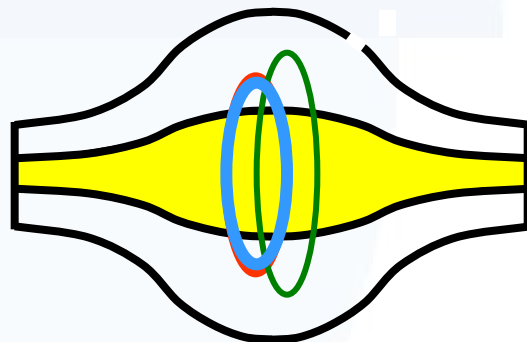


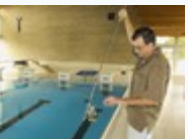


Multiclass methods in the field of drug residues: Low Resolution Mass Spectrometry versus high Resolution Mass Spectrometry

(Single Stage Orbitrap)



Anton Kaufmann
Food Control Authority Zürich, Switzerland





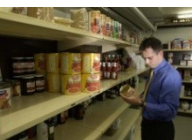
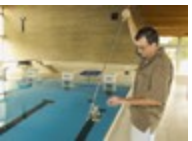
Content overview



- Why HRMS based multiresidue methods ?
- Are we selective enough ?
- Quantification
- Identification
- Strengths and limitations (Single stage Orbitrap)
- Successful example of unknown screening



Proficiency test (Rikilt)

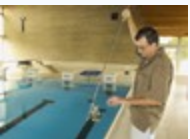




Three samples

Screening
followed by
Quantification:

Aminoglycosides
Penicillines
Macrolides
Chinolones
Sulfonamides
Tetracyclines





Multimethods ?

37 participating laboratories

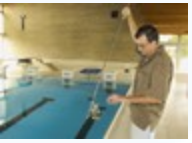
160 different methods

nevertheless only

29 laboratories correctly found Tetracyclin

23 laboratories correctly found Nafcillin

13 laboratories correctly found Neomycin





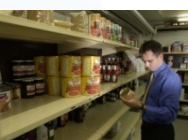
Screening results

Table 1. Overview of the false negative and false positive results for microbiological, biochemical and instrumental screening methods

Material		A		B	C
	total	NEO	OTC	OTC	NAF
No. of labs submitted results		26	33	33	32
No. of methods applied	160	34	43	43	40
False positives					
Microbiological methods					
Biochemical methods	3	1		1	1
Instrumental methods					
False negatives					
Microbiological methods					
Biochemical methods	1	1	0	0	0
Instrumental methods					

*1 false negative result was reported by not detecting NEO, although it was included in method.

**false negative result was reported by not detecting OTC, although it was included in method.





Only 5 out of 37 laboratories screened and quantified correctly !



Screening

LAB 1: Microbiological

LAB 2: HRMS

LAB 3: HRMS

LAB 4: LC-MS/MS

LAB 5: LC-MS/MS

Quantification

LC-MS/MS

LC-MS/MS

HRMS

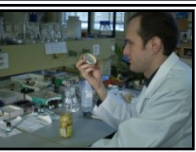
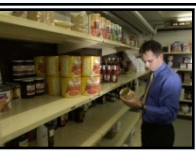
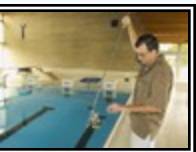
LC-MS/MS

LC-MS/MS



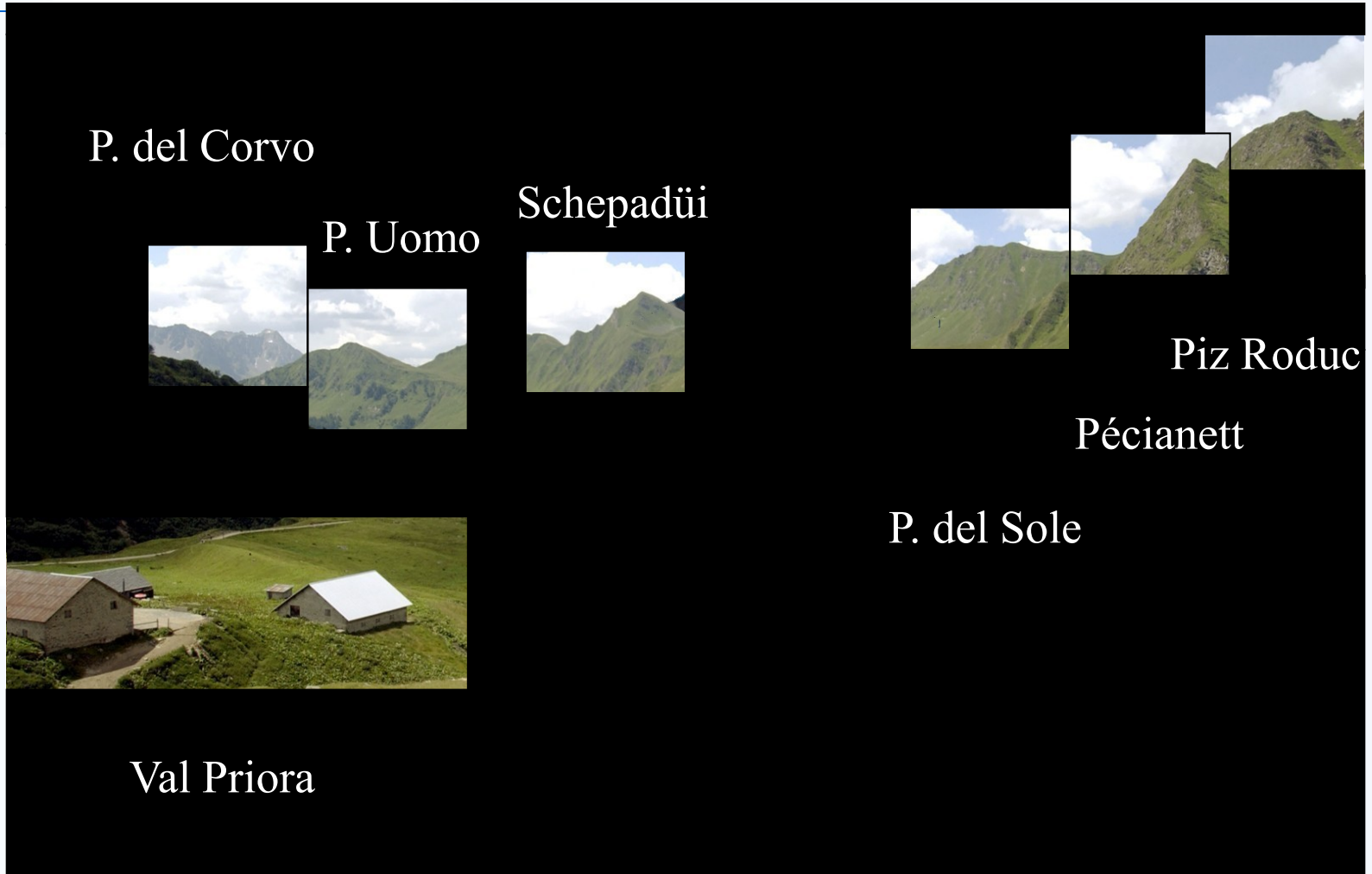
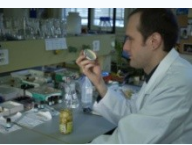
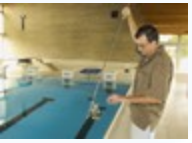
MS/MS based multiresidue methods are significantly better than microbiological tests but ...

- MS/MS requires the individual tuning of each monitored compound
- MS/MS is not made to monitor hundreds of compounds
- MS/MS finds only the things we are expecting



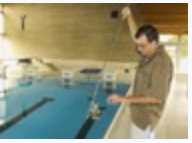


Typical Swiss scenery as seen by MS-MS





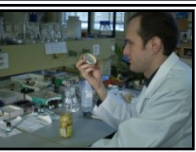
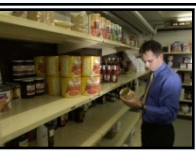
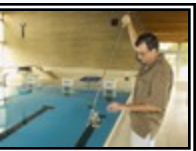
Atypical Swiss scenery not detected by MS-MS





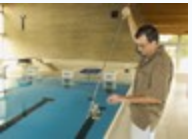
High Resolution Mass Spectrometry (HRMS)

- No tuning of compounds
- Exact masses are calculative by nature, unlike MS/MS transitions
- All ionisable compounds show up
- No Limits regarding the number of monitored compounds





Content overview



- Why HRMS based multiresidue methods ?

- Are we selective enough ?

- Quantification

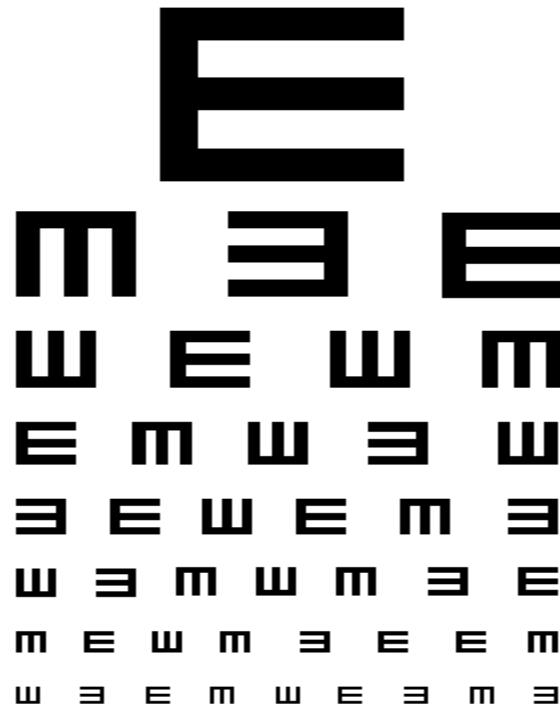
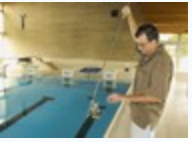
- Identification

- Strengths and limitations (Single stage Orbitrap)

- Successful example of unknown screening



How high is high enough?

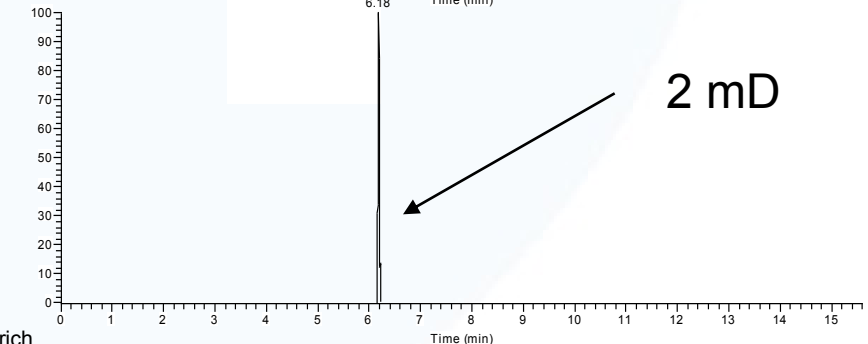
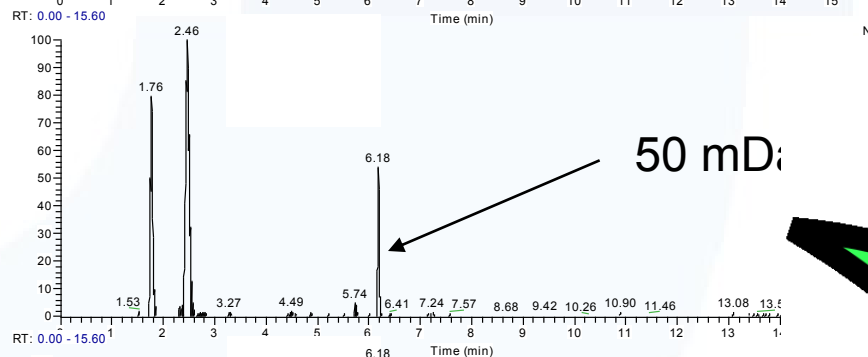
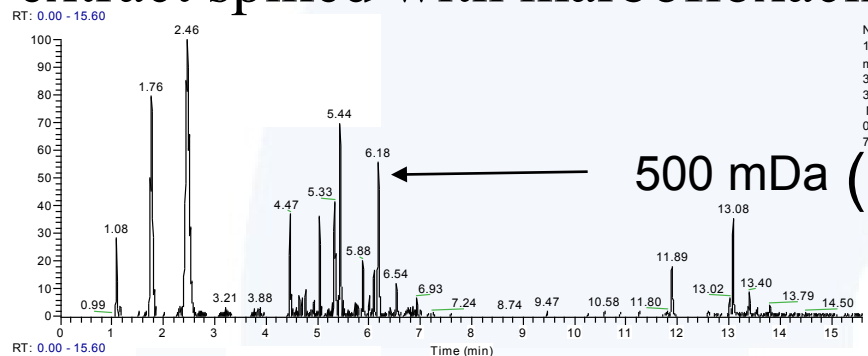


What resolution do we need ?



Second Generation TOF Resolution 10'000 FWHM

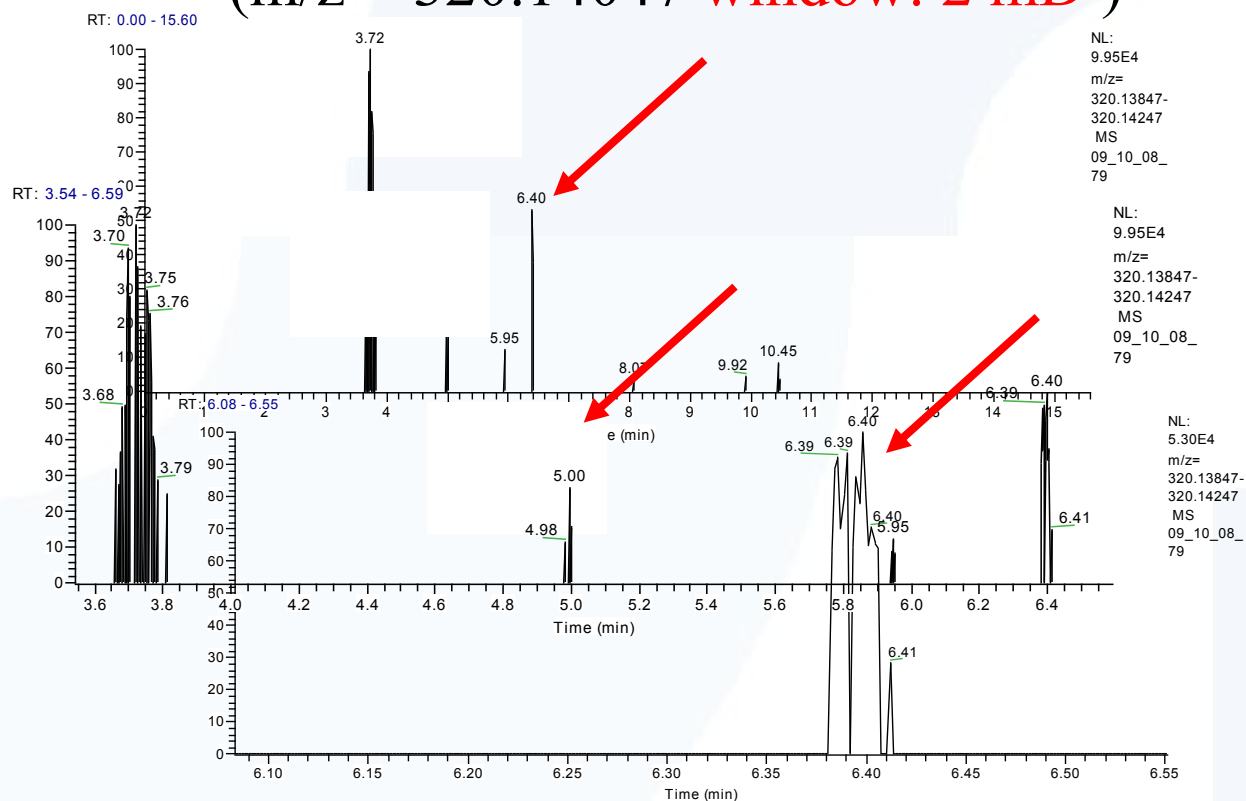
Liver extract spiked with marbofloxacin ($m/z = 363.14628$)





Second Generation TOF Resolution 10'000 FWHM

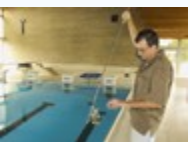
Liver extrakt spiked with norfloxacin
($m/z = 320.14047$ window: 2 mD)



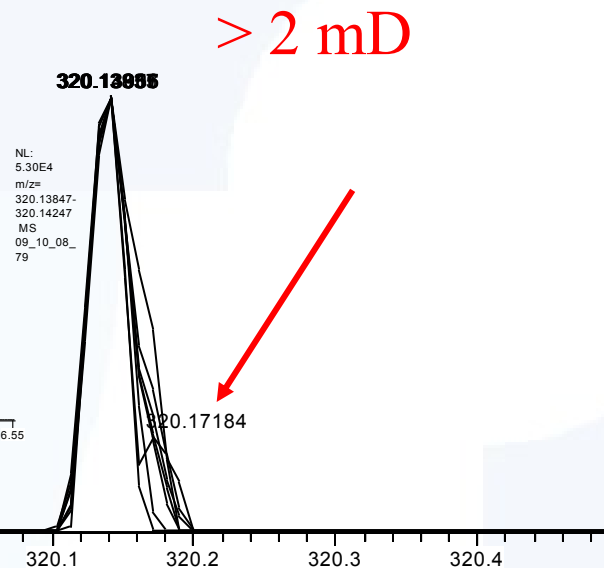
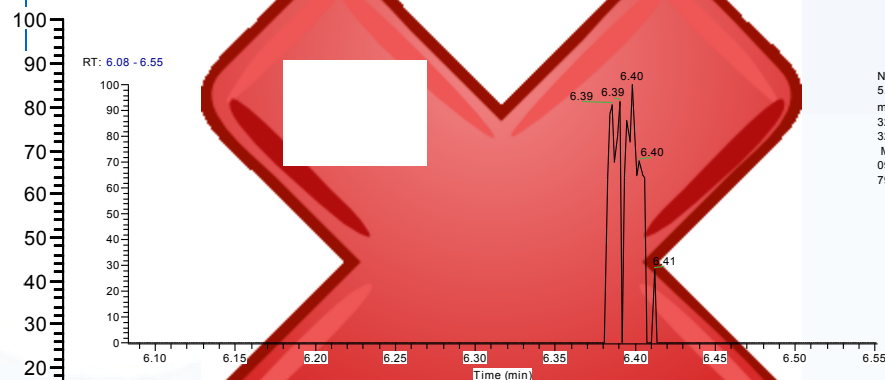


Second Generation TOF Resolution 10'000 FWHM

Norfloxacin spectrum ($m/z = 320.14047$)



09_10_08_79 #4131 RT: 6.39 AV: 1
T: FTMS {0,0} + p ESI

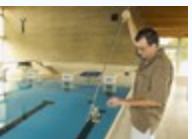
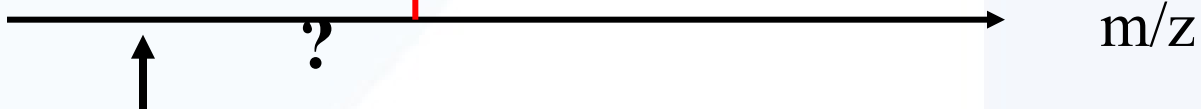




Isobaric interferences

Dom
(Analyte)

Täschhorn
(Matrix Compound)

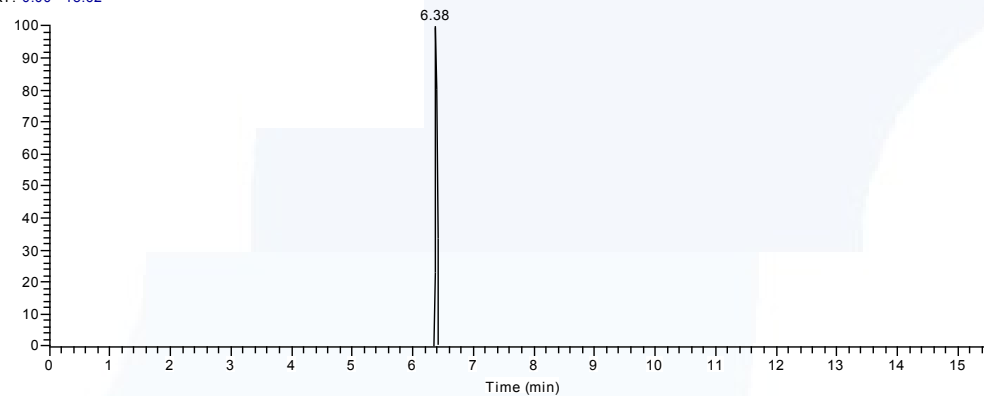




Maximum resolution 100'000 FWHM

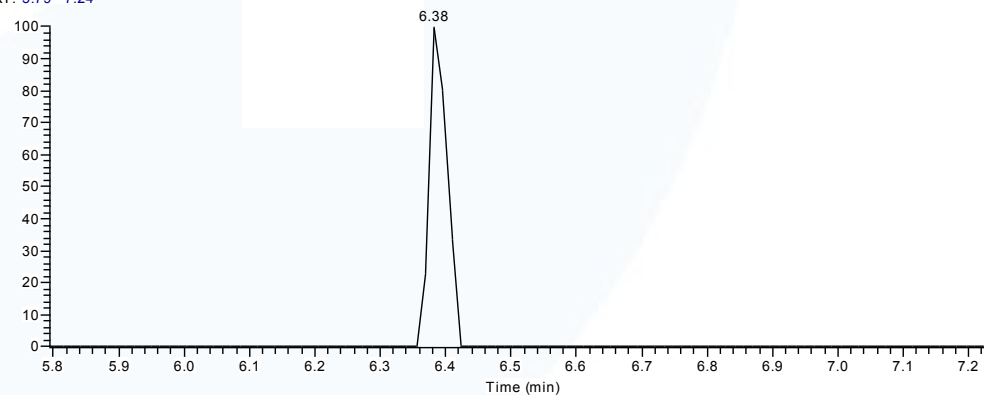
Liver with norfloxacin $m/z = 320.14047$ 1 mDa

RT: 0.00 - 15.62

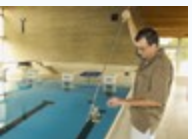


NL:
3.02E4
m/z=
320.13947-
320.14147
MS
09_10_08_
28

RT: 5.79 - 7.24



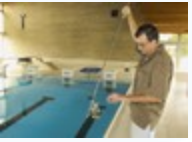
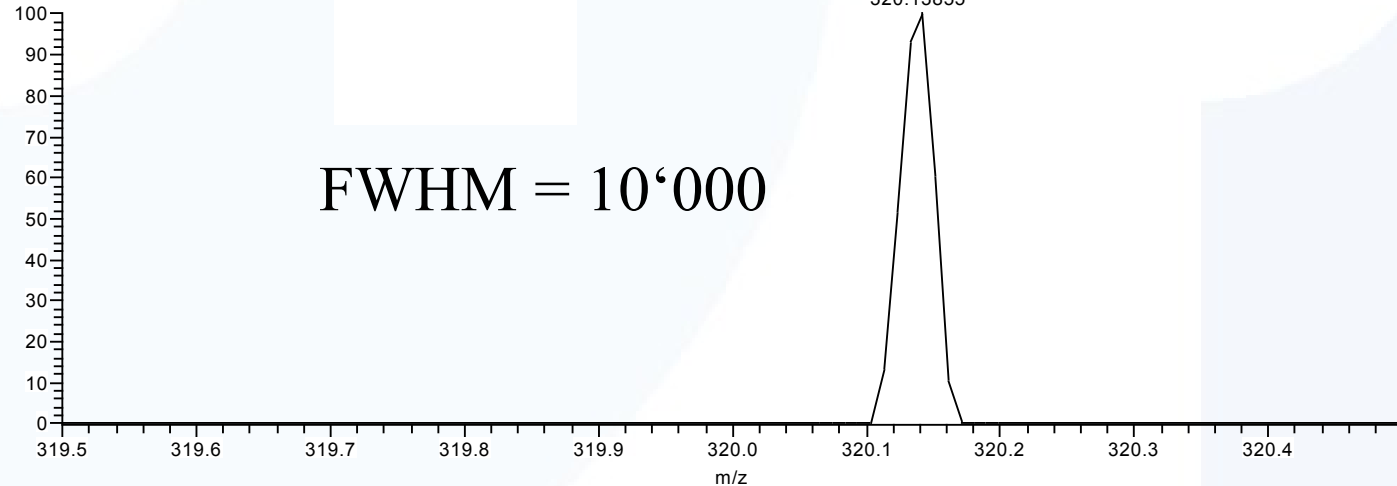
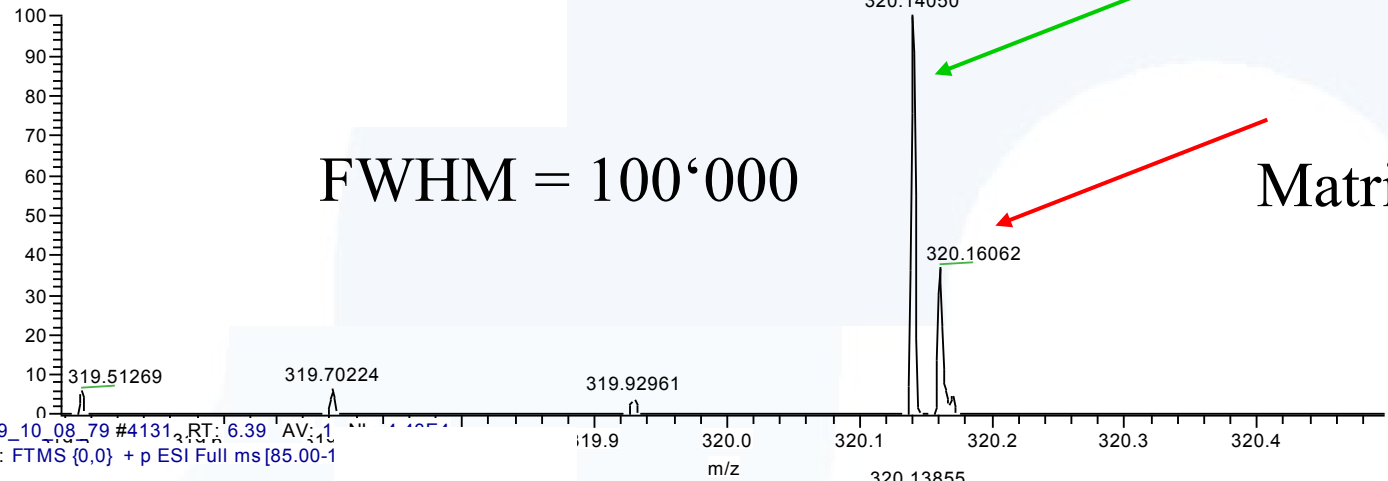
NL:
3.02E4
m/z=
320.13947-
320.14147
MS
09_10_08_
28





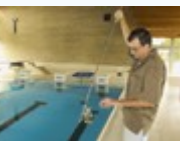
Comparison of Spectra

09_10_08_28 #473-476 RT: 6.37-6.44 AV: 1.1
T: FTMS {0,0} + p ESI Full ms[85.00-]





HRMS surpasses MS/MS selectivity



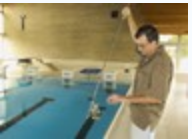
at 50'000 FWHM

Orbitrap and modern TOF reach 50'000 FWHM

A. Kaufmann, P. Butcher Anal. Chimica Acta 673 (2010) 60-72



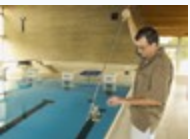
Content overview



- Why HRMS based multiresidue methods ?
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- Successful example of unknown screening



Quantification by single stage Orbitrap





Quantification: Example 1

**Antibiotics in meat (triple validation)
(5 levels; n=4; 3 days)**

Average r^2 of 35 compounds

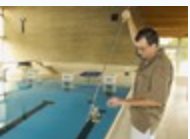
	10-100 $\mu\text{g/kg}$
Orbitrap	0.9962
TOF	0.9578
MS/MS	0.9910

Remarks:

50'000 FWHM

12'000 FWHM (TDC)

Transitions



A. Kaufmann, P. Butcher, Rapid Commun. MS 25 (2011) 979-992

Quantification: Example 2

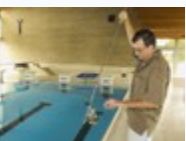


Migration of a pesticide multiresidue method (230 compounds) from MS/MS to Orbitrap

- Method was copied without any change
- 1 day invested for the finding of common interface parameters
- 1 1/2 days invested for calculating + testing accurate masses

Migration of a pesticide multiresidue method (230 compounds) from MS/MS to Orbitrap

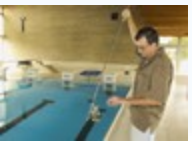
- Sensitivity was slightly poorer
- Quantitative data was very similar
- The Orbitrap handled even challenging matrices
- Data processing was clearly longer for HRMS
- Some compounds made problems in both methods



A. Kaufmann, P. Butcher Journal of AOAC, 95 (2012) 528-548



How about proficiency tests ?





Proficiency tests where we participated with HRMS

Organizer	Matrix	Analyte	Z-score
Rikilt (2010)	Muscle	Oxytetracycline	-1.05
		Sulfadimidine	0.2
		Sulfachloropyridazine	0.33
		Dapson	-1.8
FAPAS 02156	Fish	Ciprofloxacin	0.9
FAPAS 02166	Kidney	Sulfamethoxazole	0.7
		Sulfathiazole	1.3
FAPAS 02173	Kidney	Sulfadiazine	
FAPAS 02170	Fish	Oxytetracycline	0.7





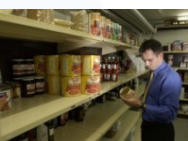
Proficiency tests where we participated with HRMS

Organizer	Matrix	Analyte	Z-score
Rikilt (2011)	Muscle	Oxytetracycline	0.4
		Oxytetracycline	0.31
		Nafcilline	1.29
FAPAS 02174	Fish	Ciprofloxacin	1.5
FAPAS 02175	Corned beef	Abamectin	
		Ivermectin	
FAPAS 02178	Kidney	Oxytetracycline	0.5





In what do we trust ?

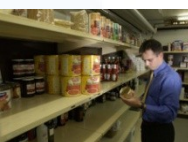


Compound	MS/MS		HRMS	
	Value	Z-score	Value	Z-score
Abamectin	13 $\mu\text{g/kg}$		44 $\mu\text{g/kg}$	
Ivermectin	16 $\mu\text{g/kg}$		25 $\mu\text{g/kg}$	

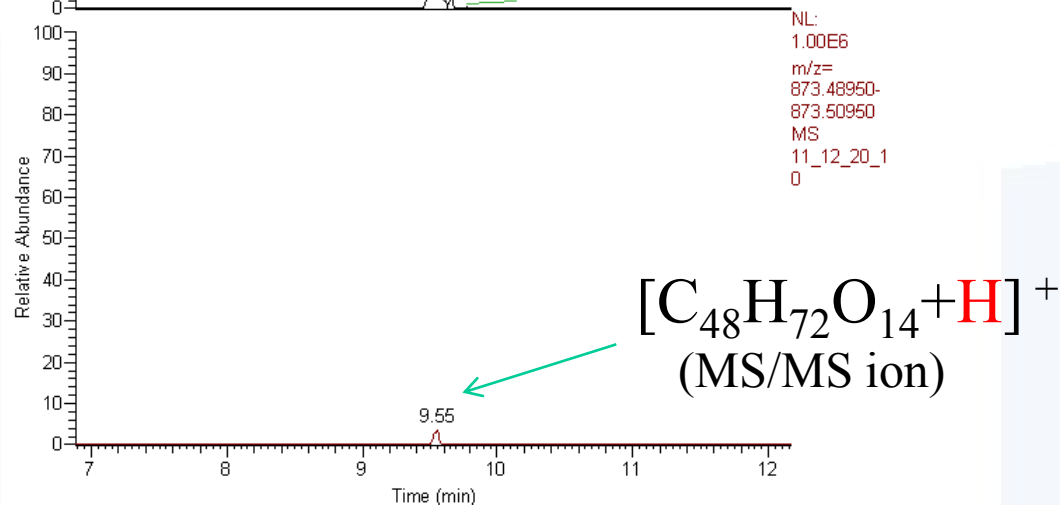
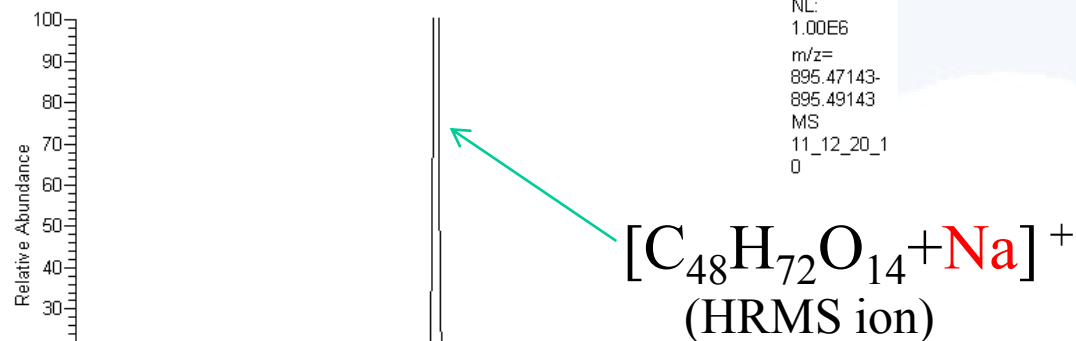


Abamectin

$[M+Na] >> [M+H]^+$ or $[M+Na]$ fragmentation

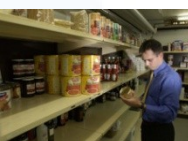


RT: 6.88 - 12.17





MS/MS or HRMS



	MS/MS		HRMS	
Compound	Value	Z-score	Value	Z-score
Abamectin	13 µg/kg		44 µg/kg	
Ivermectin	16 µg/kg		25 µg/kg	



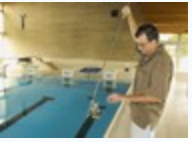
Content overview



- Why HRMS based multiresidue methods ?
- Are we selective enough ?
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- **Identification**
- Strengths and limitations (Single stage Orbitrap)
- Successful example of unknown screening



Identification





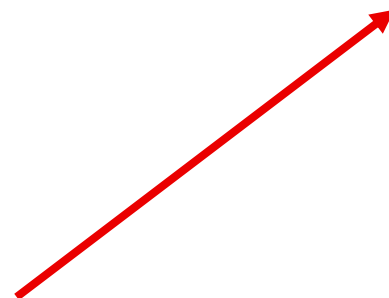
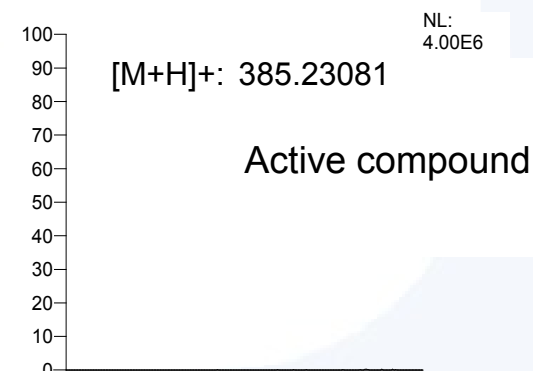
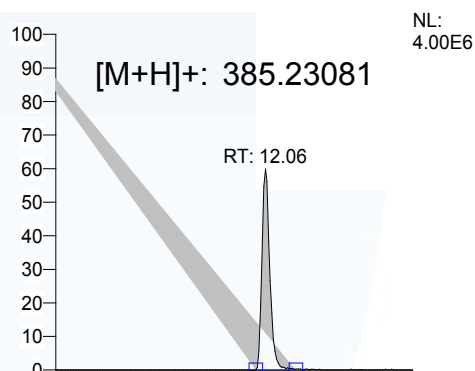
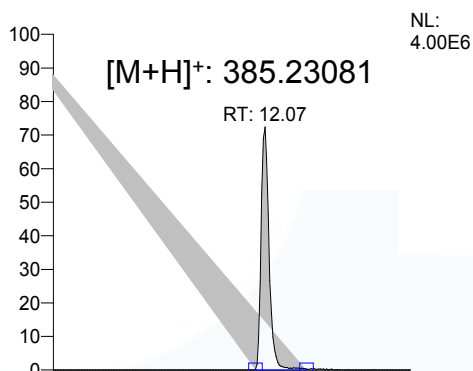
Migration of a pesticide multiresidue method (230 compounds) from MS/MS to Orbitrap

Diafenthion

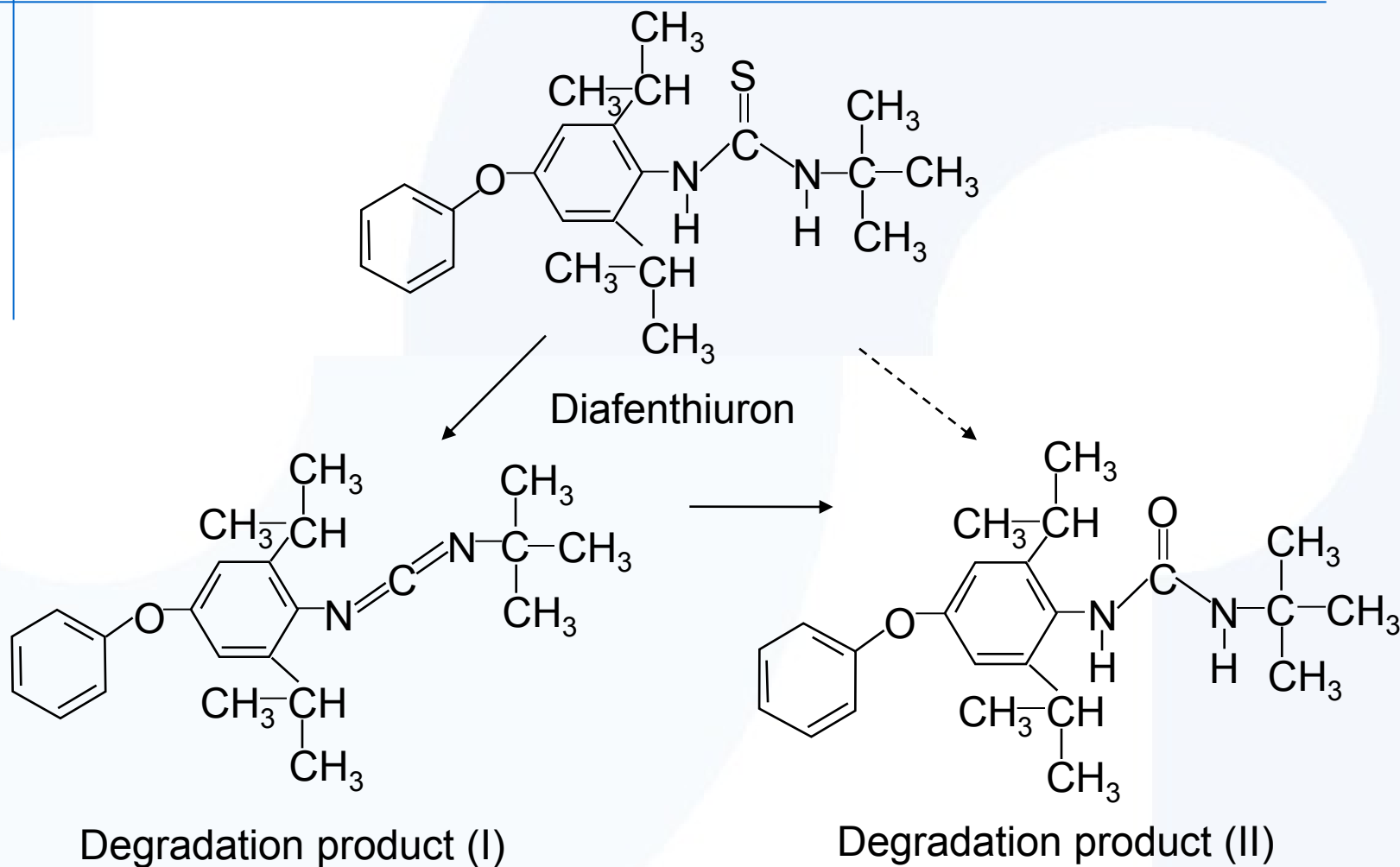
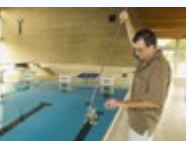
Standard 100 µg/L

Pepper 100 µg/L

Rocket 100 µg/L

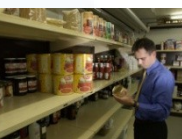


Migration of a pesticide multiresidue method (230 compounds) from MS/MS to Orbitrap





Migration of a pesticide multiresidue method (230 compounds) from MS/MS to Orbitrap

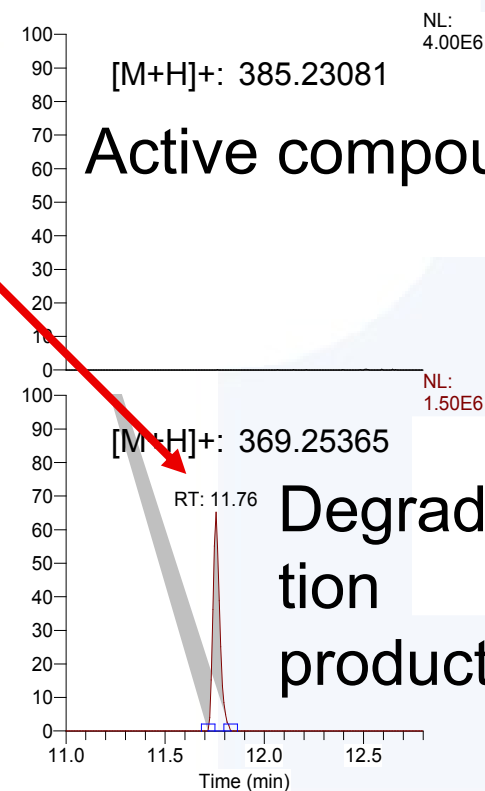
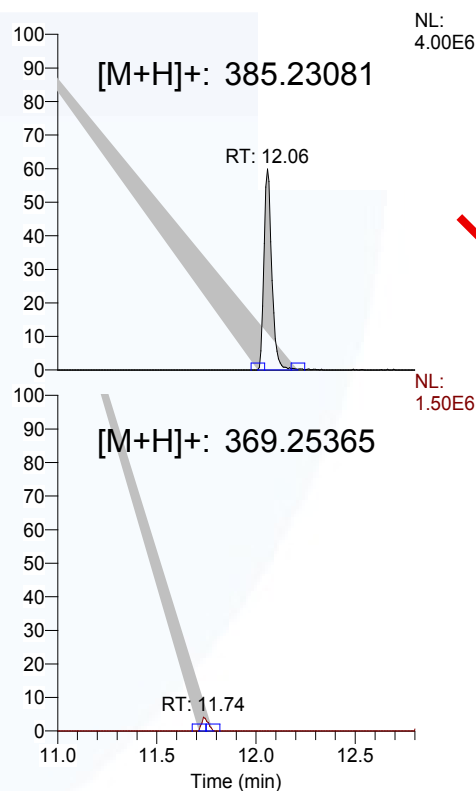
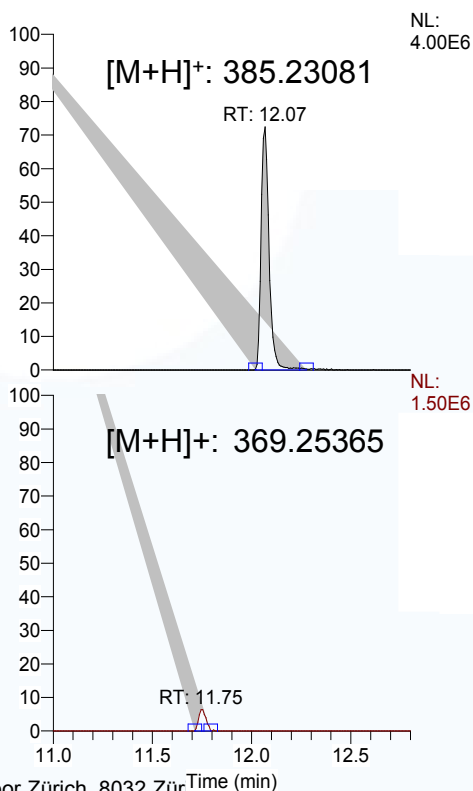


Diafenthiuron

Standard
100 µg/L

Pepper
100 µg/L

Rocket
100 µg/L



Active compound

Degradation
product II



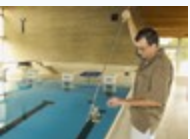
Content overview



- Why HRMS based multiresidue methods ?
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- Identification
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- Successful example of unknown screening

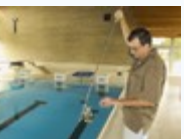


Strengths and Limitations



Quantification

- The Orbitrap is as quantitative as a MS/MS
- No limitations with dwell times and retention time windows
- Every ionizable compound is detectable



Quantification

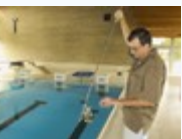
- Current data processing software is the MAIN BOTTLENECK
- Trap has a finite capacity, heavy matrix (proteins) can cause additional problems
- MS/MS is more sensitive when focusing on a few analytes





Ruggedness of the technology

- Instrument is a workhorse
- Easy to operate & calibrate
(high mass stability
no lock mass needed)
- Hardly any maintenance





High resolution mass spectrometry (HRMS) provides new insights

- Exact masses are predictable and universal, SRM are unpredictable and local
- Helps to explain what is wrong with a LC-MS/MS method (e.g signal suppression, degradation)
- A-posteriori and not a-priori hypothesis

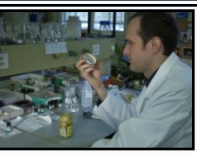
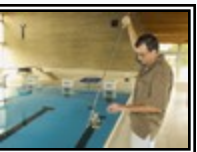




MS/MS shows only a few percent of all present compounds.

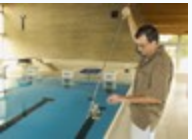
HRMS shows us all ...

**we dare
to think about
and we dare
to ask for**





Content overview



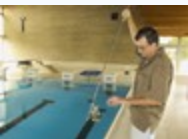
- Why HRMS based multiresidue methods ?
- Are we selective enough ?
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- Identification
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KANTONALES LABOR

ZÜRICH

Structure elucidation of an unauthorized textile dye discovered in a food product (Sumach)



Anton Kaufmann

Official Food Control Authority of the Canton of Zurich Switzerland



Sumac spices



Kingdom: Plantae

Order: Sapindales

Family: Anacardiaceae

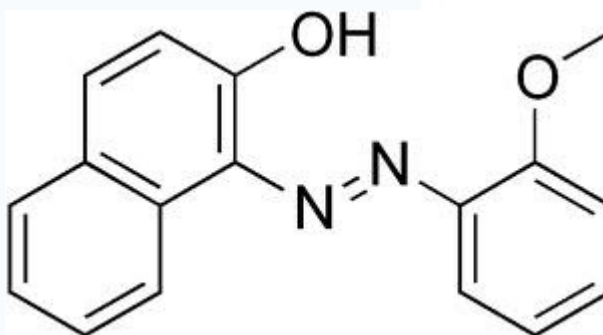
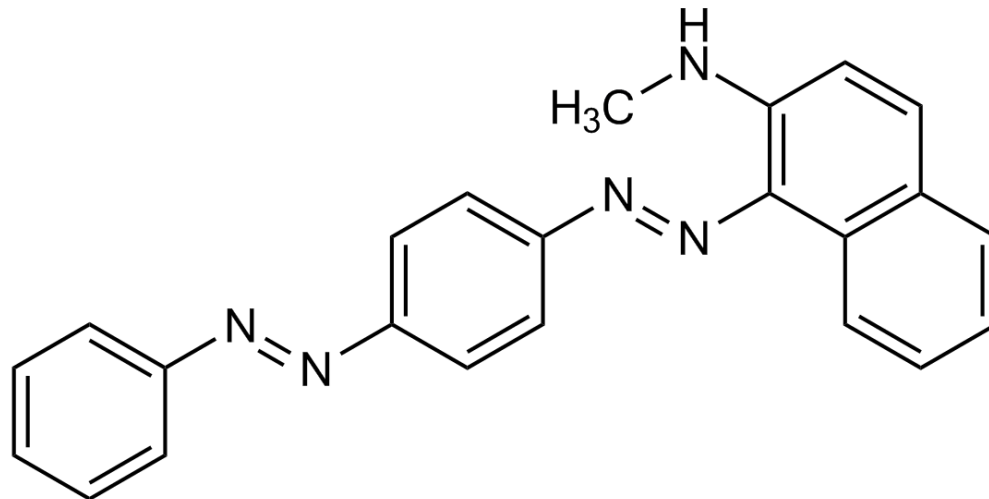
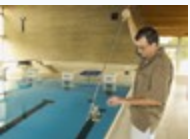
Genus: Rhus

Sumac grows undomesticated in Southern Italy, Iran and Turkey.

Sumac is a popular spice in the Turkish and Persian kitchen.



History of unauthorized use of azo dyes (Sudan red) utilized as food colorant





Toxicology of Sudan red textile dyes



Sudan I is a rodent carcinogen



Affected organs: Liver and bladder

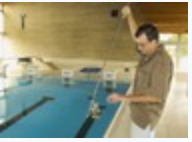


Studies on human and animal Cytochrome P 450 suggested that cancerogenicity can be extrapolated to humans





Sudan red in food



2003 Sudan I was found in curry and chilly



Soon related Sudan III and IV was found as well



A food scandal developed

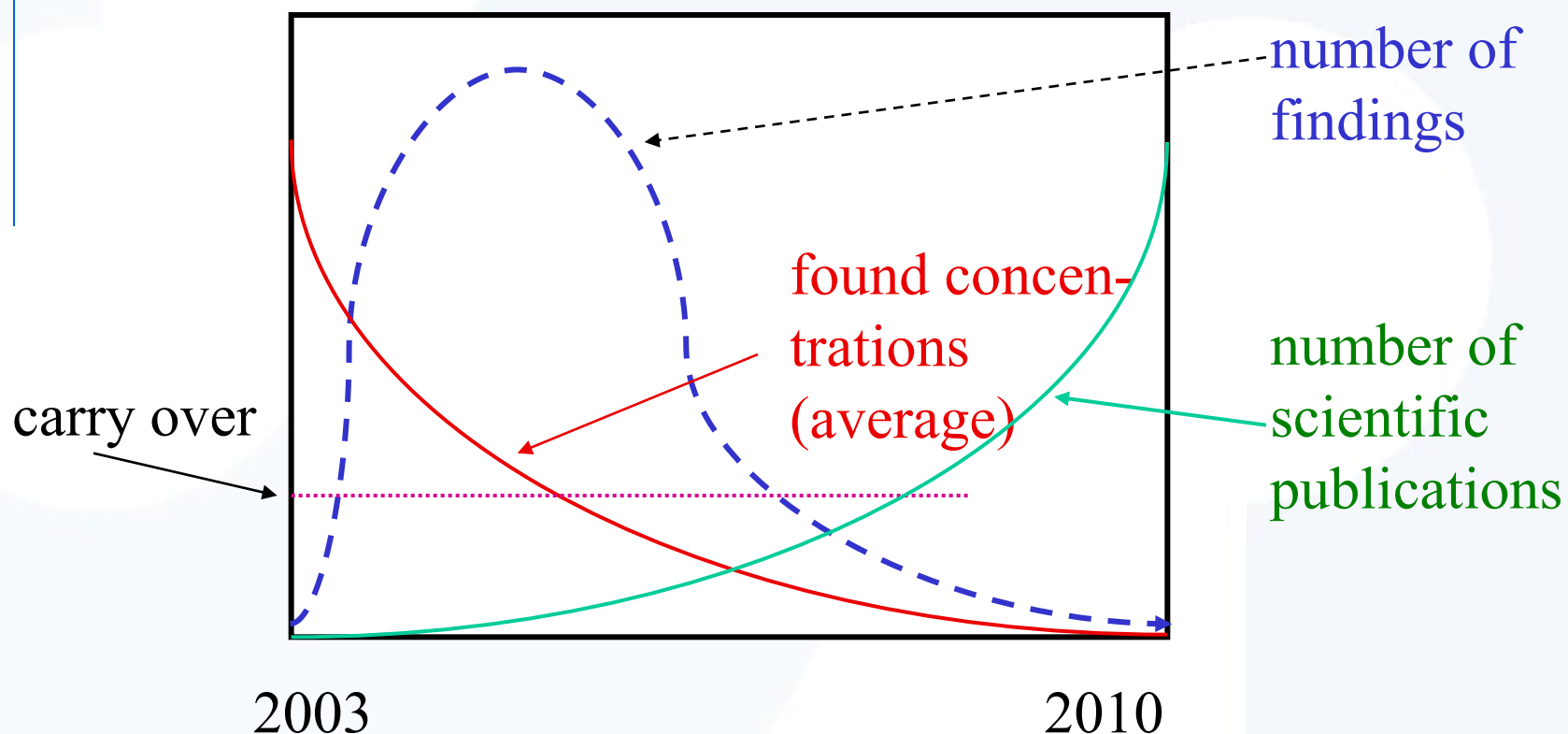


Even the government of Sudan requested to change the name of the dye



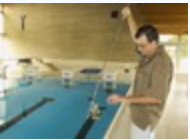
The more we analyze the less relevant the findings

EU rapid alert warnings over the years





A sumac sample contained an unidentified colouring



Investigation for non permitted food coloring (Sudan) in a collaborating institution (Kantonales Labor Thurgau)

The analytical method calls for a gel-permeation sample clean-up

The extract stained the expensive gel-permeation column

A regeneration of the column was not possible



Initial identification steps



Omitting the detrimental clean-up



UV-Vis Spectrum showed similarities with Sudan



No match with any reference compound



LC-MS/MS showed 321>196 und 321>224 transitions



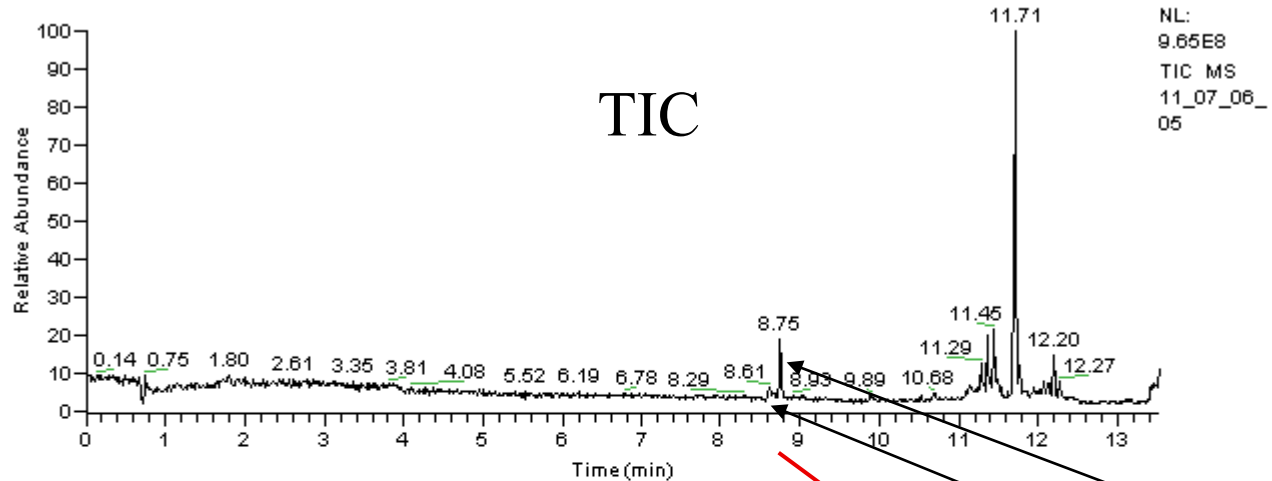


Extract analyzed with High Resolution MS

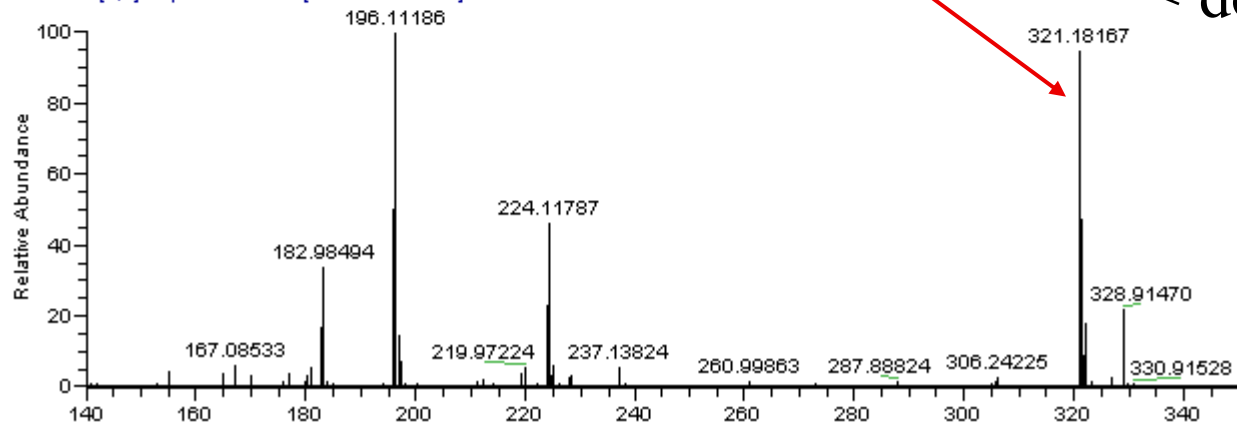
C:\Xcalibur\data\Diverses\11_07_06_05
217829 a)

7/6/2011 1:17:20 PM

RT: 0.00 - 13.51

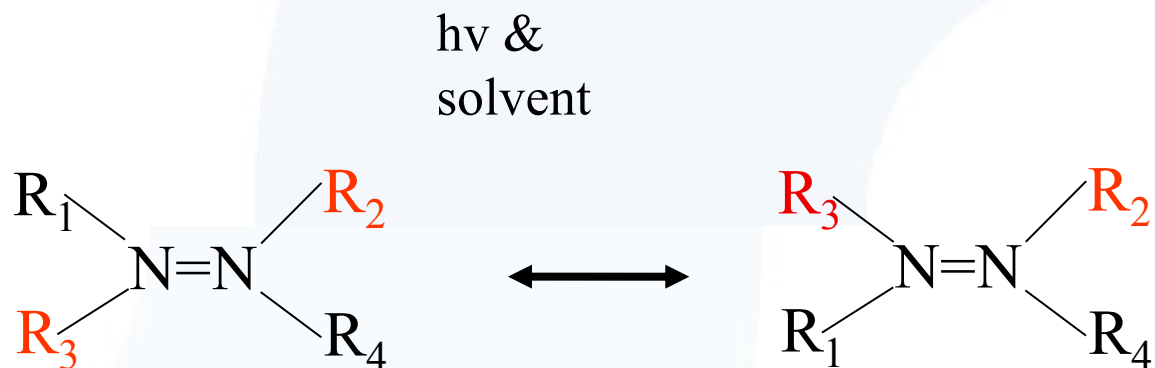


11_07_06_05 #642 RT: 8.76 AV: 1 NL: 2.27E7
T: FTMS {1,1} + p ESI Full ms [140.00-1000.00]

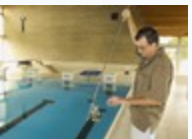




E-Z Isomisation

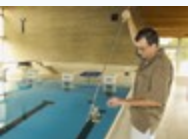


K. Mölder et. al J. Chrom. A. 1160 (2007) 227-234





Elemental composition based on exact mass



Hit Nr.	Formula	mDa	ppm
1	C18 H21 N6	11.5	-0.551
2	C17 H25 O4 N2	6.5	0.786
3	C20 H23 O N3	11	-1.894
4	C15 H23 O3 N5	7	2.129
5	C22 H25 O2	10.5	-3.237
6	C14 H27 O7 N	2	3.466
7	C13 H21 O2 N8	7.5	3.472
8	C12 H25 O6 N4	2.5	4.809

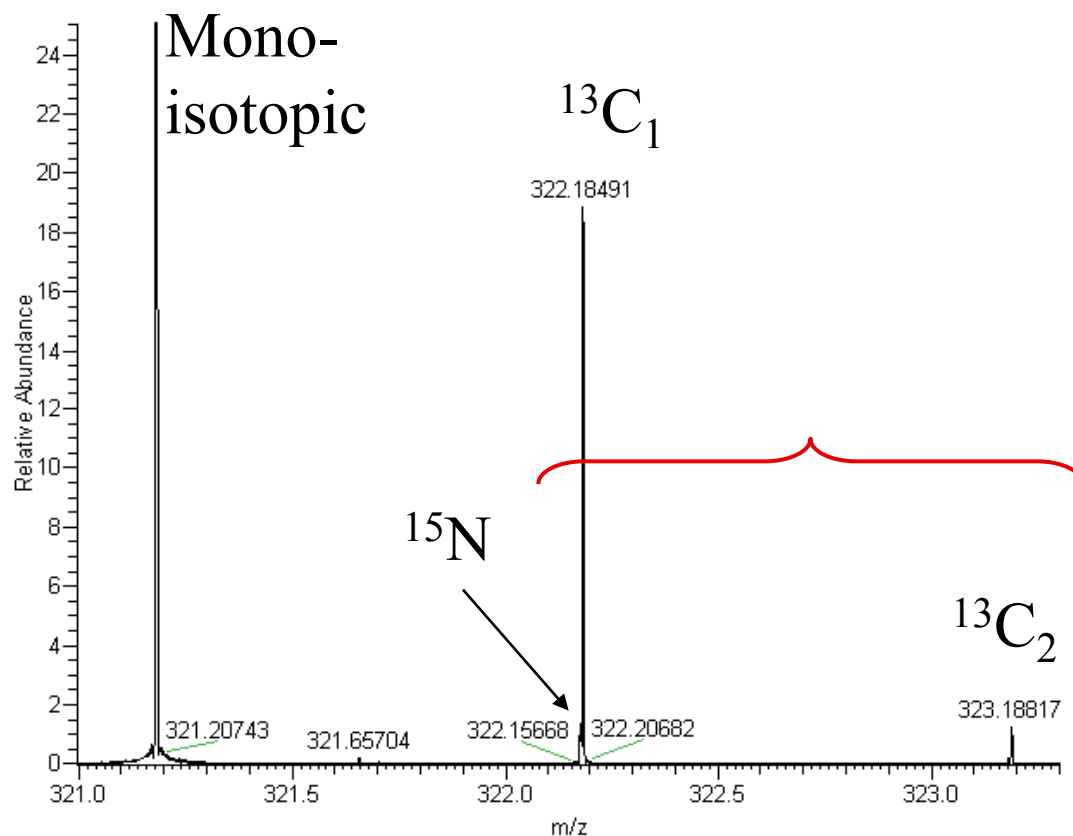


Resolution of nitrogen isotopes

C:\Xcalibur\data\Diverses\11_07_06_05
217829 a)

7/6/2011 1:17:20 PM

11_07_06_05 #642 RT: 8.76 AV: 1 NL: 2.14E7
T: FTMS {1,1} + p ESI Full ms [140.00-1000.00]



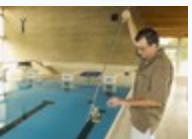
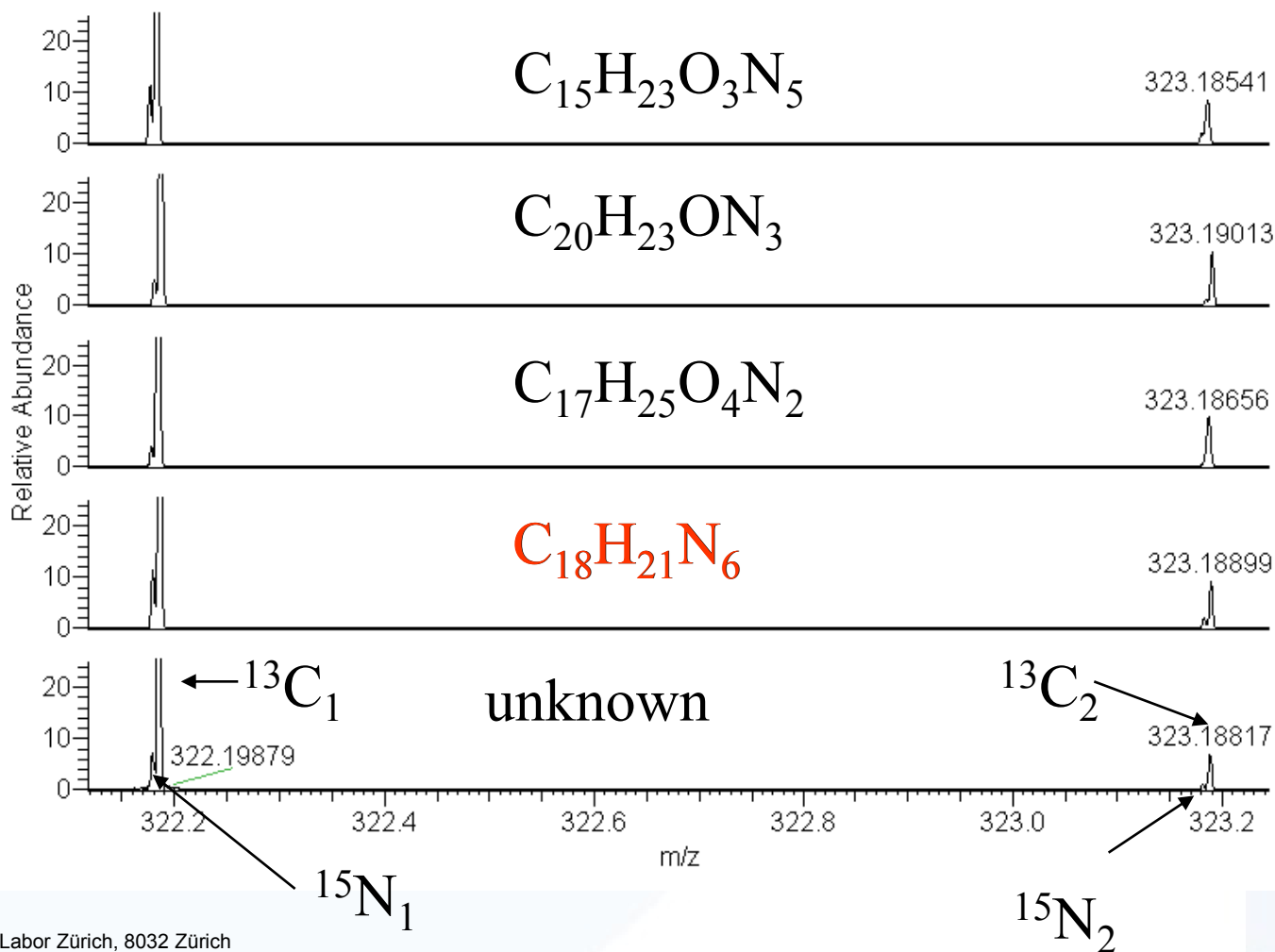


Comparison with the four best hits

C:\Xcalibur\data\Diverses\11_07_06_05
217829 a)

7/6/2011 1:17:20 PM

TG Sudanrot ? ohne Fragmentierung (100000)





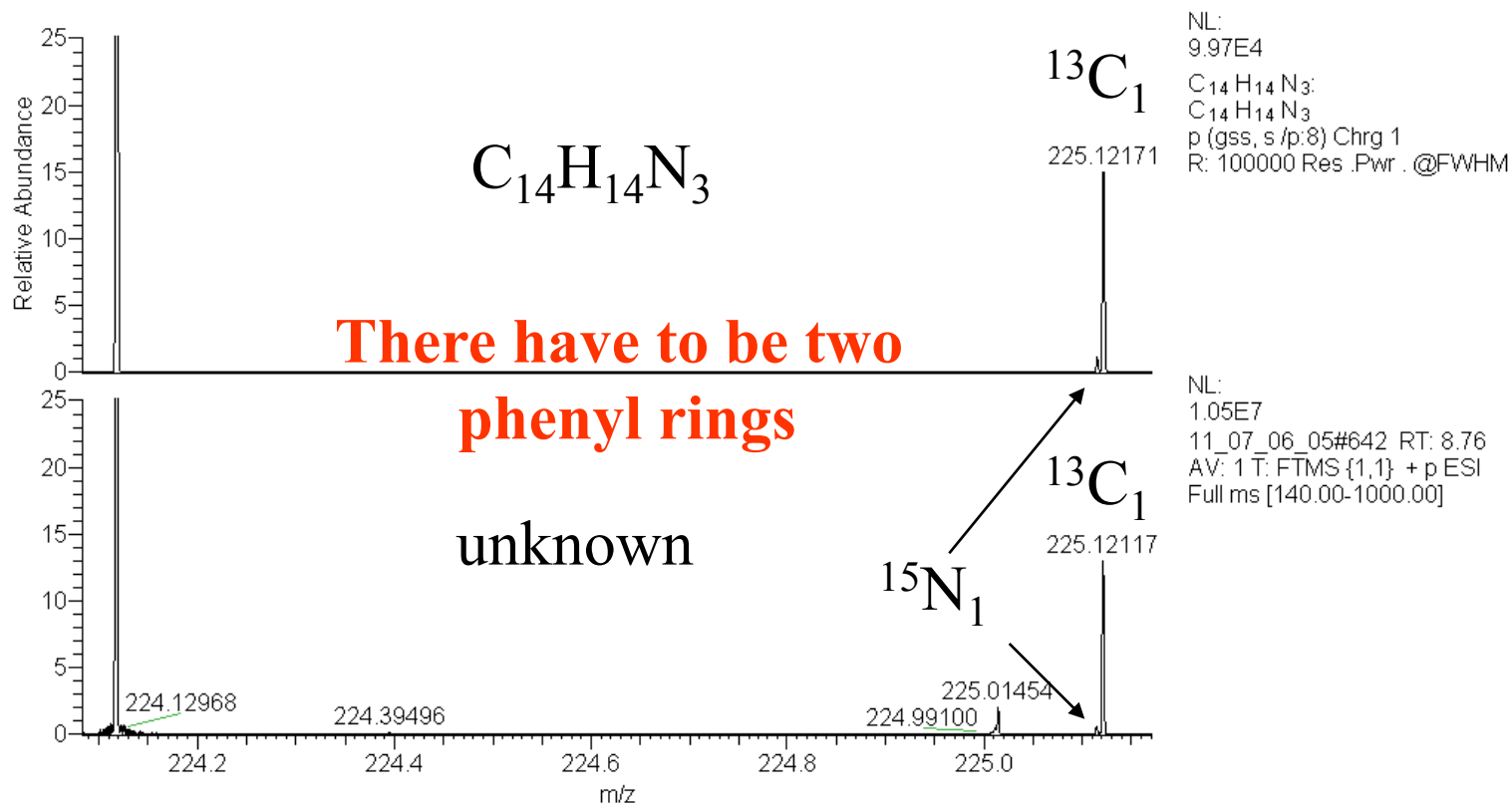
The fragment could be identified as well



C:\Xcalibur\data\Diverses\11_07_06_05
217829 a)

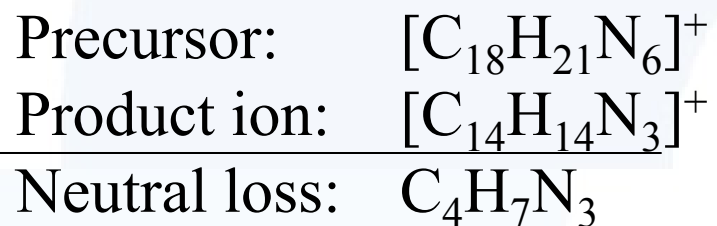
7/6/2011 1:17:20 PM

TG Sudanrot ? ohne Fragmentierung (100000)





An interesting neutral loss



So many nitrogen and so few carbon atoms.
Which moiety is split off?





Printout from Molport.com (C₁₈H₂₁N₆)-H

Molecules with molecular formula C₁₈H₂₀N₆ - Windows Internet Explorer

http://www.molport.com/buy-chemicals/molecular-formula/C18H20N6

molport
search like a chemist

draw & find

Find Chemicals

- Find Chemicals by Structure
- Custom Synthesis Request
- Directory

Buy

Sell

My Account

News and Resources

Queries

Home >> Find Chemicals >> Search Results

Molecules with molecular formula C₁₈H₂₀N₆

We searched in our molecule database and found the following results.

Items found: 100 | Display:

1.

Compound Number
MolPort-000-352-199

IUPAC
2-[(E)-2-[(3,5-dimethyl-1-phenyl-1H-pyrazol-4-yl)methylidene]hydrazin-1-yl] ...

Names
n/a

Chemical Data
SMILES: Cc1nn(c(C)c1\C=N\Nc1nc(C)cc(C)n1)-c1ccccc1
InChI Key: n/a
Molecular Formula: C₁₈H₂₀N₆
Molecular Weight: 320.3916

Buy From Stock

2.

Compound Number
MolPort-003-997-320

IUPAC
13-(1-(1-cyclopropyl-2,5-dimethyl-1H-pyrrol-3-yl)-1,8,10,12-tetraazatricyclo[7 ...

Names
n/a

Chemical Data
SMILES: Cc1cc(C2N=C(N)Nc3nc4ccccc4n23)c(C)n1C1CC1 [t:5]
InChI Key: YJMBHOITJWGCH-UHFFFAOYAN
Molecular Formula: C₁₈H₂₀N₆
Molecular Weight: 320.3916

Buy From Stock

3.

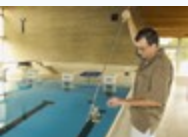
Compound Number
MolPort-004-180-626

IUPAC
N-(1-benzylpyrrolidin-3-yl)-1-phenyl-1H-1,2,3,4-tetrazol-5-amine

Names
n/a

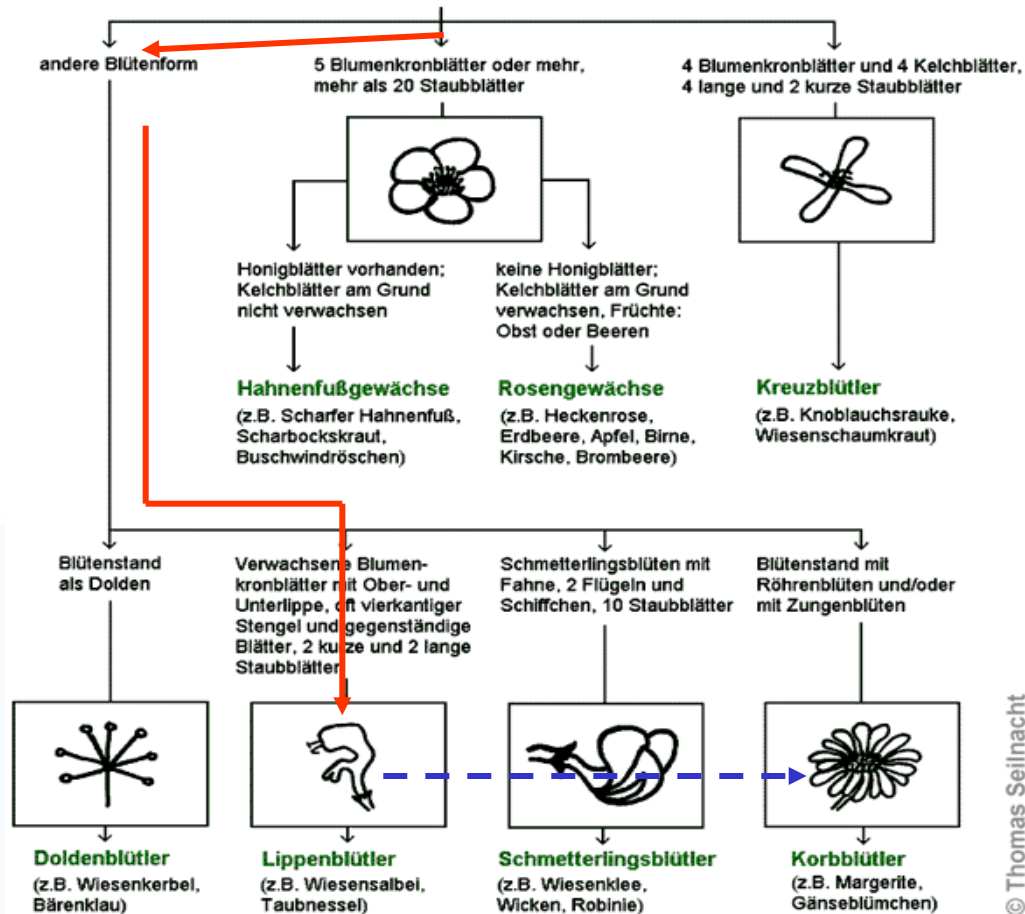
Chemical Data
SMILES: C1=CN=C(N1)Nc2ccccc2N3CCCN3Cc4ccccc4

This goes on page by page



Decision trees to not permit errors

Bestimmungsschlüssel für die 7 wichtigsten Blütenpflanzenfamilien

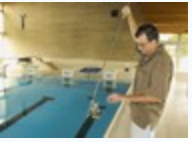


© Thomas Seilnacht





Make a detour ...





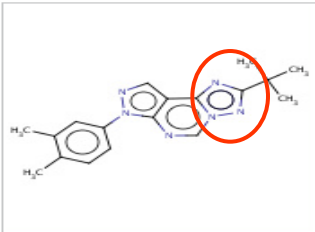
From Molport



Zurück

weiter

Optionen



Compound Number

MolPort-002-621-034

Buy From Stock

IUPAC

4-tert-butyl-10-(3,4-dimethylphenyl)-3,5,6,8,10,11-hexaazatricyclo[7.3.0.0^...

Names

2-(tert-butyl)-7-(3,4-dimethylphenyl)-7H-pyrazolo[4,3-e][1,2,4]triazolo[1,5-c]pyrimidine;

Chemical Data

SMILES:

Cc1ccc(cc1C)-n1ncc2c1ncn1nc(nc21)C(C)(C)C

InChI Key:

FZWQBBZQALUIPF-UHFFFAOYAH

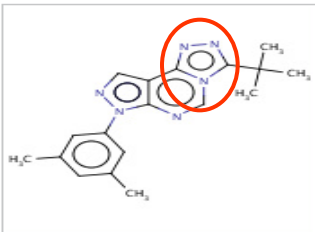
Molecular Formula:

C18H20N6

Molecular Weight:

320.3916

16.



Compound Number

MolPort-002-647-660

Buy From Stock

IUPAC

5-tert-butyl-10-(3,5-dimethylphenyl)-3,4,6,8,10,11-hexaazatricyclo[7.3.0.0^...

Names

3-(tert-butyl)-7-(3,5-dimethylphenyl)-7H-pyrazolo[4,3-e][1,2,4]triazolo[4,3-c]pyrimidine; 3-tert-butyl-7-(3,5-dimethylphenyl)-7H-pyrazolo[4,3-e][1,2,4]triazolo[4,3-c]pyrimidine;

Chemical Data

SMILES:

Cc1cc(C)cc(c1)-n1ncc2c1ncn1c(nnc21)C(C)(C)C

InChI Key:

PLYPPXFKTLJCJV-UHFFFAOYAP

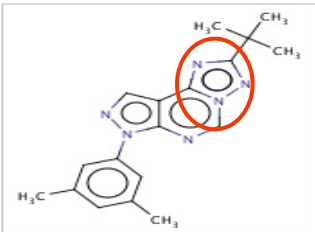
Molecular Formula:

C18H20N6

Molecular Weight:

320.3916

17.



Compound Number

MolPort-002-648-265

Buy From Stock

IUPAC

4-tert-butyl-10-(3,5-dimethylphenyl)-3,5,6,8,10,11-hexaazatricyclo[7.3.0.0^...

Names

2-(tert-butyl)-7-(3,5-dimethylphenyl)-7H-pyrazolo[4,3-e][1,2,4]triazolo[1,5-c]pyrimidine; 2-tert-butyl-7-(3,5-dimethylphenyl)-7H-pyrazolo[4,3-e][1,2,4]triazolo[1,5-c]pyrimidine;

Chemical Data

SMILES:

Cc1cc(C)cc(c1)-n1ncc2c1ncn1nc(nc21)C(C)(C)C

InChI Key:

OYXHBVYDSSCCLV-UHFFFAOYAS

Molecular Formula:

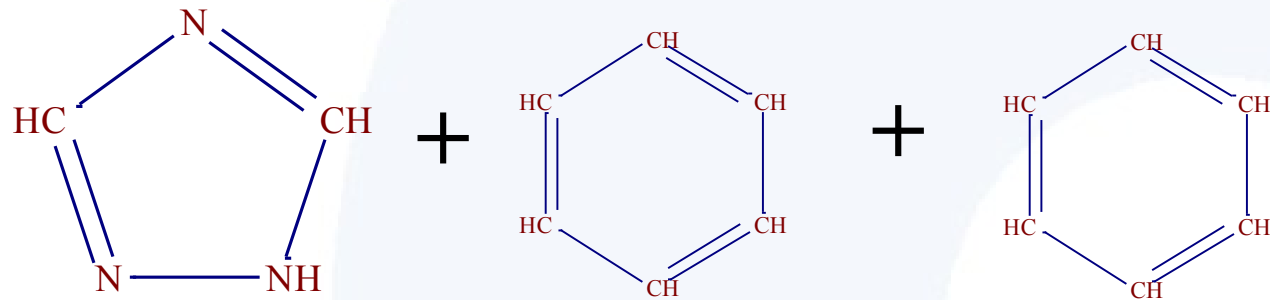
C18H20N6

Molecular Weight:

320.3916

© 060207, Kantonales Labor Zürich, 8032 Zürich

What do we know ?



+



= ?



Substructure search with Google

Google

triazolium phenyl red



SafeSearch - Moderat ▼



e know
s already !



Our firewall does not collaborate



Google-Ergebnis für <http://www.lookchem.com/300w/2010/0617/115682-08-1.jpg> - Windows Internet Explorer

http://www.google.ch/imgres?imgurl=http://www.lookchem.com/300w/2010/0617/115682-08-1.jpg&...

Datei Bearbeiten Ansicht Favoriten Extras ?

Favoriten http-www.admin.ch-ch-ds-8... Web Slice-Katalog

Research chemical suppliers... Google-Ergebnis für http://... 12221-69-1, CAS Number: 68...

Suchen: Zurück Weiter Optionen

FORTIGUARD Web Filtering **FORTINET** REAL TIME NETWORK PROTECTION

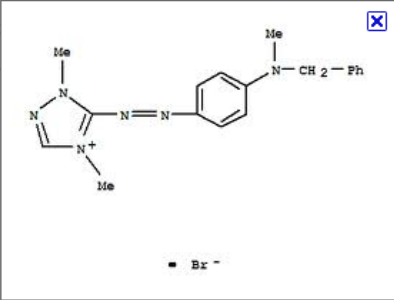
Web Page Blocked

You have tried to access a web page which is in violation of your internet usage policy.

URL: www.lookchem.com/cas-115/115682-08-1.html
Category: Health and Wellness

To have the rating of this web page

Powered by FortiGuard.



Google bilder

[Website mit diesem Bild](#)
[lookchem.com](#)

[Bild in voller Größe](#) - 1.7x größer

Größe: 443 × 329
Typ: 7KB JPG

Dieses Bild ist möglicherweise urheberrechtlich geschützt.

<http://www.lookchem.com/cas-115/115682-08-1.html>

Internet 100%



It is an industry chemical !!



http://www.lookchem.com/cas-122/12221-69-1.html

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Product Suppliers Buy offers Encyclopedia

Please Enter CAS NO, Product Name, Formula [Search](#)

Home > Products > 12221-69-1

Refine Suppliers

Supplier Location: China (Mainland) (5)

Business Type: Manufacturers (5)

Membership Level: Platinum supplier (4)

All Suppliers Suppliers Of Custom Synthesis

Select All Contact Now

12221-69-1 [Competitive Product](#)

Product name: basic red 46. Package: in 25 kg bags. We are your better partner, we can assure you of the quality of our product. Any new inquiries will be highly appreciated. [Contact Now](#)

China (Mainland)

Tel: 0571-85270505-112

Address: Flat E 16/F 1 Huazhe Plaza, Hangzhou, China

12221-69-1

BASIC RED 46

Jinan Haohua Industry CO., LTD

China (Mainland)

Chemical Structure:

CN(C)Cc1ccc(cc1)/N=N/c2nc(C)n(C)c2

$C_{18}H_{23}BrN_6$

$[C_{18}H_{23}N_6]^+(sic)$

according to Orbitrap: $C_{18}H_{21}N_6$!!!!

recounted: $C_{18}H_{21}N_6$

extremely strength product used not in the different industries. The packing is 25 kg bags

[Contact Now](#)

Click here to send inquiry

Detail of > 12221-69-1

CAS No: 12221-69-1

Name: 1H-1,2,4-Triazolium, 1,4-dimethyl-5-[2-[4-(phenyl(methyl)amino)phenyl]diazenyl]bromide(1:1)

Formula: $C_{18}H_{23}BrN_6$

Molecular Structure:

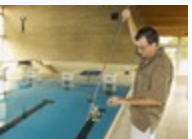
Synonyms: 1H-1,2,4-Triazolium, 1,4-dimethyl-5-[[4-[meth(phenylmethyl)amino]phenyl]azo]-, bromide (9CI); Astrazon Red FBL; Basic Red 46; C.I. Basic Red 46; Cationic Red GRL; Cationic Red SD-GRL; Cationic Red X-GRL; Estrol Red N-GSL; Kayacryl Red GRL; Kayacryl Red GRL-ED; Kayacryl Red GRL-N; Maxilon Red GRL; Maxilon Red GRL Pearls; Synacril Red G;

Molecular Weight: 403.3194

Boiling Point: 470.8 °C at 760 mmHg



Can we see through it ?





Which structure has CAS: 12221-69-1 outside China ?



ChemSpider | 5-[(E)-{4-[benzyl(methyl)amino]phenyl}diazenyl]-1,4-dimethyl-4,5-dihydro-1H-1,2,4-triazol-1-ium bromide - Win...

http://www.chemspider.com/Re

ChemSpider... x 12221-69-1 - G...

ChemSpider
The free chemical database

About | More Sea | Web APIs | Help

Search term: 12

5-[(E)-{4-[benzyl(methyl)amino]phenyl}diazenyl]-1,4-dimethyl-4,5-dihydro-1H-1,2,4-triazol-1-ium bromide

Br⁻

2D 3D Load Save Zoom

- Double-bond stereo

140211 (ChemSpider ID)

C₁₈H₂₃BrN₅

Similar

Wikibox

Mass

PhysChem Properties

Systematic name

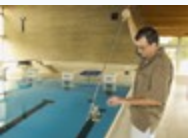
5-[(E)-{4-[benzyl(methyl)amino]phenyl}diazenyl]-1,4-dimethyl-4,5-dihydro-1H-1,2,4-triazol-1-ium bromide

Give Feedback

Leads to Insight



Inside the chemical webs



Mass according to ChemSpider: $C_{18}H_{23}BrN_6$

Mass of the free bases according to ChemSpider: $C_{18}H_{22}N_6$

Mass of protonated ESI ion according to.
ChemSpider: $[C_{18}H_{23}N_6]^+$

Mass of detected ESI ion: $[C_{18}H_{21}N_6]^+$



ChemSpider = NCBI Datenbank

basic red 46 - PubChem Public Chemical Database - Windows Internet Explorer

http://pubchem.ncbi.nlm.nih.gov/summary/summary.cgi?cid=159441

PubChem Compound

PubMed | Entrez | Structure | PubChem | Help

basic red 46 - Compound Summary (CID 159441)

Table of Contents

- BioMedical Annotation
 - Chemical Classification
 - Safety and Toxicology
 - Literature Links
- Synonyms
- Properties
- Descriptors
- Compound Information
- Substance Information
 - Category
- Exports

BioMedical Annotation: (Total: 1)

basic red 46

Related Chemical Classification

- Organic Chemicals
- Azo Compounds

Safety and Toxicology

TOXLINE - Citations to the toxicological literature

Literature

Depositor-Supplied Synonyms: (Total: 10)

Sort: Filtered

Basic red 46

5-[(e)-{4-[benzyl(methyl)amino]phenyl}diazenyl]-1,4-dimethyl-4,5-dihydro-1h-1,2,4-triazol-1-ium bromide

C.I. Basic Red 46

AC1L4LU9

AC1Q1RD7

AR-1G6689

Structure & Quick Link Bar

2D 3D

Chemical structure diagram showing the 2D structure of Basic red 46, a triazole derivative with a benzyl(methyl)amino group and a diazenyl group.

Compound ID 159441

Molecular Weight 403.31942 [g/mol]

Molecular Formula C₁₈H₂₃BrN₆

H-Bond Donor 1

H-Bond Acceptor 5

Links

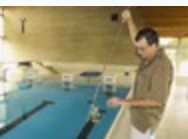
- NLM Toxicology Link
- Chemical Structure Search

Similar Compounds: 11 Links

Substances: 7 Links



CAS Nr. Search with Google



12221-69-1 - Google-Suche - Windows Internet Explorer

http://www.google.ch/search?um=1&hl=de&safe=off&biw=1920&bih=1019&as_st=y&tbm=isch&sa=1&q=12221-69-1&btnG=Suche&oq=12221-69-1&aq=f&aql=&aql=&gs_sm=s&gs_up

Datei Bearbeiten Ansicht Favoriten Extras ?

Favoriten http-www.admin.ch-ch-d-sr-8... Web Slice-Katalog

ChemSpider - Service Unavail... 12221-69-1 - Google-Suche

Web Bilder Videos Maps News Übersetzer Google Mail Mehr

Google

12221-69-1

Ungefähr 182 Ergebnisse (0.04 Sekunden)

Google.com in English Erweiterte Suche

Alles Bilder Videos News Mehr

Nach Relevanz sortiert
Nach Thema sortiert

Alle Größen
Groß
Mittel
Piktogramm

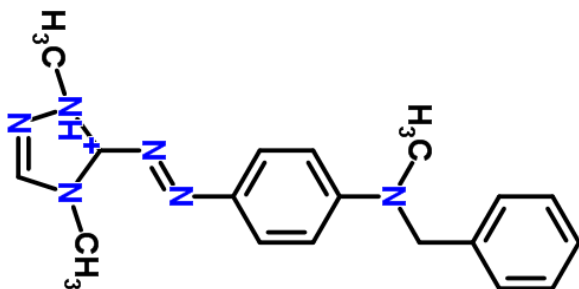
Chemical structure of 12221-69-1 (a diazo compound) is shown, along with other chemical structures and images related to the search results.



CAS: 12221-69-1 (Are we twins?)

according
to Chemspider
+ Pubchem

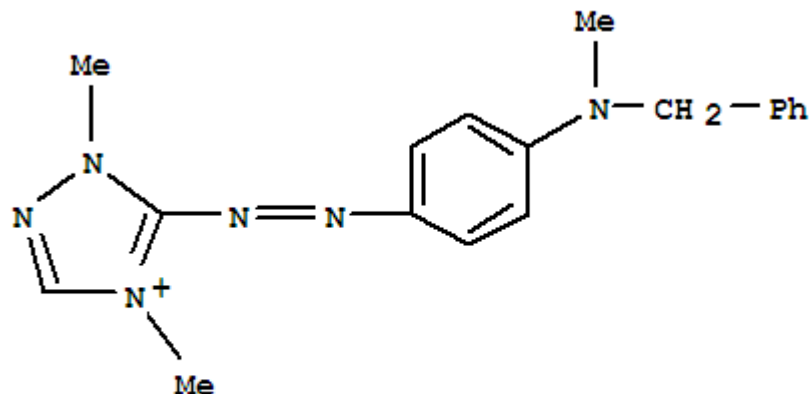
Br⁻



specified : C₁₈H₂₃BrN₆

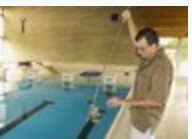
recounted: C₁₈H₂₃BrN₆

according
to Lookchem



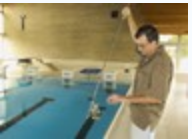
C₁₈H₂₃BrN₆

C₁₈H₂₁BrN₆





Understanding the Mystery

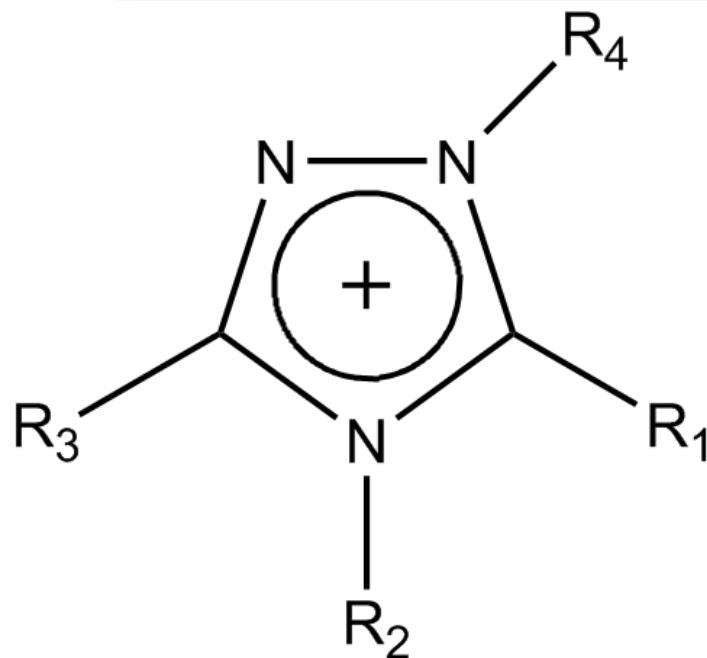


ພິເສດ...! ປະເພດກັບແກ້ມ		
ປາຂັນໝັງ	Smoked fish with vegetable	35,000K
ປາເຄິງແຊ່ເບຍ	Mystus fish with beer	40,000K
ປານິນແຊ່ເບຍ	Black fish with beer	35,000K
ປາເຄິງແຊ່ໂຊດາ	Mystus fish with soda	40,000K
ປານິນແຊ່ໂຊດາ	Black fish with soda	35,000K
ປາເຄິງລວກ	Boiled Mystus fish	40,000K
ປາຫັງລວກ	Boiled Skin fish	30,000K
ກຸ້ງເຜົາ	Grilled Prawns	35,000K
ກຸ້ງລວກ	Boiled Prawns	35,000K

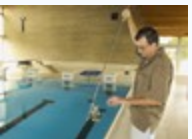


Who is right ?

All refer to Triazolium:



Stable Heterocyclic Structure; therefore we should not expect MS/MS fragmentation

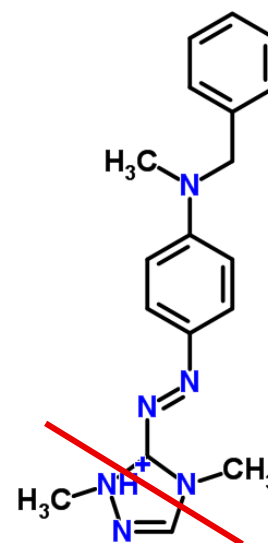




CAS: 12221-69-1 (which one ?)

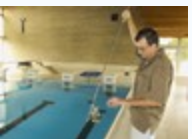
Would undergo MS fragmentation

Br^-



Is not a conjugated system, therefore not Red

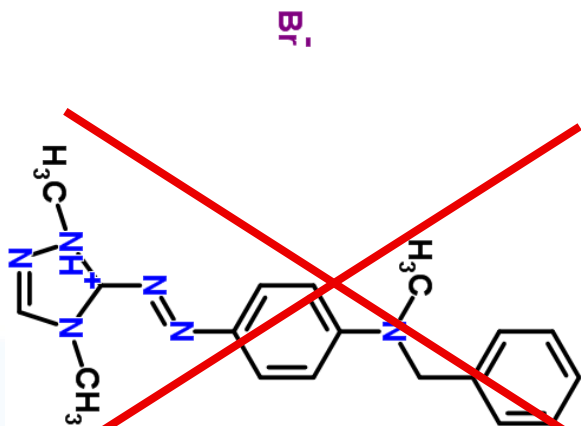
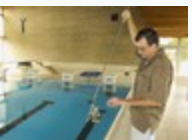
Structure is probably wrong !?!





The Hypothesis

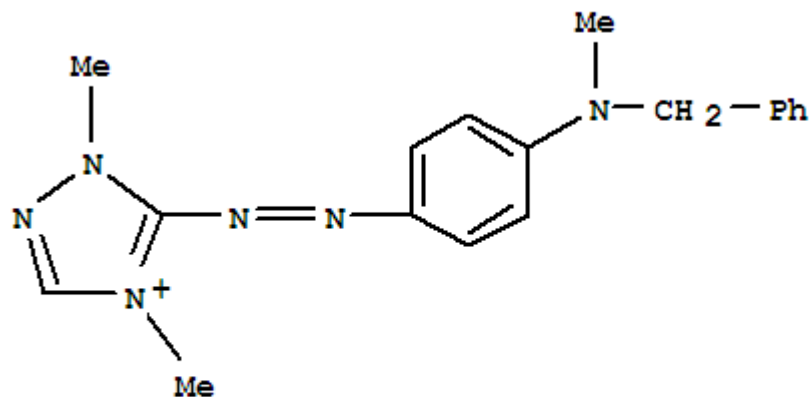
according Chemspider
+ Pubchem



~~declared: $\text{C}_{18}\text{H}_{23}\text{BrN}_6$~~

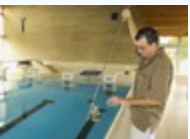
recounted: $\text{C}_{18}\text{H}_{23}\text{BrN}_6$

according Lookchem



~~$\text{C}_{18}\text{H}_{23}\text{BrN}_6$~~

$\text{C}_{18}\text{H}_{21}\text{BrN}_6$



The Americans can do
the mathematics

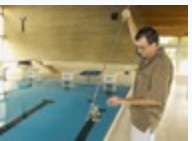
Everybody can do
Copy-Paste



Die Chinese can handle
the stokes (double bonds)



Where is Basic Red 46 used?



Google search results for "basic red 46".

Google search results for "basic red 46". The search results show several links related to the chemical, including information from chemicalbook.com and a PDF document from dormer.ca. The results also mention "Foot dermatitis caused by the textile dye Basic Red 46 in acrylic ..." and "Allergic contact dermatitis to basic red 46 occurring in an HIV ...".

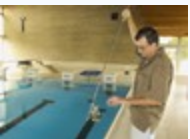
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textile dye causing dermatitis

Hair dye



To tear one's hair



Our collaborator tired to organize a reference material



No reference material supplier stocks this compound



Producers from China are willing to sell 500 kg



In-house synthesis is complex



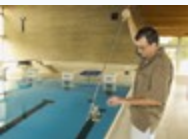
Chemists from BASF answer e-mails
(discussion about the number of double bonds)



German textile dye agent promises a commercial sample
in months



Being so close and having to wait!

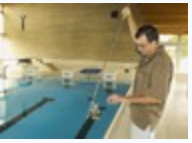




When we can't obtain the real stuff (certified reference materials)?

How about looking
for a consumer
product claiming to
contain

Basic red 46 ?





Everything about hair



Basic Red 46 haare färben

Ungefähr 4'410 Ergebnisse (0.07 Sekunden) Google.com in English Erweiterte Suche SafeSe

[Haar färben | perfecthair.ch](#) Anzeige

www.perfecthair.ch
20% auf viele Haar Produkte Hier direkt im E-Shop bestellen!

[SEMI-PERMANENT CONDITIONING HAIR COLOUR RINSE PITCH BLACK Semi ...](#) Anzeige

www.codecheck.info/.../haarpflege/haarfaerbemittel/.../SE... - Im Cache
9. Nov. 2010 – MAY CONTAIN: basic brown 17, basic brown 16, basic red 76, basic yellow 57, basic violet 3 ... basic red 46, basic orange 2, basic violet 4, basic violet 14, Viele Frauen färben ihre Haare selber und greifen zu den ...

["Rot = Pink?" Testbericht für Smart Beauty Smart Colour](#)

www.dooyoo.de > Mode & Beauty > Haarpflege - Im Cache
2. Dez. 2008 – Red - Amber Flame - Warm Blond - Cool Blond Fazit An sich bin ich ... +/-: Basic Orange 1, Basic Red 2, Basic Red 22, Basic Red 46, Basic Violet 2, ... Genauso ist es, wenn die Haare sofort brechen. Wer die Färbung ...

[La Riché Directions Test - Pink Pink Pink sind meine Haare ...](#)

www.yopi.de > ... > La Riché Directions - Im Cache
★★★★★ Erfahrungsbericht von cachmere - 16. Jun 2008
16. Juni 2008 – Platz 24 in der Kategorie "Haarfarben, Tönungen & Colorationen". ... Basic Green 4, Basic Red 46, Basic Orange 2, Basic Violet 4, Basic Violet 14, Basic Blue 7, Basic Blue 9, ... Färbt nicht nur die Haare, 06.05.2005 ...

[La Riché Directions Haarfarben - Erfahrungsbericht - Farbe bekennen!](#)

www.ciao.de > ... > Erfahrungsberichte - Im Cache
9. März 2007 – Basic Red 46, Basic Violet 4, Yellow 2, Red 1, Midnight Blue 28 ... Doch dabei bitte Beachten das die Haare leider sehr hell gefärbt sein ...

[Colorationen Produkte | Intensiv-Color-Creme, pinot red 465 ...](#)

Basic red 46



By a hair

Browser window showing the website **dooyoo Kaufberatung online**. The URL is <http://www.dooyoo.de/haarpflege/smart-beauty-smart-colour>.

The website features a navigation bar with categories: Handy, Fernseher, Audio-Hifi, Apotheke, Computer, Fotografie, Games, Haushalt, Kosmetik, and Alle Kategorien. The current page is titled "Smart Beauty Smart Colour" and displays a product image of a hair color box.

Smart Beauty Smart Colour ★★★★★

ab €

Produkttyp: [Smart Beauty Haarpflegeprodukte](#)

Neuester Testbericht: ... Red - Amber Flame - Warm Blond - Cool Blond Fazit: bin ich eigentlich ganz zufrieden mit der Färbung von Smart Colour. Diese F...

Google-Anzeigen

[Shampoo Haarausfall?](#)
[Riverside-center.ch/Shampoo+haarausfall](#) Schweizer Qualität zu bezahlbarem Preis: Dr. von Albertini, Zürich.

Buttons: [Preisvergleich](#), [1 Testbericht](#), [Testbericht schreiben](#)

Rot = Pink?
Smart Beauty Smart Colour

Name des Mitglieds: [BulmaZ](#)

[Nachricht an dieses Mitglied senden](#)

[In meinen Freundeskreis aufnehmen](#)

[Email bei neuen Testberichten](#)

Produkt:

Weitere Kategorien:

- Hautpflege
- Parfum
- Wellness
- Körperpflege
- Makeup

Wer hat diesen Testbericht bewertet? (95 Mitglieder)

Gesamtbewertung: Sehr hilfreich
[Alle 95 Bewertungen](#)

Letzte Kommentare:

- [daturaferox](#)
04.12.08
Blondierung sparen? Die steckt da einfach rein!
02.12.08
Ich mache mit der Nuance Warm blond





A rather hairy source

← → http://www.dildo-king.ch/product.php?id_product=91 Willkommen beim Kantona...

Datei Bearbeiten Ansicht Favoriten Extras ?

DILDO-KING.ch

Dildo-King Schweiz

schnell, zuverlässig

👑 DILDO-KING 👑 DILDO-KING 👑 DILDO-KING 👑 DILDO-KING 👑 DILDO-KING

TRANSLATOR

Afrikaans

SCHLAGWÖRTER/TAGS

Fun Factory Crystal Bade-Enten
BIG TEAZE TOYS Anal G-Punk
Nature Skin Bad Kitty Smile Vibe
Therapy NEXUS Be Sexy
TITANMEN Strap On Rocks-Off
Waver Bizarre Analvibrator

KATEGORIEN

Geschenkkategorie Love Paket

- ☑ Kondome - Verhütung
 - Kondome von Billy Boy
 - Kondome von Blausiegel
 - Kondome von Durex
 - Kondome von London
 - Kondome von Lümmeltüte
 - Kondome von Ritex
 - Kondome von Secura

Startseite > bikini colour carmine pink 3 g

BIKINI COLOUR CARMINE PINK 3 G



Färbemittel-Set für die Intimbehaarung. Farbe: F...
Geeignet für alle Haartyp...
Komplettes ...

Mehr Infos

12,80 Fr. i...

Artikelnummer : 0619

Menge: 1

In den Warenkorb

Zum Wunschze...

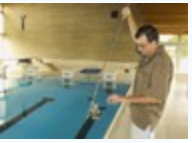
🌟 Durch den Kauf die...
können Sie die folgender...
sammeln **1 Treue-Pun**...
bringt Ihnen **1 Punkt** d...
einen Gutschein umgewar...
Höhe von 0,20 Fr..

> | Einem Freund senden
> | Drucken
> | Totalansicht





A store for punks



Tansini

Scherzartikel

Sie finden bei uns

► Ballons

► Furzkissen

► Girlanden

► Juckpulver

► Stinkbomben

► Lachsäcke

► Spinnen



► Konfetti

► Luftschlangen

Über uns

Events

Theaterschminke

Coiffeur-Zubehör

► Scherzartikel

Feuerwerk

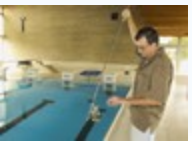
Accessoires

Kontakt

Tansini - Limmatquai 4 / Torgasse - 8001 Zürich - Tel und Fax 044 251 79 15 - Email: info@tansini.ch



No success with hair colour. Hit with Semi-permanent hair colour





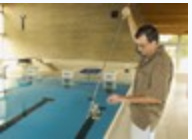
Readability ?



Basic Red 46 ?!



The disappointment



May contain:



The Proof

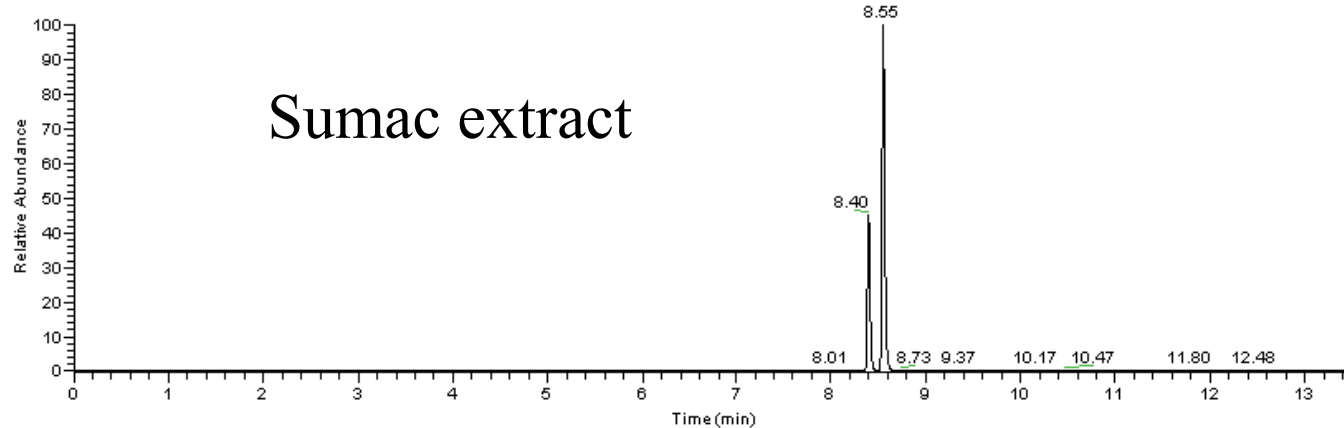


c:\calibur\11_07_15_02

7/15/2011 1:12:20 PM

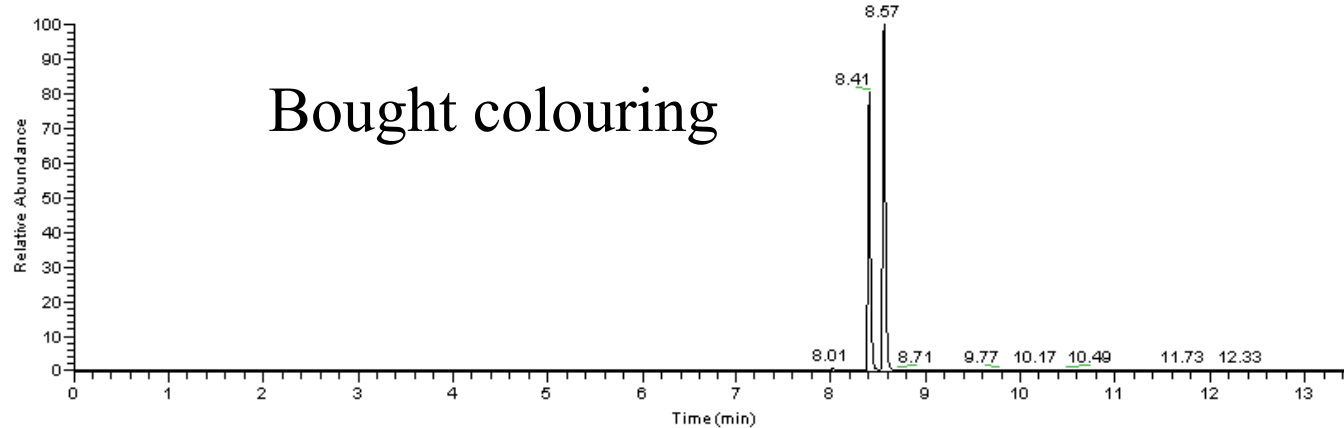
Haartoehrug Tangerine 2g/L

RT: 0.00 - 13.51



NL:
3.95E7
m/z=
321.18022-
321.18422
MS
11_07_15_
04

RT: 0.00 - 13.51



NL:
1.86E7
m/z=
321.18022-
321.18422
MS
11_07_15_
02



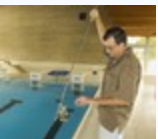
Elucidation of the structure: Where was the problem ?

Mea culpa

ESI(pos) ionization produces in 99 % of all cases an $[M+H]^+$ ion

ESI(pos) ionization produces in 1 % of all cases a free radical or the analyte is a quaternary amine

Therefore avoid the reflex to deduct an H^+ !!!
(Test: Neg. ionisation or Na^+ or D_2O infusion)





Elucidation of the structure: Where was the problem ?

The search is not always straightforward

Even a correct elemental composition [M]
may nowhere to be found

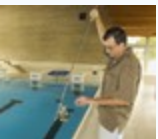
It might be recorded as

M+acetate

M+H+Br

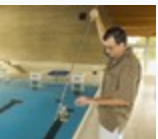
M+formate

etc.





Elucidation of the structure: Where was the problem ?



Public databanks can contain errors
(Scifinder an expensive tool contains the correct information)

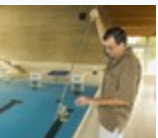
There are wrong structures
wrong elemental compositions
and structures do not always correspond
to the shown elemental compositions



How can we avoid this ?

There is a rule for an experienced traveller:
Ask three persons for the right way. Take the way which
was mentioned at least two times

For chemists involved in structural elucidation:
google the CAS Nr.
Believe only when you see the same structure 10 times





Why is finding an unknown that difficult ?



Finding an unknown additive in food is more difficult than finding an in-vitro metabolite of a known drug



A curry powder contains other natural red colours than a sumach



Food analysts have to show a high sample throughput



They have a heavy burden of administration, quality control and human resource management duties.

More and more they sit in the office.



The laboratory assistances struggle with the instruments



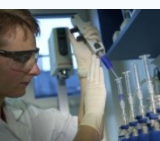
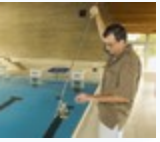
Why was Basic red 46 only discovered now?

Unauthorized use of Sudan red in food is known since 2003

Methods were developed and validated to detect sudan red

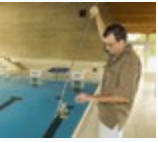
The methods were carefully optimized to produce best sensitivity, reproducibility, accuracy linearity etc.

This goal was achieved by introducing selective sample processing (e.g. gel permeation, or silica solid phase extraction)





Does too much regulation cause food unsafety ?



The validated methods worked well
for sudan red



The extensive clean up produced
nice results for sudan red but removed
Basic red 46 completely.



Is it worth quantifying a known compound with uttermost precision
while completely overlooking a closely related compound ?



Conclusion

We use harmonized analytical methods.

We have more and more sophisticated
method validation concepts.

We provide results with measurement
errors.

Is this the way to go ?

More brain in the lab;
less Regulations

