

13. BfR-Forum Verbraucherschutz

14. Juni 2012

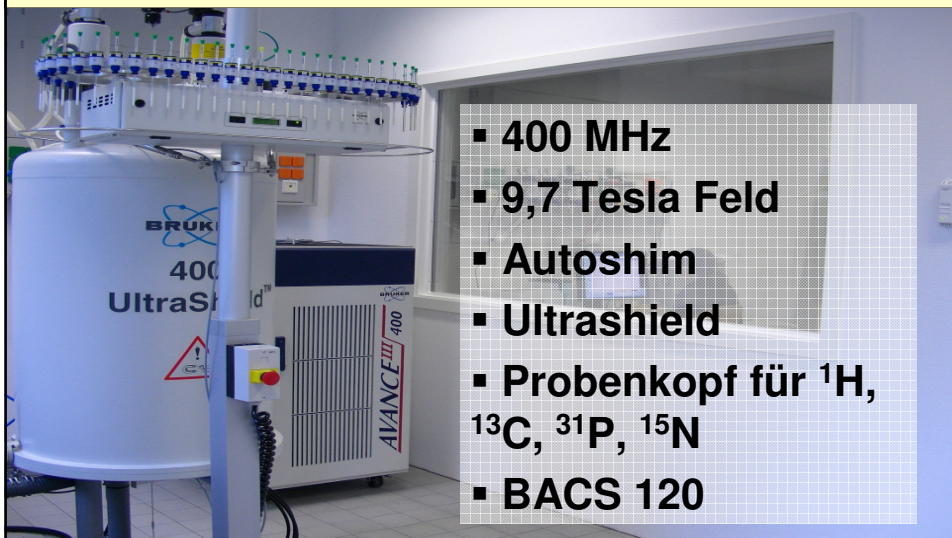
Kernspinresonanzspektroskopie (NMR)-Profiling in der Lebensmittelüberwachung

Dirk W. Lachenmeier

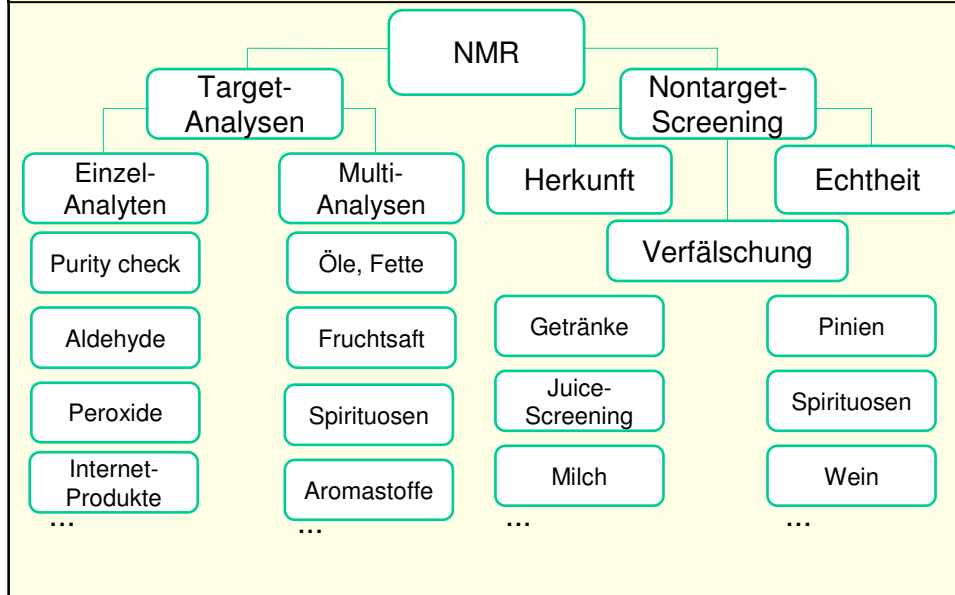


Baden-Württemberg
Chemische und Veterinäruntersuchungsämter

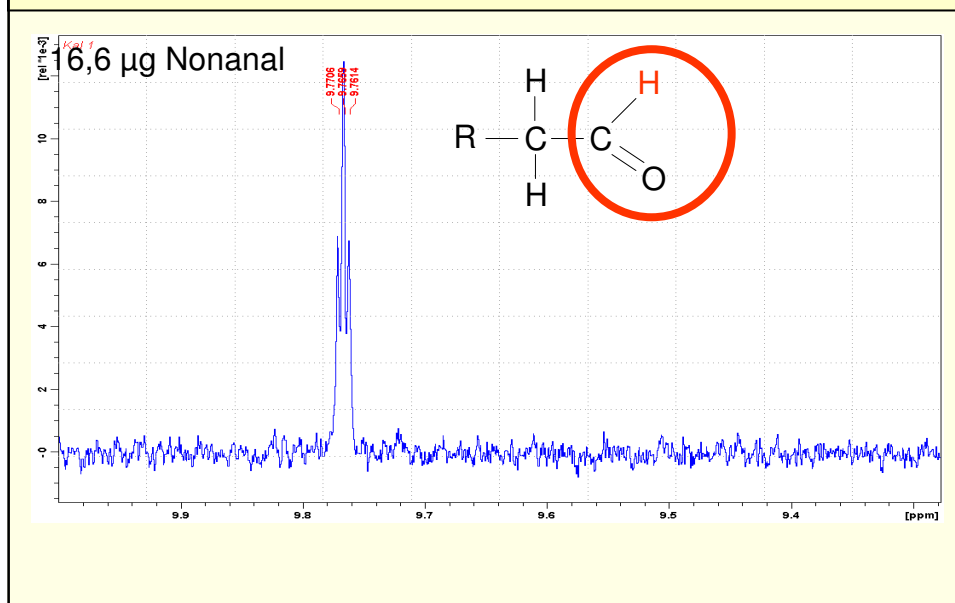
NMR am CVUA



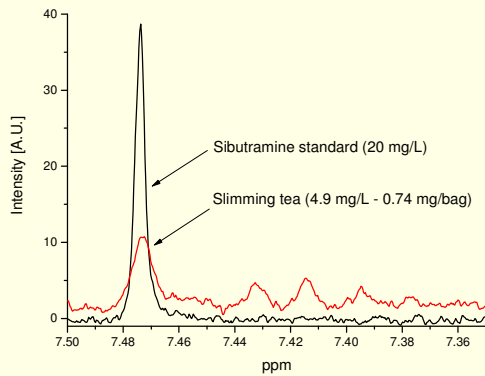
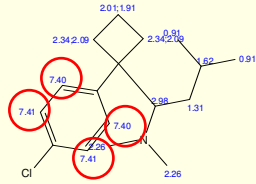
NMR in der Überwachung



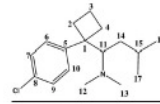
Ranzigkeit - Aldehyde



Schlankheitstee: Sibutramin



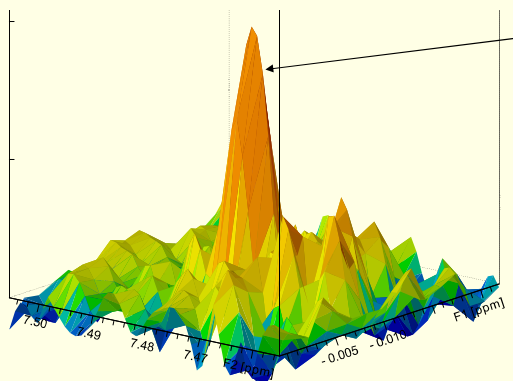
Sibutramine



Atom number	δ ^1H (ppm) ^a	Multiplicity ^b (J in Hz)
1		
2 or 4	2.60 (1H syn aromatic ring)	m
3	2.49 (1H anti aromatic ring)	m
	1.98 (1H)	m
	1.78 (1H)	m
4 or 2	2.37 (1H syn aromatic ring)	m
	2.41 (1H anti aromatic ring)	m
5	6.10	s
6	7.41	s
7	7.41	s
8		
11	3.67	t (6.3)
12 or 13	2.19	broad s
13 or 12	2.81	broad s
14	2.72	
15		
16 or 17		
17 or 16		



2D J-Resolved NMR: Vorteil bei Peakzuordnung und Integration



Sibutramin
(Singulett-Peak in
Zielregion ist
einfach zu
erkennen)

„Standardlose“ Quantifizierung

$$m_x = \frac{\text{Molecular weight}_x}{\text{Molecular weight}_{ref}} \cdot \frac{\text{Number of protons}_{ref}}{\text{Number of protons}_x} \cdot \frac{\text{Integral}_x}{\text{Integral}_{ref}} \cdot m_{ref}$$

Steroide in „Sports Nutrition“

	Product	Compound	Standardless NMR quantification	NMR quantification with calibration standard	Labelling
1.	Mesterolone	Mesterolone	16.4 mg/tablet	19.7 mg/tablet	25 mg/tablet
2.	Mesterolone		16.8 mg/tablet	17.3 mg/tablet	25 mg/tablet
3.	Oxymetholone IH 50	Oxymetholone	n.d.	n.d.	50 mg/tablet
4.	Testogain	Dehydro-epiandrosterone (DHEA)	18.8 mg/tablet	21.6 mg/tablet	25 mg/tablet
5.	Biovea DHEA		58.4 mg/tablet	68.6 mg/tablet	100 mg/capsule

NMR Bieranalytik

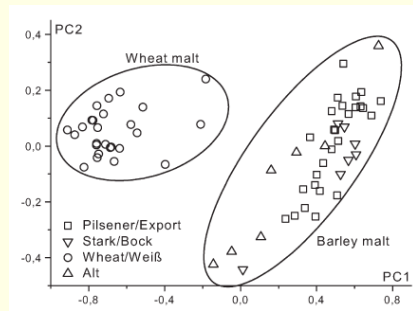


Fig. 4 Scatter plot of PCA scores for the aromatic region (7.6–6.7 ppm)

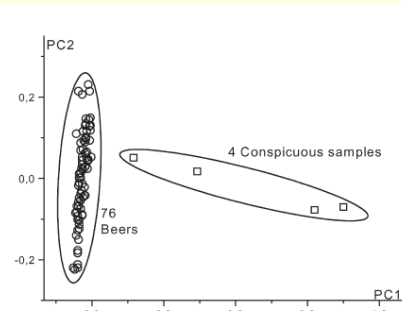
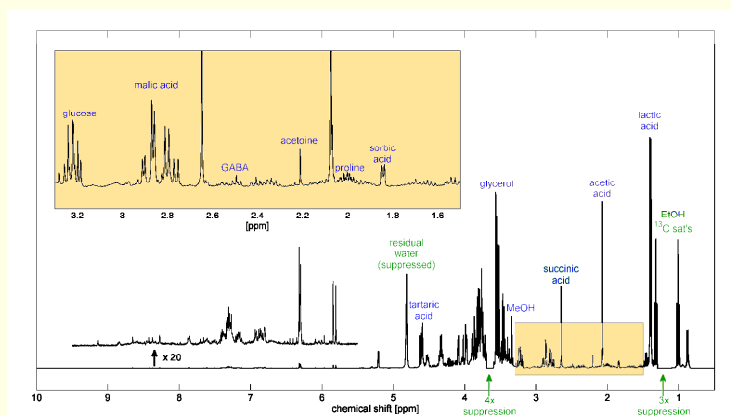









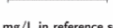
Fig. 3 Scatter plot of the PCA scores (9–0.4 ppm)

1-H-NMR spectrum of wine

Proton spectrum of wine with 8-band suppression. Details in magnification. NMR spectrum is quantitative in a dynamic range of 5-6 magnitudes. Sample acquisition and processing is automatic with 3 sample per hour.



WineScreener

Compound	Value	Unit	Official Reference		Wine-Profiling™
			Flag	max. Value	Authentic NMR Database
acetic acid	525	mg/L	○	-	165  1007
benzoic acid	<5	mg/L	●	0 mg/L	<5 mg/L in reference set
citric acid	299	mg/L	●	1000 mg/L	<200  471
ethanol	91.7	g/L	○	-	56.2  108.0
fumaric acid	<5	mg/L	○	-	<5  12
glycerol	7.1	g/L	○	-	4.5  11.1
malic acid	5.5	g/L	○	-	<0.2  8.2
methanol	53	mg/L	●	250 mg/L	6  146
shikimic acid	88	mg/L	○	-	<20  100
sorbic acid	<5	mg/L	●	200 mg/L	<5 mg/L in reference set

Zusammenfassung

- NMR sehr vorteilhaft für amtliche Überwachung
- Technik hat Routinefähigkeit erreicht
- Messungen sind sehr schnell (ca. 20 min) und erfordern nur minimale Probenvorbereitung
- Potential insbesondere für Screening großer Probenzahlen (Fruchtsaft, Wein, Öl) unter Wegfall von Referenzanalytik
- Große Vorteile bei Targetanalysen im „Lifestyle-Bereich“ (Nahrungsergänzungsmittel, Zoll- und Internetproben)

Danke für die Aufmerksamkeit

Kontakt: Lachenmeier@web.de



Chemisches und Veterinär-
Untersuchungsamt Karlsruhe