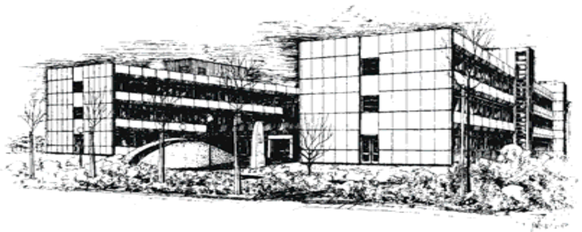
Rheinland-Pfalz
DIENSTLEISTUNGSZENTRUM
LÄNDLICHER RAUM
RHEINPFALZ

Zielkomponenten- und Profiling-Analysen in der modernen Weinaromaanalytik: Spielwiesen für den Einsatz mehrdimensionaler (gas)chromatographischer Techniken?

Hans-Georg Schmarr



Kompetenzzentrum Weinforschung

Dienstleistungszentrum Ländlicher Raum – Rheinpfalz
Abteilung Weinbau und Oenologie
Breitenweg 71
D-67435 Neustadt an der Weinstraße, Germany

Überblick 1

- Weinaroma: **Zielkomponentenanalyse**

    - **Problematik und Bedarf für High-End Analytik**

- Beispiele: Methoxypyrazine (MPs), Haloanisole
- HS-SPME-GC(×GC)-MS (SIM)
- SPE // on-line-LC-MDGC-MS (SIM)
- SPE // LV-OC-MDGC-MS (SIM)
- SPE // LV-PTV-OC-MDGC-MSMS

Überblick 2

- Weinaroma: **Profiling-Analysen**

  - **Erfassung molekularer Veränderungen durch weinbauliche und oenologische Massnahmen**

- Beispiele: Alterung; Micro-Oxygenierung
- HS-SPME-GC(×GC)-MS
- Klassische und *pixel-based* Verfahren
- Statistische Auswertung; Korrelationen
- Zusammenfassung und Ausblick

Weinaroma für den Analytiker

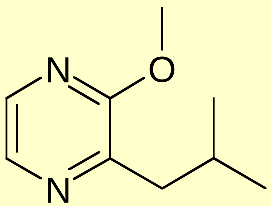
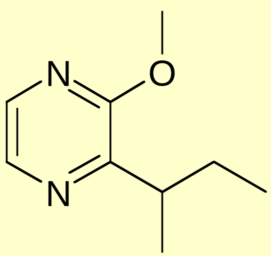
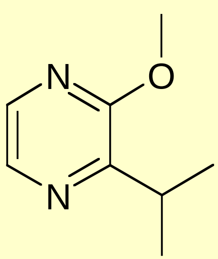
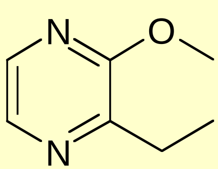
- Weinaroma ist sehr komplex zusammengesetzt
- Aromastoffe in sub-ng/L bis mg/L Konzentrationen
- Organische Chemie in breiter Abdeckung
- Matrixzusammensetzung oft problematisch für Extraktion, Chromatographie und Detektion
- **Störungen – gerade bei (sub-)ng/L Bestimmungen**

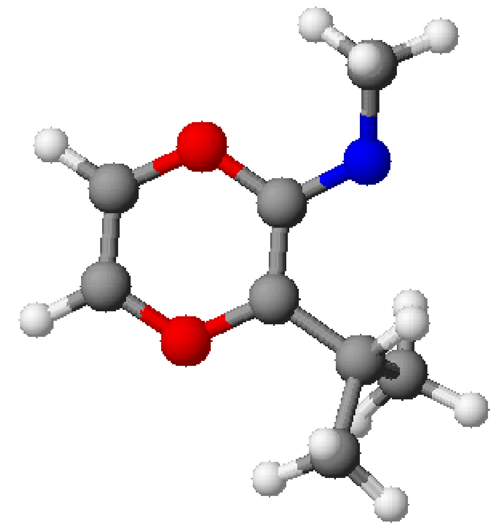
Zur Biochemie des sortentypischen Weinaromas

Wein – Qualität entscheidet sich in Nanogramm

Matthias Wüst, Chem. in Unserer Zeit 37 (2003) 8-17

Methoxypyrazine im Sauvignon Blanc

Aromastoff	*Struktur*	*Geruchs-eigenschaft*	*Geruchs-schwellenwert [ng/ l]*
2-Methoxy-3-isobutyl		grüner Paprika, Pfeffer	2
2-Methoxy-3-sec. butyl		grüne Erbsen, grüner Paprika	1
2-Methoxy-3-isopropyl		grüner Paprika, Pfeffer, erdig	2
2-Methoxy-3-ethyl		grüner Paprika, erdig	400



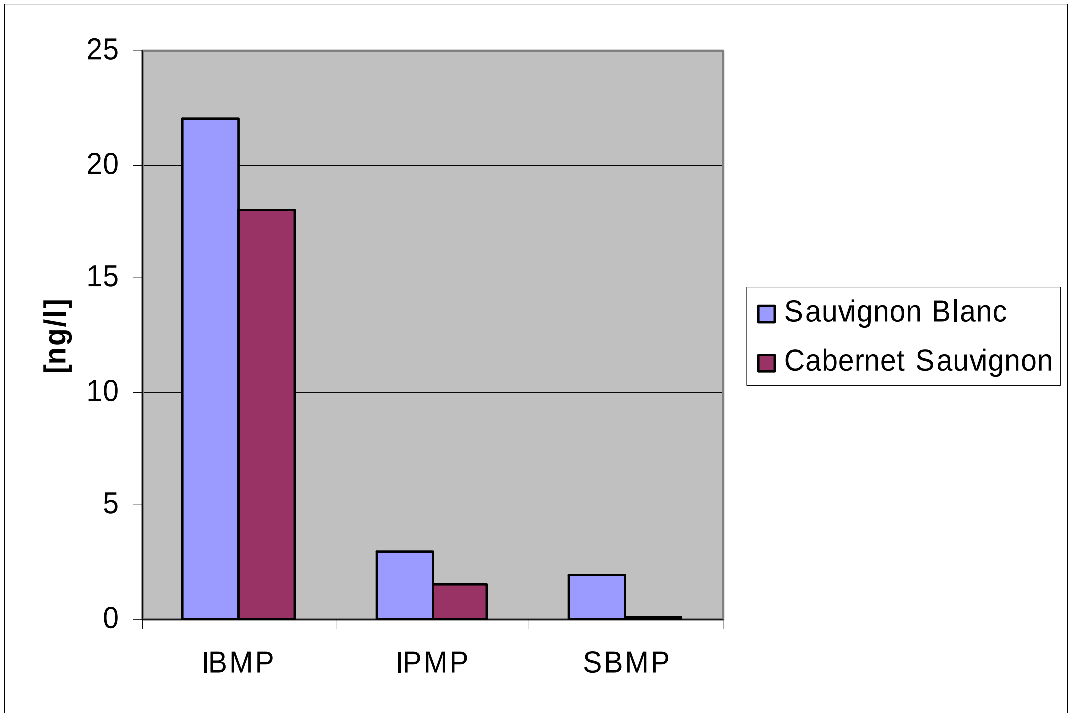
3-Isopropyl-2-Methoxypyrazin

Enger – *angenehmer* – Konzentrationsbereich von etwa 8-15 ng/L

Wüst, M., Chemie in Unserer Zeit. 37 (2003) 8 - 17

MPs in *Vitis vinifera var.*

- **Dominierendes MP ist IBMP**



MP-Konzentration hoch **in unreifen Trauben** und geringer werdend mit zunehmender Reife

Allen, M.S., Lacey, M.J., ACS Symp. Ser. 714 (1998) 31 - 38

Methoxypyrazine mal nicht aus Trauben

Klimawandel und das Vordringen des Asiatischen Marienkäfers *Harmonia axyridis* (nicht nur) in pfälzischen Weinbergen ...

- Käfer enthalten (in der Hämolymphe) IPMB, IBMP, SBMP und DMMP
- Gehalte [ng/g Käfer] gefunden in (lebenden) Käfern: IPMP (4.2); SBMP (1.9); IBMP (<0.01)[1]
- Sensorisch bestimmter Schwellenwert für off-flavor durch *H. axyridis* (in der Lit. auch “ladybug taint; LBT” genannt) liegt bei 0.3 bis 1.9 Käfer/kg Trauben[2]

Interesse der amtlichen Lebensmittelüberwachung an der Analytik von Methoxypyrazingehalten in Weinen (insbesondere Sauvignon Blanc und Cabernet Sauvignon) ... --> IBMP (173 ng/l südafrikanischer S. Blanc[3])

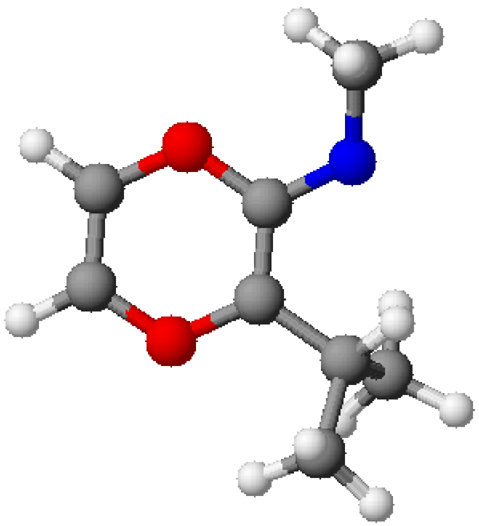
[1] Cai, L. *et al.,* J. Chromatogr. A 1147 (2007) 66 - 78

[2] Galvan, T. L. *et al.*, Am. J. Enol. Viticul. 58 (2007) 518 - 522

[3] Godelmann, R. *et al.,* Eur. Food Res. Technol. 227 (2007) 449 - 461

Methoxypyrazine

***Harmonia axyridis* ...**



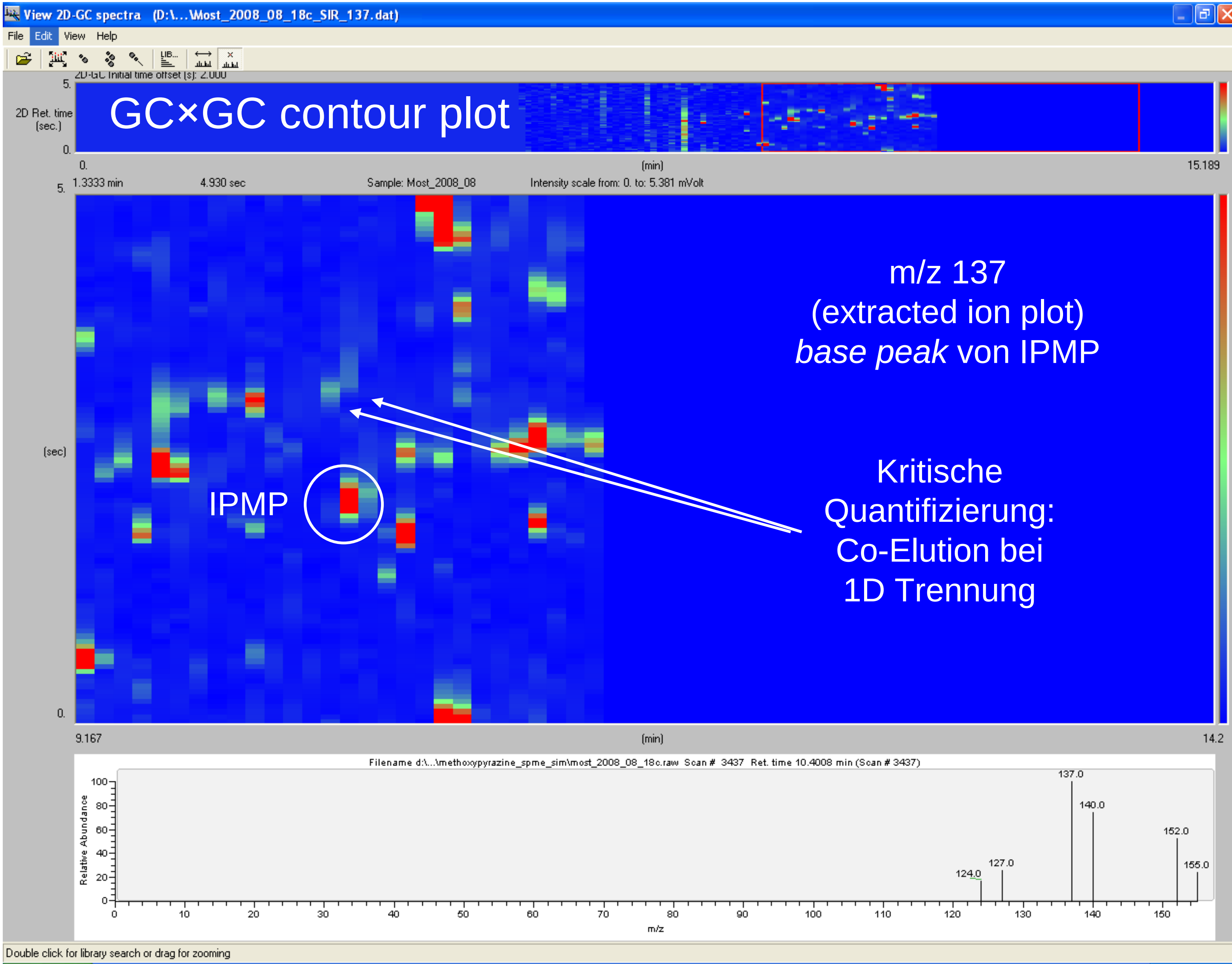
3-Isopropyl-2-
methoxypyrazin

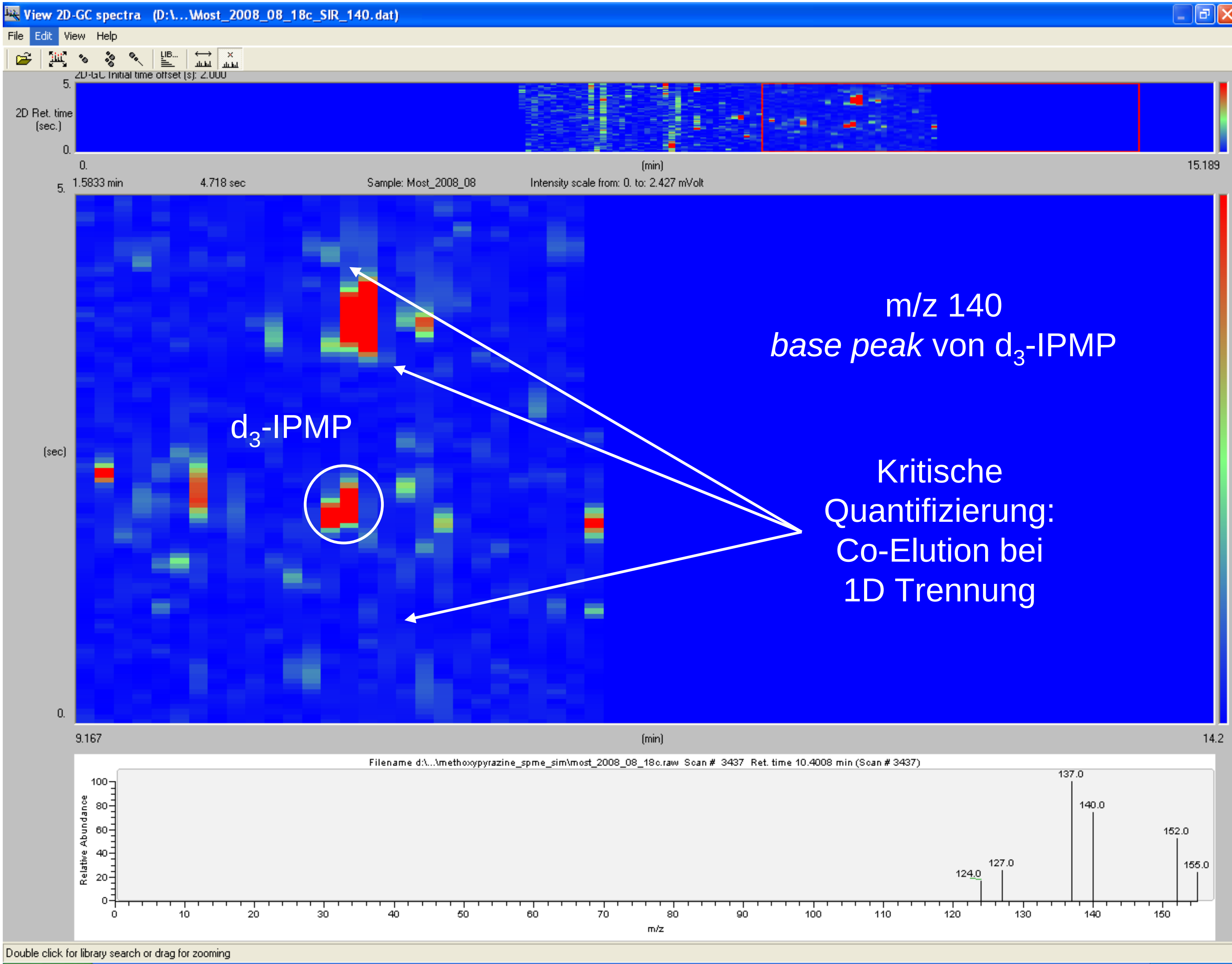
Analyse mit einer oft beschriebenen Methode:

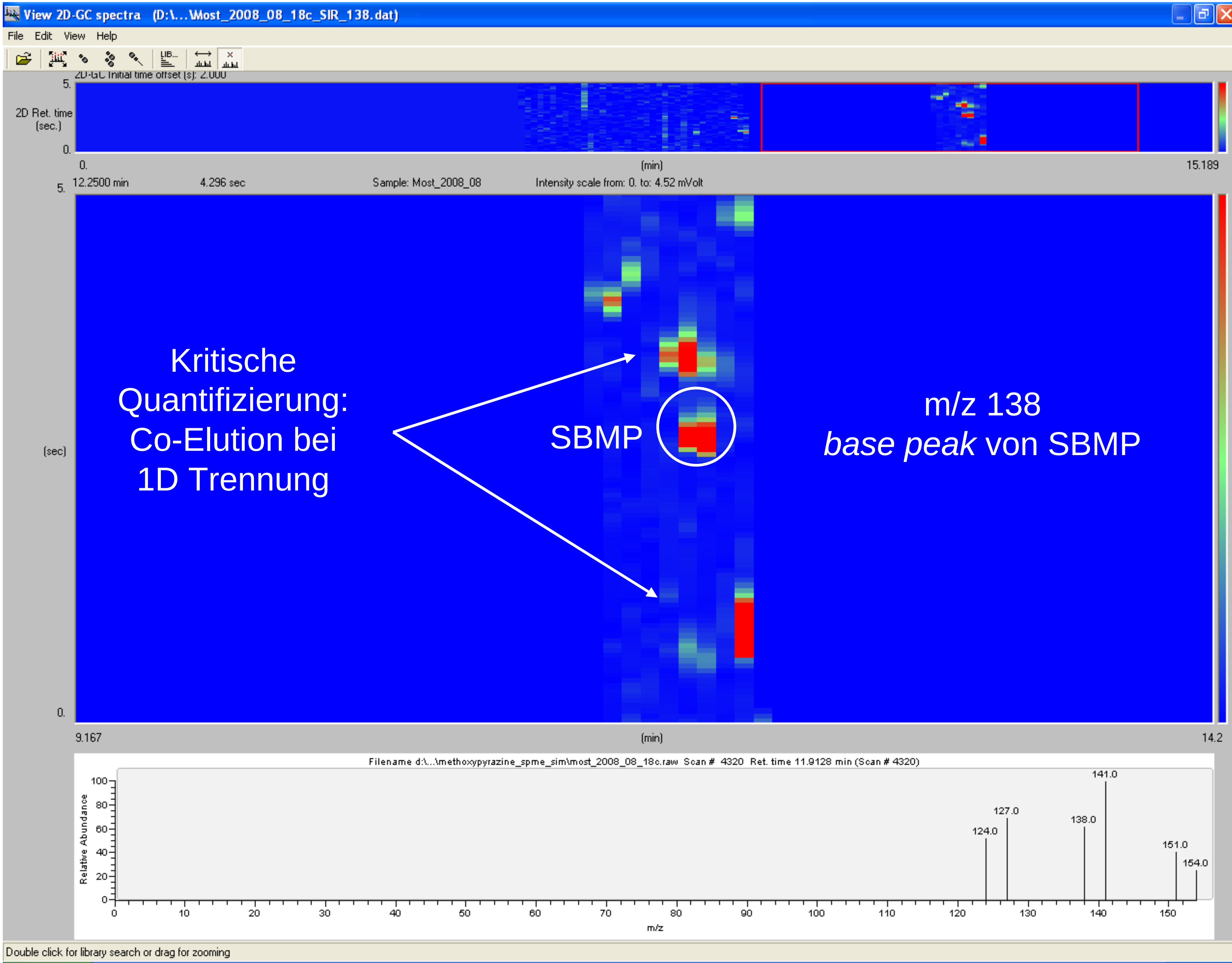
HS-SPME-GC-MS

- pH Einstellung auf 6.6
- SIDA für Quantifizierung der MPs (d_3-MPs)
- HS-SPME-GC-MS (SIM);
  Divinylbenzene/Carboxen/PDMS Faser, HP-5MS column, SIM-MS[1]
- **In unserer Situation ergaben dotierte Proben (Most) nicht die erwarteten Resultate !?**
- Spektren bzw. “Quantifier/Qualifier” Ionenverhältnisse auffällig
- GC×GC-qMS für Fehlersuche (Trace GC×GC-DSQ)

[1] Kotseridis, Y.S. *et al.*, J. Chromatogr. A, 1190 (2008) 294-301


View 2D-GC spectra (D:\...\Most_2008_08_18c_SIR_137.dat)
File Edit View Help
2D-GC Initial time offset [s]: 2.000
2D Ret. time (sec.)
GC×GC contour plot
0.
(min)
15.189
1.3333 min
4.930 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 5.381 mVolt
(sec)
m/z 137
(extracted ion plot)
base peak von IPMP
IPMP
Kritische
Quantifizierung:
Co-Elution bei
1D Trennung
9.167
(min)
14.2
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 3437 Ret. time 10.4008 min (Scan # 3437)
Relative Abundance
m/z
137.0
140.0
152.0
127.0
124.0
155.0
Double click for library search or drag for zooming


View 2D-GC spectra (D:\...\Most_2008_08_18c_SIR_140.dat)
File Edit View Help
2D-GC Initial time offset [s]: 2.000
2D Ret. time (sec.)
15.189
1.5833 min
4.718 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 2.427 mVolt
m/z 140
base peak von d3-IPMP
d3-IPMP
Kritische Quantifizierung: Co-Elution bei 1D Trennung
9.167
(min)
14.2
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 3437 Ret. time 10.4008 min (Scan # 3437)
Relative Abundance
137.0
140.0
152.0
155.0
127.0
124.0
m/z
Double click for library search or drag for zooming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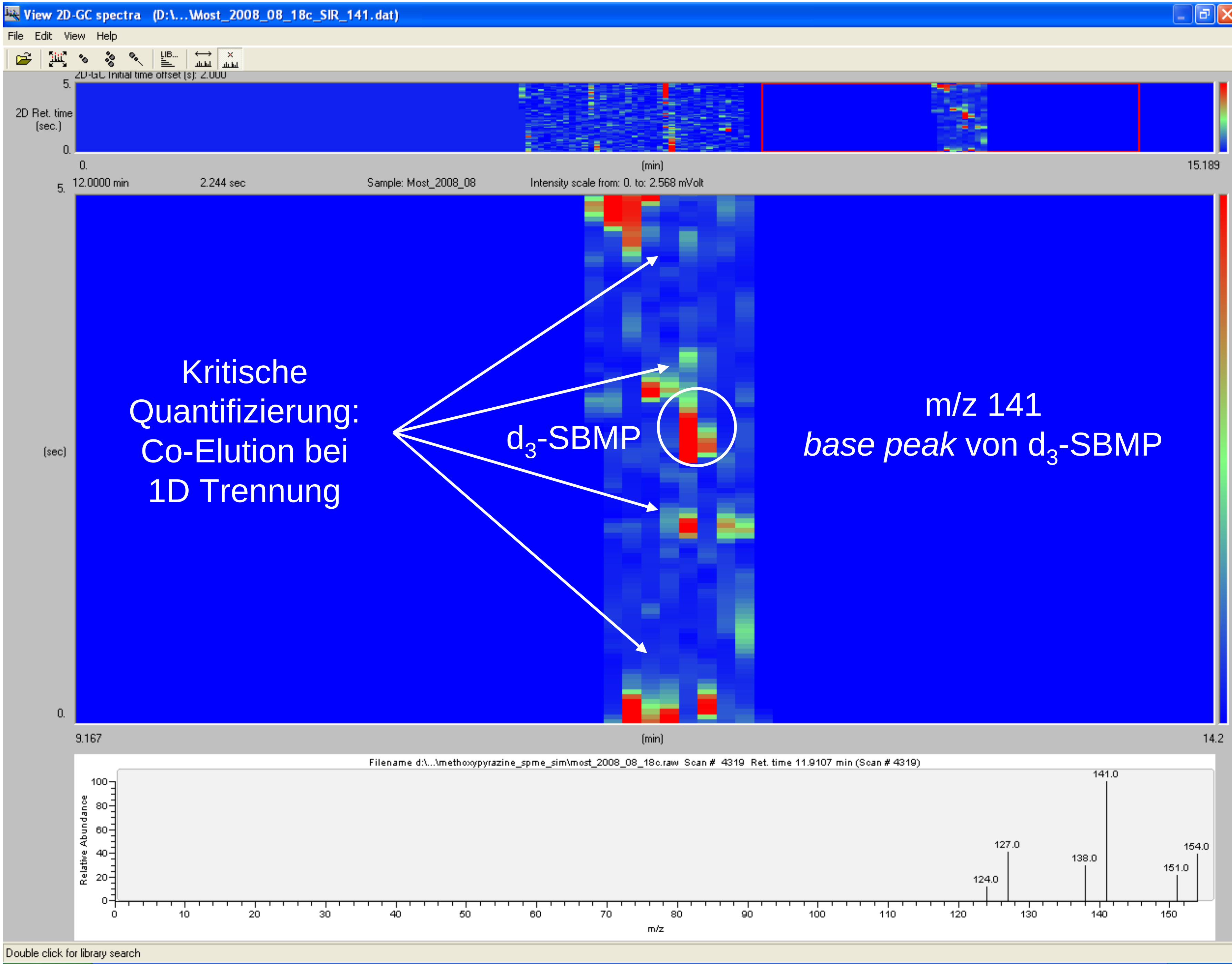
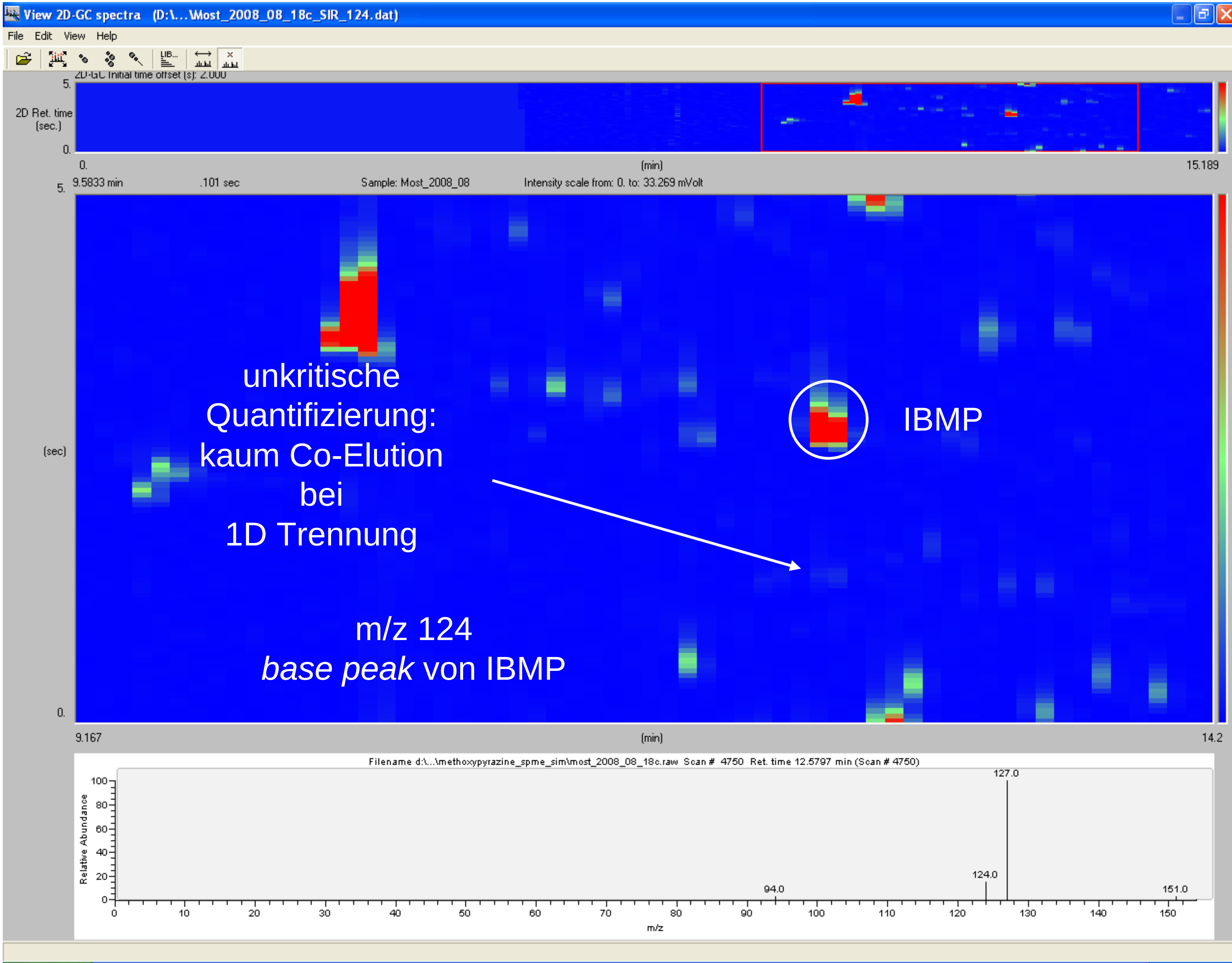
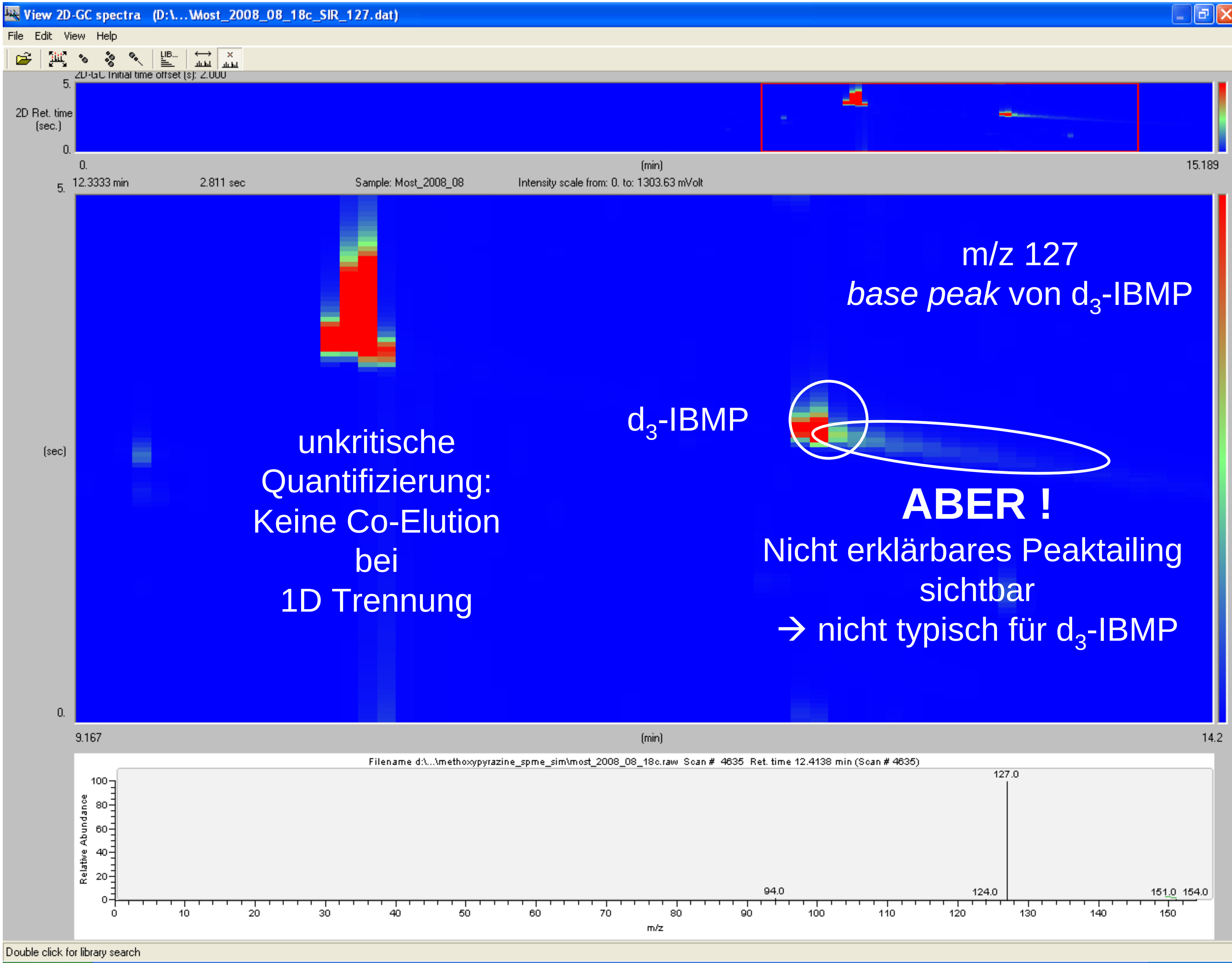
File Edit View Help
2D-GC Initial time offset [s]: 2.000
2D Ret. time (sec.)
5.
0.
0.
(min)
15.189
12.2500 min
4.296 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 4.52 mVolt
(sec)
9.167
14.2
Kritische Quantifizierung: Co-Elution bei 1D Trennung
SBMP
m/z 138
base peak von SBMP
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 4320 Ret. time 11.9128 min (Scan # 4320)
Relative Abundance
m/z
124.0
127.0
138.0
141.0
151.0
154.0
Double click for library search or drag for zooming


View 2D-GC spectra (D:\...\Most_2008_08_18c_SIR_141.dat)
File Edit View Help
2D-GC Initial time offset [s]: 2.000
2D Ret. time (sec.)
[min]
15.189
12.0000 min
2.244 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 2.568 mVolt
[sec]
9.167
14.2
Kritische Quantifizierung: Co-Elution bei 1D Trennung
d_3-SBMP
m/z 141
base peak von d_3-SBMP
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 4319 Ret. time 11.9107 min (Scan # 4319)
Relative Abundance
m/z
124.0
127.0
138.0
141.0
151.0
154.0
Double click for library search



View 2D-GC spectra (D:\...\Most_2008_08_18c_SIR_124.dat)
File Edit View Help
2D-GC Initial time offset [s]: 2.000
2D Ret. time (sec.)
5.
0.
0.
(min)
15.189
9.5833 min
.101 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 33.269 mVolt
(sec)
9.167
14.2
unkritische Quantifizierung: kaum Co-Elution bei 1D Trennung
IBMP
m/z 124
base peak von IBMP
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 4750 Ret. time 12.5797 min (Scan # 4750)
Relative Abundance
127.0
124.0
94.0
151.0
m/z


View 2D-GC spectra (D:\...\Most_2008_08_18c_SIR_127.dat)
File Edit View Help
2D-GC Initial time offset (s): 2.000
2D Ret. time (sec.)
0.
(min)
15.189
12.3333 min
2.811 sec
Sample: Most_2008_08
Intensity scale from: 0. to: 1303.63 mVolt
(sec)
9.167
14.2
m/z 127
base peak von d_3-IBMP
d_3-IBMP
unkritische Quantifizierung: Keine Co-Elution bei 1D Trennung
ABER !
Nicht erklärbares Peaktailing sichtbar
→ nicht typisch für d_3-IBMP
Filename d:\...\methoxypyrazine_spme_sim\most_2008_08_18c.raw Scan # 4635 Ret. time 12.4138 min (Scan # 4635)
Relative Abundance
127.0
94.0
124.0
151.0
154.0
m/z
Double click for library search

Alternative Analysenideen ...

- **GC×GC-ToF-MS**

  eventuell mit anderen Ionen und/oder Spektrendekonvolution

  → evtl. mit hochauflösendem ToF-MS

  → (beide) nicht verfügbar (in unserer Gruppe)

- **GC×GC-NPD**

  - evtl. → aber dann SIDA Quantifizierung problematisch mit d_3-MPs

- **Probenvorbereitung**

  → SPE mit LiChrolut® EN als Sorbenz[1]

- **Anderer chromatographischer Ansatz**

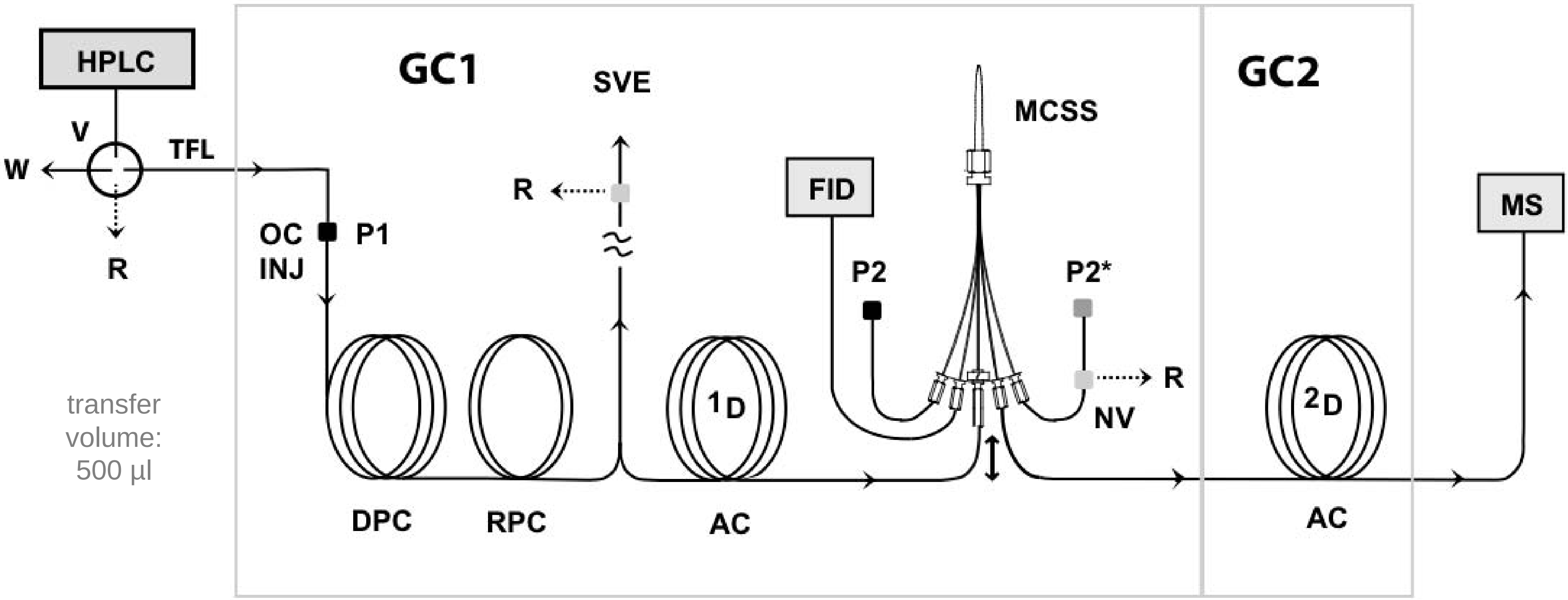
  - andere stationäre Phase(n), Säulendimension, Gasfluss, ...

  → **on-line LC-(MD)GC statt GC×GC**

[1] Culleré, L. *et al.*, J. Chromatogr. A, 1216 (2009) 4040-4045

on-line LC-(MD)GC

transfer
volume:
500 µl



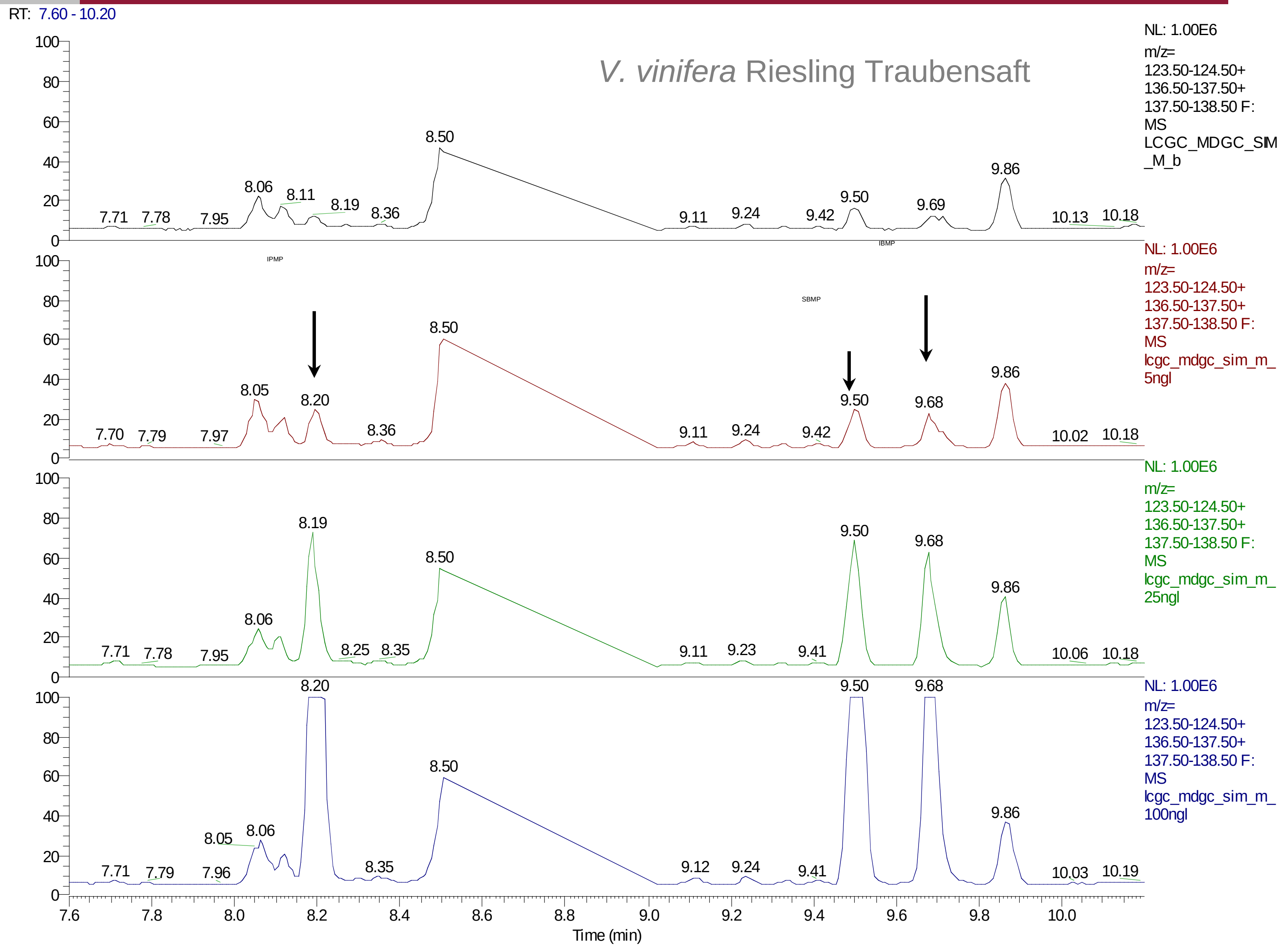
MCSS – Moving Capillary Stream Switching;
H. Sulzbach, C.E. Instruments patent

Schmarr, H.-G. *et al.*
J. Chromatogr. A, 1217 (2010) 6769-6777

SPE // on–line LC–MDGC–MS (SIM)

Probenvorbereitung:	SPE (LiChrolut EN, Merck); 100 ml Most, 1 ml Extrakt
HPLC:	Phoenix20 (Thermo) + TM 486 (Waters); UV (277 nm)
Trennsäule:	12 cm x 2 mm i.d. HyperSil Si (3µm); 20 µl Inj.vol.
Eluent:	*n*-Hexan/MTBE (70/30; v/v) @ 200 µl/min
Transfervolumen:	0.5 ml
Abdampfrate:	172 µl/min @ 72 °C, 100 kPa He
GC Vorsäulen:	10 m x 0.53 mm i.d. unbelegt, desaktiviert (Phesil) + 2 m x 0.32 mm i.d., 1.5 µm PS-255 Y-Stück, *early solvent vapor exit*
^{1}D Säule:	20 m x 0.25 mm i.d. ZB-Wax, 0.5 µm 72 °C (8 min), 8°/min to 250 °C (10 min)
Säulenschaltung:	MCSS (Domdruck @ 25 kPa)
^{2}D Säule:	15 m x 0.25 µm i.d. ZB-35, 0.5 µm 55 °C (3 min), 10°/min to 250 °C (10 min)
Detektion:	Quadrupol MS (MD800, Thermo); EI+, SIM
Quantifizierung:	SIDA mit d_3-IPMP, d_3-SBMP, d_3-IBMP

Erste Ergebnisse



Standard-Addition: nD MPs [ng/l]

0

5

25

100

extracted ion Chromatogramme nach SPE // on-line-LC-MDGC-MS (SIM)

MPs in *Riesling*

MPs in *Vitis Vinifera c.v.* Riesling (Traubensaft); Neustadt, Pfalz

Quantifizierungsmethode: **Standard Addition**

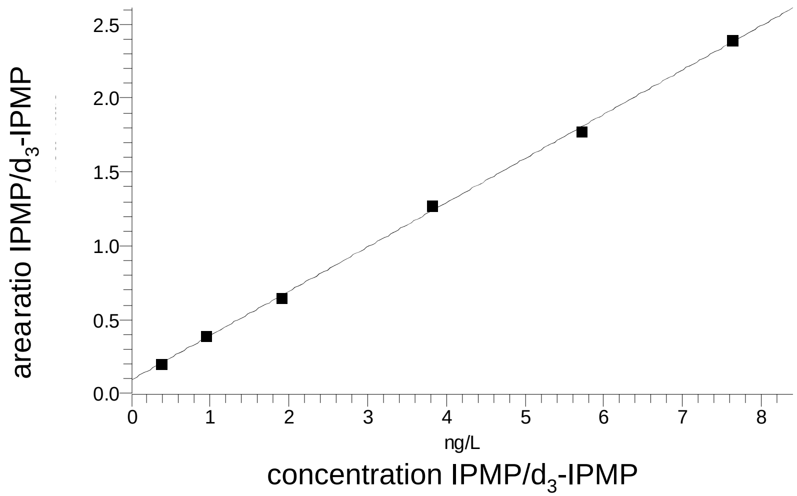
	spike level [ng/L]			[ng/L]		
	5	**25**	**100**	**av.**	**std.dev.**	**cov [%]**
IPMP	2.5	2.6	2.5	**2.5**	0.1	3.9
SBMP	5.8	5.3	5.6	**5.6**	0.3	4.6
IBMP	4.3	4.3	4.0	**4.2**	0.2	4.2

Bisher waren kaum Daten für Riesling publiziert: <0.2-6 ng IPMP oder 0.3-55 IBMP in ng/L Saft oder ng/kg in unreifen Beeren.

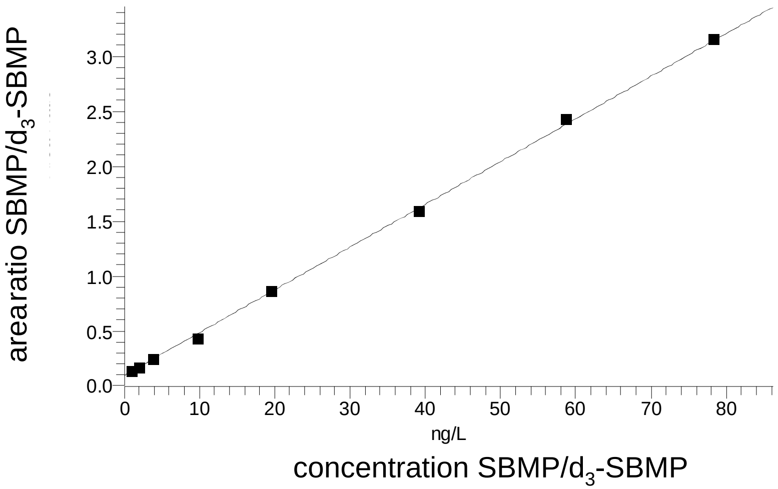
Hashizume und Samuta, Am. J. Enol. Vitic. 50 (1999) 194-198

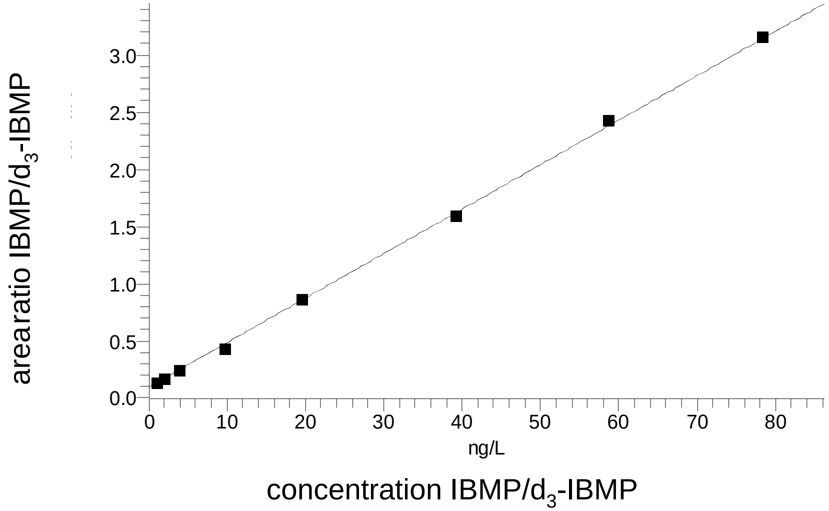
Kalibrierung (SIDA)

Kalibrierungsbereich: 0.4 – 8 ng/l



Kalibrierungsbereich: 0.2 – 8 ng/l





Kalibrierungsbereich:
1 – 80 ng/l

MP-Gehalte in Sauvignon Blanc Most

Reife

c_{MPs}

	Most[a] [ng L^{-1}]		
Lesetermin	**IPMP**	**SBMP**	**IBMP**
Serie A[c]			
08.08.07	<0.4	nd	9.6
14.08.07	<0.4	nd	6.9
23.08.07	<0.4	nd	4.6
28.08.07	<0.4	nd	1.2
09.09.07	<0.4	nd	<1.1
10.09.07[d]	0.6	nd	3.6
Serie B[c]			
14.08.07	<0.4	nd	5.3
23.08.07	<0.4	nd	3.5
28.08.07	<0.4	nd	3.4
05.09.07	<0.4	nd	2.4
11.09.07	<0.4	5.7	1.4
17.09.07	<0.4	5.0	1.8

[a] Most nach SPE mit LiChrolut® EN

[b]

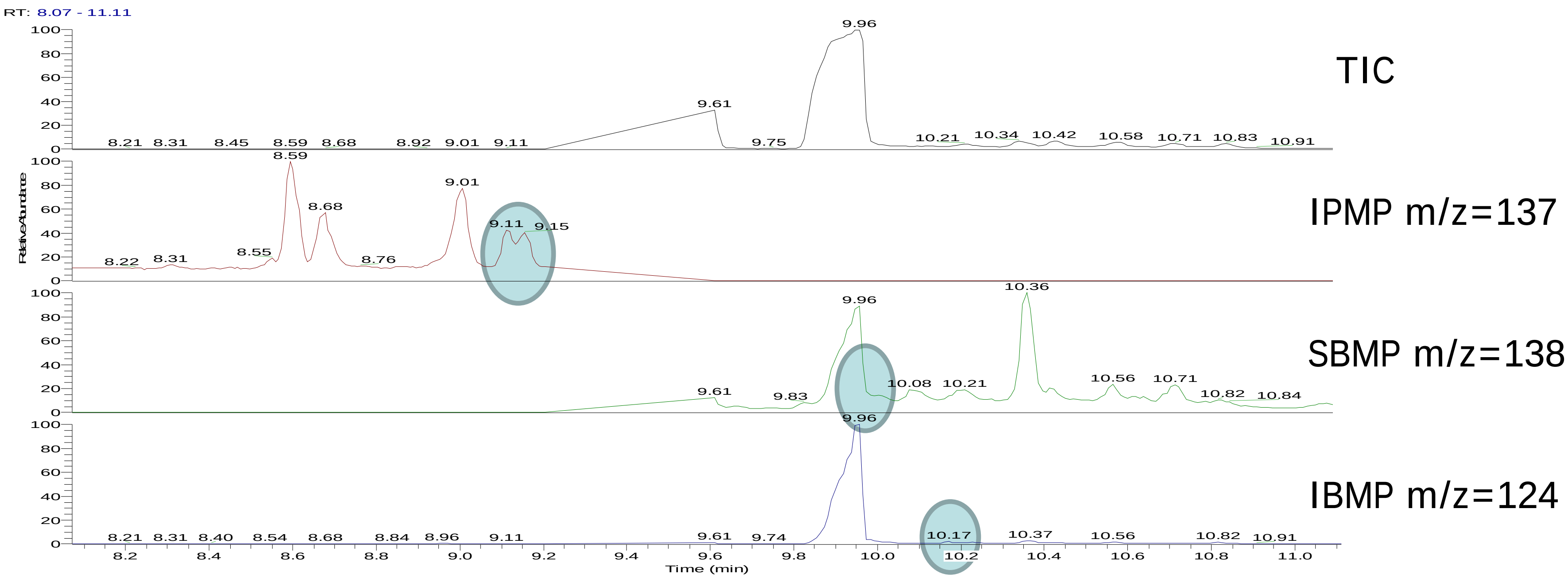
[c] Serie A von Weisenheim (Sand/Lehm; *warm*), Serie B von Hochstadt (Löß/Lehm; *kalt*), 2 unterschiedliche Standorte (Boden, Mikroklima)

[d] Lese zuvor für Reifemessungen , hier komplette Lese

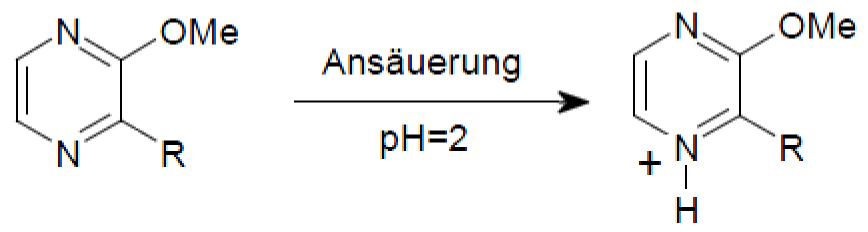
MPs in *Sauvignon Blanc* Wein

MPs in *Sauvignon Blanc* (**Wein**); aus Neuseeland, Marlborough Region (2007)

Probe	[ng/L]		
	IPMP	**SBMP**	**IBMP**
SB_NZ	?	?	?



SPE mit Kationenaustauscher



3-Alkyl-2-methoxypyrazin

3-Alkyl-2-methoxypyrazin Kation

R. Lopez *et al.*
J. Chromatogr. A 1218 (2011) 842

Plexa PCX™-Säulen (200mg):

- **Wein** (75 ml) + MPs-D3, einstellen auf pH=2
- Konditionierung: Dichlormethan (DCM)
Methanol
milli-Q Wasser
- 1. Waschschritt: milli-Q H_2O/Methanol(30%) pH=2
- Trocknung: Argon
- 2. Waschschritt: DCM
- Elution: Triethylamin in DCM (10g/L)
- 3.Waschschritt: milli-Q-H_2O (pH=3 mit Weinsäure einstellen)

MPs in *Sauvignon blanc*

Peter Schoenmakers:

Thank God for the matrix!

MPs in *Vitis Vinifera c.v.* Sauvignon blanc (**Wein**); Herkunft Neuseeland, Marlborough Region

Quantifizierung: **SIDA**

sample	[ng/L]		
	IPMP	SBMP	IBMP
sv_nz	2.3	0.4	17.1

... asking for expert(s) hyphenated techniques ...

Problem gelöst !

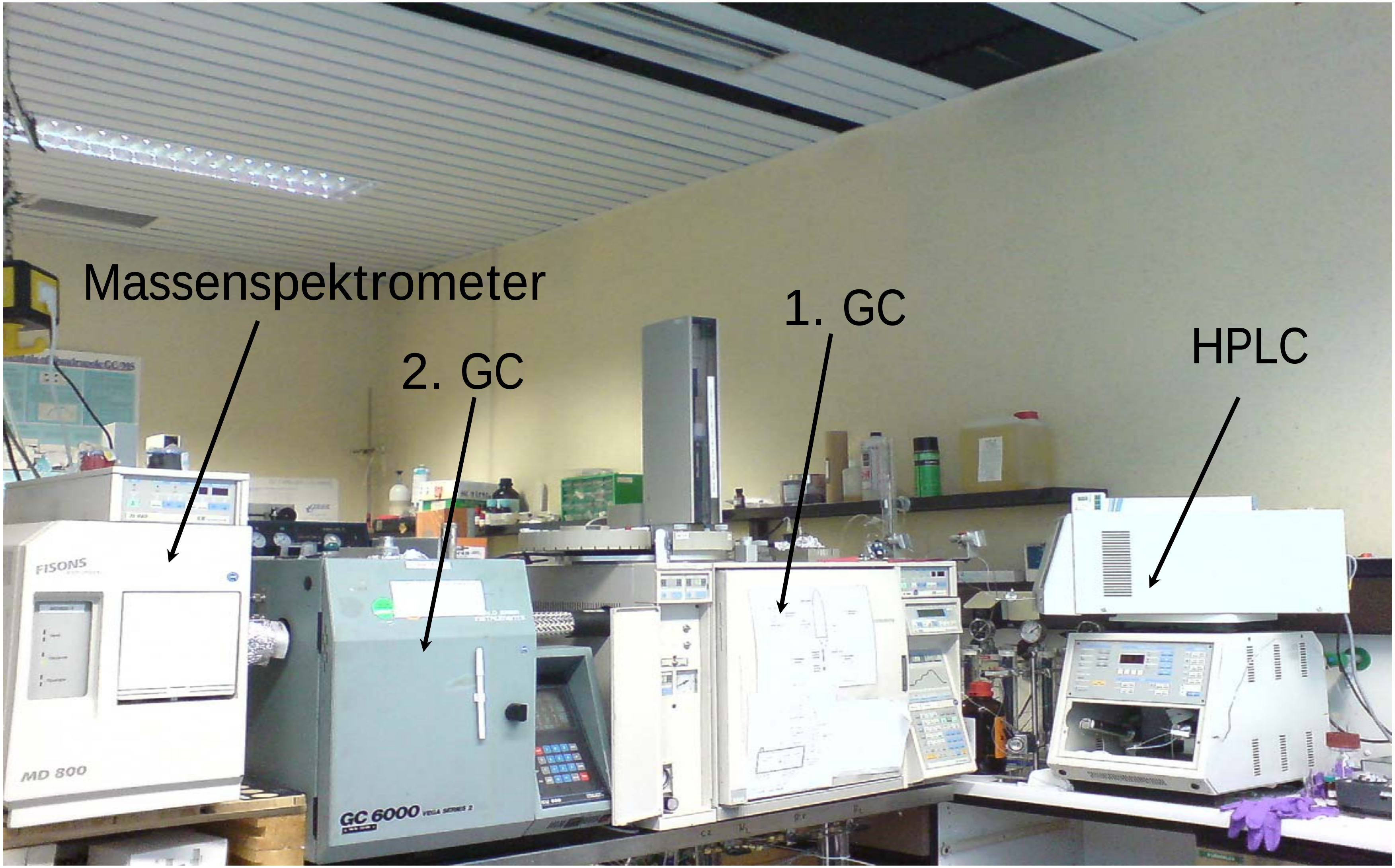
SPE mit **Mischbett Material** (Bond Elut Plexa PCX™, Varian) welches auch **starke Kationenaustausch-Funktionalität enthält**[1]

Ermöglicht wesentlich selektivere Extraktion der MPs, durch die Ausnutzung der N-Protonierung bei saurem pH.

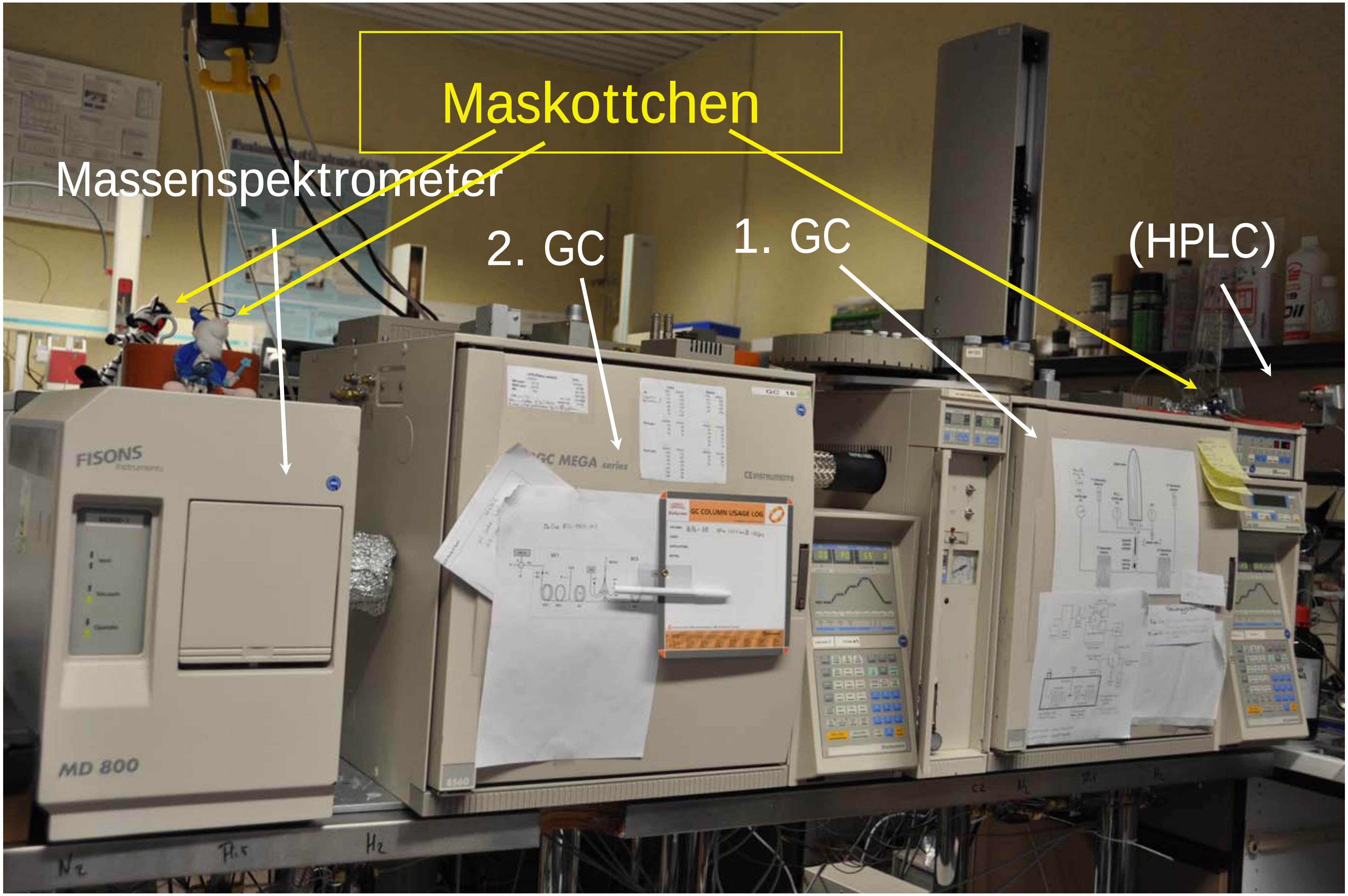
Zur Analyse von MPs in vergorenem Most (Weinen) ist dies unbedingt angeraten!

[1] R. Lopez *et al.*: J. Chromatogr. A 1218 (2011) 842

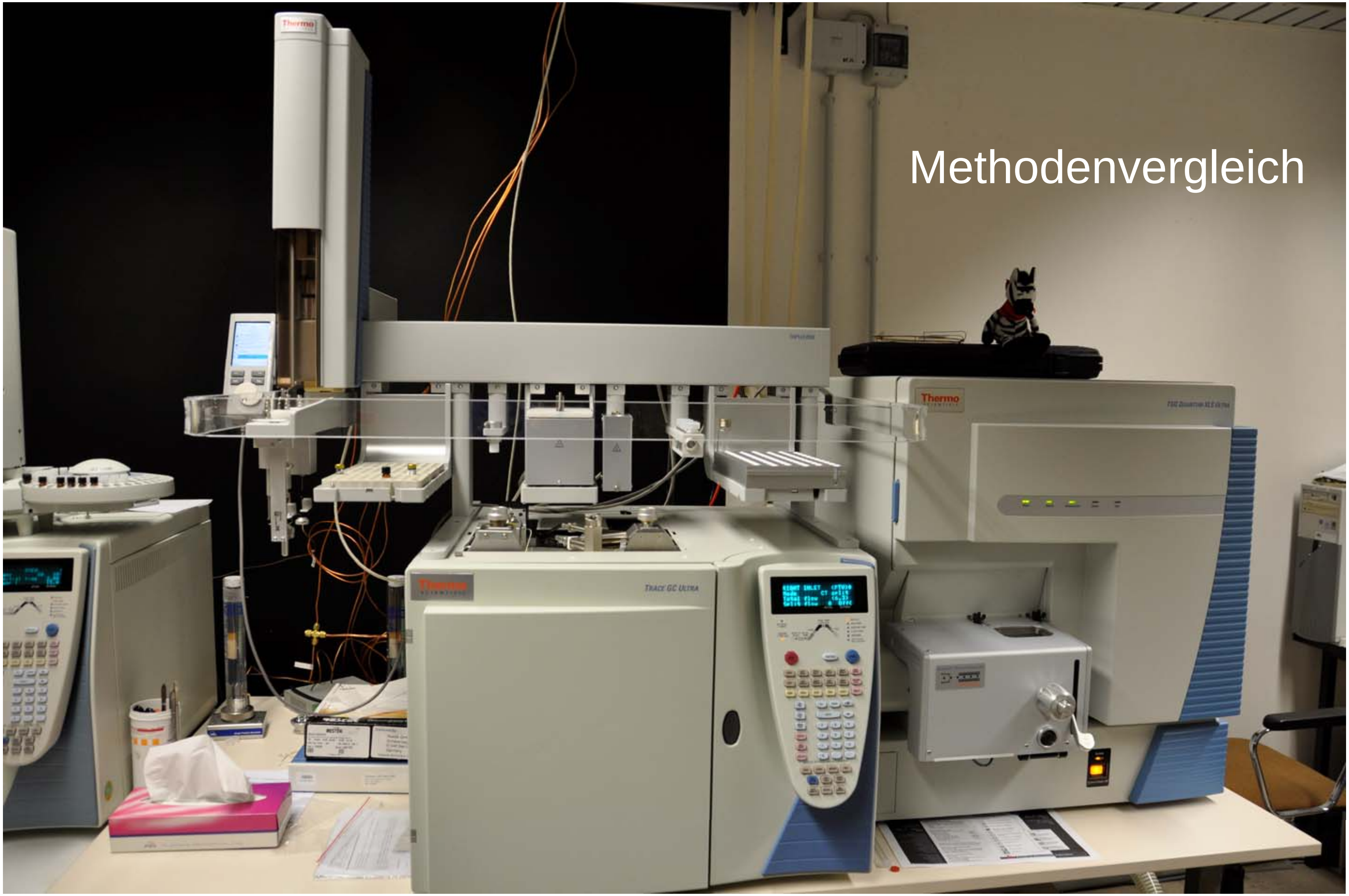
On-line LC-MDGC-MS



Verbesserte SPE und LV-OC-MDGC-MS

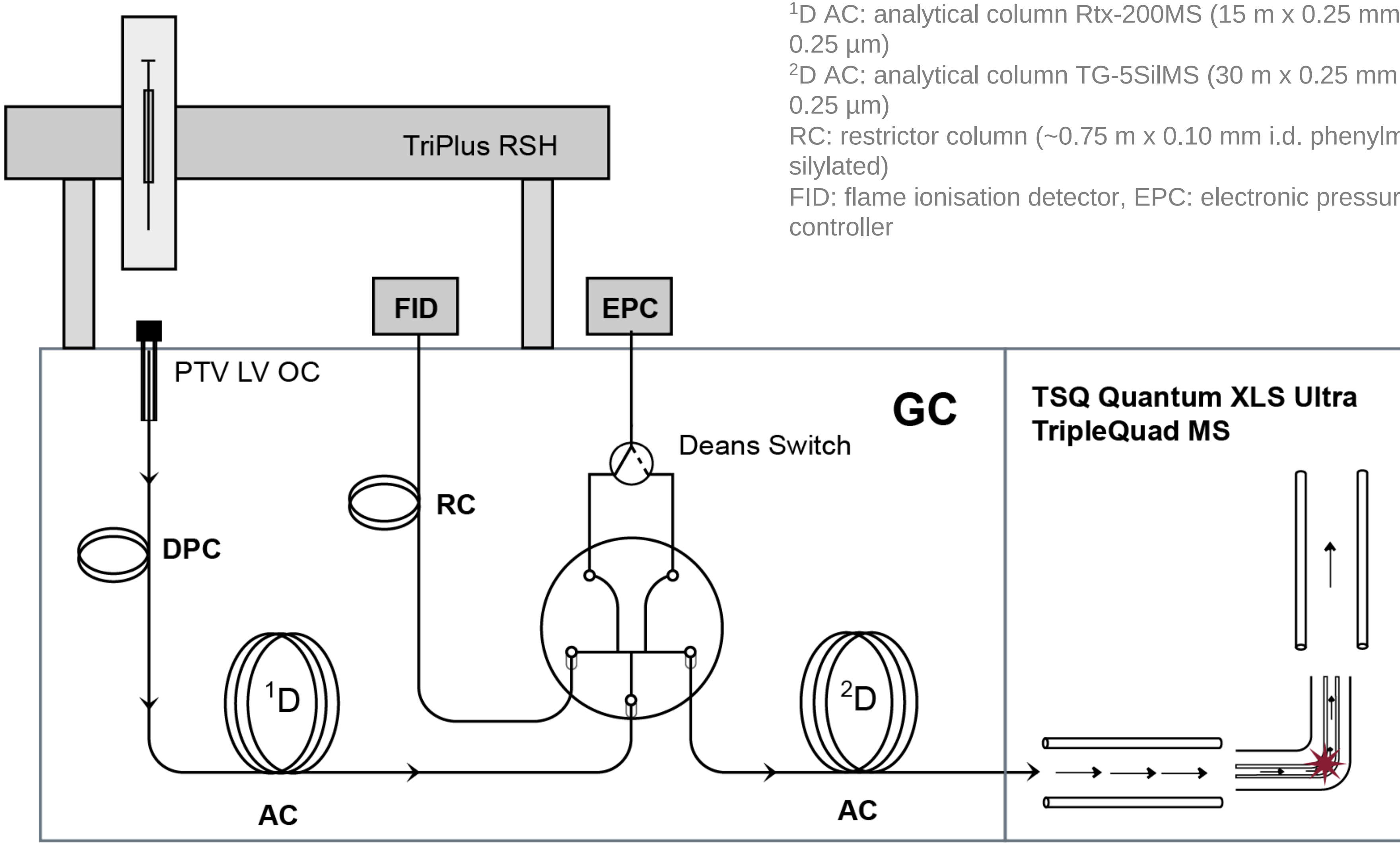


Modernes Equipment: Triplus RSH & MDGC-QqQ-MS

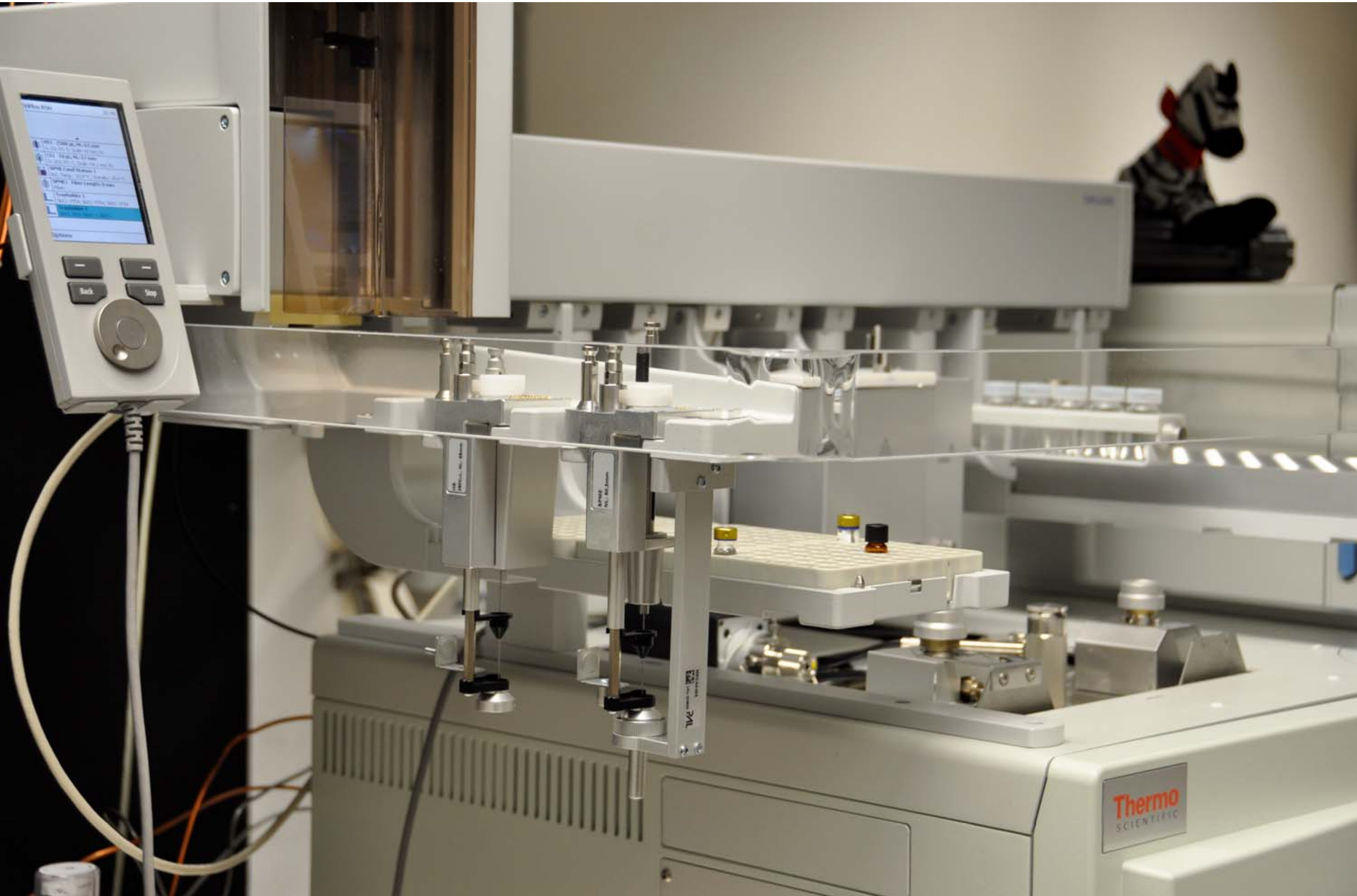


MDGC-QqQ-MS: Schematics

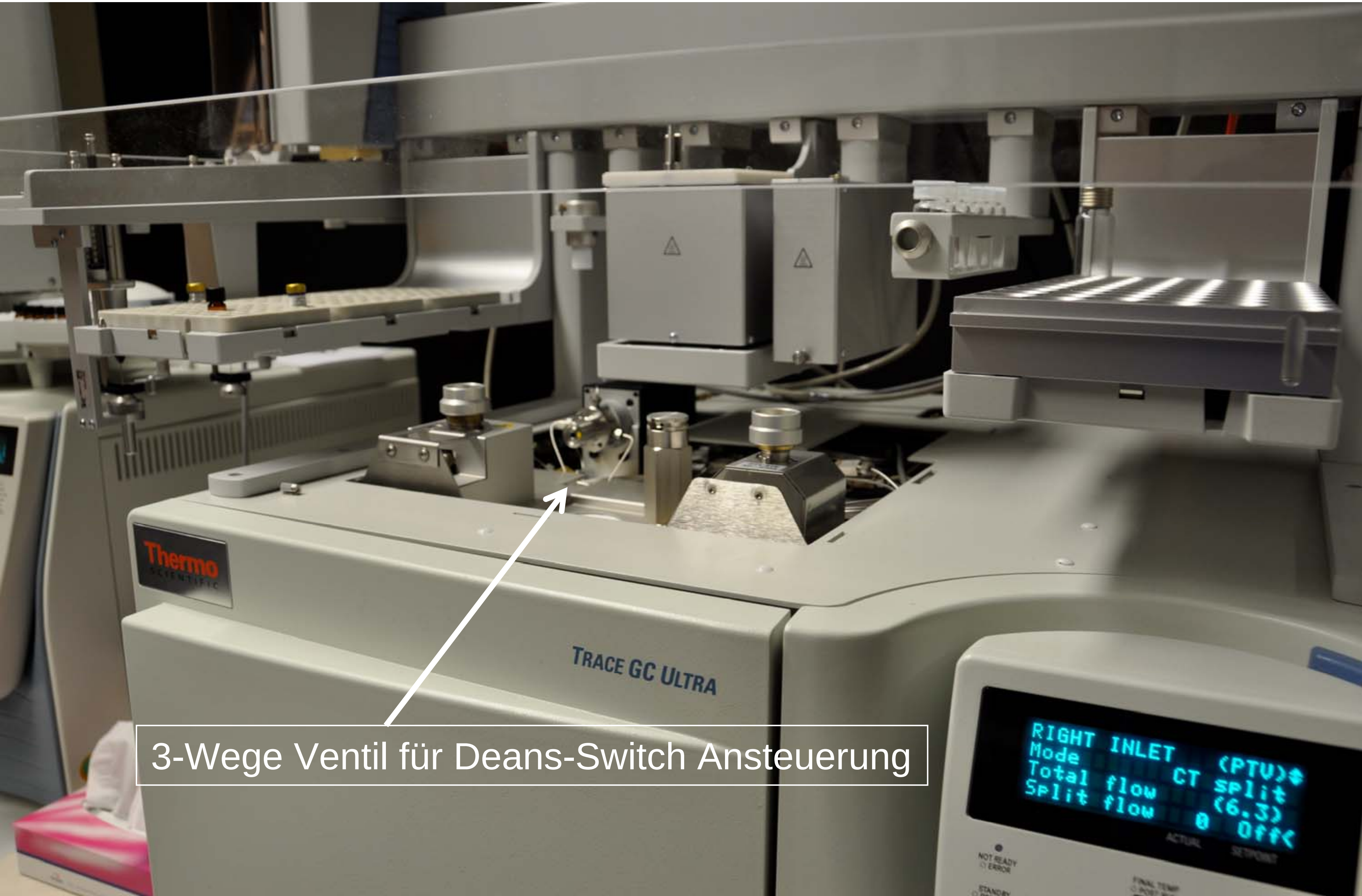
DPC: uncoated, deactivated pre-column (3 m x 0.53 mm i.d. phenylmethyl silylated)
^{1}D AC: analytical column Rtx-200MS (15 m x 0.25 mm i.d., 0.25 µm)
^{2}D AC: analytical column TG-5SilMS (30 m x 0.25 mm i.d., 0.25 µm)
RC: restrictor column (~0.75 m x 0.10 mm i.d. phenylmethyl silylated)
FID: flame ionisation detector, EPC: electronic pressure controller



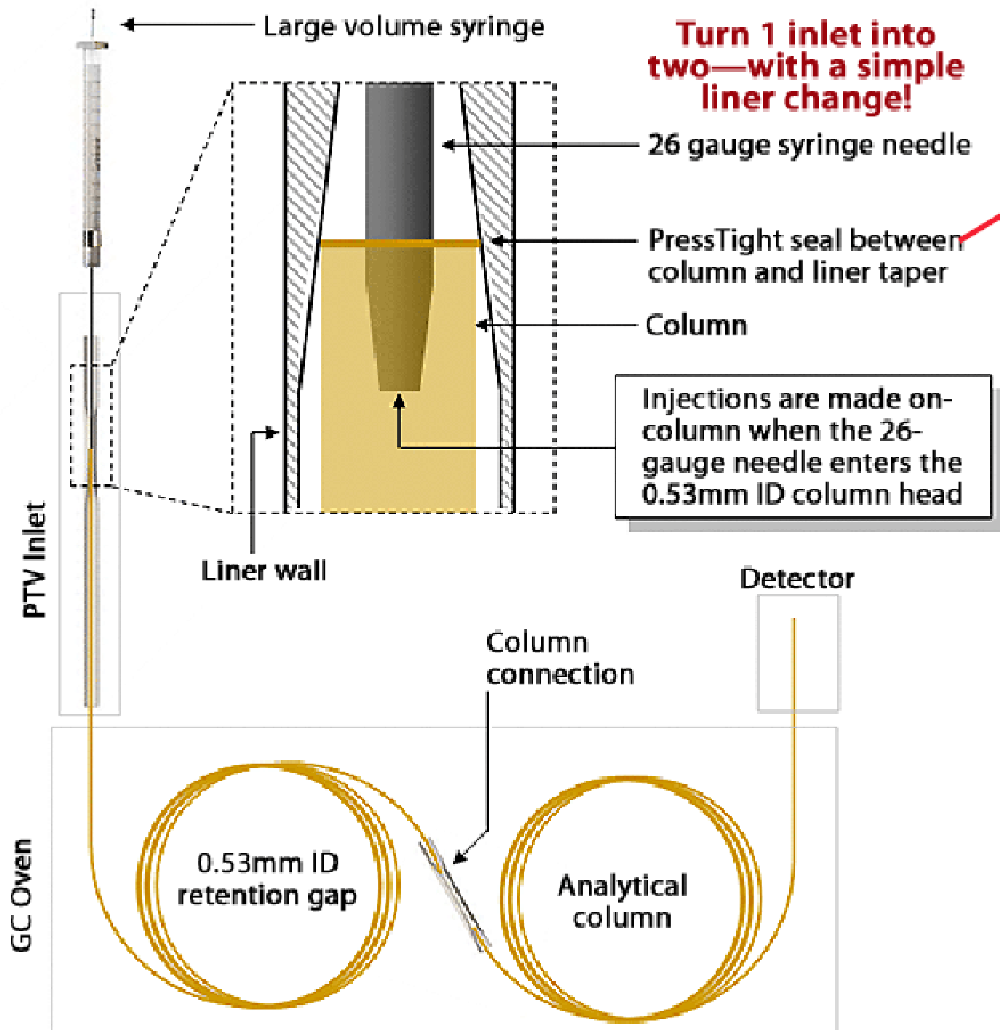
Variable Injektionstools



Ansteuerung der Deans-Schaltung



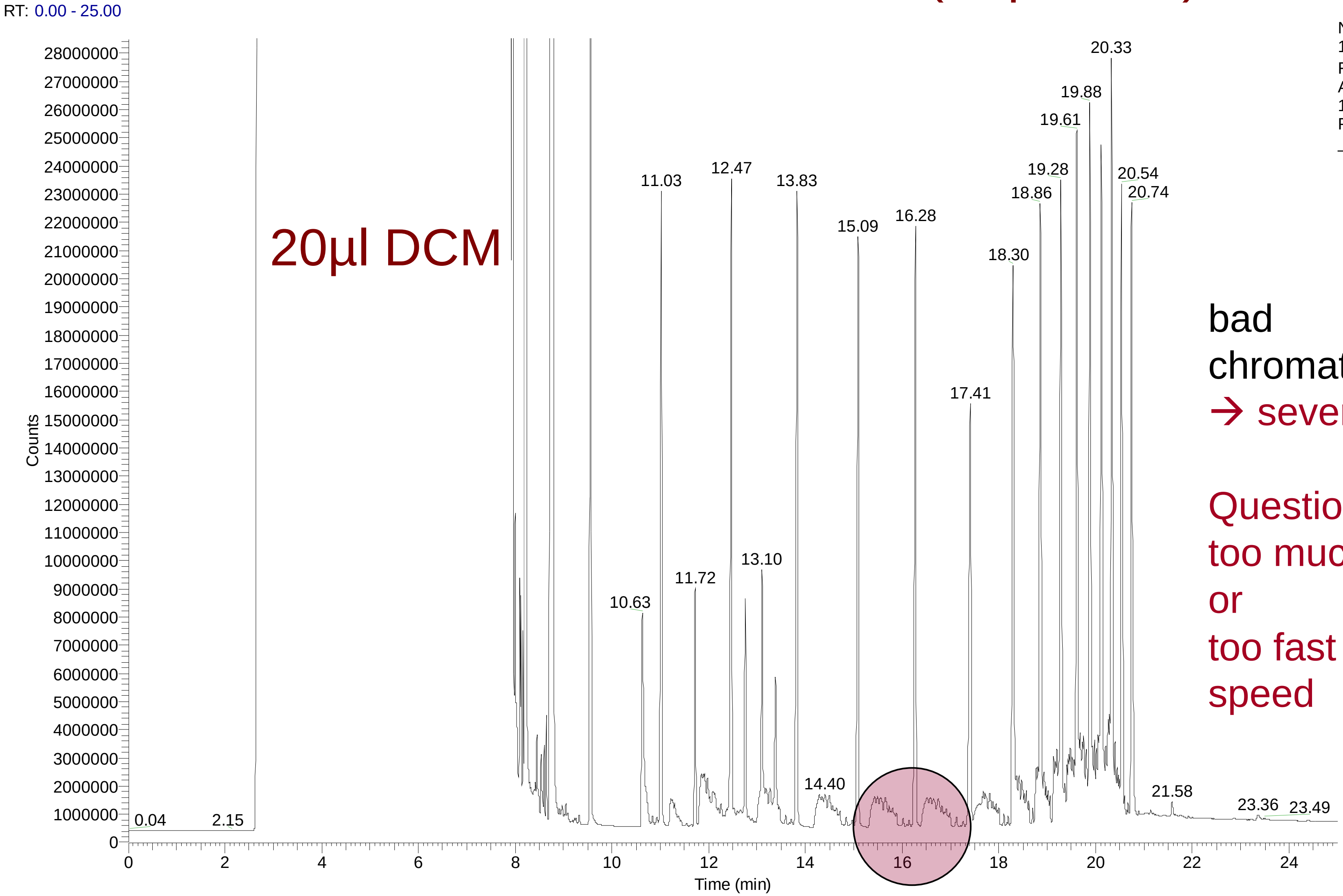
PTV On Column Injection



It is not fully a PressTight seal:
A small flow is guaranteed to avoid solvent vapours to generate a broad solvent peak in case of wrong temperature conditions

59˚C (8 min iso); He @ 2ml/min const. flow
20µl @ 2µl/sec (last 3µl w 10µl/sec)

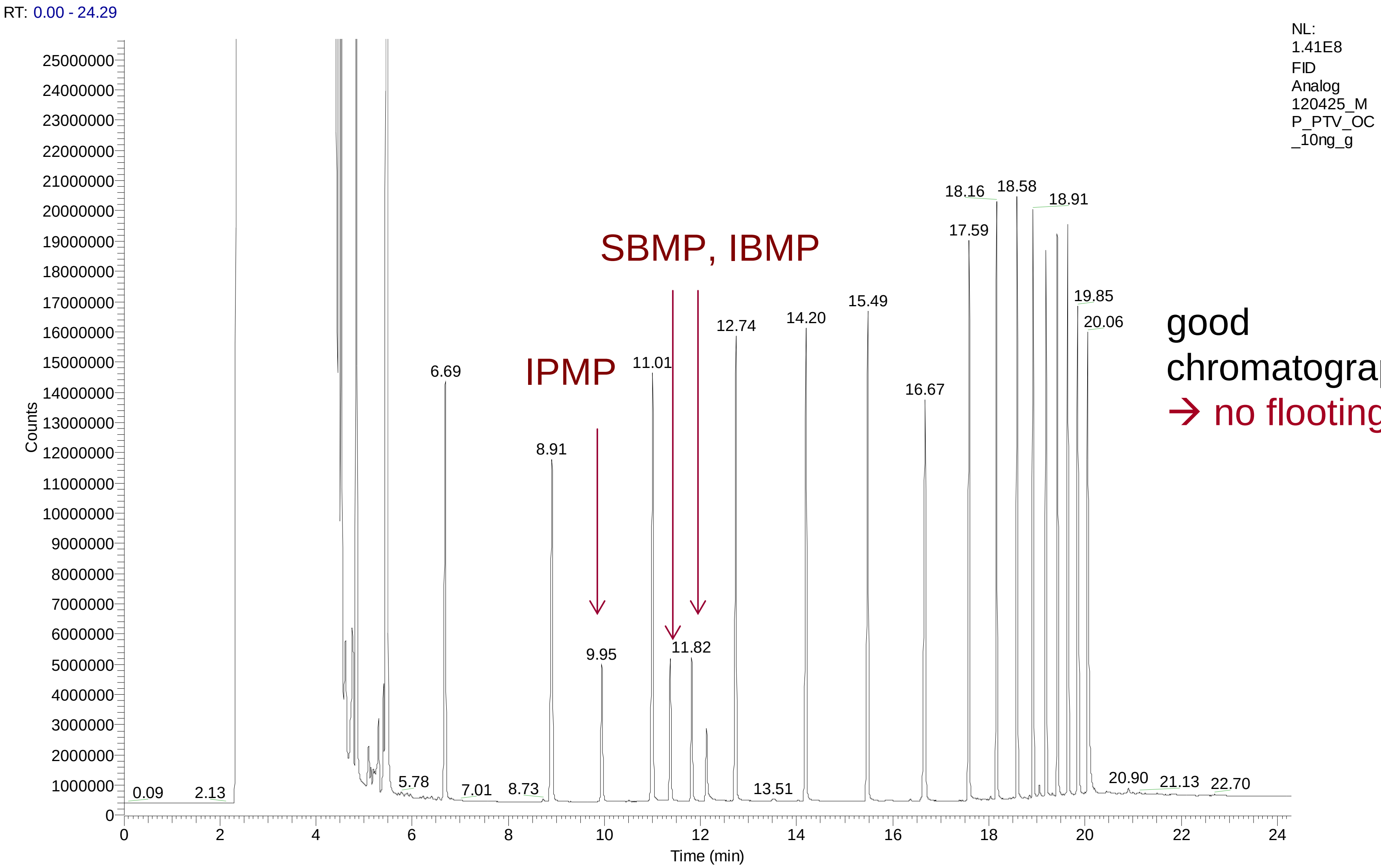
MPs und Alkanreihe in DCM (Sdp. 40°C)



bad chromatography,
→ severe flooting

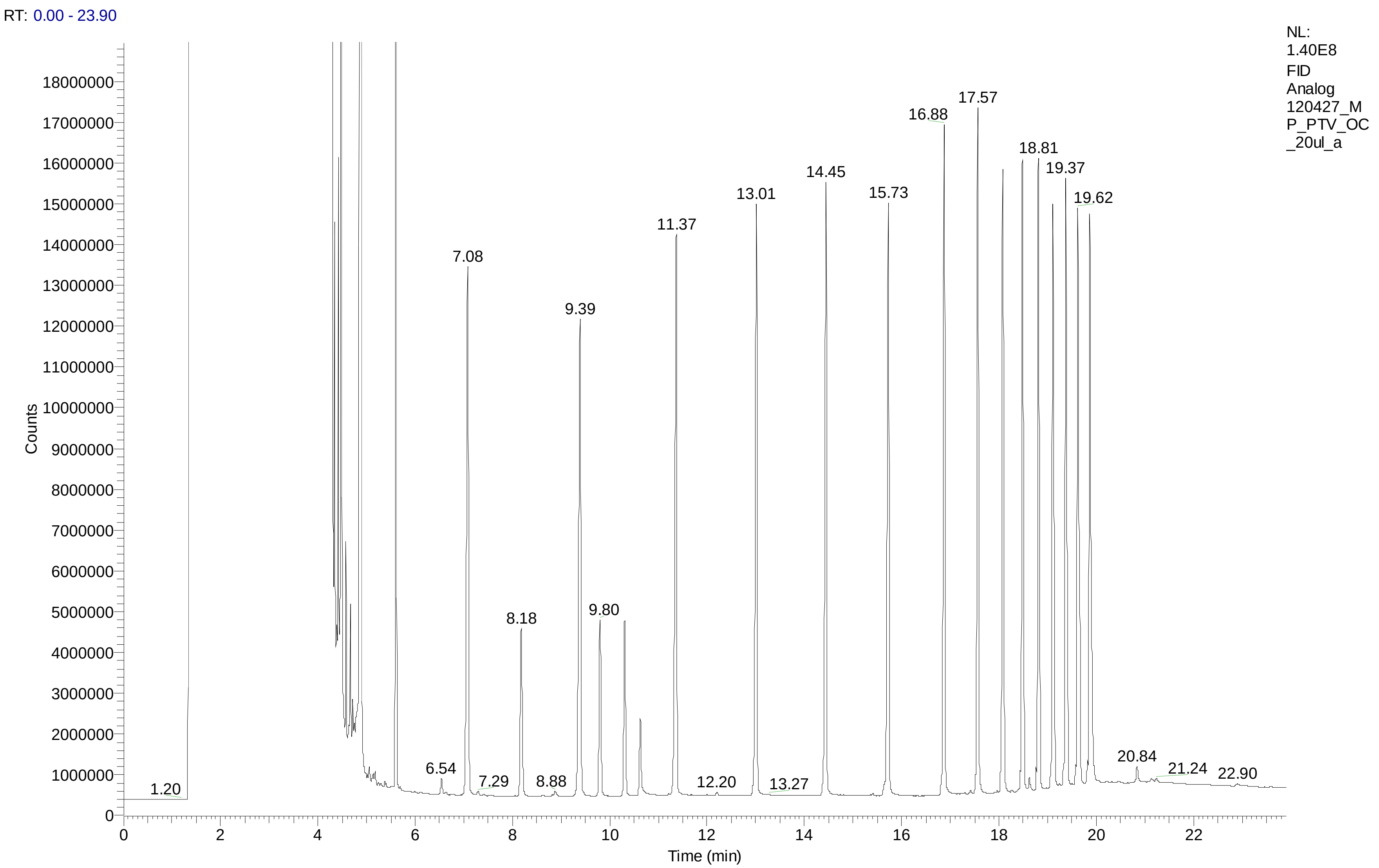
Question:
too much liquid
or
too fast injection speed

59˚C (8 min iso); He @ 2ml/min const. flow
10µl @ 2µl/sec (last 3µl w 10µl/sec)

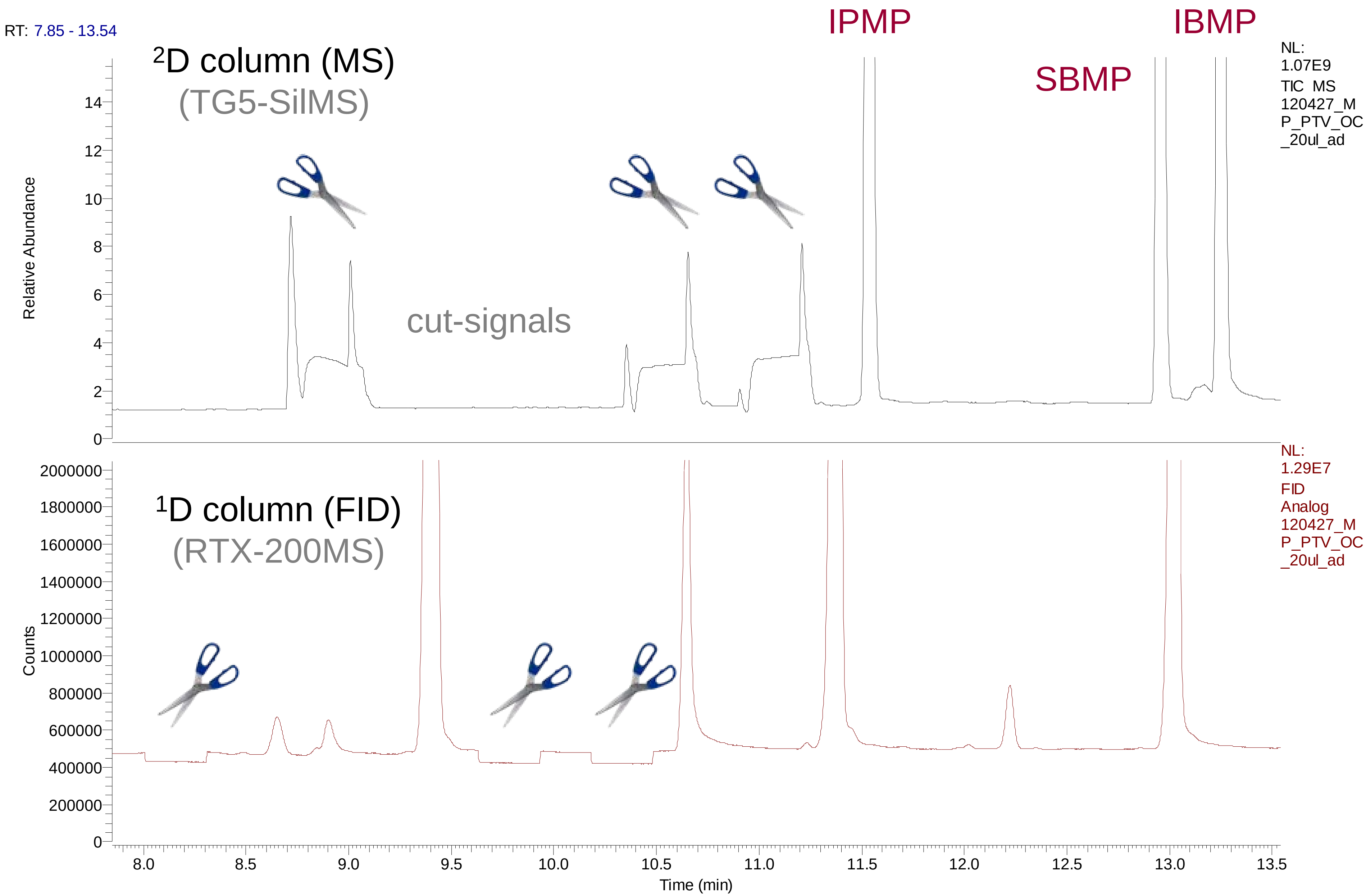


good chromatography,
→ no flooting

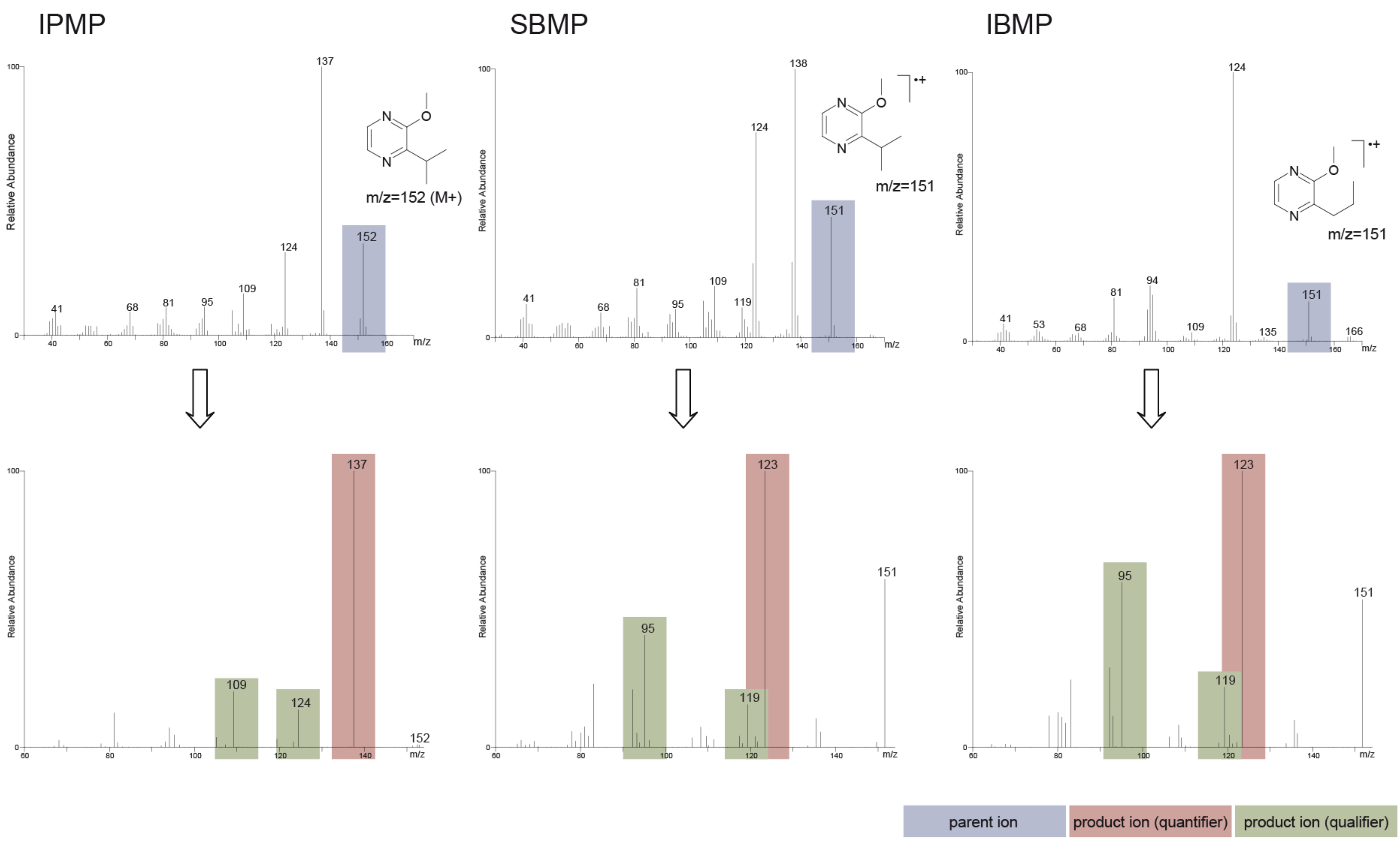
70˚C (8 min iso); 270kPa He constant pressure
20µl @ 0.5µl/sec (last 3µl w 10µl/sec)



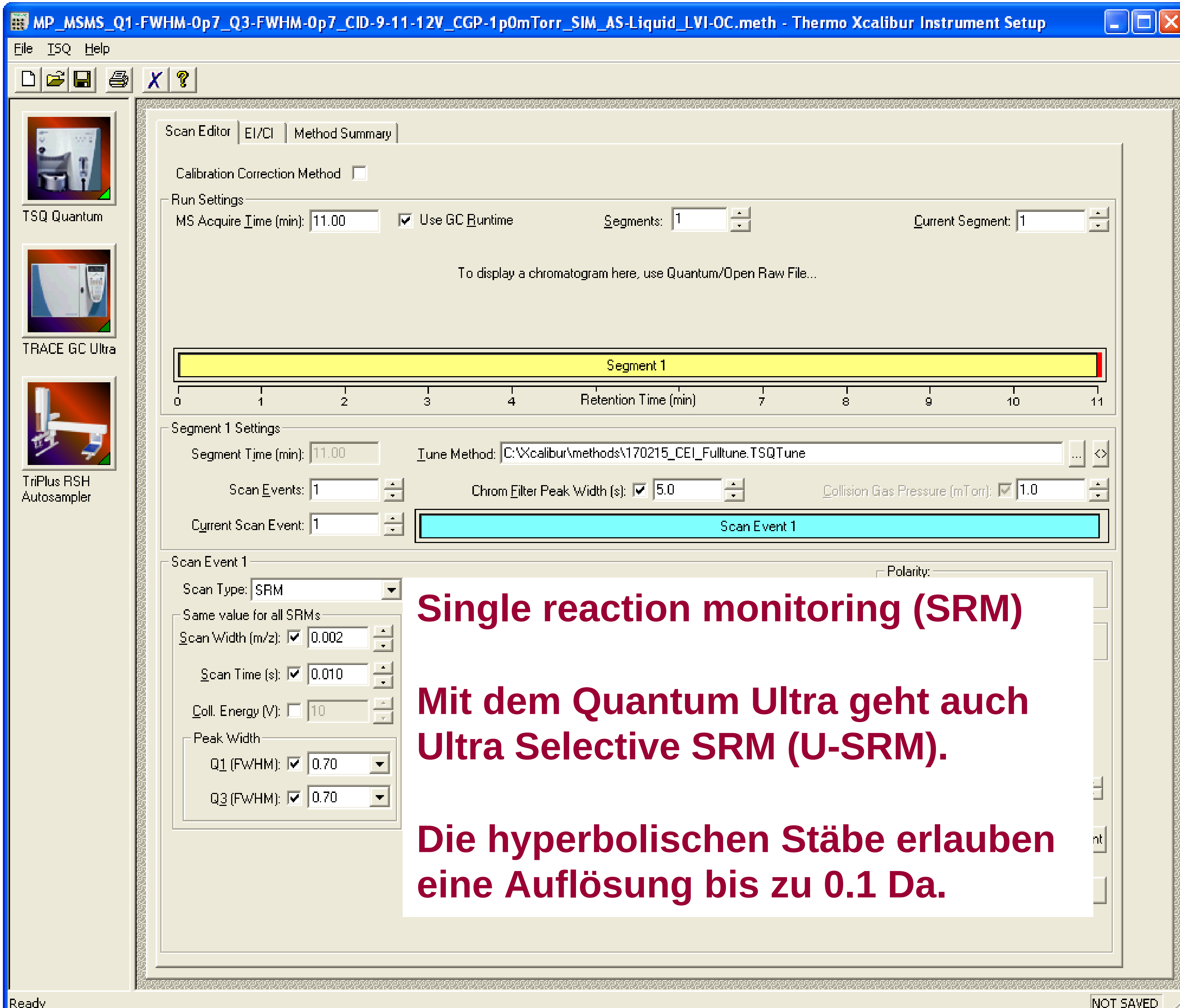
Definition der Schnittfenster



MPs: MS-MS Fragmentierung



Full scan und product scan Spektren der MPs. Qualifier und Quantifier ions für MS-MS mit entsprechenden Farben unterlegt. Strukturvorschläge für die relevanten parent ions.



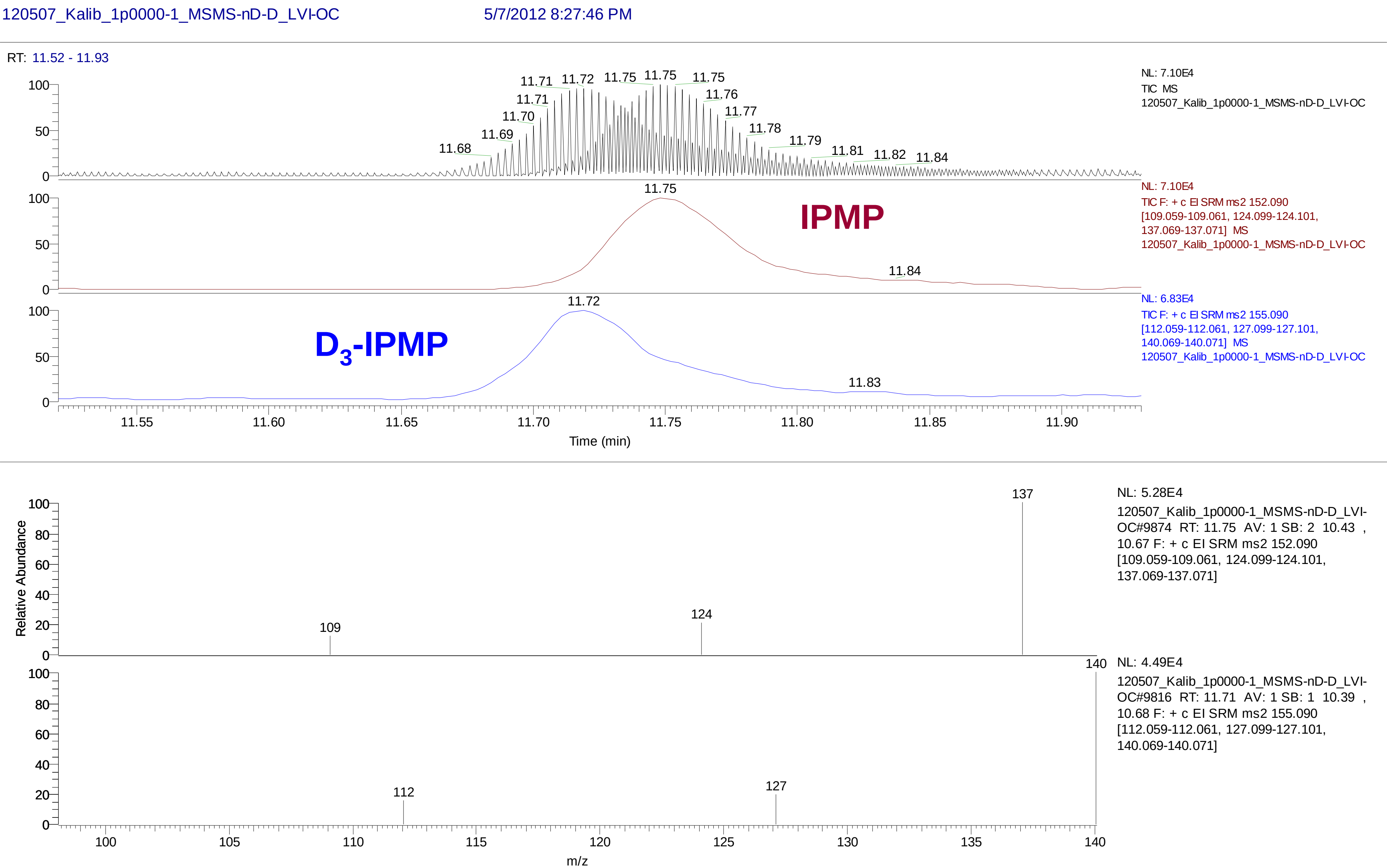
Single reaction monitoring (SRM)

Mit dem Quantum Ultra geht auch Ultra Selective SRM (U-SRM).

Die hyperbolischen Stäbe erlauben eine Auflösung bis zu 0.1 Da.

IPMP: Quan: 152 → 137

Qual: 152 → 109,124



Probe: IPMP

Thermo Xcalibur Quan Browser - Samples_20120511.XQN (Bracket 1, View All)

File View Zoom Options GoTo Help

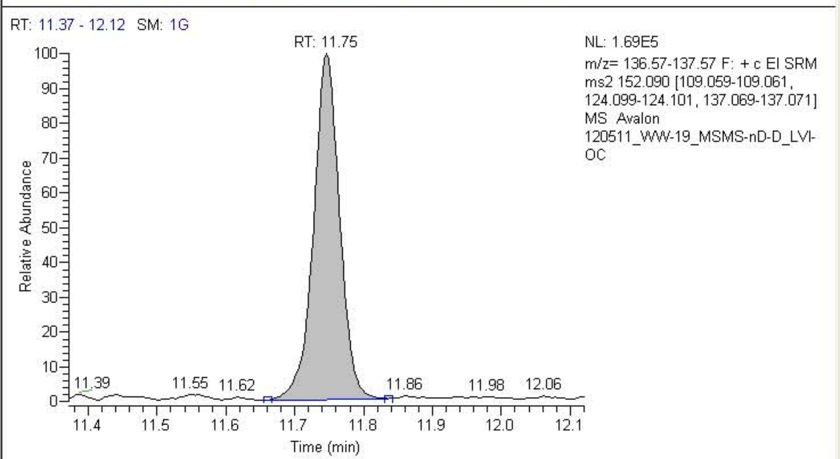
Bracket in use: Bracket 1 | Calibration File: Embedded Calibration

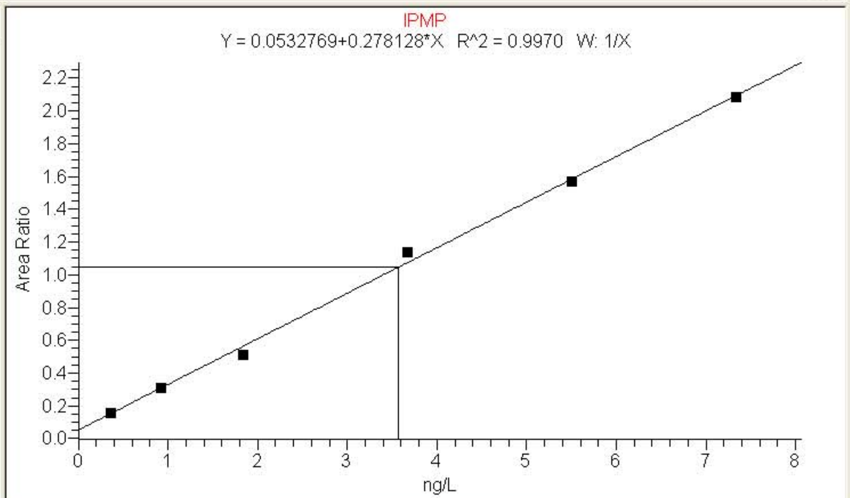
IPMP-D3, IPMP, SBMP-D3, SBMP, IBMP-D3, IBMP

	File Name	Sample Type	Sample Name	Integration Type	Area	ISTD Area	Area Ratio	Specified Amount	Calculated Amount	% Diff	%RSD-AMT	Peak Status	Level	Units
5	120507_Kalib_0p5000-1_MSMS-n	Standard	Kalib 0.5000:1; ng/L; IBMP	Method Settings	92979	182860	0.508	1.834	1.637	-10.76	0.00		0.5000:1	ng/L
6	120507_Kalib_1p0000-1_MSMS-n	Standard	Kalib 1.0000:1; ng/L; IBMP	Manual Integration	193874	170163	1.139	3.668	3.905	6.46	0.00		1.0000:1	ng/L
7	120507_Kalib_1p5000-1_MSMS-n	Standard	Kalib 2.0000:1; ng/L; IBMP	Manual Integration	287423	182866	1.572	5.501	5.460	-0.75	0.00		1.5000:1	ng/L
8	120509_Kalib_2p0000-1_MSMS-n	Standard	Kalib 2.5000:1; ng/L; IBMP	Manual Integration	419878	201254	2.086	7.335	7.310	-0.34	0.00		2.0000:1	ng/L
9	120509_Kalib_0p1000-1_MSMS-n	Standard	Kalib 0.1000:1; ng/L; IBMP	Manual Integration	30556	191563	0.160	0.367	0.382	4.08	0.00		0.1000:1	ng/L
10	120509_WW-WF_dot-1z1_MSMS-	Unknown		Manual Integration	174327	165124	1.056	NA	3.604	NA	NA		NA	ng/L
11	120511_WW-20_MSMS-nD-D_LVI	Unknown		Method Settings	319253	304462	1.049	NA	3.579	NA	NA		NA	ng/L
12	120509_WW-WF-2_dot-1z1_MSM	Unknown		Method Settings	189008	157715	1.198	NA	4.117	NA	NA		NA	ng/L
13	120509_WW-WF-2_dot-2z1_MSM	Unknown		Method Settings	279517	132522	2.109	NA	7.392	NA	NA	Response High	NA	ng/L
14	120509_WW-WF_dot-2z1_MSMS-	Unknown		Manual Integration	321325	155047	2.072	NA	7.260	NA	NA		NA	ng/L
15	120509_WW-Nativ-2_MSMS-nD-D	Unknown		Manual Integration	17952	157793	0.114	NA	0.217	NA	NA	Response Low	NA	ng/L
16	120509_WW-Blank_MSMS-nD-D_	Unknown		Manual Integration	5427	151954	0.036	NA	-0.063	NA	NA	Response Low	NA	ng/L
17	120509_WW-Nativ_MSMS-nD-D_L	Unknown		Method Settings	11621	164290	0.071	NA	0.063	NA	NA	Response Low	NA	ng/L
18	120509_WW-WF-ELISA_dot-1z1_	Unknown		Manual Integration	330592	307264	1.076	NA	3.677	NA	NA		NA	ng/L
19	120511_WW-18_MSMS-nD-D_LVI	Unknown		Manual Integration	173876	638155	0.272	NA	0.788	NA	NA		NA	ng/L
20	120511_WW-19_MSMS-nD-D_LVI	Unknown		Method Settings	442426	422942	1.046	NA	3.570	NA	NA		NA	ng/L
21	120514_WW-06_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	37356	NF	NA	NF	NF	NF	Not Found	NA	ng/L
22	120514_WW-03_MSMS-nD-D_LVI	Unknown		Manual Integration	73766	393490	0.187	NA	0.482	NA	NA		NA	ng/L
23	120514_WW-04_MSMS-nD-D_LVI	Unknown		Manual Integration	84839	455597	0.186	NA	0.478	NA	NA		NA	ng/L
24	120514_WW-05_MSMS-nD-D_LVI	Unknown		Method Settings	22198	105400	0.211	NA	0.566	NA	NA		NA	ng/L
25	120514_WW-23_MSMS-nD-D_LVI	Unknown		Manual Integration	80818	319902	0.253	NA	0.717	NA	NA		NA	ng/L
26	120514_WW-21_MSMS-nD-D_LVI	Unknown		Method Settings	70244	430442	0.163	NA	0.395	NA	NA		NA	ng/L
27	120514_WW-22_MSMS-nD-D_LVI	Unknown		Method Settings	79057	298916	0.264	NA	0.759	NA	NA		NA	ng/L

All / Standards / QCs / Blanks / Unknowns

120511_WW-19_MSMS-nD-D_LVI-OC (Method Settings) 5/11/2012 1:04:20 PM





NUM 6/6/2012 5:12 PM

Probe: SBMP

Thermo Xcalibur Quan Browser - Samples_20120511.XQN (Bracket 1, View All)

File View Zoom Options GoTo Help

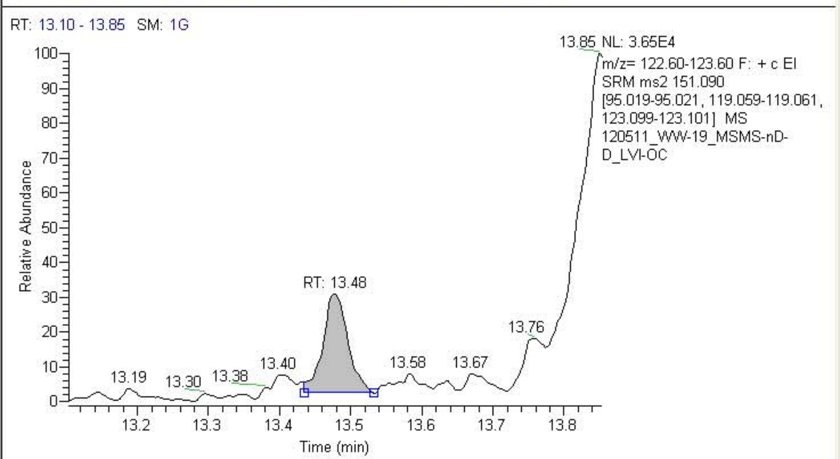
Bracket in use: Bracket 1 | Calibration File: Embedded Calibration

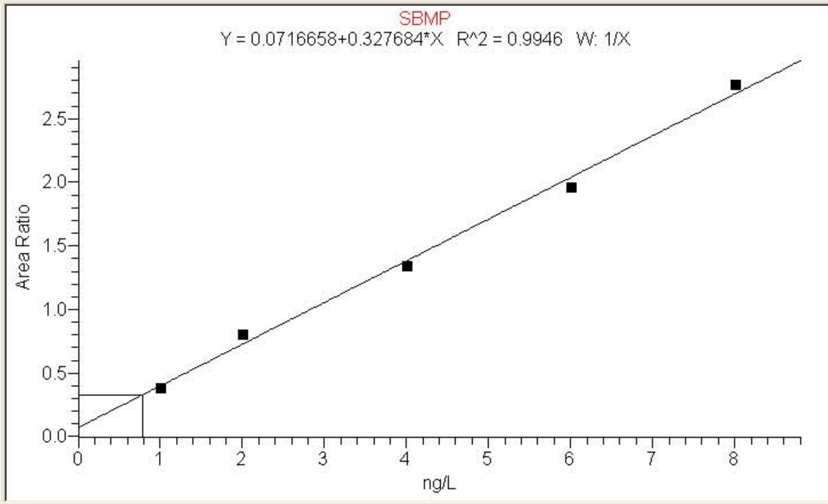
IPMP-D3
IPMP
SBMP-D3
SBMP
IBMP-D3
IBMP

	File Name	Sample Type	Sample Name	Integration Type	Area	ISTD Area	Area Ratio	Specified Amount	Calculated Amount	% Diff	%RSD-AMT	Peak Status	Level	Units
5	120507_Kalib_0p5000-1_MSMS-n	Standard	Kalib 0.5000:1; ng/L; IBMP	Manual Integration	37954	47503	0.799	2.003	2.220	10.81	0.00		0.5000:1	ng/L
6	120507_Kalib_1p0000-1_MSMS-n	Standard	Kalib 1.0000:1; ng/L; IBMP	Manual Integration	60408	45145	1.338	4.005	3.865	-3.50	0.00		1.0000:1	ng/L
7	120507_Kalib_1p5000-1_MSMS-n	Standard	Kalib 2.0000:1; ng/L; IBMP	Manual Integration	89184	45455	1.962	6.008	5.769	-3.98	0.00		1.5000:1	ng/L
8	120509_Kalib_2p0000-1_MSMS-n	Standard	Kalib 2.5000:1; ng/L; IBMP	Manual Integration	139445	50344	2.770	8.010	8.234	2.80	0.00	Response High	2.0000:1	ng/L
9	120509_Kalib_0p1000-1_MSMS-n	Standard	Kalib 0.1000:1; ng/L; IBMP	Manual Integration	NF	36710	NF	0.401	NF	NF	NF	Not Found	0.1000:1	ng/L
10	120509_WW-WF_dot-1z1_MSMS-	Unknown		Method Settings	61948	42628	1.453	NA	4.216	NA	NA		NA	ng/L
11	120511_WW-20_MSMS-nD-D_LVI	Unknown		Manual Integration	24065	70120	0.343	NA	0.829	NA	NA	Response Low	NA	ng/L
12	120509_WW-WF-2_dot-1z1_MSM	Unknown		Manual Integration	49974	32169	1.554	NA	4.522	NA	NA		NA	ng/L
13	120509_WW-WF-2_dot-2z1_MSM	Unknown		Method Settings	110512	33786	3.271	NA	9.763	NA	NA	Response High	NA	ng/L
14	120509_WW-WF_dot-2z1_MSMS-	Unknown		Manual Integration	121837	37598	3.240	NA	9.670	NA	NA	Response High	NA	ng/L
15	120509_WW-Nativ-2_MSMS-nD-D	Unknown		Manual Integration	NF	38786	NF	NA	NF	NF	NF	Not Found	NA	ng/L
16	120509_WW-Blank_MSMS-nD-D_	Unknown		Manual Integration	NF	34232	NF	NA	NF	NF	NF	Not Found	NA	ng/L
17	120509_WW-Nativ_MSMS-nD-D_L	Unknown		Manual Integration	NF	34044	NF	NA	NF	NF	NF	Not Found	NA	ng/L
18	120509_WW-WF-ELISA_dot-1z1_	Unknown		Method Settings	191998	127658	1.504	NA	4.371	NA	NA		NA	ng/L
19	120511_WW-18_MSMS-nD-D_LVI	Unknown		Manual Integration	18541	117882	0.157	NA	0.261	NA	NA	Response Low	NA	ng/L
20	120511_WW-19_MSMS-nD-D_LVI	Unknown		Manual Integration	25497	77746	0.328	NA	0.782	NA	NA	Response Low	NA	ng/L
21	120514_WW-06_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	8282	NF	NA	NF	NF	NF	Not Found	NA	ng/L
22	120514_WW-03_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	95111	NF	NA	NF	NF	NF	Not Found	NA	ng/L
23	120514_WW-04_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	105832	NF	NA	NF	NF	NF	Not Found	NA	ng/L
24	120514_WW-05_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	25292	NF	NA	NF	NF	NF	Not Found	NA	ng/L
25	120514_WW-23_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	71786	NF	NA	NF	NF	NF	Not Found	NA	ng/L
26	120514_WW-21_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	101507	NF	NA	NF	NF	NF	Not Found	NA	ng/L
27	120514_WW-22_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	67327	NF	NA	NF	NF	NF	Not Found	NA	ng/L

All | Standards | QCs | Blanks | Unknowns

120511_WW-19_MSMS-nD-D_LVI-OC (Manual Integration) 5/11/2012 1:04:20 PM





NUM 6/6/2012 5:12 PM

Probe: IBMP

Thermo Xcalibur Quan Browser - Samples_20120511.XQN (Bracket 1, View All)

File View Zoom Options GoTo Help

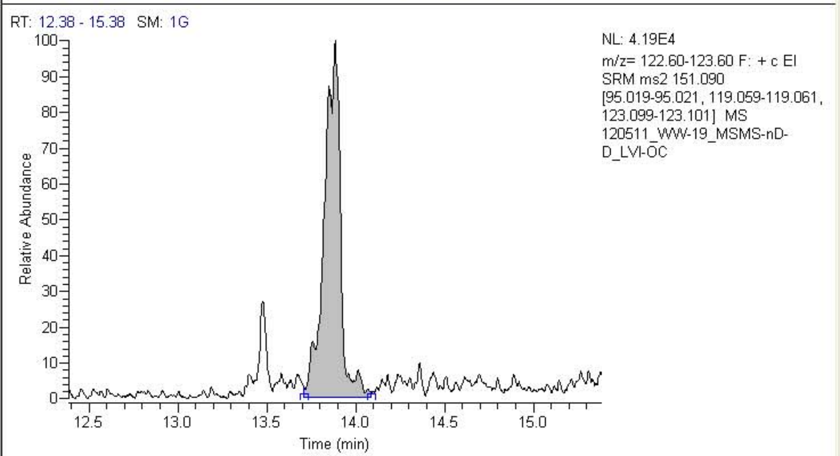
Bracket in use: Bracket 1 Calibration File: Embedded Calibration

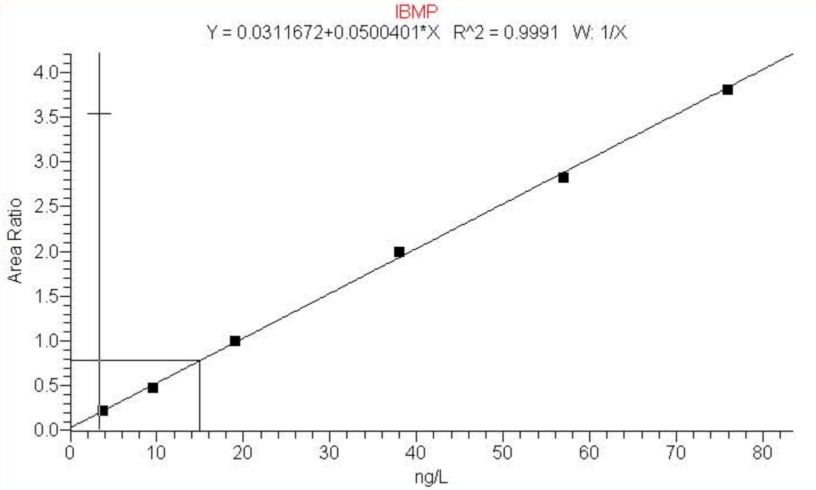
IPMP-D3
IPMP
SBMP-D3
SBMP
IBMP-D3
IBMP

	File Name	Sample Type	Sample Name	Integration Type	Area	ISTD Area	Area Ratio	Specified Amount	Calculated Amount	% Diff	%RSD-AMT	Peak Status	Level	Units
5	120507_Kalib_0p5000-1_MSMS-n	Standard	Kalib 0.5000:1; ng/L; IBMP	Method Settings	151604	150484	1.007	18.975	19.510	2.82	0.00		0.5000:1	ng/L
6	120507_Kalib_1p0000-1_MSMS-n	Standard	Kalib 1.0000:1; ng/L; IBMP	Manual Integration	303616	151821	2.000	37.950	39.342	3.67	0.00		1.0000:1	ng/L
7	120507_Kalib_1p5000-1_MSMS-n	Standard	Kalib 2.0000:1; ng/L; IBMP	Manual Integration	476657	168769	2.824	56.925	55.818	-1.94	0.00		1.5000:1	ng/L
8	120509_Kalib_2p0000-1_MSMS-n	Standard	Kalib 2.5000:1; ng/L; IBMP	Method Settings	656152	172202	3.810	75.900	75.523	-0.50	0.00		2.0000:1	ng/L
9	120509_Kalib_0p1000-1_MSMS-n	Standard	Kalib 0.1000:1; ng/L; IBMP	Manual Integration	32889	147451	0.223	3.795	3.835	1.04	0.00		0.1000:1	ng/L
10	120509_WW-WF_dot-1z1_MSMS-	Unknown		Method Settings	379675	192993	1.967	NA	38.692	NA	NA		NA	ng/L
11	120511_WW-20_MSMS-nD-D_LVI	Unknown		Manual Integration	152588	207718	0.735	NA	14.057	NA	NA		NA	ng/L
12	120509_WW-WF-2_dot-1z1_MSM	Unknown		Manual Integration	330881	184498	1.793	NA	35.217	NA	NA		NA	ng/L
13	120509_WW-WF-2_dot-2z1_MSM	Unknown		Manual Integration	633776	157226	4.031	NA	79.932	NA	NA	Response High	NA	ng/L
14	120509_WW-WF_dot-2z1_MSMS-	Unknown		Method Settings	648292	174835	3.708	NA	73.478	NA	NA		NA	ng/L
15	120509_WW-Nativ-2_MSMS-nD-D	Unknown		Manual Integration	NF	202526	NF	NA	NF	NF	NF	Not Found	NA	ng/L
16	120509_WW-Blank_MSMS-nD-D_	Unknown		Manual Integration	NF	177780	NF	NA	NF	NF	NF	Not Found	NA	ng/L
17	120509_WW-Nativ_MSMS-nD-D_L	Unknown		Manual Integration	NF	163612	NF	NA	NF	NF	NF	Not Found	NA	ng/L
18	120509_WW-WF-ELISA_dot-1z1_	Unknown		Method Settings	1533766	757612	2.024	NA	39.834	NA	NA		NA	ng/L
19	120511_WW-18_MSMS-nD-D_LVI	Unknown		Manual Integration	88490	315527	0.280	NA	4.982	NA	NA		NA	ng/L
20	120511_WW-19_MSMS-nD-D_LVI	Unknown		Manual Integration	264279	338804	0.780	NA	14.965	NA	NA		NA	ng/L
21	120514_WW-06_MSMS-nD-D_LVI	Unknown		Manual Integration	NF	53267	NF	NA	NF	NF	NF	Not Found	NA	ng/L
22	120514_WW-03_MSMS-nD-D_LVI	Unknown		Manual Integration	112227	455151	0.247	NA	4.305	NA	NA		NA	ng/L
23	120514_WW-04_MSMS-nD-D_LVI	Unknown		Manual Integration	111374	474215	0.235	NA	4.071	NA	NA		NA	ng/L
24	120514_WW-05_MSMS-nD-D_LVI	Unknown		Manual Integration	29679	131346	0.226	NA	3.893	NA	NA		NA	ng/L
25	120514_WW-23_MSMS-nD-D_LVI	Unknown		Manual Integration	30359	216213	0.140	NA	2.183	NA	NA	Response Low	NA	ng/L
26	120514_WW-21_MSMS-nD-D_LVI	Unknown		Manual Integration	31526	283089	0.111	NA	1.603	NA	NA	Response Low	NA	ng/L
27	120514_WW-22_MSMS-nD-D_LVI	Unknown		Manual Integration	76832	336501	0.228	NA	3.940	NA	NA		NA	ng/L

All / Standards / QCs / Blanks / Unknowns

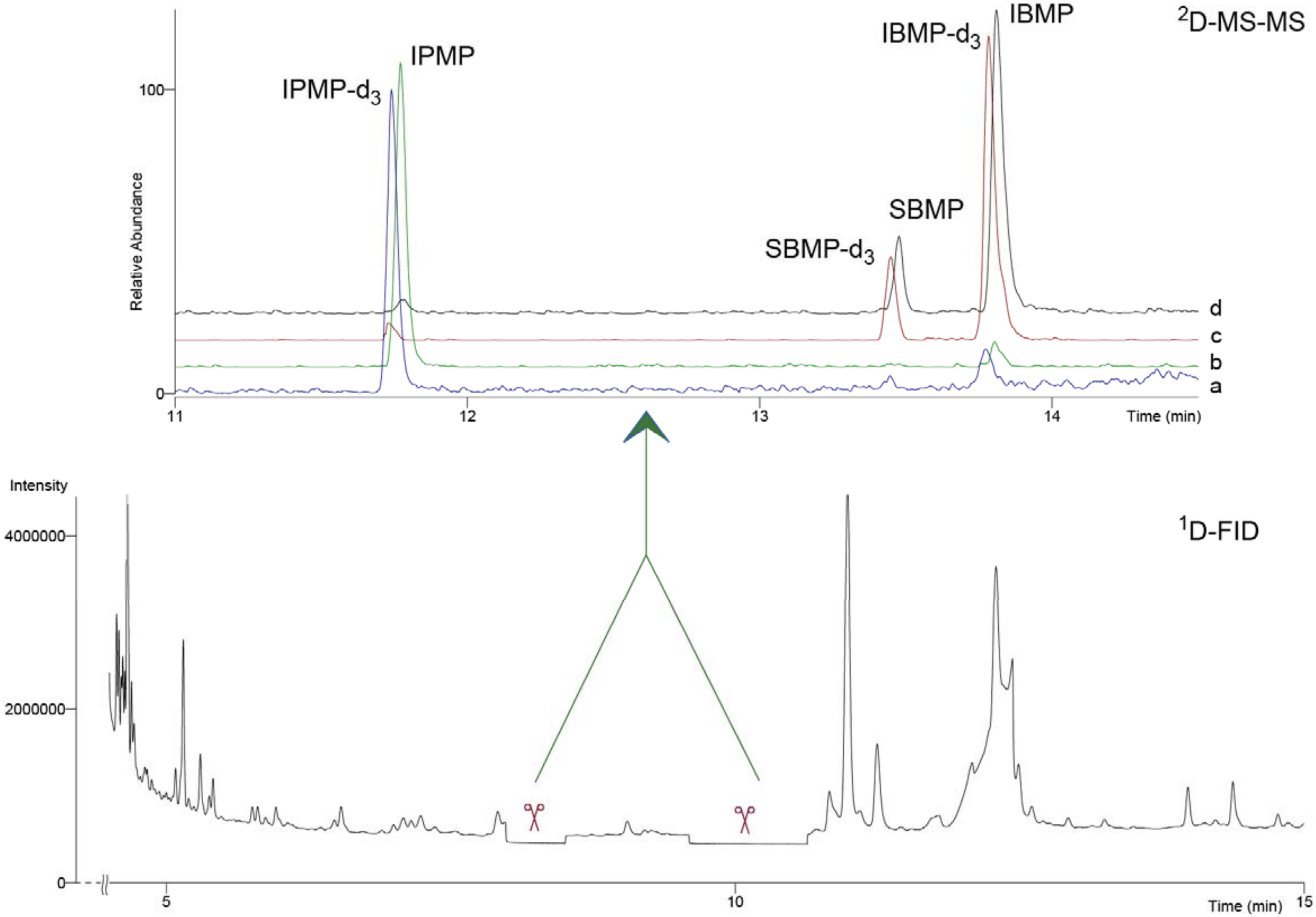
120511_WW-19_MSMS-nD-D_LVI-OC (Manual Integration) 5/11/2012 1:04:20 PM





x: 3.29, y: 3.53 NUM 6/6/2012 5:13 PM

MDGC-QqQ-MS: MPs-Analysenbeispiel



[1]D GC Trennung (FID) mit heart-cuts eines Weinextraktes, spiked mit 4 ng/L (IPMP, SBMP) und 40 ng/L (IBMP) sowie den Deuterierten MPs (unten). Im Bild oben sind die Ionenspuren dargestellt (m/z; Quantifier und Qualifier) für MPs: a) 140, 127, 112; b) 137, 124, 109; c) 126, 119, 98; d) 123, 119, 95

MDGC-SIM-MS *versus* MDGC-QqQ-MS

In der Regel ergaben beide Verfahren vergleichbare Ergebnisse für die in einer weinbaulichen und oenologischen Studie untersuchten Proben !

(Ergebnisse z.T. in *das deutsche weinmagazin*, Juni 2012, S. 30-34)

Allerdings wurden mit SIM-MS einige Proben mit **auffälligen** und **unplausiblen** SBMP-Gehalten gemessen.

→ **Hier ergab die MS-MS Messung, dass es sich um falsch-positive Werte handelte !**

Table 1. Matrix effect for MP analysis with critical samples [ng/L]

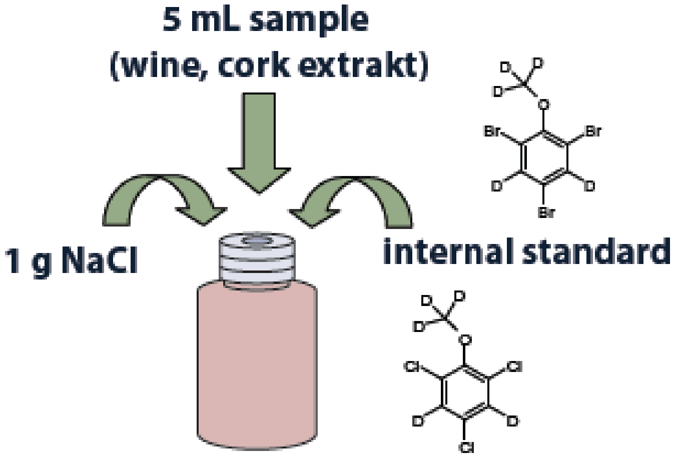
Sample	IPMP		SBMP		IBMP	
Sauv. Blanc A	2.9[a]	0.7[b]	3.9[a]	n.d.[b]	4.8[a]	2.2[b]
Sauv. Blanc B	2.1[a]	0.6[b]	2.2[a]	n.d.[b]	5.6[a]	5.4[b]

n.d. not detectable; [a] LVOC-MDGC-SIM-MS; [b] PTV-LVOC-MDGC-MS-MS

Haloanisole mit HS-SPME-MDGC-ECD / QqQ-MS

- 2,4,6-Trichloranisol (TCA) als Leitsubstanz für muffige Korktöne; daneben auch Tetrachloranisol und 2,4,6-Tribromanisol (TeCA und TBA)

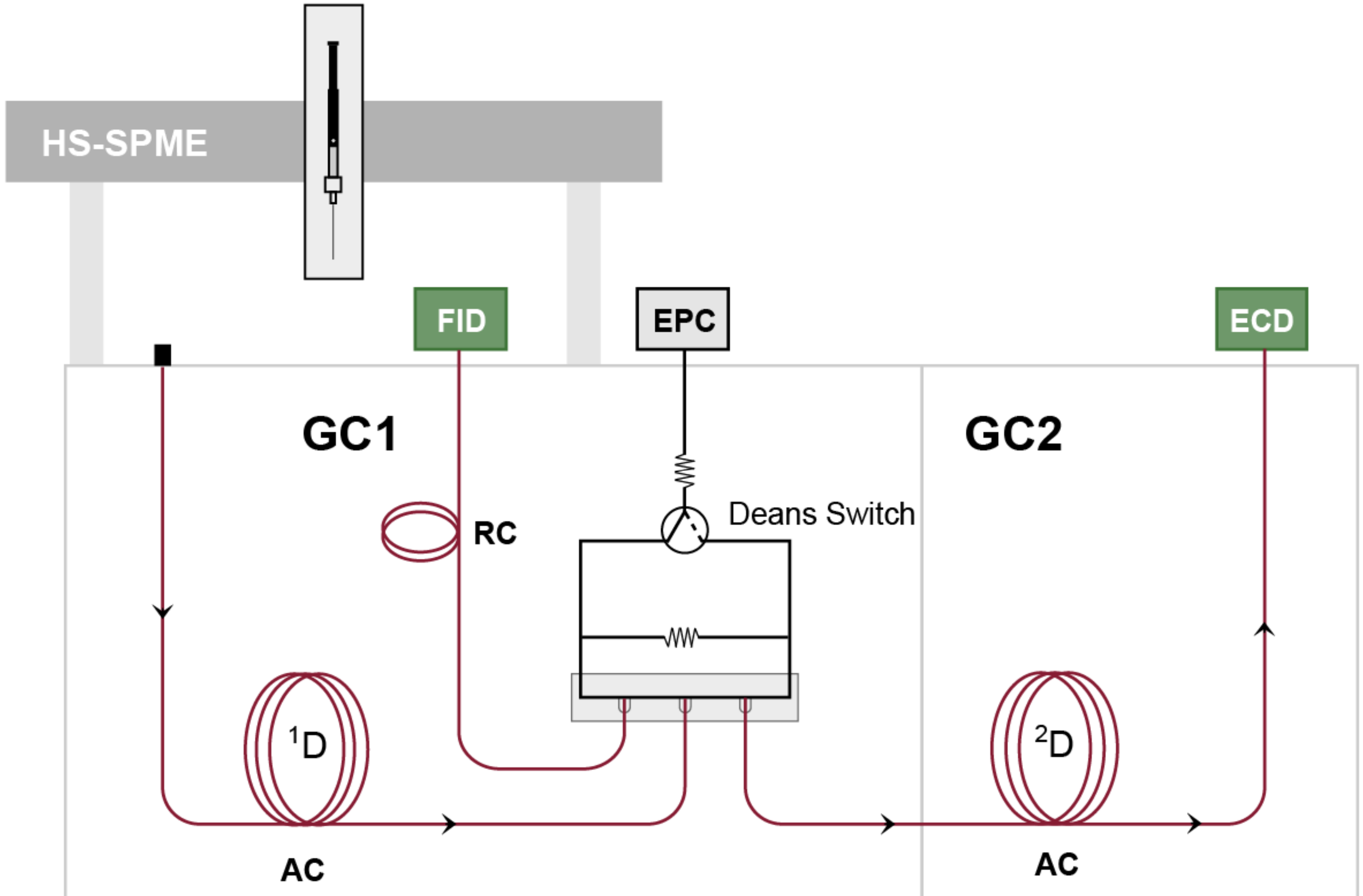
- Routineanalytik mit HS-SPME-¹D-GC-ECD, jüngst wegen Matrixproblemen in Wein umgestellt auf HS-SPME-MDGC-ECD
(Poster ISCC 2012, Riva del Garda)



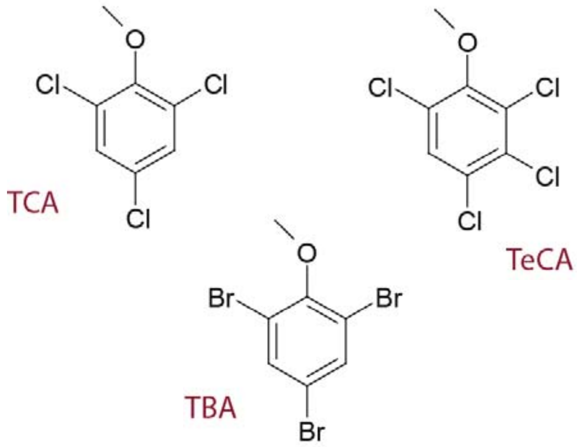
- Quantifizierung über D_5-Isotopenstandards (SIDA)

Haloanisole mit HS-SPME-MDGC-ECD

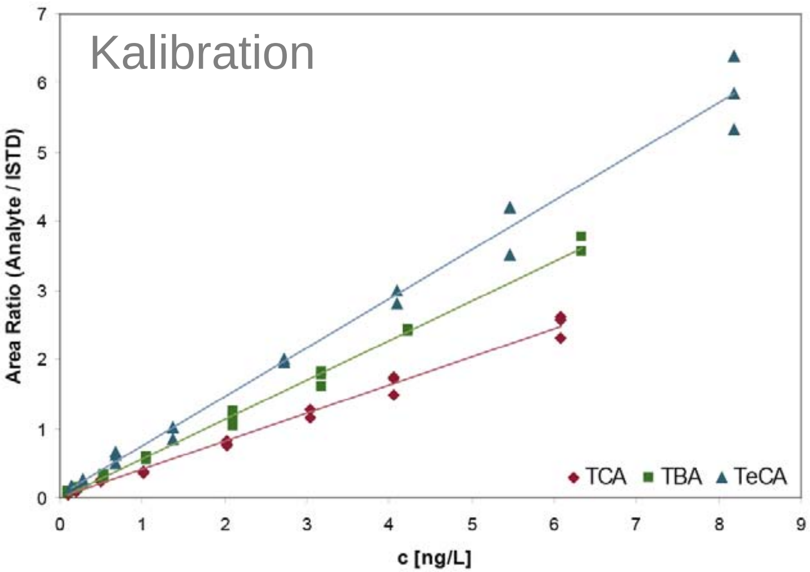
Stable Isotope Dilution Analysis of Corky Off-Flavour Compounds at Ultra Trace Level with Multidimensional Gas Chromatography–Electron Capture Detection



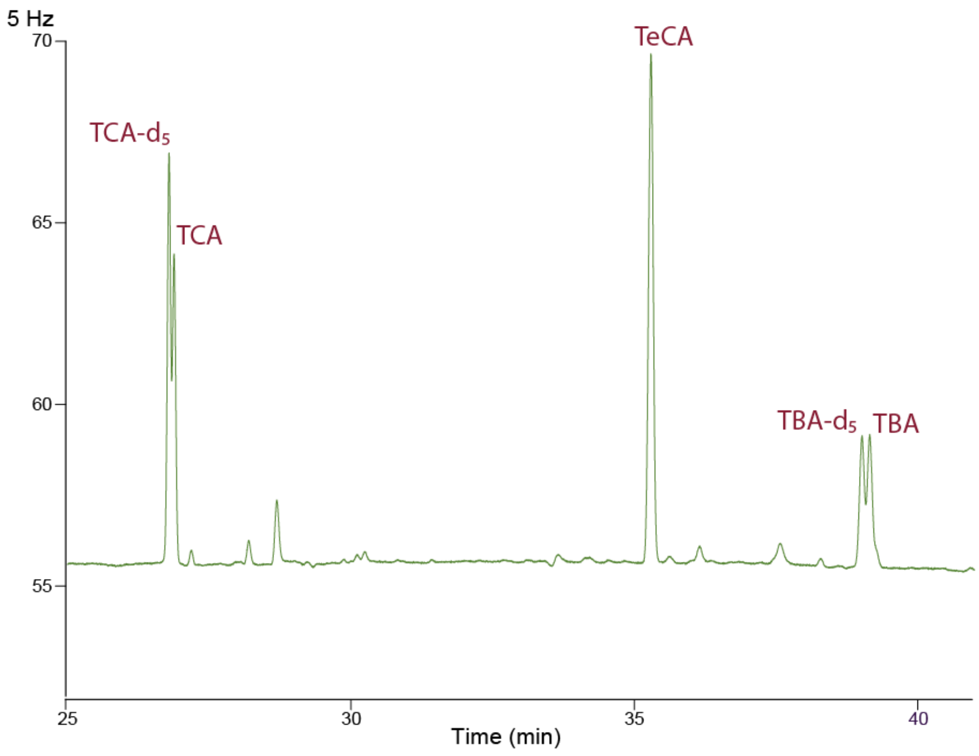
Haloanisole mit HS-SPME-MDGC-ECD



Geruchsschwellenwerte z.T. bei 1-3 ng/L Wein; Qualitätskontrolle bis < 1 ng/L



Standard-Chromatogramm



Haloanisole mit HS-SPME-MDGC-ECD

Kork-Extrakt
(10% vol. Ethanol)

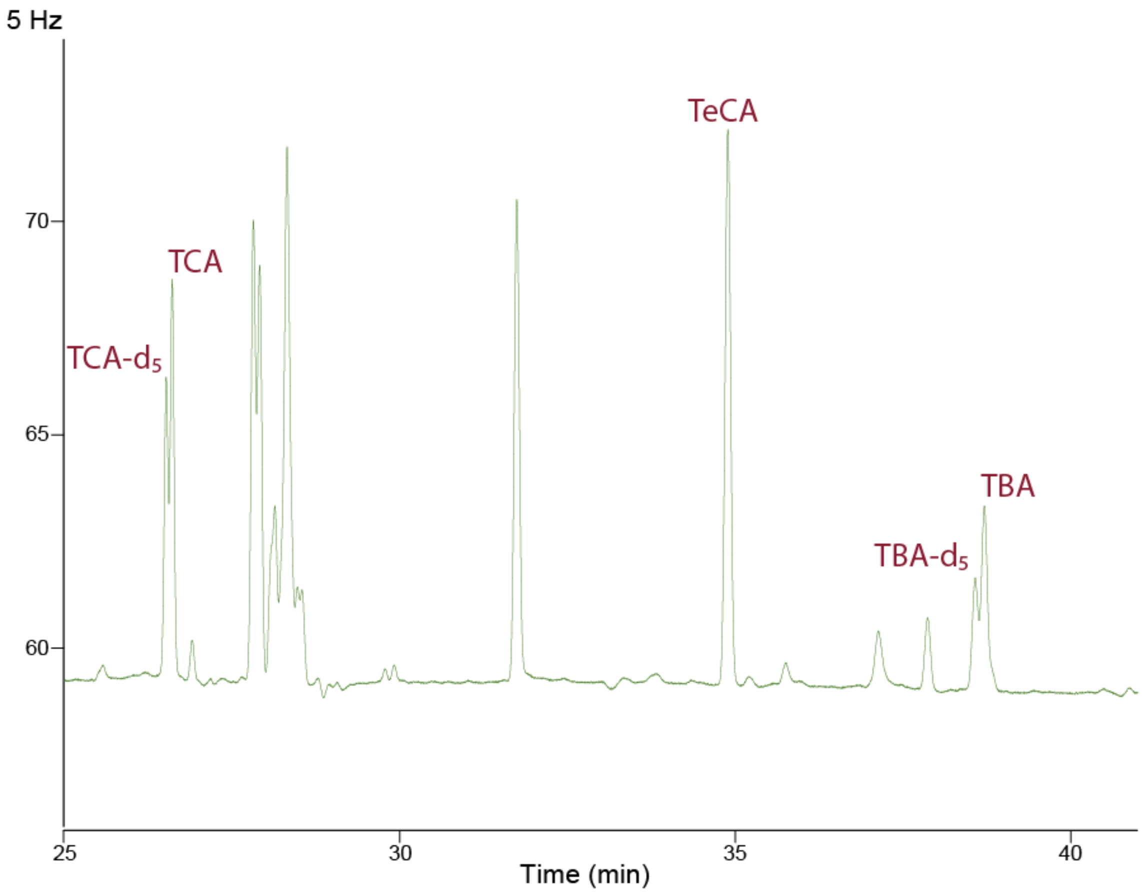


Figure 5 HS–SPME–MDGC–ECD chromatogram of a cork extract sample (3 ng/L)

Haloanisole mit HS-SPME-MDGC-ECD

Wein
(ca. 12% vol. Ethanol)

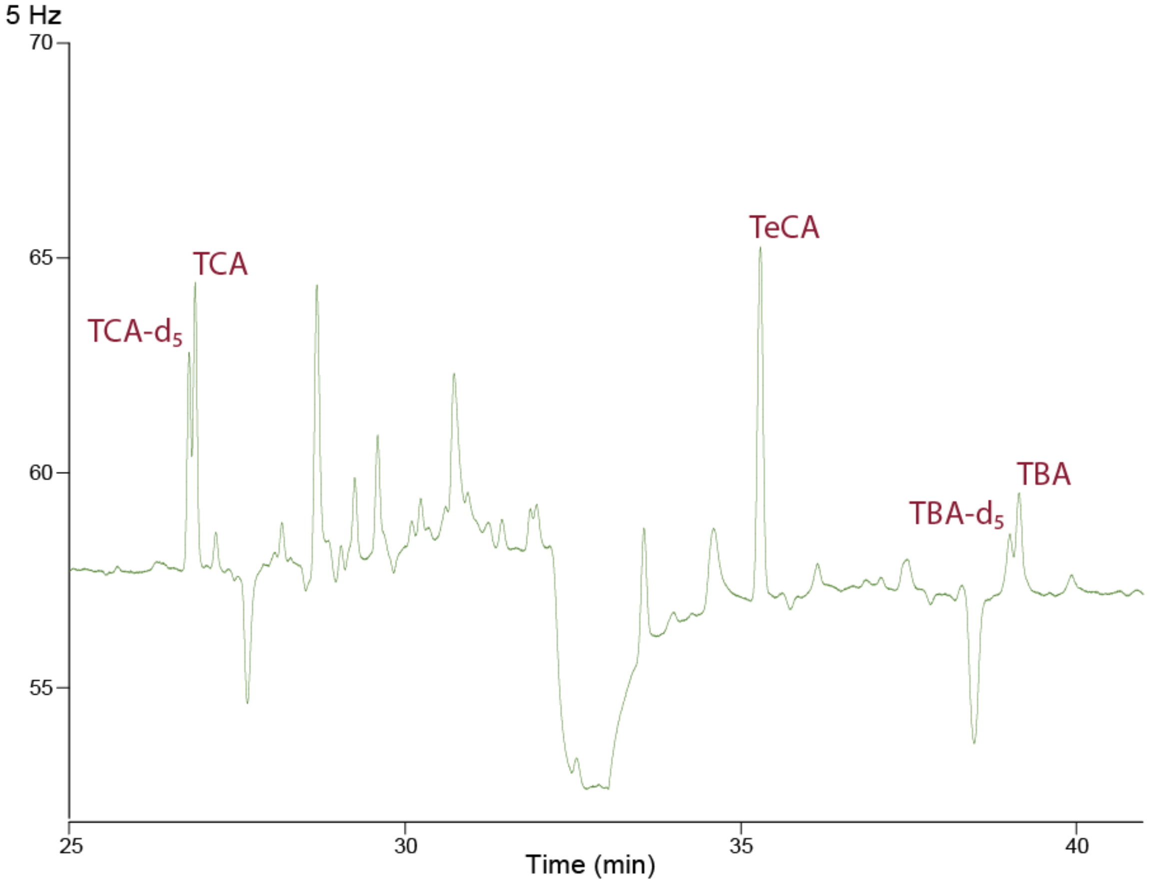


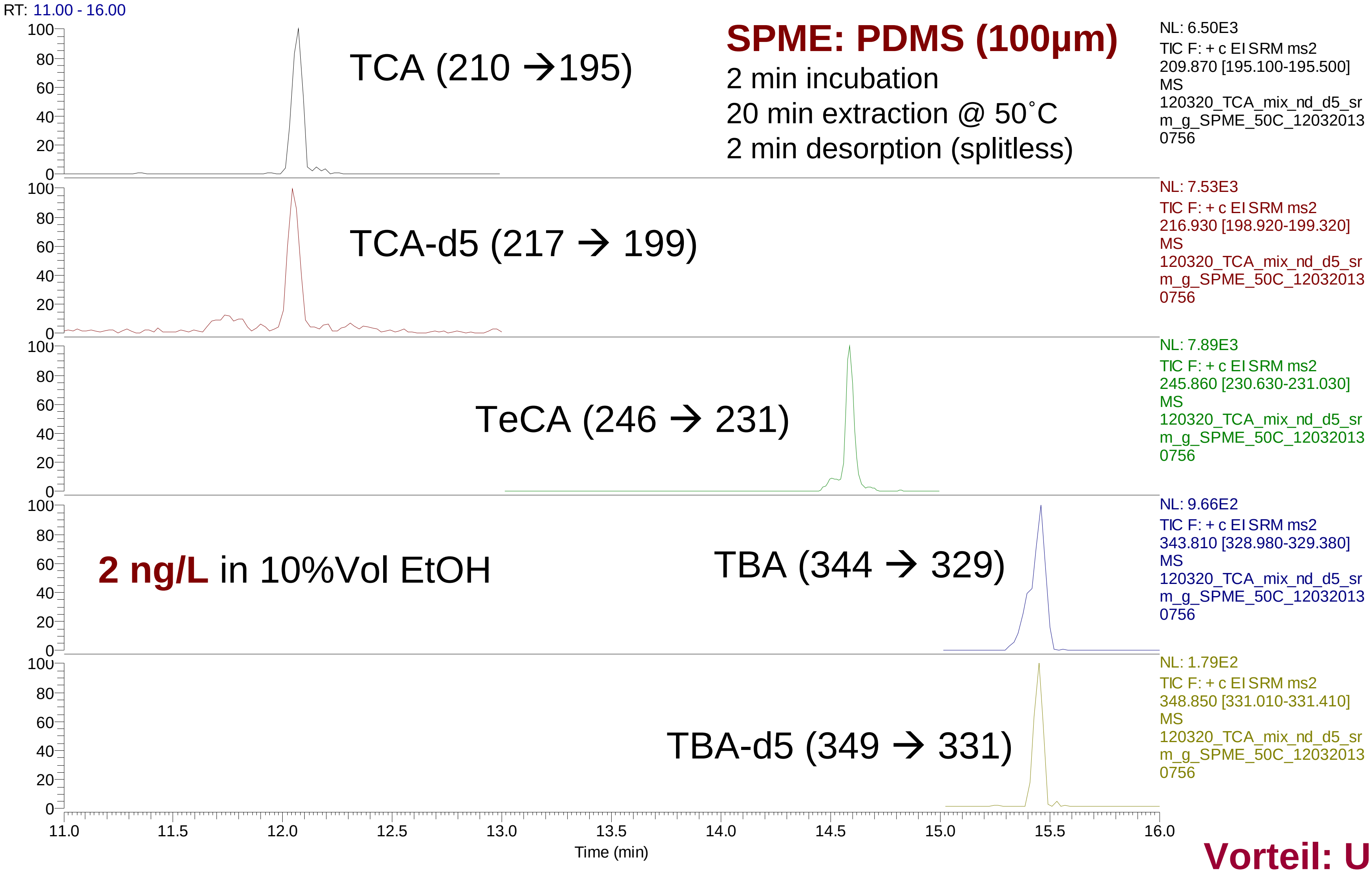
Figure 6 HS–SPME–MDGC–ECD chromatogram of a wine sample (3 ng/L)

Haloanisole mit HS-SPME-MDGC-ECD

Analyte	LOD	LOQ
TCA	0.12 ng/L	0.43 ng/L
TeCA	0.10 ng/L	0.37 ng/L
TBA	0.12 ng/L	0.45 ng/L

Analyte	Repeatability (n=3) wine	cork extract	Recovery (n=3) wine	cork extract
TCA	3.6 (±0.3) ng/L	3.4 (±0.4) ng/L	112.7 (±10.3) %	108.3 (±11.1) %
TeCA	3.7 (±0.4) ng/L	3.8 (±0.3) ng/L	85.1 (±5.6) %	93.8 (±6.2) %
TBA	3.2 (±0.4) ng/L	3.1 (±0.3) ng/L	100.9 (±13.8) %	98.8 (±8.9) %

Haloanisole mit HS-SPME-MDGC-QqQ-MS



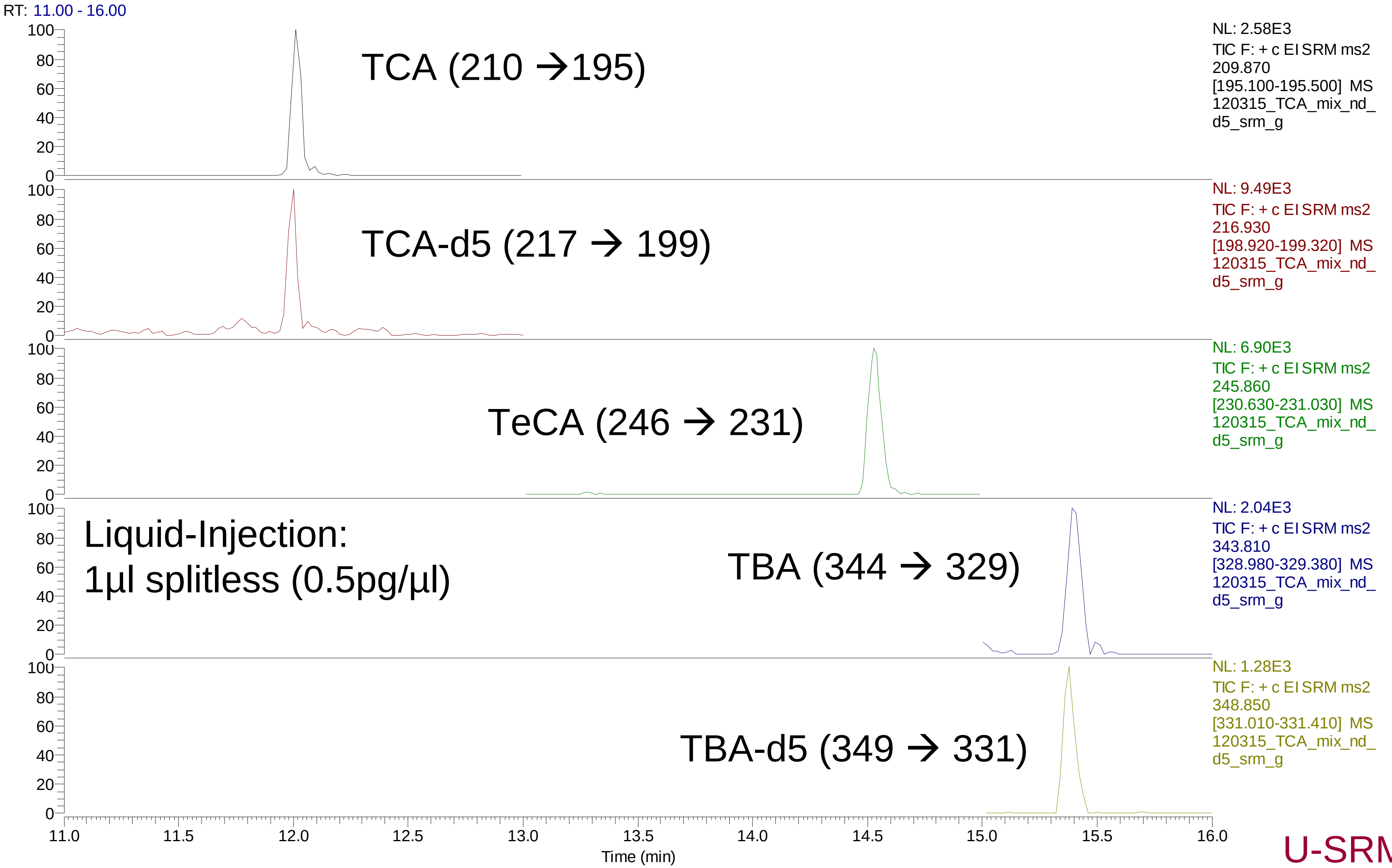
SPME: PDMS (100µm)
2 min incubation
20 min extraction @ 50˚C
2 min desorption (splitless)

60˚C (2 min)10˚/min / 210˚C / 50˚/min / 270˚C (5min)

collision energy 15V

Vorteil: U-SRM
Q1 0.3 Da
Q3 0.7 Da

Haloanisole mit HS-SPME-MDGC-QqQ-MS



U-SRM
Q1 0.3 Da
Q3 0.7 Da

60˚C (2 min)10˚/min / 210˚C / 50˚/min / 270˚C (5min)

collision energy 15V

Profiling-Analysen

- Oenologie
  ... die Wissenschaft vom *Weinmachen*
  (ausser weinbaulichen Aspekten)

- Wein-Aroma
  Flüchtige Stoffe, die unseren Sinnen (Nase) und den analytischen Methoden (GC) zugänglich sind

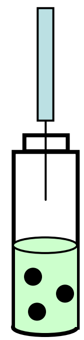
- (Multivariate) Statistische Analysen
  Principal Component Analysis (PCA):
  Linear Discriminant Analysis (LDA)
  Partial Least Squares Regression (PLSR)
  Analysis of Variance (ANOVA)

- Applikationen
  Alterung, Mikro-Oxygenierung (MOX)

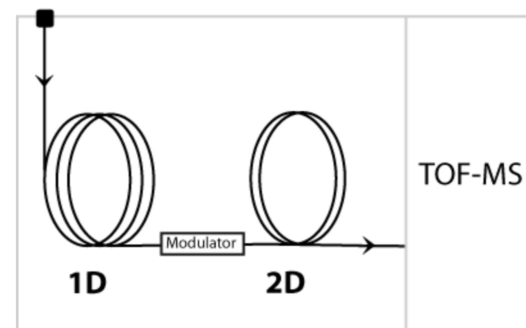
Analysis: HS-SPME-GC×GC-MS

38 samples
10 origins
4 vintages

subset of a larger number of samples from the Palatinate *Terroir* project



HS-SPME
internal standards

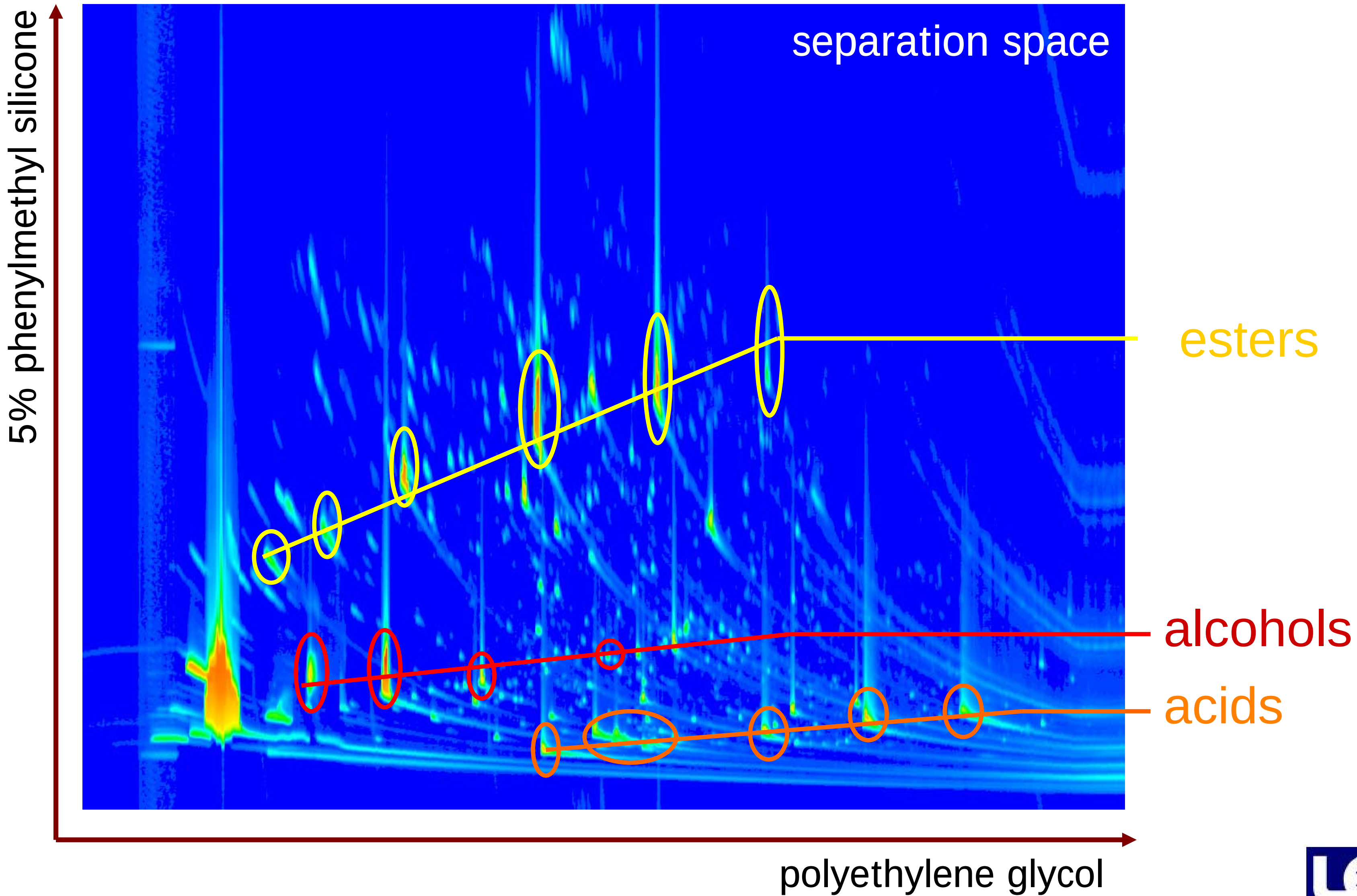


analysis via
GC×GC-qMS
or
GC×GC-TOF-MS

quantification *via* internal standards

ToF-MS in co-operation with

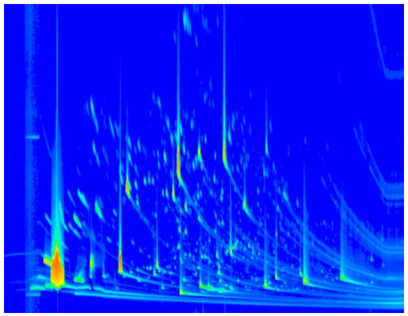
Analysis: ***Reversed*** column set-up

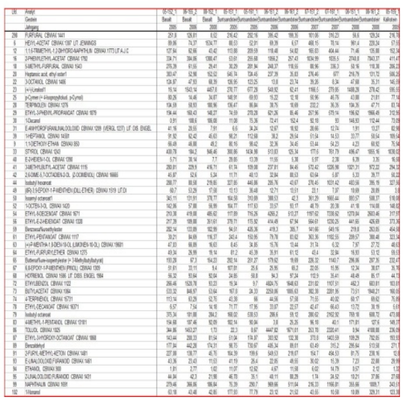


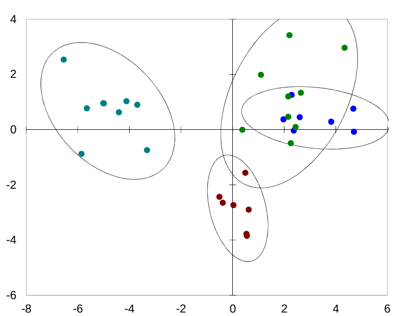
Alterung von (Riesling) Wein

- Nach Ende der Gärung, umfasst die einsetzende Reife (Alterung) viele komplexe substanzielle Veränderungen
- Farbe, Mundgefühl, Aroma und Geschmack unterliegen z.T. drastischen Veränderungen
- Sauerstoff und konzentrations-abhängige molekulare Veränderungen (MWG), aber auch Zersetzungs- und andere Reaktionen
- Neben individueller Präfe-renzen für alte Weine ist die Kenntnis des Alterungspoten-tiales wichtig für die Industrie

Aging of Riesling Wine





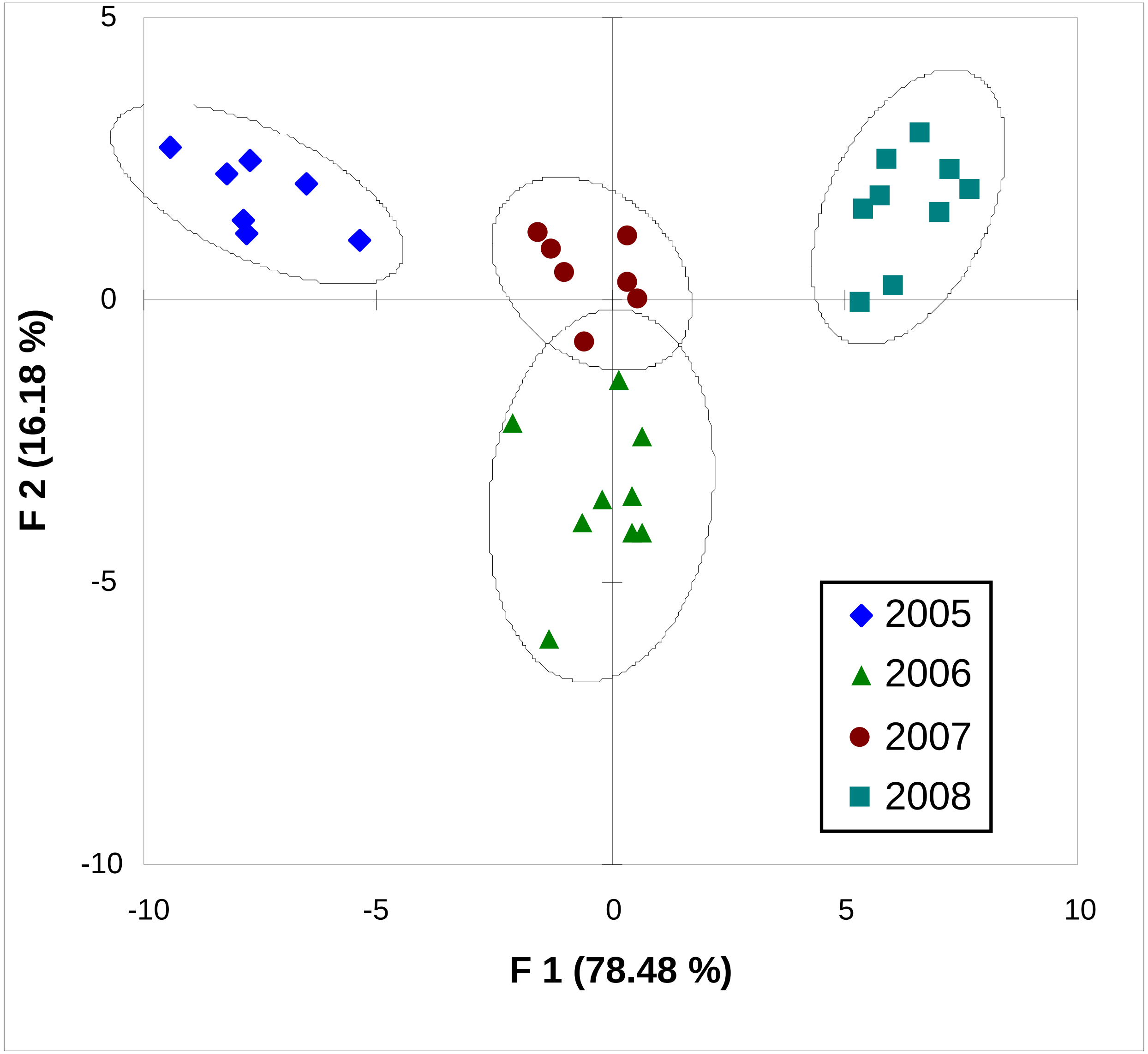


- 1200 peaks detected
- 300 peaks identified according to their mass spectra and retention indices
- random selection of a chromatogram as reference
- 130 analytes present in all samples
- quantification relative to a reference standard (2,6-dimethylhept-6-en-2-ol, [$^2H_5$]-ethyl cyclohexanoate)
- statistics with individual peak areas — the *classical* workflow, based on integrated peaks

Non-Targeted Multi-Component Profiling

… biased …

Riesling Aging (ChromaToF), LDA, F1 F2, 15 most relevant signals



confidence intervals @ 95%

Riesling Aging (ChromaToF), LDA, 15 most relevant signals

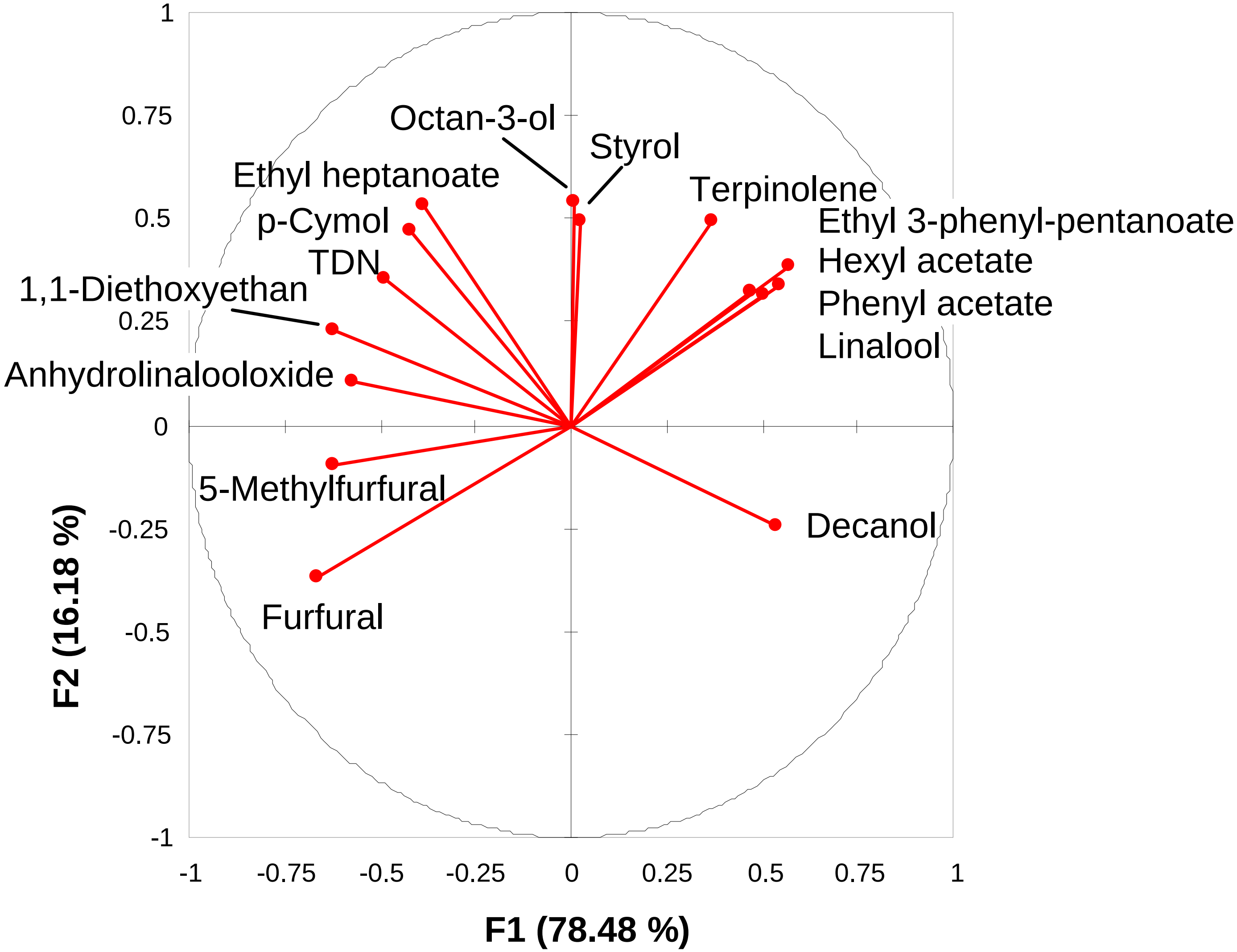
Confusion matrix

Validation: ~15 % Test data set
85 % Model data set
20x Repetition

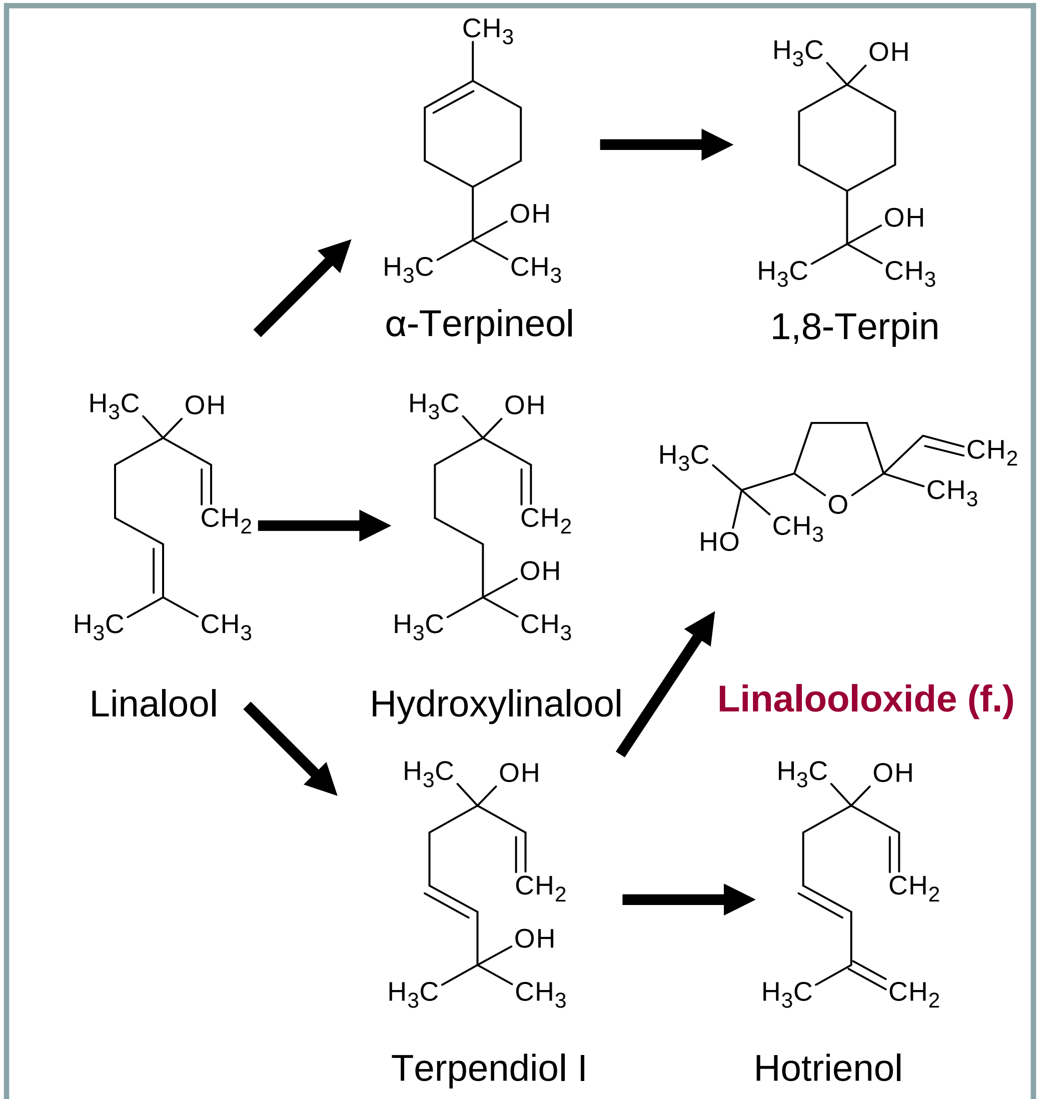
	2005	**2006**	**2007**	**2008**
2005	95 %	0 %	5 %	0 %
2006	0 %	84 %	16 %	0 %
2007	6 %	6 %	89 %	0 %
2008	0 %	8 %	21 %	71 %

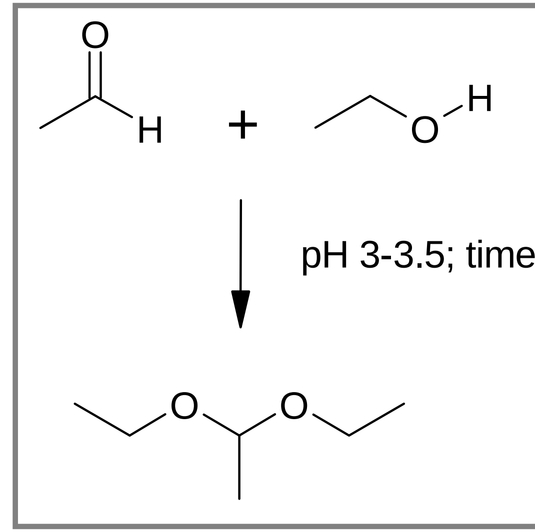
~ 84 % correct correlation
(differing sample number in individual groups, due to randomization of vintages)

Riesling Aging (ChromaToF), LDA, F1 F2, 15 most relevant signals, loadings



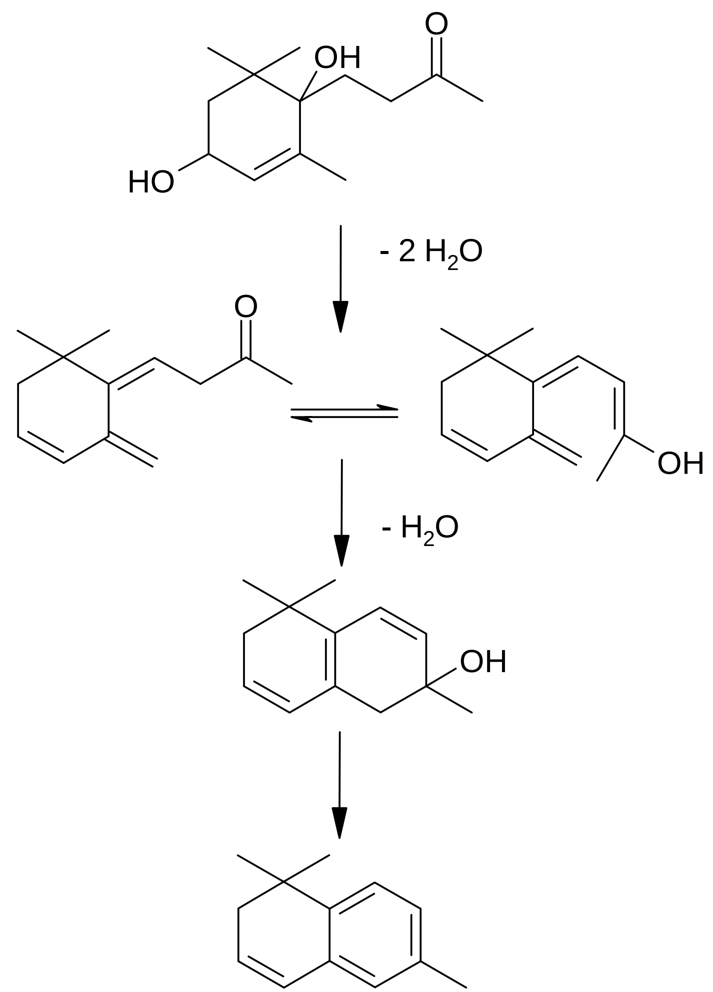
Aging – Chemical Issues





1,1-Diethoxyethane,
Acetaldehyde diethyl acetal

Riesling Aging: Petrol or Kerosine note



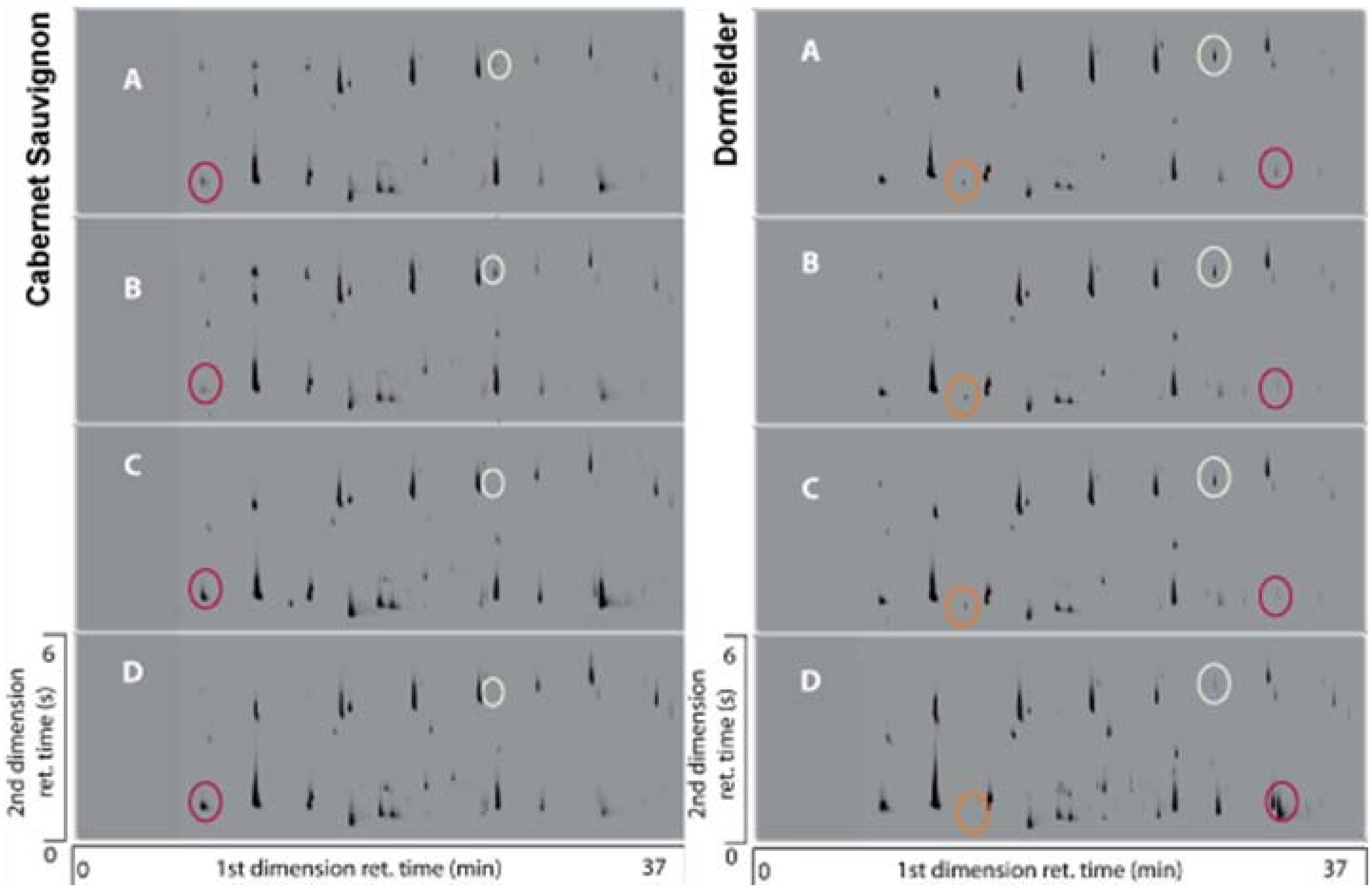
1,1,6-Trimethyl-1,2-dihydronaphthalene (TDN)

Proposed pathway of TDN formation (from Winterhalter, JAFC 39 **(1991)** 1825)

Profiling – via Fingerprint Analysis

Clearly non-targeted: Visual comparison

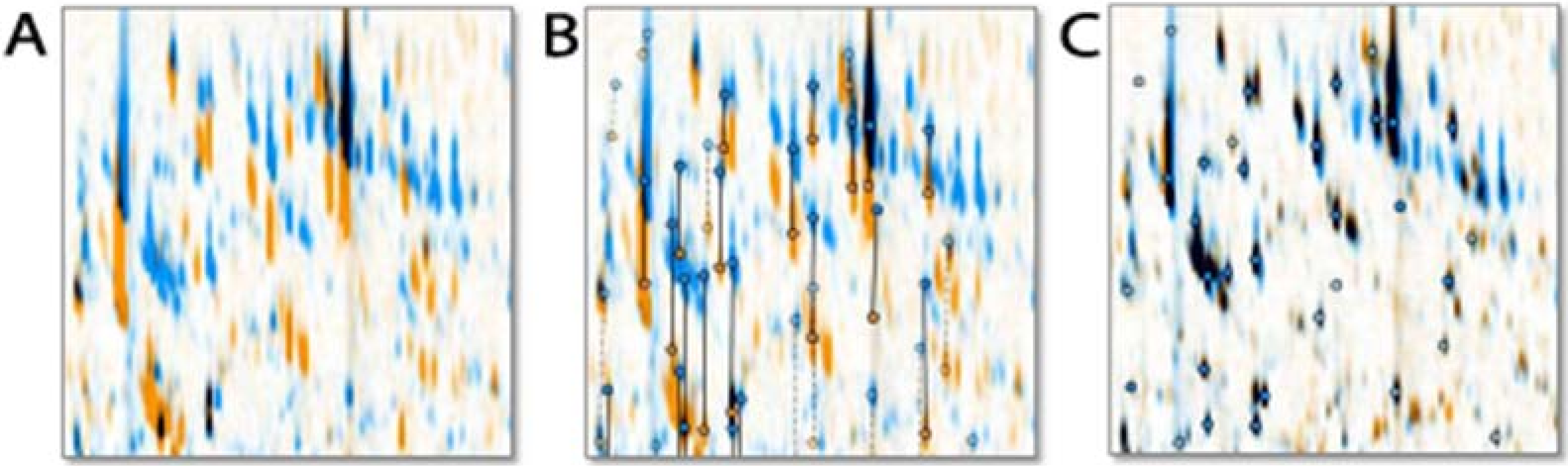
only for limited sample numbers



Advanced Image Processing

Image warping

compensates for fluctuations in signal position



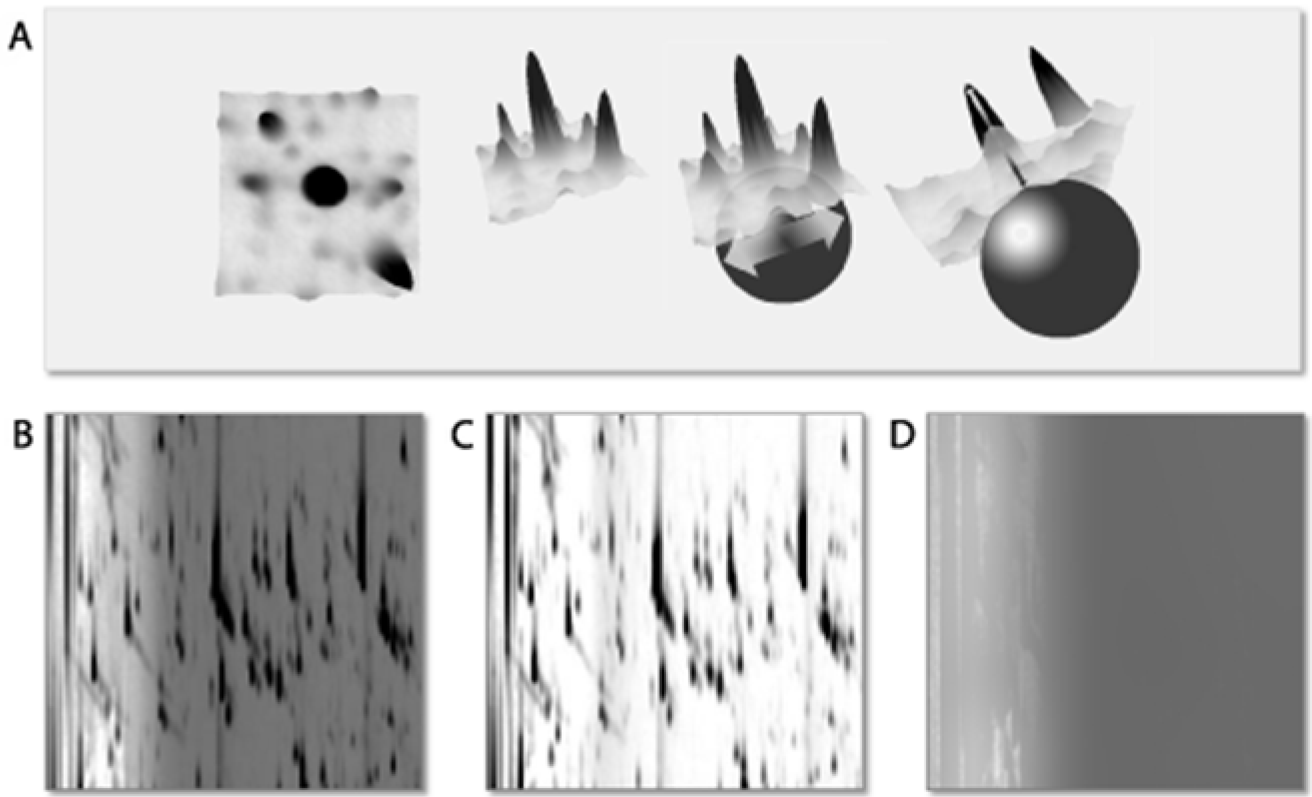
... image/pixel-based analysis ...

H.-G. Schmarr, J. Bernhardt, *J. Chromatogr. A* 1217 (2010) 565

Advanced Image Processing

Rolling ball model

applied for background substraction



H.-G. Schmarr, J. Bernhardt, *J. Chromatogr. A* 1217 (2010) 565

Volatile (aroma) - Profiling

fingerprint analysis;
approach is based on image processing techniques


Journal of Chromatography A, 1217 (2010) 565–574

Contents lists available at ScienceDirect

Journal of Chromatography A

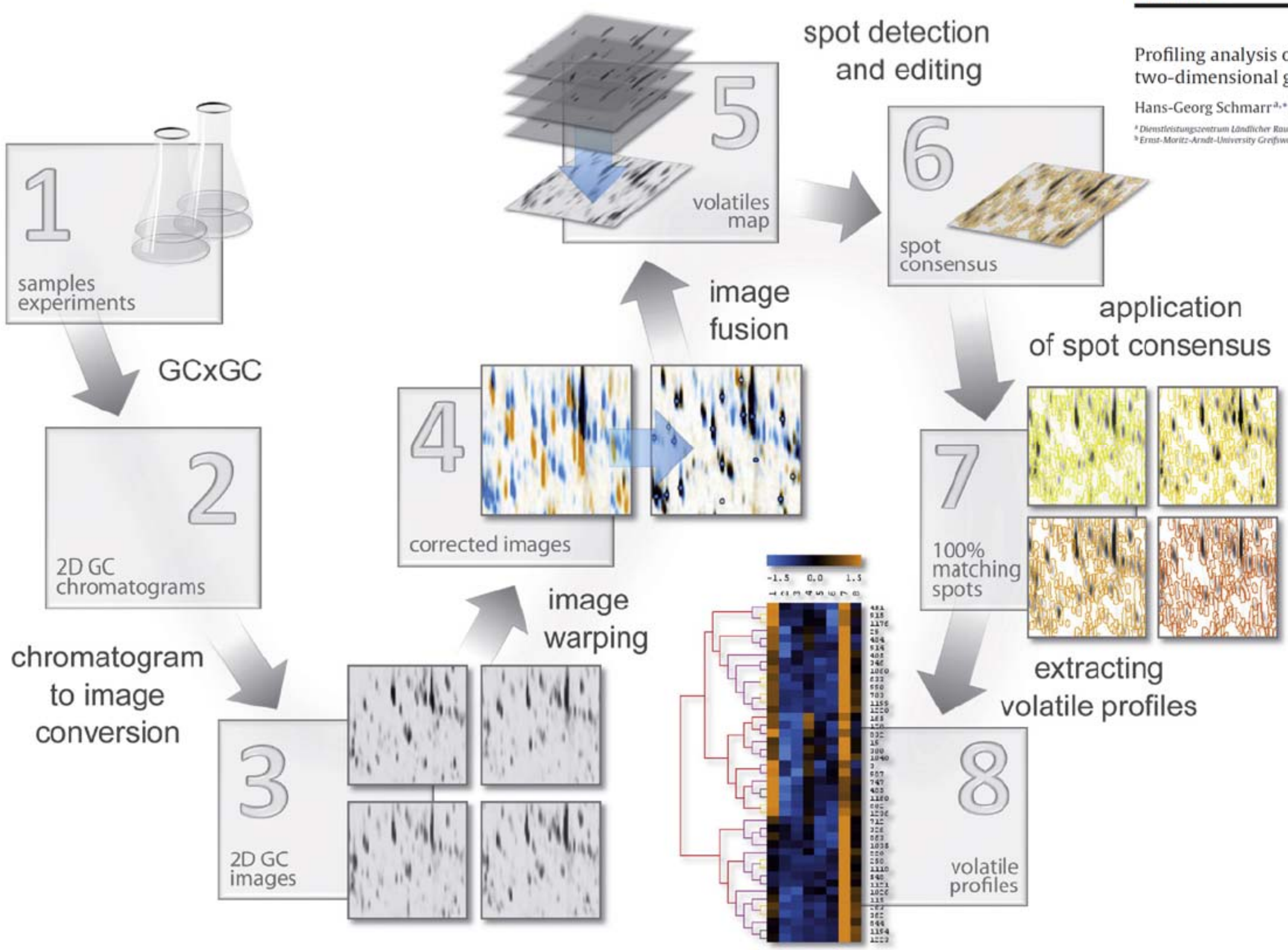
journal homepage: www.elsevier.com/locate/chroma

Profiling analysis of volatile compounds from fruits using comprehensive two-dimensional gas chromatography and image processing techniques

Hans-Georg Schmarr[a,*], Jörg Bernhardt[b]

[a] Dienstleistungszentrum Ländlicher Raum (DLR) Rheinpfalz, Viticulture and Enology Group, Breitenweg 71, D-67435 Neustadt a.d. Weinstraße, Germany
[b] Ernst-Moritz-Arndt-University Greifswald, Institute of Microbiology, Friedrich Ludwig Jahn Straße 15, D-17487 Greifswald, Germany

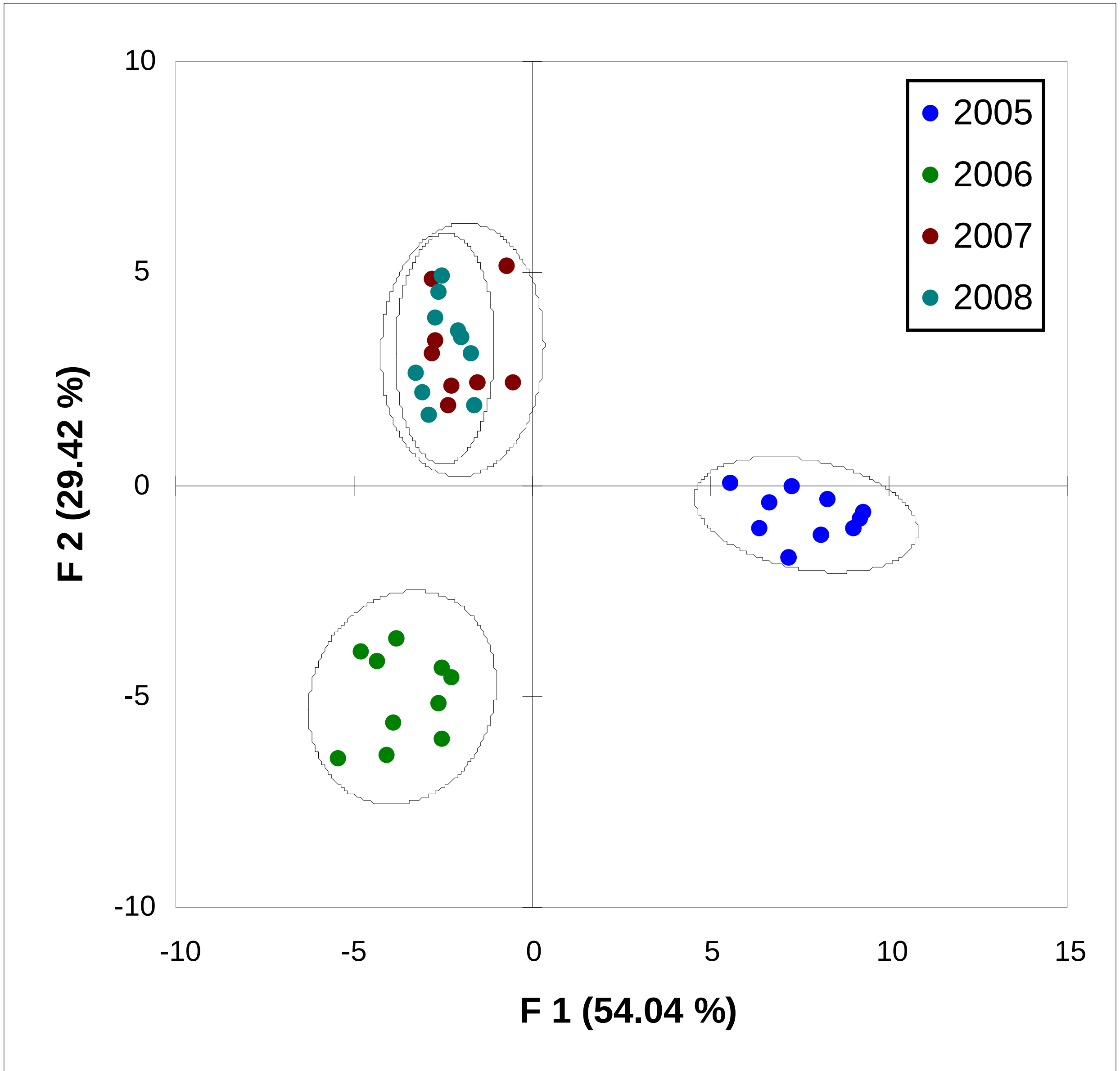
H.-G. Schmarr, J. Bernhardt / J. Chromatogr. A 1217 (2010) 565–574



Delta2D software, well established in 2D gel electrophoresis

Decodon, Greifswald, Germany

Riesling Aging (Delta2D), LDA F1 F2, 15 most relevant signals



confidence
intervals @ 95%

Riesling Aging (Delta2D), LDA F1 F2, 15 most relevant signals

Confusion matrix

Validation: ~15 % Test data set
85 % Model data set
10x Repetition

	2005	2006	2007	2008
2005	100 %	0 %	0 %	0 %
2006	0 %	100 %	0 %	0 %
2007	0 %	0 %	100 %	0 %
2008	0 %	0 %	0 %	100 %

Potential explanation for better validation of the model:
ANOVA yielded 15 most relevant signals - but considering all signal information (unbiased)

Analytica Chimica Acta 672 (2010) 114–123

Contents lists available at ScienceDirect

Analytica Chimica Acta

journal homepage: www.elsevier.com/locate/aca

Enology, Winemaking

Two-dimensional gas chromatographic profiling as a tool for a rapid screening of the changes in volatile composition occurring due to microoxygenation of red wines

Hans-Georg Schmarr[a,*,1], Jörg Bernhardt[b,1], Ulrich Fischer[a], Alexander Stephan[a,c,2], Patrick Müller[b], Dominik Durner[a,1]

[a] Dienstleistungszentrum Ländlicher Raum (DLR) Rheinpfalz, Viticulture and Enology Department, Breitenweg 71, D-67435 Neustadt a. d. Weinstraße, Germany
[b] Ernst-Moritz-Arndt-University Greifswald, Institute of Microbiology, Friedrich Ludwig Jahn Straße 15, D-17487 Greifswald, Germany
[c] Technical University of Kaiserslautern, Food Chemistry and Environmental Toxicology, Erwin-Schrödinger-Straße 52, D-67663 Kaiserslautern, Germany

MOX treatment of red wines

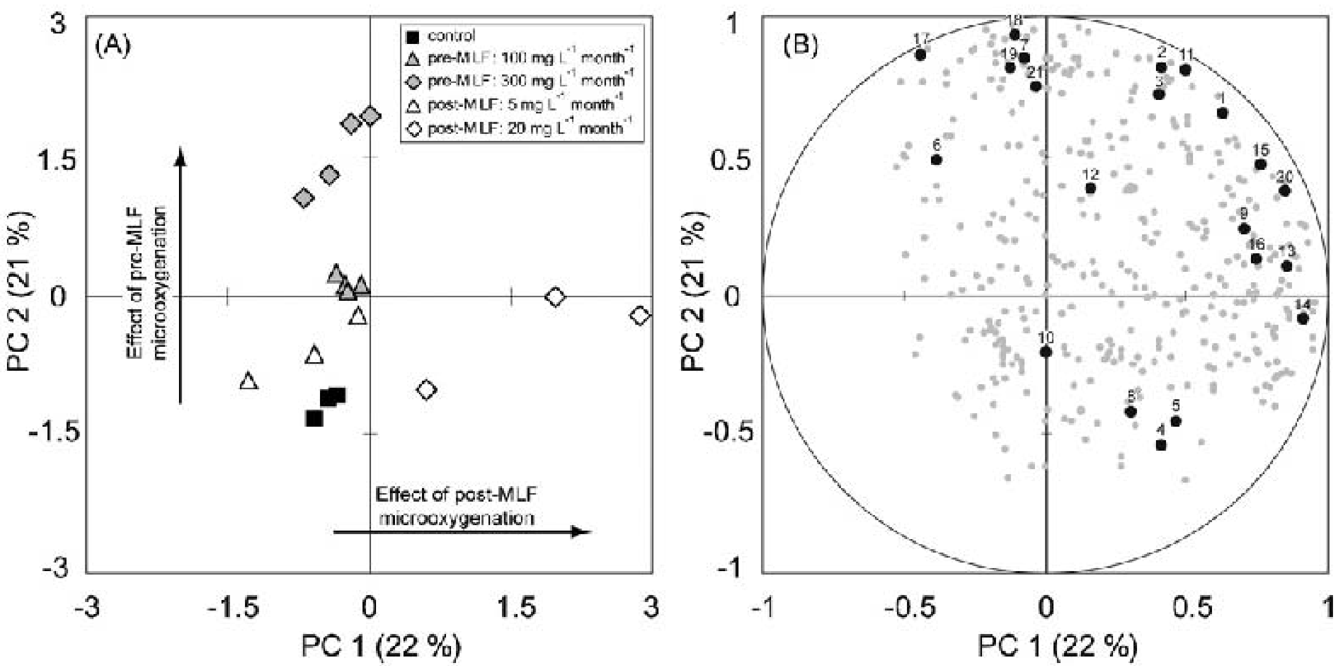


Fig. 5. Principal component analysis (PCA) of volatiles in MOX treated Cabernet Sauvignon wines. (A) Scores plot and (B) loadings plot. Bold circles and numbers in (B) refer to components listed in Table 3.

MOX: Changes in composition

"Fermentation-Metabolom" dependend on

- oxygen dosage and timing
- grape variety (polyphenol structure)

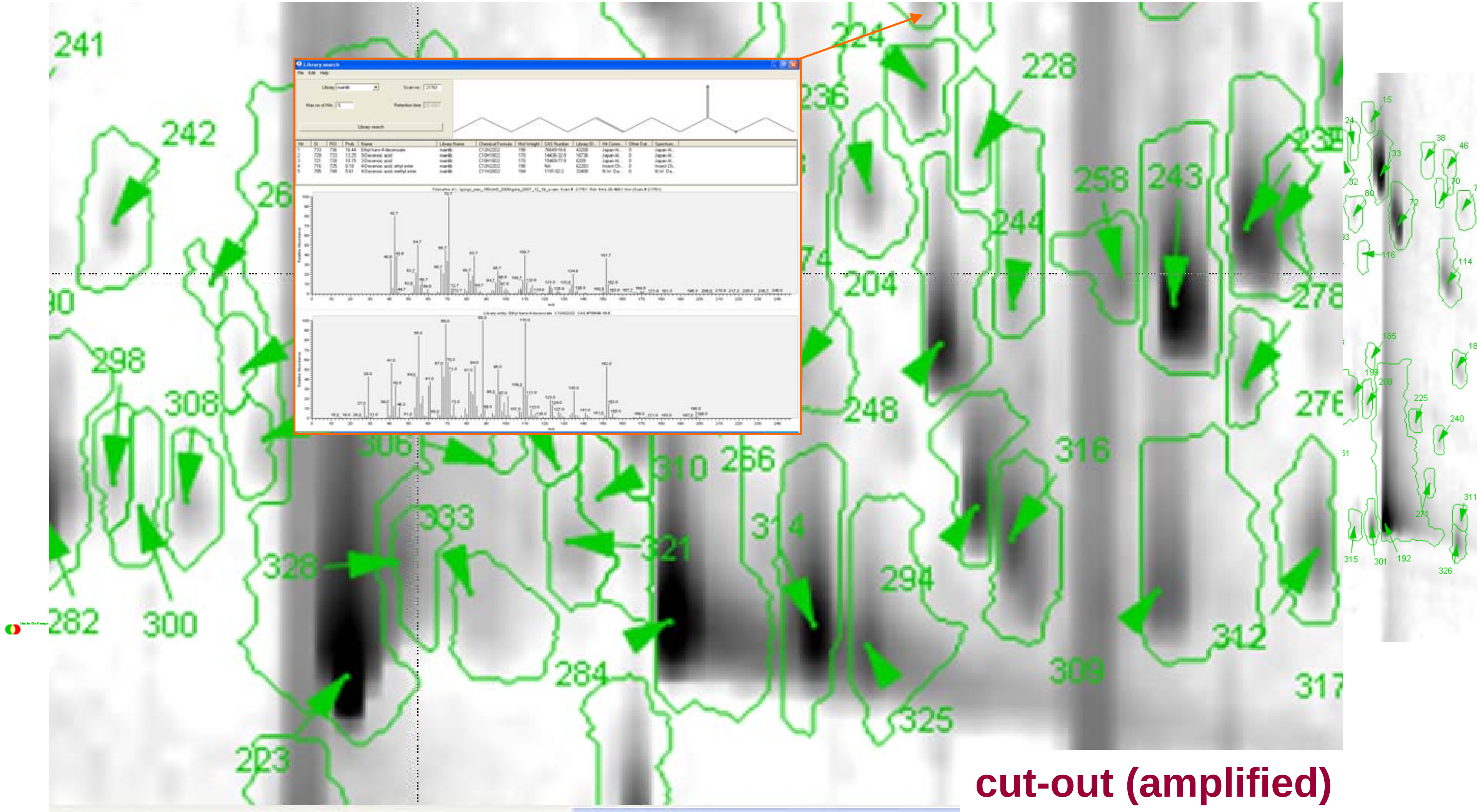
loadings on PC1-PC3 were multiplied by their percentage of explained variance, summed, and arranged according to size

Loadings of tentative marker compounds on PC1, PC2 and PC3 and their contribution to discriminate between pre- and post-MLF–MOX.

Loadings: Contribution to:	Assigned number	PC1	PC2	PC3	PC1 Post-MLF	PC2 Pre-MLF	PC3 Pre + post-MLF
Alcohols							
2-Phenylethanol	1	0.631	0.651	−0.158	+	+	−
1-Hexanol	2	0.411	0.811	0.185	+	+	+
3-Methylthio-1-propanol	3	0.410	0.722	0.344	+	+	+
2,3-Butandiol	4	0.411	−0.539	0.484	+	−	+
2,3-Butandiol	5	0.461	−0.459	0.406	+	−	+
2-Hexen-1-ol	6	−0.385	0.479	0.216	−	+	+
3-Hexen-1-ol	7	−0.067	0.843	0.233	−	+	+
Aldehydes and ketones							
Octanal	8	0.306	−0.423	0.433	+	−	+
Decanal	9	0.704	0.235	−0.002	+	+	−
3-Hydroxy-2-butanone, acetoin	10	0.005	−0.206	0.681	+	−	+
Esters							
2/3-Methylbutyl butanoate	11	0.499	0.808	−0.039	+	+	−
Ethyl hexanoate	12	0.164	0.375	0.380	+	+	+
Ethyl octanoate	13	0.854	0.094	−0.015	+	+	−
Ethyl decanoate	14	0.919	−0.086	−0.095	+	−	−
Ethyl dec-2-enoate	15	0.765	0.467	0.198	+	+	+
Ethyl dodecanaote	16	0.750	0.124	0.082	+	+	+
Ethyl hexadecanoate	17	−0.436	0.855	0.063	−	+	+
2-Phenylethyl acetate	18	−0.106	0.931	−0.195	−	+	−
Miscellaneous							
β-Damascenone	19	−0.124	0.809	−0.279	−	+	−
TDN (1,1,6-trimethyl-1,2-dihydronaphtalene)	20	0.848	0.366	−0.021	+	+	−

Image Analysis and Signal Labeling

Drawback: Off-line Signal Identification



cut-out (amplified)

Zusammenfassung

Komplexe Matrices (z.B. Wein) erfordern mitunter besondere Behandlung für Zielkomponentenanalytik!
→ Ansonsten drohen **falsche Resultate!**

- Möglichkeiten für verbesserte Analytik seitens:
  - Probenvorbereitung
  - **Separation (Chromatographie)**
  - **Detektion**

- **MDGC (Deans-Switch oder MCSS)**

- TriPlus RSH
  - Variable Injektionsmöglichkeiten

- **Quantum Ultra**
  - **QqQ-MS und evtl. U-SRM**

Ausblick und Erwartungen

- Profiling – Analysen und Statistische Auswerteverfahren zur Erfassung komplexer Veränderungen im Weinaroma (Oenologie)

- Advanved Image Processing für GC x GC-Daten sehr vielversprechend (stoffliche Zuordnung derzeit *off-line*)

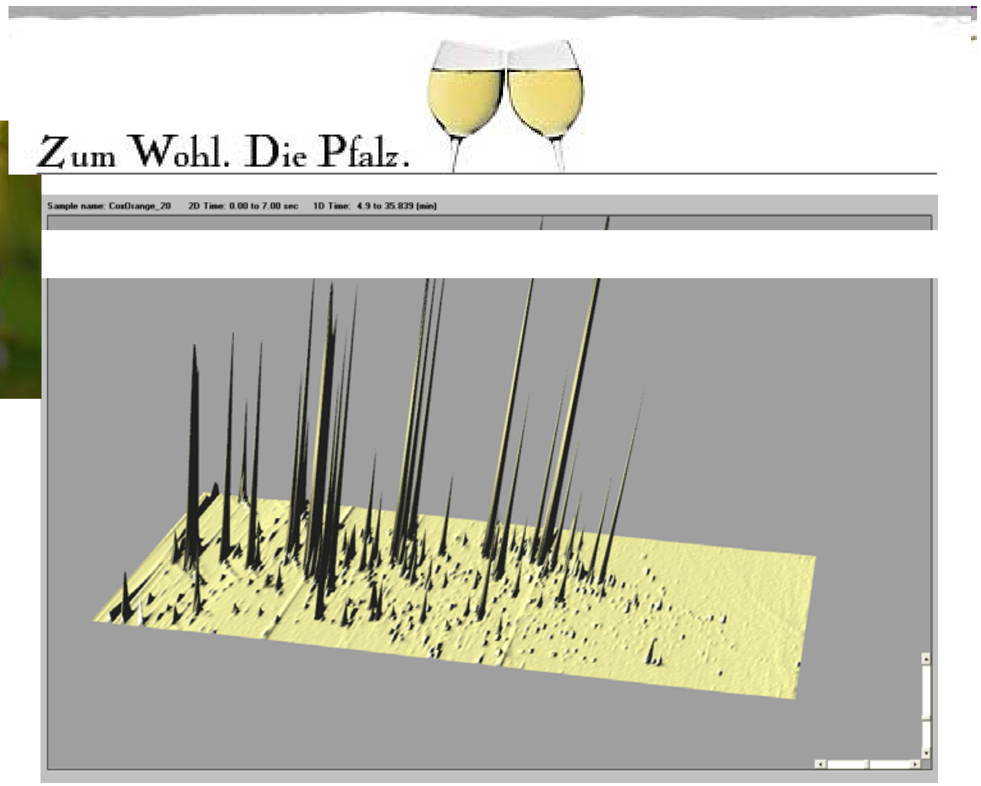
- Korrelation oenologischer/sensorischer Daten mit chromatographischer und spektroskopischer (UV/Vis, IR, NMR) Information

- → **Oenometabolomics**

Vielen Dank für Ihre Aufmerksamkeit!

RheinlandPfalz
DIENSTLEISTUNGSZENTRUM
LÄNDLICHER RAUM
RHEINPFALZ

Competence Center
of Wine Research



Dank an:
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Theodoros Potouridis,
Johannes Langen,
Elisa Garcia-Moreno
u.a.
für Ihre Mitarbeit

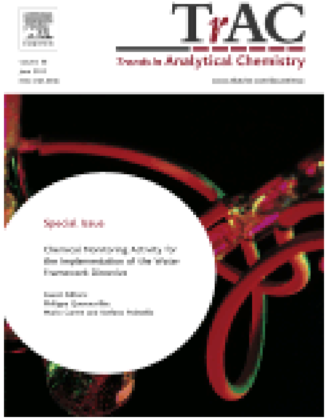
Dank an das MULEWF, Rheinland-Pfalz, für finanzielle Unterstützung

MDGC: A comprehensive review ...

Trends in Analytical Chemistry, Vol. 34, 2012

Multidimensional gas chromatography

Philip J. Marriott, Sung-Tong Chin, Bussayarat Maikhunthod, Hans-Georg Schmarr, Stefan Bieri



Most Downloaded Trends in Analytical Chemistry Articles

8. Multidimensional gas chromatography

April 2012

Philip J. Marriott | Sung-Tong Chin | Bussayarat Maikhunthod | Hans-Georg Schmarr | Stefan Bieri

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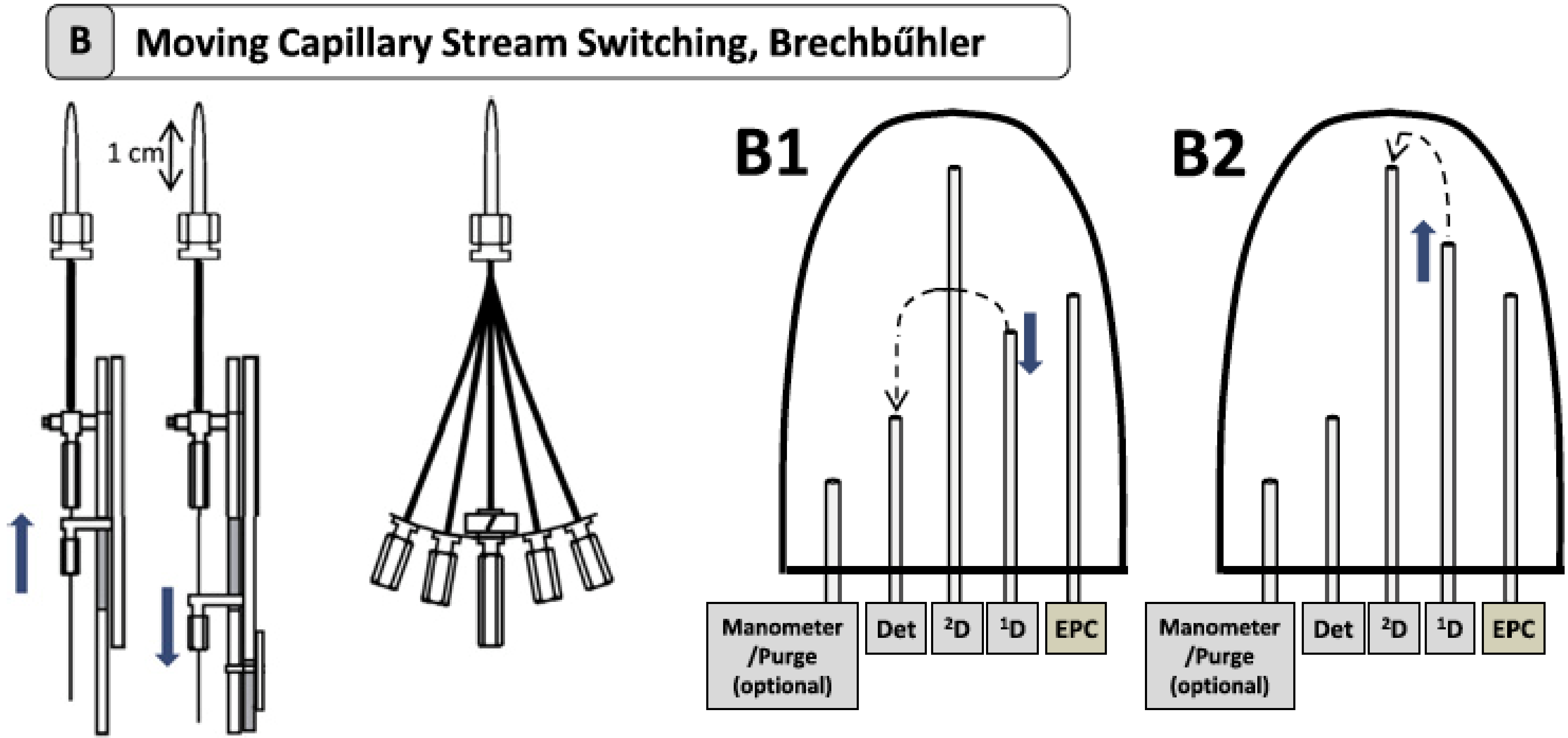
AbstractAnalytical multidimensional gas chromatography (MDGC) and the excellent separation efficiency it achieves serve advanced characterization of complex volatile and semi-volatile samples, which is unlikely to be accomplished by single-dimensional chromatography. Here, we provide a technical overview of recent method implementation in MDGC, for both the classical sense (i.e. conventional heart-cut MDGC), including recent approaches to MDGC, and the comprehensive two-dimensional gas chromatography (GC×GC)

MDGC: MCSS



TrAC Trends in Analytical Chemistry 34 (2012) 1 - 21

MDGC: SilFlow Deans Switch

E SilFlow Micro-channel device, SGE Analytical