

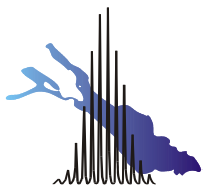
ANALYTISCHE CHEMIE I

Trennmethoden

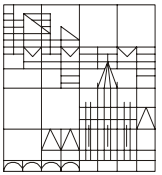
5. Prozess-Analytik

GC-MS , LC-MS

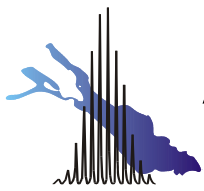
WS 2007/2008



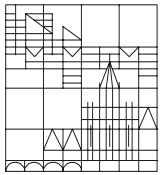
Prozeßanalytik



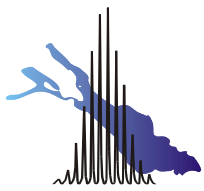
- Analyse von Prozessen durch Messungen des zeitlichen Verlaufes physikalischer Probenveränderungen und von chemischen Reaktionen
- Anwendungsgebiete sind z.B. die kontinuierliche Kontrolle strömender Flüssigkeiten und Gase oder der Ablauf chemischer Reaktionen
- Prozeßanalytische Verfahren sind in der Regel quantitative Analysen
- Die Prozeßanalytik ist hauptsächlich in der Industrie von Bedeutung



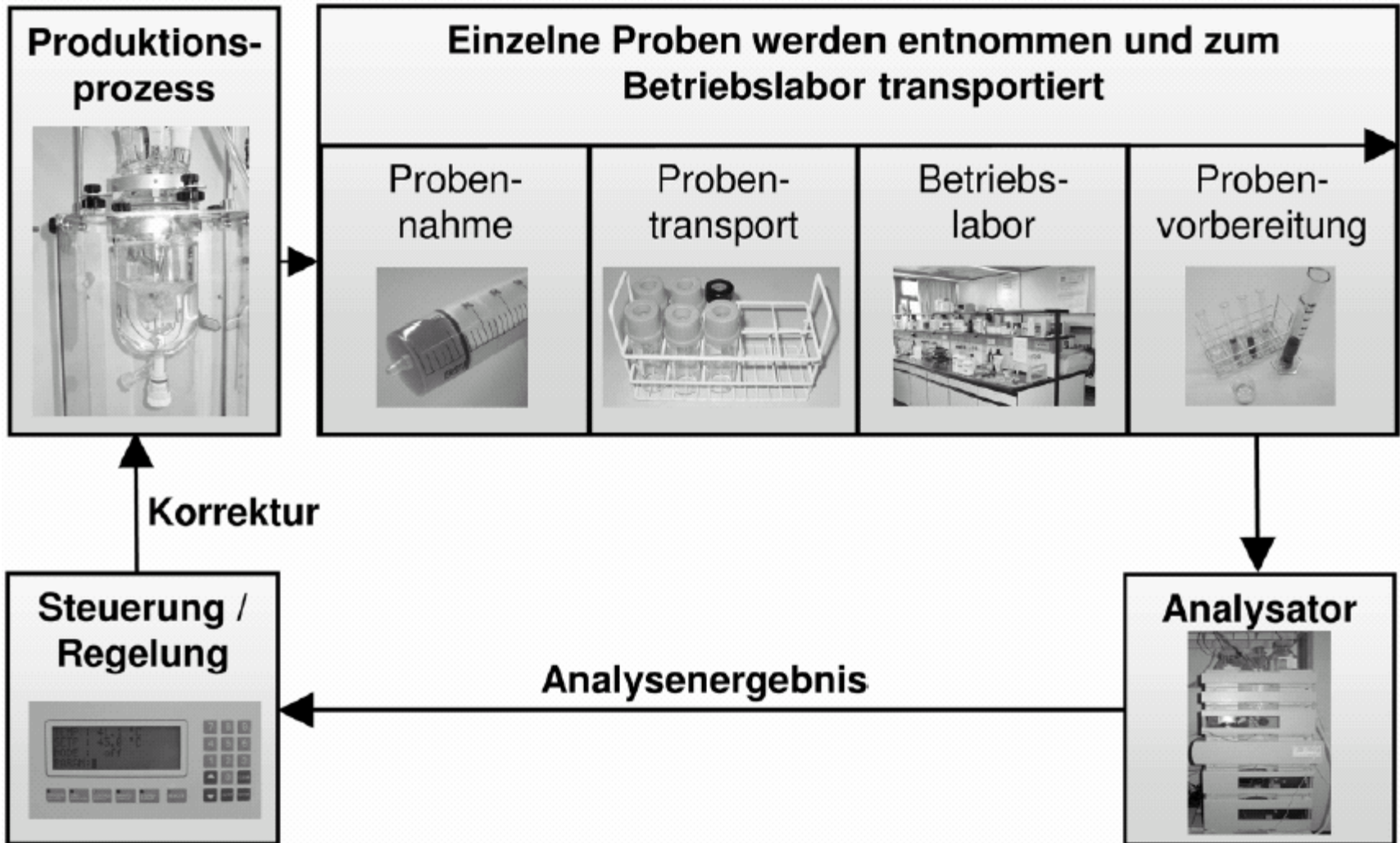
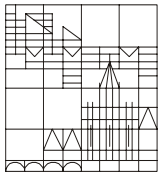
Anforderungen an die Prozeßanalytik

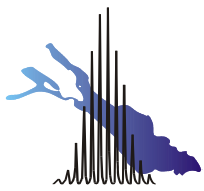


- Schnelle Analysen für Hochgeschwindigkeitsprozesse
- Einfache Funktionsweise eines Prozeßanalytors
- Hohe Reproduzierbarkeit und Zuverlässigkeit
- Widerstandsfähigkeit des Analysators muß gewährleistet sein
- Einfache Bedienbarkeit der Analytoren
- Besondere Anforderungen an die Probennahme und Probenvorbereitung

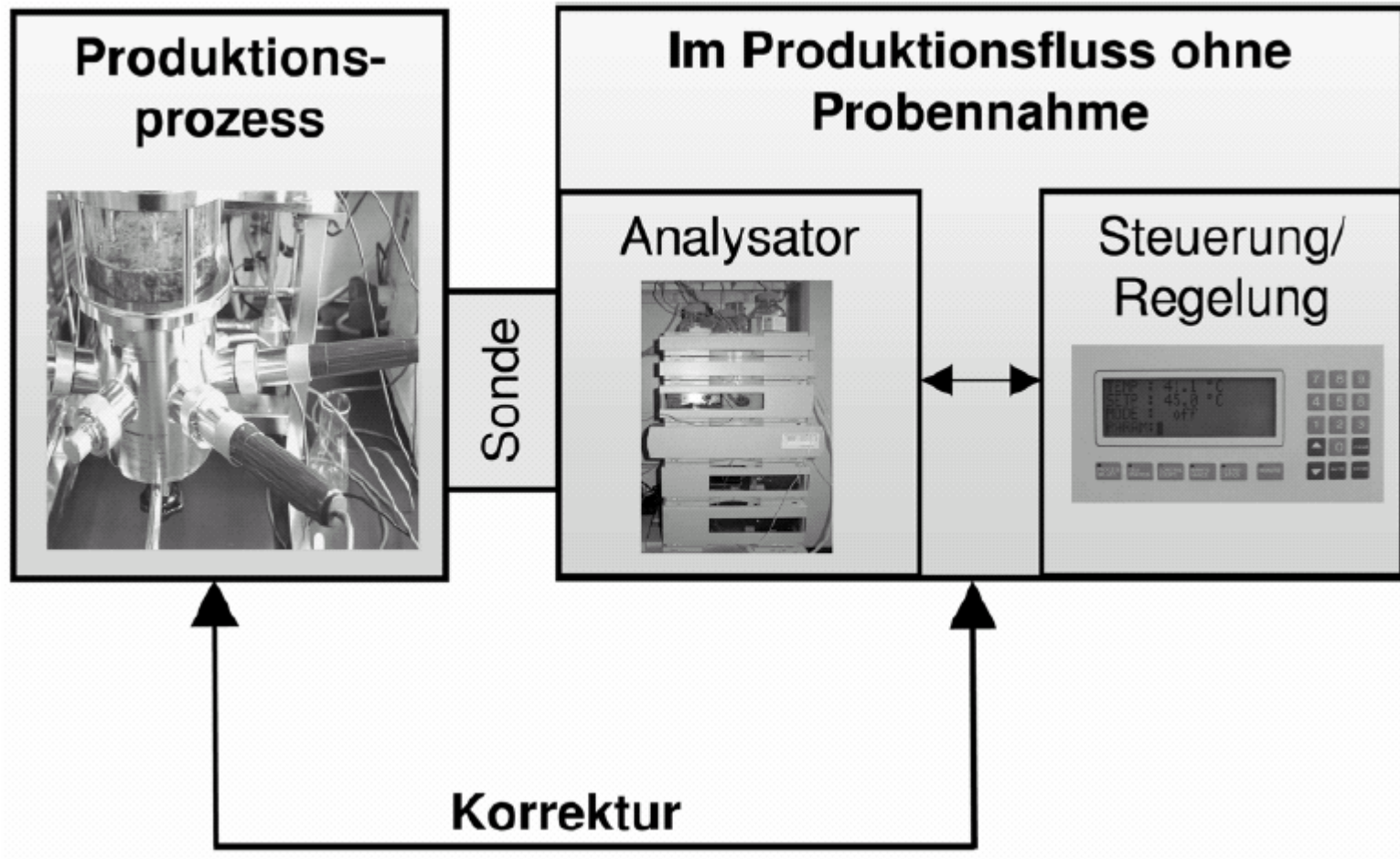
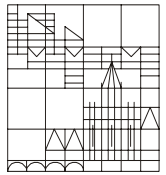


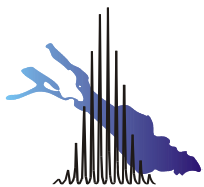
Prozeßanalytik - Offline



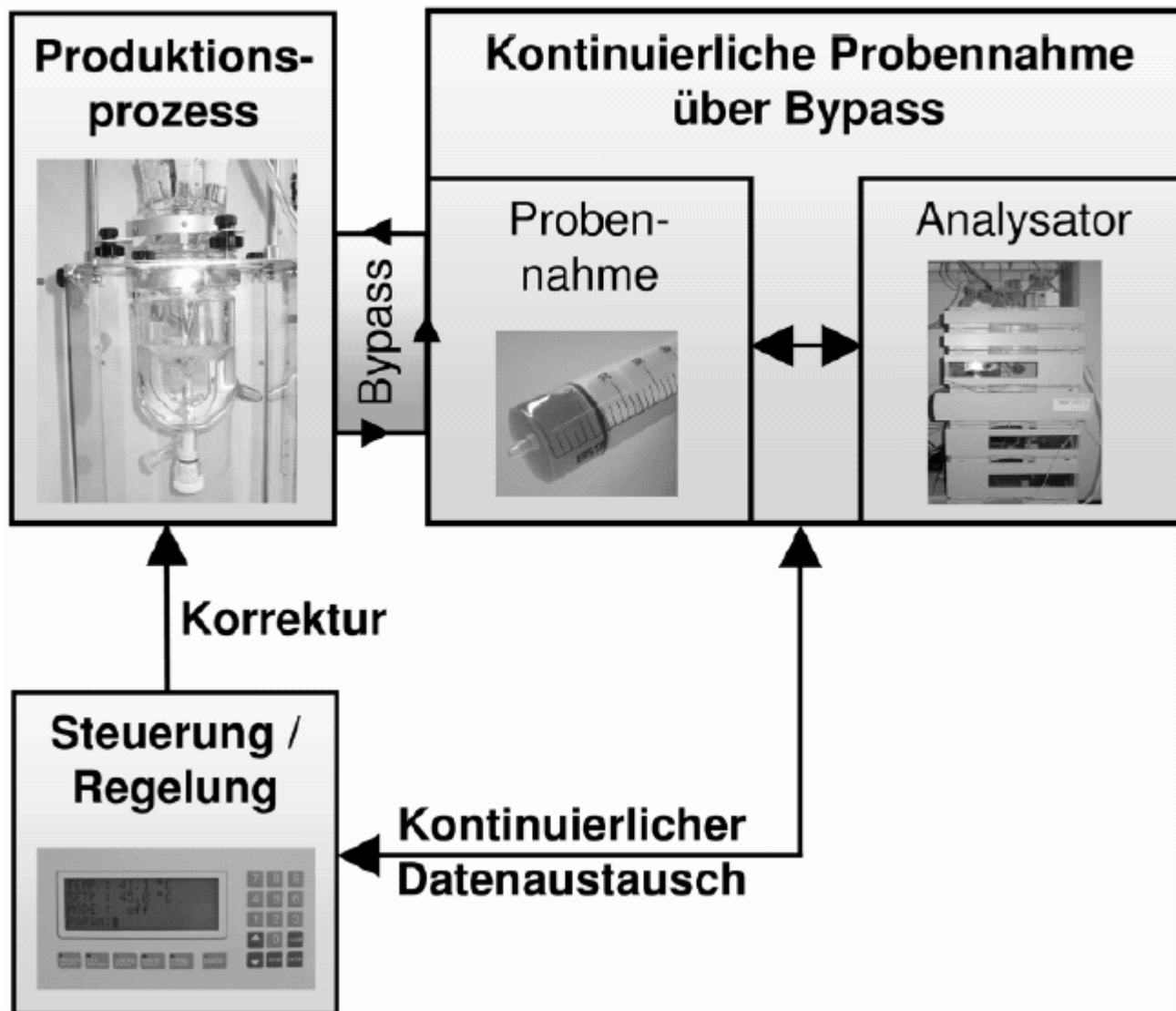
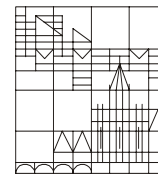


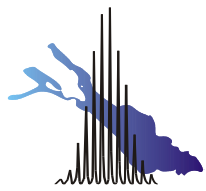
Prozeßanalytik - Inline



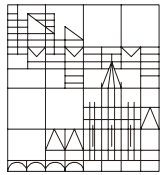


Prozeßanalytik - Online

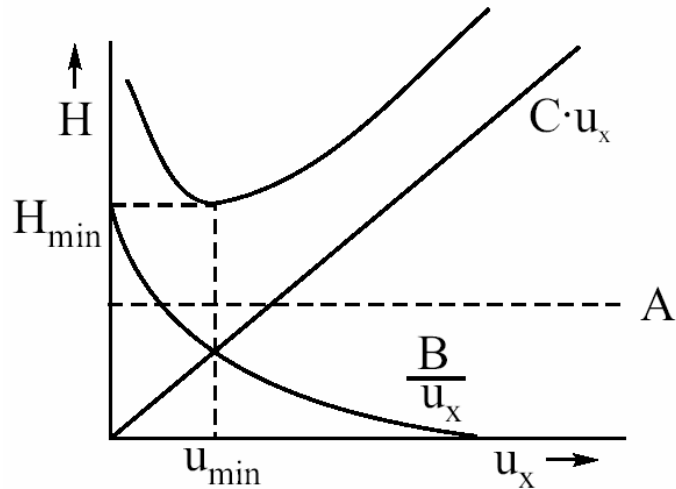




Schnell und effizient: HPLC, UFLC, RRLC...



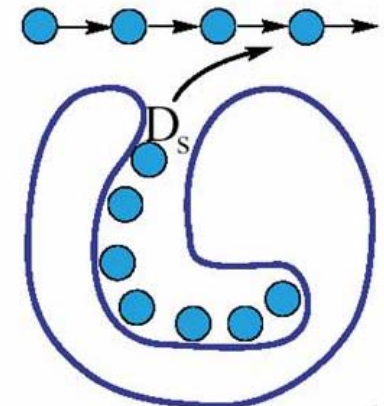
van Deemter-Gleichung

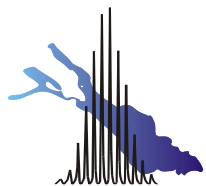


$$H = A + \frac{B}{u_x} + C \cdot u_x$$

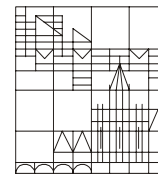
- A Die Streudiffusion (Eddy Diffusion)
- B / u_x Longitudinale (axiale) Diffusion
- $C \cdot u_x$ **Massentransfer**

	Abbrev.	Column		
		Particle Size	I.D.	Length
Rapid Resclution LC	RRLC	1.8 μm^*	1 – 4.6 mm	15 – 150 mm
Ultra-fast LC**	UFLC	1.8 μm^*	1 – 4.6 mm	15 – 50 mm
Conventional HPLC	HPLC	2 – 10 μm	2 – 4.6 mm	15 – 300 mm
Standard Bore LC		1.8* – 10 μm	3 – 4.6 mm	15 – 300 mm
Narrow Bore LC		1.8* – 5 μm	1 – 2 mm	15 – 300 mm
Capillary LC, Micro LC	CapLC, μLC	1.8* – 5 μm	0.2 – 1 mm	15 – 300 mm

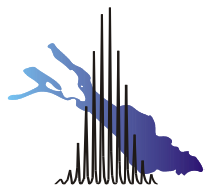




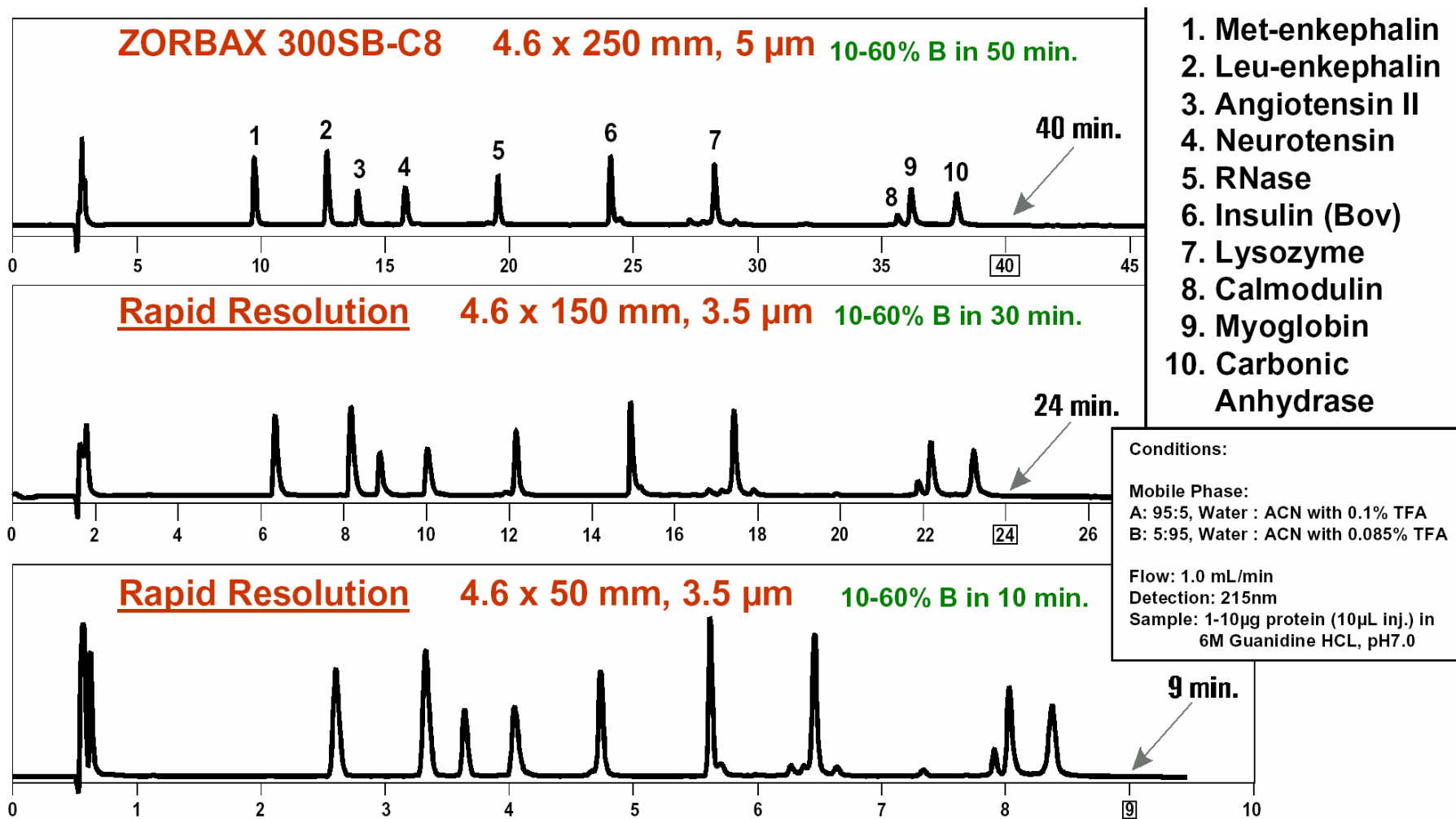
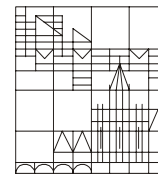
Schnell und effizient: HPLC, UFLC, RRLC...

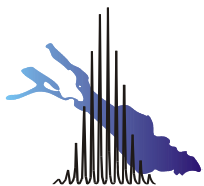


	5 μ m	3.5 μ m	5 μ m	3.5 μ m
Dimension	250 x 4.6 mm	150 x 4.6 mm	150 x 4.6 mm	75 x 4.6 mm
Analysis Time (min)	30 min.	18 min.	18 min.	9 min.
		40% reduction		50% reduction
Solvent Waste, mL	30 mL	18 mL	18 mL	9 mL
		40% reduction		50% reduction
N	20,000	20,000	12,000	10,000
Resolution $\propto N^{1/2}$	Unchanged		9% Difference	

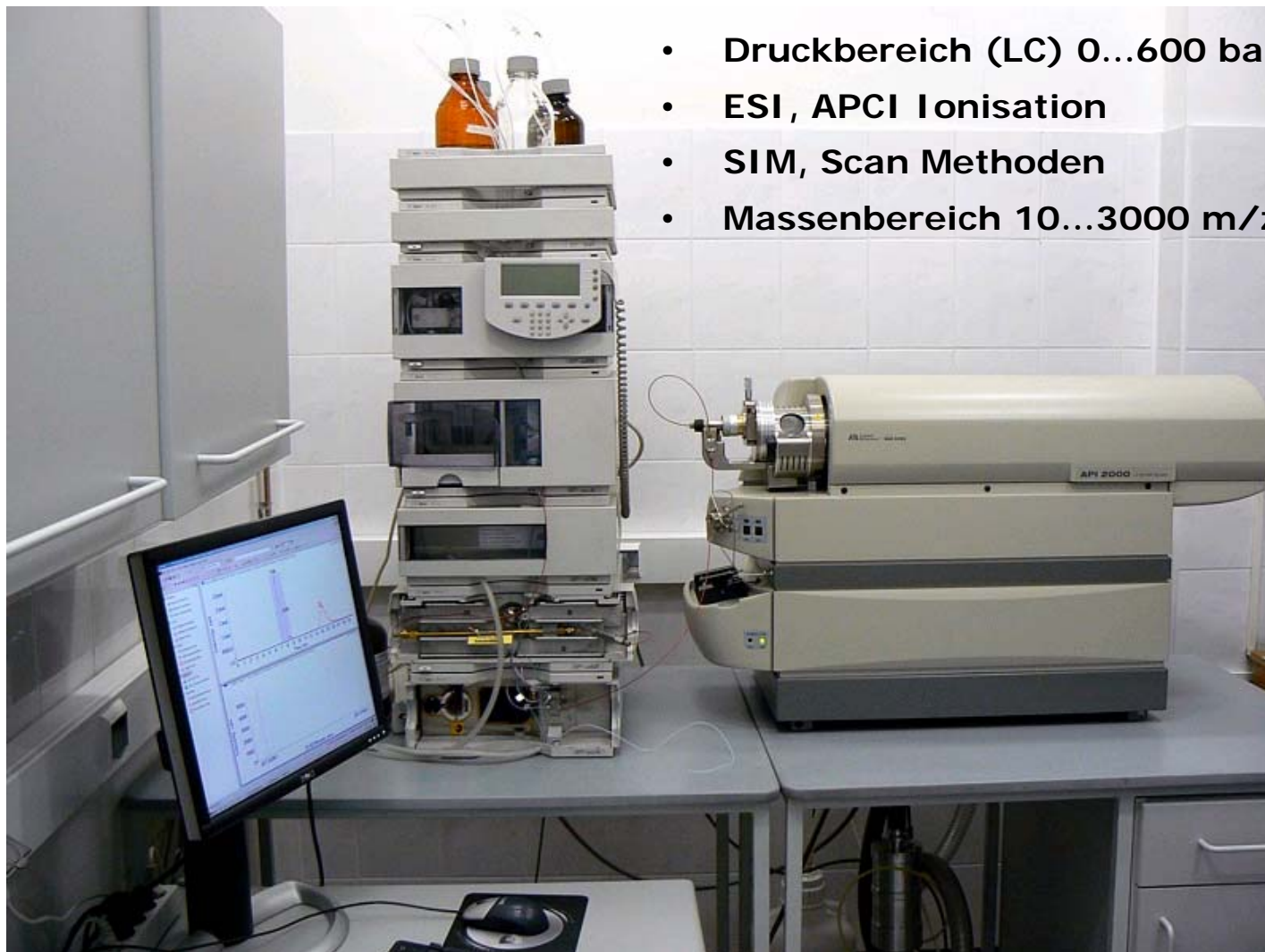
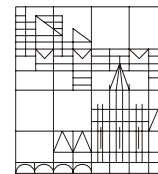


Schnell und effizient: HPLC, UFLC, RRLC...

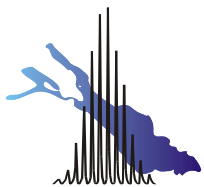




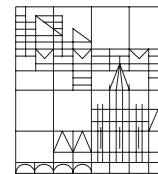
HPLC-MS-Kopplung (LC-MS)



- Druckbereich (LC) 0...600 bar
- ESI, APCI Ionisation
- SIM, Scan Methoden
- Massenbereich 10...3000 m/z



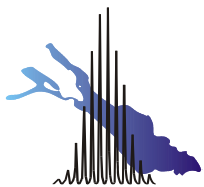
Schnell und effizient: GC-MS, Prozess-Gas-Chromatograph



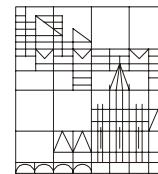
- **Temperaturbereich (GC) 50 ... 350 °C**
- **Temperaturgradient 1...20 °C/min**
- **EI, CI Ionisation**
- **SIM, Scan Methoden**
- **Massenbereich 10...1050 m/z**



- **Temperaturbereich 60 ... 165°C isotherm**
- **Multidimensionale Chromatographie mit Rückspülung und Schnitt**
- **WL Detektoren**



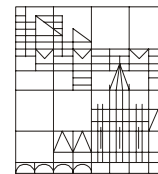
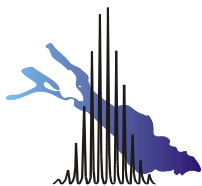
Prozess-Gas-Chromatograph MicroSAM (Siemens)



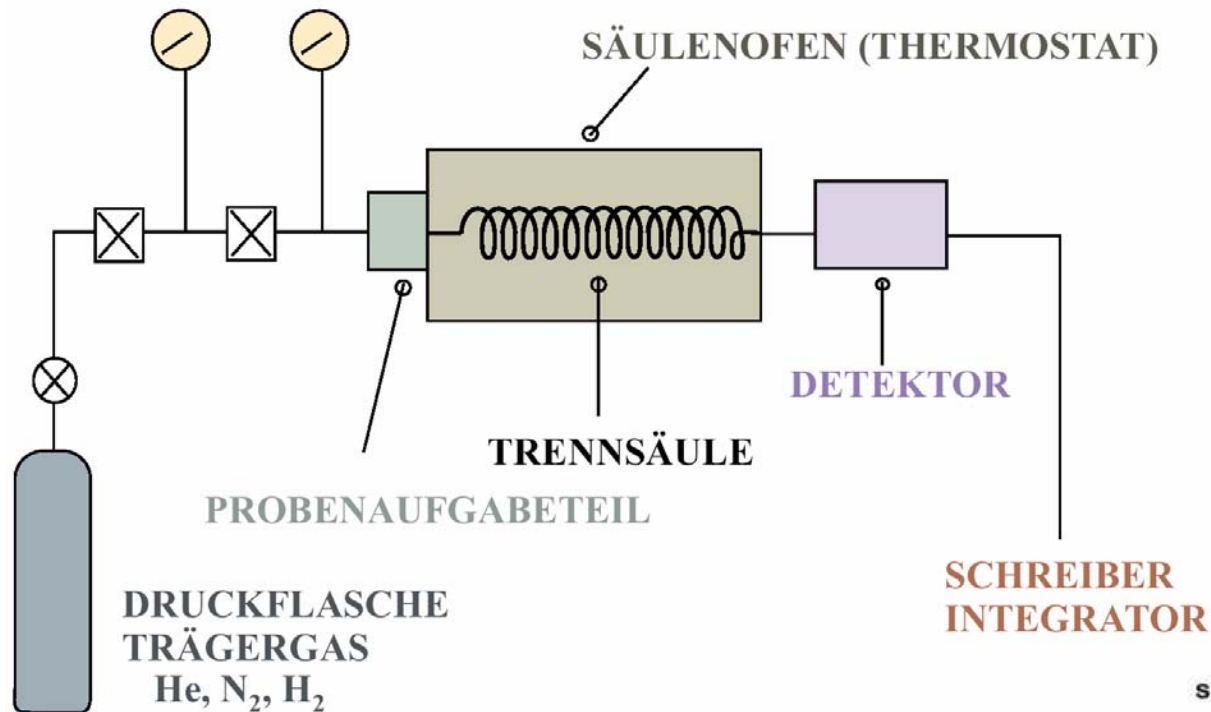
Carrier gas



Dosierung in Gas-Chromatographie



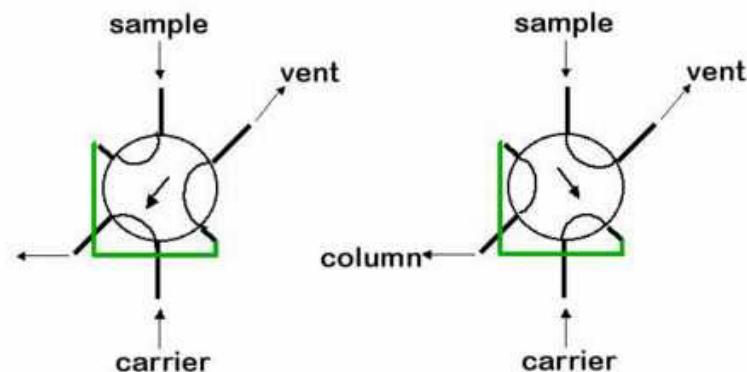
MANOMETER

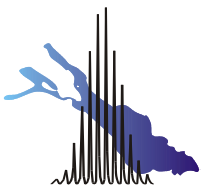


Gasschleife

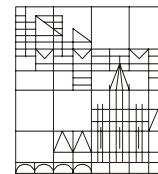
PROBENAUFGABETEIL

- 1. gasförmig → Gasschleife (0,5 - 5 ml)
- 2. flüssig → Injektionspritze (1 - 10 µl)
- 3. flüssig/fest → Einspritzblock (beheizbar)

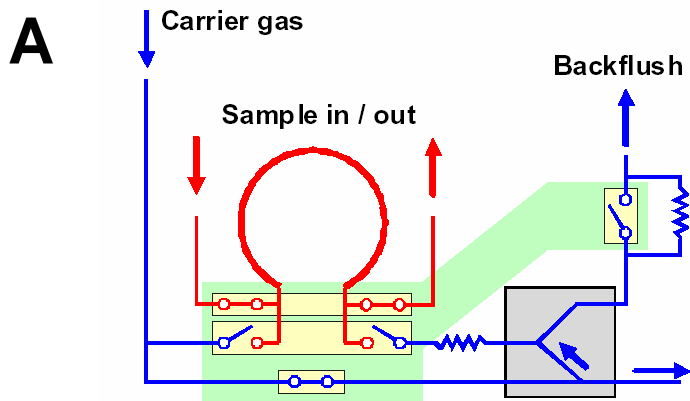




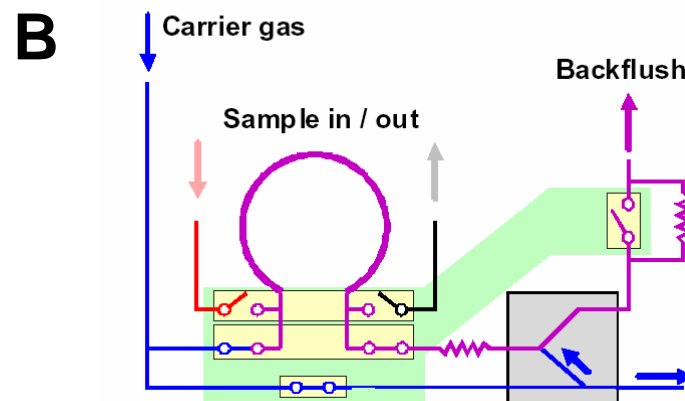
Live-Dosierung in Prozess-Gas-Chromatographie



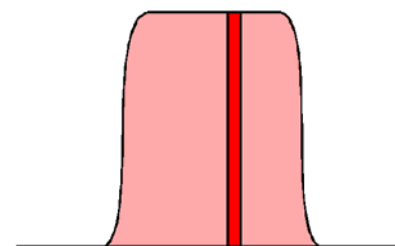
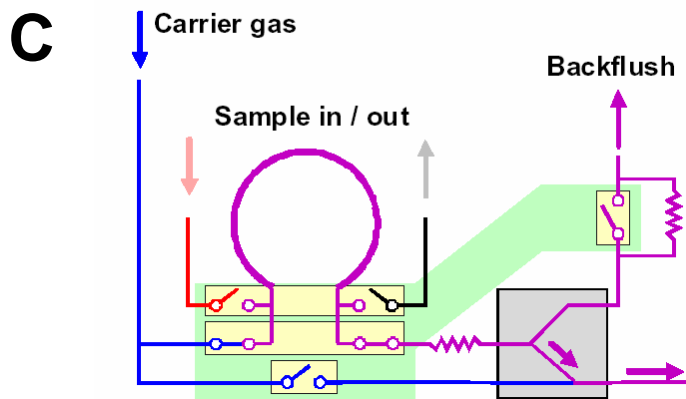
Dosierschleife füllen



Dosierschleife leeren

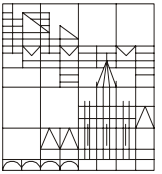
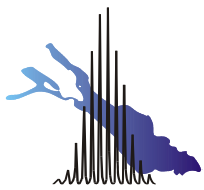


Dosieren

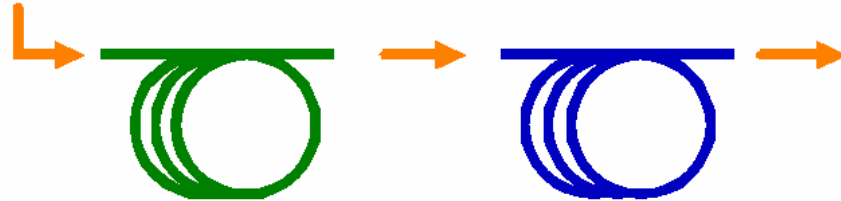


Durch Verändern der Pulsdauer für das Dosierventil lässt sich die Dosiermenge einstellen.

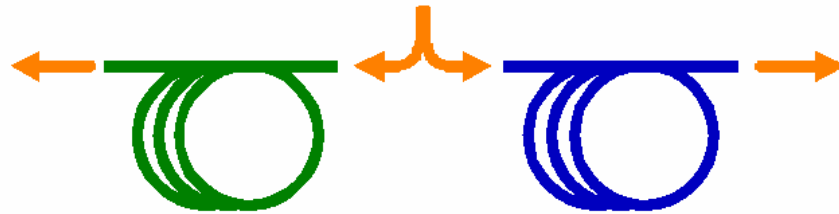
Multidimensionale Prozess-Gas-Chromatographie



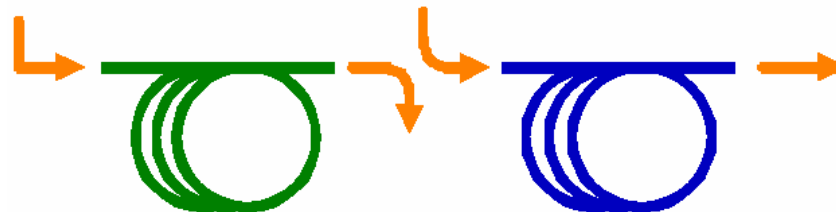
Geradeaus

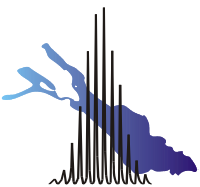


Rückspülen

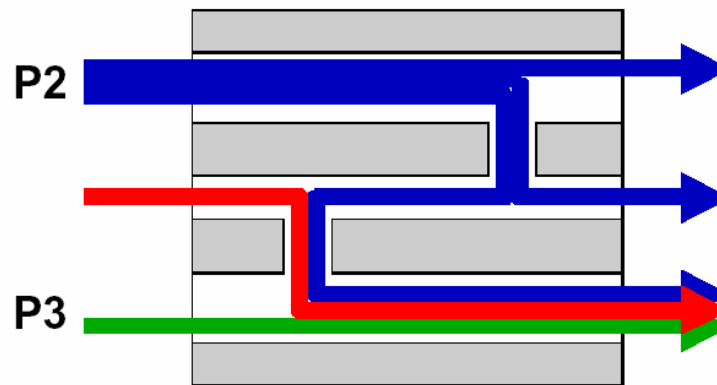
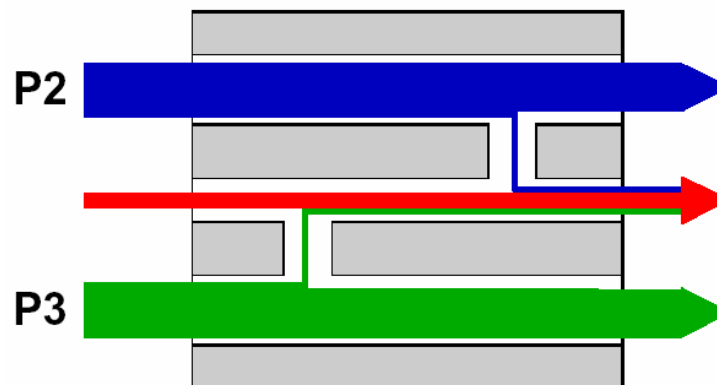
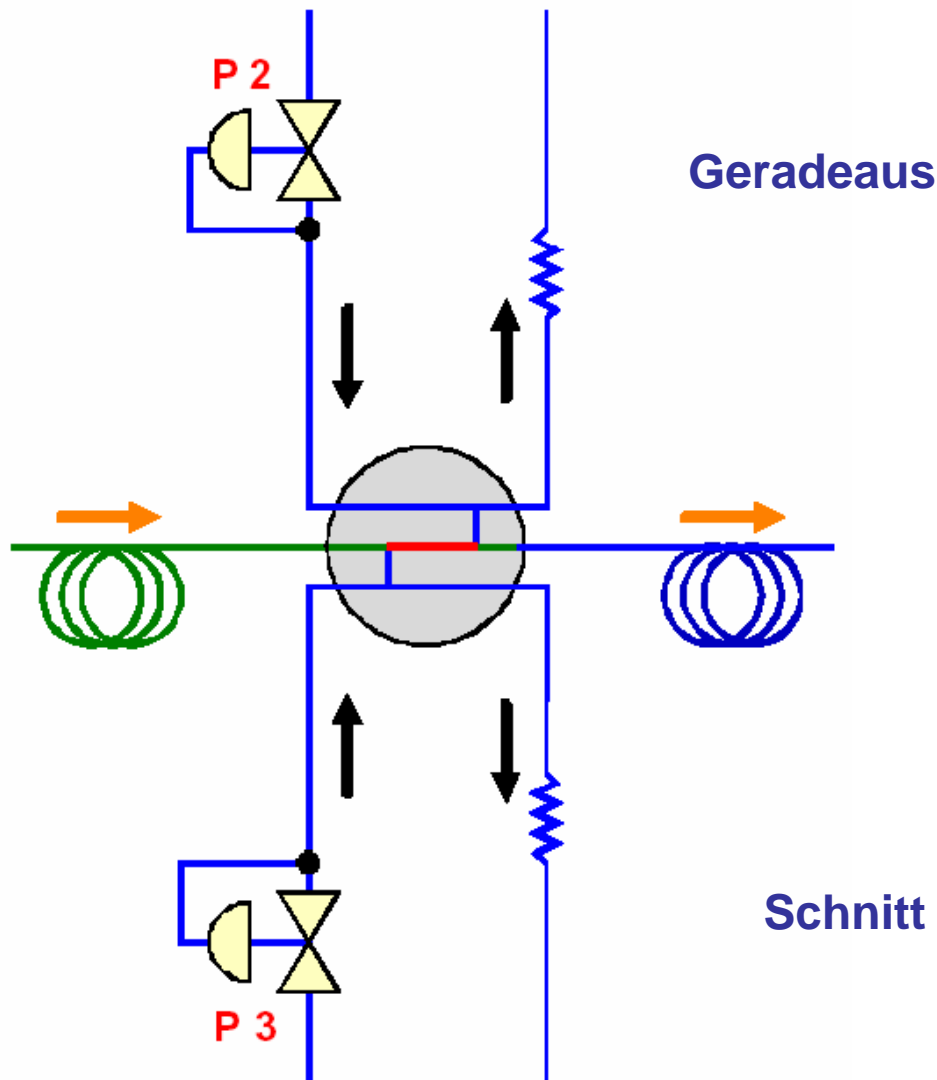
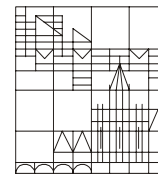


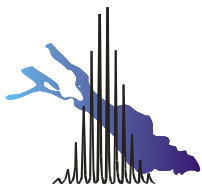
Schnitt



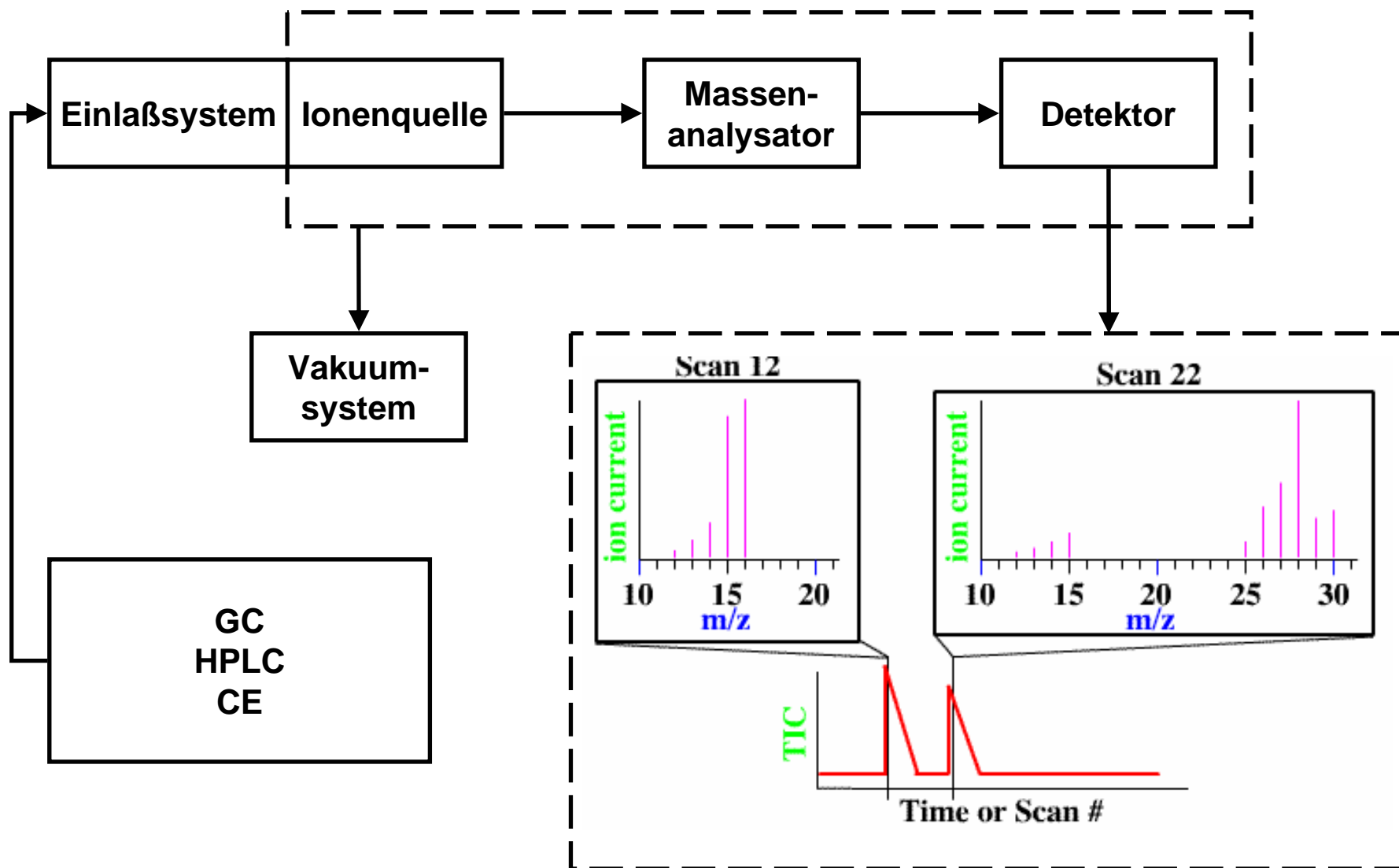
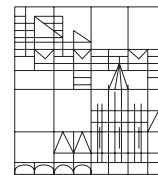


Live-Trennsäulenschaltung in Prozess-Gas-Chromatographie

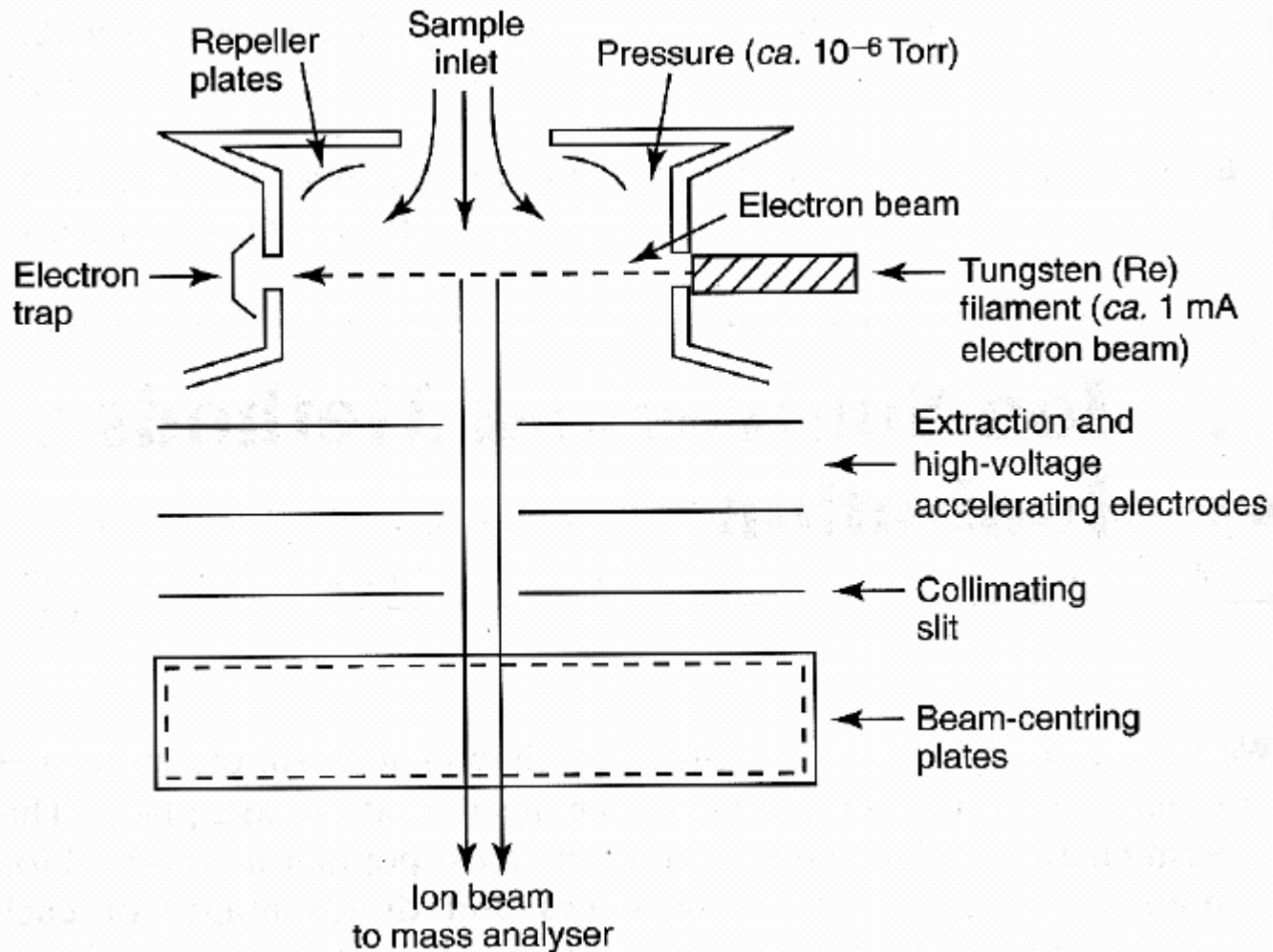




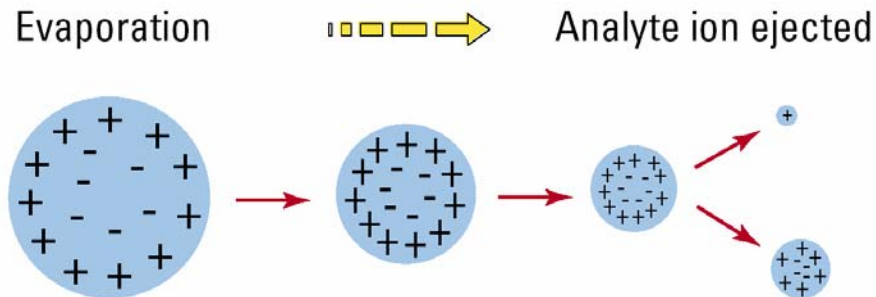
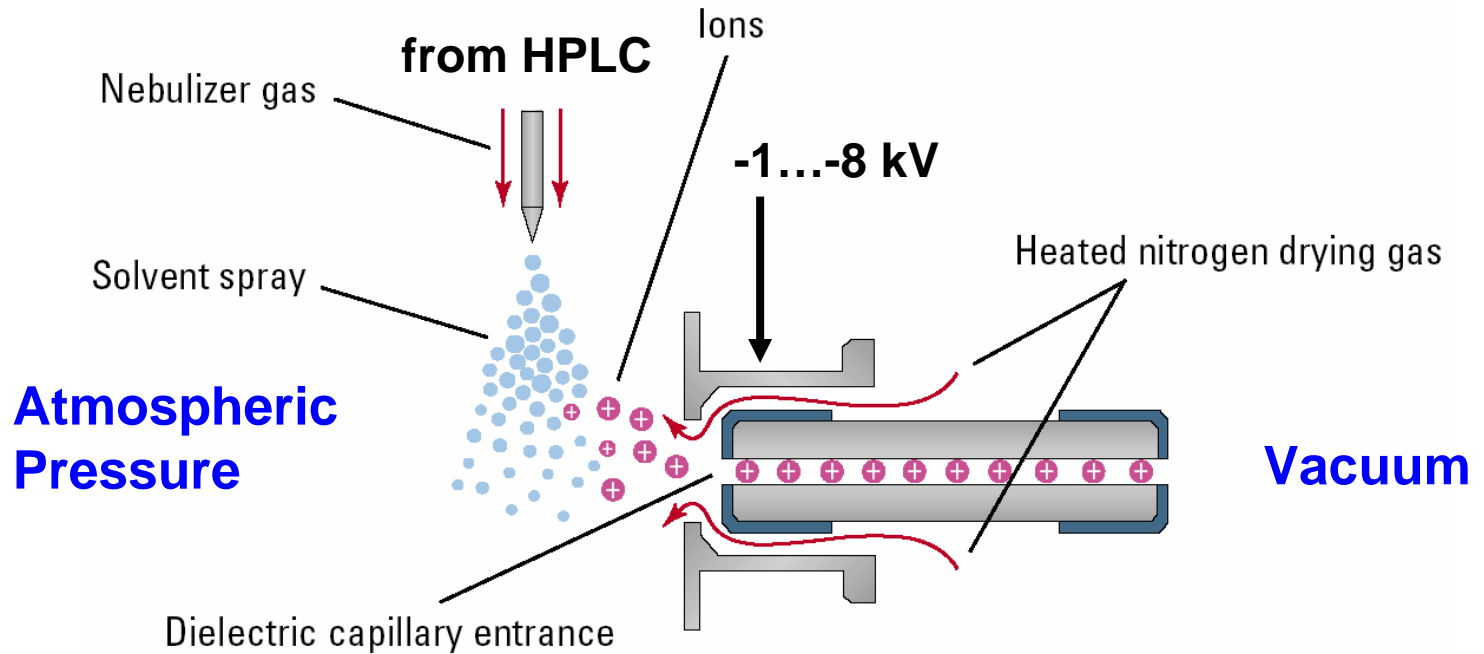
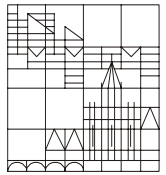
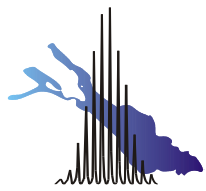
Massenspektrometrie

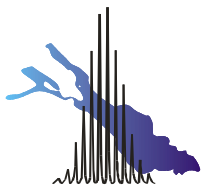


EI (Electron impact Ionization)

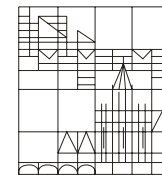


API-ESI (Atmospheric Pressure Interface-Electrospray Ionization)

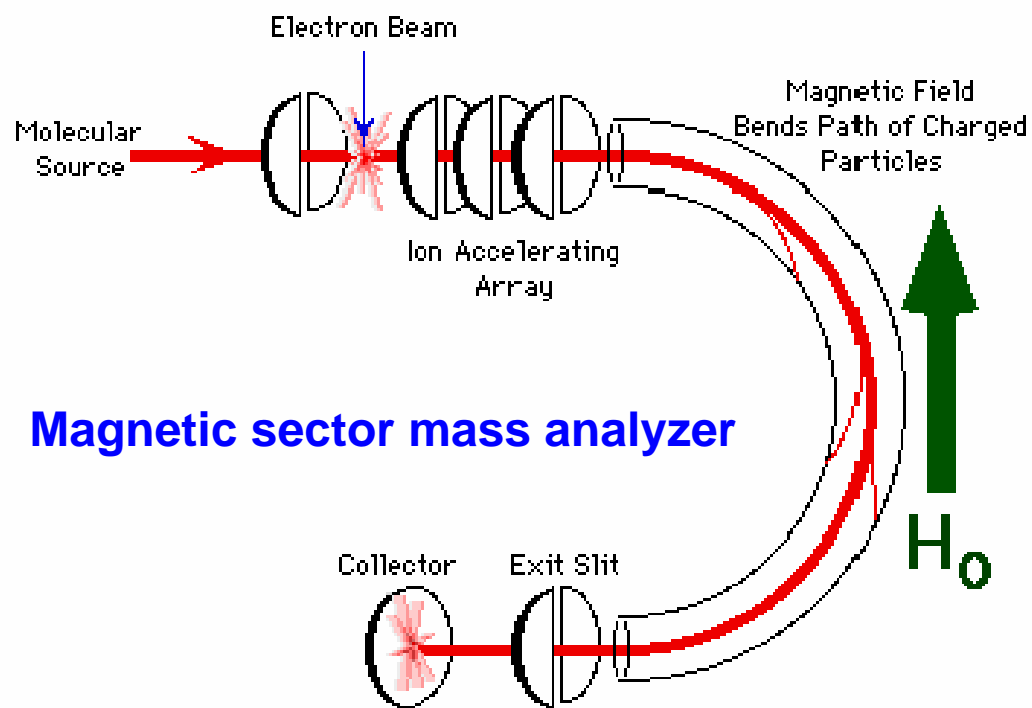
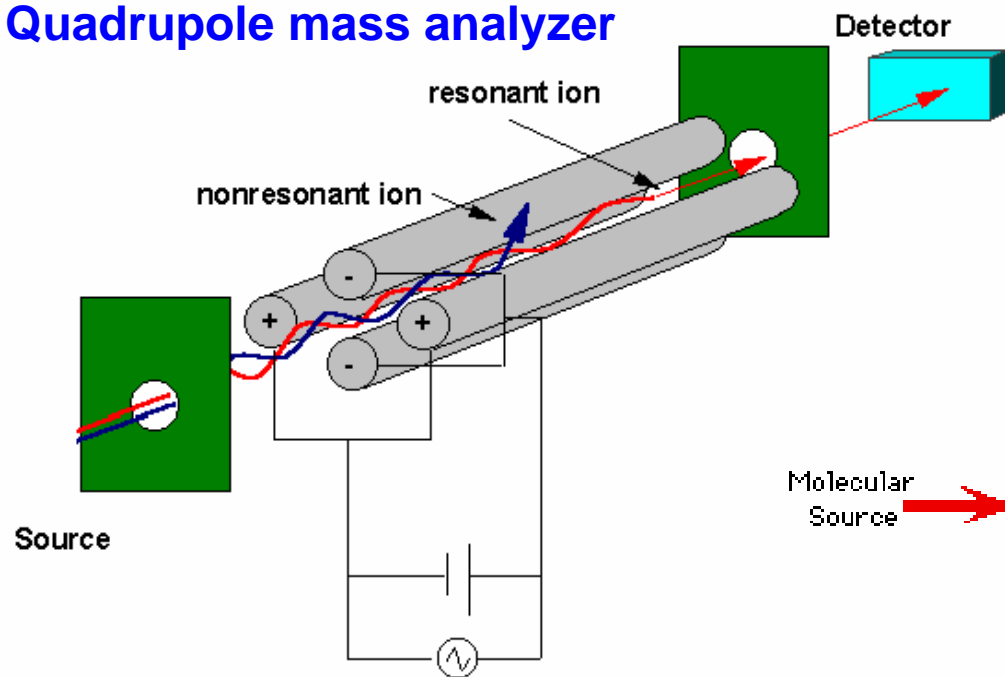




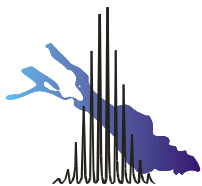
Massenanalysator



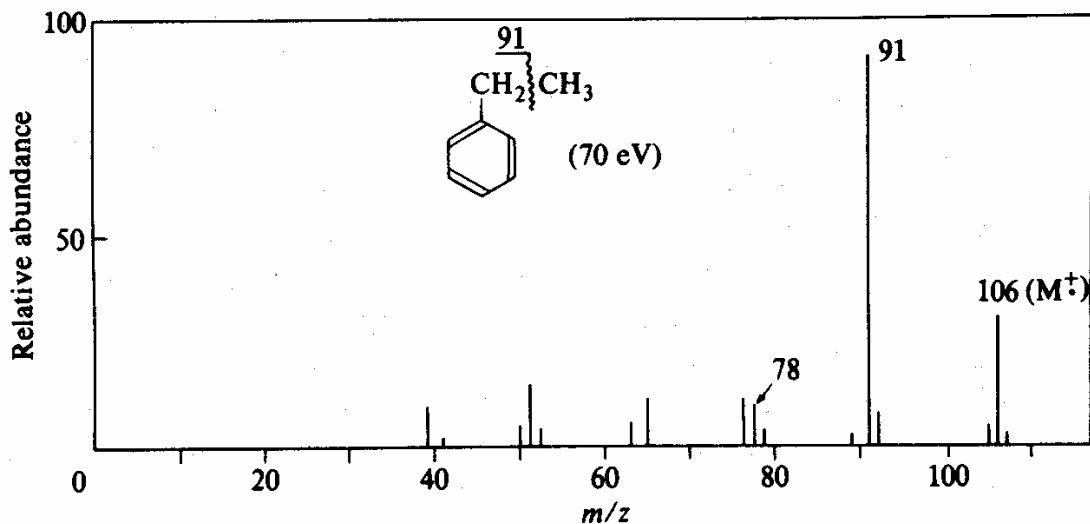
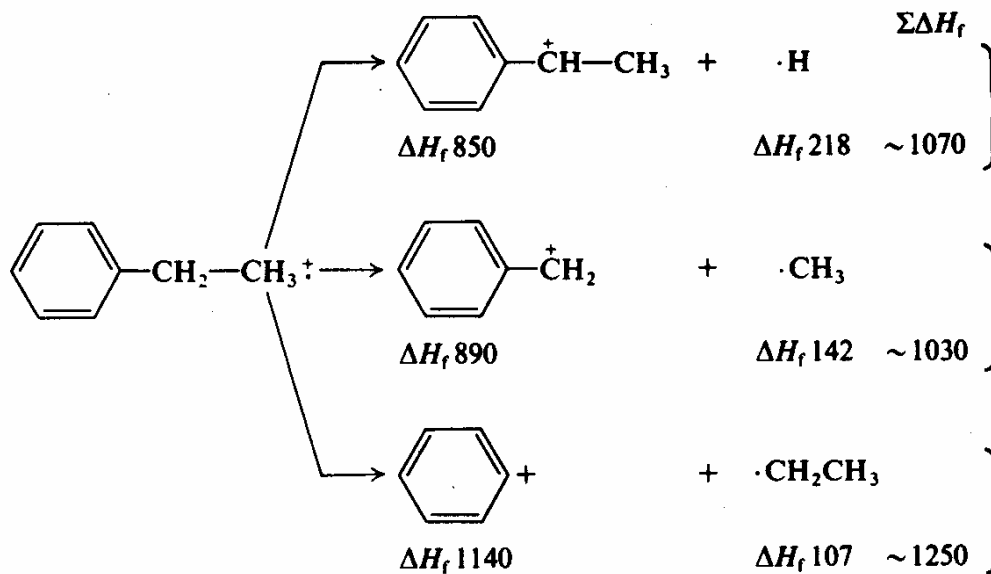
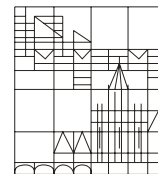
Quadrupole mass analyzer

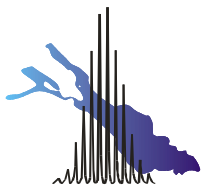


Magnetic sector mass analyzer

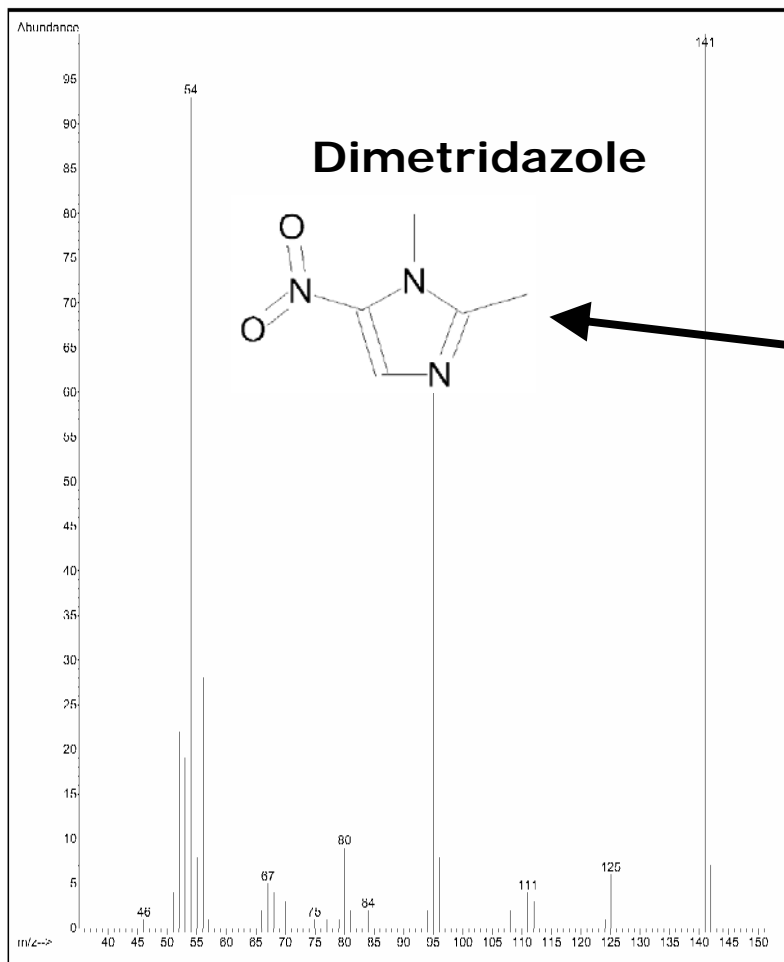
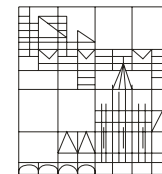


EI-MS

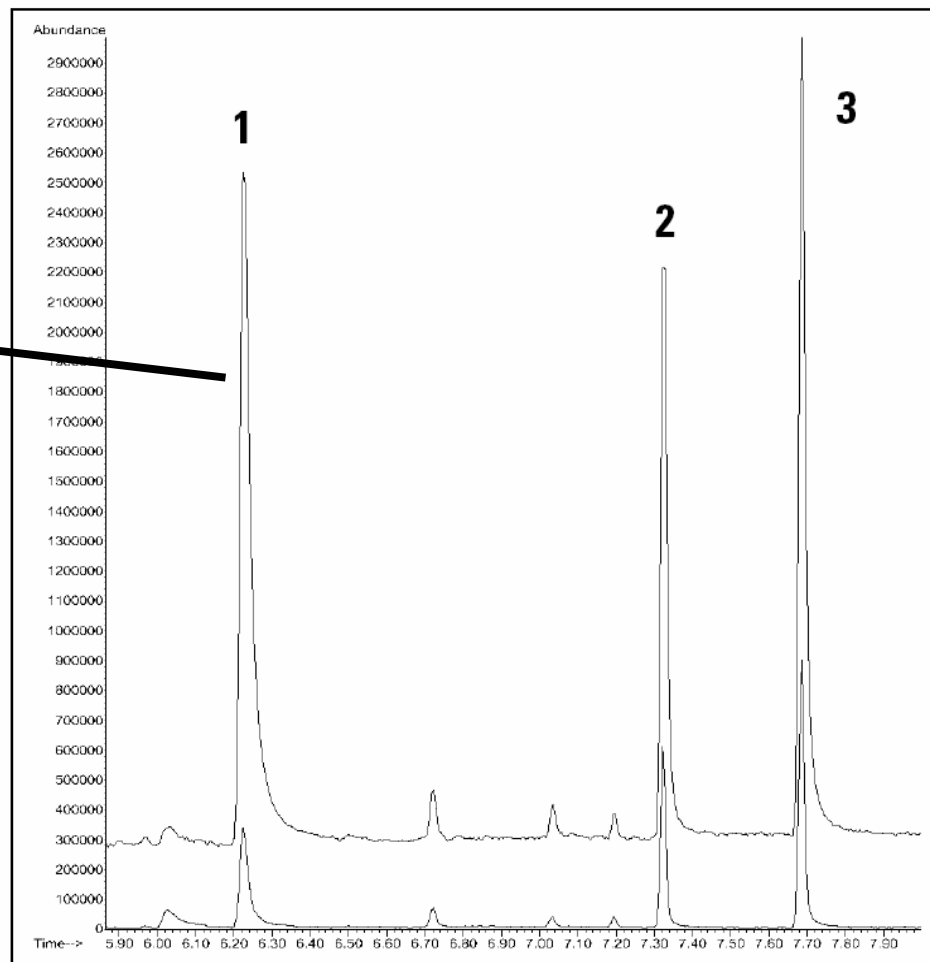




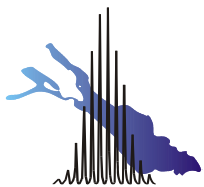
Beispiel GC/MS-Kopplung Nitroimidazoles



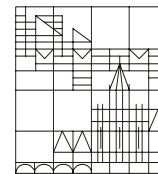
El-Spectrum, Dimetridazole, m/z 141; M^+



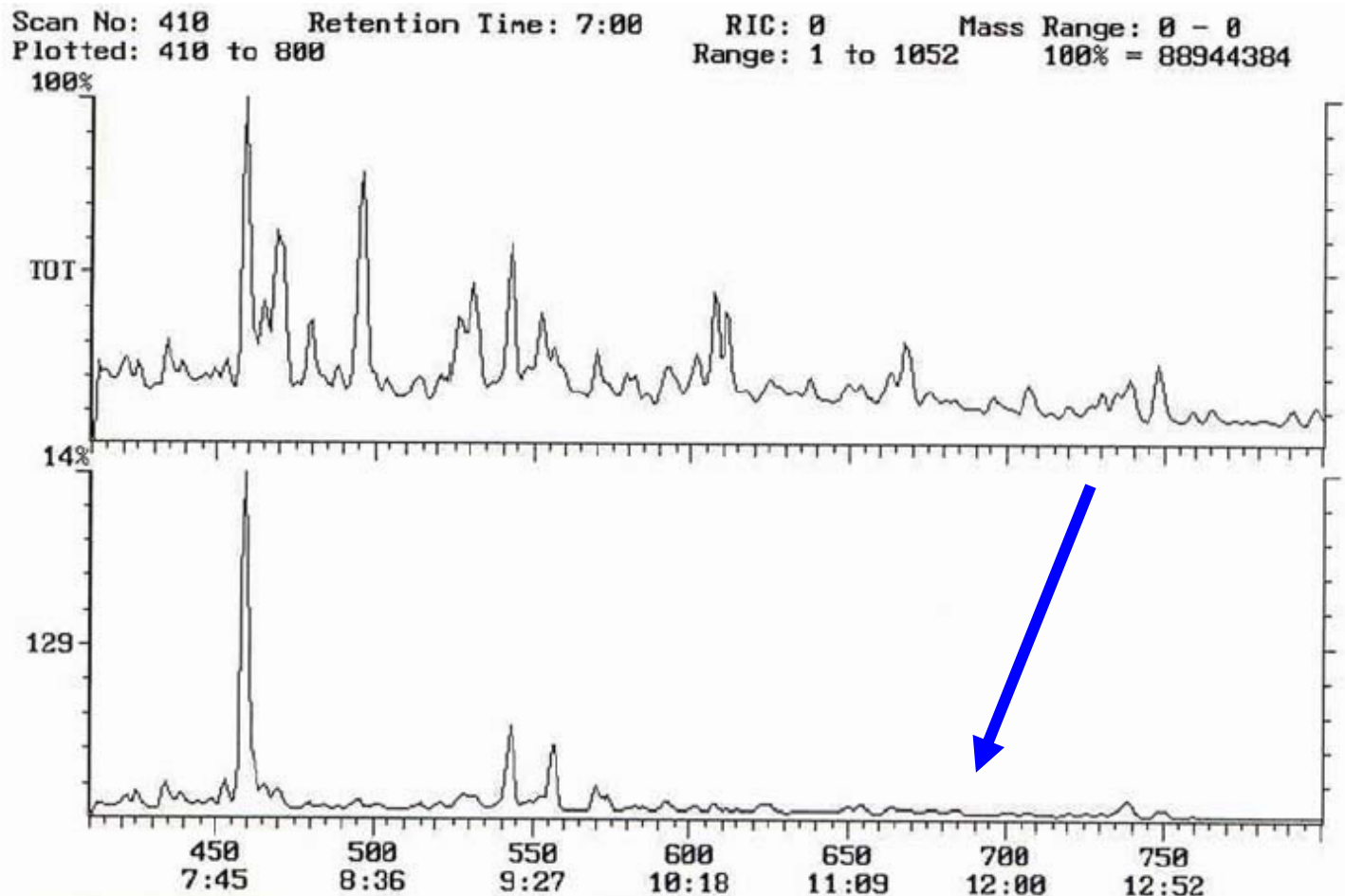
TIC of Dimetridazole(1), Ronidazole(2), Metronidazole(3)
Buffer Gas: Methane, bottom; Ammonia, top

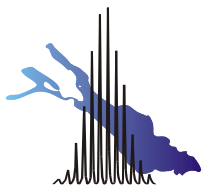


GC-MS Urinanalyse von einem Pferd

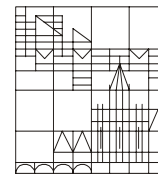


Gaschromatogramm von Metabolitextrakten (oben) und Ionenchromatogramm des Ringfragmentes (m/z 129) (unten)





GC-MS Urinanalyse von einem Pferd



Dehydroepiandrosteron (Oxim-TBDMS-Derivat)

