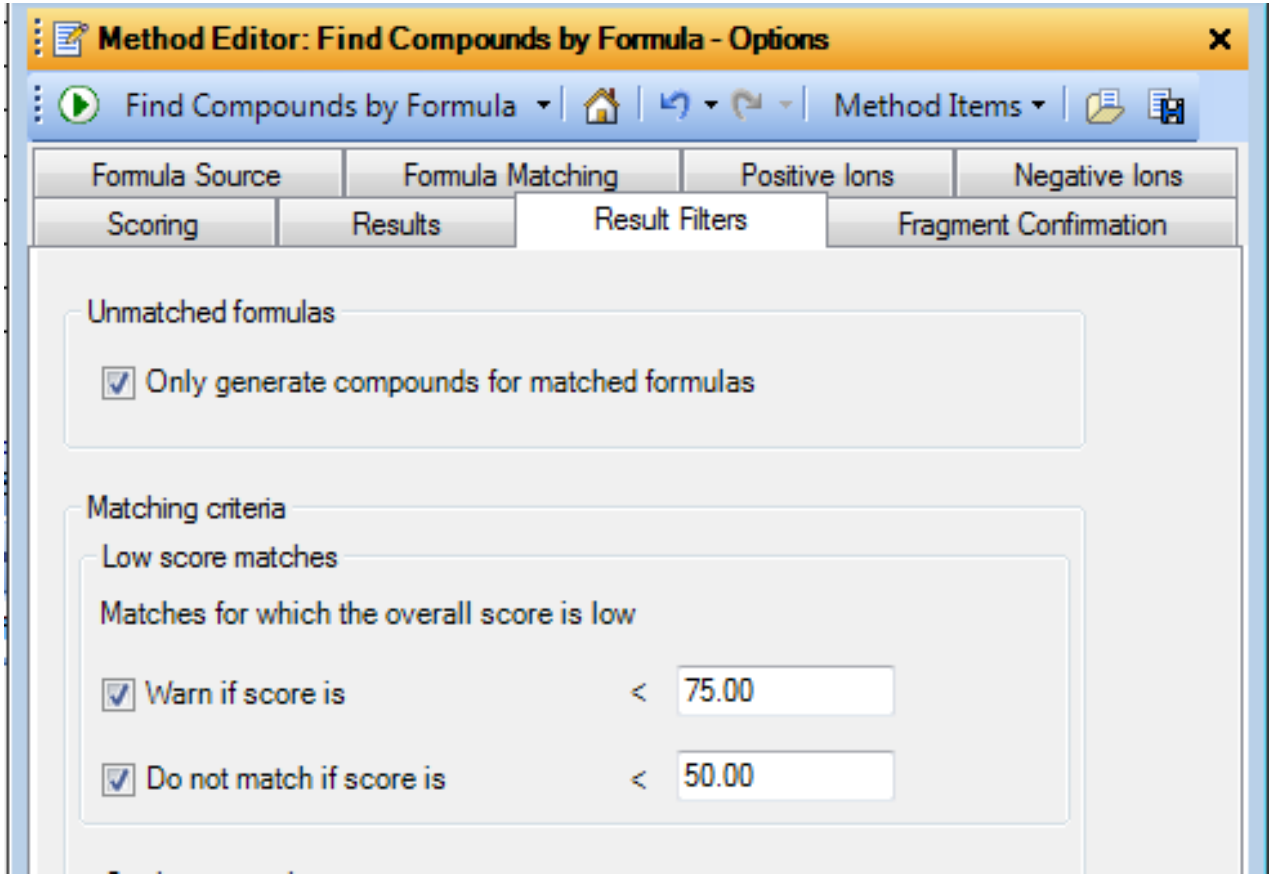
Screening criterea– Match tolerance



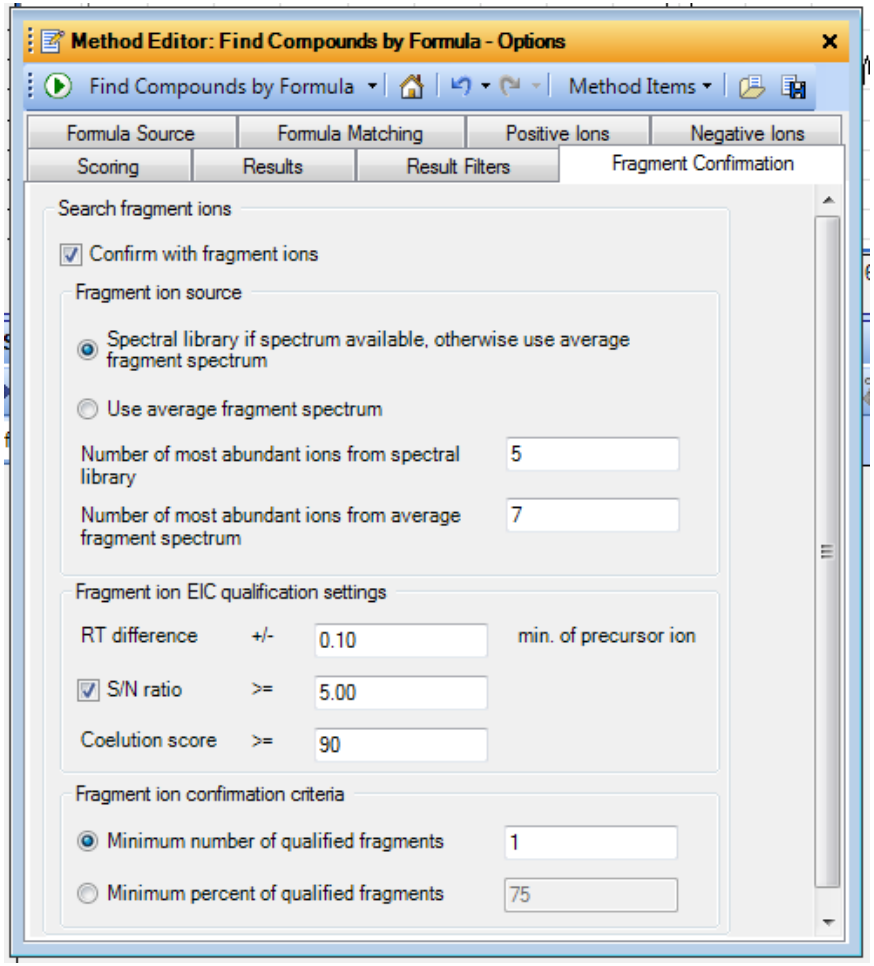
Library Search - scoring



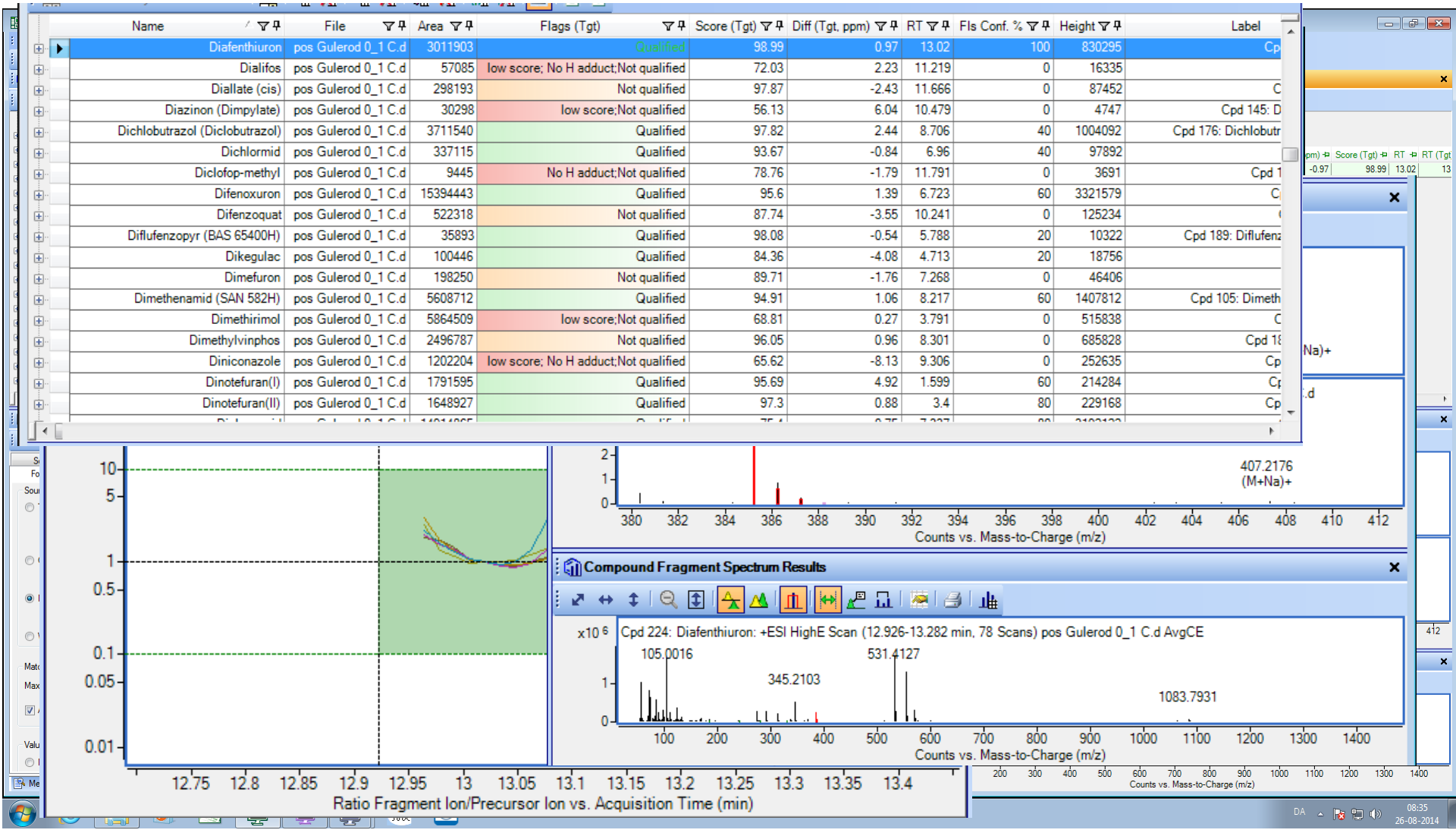
Library Search - scoring



Library Search – fragment ions

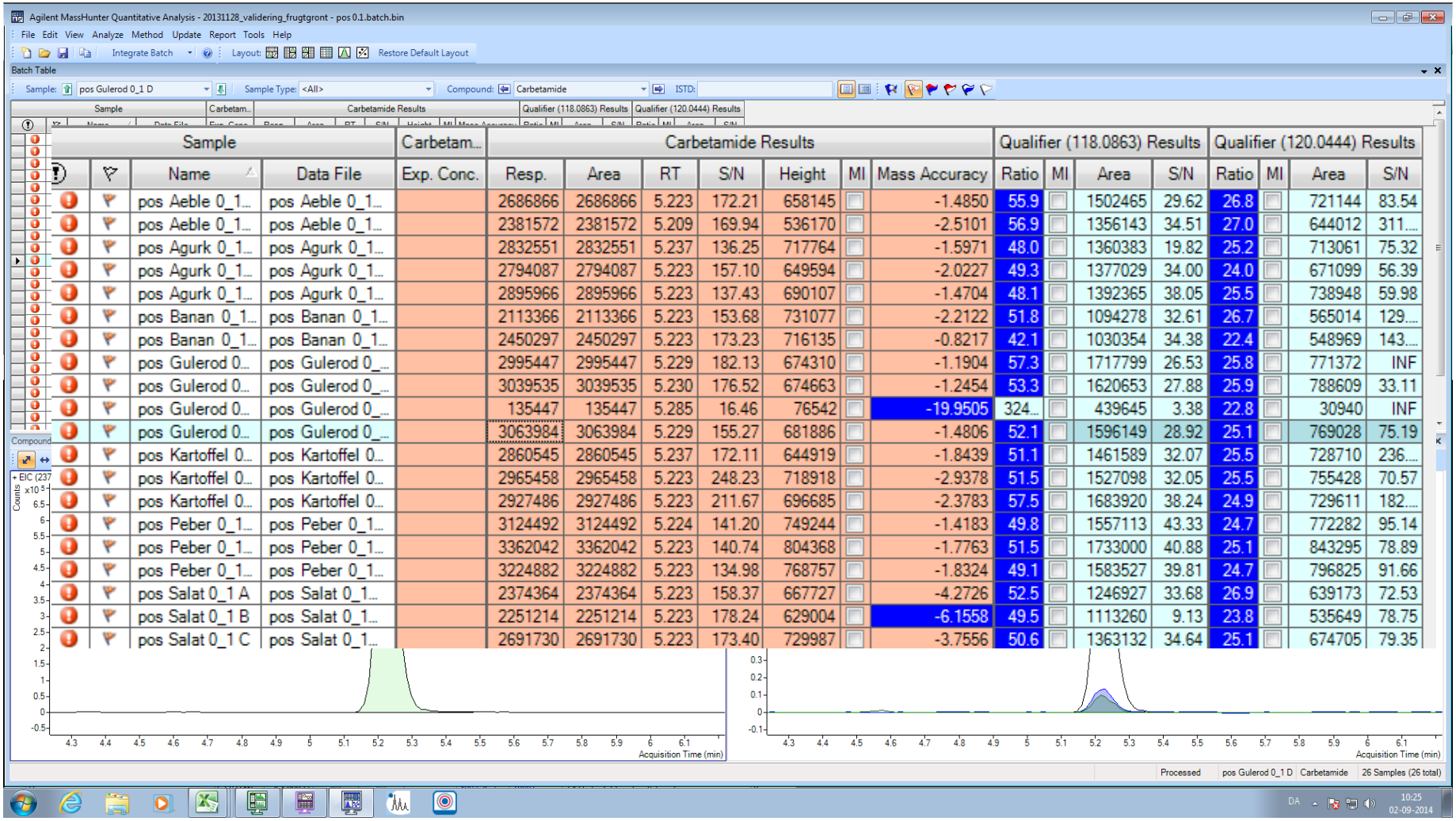


Compounds Detail View



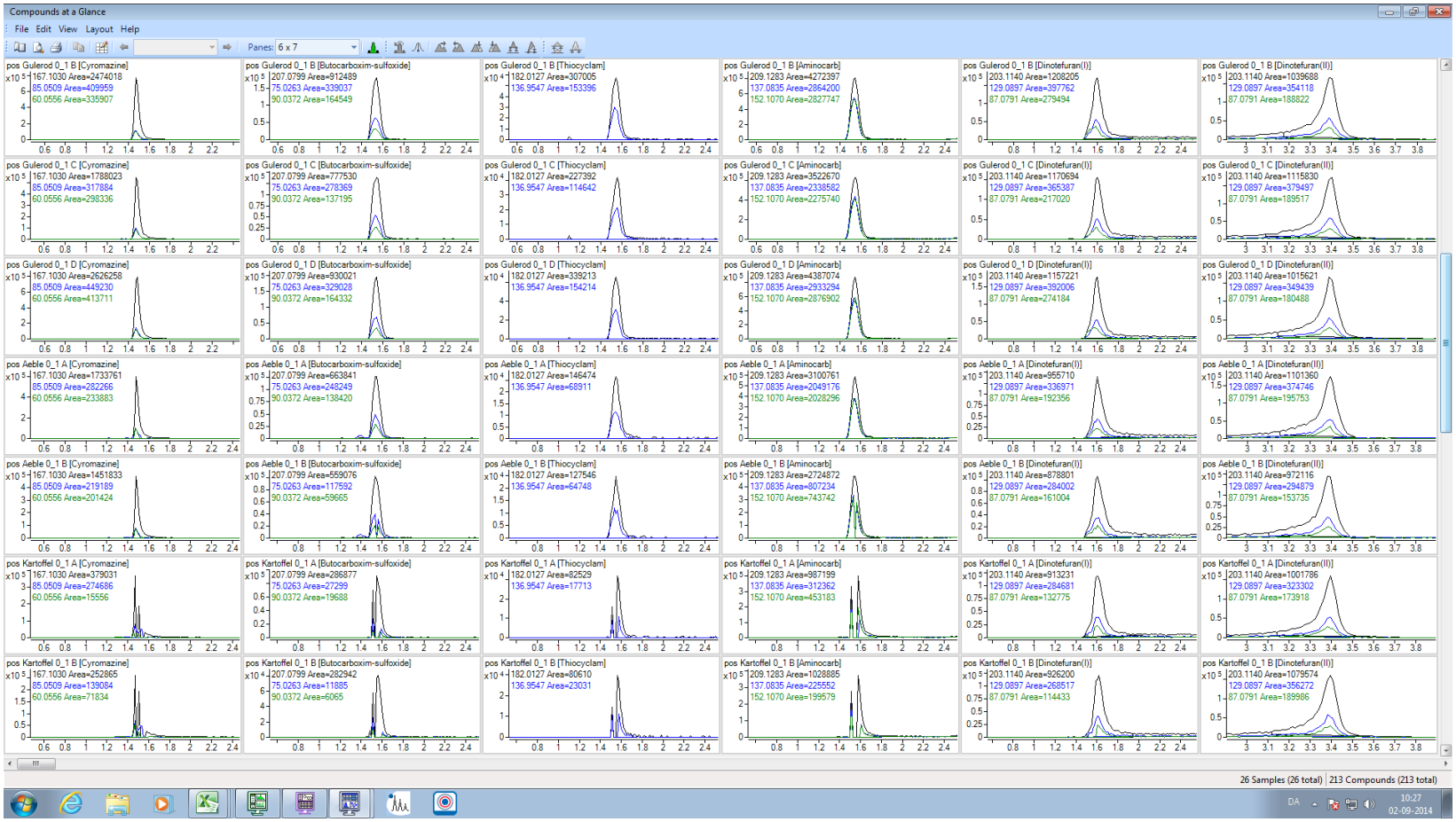
Name	File	Area	Flags (Tgt)	Score (Tgt)	Diff (Tgt, ppm)	RT	Fls Conf. %	Height	Label
Diafenthiuron	pos Gulerod 0_1 C.d	3011903	Qualified	98.99	0.97	13.02	100	830295	Cp
Dialifos	pos Gulerod 0_1 C.d	57085	low score; No H adduct;Not qualified	72.03	2.23	11.219	0	16335	
Diallate (cis)	pos Gulerod 0_1 C.d	298193	Not qualified	97.87	-2.43	11.666	0	87452	C
Diazinon (Dimpylate)	pos Gulerod 0_1 C.d	30298	low score;Not qualified	56.13	6.04	10.479	0	4747	Cpd 145: D
Dichlobutrazol (Diclobutrazol)	pos Gulerod 0_1 C.d	3711540	Qualified	97.82	2.44	8.706	40	1004092	Cpd 176: Dichlobutr
Dichlormid	pos Gulerod 0_1 C.d	337115	Qualified	93.67	-0.84	6.96	40	97892	
Diclofop-methyl	pos Gulerod 0_1 C.d	9445	No H adduct;Not qualified	78.76	-1.79	11.791	0	3691	Cpd 1
Difenoxuron	pos Gulerod 0_1 C.d	15394443	Qualified	95.6	1.39	6.723	60	3321579	C
Difenzoquat	pos Gulerod 0_1 C.d	522318	Not qualified	87.74	-3.55	10.241	0	125234	
Diflufenzopyr (BAS 65400H)	pos Gulerod 0_1 C.d	35893	Qualified	98.08	-0.54	5.788	20	10322	Cpd 189: Diflufenz
Dikegulac	pos Gulerod 0_1 C.d	100446	Qualified	84.36	-4.08	4.713	20	18756	
Dimefuron	pos Gulerod 0_1 C.d	198250	Not qualified	89.71	-1.76	7.268	0	46406	
Dimethenamid (SAN 582H)	pos Gulerod 0_1 C.d	5608712	Qualified	94.91	1.06	8.217	60	1407812	Cpd 105: Dimeth
Dimethirimol	pos Gulerod 0_1 C.d	5864509	low score;Not qualified	68.81	0.27	3.791	0	515838	C
Dimethylvinphos	pos Gulerod 0_1 C.d	2496787	Not qualified	96.05	0.96	8.301	0	685828	Cpd 18
Diniconazole	pos Gulerod 0_1 C.d	1202204	low score; No H adduct;Not qualified	65.62	-8.13	9.306	0	252635	Cp
Dinotefuran(I)	pos Gulerod 0_1 C.d	1791595	Qualified	95.69	4.92	1.599	60	214284	Cp
Dinotefuran(II)	pos Gulerod 0_1 C.d	1648927	Qualified	97.3	0.88	3.4	80	229168	Cp

MassHunter - Quantitative

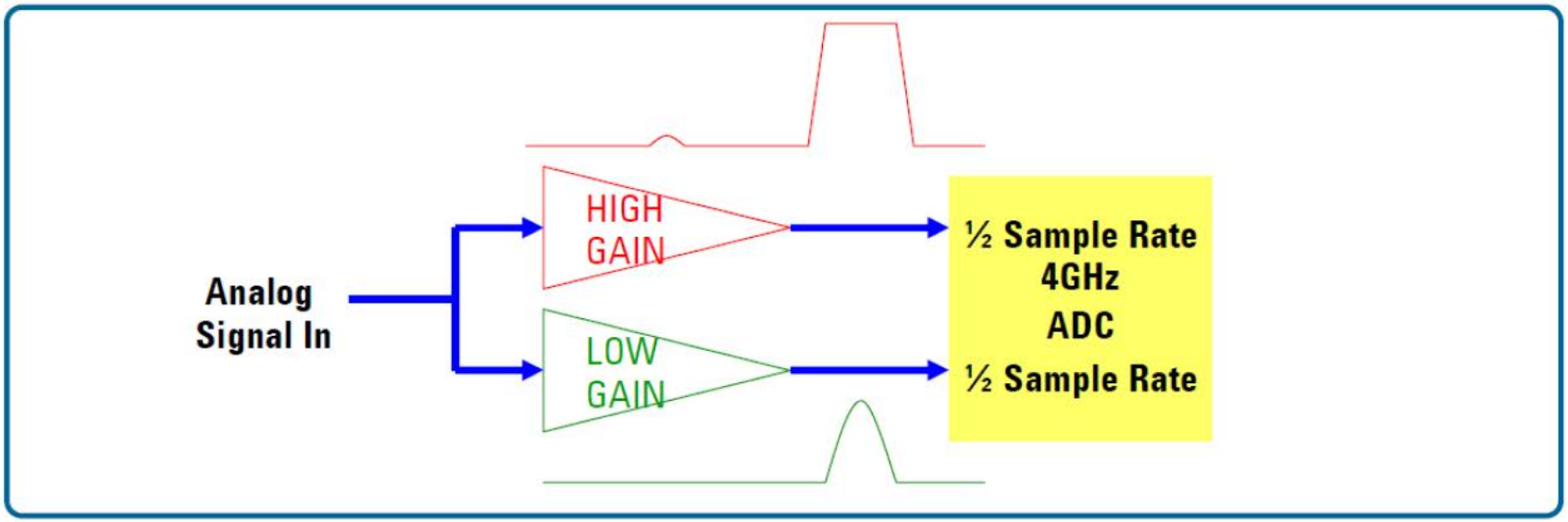


Sample		Carbetam...	Carbetamide Results							Qualifier (118.0863) Results				Qualifier (120.0444) Results			
Name	Data File	Exp. Conc.	Resp.	Area	RT	S/N	Height	MI	Mass Accuracy	Ratio	MI	Area	S/N	Ratio	MI	Area	S/N
pos Aeble 0_1...	pos Aeble 0_1...		2686866	2686866	5.223	172.21	658145		-1.4850	55.9		1502465	29.62	26.8		721144	83.54
pos Aeble 0_1...	pos Aeble 0_1...		2381572	2381572	5.209	169.94	536170		-2.5101	56.9		1356143	34.51	27.0		644012	311....
pos Agurk 0_1...	pos Agurk 0_1...		2832551	2832551	5.237	136.25	717764		-1.5971	48.0		1360383	19.82	25.2		713061	75.32
pos Agurk 0_1...	pos Agurk 0_1...		2794087	2794087	5.223	157.10	649594		-2.0227	49.3		1377029	34.00	24.0		671099	56.39
pos Agurk 0_1...	pos Agurk 0_1...		2895966	2895966	5.223	137.43	690107		-1.4704	48.1		1392365	38.05	25.5		738948	59.98
pos Banan 0_1...	pos Banan 0_1...		2113366	2113366	5.223	153.68	731077		-2.2122	51.8		1094278	32.61	26.7		565014	129....
pos Banan 0_1...	pos Banan 0_1...		2450297	2450297	5.223	173.23	716135		-0.8217	42.1		1030354	34.38	22.4		548969	143....
pos Gulerod 0...	pos Gulerod 0_...		2995447	2995447	5.229	182.13	674310		-1.1904	57.3		1717799	26.53	25.8		771372	INF
pos Gulerod 0...	pos Gulerod 0_...		3039535	3039535	5.230	176.52	674663		-1.2454	53.3		1620653	27.88	25.9		788609	33.11
pos Gulerod 0...	pos Gulerod 0_...		135447	135447	5.285	16.46	76542		-19.9505	324...		439645	3.38	22.8		30940	INF
pos Gulerod 0...	pos Gulerod 0_...		3063984	3063984	5.229	155.27	681886		-1.4806	52.1		1596149	28.92	25.1		769028	75.19
pos Kartoffel 0...	pos Kartoffel 0...		2860545	2860545	5.237	172.11	644919		-1.8439	51.1		1461589	32.07	25.5		728710	236....
pos Kartoffel 0...	pos Kartoffel 0...		2965458	2965458	5.223	248.23	718918		-2.9378	51.5		1527098	32.05	25.5		755428	70.57
pos Kartoffel 0...	pos Kartoffel 0...		2927486	2927486	5.223	211.67	696685		-2.3783	57.5		1683920	38.24	24.9		729611	182....
pos Peber 0_1...	pos Peber 0_1...		3124492	3124492	5.224	141.20	749244		-1.4183	49.8		1557113	43.33	24.7		772282	95.14
pos Peber 0_1...	pos Peber 0_1...		3362042	3362042	5.223	140.74	804368		-1.7763	51.5		1733000	40.88	25.1		843295	78.89
pos Peber 0_1...	pos Peber 0_1...		3224882	3224882	5.223	134.98	768757		-1.8324	49.1		1583527	39.81	24.7		796825	91.66
pos Salat 0_1 A	pos Salat 0_1...		2374364	2374364	5.223	158.37	667727		-4.2726	52.5		1246927	33.68	26.9		639173	72.53
pos Salat 0_1 B	pos Salat 0_1...		2251214	2251214	5.223	178.24	629004		-6.1558	49.5		1113260	9.13	23.8		535649	78.75
pos Salat 0_1 C	pos Salat 0_1...		2691730	2691730	5.223	173.40	729987		-3.7556	50.6		1363132	34.64	25.1		674705	79.35

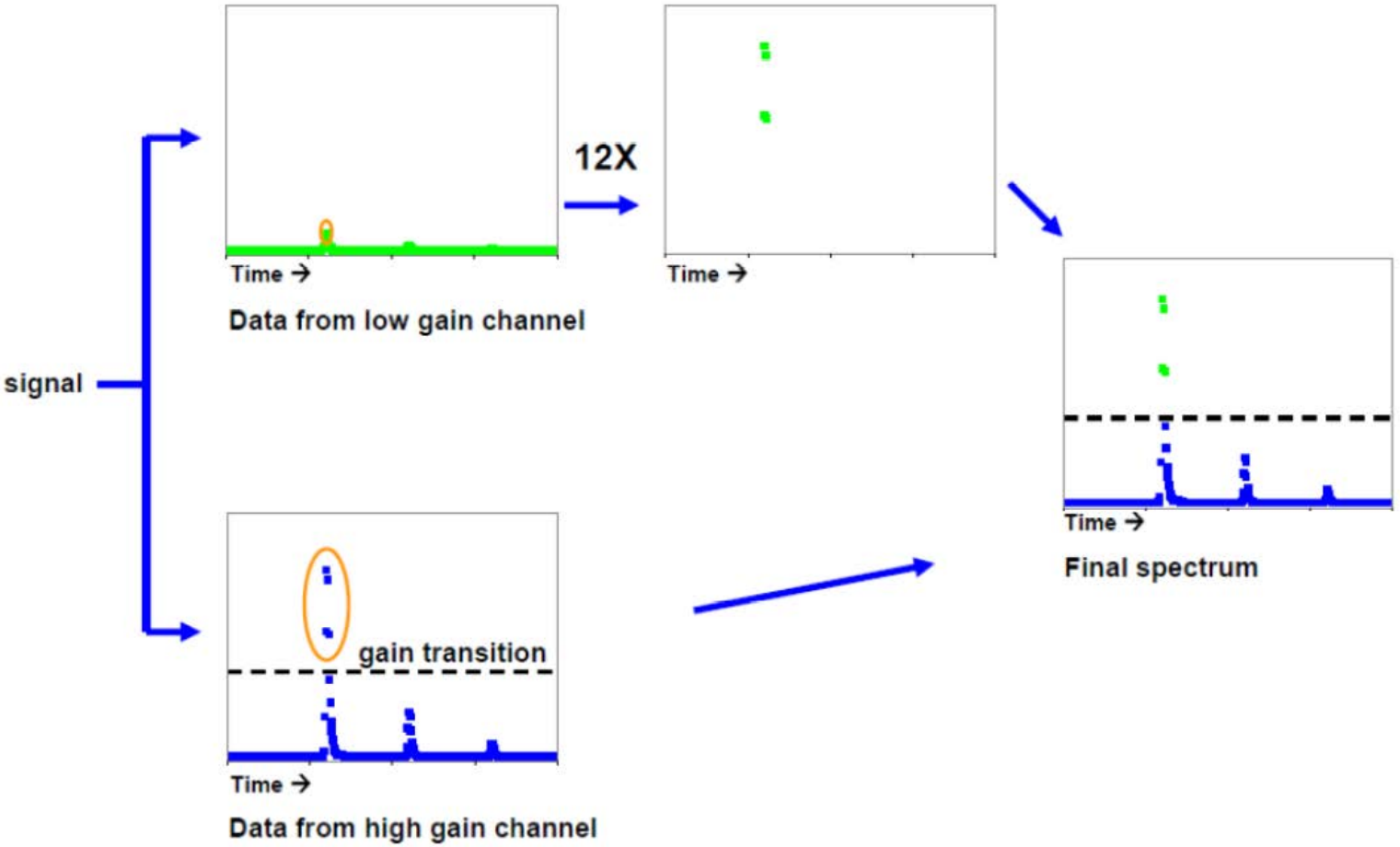
Compound at a glance



2GHz Extended Dynamic Range



2GHz Extended Dynamic Range



Disposition

- LC-QTOF screening - MassHunter Qualitative
- **LC-QTOF - Validation**
- GC-QTOF screening - MassHunter Quantitative
- GC-QTOF - Validation

Validation

- SANCO/12571/2013:

G9 The validation of a screening method based on a SDL can be focused on detectability. For each commodity group (see Annex 1), a basic validation should involve analysis of at least 20 samples spiked at the estimated SDL. The samples selected should represent multiple commodity categories from the commodity group, with a minimum of two different samples for each commodity category and should be representative for the intended scope of the laboratory. Additional validation data can be collected from on-going AQC-data and method performance verification during routine analysis.

Method performance acceptability criteria

G10 When the screening method is only intended to be used as a qualitative method, there are no requirements with regard to recovery of the analytes. In order to determine the selectivity, the presence of false detects should be verified using non-spiked (preferably "blank") samples. Provided the analytes that are tentatively detected by the screening method are identified and confirmed by a second analysis of the sample using an appropriate confirmatory method, there is no need for a strict criterion for the number of false detects. The SDL of the qualitative screening method is the lowest level at which an analyte has been detected (not necessarily meeting the MS-identification criteria) in at least 95% of the samples (i.e. an acceptable false-negative rate of 5%).

Validation – LC-QTOF

- We have validated at 3 different concentration levels in rye, oat, barley, wheat and rice.

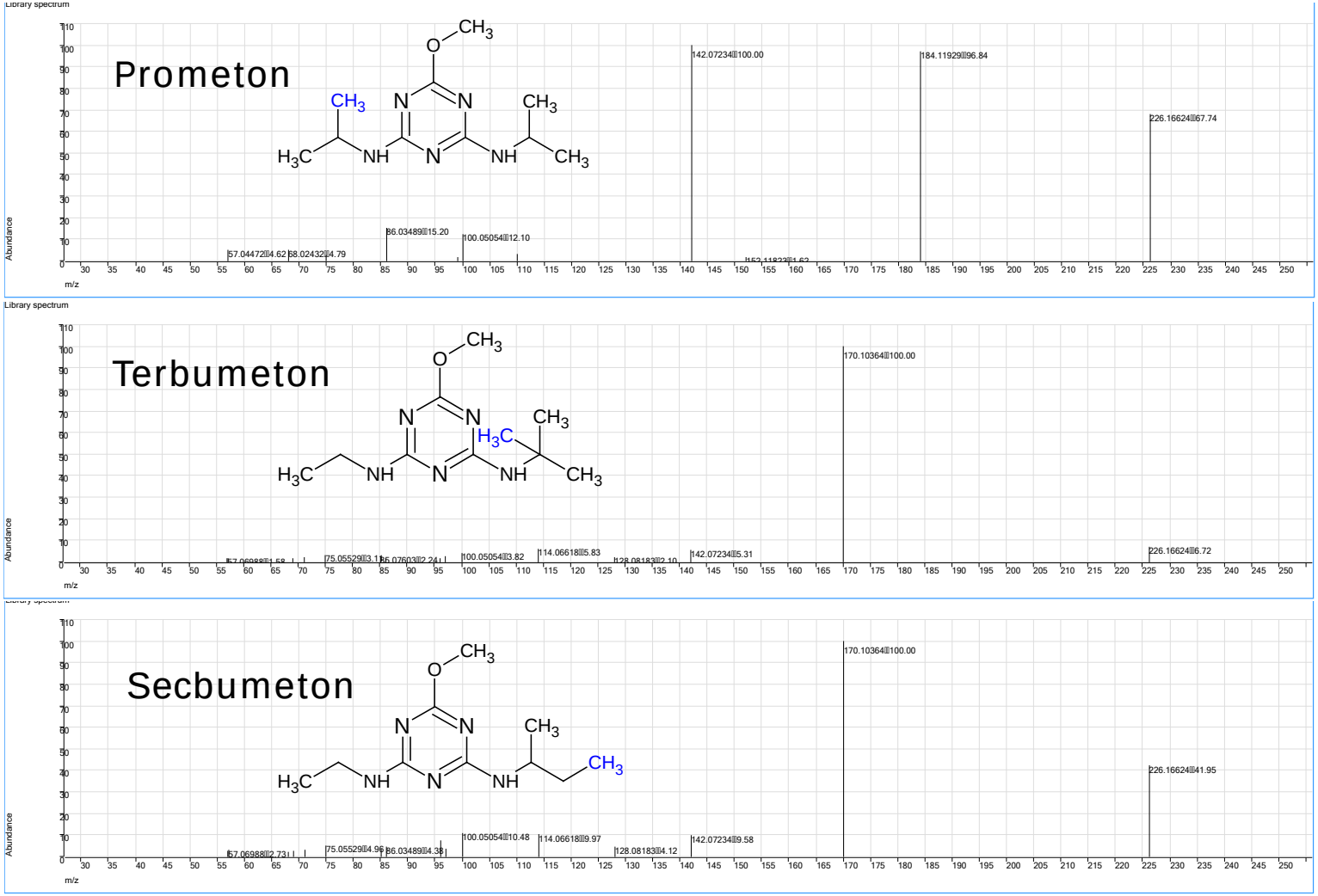
- Amounts of pesticides (including isomers) validated:

SDL (mg/kg)	Pesticides validated
0.01	246
0.02	41
0.1	39

Standard mixtures

Mixtures	50-100 pesticides
Isomers	Avoiding isomers in the mixtures
Validation	2-6 mixtures together

Spectra of isomers in Agilents library



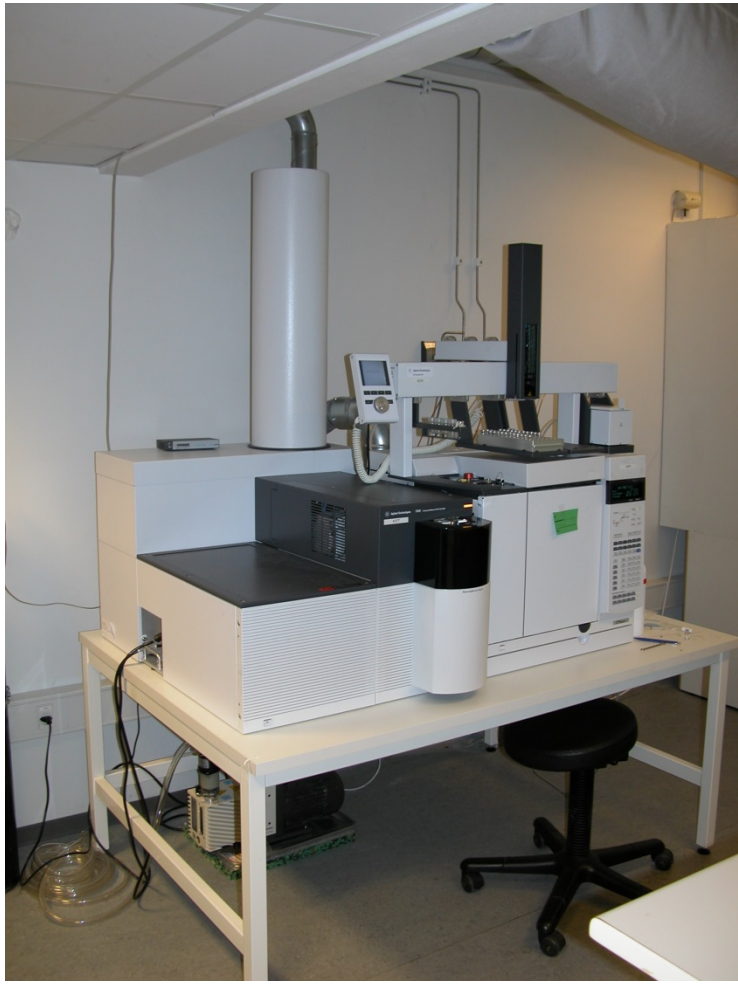
Disposition

- LC-QTOF screening - MassHunter Qualitative
- LC-QTOF - Validation
- **GC-QTOF screening - MassHunter Quantitative**
- GC-QTOF - Validation

EURL-CF and GC-QTOF

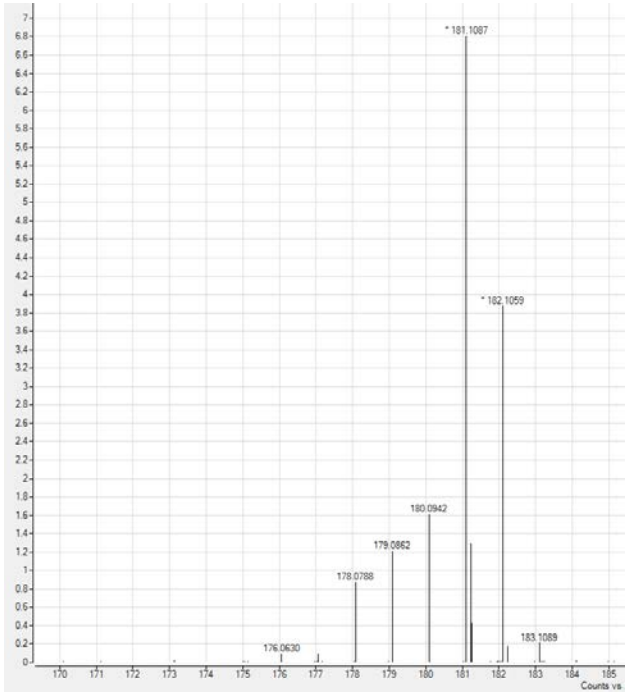
- TOF instrument: 7200 GC/Q-TOF, Agilent Technologies

- GC-system: 7890B, Agilent Technologies
  - back flush,
  - Gerstel PTV injector.
  - Two HP-5MS UI, 15 m, 0.250 mm dia, 0.25 mm filmtickness
  - PAL autosampler system and



Conditions

- Ionisation mode: EI positive
- Acquisition rate: 4GHz
- Acquisition mode: Centroid or profile
- Source temperature 230 ºC
- Software: MassHunter B 06.01.1312,



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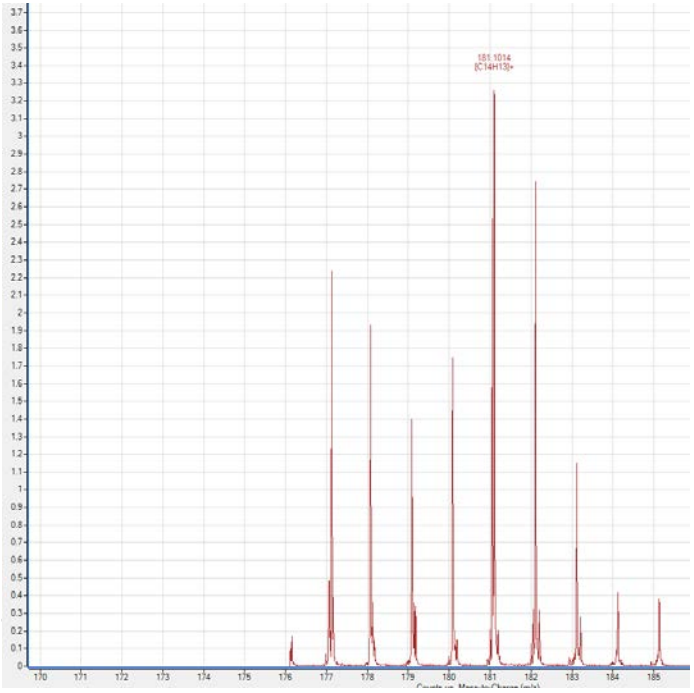


PCDL library

- Exact masses of pesticides and fragments
- Retention time

	A	B	C	D
1	# Compound database			
2	#			
3	#			
4	# This is a compound formula database file used by Agilent MassHunter applications.			
5	# Copy to a different file if you wish to make changes to database contents			
6	# You can set the version (below) to any string value.			
7	# We recommend changing it when you update the database contents.			
8	#			
9	#			
10	# Version: the most recent-one			
11	#			
12	#			
13	#			
14	# WARNING: User should not change the format of this file.			
15	#			
16	# Each record in this file contins 5 columns described in the last comment in this comment block.			
17	# Retention time and Description are optional.			
18	# Each record should be kept on an individual line.			
19	# Fields are separated by comma. Use quotes around a field that contains a comma.			
20	#			
21	# The first two lines have to be comments which start with the '#' character.			
22	# First line is 'Agilent TOF Formula data store'			
23	# Second line is 'Version:' followed by a version number			
24	#			
25	# Additior comments (such as these) may be inserted on individual			
26	# lines by specifying a '#' character at the beginning of the line			
27	#			
28	#			
29	### Formu	Retentior	Mass	Compound name
30	# Formula	RT	Mass	Cpd
31	C4H7O4P1	6.901	219.9459	Diclorvos
32	C4H7O4P1	6.901	184.977	Diclorvos F1
33	C2H6O3P1	6.901	109.0055	Diclorvos F2
34	C2H3Cl2O	6.901	143.9299	Diclorvos F3
35	C12H15NC	7.182	221.1052	Carbofuran
36	C10H12O2	7.182	164.0837	Carbofuran F1
37	C8H7NO2	7.182	149.0477	Carbofuran F2
38	C8H5NO	7.182	131.0371	Carbofuran F3
39	C7H5O2	7.182	121.029	Carbofuran F4
40	C14H9N2C	7.261	310.0321	Diflubenzuron
41	C7H5NOF2	7.261	157.0339	Diflubenzuron F1
42	C7H3OF2	7.261	141.0152	Diflubenzuron F2
43	C6H3F2	7.261	113.0203	Diflubenzuron F3
44	C4H10NO3	7.933	183.0119	Acephate

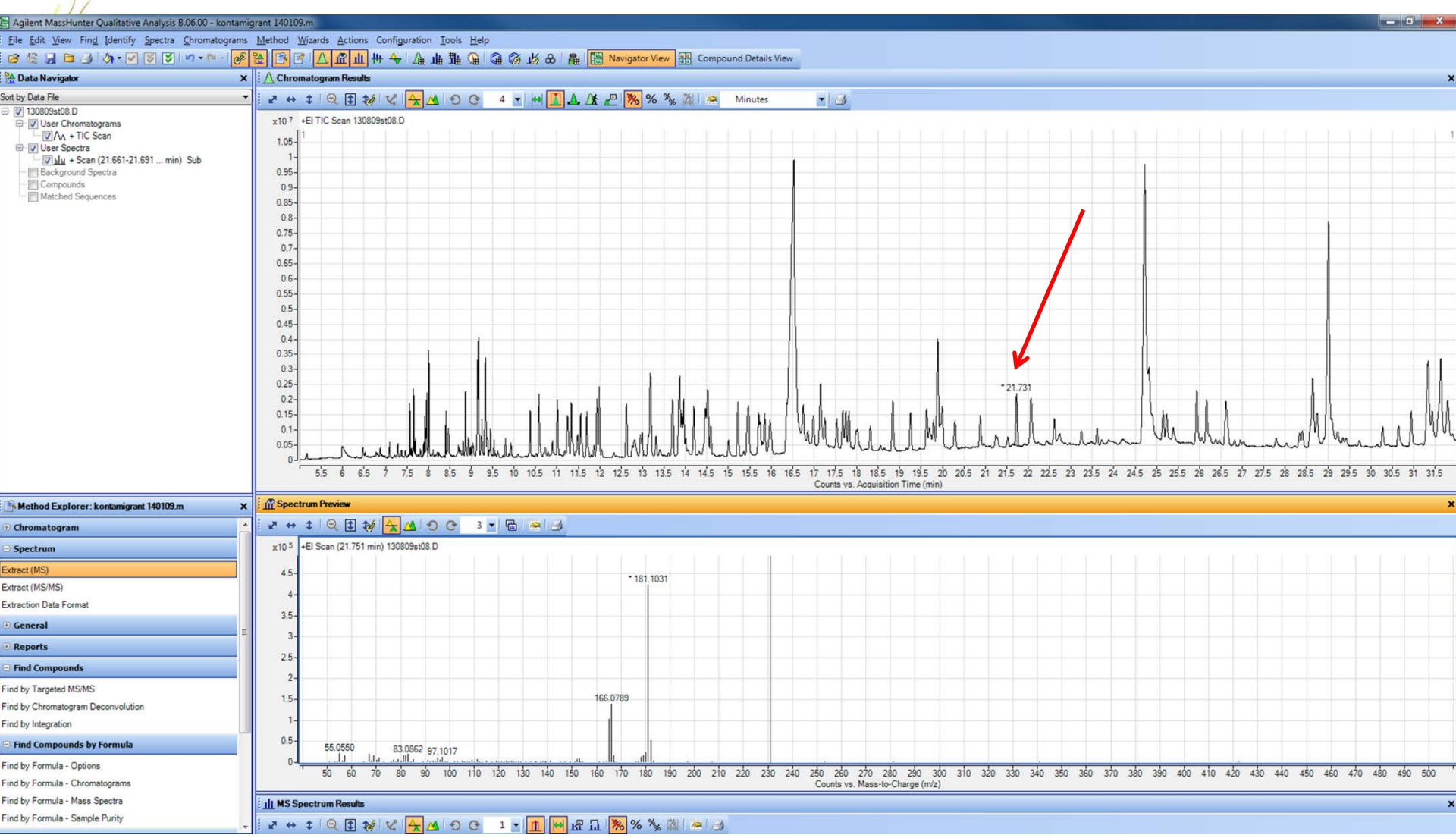
A454 =elem(G$1;G454)&elem(H$1;H454)&elem(I$1;I454)&elem(J$1;J454)&elem(K$1;K454)&elem(L$1;L454)&elem(M$1;M454)&elem(N$1;N454)&elem(O$1;O454)&elem(P$1;P454)&elem(Q$1;Q454)

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q
1	**Formula**	**Retention Time**	**Mass**	**Compound**		**Description**	**C**	**H**	**D**	**N**	**O**	**P**	**S**	**Cl**	**F**	**Br**	**Si**
2							12.000000	1.007825	2.014102	14.003074	15.994915	30.973762	31.972071	34.968853	18.998403	78.918338	27.976927
423	C12H7OCl	28.111	202.0185426	Difenoconazole 1 F3	6		12	7			1			1			
424	C19H17N3O3Cl2	28.24	405.0646968	Difenoconazole 2	6		19	17		3	3			2			
425	C16H13O3Cl2	28.24	323.0241747	Difenoconazole 2 F1	6		16	13			3			2			
426	C13H7O2Cl2	28.24	264.9823099	Difenoconazole 2 F2	6		13	7			2			2			
427	C12H7OCl	28.24	202.0185426	Difenoconazole 2 F3	6		12	7			1			1			
428	C22H19NO3Br2	28.385	502.9731687	Deltamethrin 1	6		22	19		1	3					2	
429	C7H9Br2	28.385	250.9071005	Deltamethrin 1 F1	6		7	9								2	
430	C7H9Br	28.385	171.9887629	Deltamethrin 1 F2	6		7	9								1	
431	C22H19NO3Br2	28.776	502.9731687	Deltamethrin 2	6		22	19		1	3					2	
432	C7H9Br2	28.776	250.9071005	Deltamethrin 2 F1	6		7	9								2	
433	C7H9Br	28.776	171.9887629	Deltamethrin 2 F2	6		7	9								1	
434	C12H9NO3Cl2	12.3	284.9959486	Vinclozolin	6		12	9		1	3			2			
435	C7H3NOCl2	12.3	186.9591691	Vinclozolin F1	6		7	3		1	1			2			
436	C9H5NOCl2	12.3	212.9748192	Vinclozolin F2	6		9	5		1	1			2			
437	C10H8NCl2	12.3	212.0033797	Vinclozolin F3	6		10	8		1				2			
438	C10H16O	7.11	152.1201151	Carvone			10	16			1						
439	C10H14O	7.11	150.1044651	Carvone F1			10	14			1						
440	C8H14	7.11	110.1095504	Carvone F2			8	14									
441	C7H11	7.11	95.08607535	Carvone F3			7	11									
442	C20H32N2O3S		380.2133636	Carbosulfan			20	32		2	3		1				
443	C8H18NS		160.1159953	Carbosulfan F1			8	18		1			1				
444	C10H11O2		163.0759046	Carbosulfan F2			10	11			2						
445	C7H3O3		135.008219	Carbosulfan F3			7	3			3						
446	C6H3O2		107.0133043	Carbosulfan F4			6	3			2						
447	C12H8OCl6		377.8706311	Endrin			12	8			1			6			
448	C7H2Cl5		260.8599136	Endrin F1			7	2						5			
449	C5H5O		81.03403978	Endrin F2			5	5			1						
450	C12H8OCl5		342.9017784	Endrin F3			12	8			1			5			
451	C23H26O3		350.1881947	Phenothrin			23	26			3						
452	C13H11O		183.08099	Phenothrin F1			13	11			1						
453	C9H15		123.1173755	Phenothrin F2			9	15									
454			0														
455			0														
456			0														
457			0														
458			0														
459			0														
460			0														

Instructions | Calculator | CSV

Marker destinationsområde, og tryk på Enter

130%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Navigator View
Compound Details View
Data Navigator
Sort by Data File
130809st08.D
User Chromatograms
+ TIC Scan
User Spectra
+ Scan (21.661-21.691 ... min) Sub
Background Spectra
Compounds
Matched Sequences
Chromatogram Results
x10 7 +EI TIC Scan 130809st08.D
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Minutes
Counts vs. Acquisition Time (min)
Method Explorer: kontamigrant 140109.m
Chromatogram
Spectrum
Extract (MS)
Extract (MS/MS)
Extraction Data Format
General
Reports
Find Compounds
Find by Targeted MS/MS
Find by Chromatogram Deconvolution
Find by Integration
Find Compounds by Formula
Find by Formula - Options
Find by Formula - Chromatograms
Find by Formula - Mass Spectra
Find by Formula - Sample Purity
Spectrum Preview
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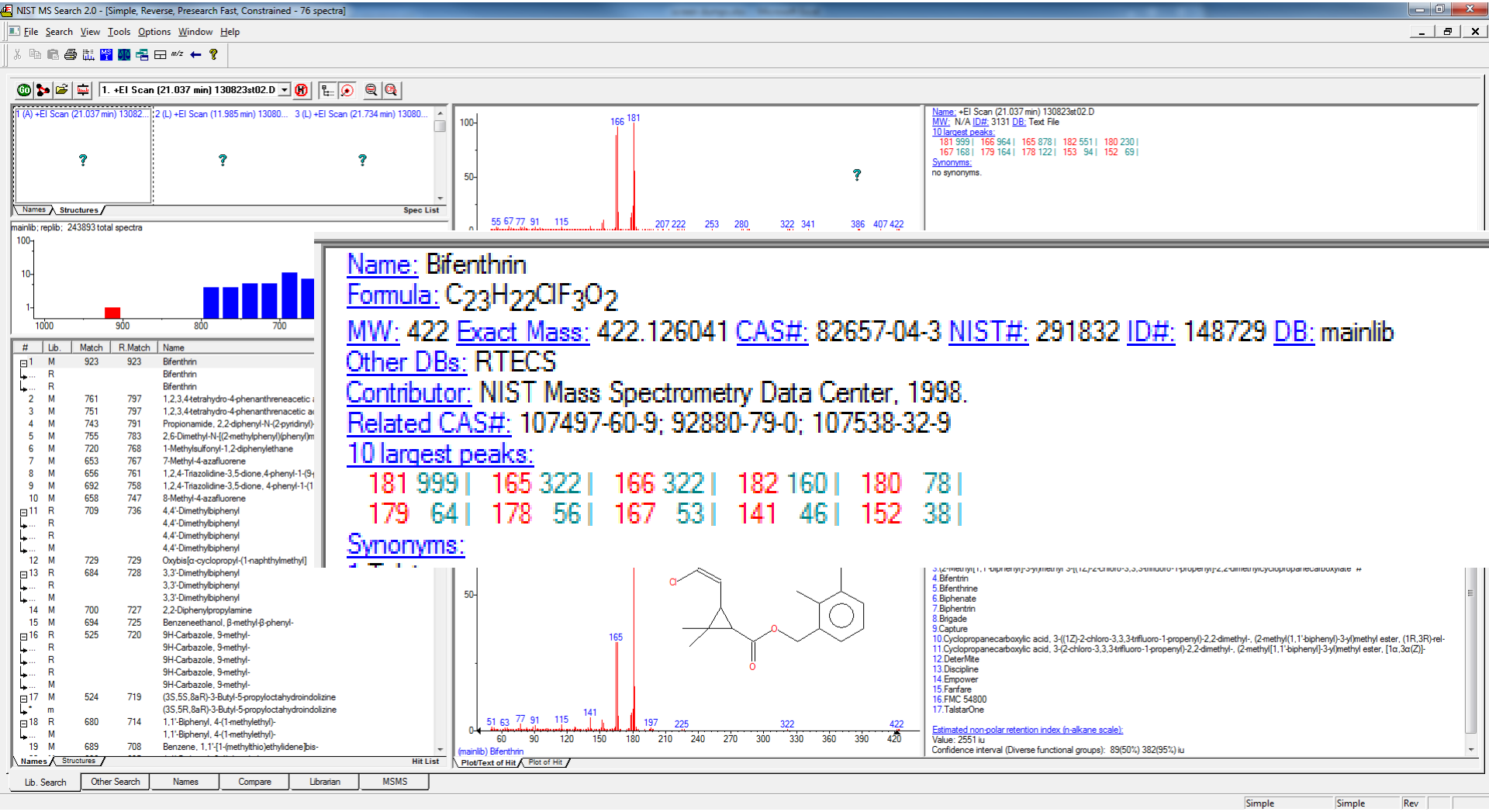
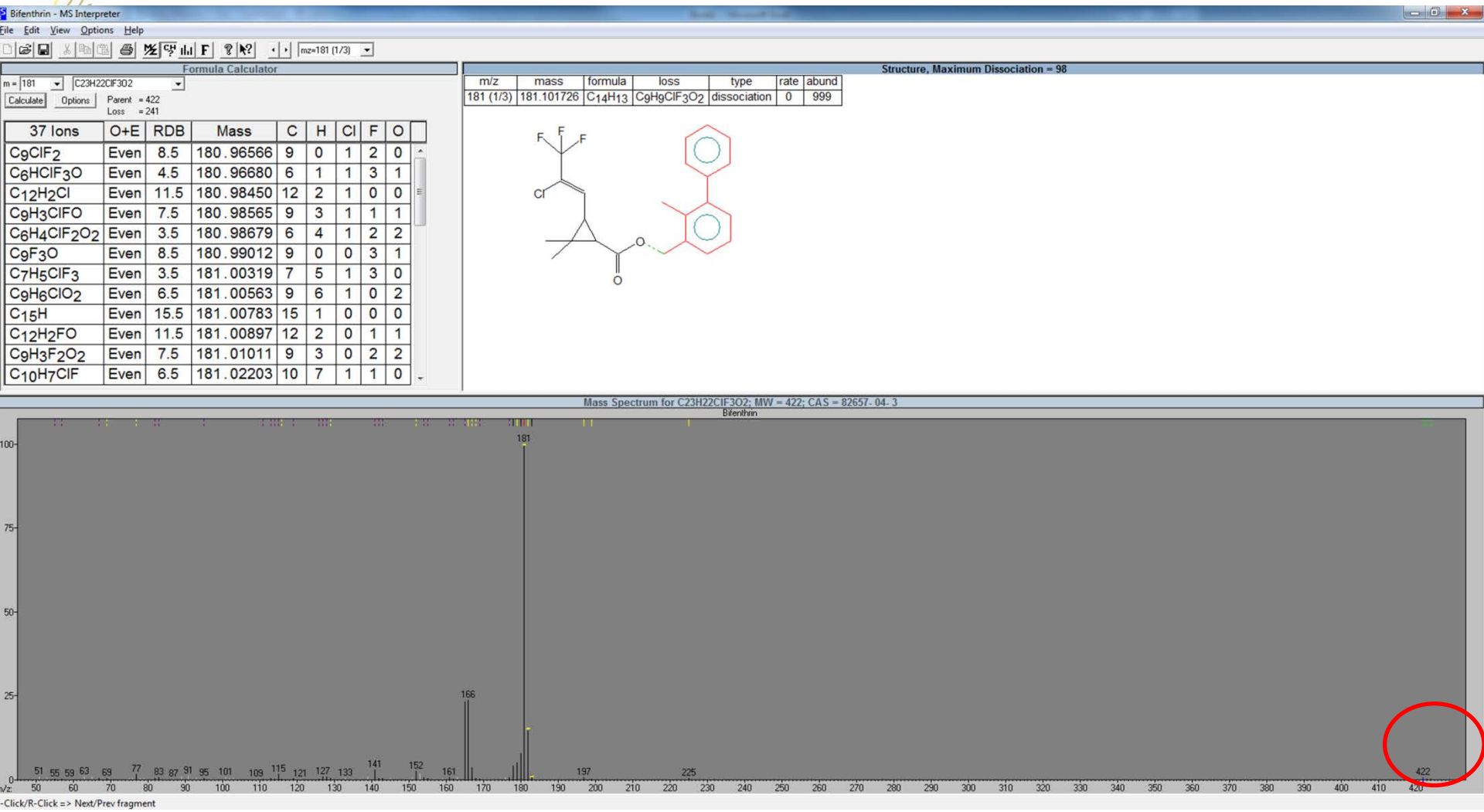
83.0862
97.1017
Counts vs. Mass-to-Charge (m/z)
MS Spectrum Results

Exact masses of fragments





- Bifentrin – mass 422.126041

Bifenthrin - MS Interpreter

File Edit View Options Help

Formula Calculator

m = 166 C23H22ClF3O2

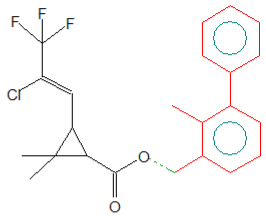
Calculate Options Parent = 422 Loss = 256

32 Ions	O+E	RDB	Mass	C	H	Cl	F	O
C_8ClFO	Odd	8	165.96217	8	0	1	1	1
$C_5HClF_2O_2$	Odd	4	165.96331	5	1	1	2	2
$C_6H_2ClF_3$	Odd	4	165.97971	6	2	1	3	0
$C_8H_3ClO_2$	Odd	7	165.98216	8	3	1	0	2
$C_8F_2O_2$	Odd	8	165.98664	8	0	0	2	2
C_9H_4ClF	Odd	7	165.99856	9	4	1	1	0
$C_6H_5ClF_2O$	Odd	3	165.99970	6	5	1	2	1
C_9HF_3	Odd	8	166.00303	9	1	0	3	0
$C_{11}H_2O_2$	Odd	11	166.00548	11	2	0	0	2
C_9H_7ClO	Odd	6	166.01854	9	7	1	0	1
$C_6H_8ClFO_2$	Odd	2	166.01969	6	8	1	1	2
$C_{12}H_3F$	Odd	11	166.02188	12	3	0	1	0

Structure, Maximum Dissociation = 98

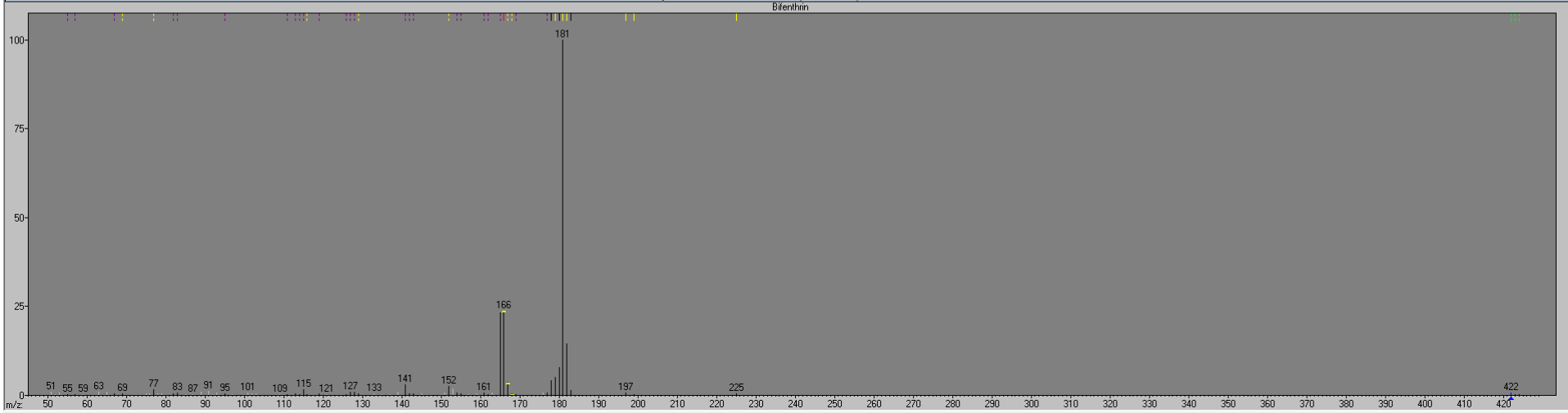
m/z	mass	formula	loss	type	rate	abund
166	166.07825	$C_{13}H_{10}$	$C_9HgClF_3O_2 + CH_3$	dissociation	0	236

- CH3



Mass Spectrum for C23H22ClF3O2; MW = 422; CAS = 82657-04-3

Bifenthrin



For Help, press F1

Bifenthrin - MS Interpreter

File Edit View Options Help

mz=165 (1/3)

Formula Calculator

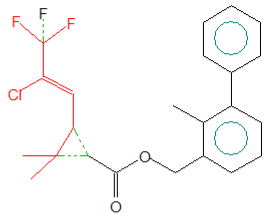
m = 165 C23H22ClF3O2

Calculate Options Parent = 422 Loss = 257

34 Ions	O+E	RDB	Mass	C	H	Cl	F	O
$C_5ClF_2O_2$	Even	4.5	164.95549	5	0	1	2	2
C_6HClF_3	Even	4.5	164.97189	6	1	1	3	0
$C_8H_2ClO_2$	Even	7.5	164.97433	8	2	1	0	2
C_9H_3ClF	Even	7.5	164.99073	9	3	1	1	0
$C_6H_4ClF_2O$	Even	3.5	164.99187	6	4	1	2	1
$C_3H_5ClF_3O_2$	+	0	164.99302	3	5	1	3	2
C_9F_3	Even	8.5	164.99521	9	0	0	3	0
$C_{11}HO_2$	Even	11.5	164.99765	11	1	0	0	2
C_9H_6ClO	Even	6.5	165.01072	9	6	1	0	1
$C_6H_7ClFO_2$	Even	2.5	165.01186	6	7	1	1	2
$C_{12}H_2F$	Even	11.5	165.01405	12	2	0	1	0
$C_9H_3F_2O$	Even	7.5	165.01520	9	3	0	2	1

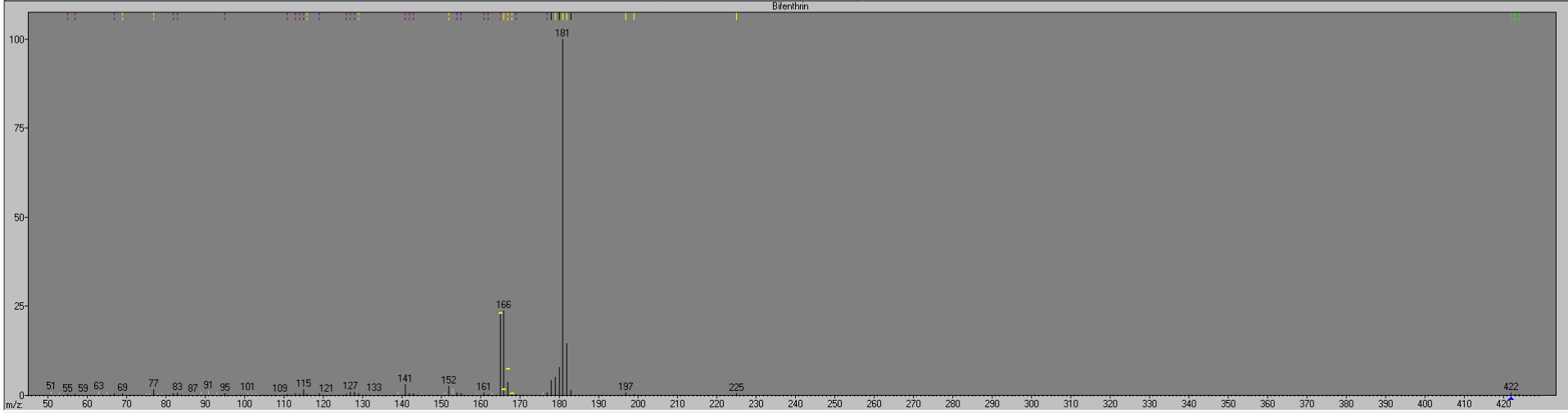
Structure, Maximum Dissociation = 98

m/z	mass	formula	loss	type	rate	abund
165 (1/3)	165.028259	$C_7H_8ClF_2$	$C_{16}H_{14}FO_2$	unspecified	N/A	231

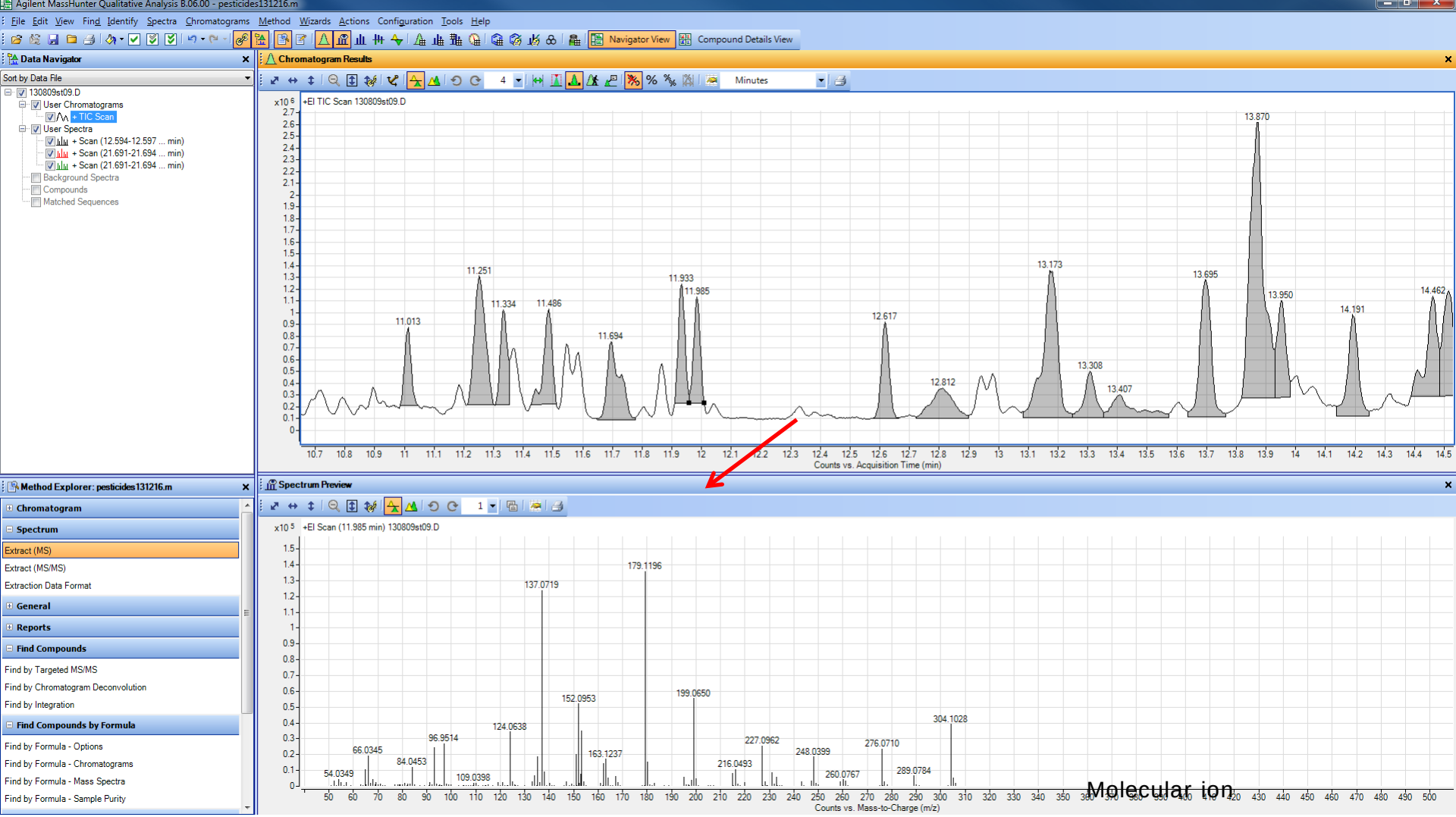


Mass Spectrum for C23H22ClF3O2; MW = 422; CAS = 82657-04-3

Bifenthrin



Exact molecular weight. Click to sort.


Agilent MassHunter Qualitative Analysis B.06.00 - pesticides131216.m
File Edit View Find Identify Spectra Chromatograms Method Wizards Actions Configuration Tools Help
Navigator View
Compound Details View
Data Navigator
Sort by Data File
130809st09.D
User Chromatograms
+ TIC Scan
User Spectra
+ Scan (12.594-12.597 ... min)
+ Scan (21.691-21.694 ... min)
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Compounds
Matched Sequences
Chromatogram Results
Minutes
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11.694
11.933
11.985
12.617
12.812
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13.407
13.695
13.870
13.950
14.191
14.462
Counts vs. Acquisition Time (min)
Method Explorer: pesticides131216.m
Chromatogram
Spectrum
Extract (MS)
Extract (MS/MS)
Extraction Data Format
General
Reports
Find Compounds
Find by Targeted MS/MS
Find by Chromatogram Deconvolution
Find by Integration
Find Compounds by Formula
Find by Formula - Options
Find by Formula - Chromatograms
Find by Formula - Mass Spectra
Find by Formula - Sample Purity
Spectrum Preview
+EI Scan (11.985 min) 130809st09.D
179.1196
137.0719
152.0953
199.0650
304.1028
124.0638
96.9514
66.0345
84.0453
54.0349
109.0398
163.1237
216.0493
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248.0399
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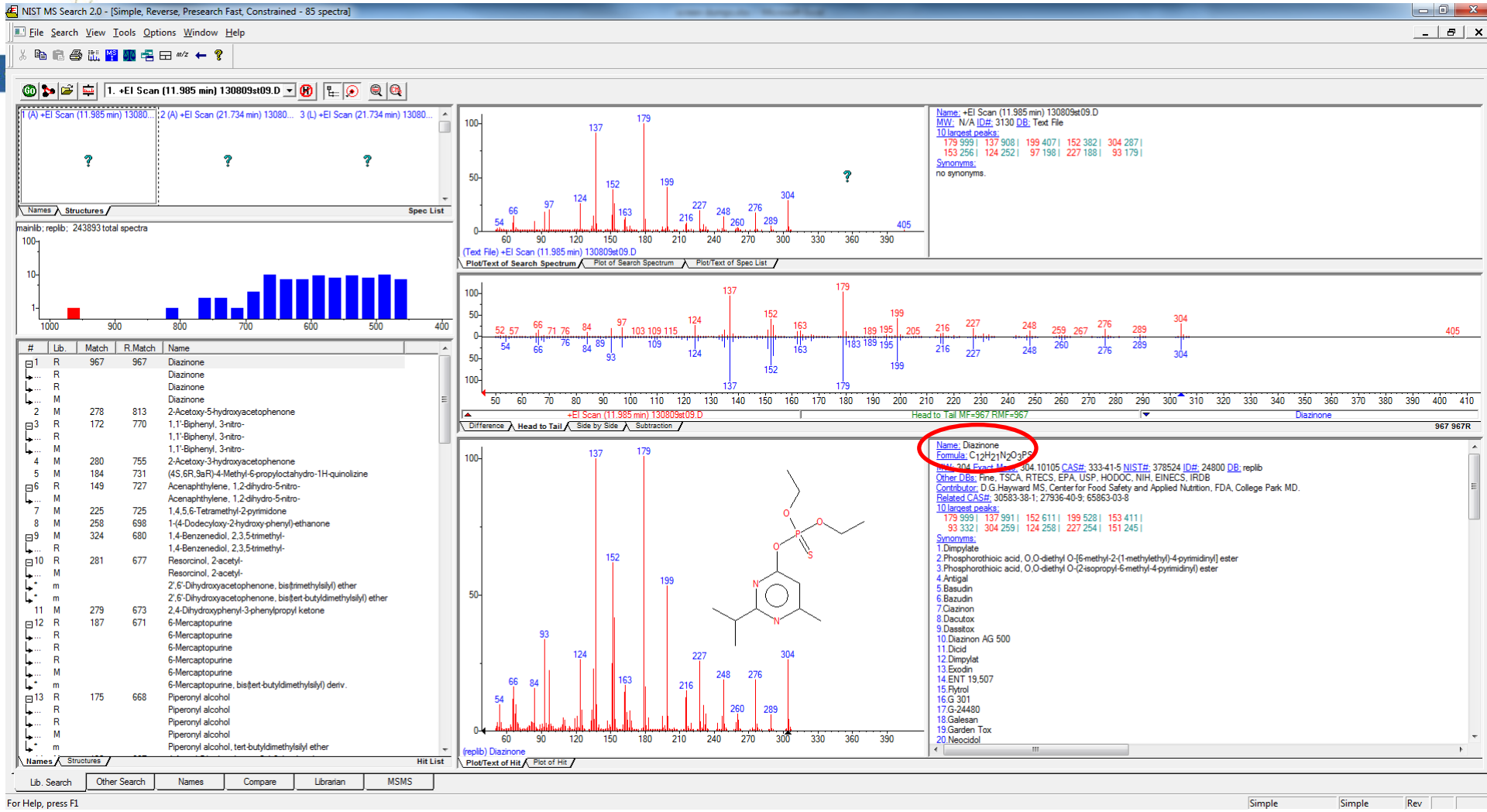
289.0784
Counts vs. Mass-to-Charge (m/z)
Molecular ion



National Food Institute, Technical University of Denmark

Diazinone - MS Interpreter

File Edit View Options Help

Formula Calculator

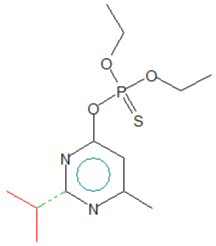
m = 179 C12H21N2O3PS

Calculate Options Parent = 304 Loss = 125

84 Ions	O+E	RDB	Mass	C	H	N	O	P
C_7O_2PS	Even	8.5-11.5	178.93566	7	0	0	2	1
C_6N_2OPS	Even	8.5-13.5	178.94690	6	0	2	1	1
C_7H_2NOPS	Odd	8-12	178.95947	7	2	1	1	1
$C_6N_2O_3P$	Even	8.5-11.5	178.96465	6	0	2	3	1
C_7HNO_3S	Odd	8-11	178.96771	7	1	1	3	0
$C_3H_4N_2O_3PS$	Even	3.5-8.5	178.96802	3	4	2	3	1
$C_{11}OP$	Even	12.5-13.5	178.96868	11	0	0	1	1
C_8H_4OPS	Even	7.5-10.5	178.97205	8	4	0	1	1
$C_7H_2NO_3P$	Odd	8-10	178.97723	7	2	1	3	1
$C_{10}N_2P$	Even	12.5-15.5	178.97991	10	0	2	0	1
$C_8H_3O_3S$	Even	7.5-9.5	178.98029	8	3	0	3	0

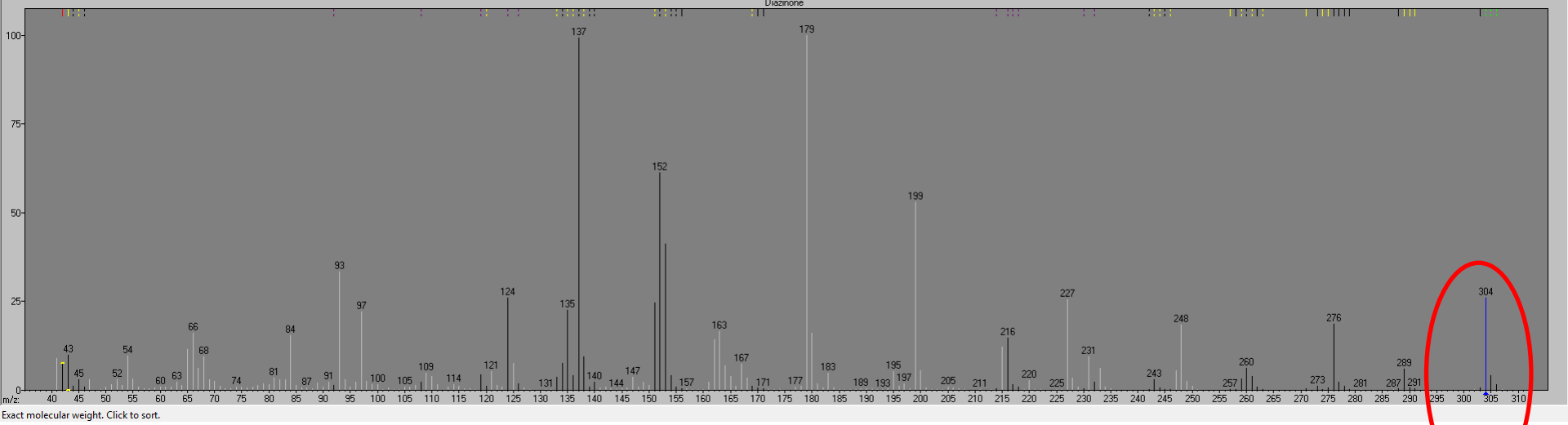
Structure, Maximum Dissociation = 93

m/z	mass	formula	loss	type	H	rate	abund
42	42.04695	C_3H_6	$C_9H_{15}N_2O_3PS$	dissociation	-1	-15*	77



Mass Spectrum for C12H21N2O3PS; MW = 304; CAS = 333-41-5

Diazinone



Exact molecular weight. Click to sort.

Diazinone - MS Interpreter

File Edit View Options Help

Formula Calculator

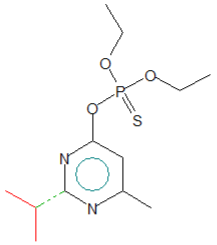
m = 179 C12H21N2O3PS

Calculate Options Parent = 304 Loss = 125

84 Ions	O+E	RDB	Mass	C	H	N	O	P
C_7O_2PS	Even	8.5-11.5	178.93566	7	0	0	2	1
C_6N_2OPS	Even	8.5-13.5	178.94690	6	0	2	1	1
C_7H_2NOPS	Odd	8-12	178.95947	7	2	1	1	1
$C_6N_2O_3P$	Even	8.5-11.5	178.96465	6	0	2	3	1
C_7HNO_3S	Odd	8-11	178.96771	7	1	1	3	0
$C_3H_4N_2O_3PS$	Even	3.5-8.5	178.96802	3	4	2	3	1
$C_{11}OP$	Even	12.5-13.5	178.96868	11	0	0	1	1
C_8H_4OPS	Even	7.5-10.5	178.97205	8	4	0	1	1
$C_7H_2NO_3P$	Odd	8-10	178.97723	7	2	1	3	1
$C_{10}N_2P$	Even	12.5-15.5	178.97991	10	0	2	0	1
$C_8H_3O_3S$	Even	7.5-9.5	178.98029	8	3	0	3	0

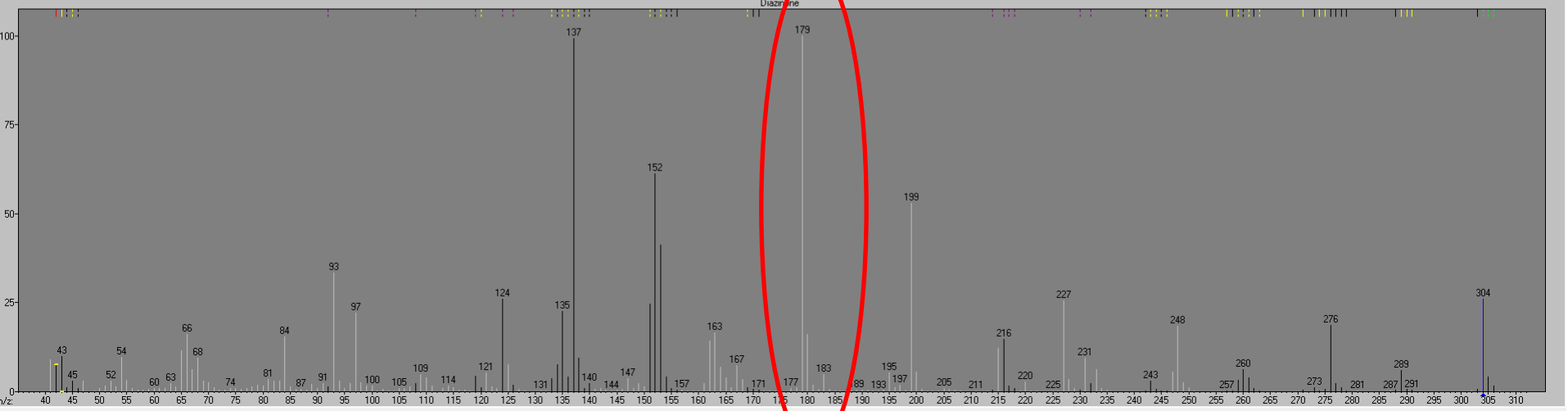
Structure, Maximum Dissociation = 93

m/z	mass	formula	loss	type	H	rate	abund
42	42.04695	C_3H_6	$C_9H_{15}N_2O_3PS$	dissociation	-1	-15*	77



Mass Spectrum for C12H21N2O3PS; MW = 304; CAS = 333-41-5

Diazinone



xact molecular weight. Click to sort.

e - MS Interpreter

View Options Help

mz=216 (1/2)

Formula Calculator

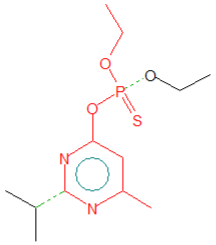
C12H21N2O3PS

Options Parent = 304 Loss = 88

9 Ions	O+E	RDB	Mass	C	H	N	O	P
O_2PS	Odd	11-14	215.94349	10	1	0	2	1
I_2OPS	Odd	11-16	215.95472	9	1	2	1	1
O_3S	Odd	11-15	215.96296	9	0	2	3	0
$_3NOPS$	Even	10.5-14.5	215.96730	10	3	1	1	1
I_2O_3P	Odd	11-14	215.97248	9	1	2	3	1
$_2NO_3S$	Even	10.5-13.5	215.97554	10	2	1	3	0
N_2O_3PS	Odd	6-11	215.97585	6	5	2	3	1
$_5OPS$	Odd	10-13	215.97987	11	5	0	1	1
$_3NO_3P$	Even	10.5-12.5	215.98505	10	3	1	3	1
$_4O_3S$	Odd	10-12	215.98811	11	4	0	3	0
NO_3PS	Even	5.5-9.5	215.98843	7	7	1	3	1

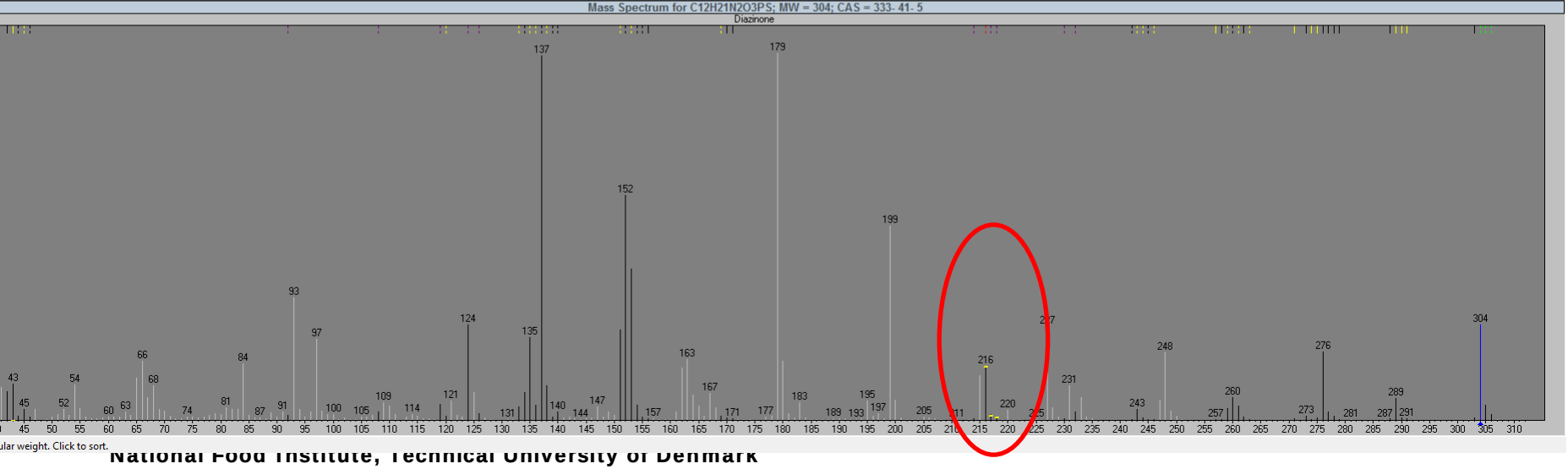
Structure, Maximum Dissociation = 93

m/z	mass	formula	loss	type	rate	abund
216 (1/2)	216.012236	$C_7HgN_2O_2PS$	$C_5H_{12}O$	unspecified	N/A	146



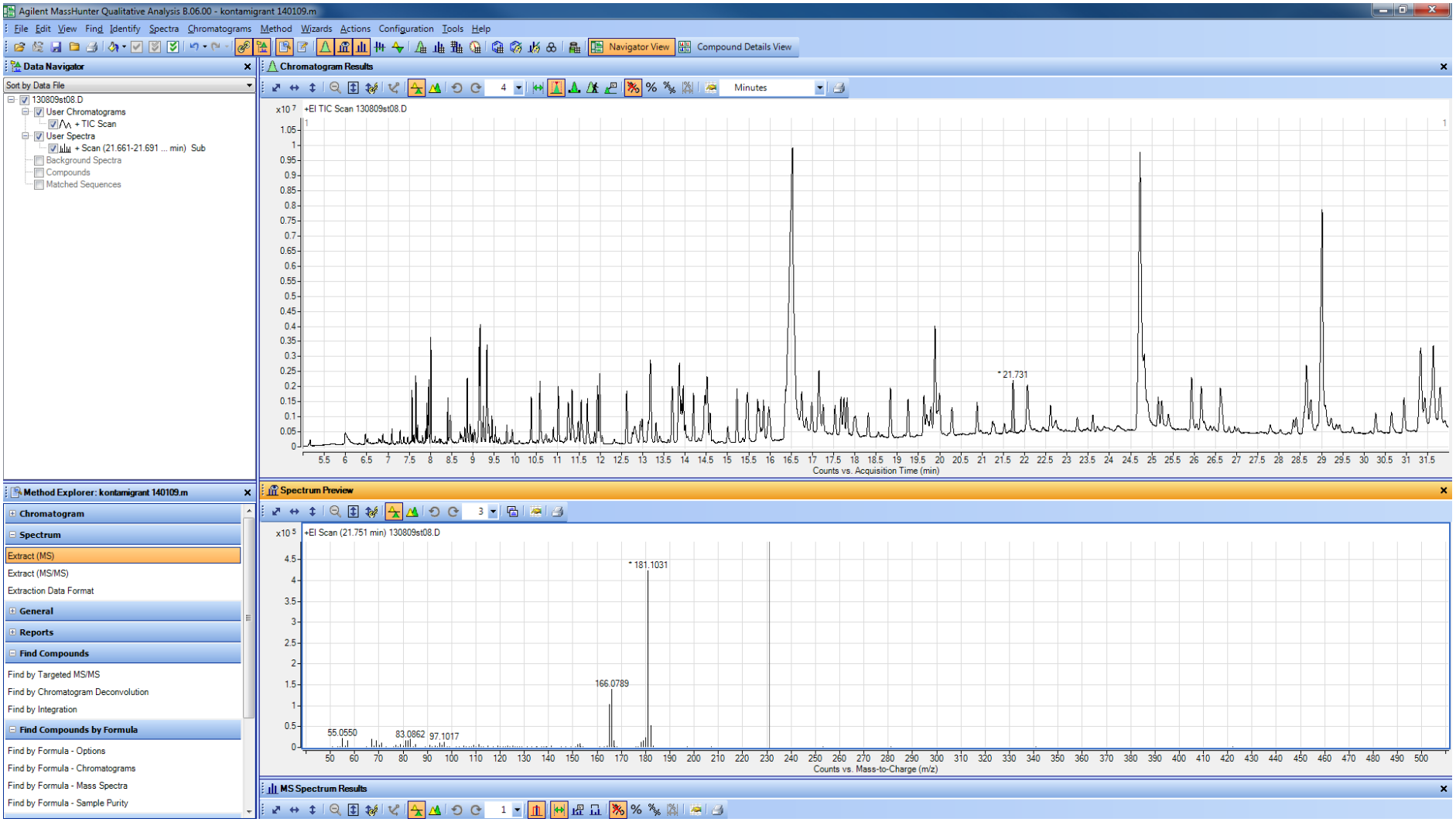
Mass Spectrum for C12H21N2O3PS; MW = 304; CAS = 333-41-5

Diazinone



ular weight. Click to sort.

Qualitative software



Compound List

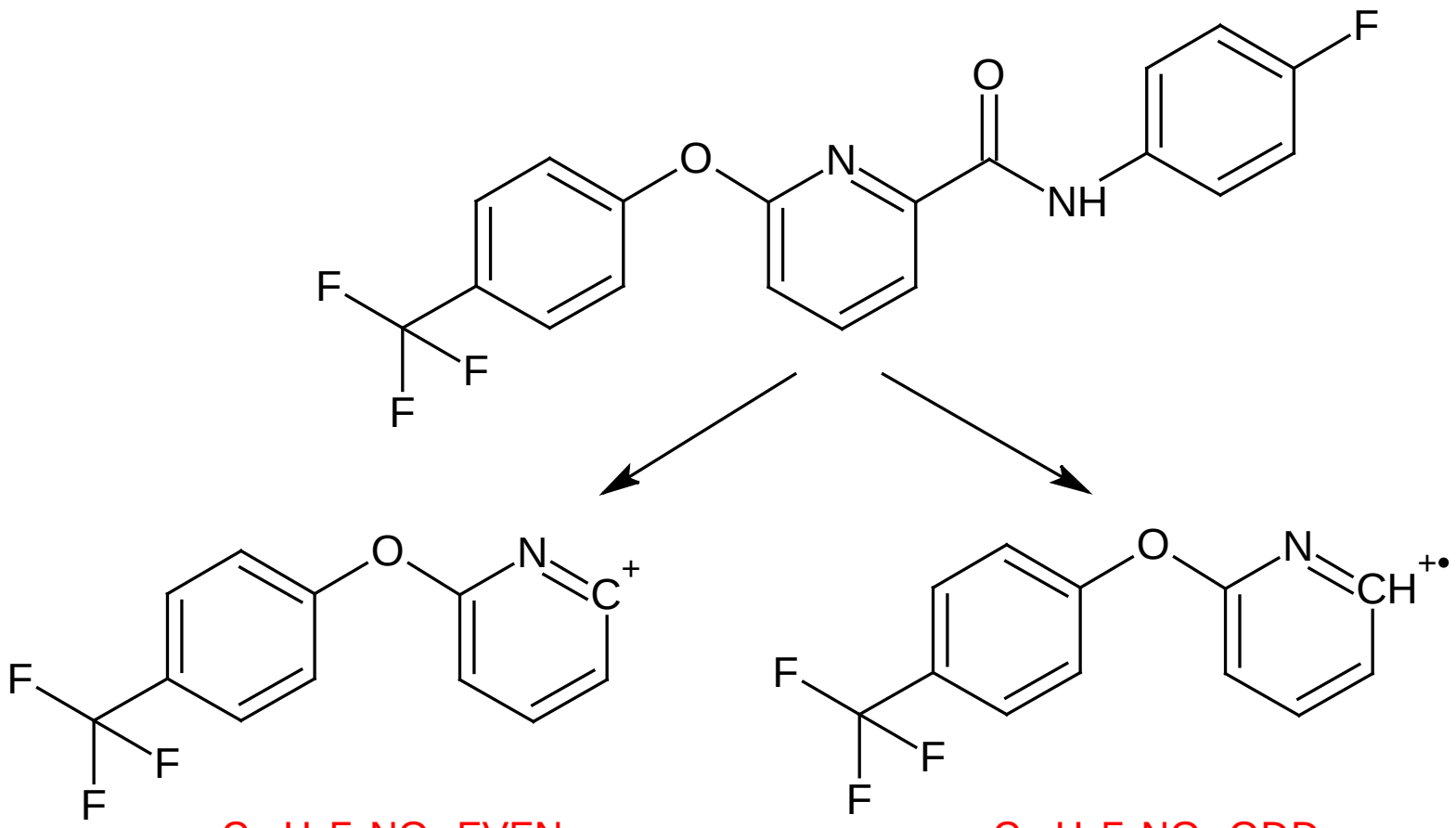
Automatically Show Columns

Name	RT (Tgt)	RT	Height	Mass (DB)	Mass	Diff (DB, pp	Score	Flags (Tgt)	Flag Severity (Tgt)	Formula	Ions	File
Atrazin	10.72	10.71	33758	215.0938	215.0946	-3.69	93.88		Pass	C8 H14 Cl N5	5	130923st0
Atrazin F1	10.72	10.71	66926	200.0703	200.0712	-4.48	96.5		Pass	C7 H11 Cl N5	5	130923st0
Atrazin F2	10.72	10.71	19056	173.0468	173.0462	3.71	76.83		Pass	C5 H8 Cl N5	4	130923st0
Atrazin F3	10.72	10.71	12094	172.039	172.0402	-7.11	59.21	low score; ion count	Warning	C5 H7 Cl N5	1	130923st0
Bromoxynil	10.06	10.038	26297	274.8581	274.8588	-2.55	97.19		Pass	C7 H3 Br2 N O	6	130923st0
Clomazone F1	10.84	10.816	16023	204.1025	204.103	-2.66	97.44		Pass	C12 H14 N O2	3	130923st0
Clomazone F2	10.84	10.812	23311	125.0158	125.0159	-0.84	97.9		Pass	C7 H6 Cl	4	130923st0
Cyfluthrin F1	25.75	25.743	2109	226.0668	226.0673	-2.23	63.63	low score; ion count	Warning	C14 H9 F N O	1	130923st0
Cyfluthrin F2	25.75	25.739	9220	163.0081	163.0087	-3.41	88.91		Pass	C7 H9 Cl2	4	130923st0
Cyfluthrin F3	25.75	25.749	2858	199.0559	199.0572	-6.69	68.13	low score	Warning	C13 H8 F O	2	130923st0
Cyfluthrin F4	25.75	25.759	923	227.0746	227.0741	2.25	74.82	low score	Warning	C14 H10 F N O	2	130923st0
Dieldrin	16.66	16.632	4149	377.8706	377.8708	-0.56	96.55		Pass	C12 H8 Cl6 O	9	130923st0
Dieldrin F1	16.66	16.639	8759	274.8756	274.8761	-2.01	88.88		Pass	C8 H4 Cl5	8	130923st0
Dieldrin F2	16.66	16.639	13372	260.8599	260.8609	-3.68	76.51		Pass	C7 H2 Cl5	4	130923st0
Flufenacet	13.94	13.904	603	363.0665	363.0664	0.22	81.24		Pass	C14 H13 F4 N3 O2 S	2	130923st0
Flufenacet F1	13.94	13.908	9049	210.9789	210.9796	-3.23	95		Pass	C5 H2 F3 N2 O2 S	3	130923st0
Flufenacet F2	13.94	13.904	13296	151.0797	151.08	-2.01	82.03		Pass	C9 H10 F N	2	130923st0
Flufenacet F3	13.94	13.911	5285	136.0563	136.0567	-3.12	77.59		Pass	C8 H7 F N	2	130923st0
Metramitron	16.95	16.92	2588	202.0855	202.0861	-3.31	86.6		Pass	C10 H10 N4 O	2	130923st0
Metramitron F1	16.95	16.923	2201	174.0905	174.0917	-6.75	79.2		Pass	C9 H10 N4	2	130923st0
Metramitron F2	16.95	16.92	3401	104.05	104.0501	-0.44	86.45		Pass	C7 H6 N	2	130923st0
Metramitron F3	16.95	16.917	1061	173.0827	173.0831	-2.34	61.33	low score; ion count	Warning	C9 H9 N4	1	130923st0
Metribuzin F1	12.32	12.287	18809	198.0701	198.0705	-2.02	88.61		Pass	C8 H12 N3 O S	4	130923st0
Metribuzin F2	12.32	12.287	2130	182.0388	182.0393	-2.49	93.63		Pass	C7 H8 N3 O S	3	130923st0
Metribuzin F3	12.32	12.284	1934	144.047	144.0471	-0.75	95.25		Pass	C4 H8 N4 S	3	130923st0
Molinate	8.81	8.802	1185	187.1031	187.1032	-0.38	96.99		Pass	C9 H17 N O S	3	130923st0
Molinate F1	8.81	8.798	11463	126.0919	126.092	-1.19	99.28		Pass	C7 H12 N O	3	130923st0
Molinate F2	8.81	8.805	2809	98.097	98.0972	-2.64	91.37		Pass	C6 H12 N	2	130923st0
Oxamyl F2	8.74	8.719	725	145.0436	145.0445	-6.39	76.35		Pass	C5 H9 N2 O S	3	130923st0
Picolinafen	21	20.974	31497	376.0835	376.0843	-2.13	96.92		Pass	C19 H12 F4 N2 O2	4	130923st0
Picolinafen F1	21	20.971	25213	239.0558	239.0558	-0.05	96.61		Pass	C12 H8 F3 N O	3	130923st0
Picolinafen F2	21	20.971	40926	238.048	238.0489	-3.84	60.18	low score; ion count	Warning	C12 H7 F3 N O	1	130923st0
Prosulfocarb	12.94	12.922	10355	251.1344	251.1352	-3.33	96.32		Pass	C14 H21 N O S	4	130923st0
Prosulfocarb F1	12.94	12.922	3045	160.0796	160.0801	-3.05	92.11		Pass	C7 H14 N O S	3	130923st0
Prosulfocarb F2	12.94	12.922	26712	128.1075	128.1076	-0.86	99		Pass	C7 H14 N O	3	130923st0
Quinoclamine	13.4	13.388	4839	207.0087	207.0108	-9.88	87.71		Pass	C10 H6 Cl N O2	4	130923st0
Quinoclamine F1	13.4	13.392	3570	172.0399	172.0403	-2.83	89.79		Pass	C10 H6 N O2	2	130923st0
Quinoclamine F2	13.4	13.392	983	144.0449	144.0453	-2.36	91.14		Pass	C9 H6 N O	2	130923st0
Triflumizole F1	15.4	15.369	1669	278.056	278.0563	-1.19	92		Pass	C12 H12 Cl F3 N O	3	130923st0
Triflumizole F2	15.4	15.376	1139	287.0437	287.043	2.44	74.8	low score	Warning	C12 H9 Cl F3 N3	3	130923st0
Triflumizole F3	15.4	15.372	1495	205.9984	205.9995	-5.08	59.35	low score; ion count	Warning	C8 H4 Cl F3 N	1	130923st0

Score system

- *Mass Match Score* is the weighted aggregate of the individual metrics calculated when comparing an actual spectrum with a spectrum pattern synthesized from a formula.
  - *Mass Spacing Score*
  - *Mass Accuracy Score*
  - *Mass Abundance Score*
  - *Isotope abundance score*
  - *Isotope spacing score*

Picolinafen - overlapping fragments

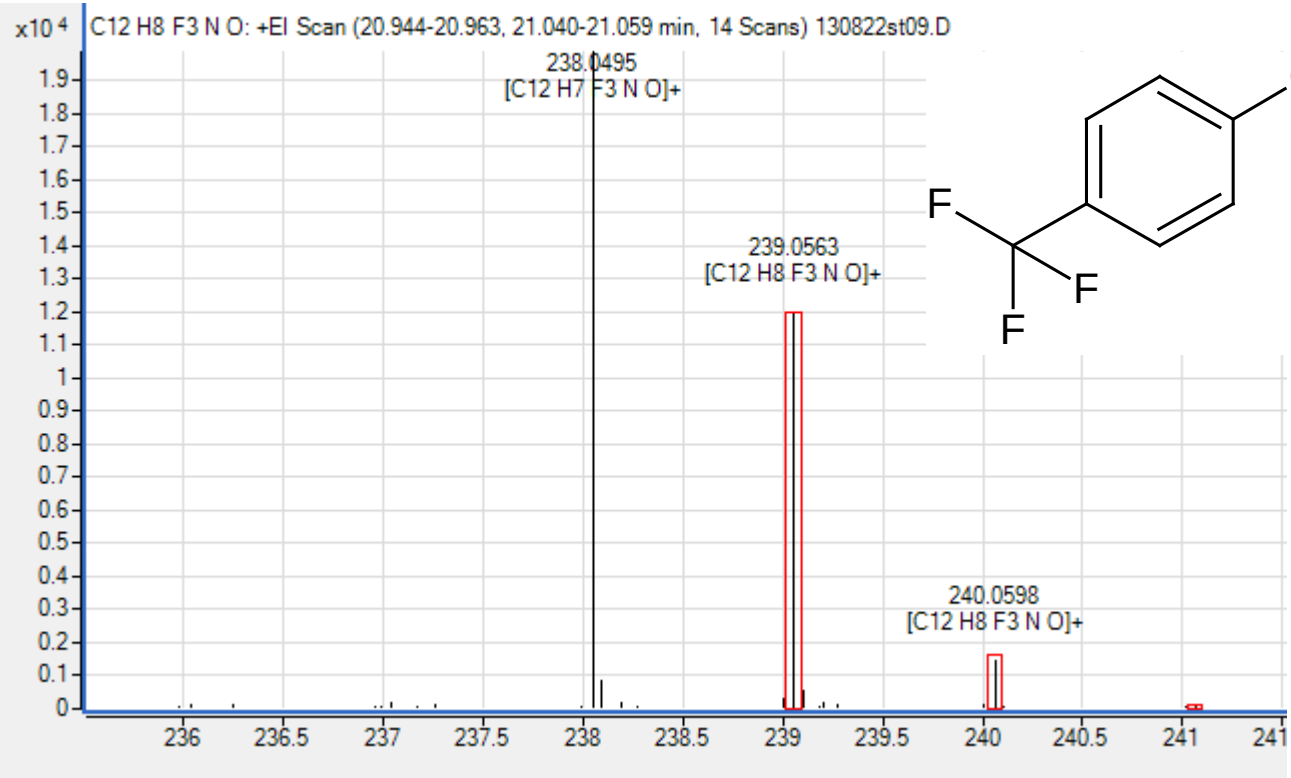


$C_{12}H_7F_3NO$, EVEN

$C_{12}H_8F_3NO$, ODD

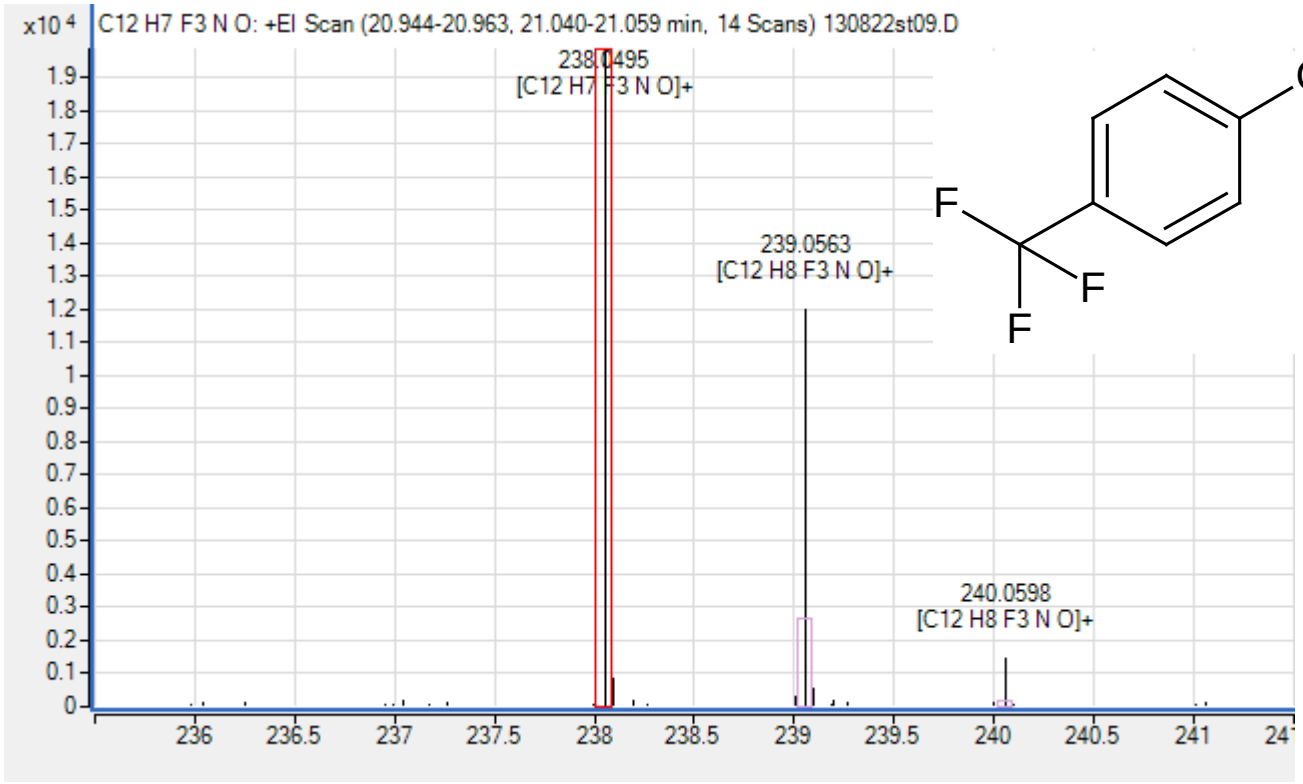
Picolinafen - overlapping fragments

$C_{12}H_8F_3NO$, ODD



Picolinafen - overlapping fragments

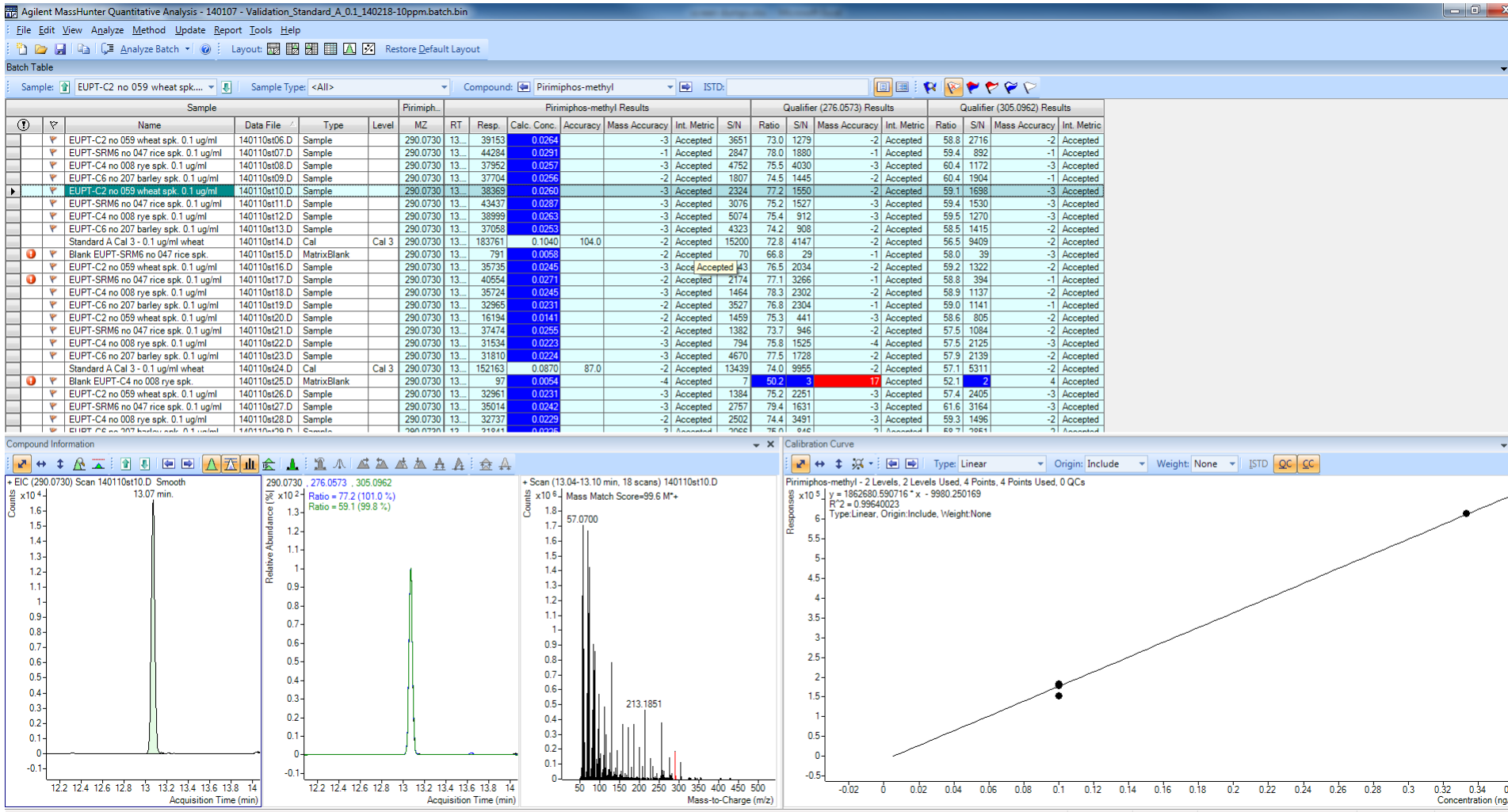
$C_{12}H_7F_3NO$, EVEN



Low score - poor isotop pattern

Show/Hide	Cpd	Label	Name	Formula	Score	Mass
☑	1	Cpd 1: Picolinafen	Picolinafen	C19 H12 F4 N2 O2	85.46	376.0863
☑	2	Cpd 2: Picolinafen F2	Picolinafen F2	C12 H7 F3 N O	54.51	238.05
☑	3	Cpd 3: Picolinafen F1	Picolinafen F1	C12 H8 F3 N O	93.43	239.0571

Quantitativ processing used qualitative

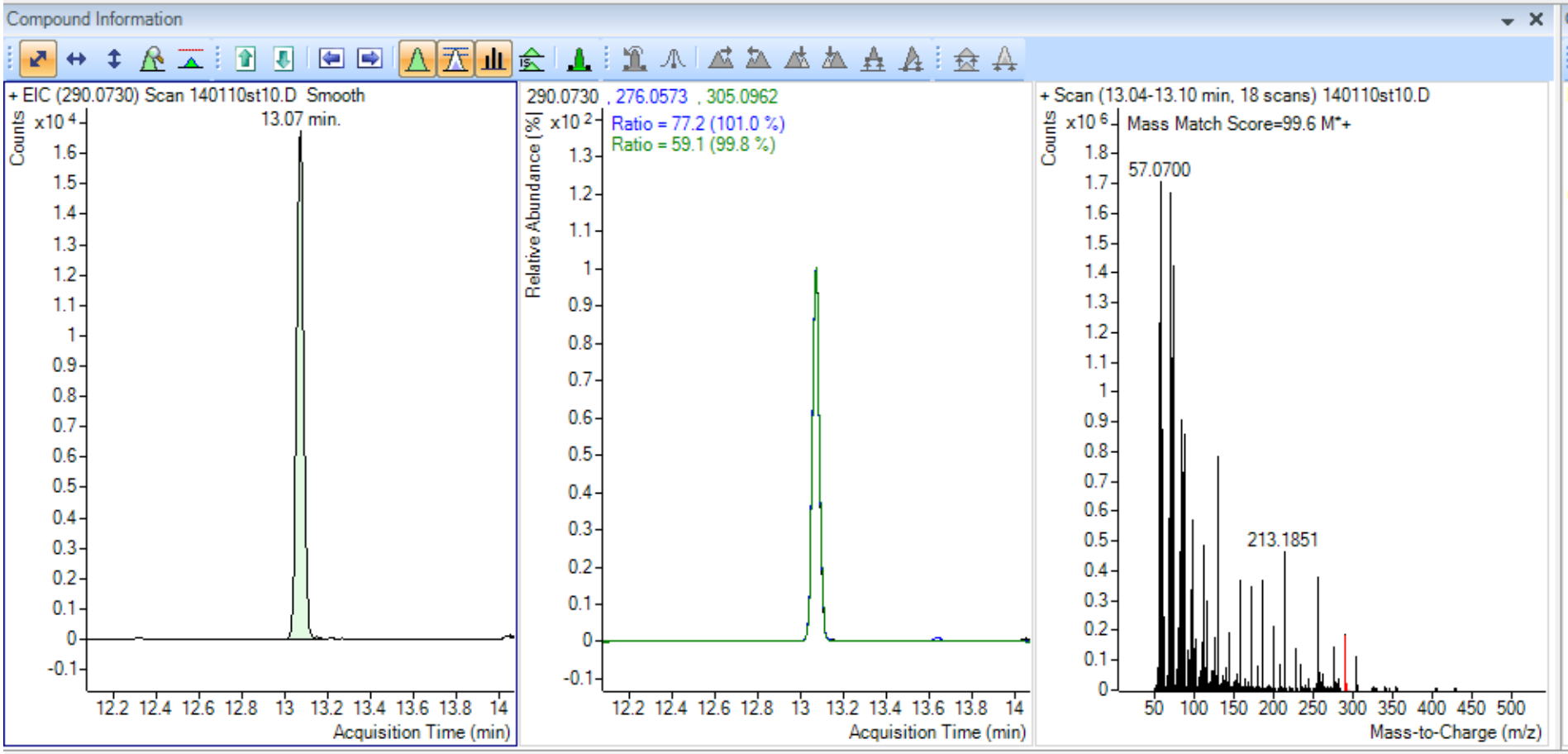


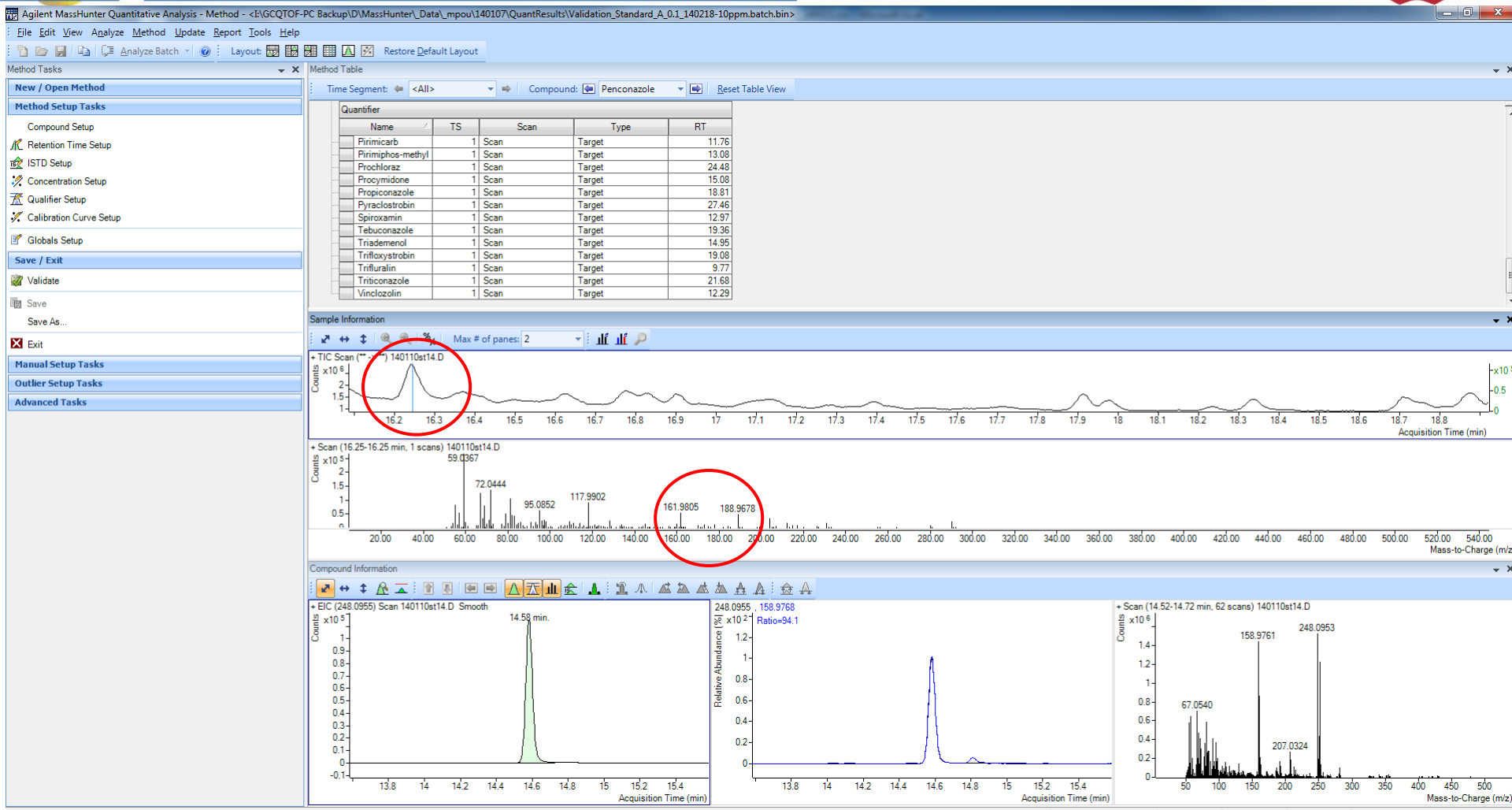
Quantitativ processing used qualitative

EUPT-C2 no 059 wheat spk.... | Sample Type: <All> | Compound: Pirimiphos-methyl | ISTD:

Sample				Pirimiph...	Pirimiphos-methyl Results							Qualifier (276.0573) Results				Qualifier (305.0962) Results			
Name	Data File	Type	Level	MZ	RT	Resp.	Calc. Conc.	Accuracy	Mass Accuracy	Int. Metric	S/N	Ratio	S/N	Mass Accuracy	Int. Metric	Ratio	S/N	Mass Accuracy	Int. Metric
EUPT-C2 no 059 wheat spk. 0.1 ug/ml	140110st06.D	Sample		290.0730	13....	39153	0.0264		-3	Accepted	3651	73.0	1279	-2	Accepted	58.8	2716	-2	Accepted
EUPT-SRM6 no 047 rice spk. 0.1 ug/ml	140110st07.D	Sample		290.0730	13....	44284	0.0291		-1	Accepted	2847	78.0	1880	-1	Accepted	59.4	892	-1	Accepted
EUPT-C4 no 008 rye spk. 0.1 ug/ml	140110st08.D	Sample		290.0730	13....	37952	0.0257		-3	Accepted	4752	75.5	4030	-3	Accepted	60.4	1172	-3	Accepted
EUPT-C6 no 207 barley spk. 0.1 ug/ml	140110st09.D	Sample		290.0730	13....	37704	0.0256		-2	Accepted	1807	74.5	1445	-2	Accepted	60.4	1904	-1	Accepted
EUPT-C2 no 059 wheat spk. 0.1 ug/ml	140110st10.D	Sample		290.0730	13....	38369	0.0260		-3	Accepted	2324	77.2	1550	-2	Accepted	59.1	1698	-3	Accepted
EUPT-SRM6 no 047 rice spk. 0.1 ug/ml	140110st11.D	Sample		290.0730	13....	43437	0.0287		-3	Accepted	3076	75.2	1527	-3	Accepted	59.4	1530	-3	Accepted
EUPT-C4 no 008 rye spk. 0.1 ug/ml	140110st12.D	Sample		290.0730	13....	38999	0.0263		-3	Accepted	5074	75.4	912	-3	Accepted	59.5	1270	-3	Accepted
EUPT-C6 no 207 barley spk. 0.1 ug/ml	140110st13.D	Sample		290.0730	13....	37058	0.0253		-3	Accepted	4323	74.2	908	-2	Accepted	58.5	1415	-2	Accepted
Standard A Cal 3 - 0.1 ug/ml wheat	140110st14.D	Cal	Cal 3	290.0730	13....	183761	0.1040	104.0	-2	Accepted	15200	72.8	4147	-2	Accepted	56.5	9409	-2	Accepted
Blank EUPT-SRM6 no 047 rice spk.	140110st15.D	MatrixBlank		290.0730	13....	791	0.0058		-2	Accepted	70	66.8	29	-1	Accepted	58.0	39	-3	Accepted
EUPT-C2 no 059 wheat spk. 0.1 ug/ml	140110st16.D	Sample		290.0730	13....	35735	0.0245		-3	Acce	Accepted	43	76.5	2034	-2	Accepted	59.2	1322	-2	Accepted
EUPT-SRM6 no 047 rice spk. 0.1 ug/ml	140110st17.D	Sample		290.0730	13....	40554	0.0271		-2	Accepted	2174	77.1	3266	-1	Accepted	58.8	394	-1	Accepted
EUPT-C4 no 008 rye spk. 0.1 ug/ml	140110st18.D	Sample		290.0730	13....	35724	0.0245		-3	Accepted	1464	78.3	2302	-2	Accepted	58.9	1137	-2	Accepted
EUPT-C6 no 207 barley spk. 0.1 ug/ml	140110st19.D	Sample		290.0730	13....	32965	0.0231		-2	Accepted	3527	76.8	2304	-1	Accepted	59.0	1141	-1	Accepted
EUPT-C2 no 059 wheat spk. 0.1 ug/ml	140110st20.D	Sample		290.0730	13....	16194	0.0141		-2	Accepted	1459	75.3	441	-3	Accepted	58.6	805	-2	Accepted
EUPT-SRM6 no 047 rice spk. 0.1 ug/ml	140110st21.D	Sample		290.0730	13....	37474	0.0255		-2	Accepted	1382	73.7	946	-2	Accepted	57.5	1084	-2	Accepted
EUPT-C4 no 008 rye spk. 0.1 ug/ml	140110st22.D	Sample		290.0730	13....	31534	0.0223		-3	Accepted	794	75.8	1525	-4	Accepted	57.5	2125	-3	Accepted
EUPT-C6 no 207 barley spk. 0.1 ug/ml	140110st23.D	Sample		290.0730	13....	31810	0.0224		-3	Accepted	4670	77.5	1728	-2	Accepted	57.9	2139	-2	Accepted
Standard A Cal 3 - 0.1 ug/ml wheat	140110st24.D	Cal	Cal 3	290.0730	13....	152163	0.0870	87.0	-2	Accepted	13439	74.0	9955	-2	Accepted	57.1	5311	-2	Accepted
Blank EUPT-C4 no 008 rye spk.	140110st25.D	MatrixBlank		290.0730	13....	97	0.0054		-4	Accepted	7	50.2	3	17	Accepted	52.1	2	4	Accepted
EUPT-C2 no 059 wheat spk. 0.1 ug/ml	140110st26.D	Sample		290.0730	13....	32961	0.0231		-3	Accepted	1384	75.2	2251	-3	Accepted	57.4	2405	-3	Accepted
EUPT-SRM6 no 047 rice spk. 0.1 ug/ml	140110st27.D	Sample		290.0730	13....	35014	0.0242		-3	Accepted	2757	79.4	1631	-3	Accepted	61.6	3164	-3	Accepted
EUPT-C4 no 008 rye spk. 0.1 ug/ml	140110st28.D	Sample		290.0730	13....	32737	0.0229		-2	Accepted	2502	74.4	3491	-3	Accepted	59.3	1496	-2	Accepted
EUPT-C6 no 207 barley spk. 0.1 ug/ml	140110st29.D	Sample		290.0730	13....	31841	0.0225		-2	Accepted	2066	75.0	846	-2	Accepted	58.7	2851	-2	Accepted

Quantitativ processing used qualitative

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Method Tasks
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Compound Setup
Retention Time Setup
ISTD Setup
Concentration Setup
Qualifier Setup
Calibration Curve Setup
Globals Setup
Save / Exit
Validate
Save
Save As...
Exit
Manual Setup Tasks
Outlier Setup Tasks
Advanced Tasks
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Reset Table View
Quantifier
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Pirimiphos-methyl 1 Scan Target 13.08
Prochloraz 1 Scan Target 24.48
Procymidone 1 Scan Target 15.08
Propiconazole 1 Scan Target 18.81
Pyraclostrobin 1 Scan Target 27.46
Spiroxamin 1 Scan Target 12.97
Tebuconazole 1 Scan Target 19.36
Triademenol 1 Scan Target 14.95
Trifloxystrobin 1 Scan Target 19.08
Trifluralin 1 Scan Target 9.77
Triticonazole 1 Scan Target 21.68
Vinclozolin 1 Scan Target 12.29
Sample Information
Max # of panes: 2
+ TIC Scan (** - **) 140110st14.D
Counts
Acquisition Time (min)
+ Scan (16.25-16.25 min, 1 scans) 140110st14.D
59.0367
72.0444
95.0852
117.9902
161.9805
188.9678
Mass-to-Charge (m/z)
Compound Information
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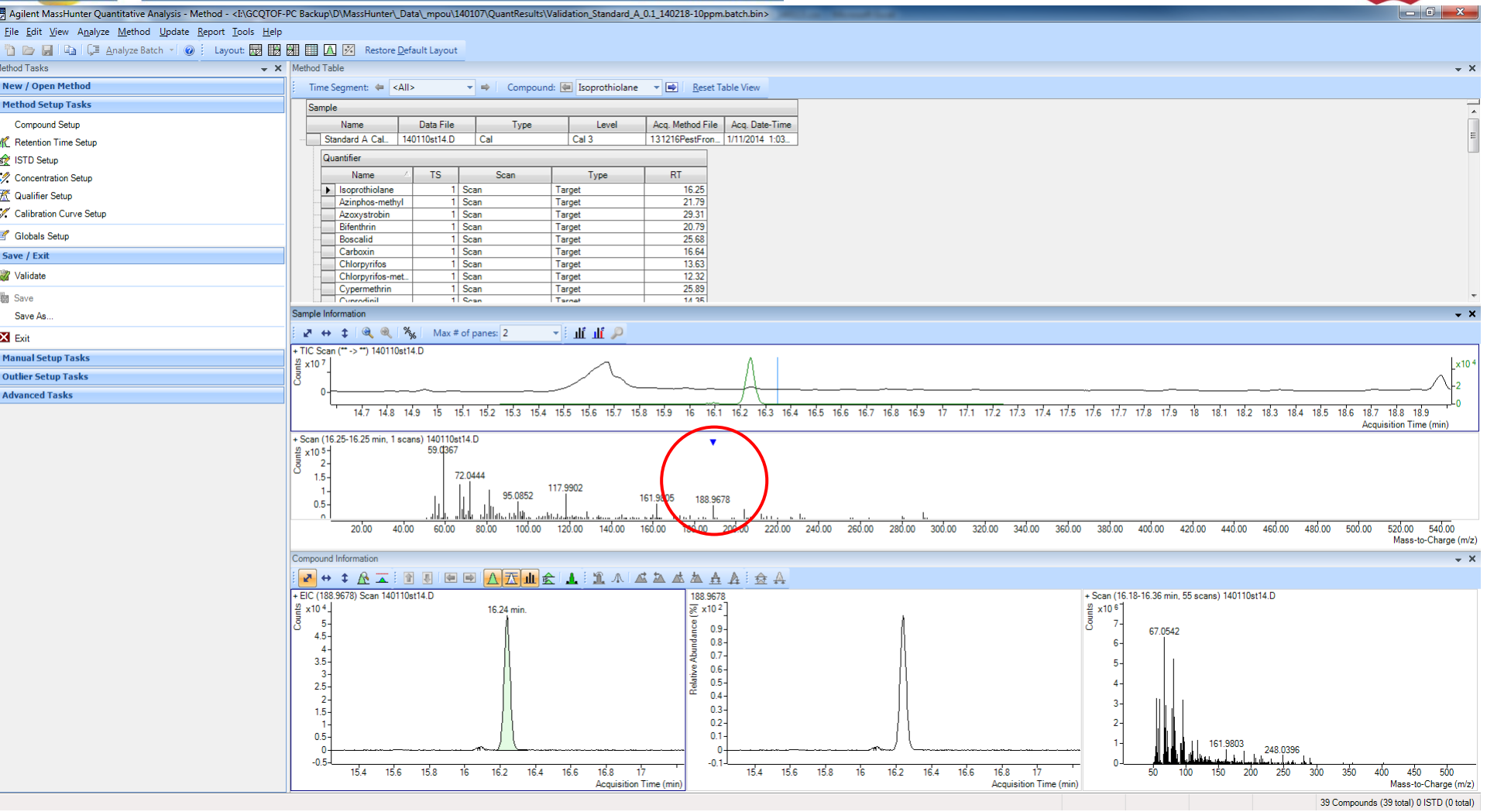
14.58 min.
248.0955 , 158.9768
Ratio=94.1
Relative Abundance (%)
+ Scan (14.52-14.72 min, 62 scans) 140110st14.D
158.9761
248.0953
67.0540
207.0324
X 178.2459 Y 62162.828
38 Compounds (38 total) 0 ISTD (0 total)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Compound Setup
Retention Time Setup
ISTD Setup
Concentration Setup
Qualifier Setup
Calibration Curve Setup
Globals Setup
Save / Exit
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Exit
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Outlier Setup Tasks
Advanced Tasks
Method Table
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Compound: Isoprothiolane
Reset Table View
Sample
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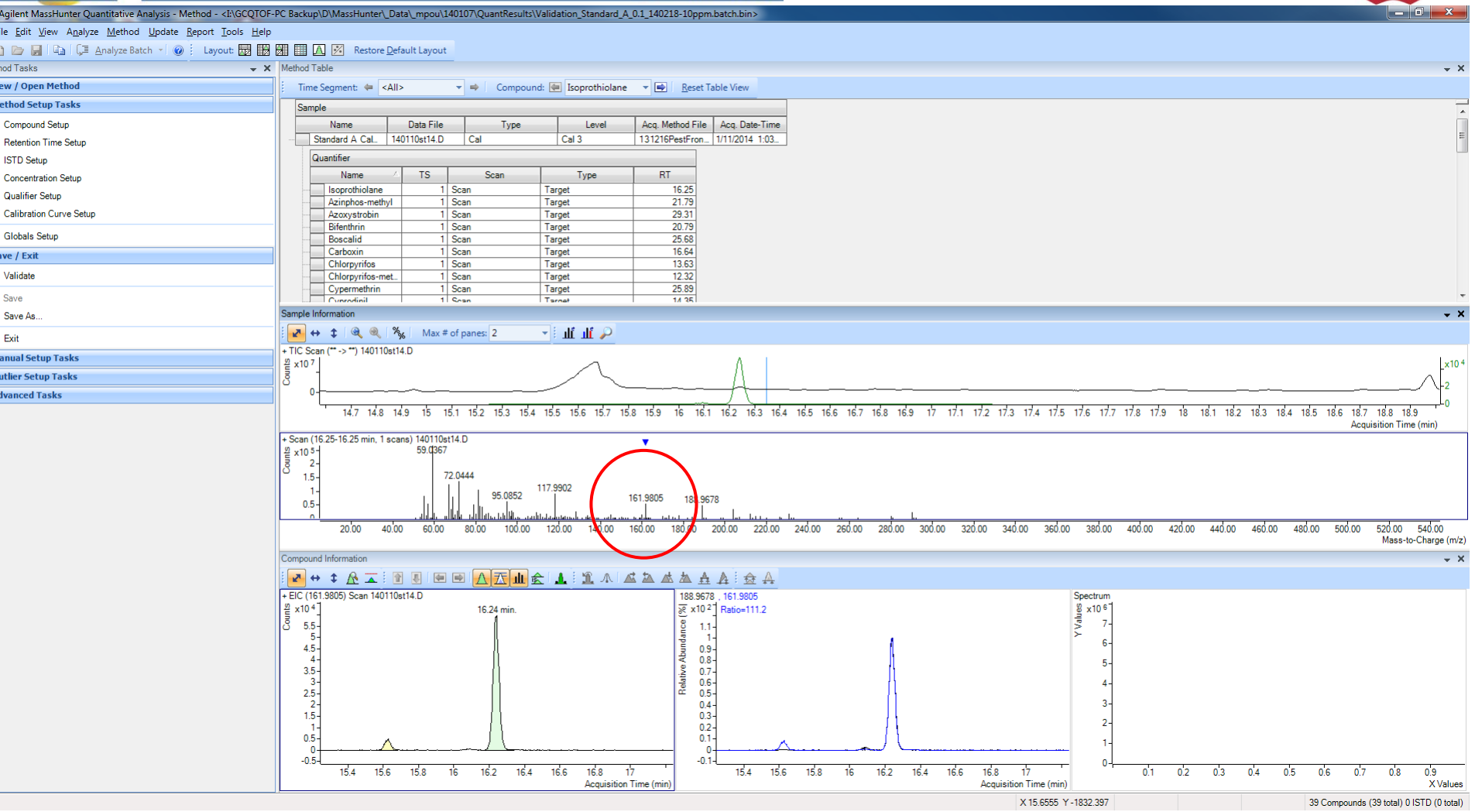
Quantifier
Name | TS | Scan | Type | RT
Isoprothiolane | 1 | Scan | Target | 16.25
Azinphos-methyl | 1 | Scan | Target | 21.79
Azoxystrobin | 1 | Scan | Target | 29.31
Bifenthrin | 1 | Scan | Target | 20.79
Boscalid | 1 | Scan | Target | 25.68
Carboxin | 1 | Scan | Target | 16.64
Chlorpyrifos | 1 | Scan | Target | 13.63
Chlorpyrifos-met.. | 1 | Scan | Target | 12.32
Cypermethrin | 1 | Scan | Target | 25.89
Sample Information
Max # of panes: 2
+ TIC Scan (** -> **) 140110st14.D
Counts
Acquisition Time (min)
+ Scan (16.25-16.25 min, 1 scans) 140110st14.D
59.0367
72.0444
95.0852
117.9902
161.9805
188.9678
Mass-to-Charge (m/z)
Compound Information
+ EIC (188.9678) Scan 140110st14.D
16.24 min.
Acquisition Time (min)
188.9678
Relative Abundance (%)
Acquisition Time (min)
+ Scan (16.18-16.36 min, 55 scans) 140110st14.D
67.0542
161.9803
248.0396
Mass-to-Charge (m/z)
39 Compounds (39 total) 0 ISTD (0 total)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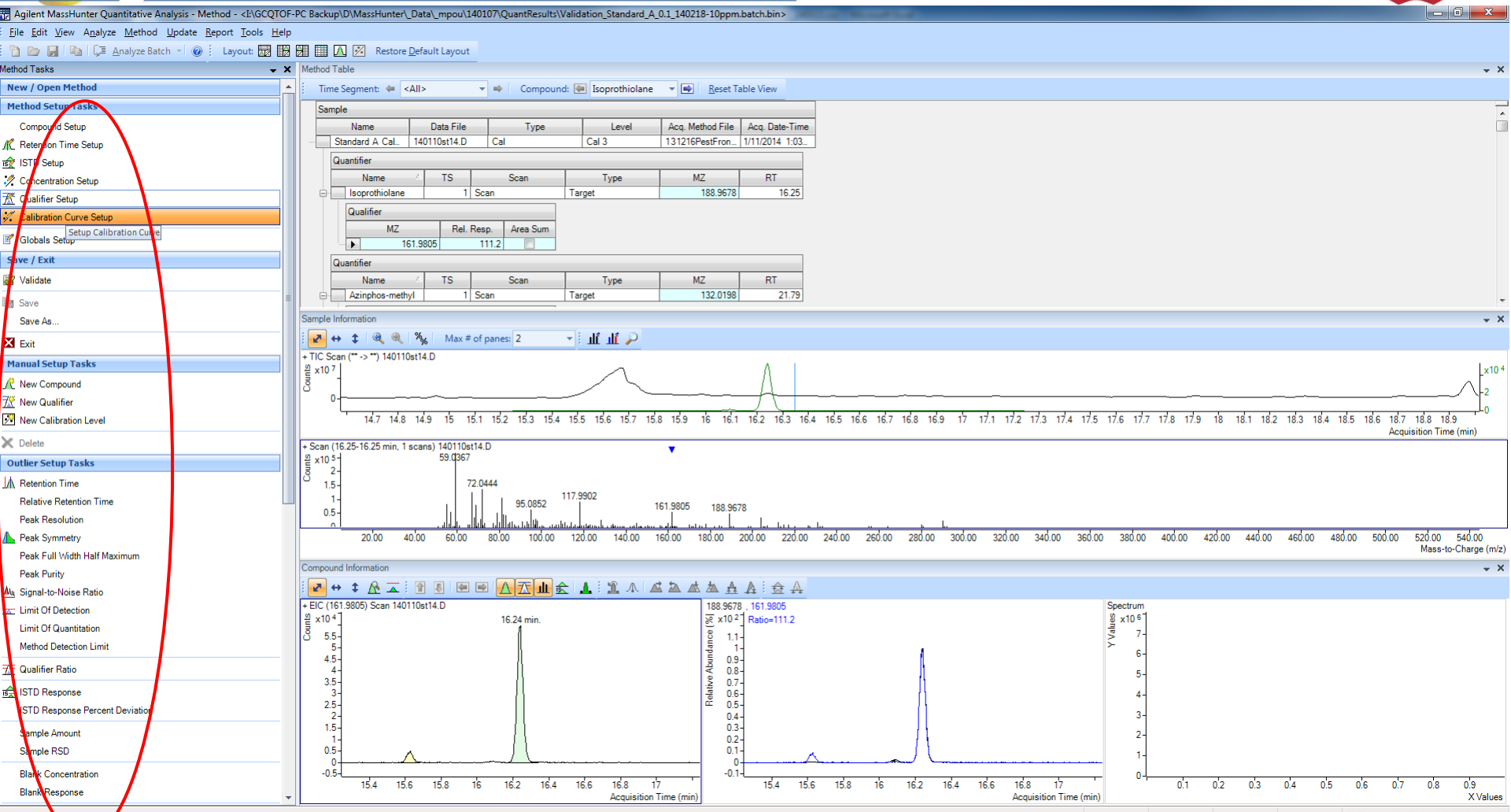
Save As...
Exit
Manual Setup Tasks
Outlier Setup Tasks
Advanced Tasks
Method Table
Time Segment: <All>
Compound: Isoprothiolane
Reset Table View
Sample
Name | Data File | Type | Level | Acq. Method File | Acq. Date-Time
Standard A Cal.. | 140110st14.D | Cal | Cal 3 | 131216PestFron.. | 1/11/2014 1:03..
Quantifier
Name | TS | Scan | Type | RT
Isoprothiolane | 1 | Scan | Target | 16.25
Azinphos-methyl | 1 | Scan | Target | 21.79
Azoxystrobin | 1 | Scan | Target | 29.31
Bifenthrin | 1 | Scan | Target | 20.79
Boscalid | 1 | Scan | Target | 25.68
Carboxin | 1 | Scan | Target | 16.64
Chlorpyrifos | 1 | Scan | Target | 13.63
Chlorpyrifos-met.. | 1 | Scan | Target | 12.32
Cypermethrin | 1 | Scan | Target | 25.89
Sample Information
Max # of panes: 2
+ TIC Scan (** -> **) 140110st14.D
Counts
Acquisition Time (min)
+ Scan (16.25-16.25 min, 1 scans) 140110st14.D
59.0367
72.0444
95.0852
117.9902
161.9805
188.9678
Mass-to-Charge (m/z)
Compound Information
+ EIC (161.9805) Scan 140110st14.D
16.24 min.
Acquisition Time (min)
188.9678 , 161.9805
Ratio=111.2
Relative Abundance (%)
Acquisition Time (min)
Spectrum
Y Values
X Values
X 15.6555 Y -1832.397
39 Compounds (39 total) 0 ISTD (0 total)

C7H9O2S2	16.36	189.0044	Isoprothiolane F1
C9H11O3S2	16.36	231.015	Isoprothiolane F2
C6H4O4S2	16.36	203.9551	Isoprothiolane F3
C5H6O2S2	16.36	161.9809	Isoprothiolane F4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File Edit View Analyze Method Update Report Tools Help
Analyze Batch
Layout:
Restore Default Layout
Method Tasks
New / Open Method
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Retention Time Setup
ISTD Setup
Concentration Setup
Qualifier Setup
Calibration Curve Setup
Setup Calibration Curve
Globals Setup
Save / Exit
Validate
Save
Save As...
Exit
Manual Setup Tasks
New Compound
New Qualifier
New Calibration Level
Delete
Outlier Setup Tasks
Retention Time
Relative Retention Time
Peak Resolution
Peak Symmetry
Peak Full Width Half Maximum
Peak Purity
Signal-to-Noise Ratio
Limit Of Detection
Limit Of Quantitation
Method Detection Limit
Qualifier Ratio
ISTD Response
ISTD Response Percent Deviation
Sample Amount
Sample RSD
Blank Concentration
Blank Response
Method Table
Time Segment: <All>
Compound: Isoprothiolane
Reset Table View
Sample
Name Data File Type Level Acq. Method File Acq. Date-Time
Standard A Cal.. 140110st14.D Cal Cal 3 131216PestFron.. 1/11/2014 1:03..
Quantifier
Name TS Scan Type MZ RT
Isoprothiolane 1 Scan Target 188.9678 16.25
Qualifier
MZ Rel. Resp. Area Sum
161.9805 111.2
Quantifier
Name TS Scan Type MZ RT
Azinphos-methyl 1 Scan Target 132.0198 21.79
Sample Information
Max # of panes: 2
+ TIC Scan (** -> **) 140110st14.D
Counts
Acquisition Time (min)
+ Scan (16.25-16.25 min, 1 scans) 140110st14.D
59.0367
72.0444
95.0852
117.9902
161.9805
188.9678
Mass-to-Charge (m/z)
Compound Information
+ EIC (161.9805) Scan 140110st14.D
16.24 min.
Acquisition Time (min)
188.9678 , 161.9805
Ratio=111.2
Relative Abundance (%)
Acquisition Time (min)
Spectrum
Y Values
X Values
39 Compounds (39 total) 0 ISTD (0 total)

Disposition

- LC-QTOF screening - MassHunter Qualitative
- LC-QTOF - Validation
- GC-QTOF screening - MassHunter Quantitative
- GC-QTOF - Validation

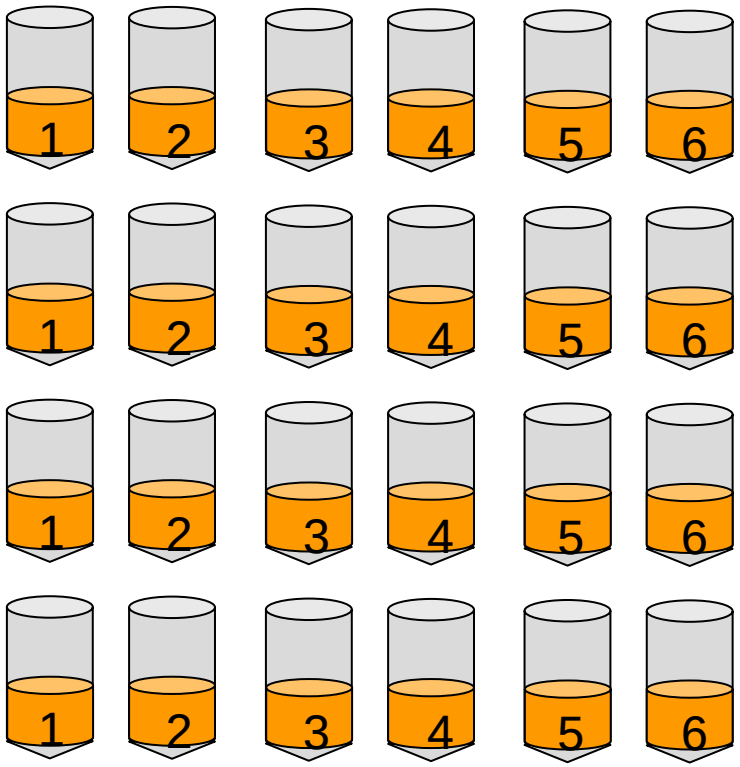
Validation of screening method

- QuEChERS method combined with a GC-QTOF from Agilent
- The method was validated in cereals two spike levels, 0.01 and 0.05 mg/kg

- Purpose:
  - to be familiar with the instrument, be able to analyse and process data, work with well known pesticides.
  - Use the Sanco approach

Design of validation

- 6 samples of four different types of cereal samples blank EUPT blank test material



Wheat

Rye

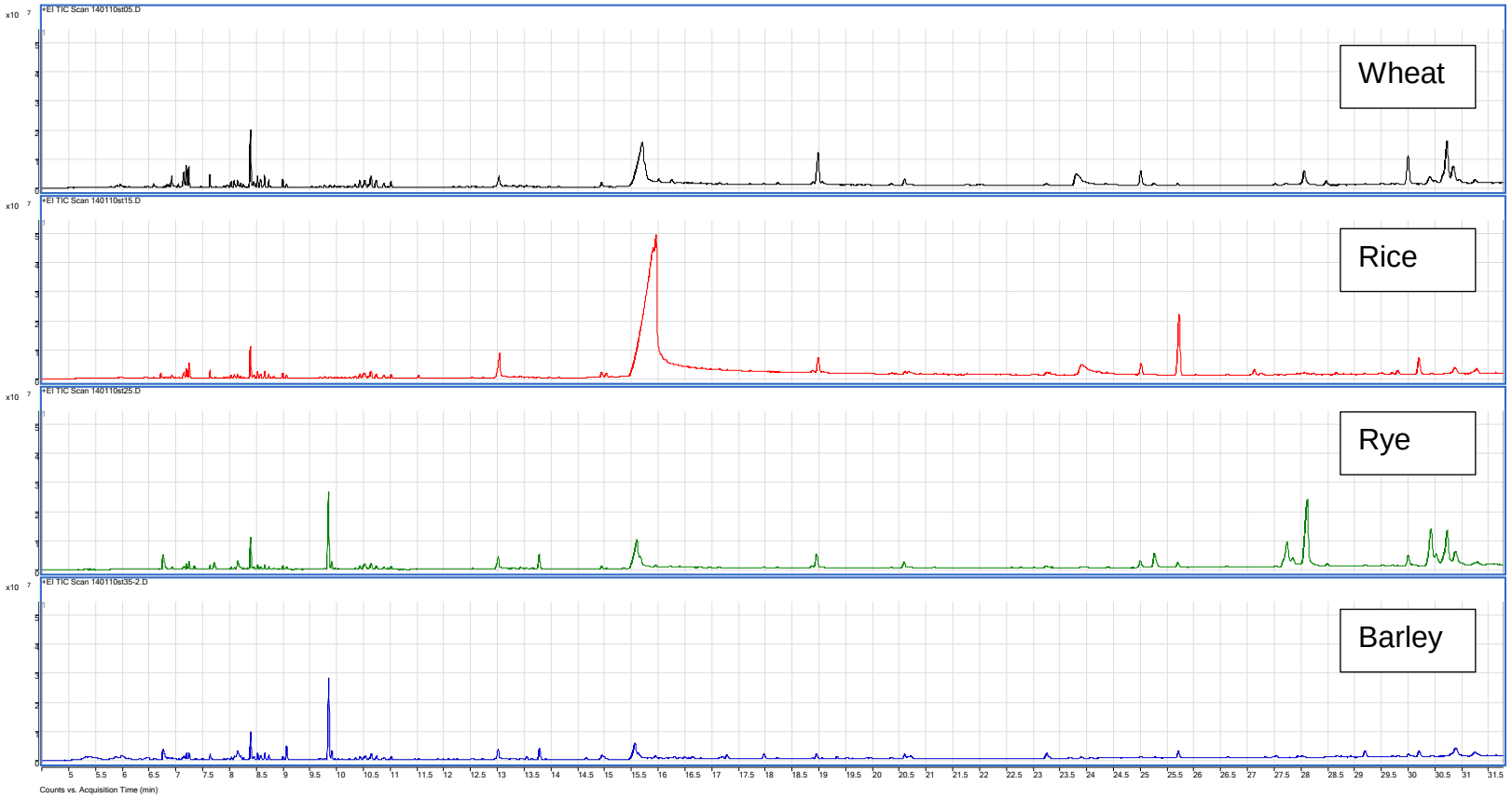
Rice

Barley

Design of validation

- Spiked at 0.01 and 0.05 mg/kg
  - 5 standard mixtures called (A, B, C, D and E), containing 356 different compounds altogether.
  - Not all of them can be detected on GC.
- The 24 spiked samples plus 4 blank cereal samples and 4 EUPT tests materials were extracted and cleaned up.
- Only 38 of the compounds have currently been evaluated.
- According to SANCO at least 95% of the samples should be detected (a false-negative rate of 5% is accepted).
  - This means that only 1 out of 20 spiked samples are allowed to be non-detected.

Matrix



Screening criteria

- DTU:
  - Retention time (RT): ± 0.1 min
  - Mass accuracy:
    - 5 ppm for at least 1 fragment ion or
    - 10 ppm for at least 2 fragment ions
  - Signal to noise ratio (S/N): 6

- Sanco:
  - Retention time (RT): ±0.2 min
  - Signal to noise ratio (S/N): 3
  - ≥ 2 diagnostic ions, preferably including the (quasi) molecular ion;
  - mass accuracy < 5 ppm; at least one fragment ion

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.075	39153	0.0264		3651.28	-3.2	28584	1278.97	-1.8	23008	2716.33	-2
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.072	38369	0.026		2324.2	-3	29625	1550.49	-2.4	22663	1698.24	-3.3
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	35735	0.0245		1343.28	-2.8	27321	2033.78	-2.3	21155	1322.36	-1.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	16194	0.0141		1458.87	-2.2	12195	441.2	-3	9489	804.92	-2.4
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.07	32961	0.0231		1383.74	-3.1	24785	2251.17	-3.3	18905	2404.96	-2.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.074	32121	0.0226		4369.78	-2.3	24485	3114.78	-2	18770	1880.56	-1.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.074	37952	0.0257		4751.72	-2.9	28667	4029.74	-2.5	22921	1172.1	-2.8
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.071	38999	0.0263		5074.47	-3	29420	911.76	-3.1	23221	1270.08	-3.2
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	35724	0.0245		1463.7	-3	27982	2302.36	-1.8	21053	1137	-2.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	31534	0.0223		794.35	-3.5	23890	1524.84	-3.9	18126	2125.3	-2.5
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.076	32737	0.0229		2501.79	-1.6	24351	3491.24	-3	19416	1495.89	-1.9
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	33346	0.0233		2319.11	-2.1	24888	2354.36	-2.1	20084	1222.18	-1.8
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37704	0.0256		1807.3	-1.8	28091	1445.26	-1.8	22763	1903.57	-1.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37058	0.0253		4323.19	-2.8	27506	908.04	-1.8	21677	1415.06	-2.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.075	32965	0.0231		3526.99	-1.5	25306	2304.1	-1.4	19463	1141.04	-0.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.072	31810	0.0224		4670.4	-2.9	24662	1727.86	-1.5	18415	2138.92	-2.3
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.076	31841	0.0225		2065.66	-2.6	23886	846.34	-2	18677	2851.06	-1.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	20225	0.0162		1979.85	-3.3	14795	773.9	-2.6	11817	834.86	-2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	44284	0.0291		2847.2	-1.4	34532	1879.5	-1.3	26325	891.77	-0.8
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	43437	0.0287		3075.92	-3.2	32685	1526.54	-3.1	25795	1530.19	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	40554	0.0271		2173.58	-1.7	31286	3265.97	-1	23856	394.38	-1.2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	37474	0.0255		1382.28	-2.3	27608	945.67	-2	21555	1084.01	-1.7
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	35014	0.0242		2757.14	-2.7	27814	1631.3	-2.9	21560	3163.66	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.077	37104	0.0253		2806.35	-1.6	28705	1403.56	-1.3	22501	2165.39	-0.2
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6854	0.009		683.48	2.2	5144	575.62	2.4	3890	380.68	3.7
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.07	6354	0.0088		722.02	-2.8	4926	227.87	-3.5	3927	367.36	-3.5
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6367	0.0088		765.17	-2.2	5060	200.18	-1.3	3772	252.95	0.6
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.072	6378	0.0088		536.46	-5.4	5183	380.78	-2.2	4120	493.45	-3.3
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6467	0.0088		977.58	-3	5293	595.49	-4.6	4250	464.58	-3.4
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6520	0.0089		568.07	-3.9	5259	610.78	-3.7	4041	475.94	-1.9
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.071	6646	0.0089		947.45	4.2	5286	456.55	3.2	3940	554.06	5.8
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.07	6172	0.0087		650.8	-1	4872	285.24	-2.9	3674	339.2	-1.2
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6930	0.0091		365.33	-4.4	5509	367.1	-3.9	4004	198.03	-3.6
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6828	0.009		983.29	-3.5	5399	486.36	-4.8	4115	315.36	-4.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6420	0.0088		433.69	-4.7	5223	462.55	-4.4	3809	443.77	-3.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6450	0.0088		868.4	-4.1	5035	411.47	-2.4	3625	373.62	-4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.074	6841	0.009		370.19	3.9	5545	146.59	4.6	3964	381.75	5.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6707	0.009		746.95	-2.7	5335	484.64	-1.9	3984	195.52	-2.6
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.073	6509	0.0089		1103.73	-4.6	5089	471.98	-3.6	4150	511.74	-2.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6953	0.0091		511.84	-2.3	5442	723.35	-4.4	3813	551.36	-3.4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.072	6711	0.009		548.35	-6.7	4969	530.79	-2.7	3827	176.45	-4.5
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.076	6531	0.0089		374.2	-4.7	4964	201.78	-5.4	3972	350.87	-5.2
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.075	9614	0.0105		480.15	2.7	7403	544.11	3.5	5949	368.61	2.4
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8835	0.0101		606.26	-2.8	7033	347.19	-2	5247	684.4	-3
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8821	0.0101		684.85	-3.2	6808	452.2	-1.6	5851	387.83	-1.9
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9276	0.0103		972.69	-3.6	6993	326.86	-4	5330	376.24	-1.6
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9958	0.0107		890.19	-3.8	7746	331.17	-3.4	6121	368.32	-3.1
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8784	0.0101		260.19	-2.6	7154	611.4	-2.7	5606	373.43	-1.9

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.075	39153	0.0264		3651.28	-3.2	28584	1278.97	-1.8	23008	2716.33	-2
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.072	38369	0.026		2324.2	-3	29625	1550.49	-2.4	22663	1698.24	-3.3
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	35735	0.0245		1343.28	-2.8	27321	2033.78	-2.3	21155	1322.36	-1.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	16194	0.0141		1458.87	-2.2	12195	441.2	-3	9489	804.92	-2.4
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.07	32961	0.0231		1383.74	-3.1	24785	2251.17	-3.3	18905	2404.96	-2.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.074	32121	0.0226		4369.78	-2.3	24485	3114.78	-2	18770	1880.56	-1.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.074	37952	0.0257		4751.72	-2.9	28667	4029.74	-2.5	22921	1172.1	-2.8
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.071	38999	0.0263		5074.47	-3	29420	911.76	-3.1	23221	1270.08	-3.2
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	35724	0.0245		1463.7	-3	27982	2302.36	-1.8	21053	1137	-2.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	31534	0.0223		794.35	-3.5	23890	1524.84	-3.9	18126	2125.3	-2.5
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.076	32737	0.0229		2501.79	-1.6	24351	3491.24	-3	19416	1495.89	-1.9
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	33346	0.0233		2319.11	-2.1	24888	2354.36	-2.1	20084	1222.18	-1.8
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37704	0.0256		1807.3	-1.8	28091	1445.26	-1.8	22763	1903.57	-1.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37058	0.0253		4323.19	-2.8	27506	908.04	-1.8	21677	1415.06	-2.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.075	32965	0.0231		3526.99	-1.5	25306	2304.1	-1.4	19463	1141.04	-0.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.072	31810	0.0224		4670.4	-2.9	24662	1727.86	-1.5	18415	2138.92	-2.3
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.076	31841	0.0225		2065.66	-2.6	23886	846.34	-2	18677	2851.06	-1.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	20225	0.0162		1979.85	-3.3	14795	773.9	-2.6	11817	834.86	-2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	44284	0.0291		2847.2	-1.4	34532	1879.5	-1.3	26325	891.77	-0.8
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	43437	0.0287		3075.92	-3.2	32685	1526.54	-3.1	25795	1530.19	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	40554	0.0271		2173.58	-1.7	31286	3265.97	-1	23856	394.38	-1.2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	37474	0.0255		1382.28	-2.3	27608	945.67	-2	21555	1084.01	-1.7
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	35014	0.0242		2757.14	-2.7	27814	1631.3	-2.9	21560	3163.66	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.077	37104	0.0253		2806.35	-1.6	28705	1403.56	-1.3	22501	2165.39	-0.2
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6854	0.009		683.48	2.2	5144	575.62	2.4	3890	380.68	3.7
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.07	6354	0.0088		722.02	-2.8	4926	227.87	-3.5	3927	367.36	-3.5
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6367	0.0088		765.17	-2.2	5060	200.18	-1.3	3772	252.95	0.6
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.072	6378	0.0088		536.46	-5.4	5183	380.78	-2.2	4120	493.45	-3.3
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6467	0.0088		977.58	-3	5293	595.49	-4.6	4250	464.58	-3.4
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6520	0.0089		568.07	-3.9	5259	610.78	-3.7	4041	475.94	-1.9
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.071	6646	0.0089		947.45	4.2	5286	456.55	3.2	3940	554.06	5.8
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.07	6172	0.0087		650.8	-1	4872	285.24	-2.9	3674	339.2	-1.2
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6930	0.0091		365.33	-4.4	5509	367.1	-3.9	4004	198.03	-3.6
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6828	0.009		983.29	-3.5	5399	486.36	-4.8	4115	315.36	-4.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6420	0.0088		433.69	-4.7	5223	462.55	-4.4	3809	443.77	-3.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6450	0.0088		868.4	-4.1	5035	411.47	-2.4	3625	373.62	-4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.074	6841	0.009		370.19	3.9	5545	146.59	4.6	3964	381.75	5.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6707	0.009		746.95	-2.7	5335	484.64	-1.9	3984	195.52	-2.6
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.073	6509	0.0089		1103.73	-4.6	5089	471.98	-3.6	4150	511.74	-2.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6953	0.0091		511.84	-2.3	5442	723.35	-4.4	3813	551.36	-3.4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.072	6711	0.009		548.35	-6.7	4969	530.79	-2.7	3827	176.45	-4.5
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.076	6531	0.0089		374.2	-4.7	4964	201.78	-5.4	3972	350.87	-5.2
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.075	9614	0.0105		480.15	2.7	7403	544.11	3.5	5949	368.61	2.4
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8835	0.0101		606.26	-2.8	7033	347.19	-2	5247	684.4	-3
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8821	0.0101		684.85	-3.2	6808	452.2	-1.6	5851	387.83	-1.9
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9276	0.0103		972.69	-3.6	6993	326.86	-4	5330	376.24	-1.6
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9958	0.0107		890.19	-3.8	7746	331.17	-3.4	6121	368.32	-3.1
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8784	0.0101		260.19	-2.6	7154	611.4	-2.7	5606	373.43	-1.9

Pirimiphos-methyl Results

							Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.075	39153	0.0264		3651.28	-3.2	28584	1278.97	-1.8	23008	2716.33	-2
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.072	38369	0.026		2324.2	-3	29625	1550.49	-2.4	22663	1698.24	-3.3
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	35735	0.0245		1343.28	-2.8	27321	2033.78	-2.3	21155	1322.36	-1.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	16194	0.0141		1458.87	-2.2	12195	441.2	-3	9489	804.92	-2.4
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.07	32961	0.0231		1383.74	-3.1	24785	2251.17	-3.3	18905	2404.96	-2.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.074	32121	0.0226		4369.78	-2.3	24485	3114.78	-2	18770	1880.56	-1.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.074	37952	0.0257		4751.72	-2.9	28667	4029.74	-2.5	22921	1172.1	-2.8
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.071	38999	0.0263		5074.47	-3	29420	911.76	-3.1	23221	1270.08	-3.2
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	35724	0.0245		1463.7	-3	27982	2302.36	-1.8	21053	1137	-2.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	31534	0.0223		794.35	-3.5	23890	1524.84	-3.9	18126	2125.3	-2.5
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.076	32737	0.0229		2501.79	-1.6	24351	3491.24	-3	19416	1495.89	-1.9
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	33346	0.0233		2319.11	-2.1	24888	2354.36	-2.1	20084	1222.18	-1.8
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37704	0.0256		1807.3	-1.8	28091	1445.26	-1.8	22763	1903.57	-1.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37058	0.0253		4323.19	-2.8	27506	908.04	-1.8	21677	1415.06	-2.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.075	32965	0.0231		3526.99	-1.5	25306	2304.1	-1.4	19463	1141.04	-0.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.072	31810	0.0224		4670.4	-2.9	24662	1727.86	-1.5	18415	2138.92	-2.3
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.076	31841	0.0225		2065.66	-2.6	23886	846.34	-2	18677	2851.06	-1.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	20225	0.0162		1979.85	-3.3	14795	773.9	-2.6	11817	834.86	-2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	44284	0.0291		2847.2	-1.4	34532	1879.5	-1.3	26325	891.77	-0.8
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	43437	0.0287		3075.92	-3.2	32685	1526.54	-3.1	25795	1530.19	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	40554	0.0271		2173.58	-1.7	31286	3265.97	-1	23856	394.38	-1.2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	37474	0.0255		1382.28	-2.3	27608	945.67	-2	21555	1084.01	-1.7
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	35014	0.0242		2757.14	-2.7	27814	1631.3	-2.9	21560	3163.66	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.077	37104	0.0253		2806.35	-1.6	28705	1403.56	-1.3	22501	2165.39	-0.2
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6854	0.009		683.48	2.2	5144	575.62	2.4	3890	380.68	3.7
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.07	6354	0.0088		722.02	-2.8	4926	227.87	-3.5	3927	367.36	-3.5
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6367	0.0088		765.17	-2.2	5060	200.18	-1.3	3772	252.95	0.6
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.072	6378	0.0088		536.46	-5.4	5183	380.78	-2.2	4120	493.45	-3.3
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6467	0.0088		977.58	-3	5293	595.49	-4.6	4250	464.58	-3.4
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6520	0.0089		568.07	-3.9	5259	610.78	-3.7	4041	475.94	-1.9
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.071	6646	0.0089		947.45	4.2	5286	456.55	3.2	3940	554.06	5.8
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.07	6172	0.0087		650.8	-1	4872	285.24	-2.9	3674	339.2	-1.2
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6930	0.0091		365.33	-4.4	5509	367.1	-3.9	4004	198.03	-3.6
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6828	0.009		983.29	-3.5	5399	486.36	-4.8	4115	315.36	-4.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6420	0.0088		433.69	-4.7	5223	462.55	-4.4	3809	443.77	-3.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6450	0.0088		868.4	-4.1	5035	411.47	-2.4	3625	373.62	-4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.074	6841	0.009		370.19	3.9	5545	146.59	4.6	3964	381.75	5.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6707	0.009		746.95	-2.7	5335	484.64	-1.9	3984	195.52	-2.6
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.073	6509	0.0089		1103.73	-4.6	5089	471.98	-3.6	4150	511.74	-2.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6953	0.0091		511.84	-2.3	5442	723.35	-4.4	3813	551.36	-3.4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.072	6711	0.009		548.35	-6.7	4969	530.79	-2.7	3827	176.45	-4.5
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.076	6531	0.0089		374.2	-4.7	4964	201.78	-5.4	3972	350.87	-5.2
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.075	9614	0.0105		480.15	2.7	7403	544.11	3.5	5949	368.61	2.4
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8835	0.0101		606.26	-2.8	7033	347.19	-2	5247	684.4	-3
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8821	0.0101		684.85	-3.2	6808	452.2	-1.6	5851	387.83	-1.9
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9276	0.0103		972.69	-3.6	6993	326.86	-4	5330	376.24	-1.6
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9958	0.0107		890.19	-3.8	7746	331.17	-3.4	6121	368.32	-3.1
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8784	0.0101		260.19	-2.6	7154	611.4	-2.7	5606	373.43	-1.9

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.075	39153	0.0264		3651.28	-3.2	28584	1278.97	-1.8	23008	2716.33	-2
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.072	38369	0.026		2324.2	-3	29625	1550.49	-2.4	22663	1698.24	-3.3
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	35735	0.0245		1343.28	-2.8	27321	2033.78	-2.3	21155	1322.36	-1.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.073	16194	0.0141		1458.87	-2.2	12195	441.2	-3	9489	804.92	-2.4
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.07	32961	0.0231		1383.74	-3.1	24785	2251.17	-3.3	18905	2404.96	-2.6
EUPT-C2 no 059 wheat spk. 0.05 ug/ml	13.074	32121	0.0226		4369.78	-2.3	24485	3114.78	-2	18770	1880.56	-1.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.074	37952	0.0257		4751.72	-2.9	28667	4029.74	-2.5	22921	1172.1	-2.8
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.071	38999	0.0263		5074.47	-3	29420	911.76	-3.1	23221	1270.08	-3.2
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	35724	0.0245		1463.7	-3	27982	2302.36	-1.8	21053	1137	-2.3
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	31534	0.0223		794.35	-3.5	23890	1524.84	-3.9	18126	2125.3	-2.5
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.076	32737	0.0229		2501.79	-1.6	24351	3491.24	-3	19416	1495.89	-1.9
EUPT-C4 no 008 rye spk. 0.05 ug/ml	13.073	33346	0.0233		2319.11	-2.1	24888	2354.36	-2.1	20084	1222.18	-1.8
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37704	0.0256		1807.3	-1.8	28091	1445.26	-1.8	22763	1903.57	-1.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	37058	0.0253		4323.19	-2.8	27506	908.04	-1.8	21677	1415.06	-2.5
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.075	32965	0.0231		3526.99	-1.5	25306	2304.1	-1.4	19463	1141.04	-0.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.072	31810	0.0224		4670.4	-2.9	24662	1727.86	-1.5	18415	2138.92	-2.3
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.076	31841	0.0225		2065.66	-2.6	23886	846.34	-2	18677	2851.06	-1.6
EUPT-C6 no 207 barley spk. 0.05 ug/ml	13.073	20225	0.0162		1979.85	-3.3	14795	773.9	-2.6	11817	834.86	-2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	44284	0.0291		2847.2	-1.4	34532	1879.5	-1.3	26325	891.77	-0.8
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	43437	0.0287		3075.92	-3.2	32685	1526.54	-3.1	25795	1530.19	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	40554	0.0271		2173.58	-1.7	31286	3265.97	-1	23856	394.38	-1.2
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.073	37474	0.0255		1382.28	-2.3	27608	945.67	-2	21555	1084.01	-1.7
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.075	35014	0.0242		2757.14	-2.7	27814	1631.3	-2.9	21560	3163.66	-2.9
EUPT-SRM6 no 047 rice spk. 0.05 ug/ml	13.077	37104	0.0253		2806.35	-1.6	28705	1403.56	-1.3	22501	2165.39	-0.2
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6854	0.009		683.48	2.2	5144	575.62	2.4	3890	380.68	3.7
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.07	6354	0.0088		722.02	-2.8	4926	227.87	-3.5	3927	367.36	-3.5
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6367	0.0088		765.17	-2.2	5060	200.18	-1.3	3772	252.95	0.6
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.072	6378	0.0088		536.46	-5.4	5183	380.78	-2.2	4120	493.45	-3.3
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.074	6467	0.0088		977.58	-3	5293	595.49	-4.6	4250	464.58	-3.4
EUPT-C2 no 059 wheat spk. 0.01 ug/ml	13.073	6520	0.0089		568.07	-3.9	5259	610.78	-3.7	4041	475.94	-1.9
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.071	6646	0.0089		947.45	4.2	5286	456.55	3.2	3940	554.06	5.8
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.07	6172	0.0087		650.8	-1	4872	285.24	-2.9	3674	339.2	-1.2
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6930	0.0091		365.33	-4.4	5509	367.1	-3.9	4004	198.03	-3.6
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6828	0.009		983.29	-3.5	5399	486.36	-4.8	4115	315.36	-4.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.074	6420	0.0088		433.69	-4.7	5223	462.55	-4.4	3809	443.77	-3.1
EUPT-C4 no 008 rye spk. 0.01 ug/ml	13.075	6450	0.0088		868.4	-4.1	5035	411.47	-2.4	3625	373.62	-4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.074	6841	0.009		370.19	3.9	5545	146.59	4.6	3964	381.75	5.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6707	0.009		746.95	-2.7	5335	484.64	-1.9	3984	195.52	-2.6
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.073	6509	0.0089		1103.73	-4.6	5089	471.98	-3.6	4150	511.74	-2.9
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.075	6953	0.0091		511.84	-2.3	5442	723.35	-4.4	3813	551.36	-3.4
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.072	6711	0.009		548.35	-6.7	4969	530.79	-2.7	3827	176.45	-4.5
EUPT-C6 no 207 barley spk. 0.01 ug/ml	13.076	6531	0.0089		374.2	-4.7	4964	201.78	-5.4	3972	360.87	-5.2
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.075	9614	0.0105		480.15	2.7	7403	544.11	3.5	5949	368.61	2.4
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8835	0.0101		606.26	-2.8	7033	347.19	-2	5247	684.4	-3
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8821	0.0101		684.85	-3.2	6808	452.2	-1.6	5851	387.83	-1.9
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9276	0.0103		972.69	-3.6	6993	326.86	-4	5330	376.24	-1.6
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.074	9958	0.0107		890.19	-3.8	7746	331.17	-3.4	6121	368.32	-3.1
EUPT-SRM6 no 047 rice spk. 0.01 ug/ml	13.073	8784	0.0101		260.19	-2.6	7154	611.4	-2.7	5606	373.43	-1.9

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Spike level, mg/kg		0.01	0.01	0.05	0.05	
			No. of detects with mass accuracy			
Mass accuracy difference	Molecular or fragment ion*	≤5ppm	≤10ppm	≤5ppm	≤10ppm	SDL, mg/kg
Pirimiphos-methyl	1	22	24	24	24	0.01
Pirimiphos-methyl	2	23	24	24	24	
Pirimiphos-methyl	3	21	24	24	24	

Spike level, mg/kg		0.01	0.01	0.05	0.05	
				No. of detects with mass accuracy		
Mass accuracy difference	Molecular or fragment ion*	≤5ppm	≤10ppm	≤5ppm	≤10ppm	SDL, mg/kg
Pirimiphos-methyl	1	22	24	24	24	0.01
Pirimiphos-methyl	2	23	24	24	24	
Pirimiphos-methyl	3	21	24	24	24	

Spike level, mg/kg		0.01	0.01	0.05	0.05	
			No. of detects with mass accuracy			
Mass accuracy difference	Molecular or fragment ion*	≤5ppm	≤10ppm	≤5ppm	≤10ppm	SDL, mg/kg
Pirimiphos-methyl	1	22	24	24	24	0.01
Pirimiphos-methyl	2	23	24	24	24	
Pirimiphos-methyl	3	21	24	24	24	

Spike level, mg/kg		0.01	0.01	0.05	0.05	
			No. of detects with mass accuracy			
Mass accuracy difference	Molecular or fragment ion*	≤5ppm	≤10ppm	≤5ppm	≤10ppm	SDL, mg/kg
Pirimiphos-methyl	1	22	24	24	24	0.01
Pirimiphos-methyl	2	23	24	24	24	
Pirimiphos-methyl	3	21	24	24	24	

Spike level, mg/kg		0.01	0.01	0.05	0.05	
				No. of detects with mass accuracy		
Mass accuracy difference	Molecular or fragment ion*	≤5ppm	≤10ppm	≤5ppm	≤10ppm	SDL, mg/kg
Pirimiphos-methyl	1	22	24	24	24	0.01
Pirimiphos-methyl	2	23	24	24	24	
Pirimiphos-methyl	3	21	24	24	24	

Validation results – spike experiments

- SDL =0.01 mg/kg (17)
  - Bifenthrin, Chlorpyrifos, Chlorpyrifos-methyl, Diazinon, Fenitrothion, Fipronil, Flutriafol, Iprodione, Krexoxim-methyl, Malathion, Methacrifos, Penconazole, Pirimicarb, Pirimiphos-methyl, Propiconazole, Trifluralin and Vinclozolin
- SDL = 0.05 mg/kg (19)
  - Azoxystrobin, Boscalid, Carboxin, Cypermethrin, Cyprodinil, Diclorvos, Difenoconazole, Epoxiconazole, Fenbuconazole, Lambda-cyhalothrin, Pendimethanil, Prochloraz, Procymidone, Pyraclostrobin, Spiroxamin, Tebuconazole, Triademenol, Trifloxystrobin, Triticonazole
- Not validated (2)
  - Azinphos-methyl, Metconazole

Validation results – PT test material

- The test materials contained 46 residues of 27 different pesticides.
- All pesticides were detected apart from one residue of lambda-cyhalothrin.
  - assigned value = 0.25 mg/kg; SDL = 0.05 mg/kg
- No false positive results were seen.

Validation results – PT test material

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 003 wheat	13.07	158	0.0054		10.99	13.6	164	10.79	7.7	99	9.59	5.2
EUPT-S5 no 105	13.077	26638	0.0197		2974.89	0.7	20537	2572.68	1.6	15978	1740.4	0.4
EUPT-C4 no 143 rye	13.076	32283	0.0227		2519.07	2.4	23981	3377.95	4.1	18355	2167.42	4.1
EUPT-C6 no 032 barleyspk	12.634	20	0.0054		0.17	-79.9	13	0.21	-8.4	10	0.6	99.5

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Validation results – PT test material

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 003 wheat	13.07	158	0.0054		10.99	13.6	164	10.79	7.7	99	9.59	5.2
EUPT-S5 no 105	13.077	26638	0.0197		2974.89	0.7	20537	2572.68	1.6	15978	1740.4	0.4
EUPT-C4 no 143 rye	13.076	32283	0.0227		2519.07	2.4	23981	3377.95	4.1	18355	2167.42	4.1
EUPT-C6 no 032 barleyspk	12.634	20	0.0054		0.17	-79.9	13	0.21	-8.4	10	0.6	99.5

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Validation results – PT test material

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 003 wheat	13.07	158	0.0054		10.99	13.6	164	10.79	7.7	99	9.59	5.2
EUPT-S5 no 105	13.077	26638	0.0197		2974.89	0.7	20537	2572.68	1.6	15978	1740.4	0.4
EUPT-C4 no 143 rye	13.076	32283	0.0227		2519.07	2.4	23981	3377.95	4.1	18355	2167.42	4.1
EUPT-C6 no 032 barleyspk	12.634	20	0.0054		0.17	-79.9	13	0.21	-8.4	10	0.6	99.5

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Validation results – PT test material

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 003 wheat	13.07	158	0.0054		10.99	13.6	164	10.79	7.7	99	9.59	5.2
EUPT-S5 no 105	13.077	26638	0.0197		2974.89	0.7	20537	2572.68	1.6	15978	1740.4	0.4
EUPT-C4 no 143 rye	13.076	32283	0.0227		2519.07	2.4	23981	3377.95	4.1	18355	2167.42	4.1
EUPT-C6 no 032 barleyspk	12.634	20	0.0054		0.17	-79.9	13	0.21	-8.4	10	0.6	99.5

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< -10

> 10

Validation results – PT test material

	Pirimiphos-methyl Results						Qualifier (276.0573) Results			Qualifier (305.0962) Results		
Name	RT	Area	Final Conc	Accuracy	S/N	Mass Accu	Area	S/N	Mass Accu	Area	S/N	Mass Accu
EUPT-C2 no 003 wheat	13.07	158	0.0054		10.99	13.6	164	10.79	7.7	99	9.59	5.2
EUPT-S5 no 105	13.077	26638	0.0197		2974.89	0.7	20537	2572.68	1.6	15978	1740.4	0.4
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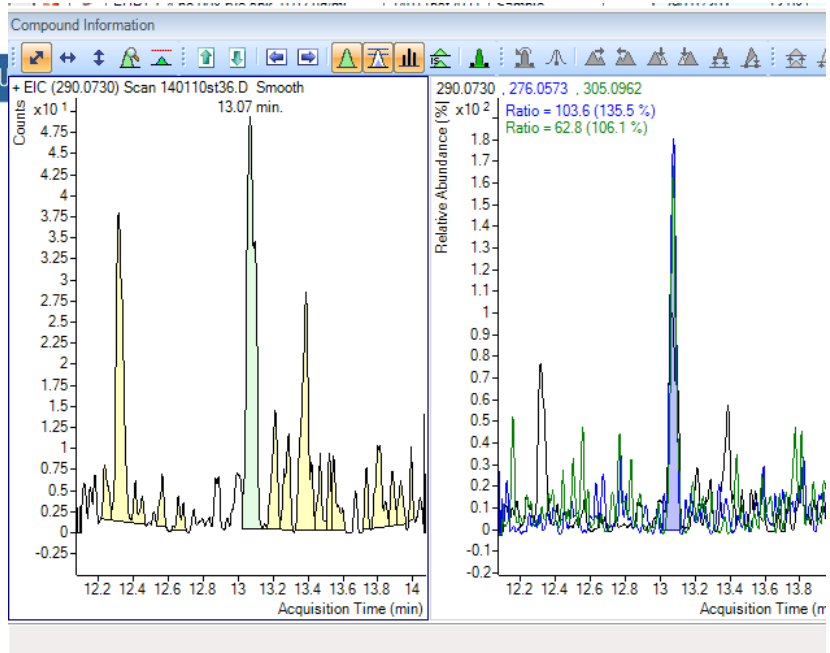
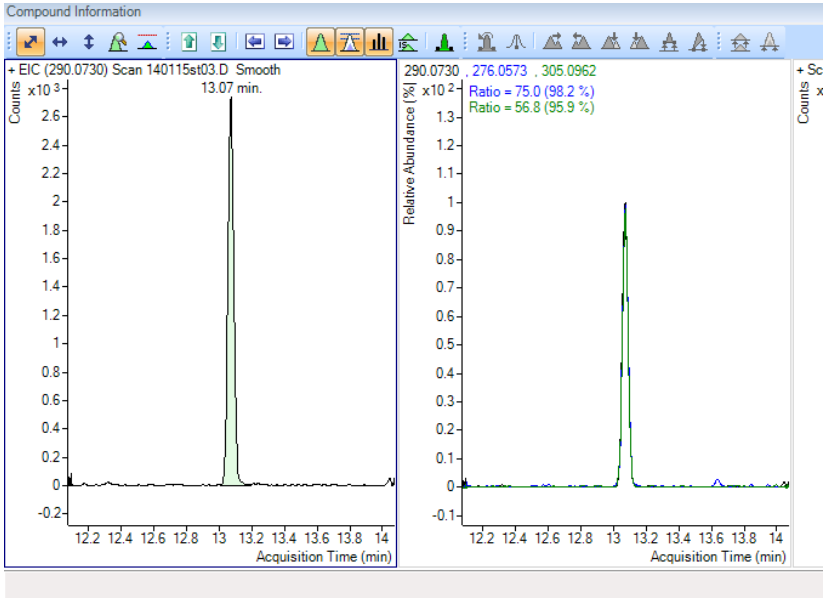

Compound Information
+ EIC (290.0730) Scan 140110st36.D Smooth
13.07 min.
Counts
Acquisition Time (min)
290.0730 , 276.0573 , 305.0962
Ratio = 103.6 (135.5 %)
Ratio = 62.8 (106.1 %)
Relative Abundance (%)
Acquisition Time (m


Compound Information
+ EIC (290.0730) Scan 140115st03.D Smooth
13.07 min.
Counts
Acquisition Time (min)
290.0730 , 276.0573 , 305.0962
Ratio = 75.0 (98.2 %)
Ratio = 56.8 (95.9 %)
Relative Abundance (%)
Acquisition Time (min)


Thank you for your attention