

(NEVŠEDNÍ) DETEKCE

Pokročilá kapalinová
chromatografie

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DETEKCE

Selektivní – odezva pouze na koncentraci analyzované látky

Univerzální – odezva úměrná celkové vlastnosti eluátu

Ideální detektor

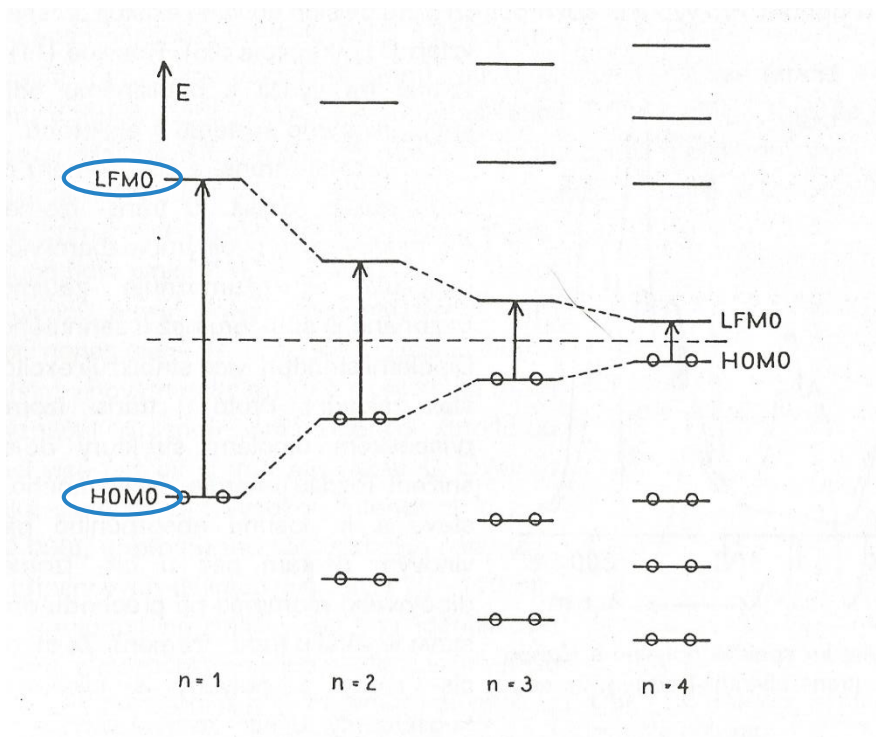
citlivý, malý šum, okamžitá odezva, lineární v širokém koncentračním rozsahu, minimální příspěvek k rozšiřování píků, málo citlivý ke změnám tlaku, teploty a průtoku, možnost práce s gradientem – *kompromis*

Nejpoužívanější

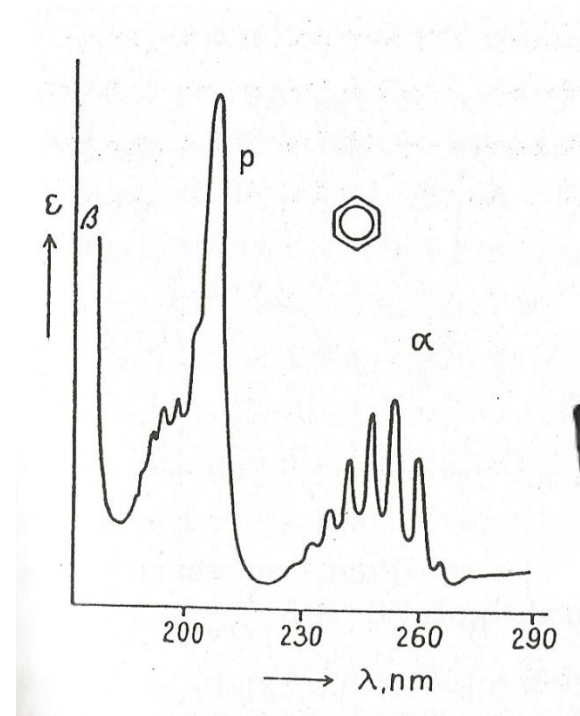
spektrofotometrický UV – VIS, fluorimetrický, rozptyl světla, elektrochemický (amperometrický, coulometrický), vodivostní, hmotnostní (HPLC/MS)

SPEKTROFOTOMETRIE (UV)

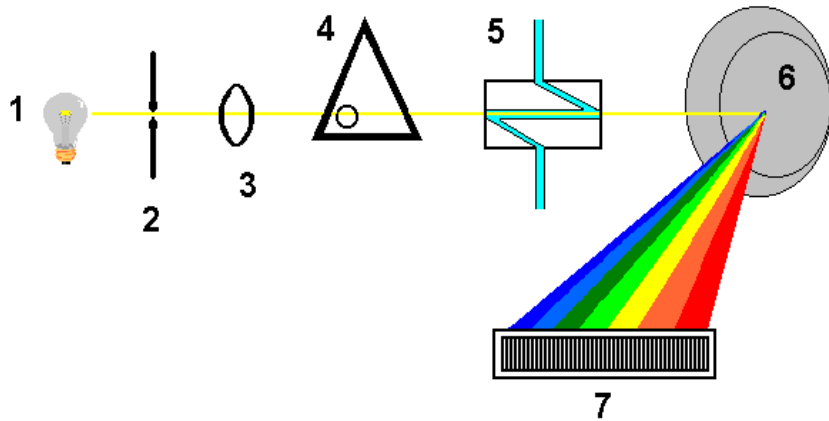
Konjugace dvojných vazeb



UV spektrum benzenu

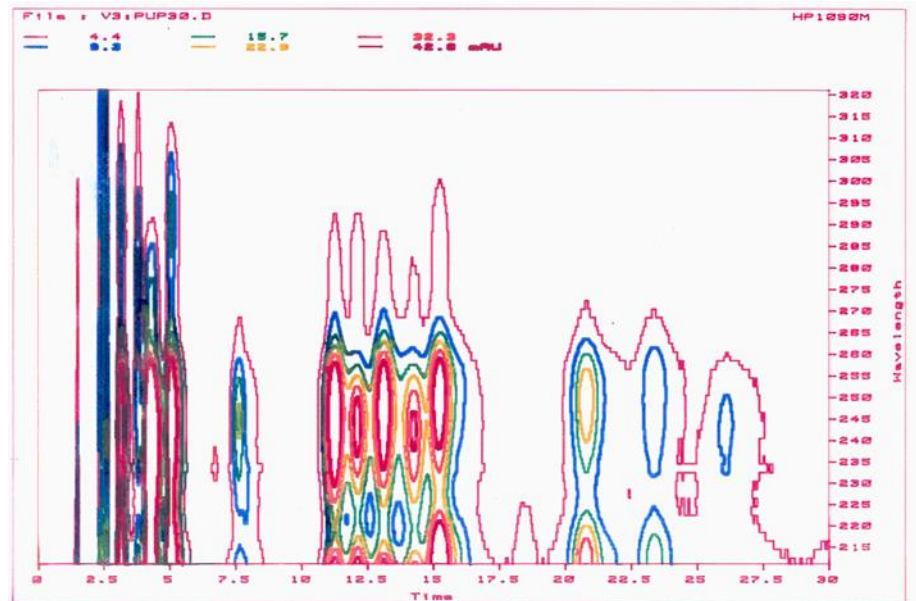
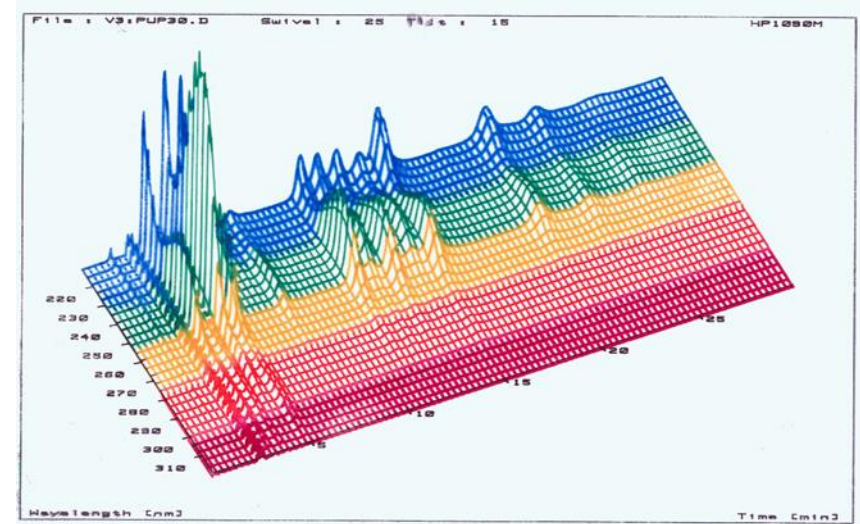


SPEKTROMETRICKÝ DETEKTOR

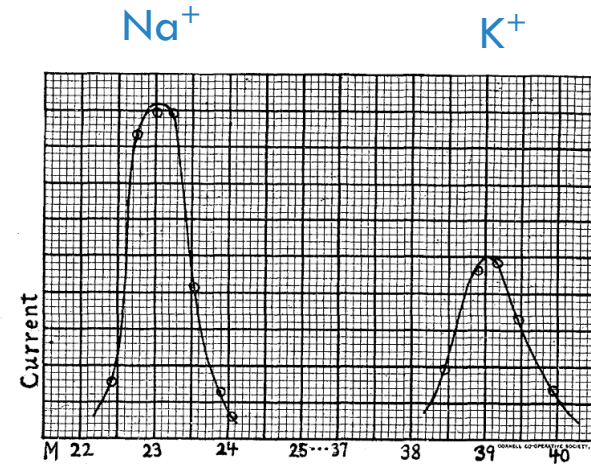
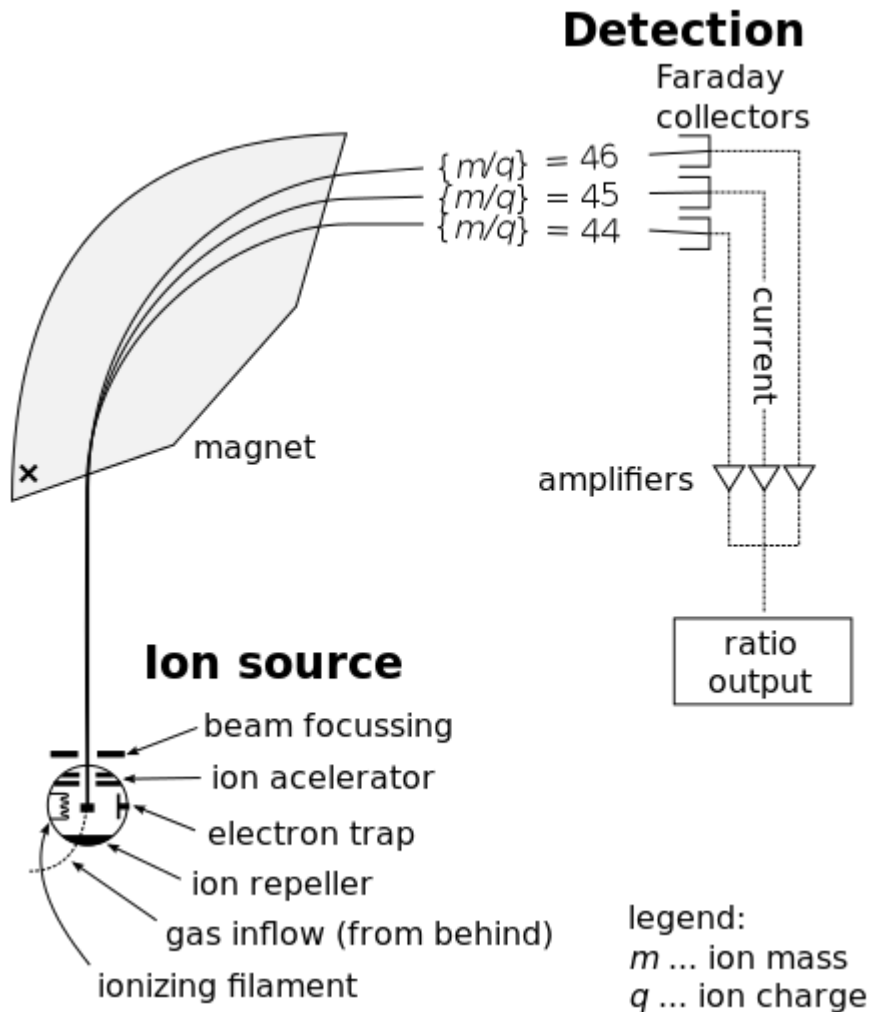


(1) záření ze zdroje, (2) štěrбина, (3) čočka,
(4) clona, (5) měrná cela detektoru, (6) holografická
mřížka, (7) pole fotodiód.

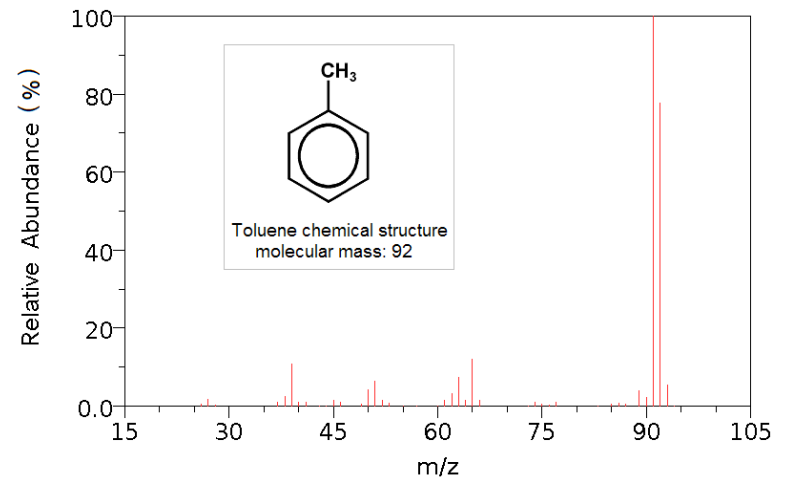
Poskytuje v aktuálním čase
UV spektrum pro každý pik



HMOTNOSTNÍ SPEKTROMETRIE (MS)

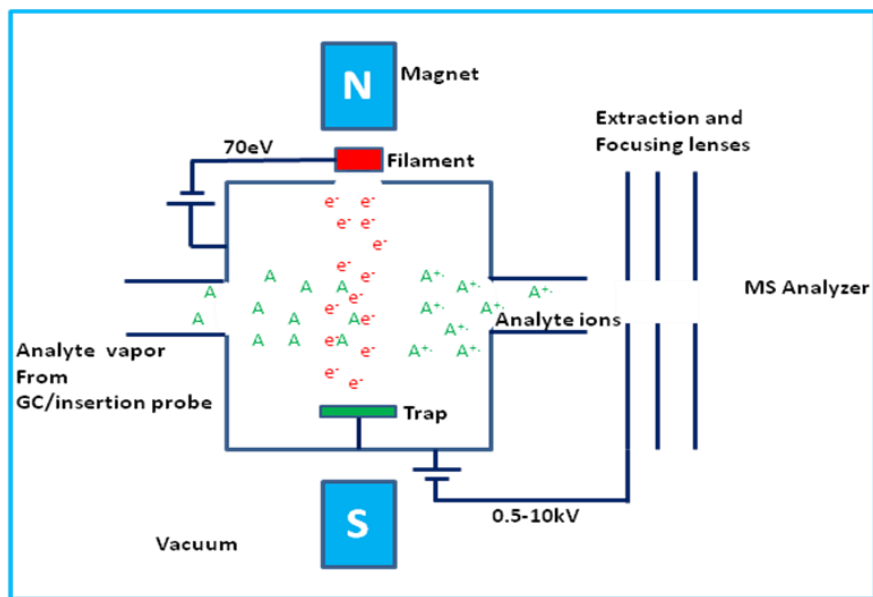


"A new Method of Positive Ray Analysis" Phys. Rev. 11 (1918) 316.

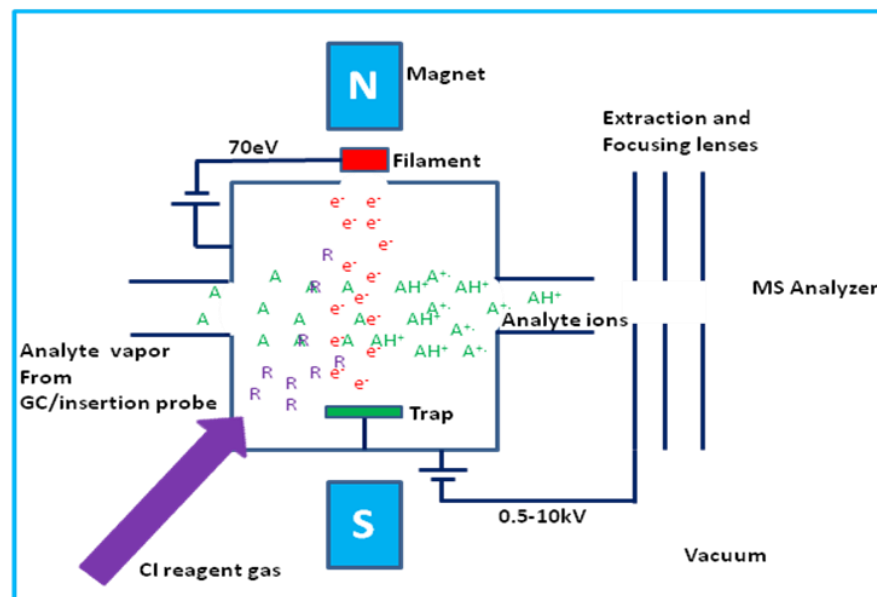


IONIZACE

Elektronová ionizace

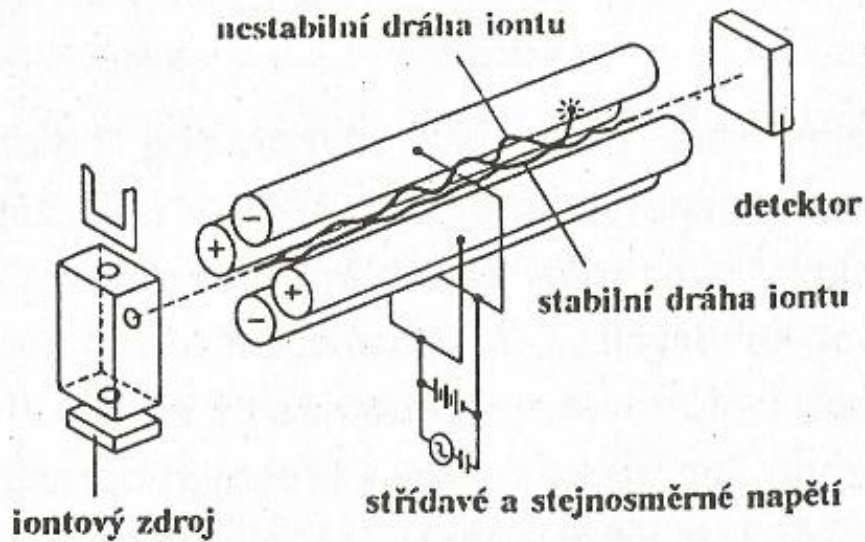


Chemická ionizace

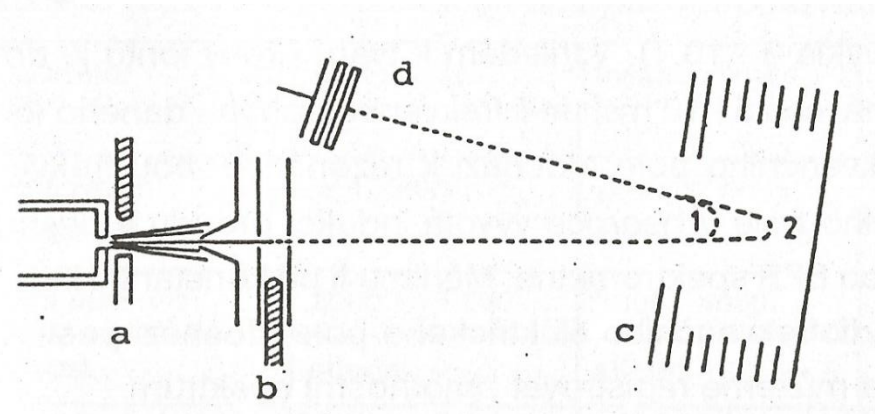


ANALYZÁTORY

Kvadrupol

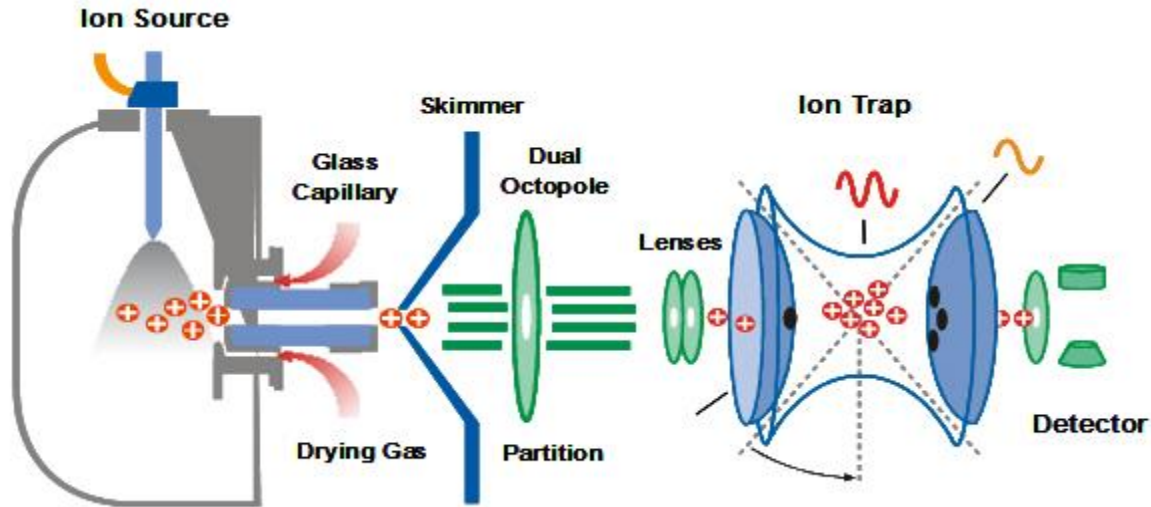


Time of flight

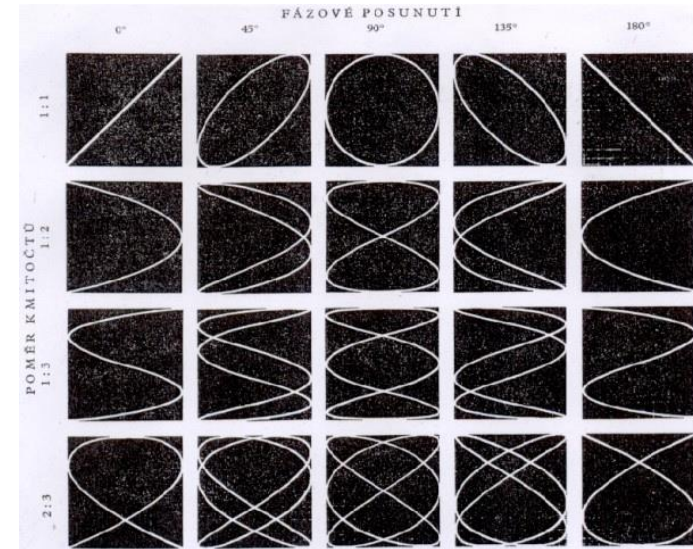


ANALYZÁTOR

Iontová past

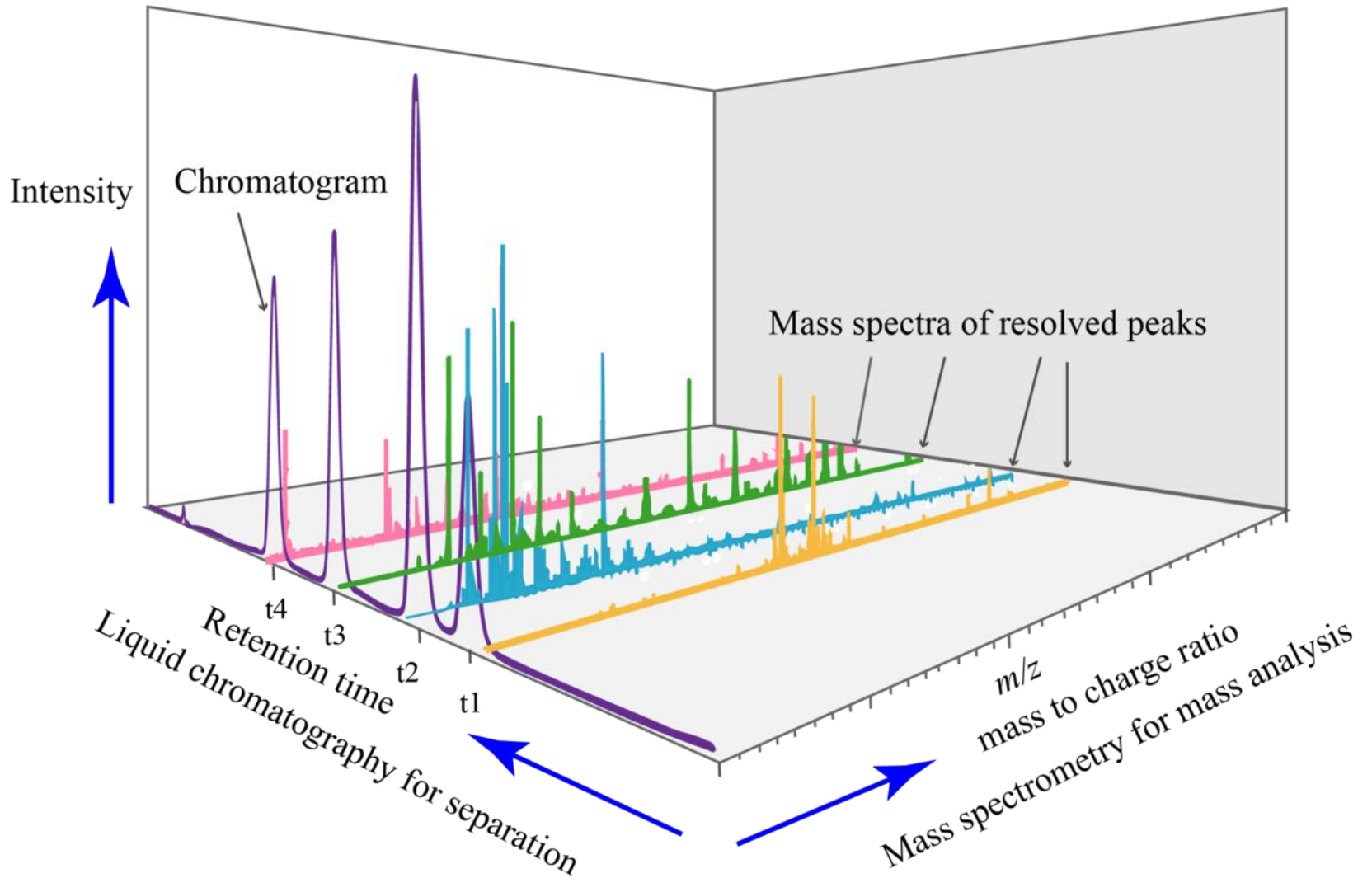


penyfan.ugent.be

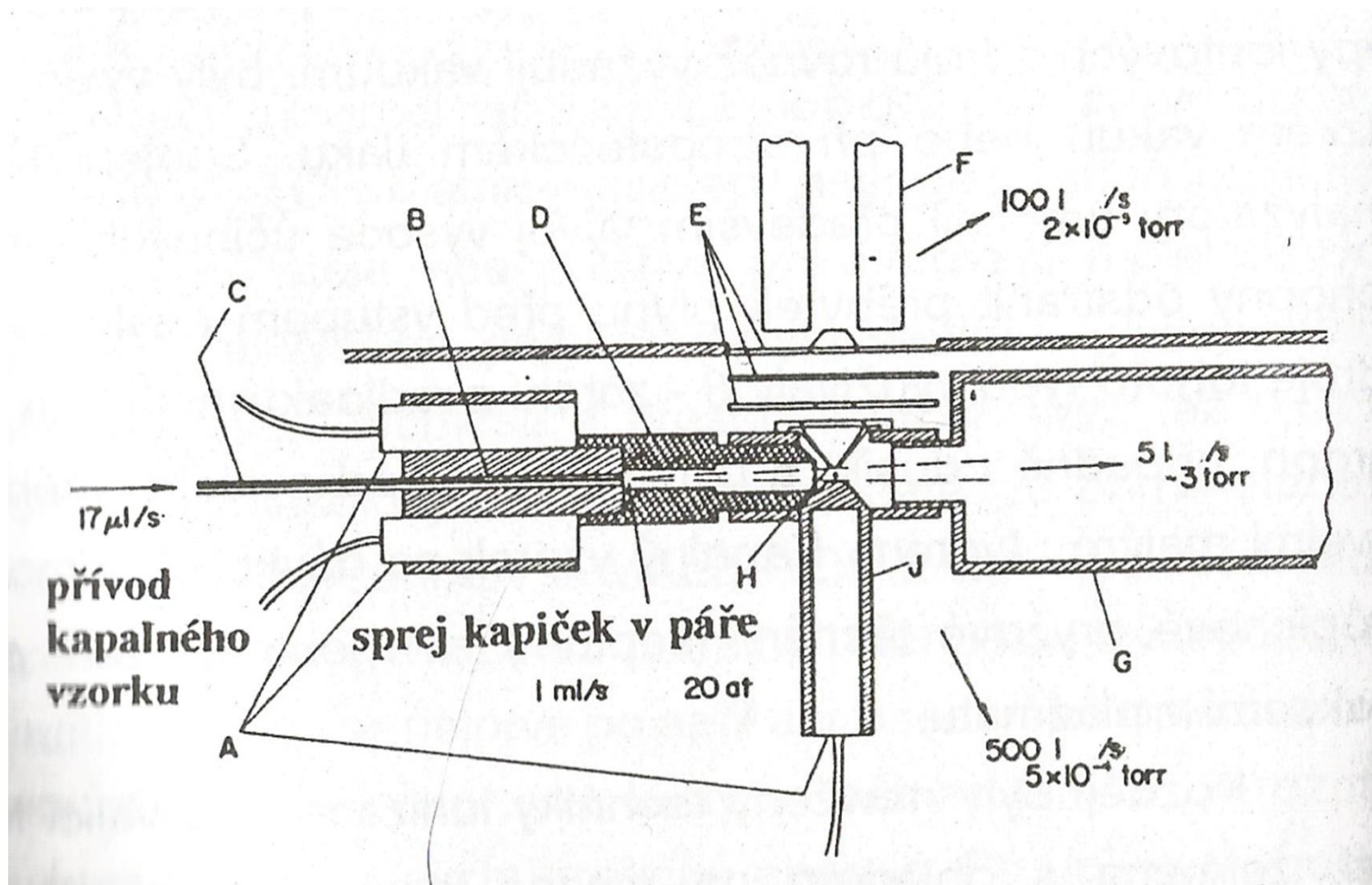


Jan Ptáčník - GJVJ - Fyzika - 3.A

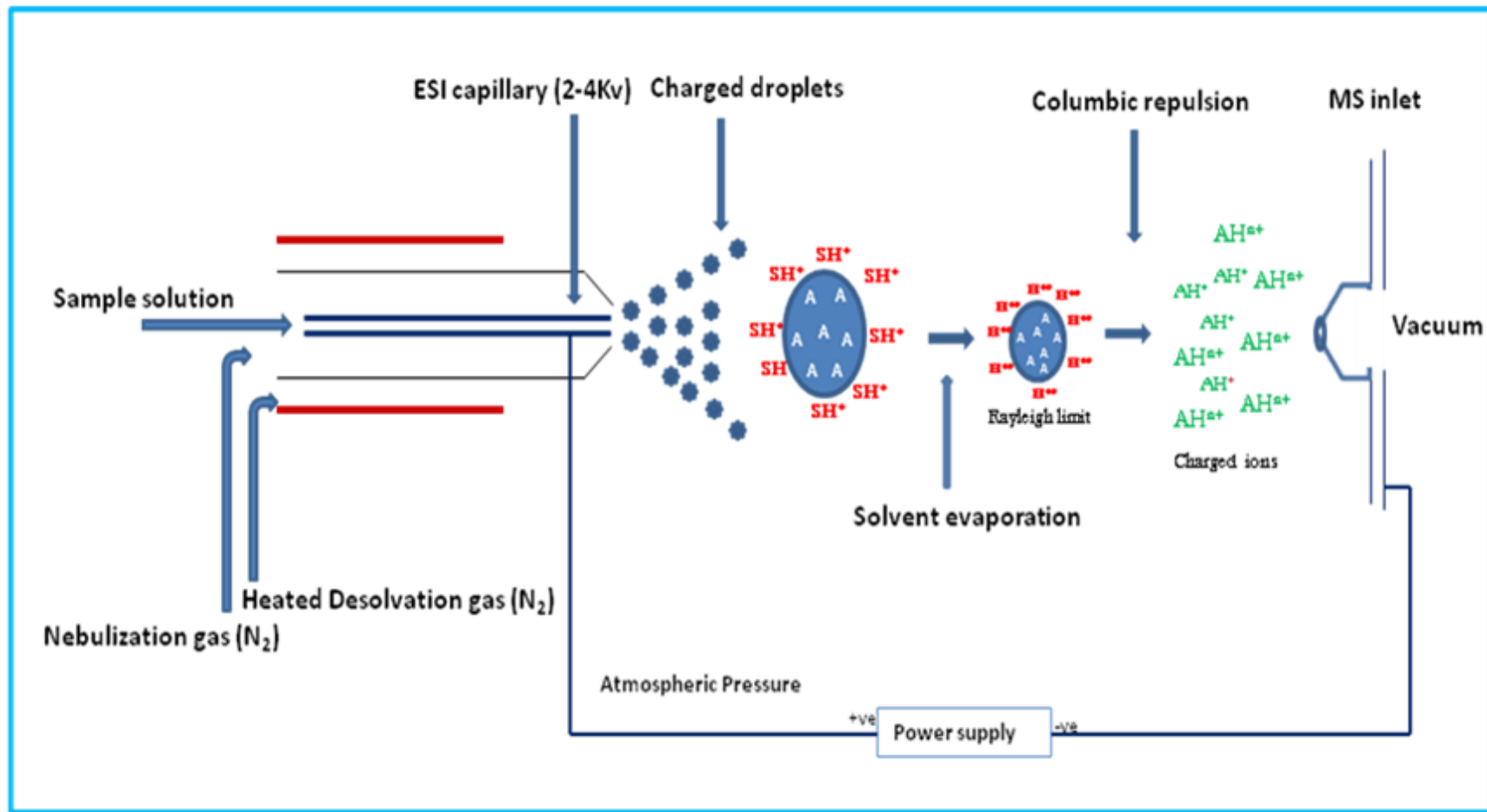
HPLC-MS



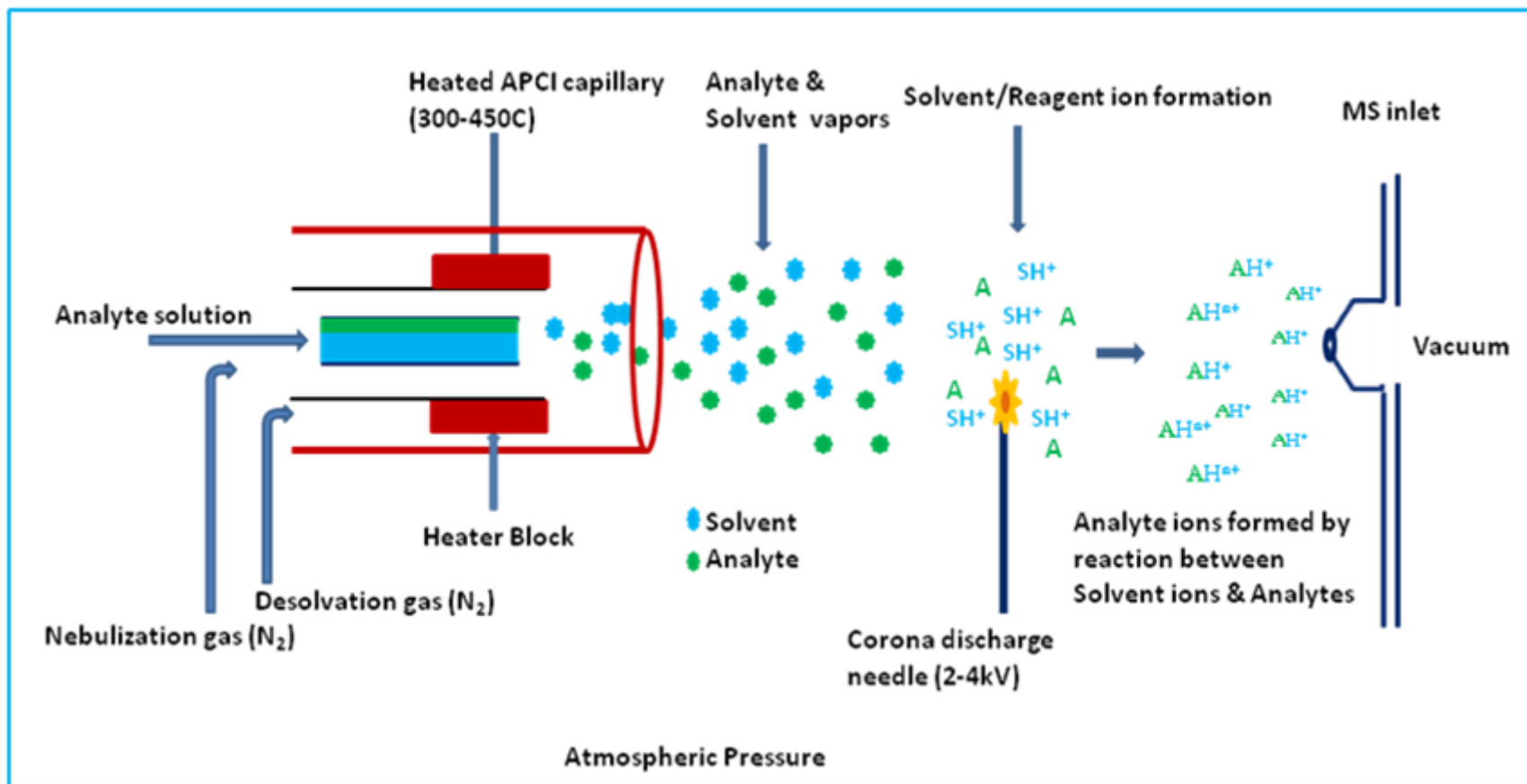
TERMOSPREJ



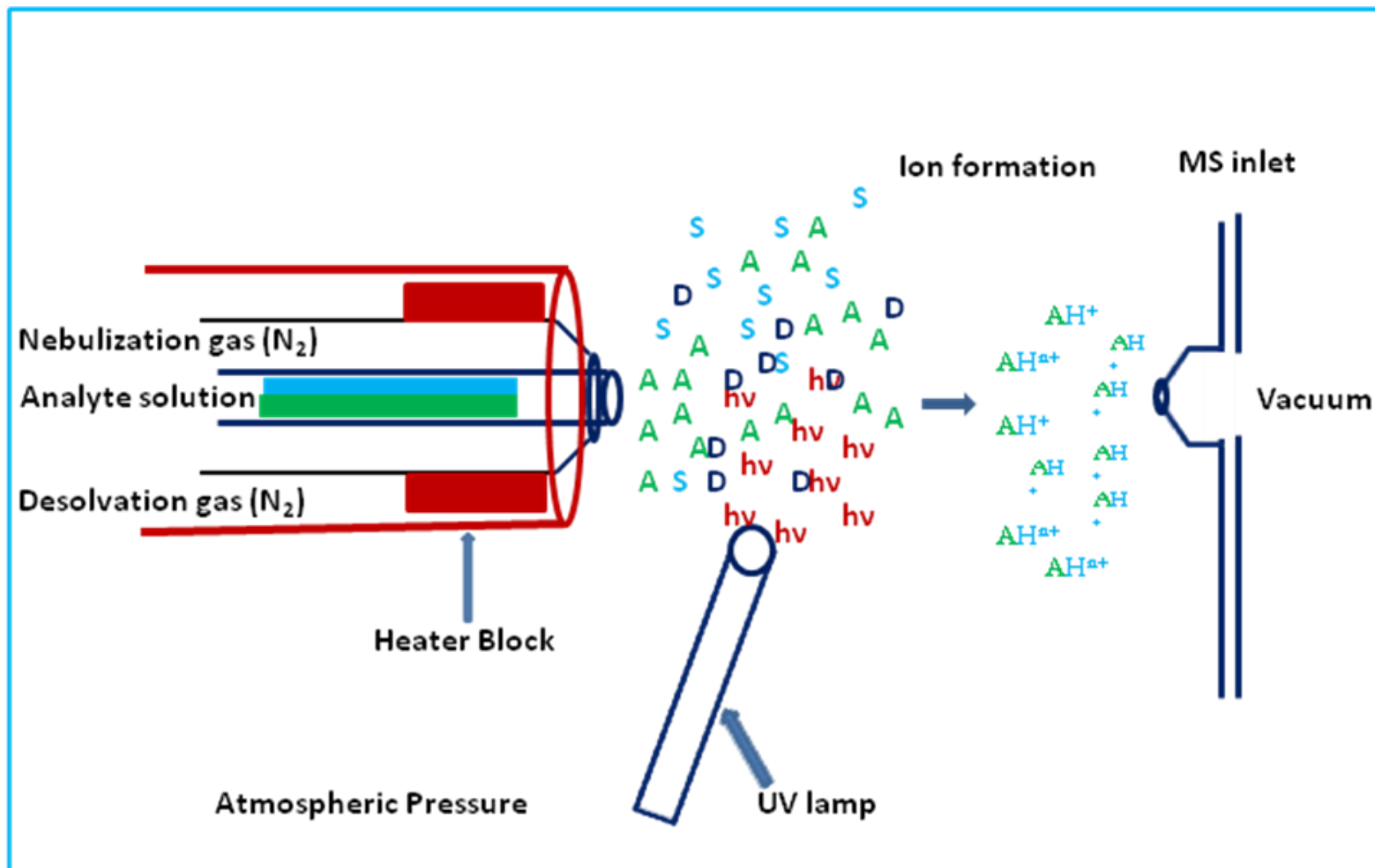
ELEKTROSPREJ (ESI)



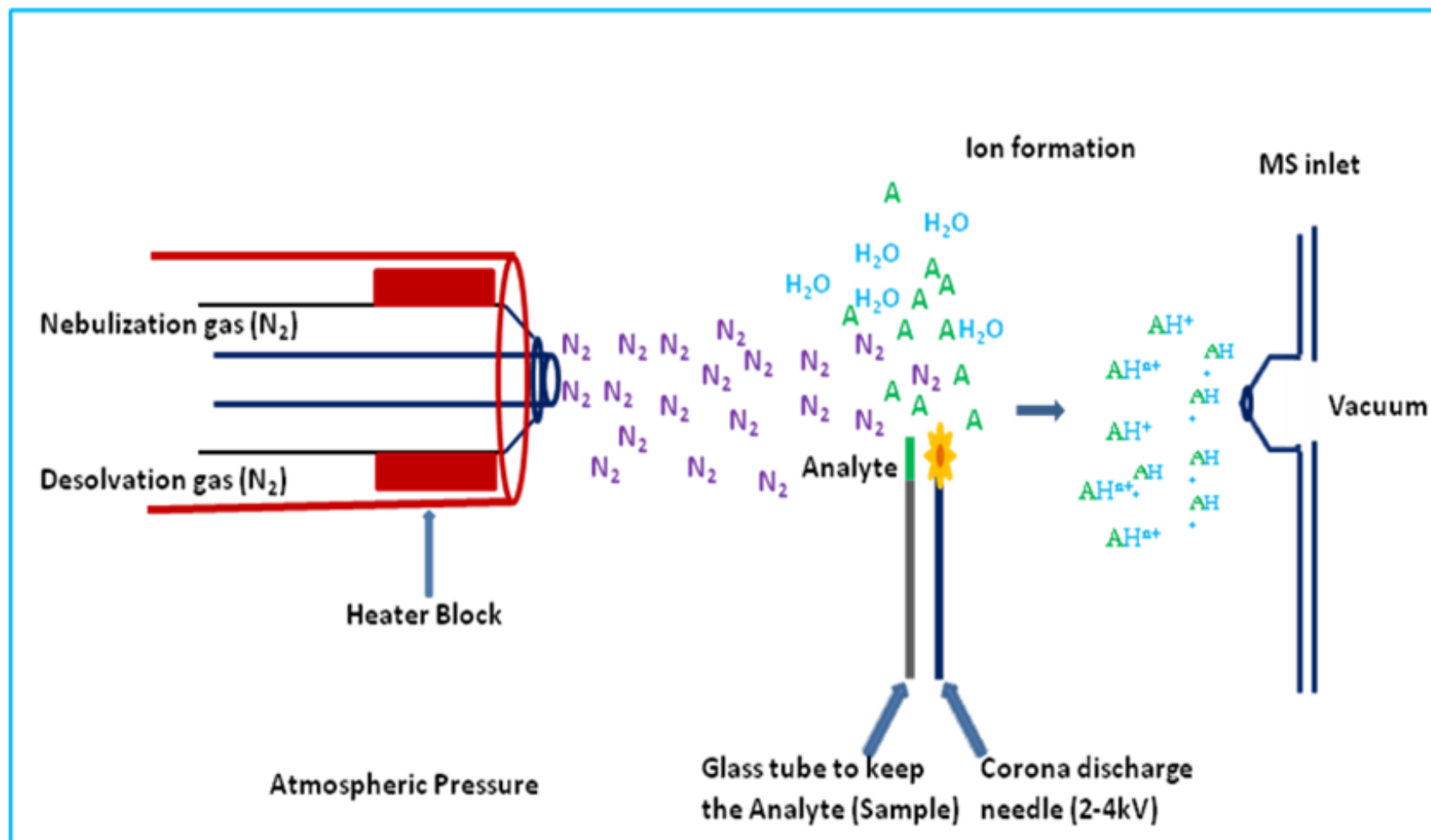
ATMOSPHERIC PRESSURE CHEMICAL IONIZATION (APCI)



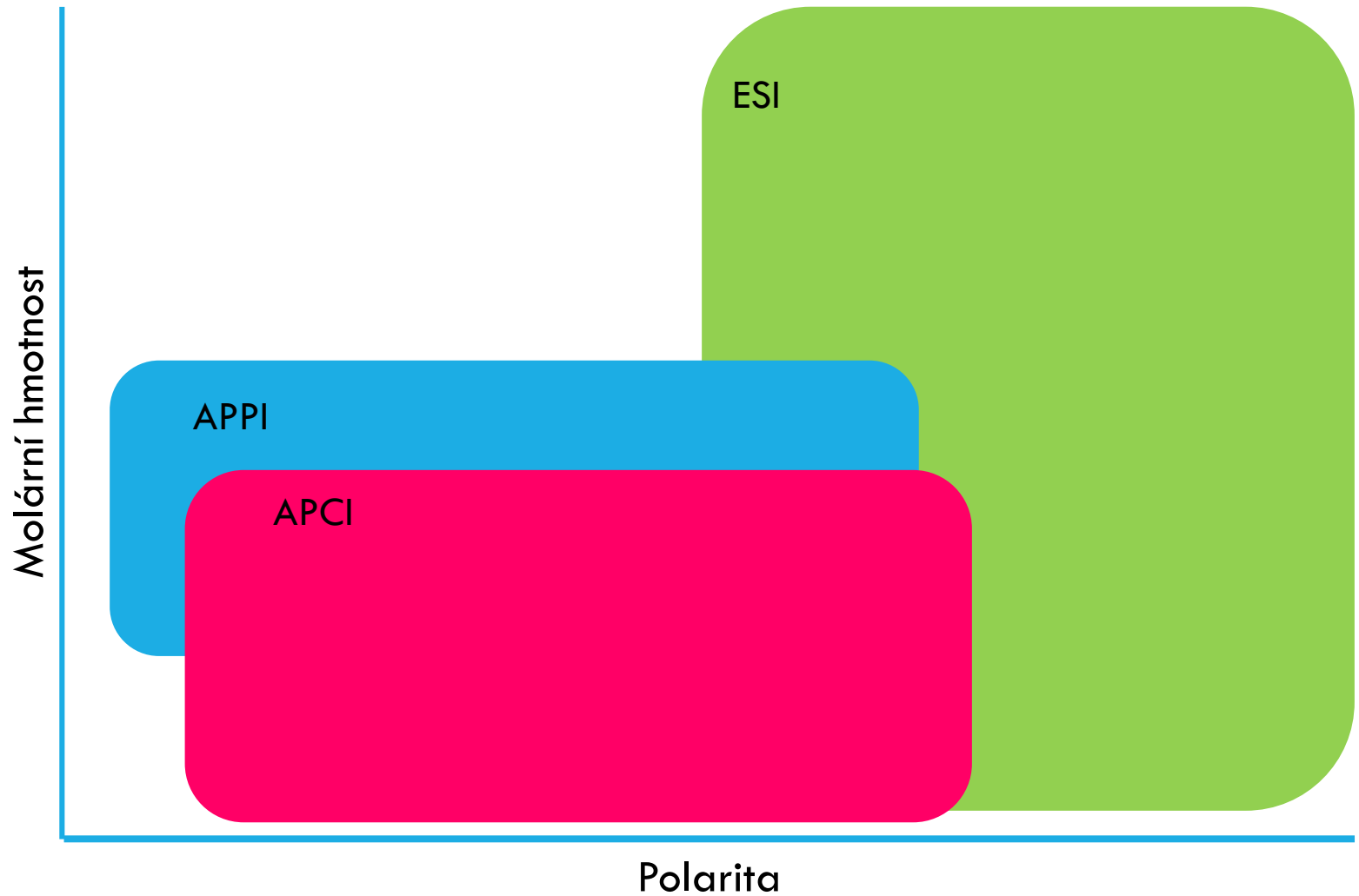
ATMOSPHERIC PRESSURE PHOTO IONIZATION (APPI)



ATMOSPHERIC SOLID ANALYSIS PROBE IONIZATION (ASAP)



VÝBĚR TECHNIKY



FLUORESCENCE

Měří sekundární emisi záření, selektivní, citlivý.

Zdroj záření

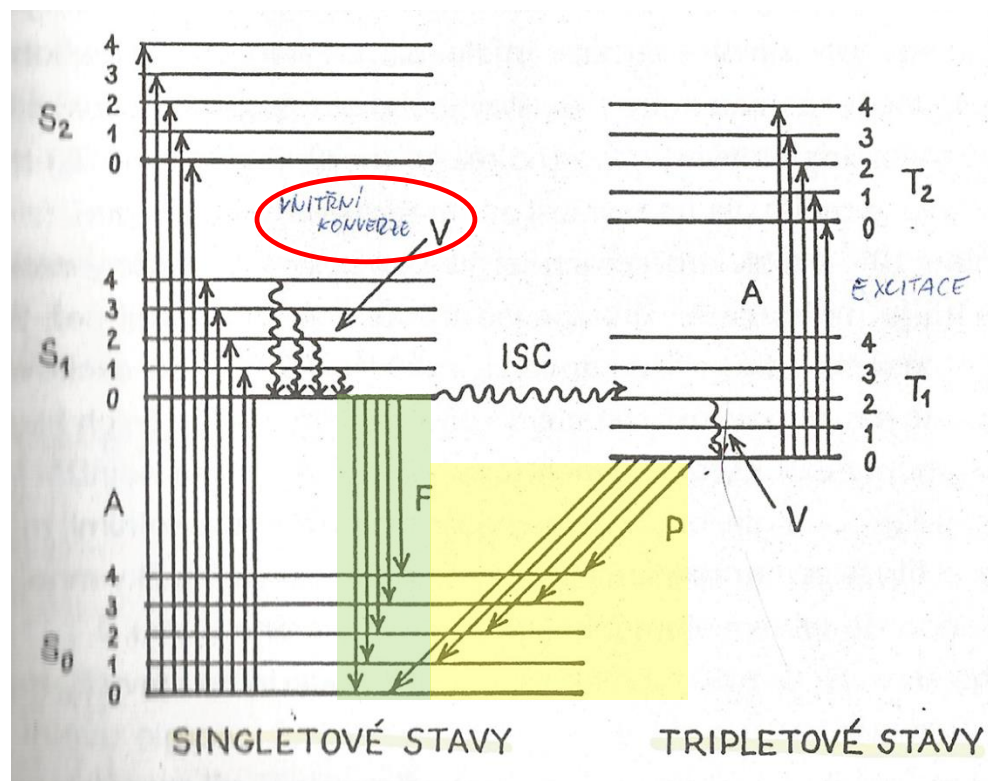
- Nízkotlaká rtuťová výbojka
- Laser - vysoká citlivosti detekce - kapilární HPLC.
- Xenonová oblouková výbojka

Dva monochromátory pro budicí i emitované záření,

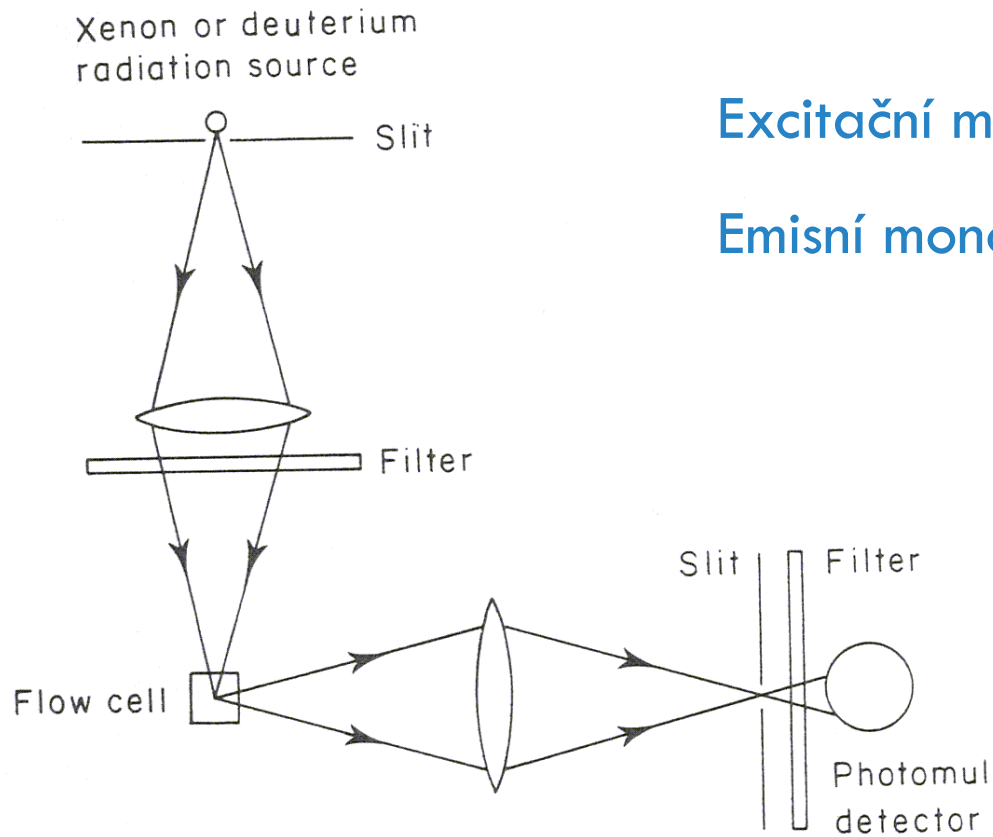
Měření fosforescence po ukončení výboje budicího záření

Derivatizace

aminy, aminokyseliny.



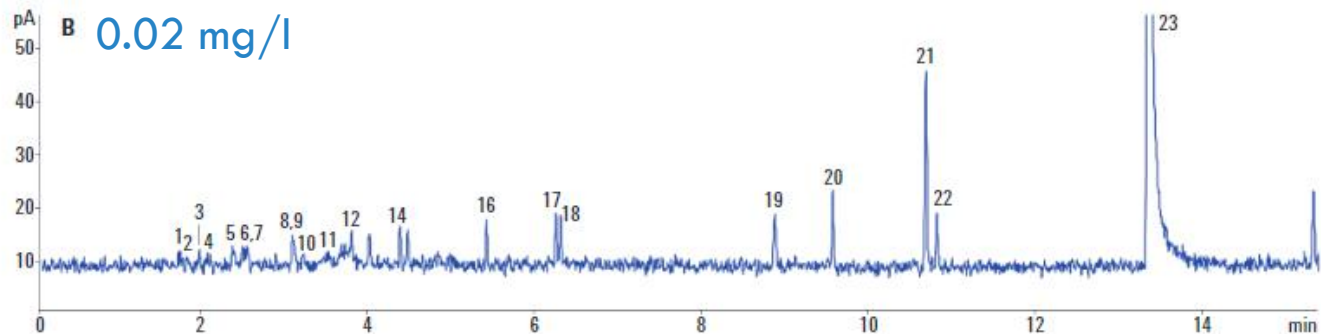
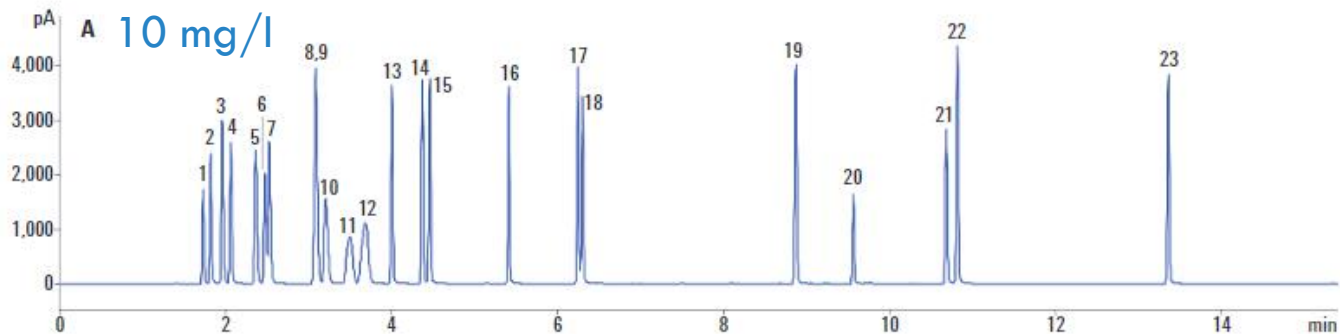
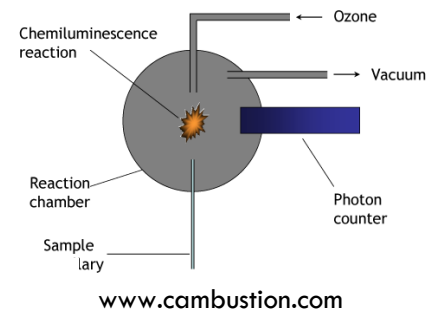
FLUORESCENCE – SCHÉMA PŘÍSTROJE



Excitační monochromátor (vlnová délka)

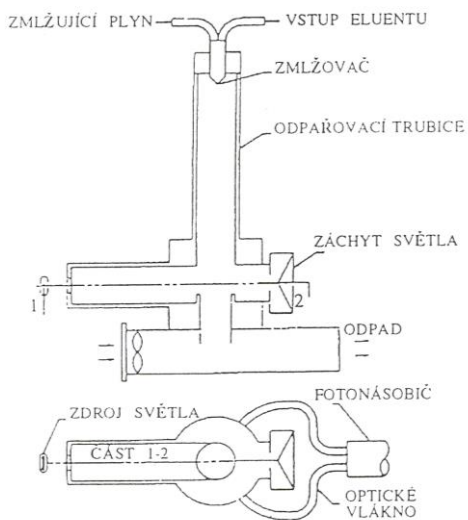
Emisní monochromátor (vlnová délka)

CHEMILUMINESCENCE (SCD)

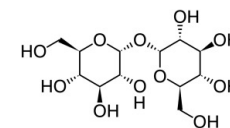
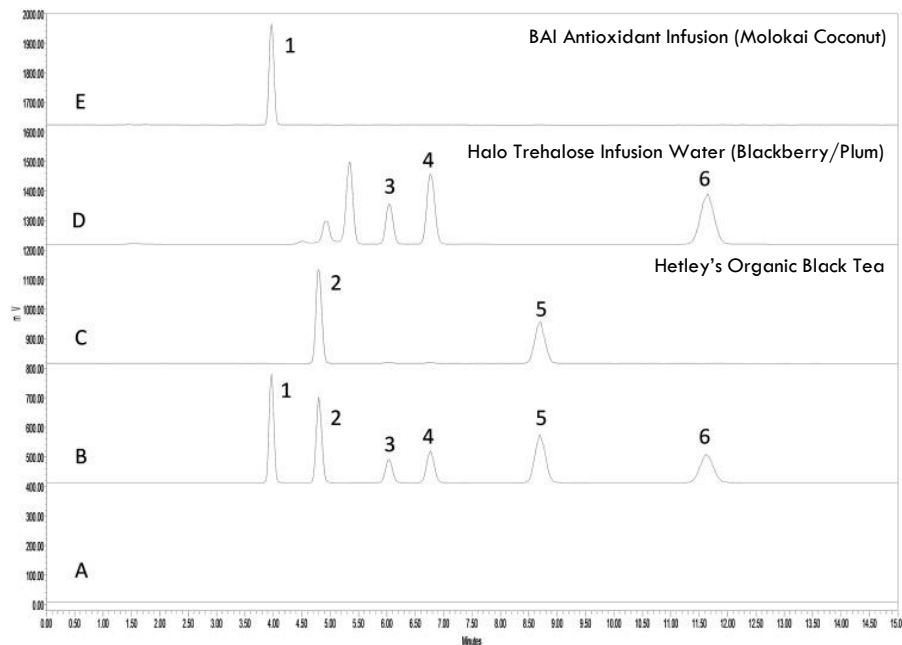


- Sířa, dusík
- GC, SFC, (LC)
- petrochemie

DETEKTOR ROZPTYLU SVĚTLA (ELSD)



Schema detektoru rozptylu světla (ELSD)



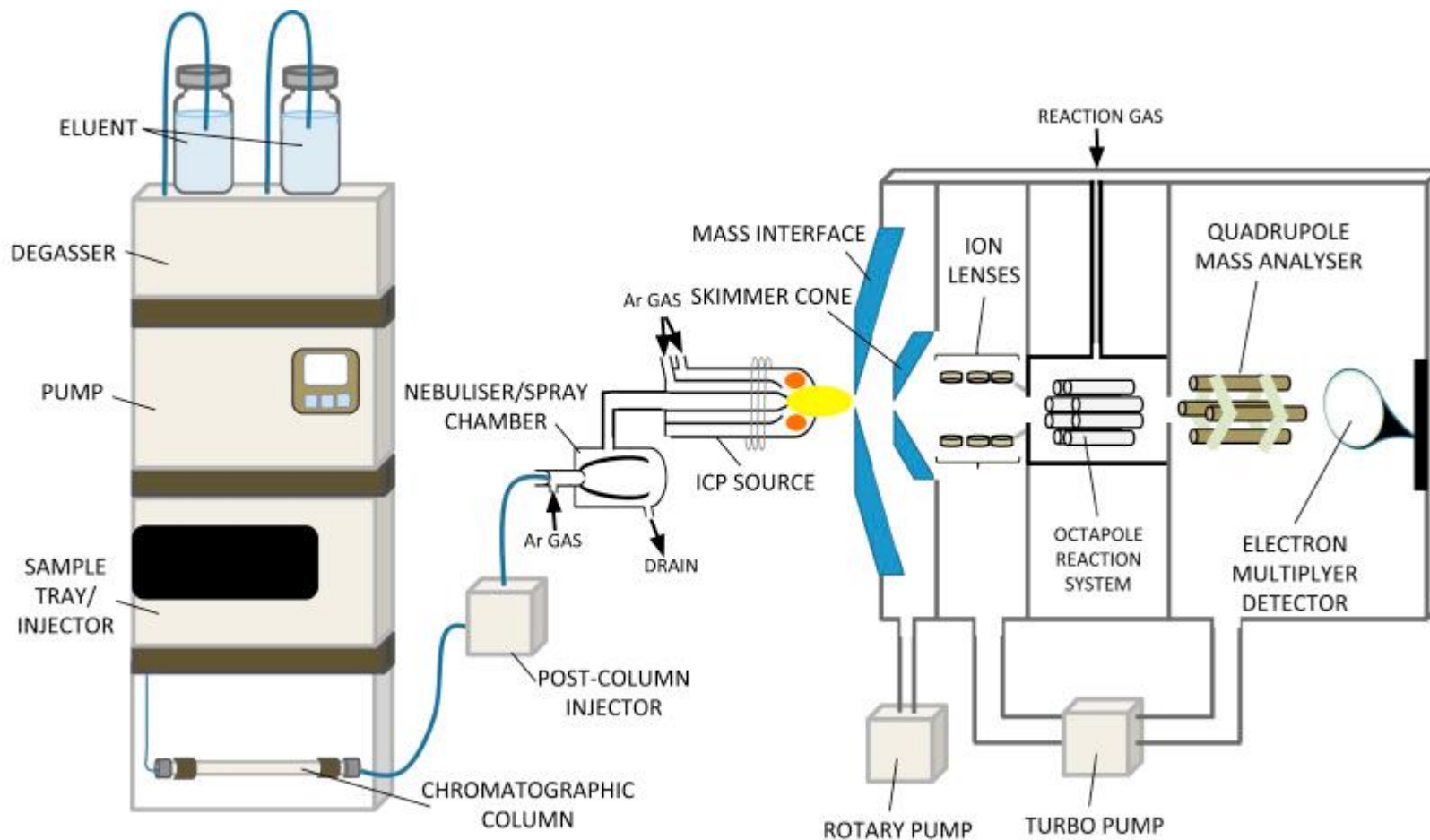
trehalóza

J. Chromatogr. A 1489 (2017) 65–74.

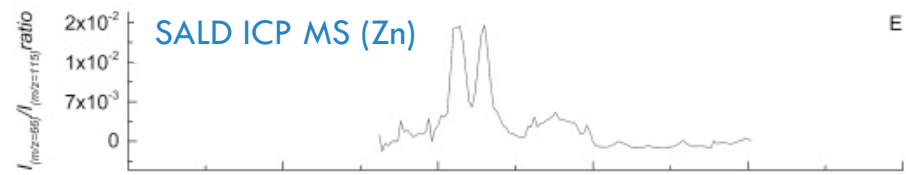
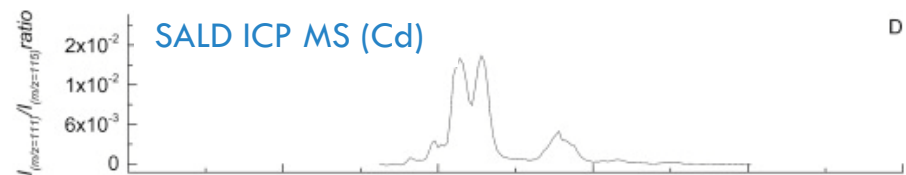
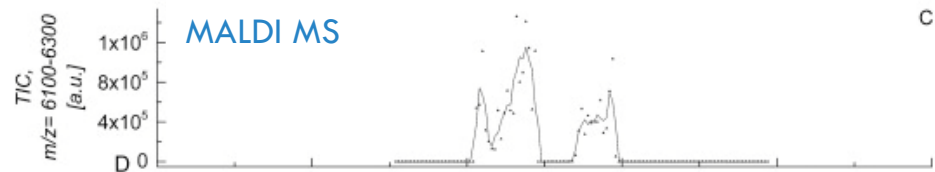
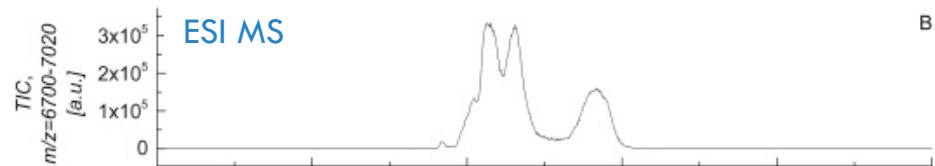
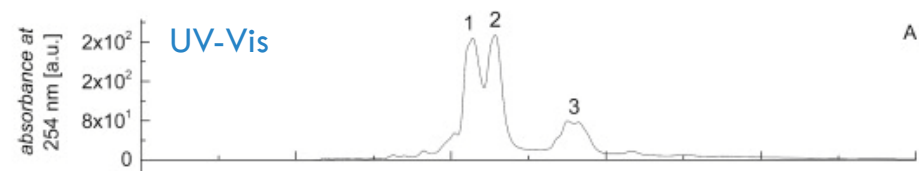
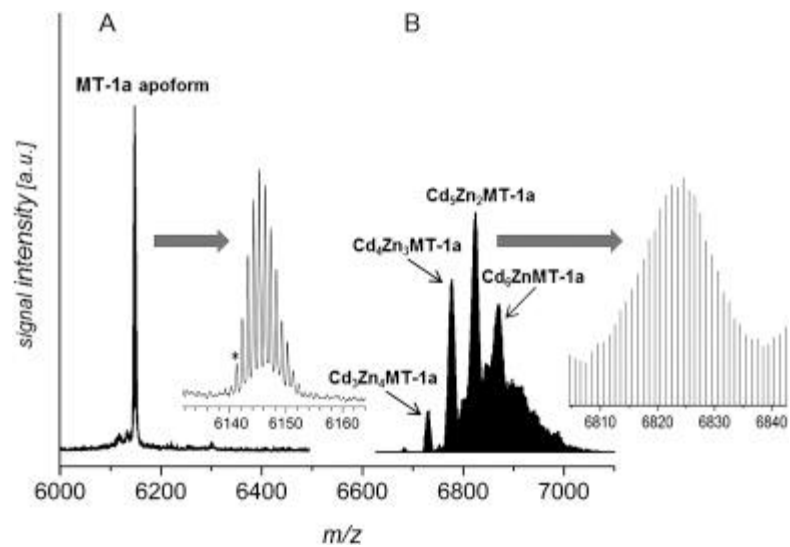
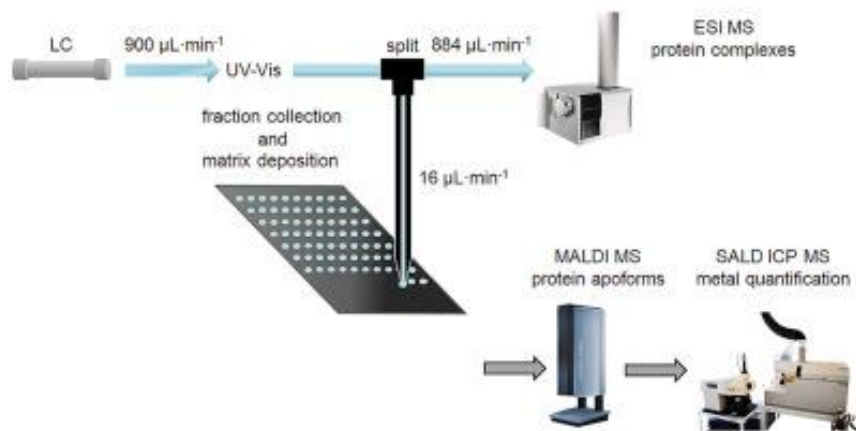
- Univerzální
- Netěkavé látky
- Cukry, AK
- Gradientová eluce



ATOMOVÁ SPEKTROMETRIE (ICP)

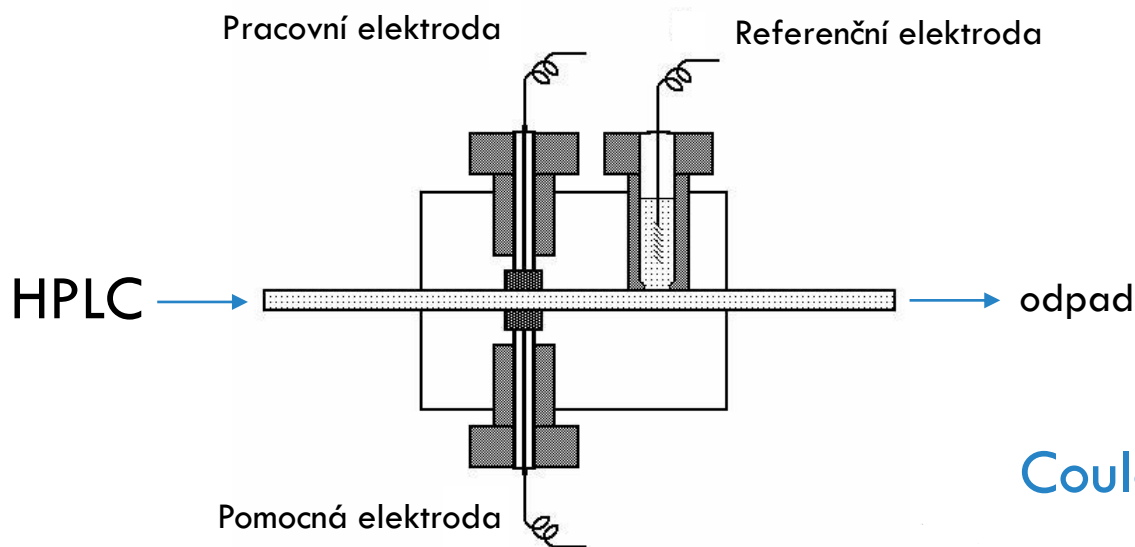


METALPROTEIN



t_R [min]

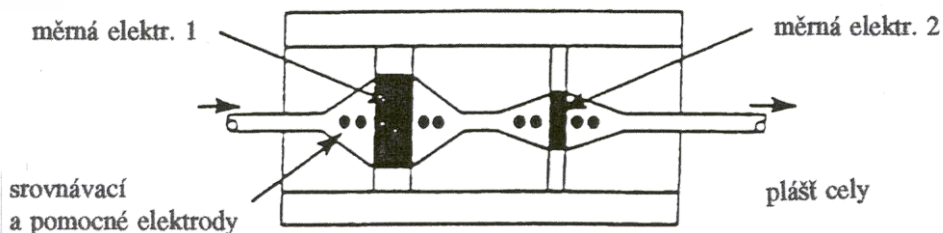
ELEKTROCHEMICKÁ DETEKCE (EC)



- Změna proudu při oxidaci nebo redukci analytu

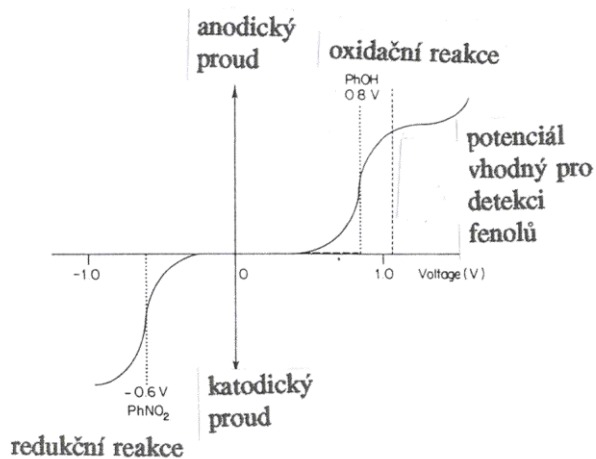
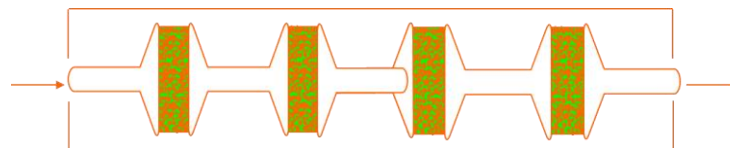
$$\text{Ox} + n \cdot e^- \rightarrow \text{Red}$$
- Uměrná koncentraci látky

Coulometrický detektor



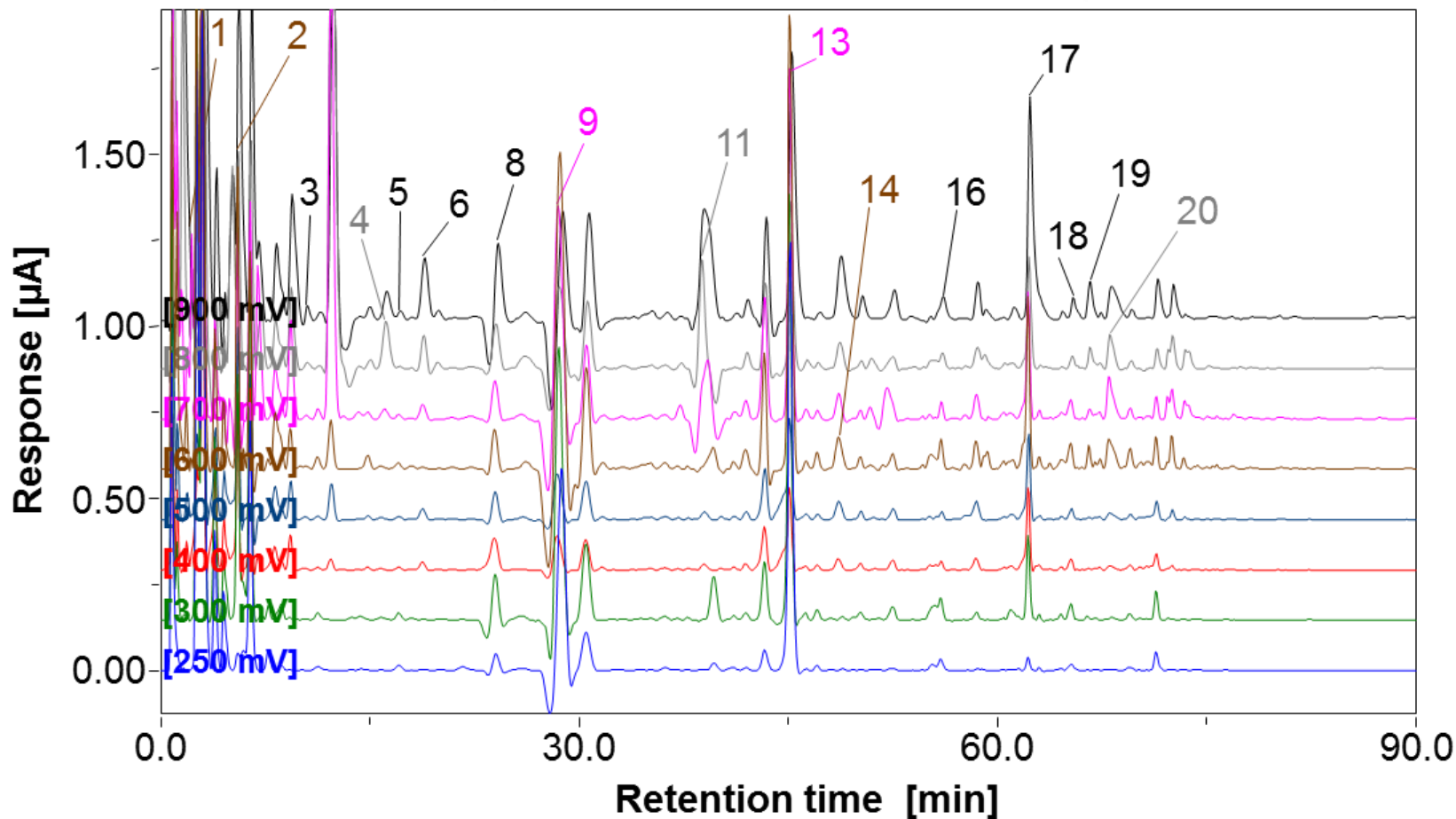
Průtočné elektrody z porézního grafitu

Elektrodové pole
CoulArray (4 – 12)



Vysoká citlivost neurotransmitery, fenoly, aminy

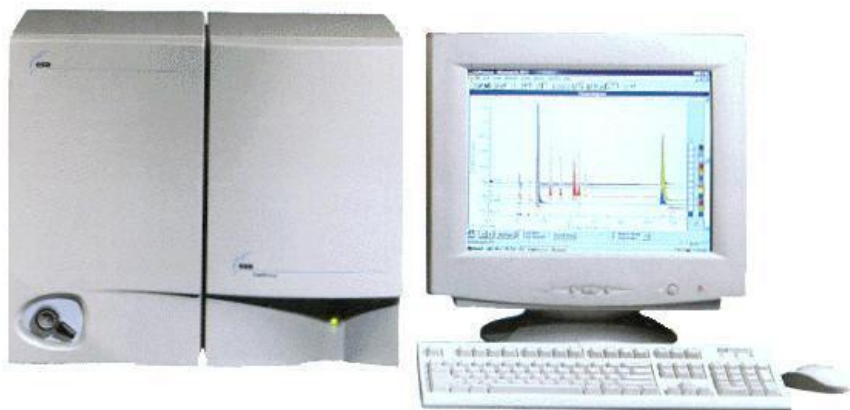
BÍLÉ VÍNO (LC-EC)



1: Galová kys., 2: Protokatechová kys., 3: 4-Hydroxybenzoová kys., 4: 4-hydroxyfenyloct. kys., 5: Salicylová kys., 6: Vanilová kys., 7: Chlorogenová kys., 8: Kávnová kys., 9: (+)-Katechin, 10: Syringová kys., 11: 4-Kumarová kys., 12: Umbelliferon, 13: (-)-Epikatechin, 14: Ferulová kys., 15: Sinapová kys., 16: 4-Hydroxykumarin, 17: Rutin, 18: Kvercetin-3-arabinosid, 19: Naringin, 20: Resveratrol

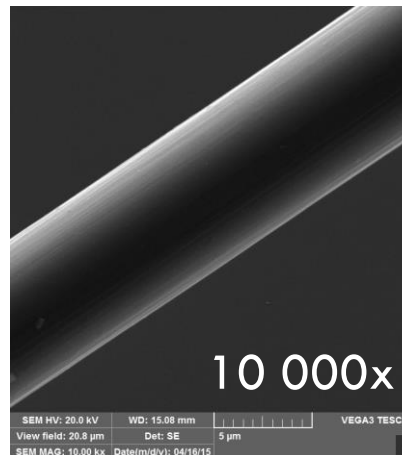
MINIATURIZOVANÁ EC DETEKCE

Komerčně dostupné systémy



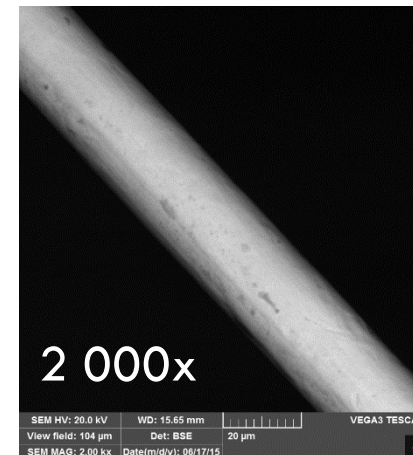
J. Chromatogr. A (2017) zasláno.

Pracovní elektroda

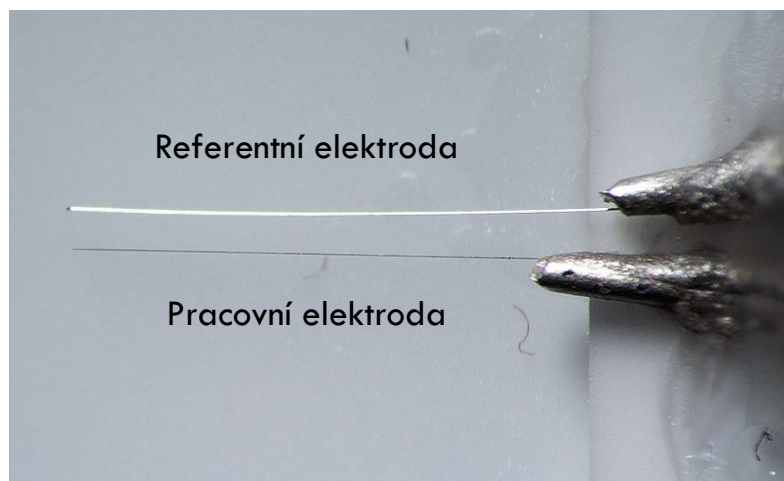


Uhlíkové mikrovlákno, 7 μm

Referentní elektroda

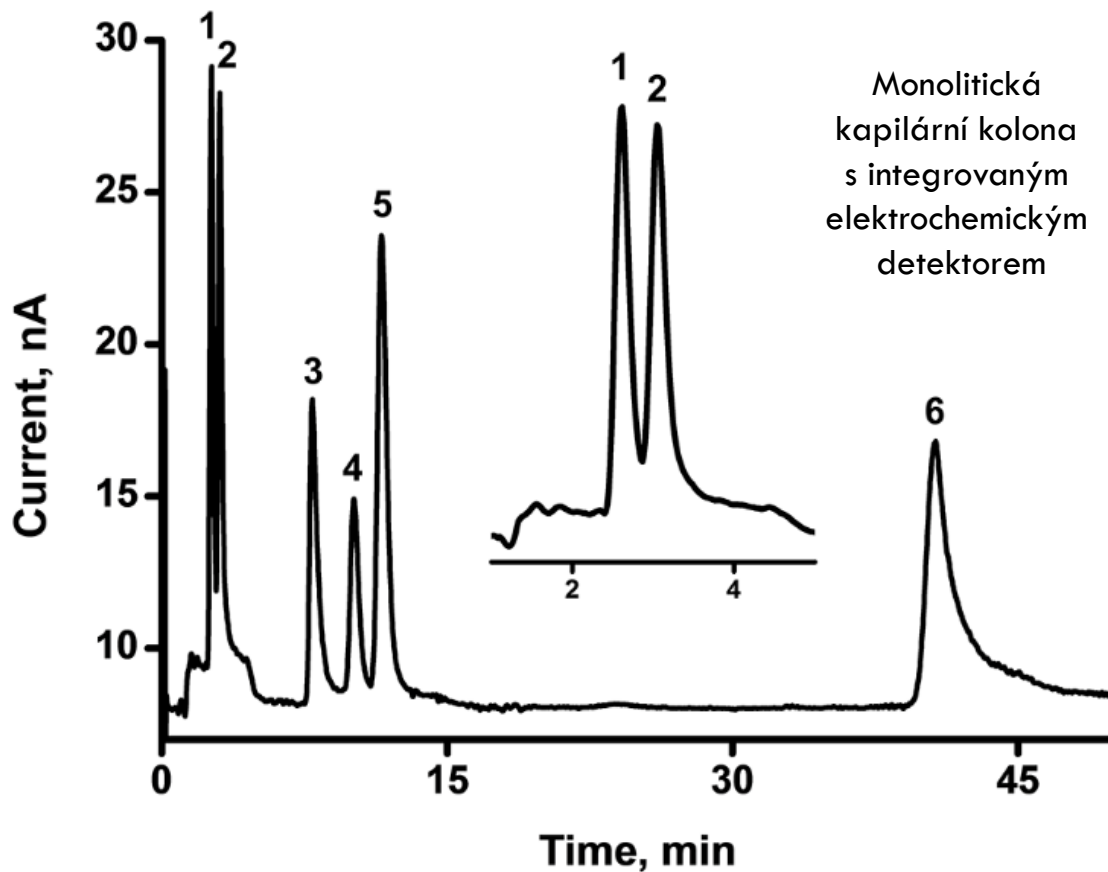


Stříbrný mikrodrát, 25 μm



INTEGROVANÁ ELEKTROCHEMICKÁ DETEKCE

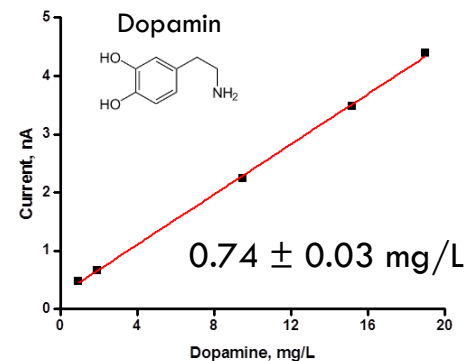
Separace a detekce neurotransmiterů



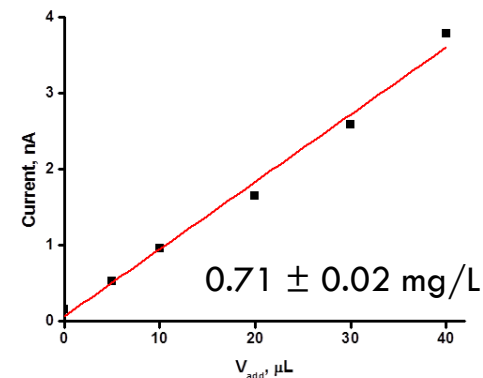
Limit detekce 24 pg nadávkovaného dopaminu

Dopamin v moči

Kalibrační křivka



Standardní přidavek



PŘENOSNÉ ANALYTICKÉ SYSTÉMY

LC-EC v krabici od bot

Monolitické kapilární kolony

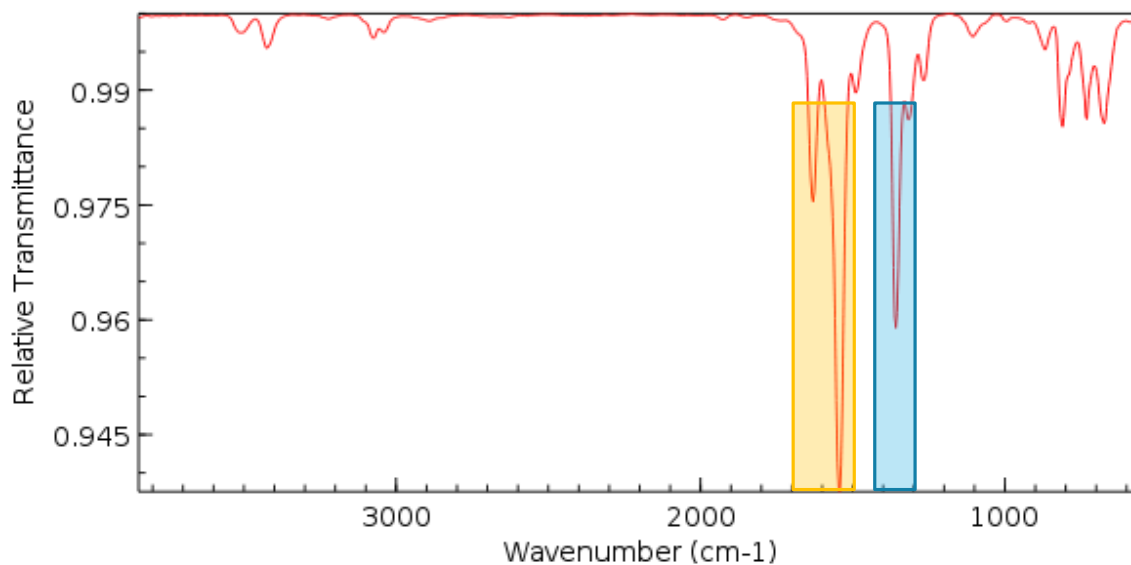
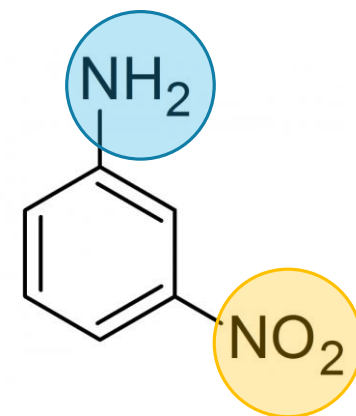
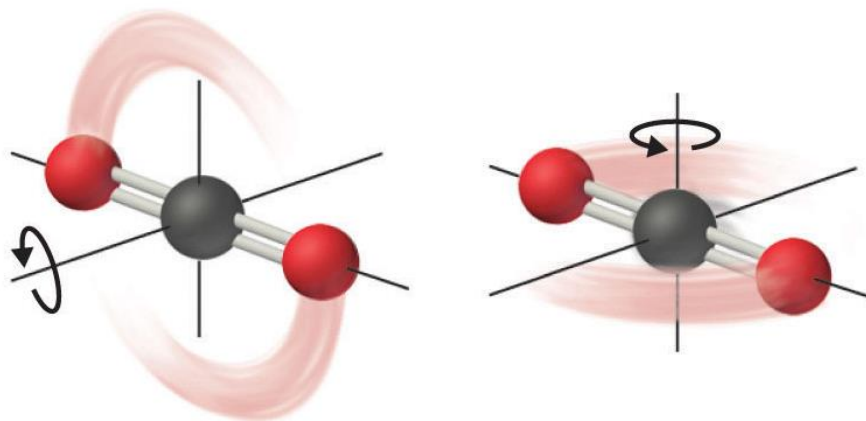
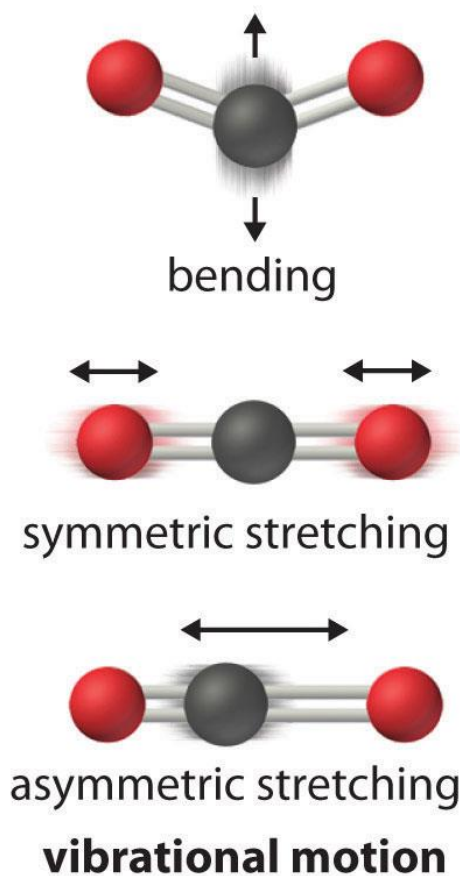
- Snadná příprava
- Cílená změna povrchové chemie
- Miniaturizace
- Nízká cena

Elektrochemické detekce

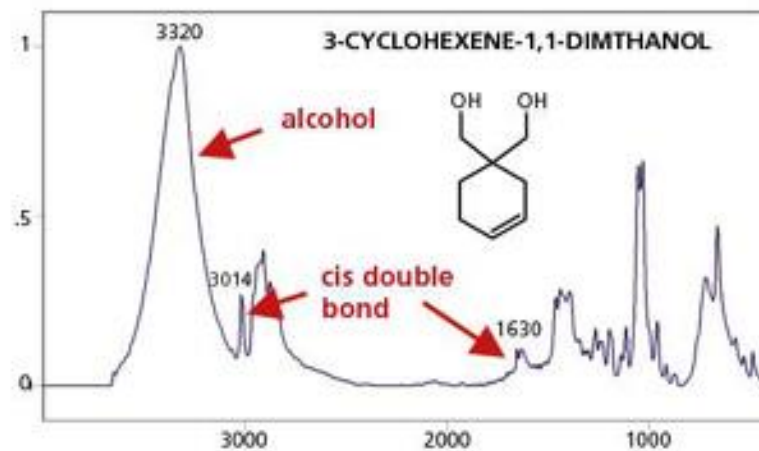
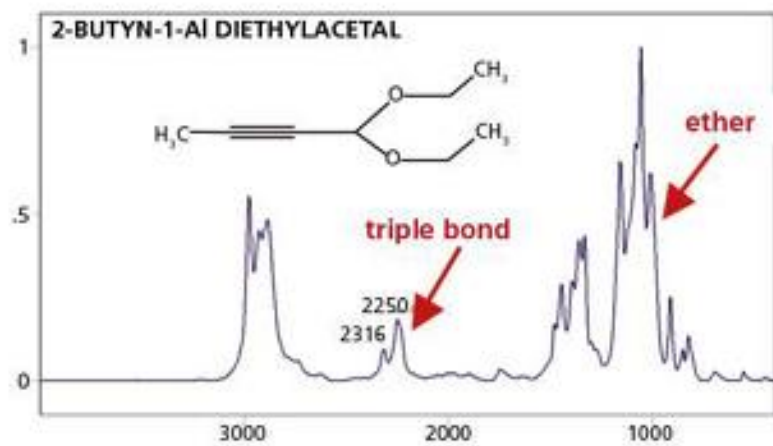
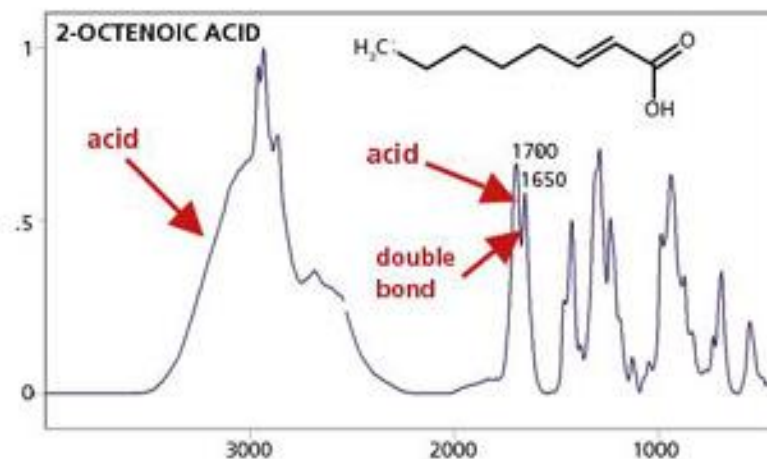
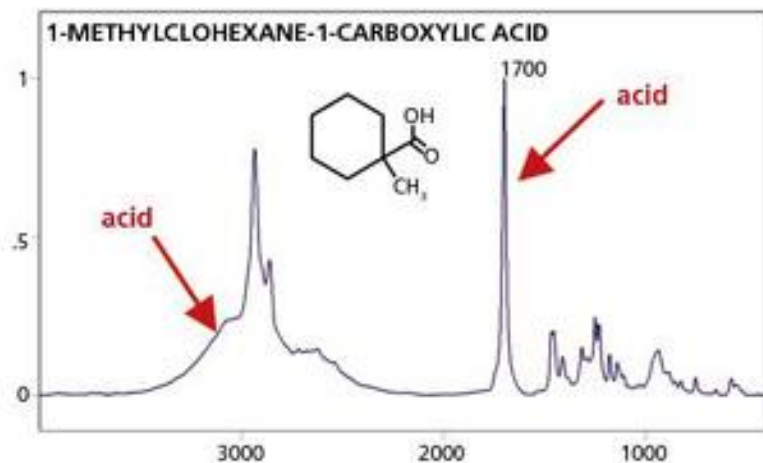
- Vysoká citlivost
- Miniaturizace
- Nízká cena



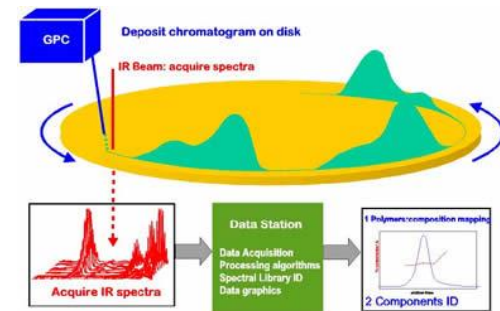
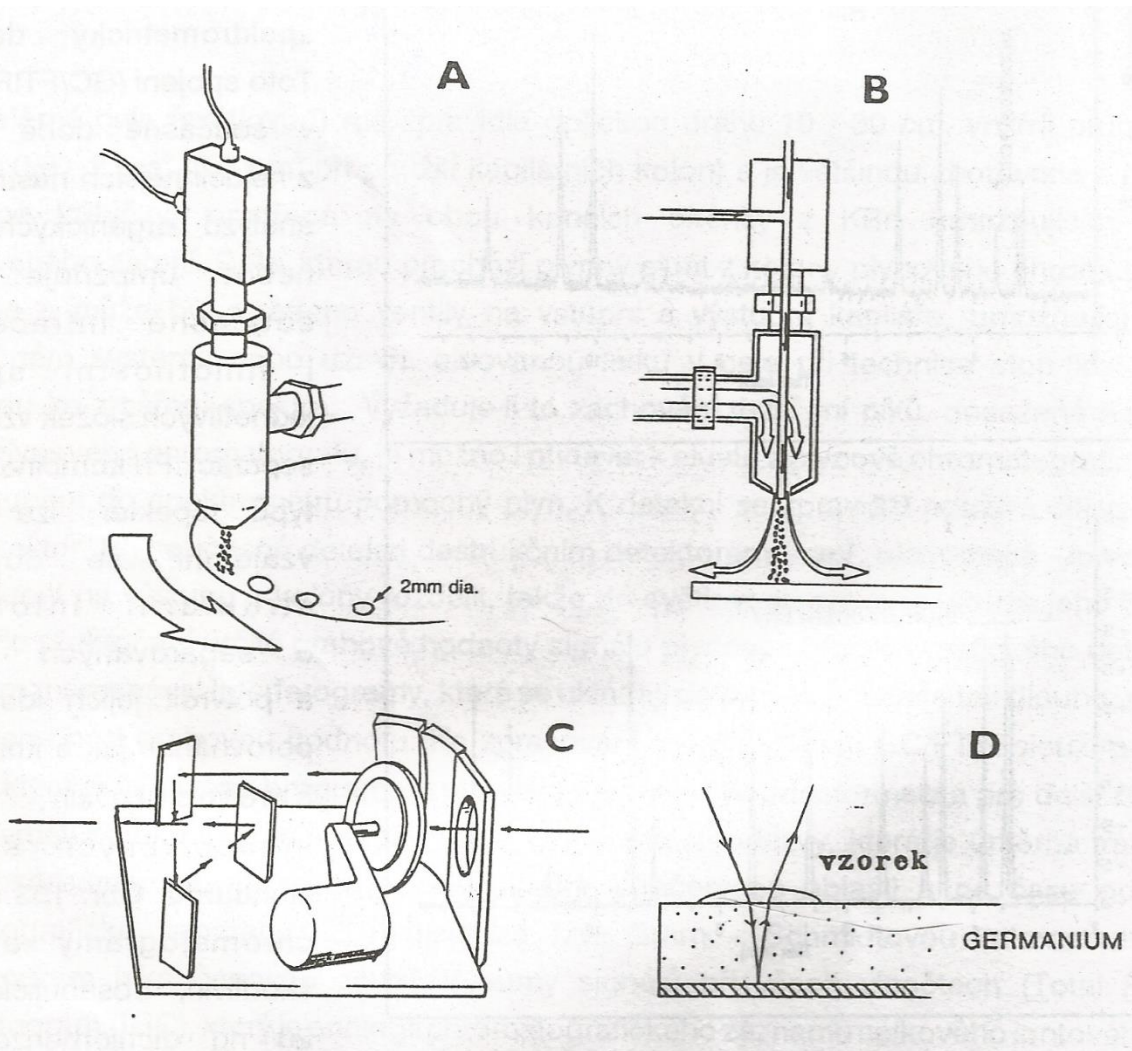
INFRAČERVENÁ SPEKTROSKOPIE (IR)



FTIR – IDENTIFIKACE LÁTEK

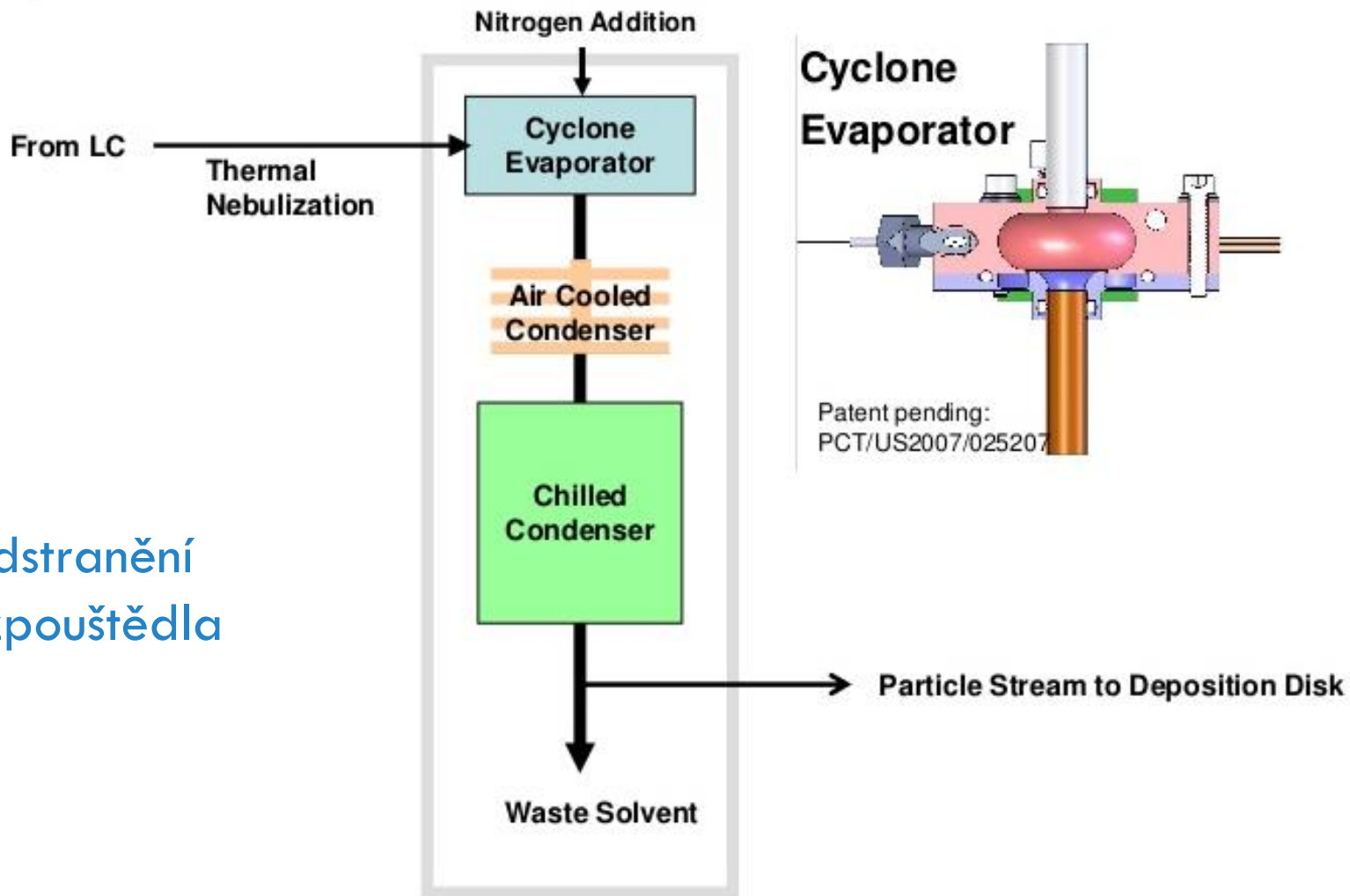


LC-FTIR SPOJENÍ

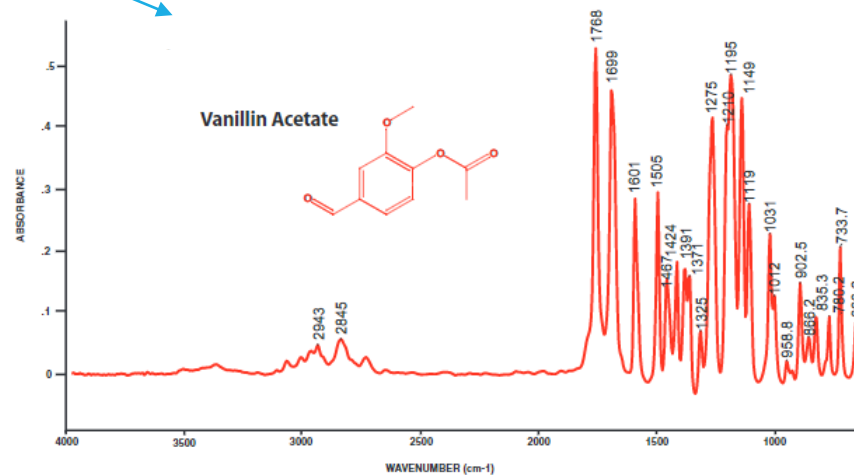
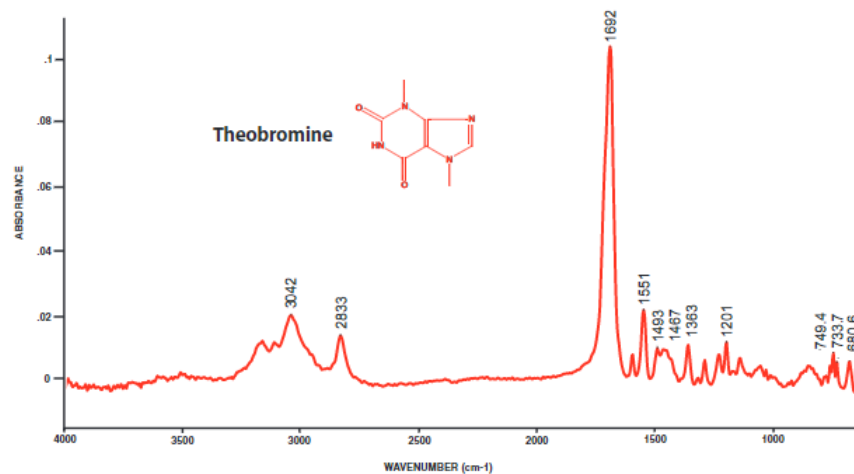
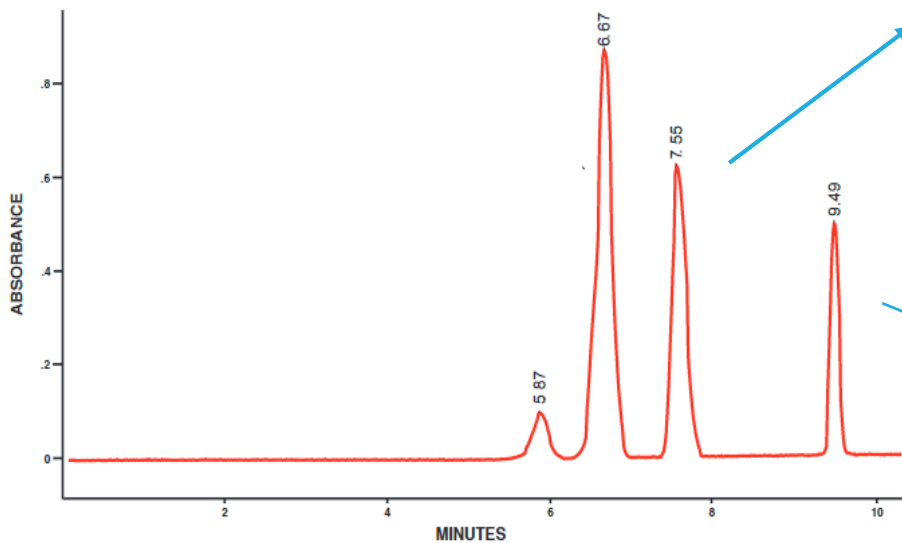


LC-FTIR

Odstranění
rozpuštědla

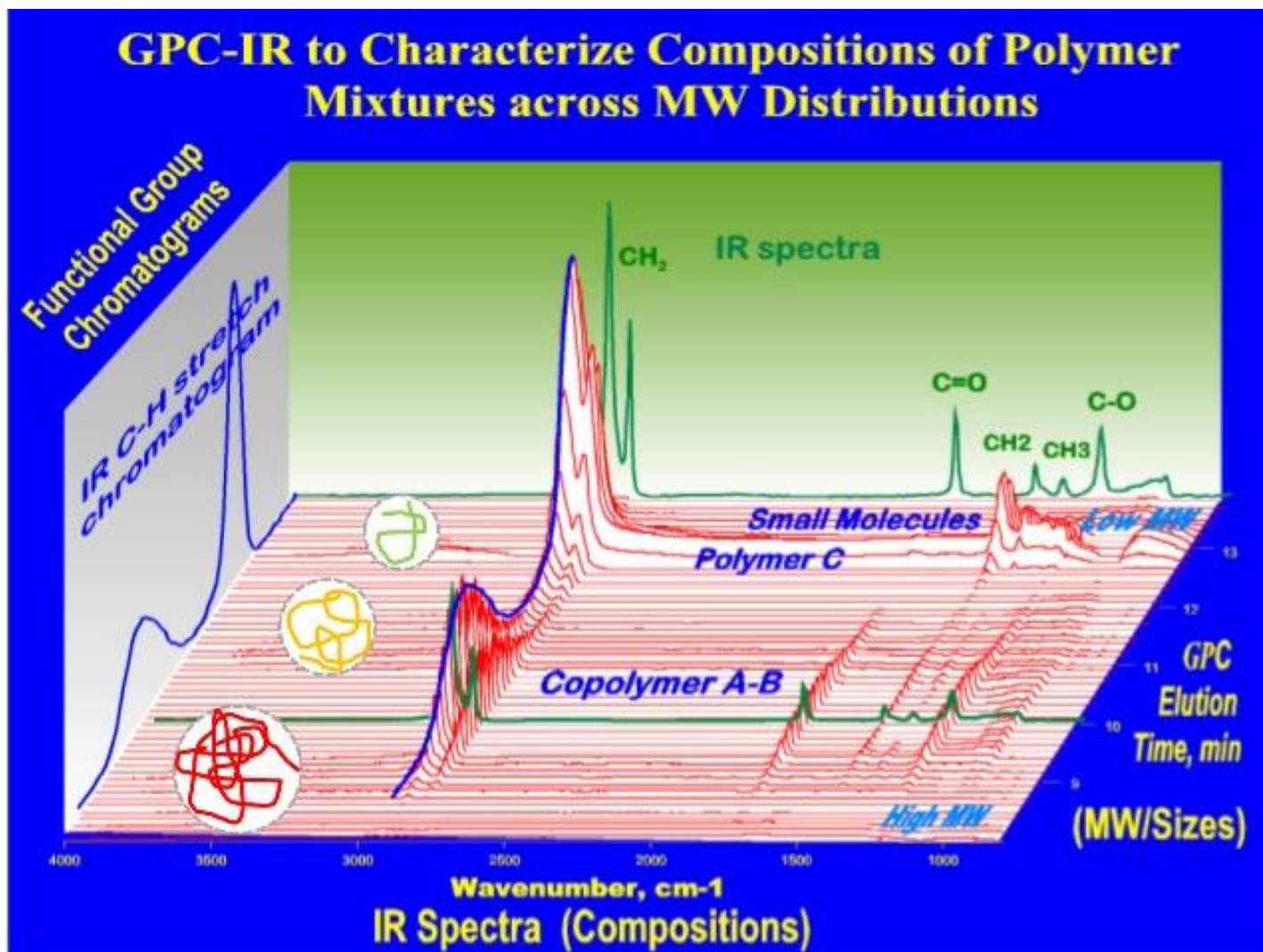


LC-FTIR

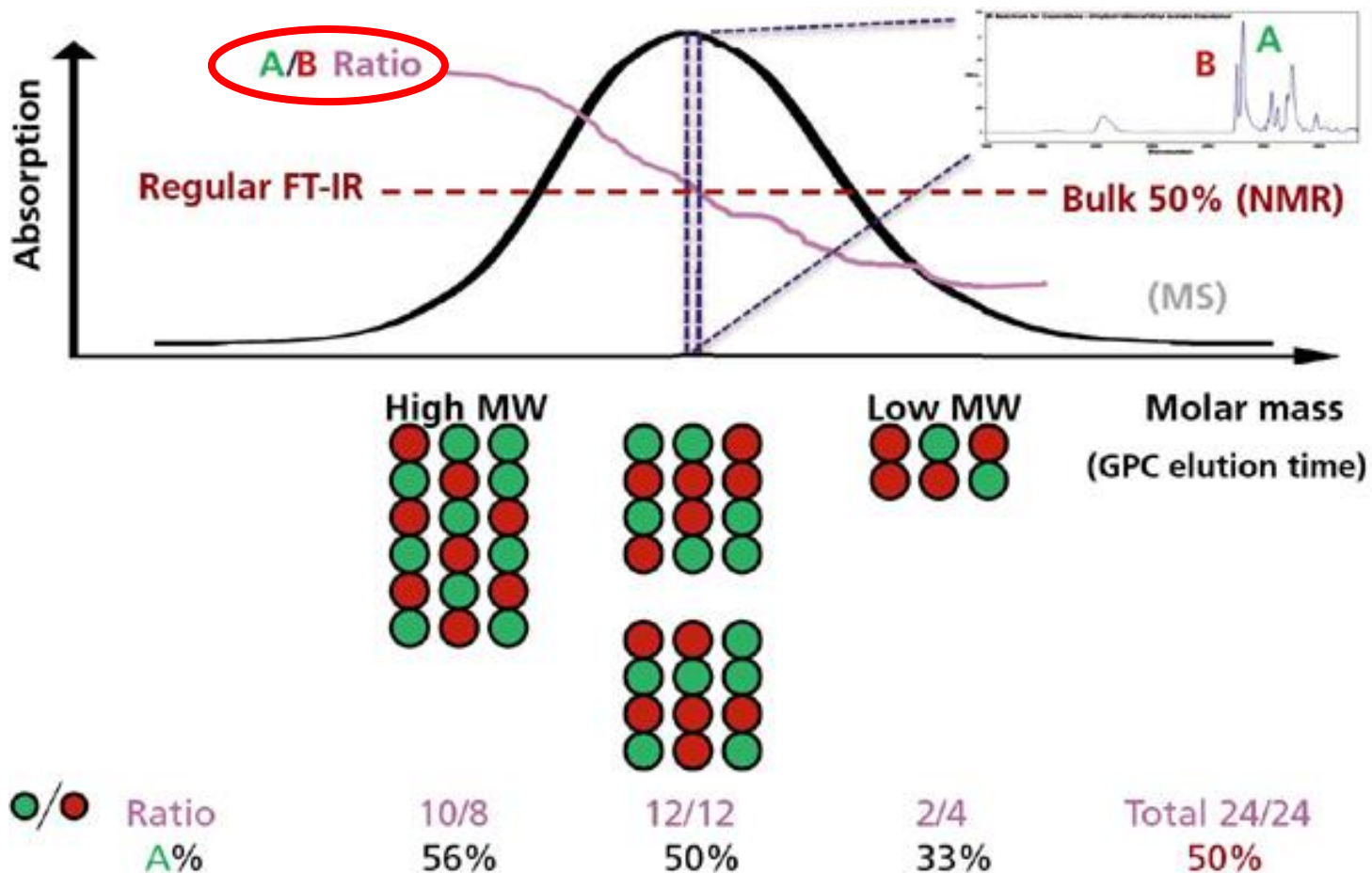


Chromatogram odpovídá maximálnímu IR signálu v průběhu času

LC-FTIR

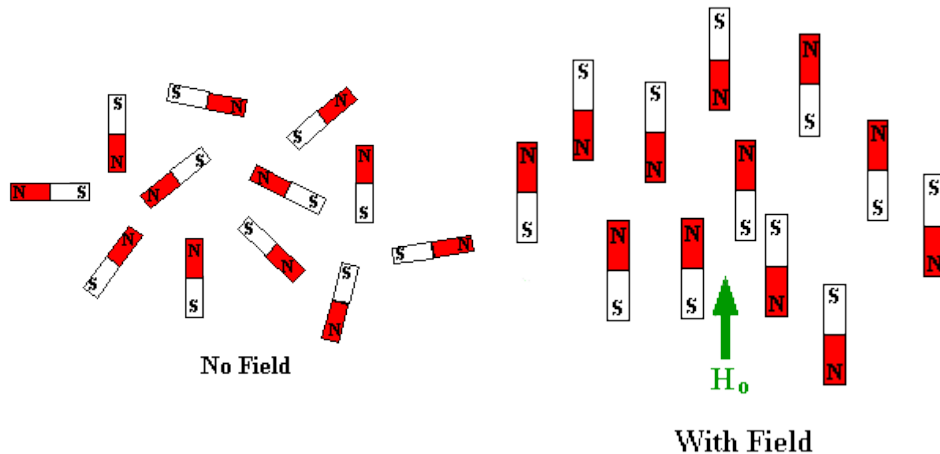
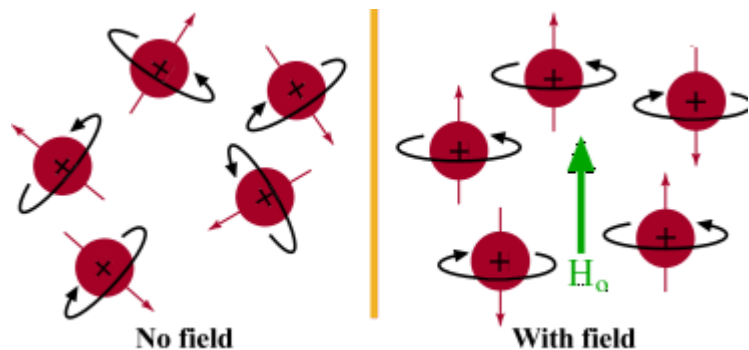


DISTRIBUCE KOPOLYMERU

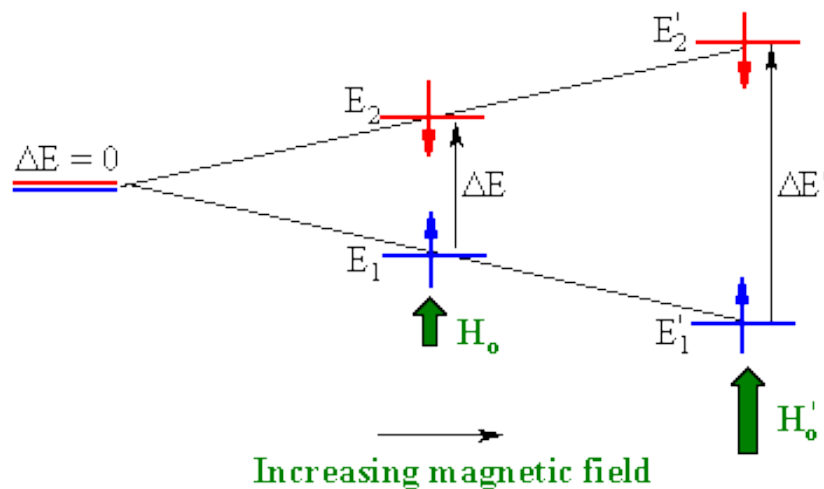
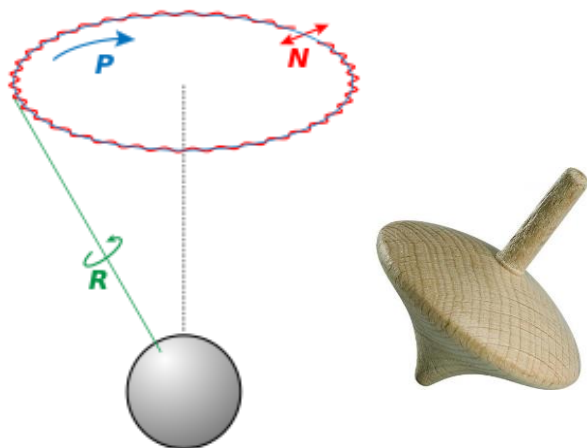


PRINCIP NMR

<http://www.mhhe.com/physsci/chemistry/carey/student/olc/ch13nmr.html>

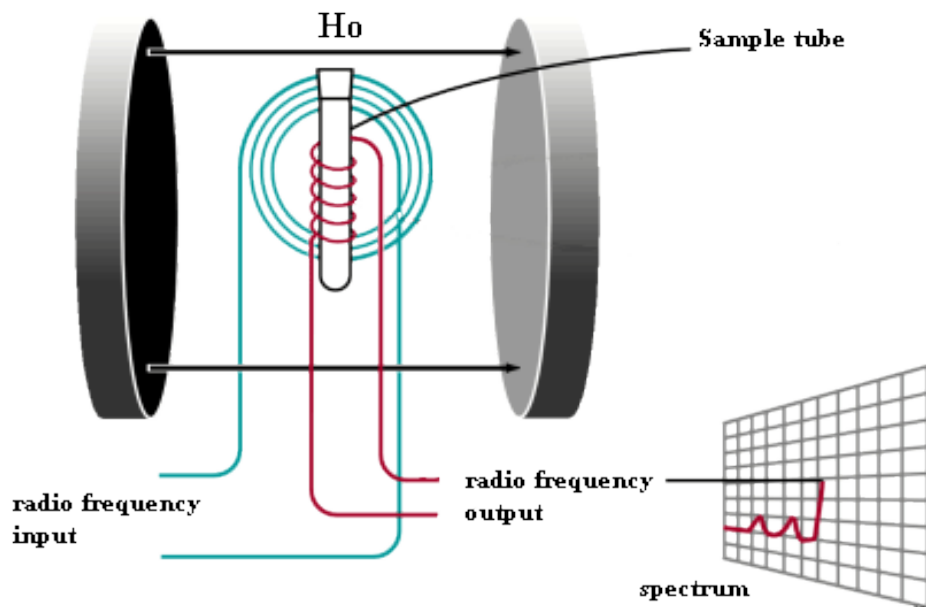


Precesní pohyb

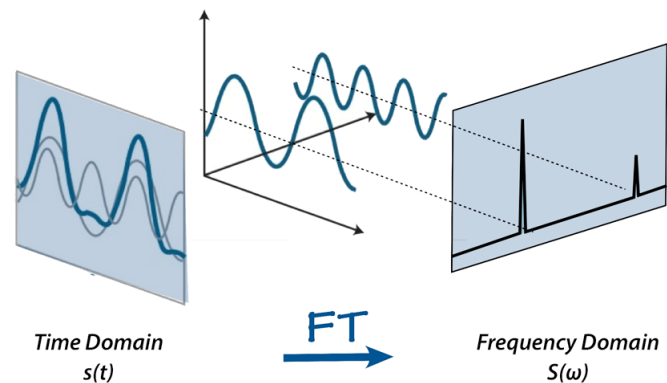
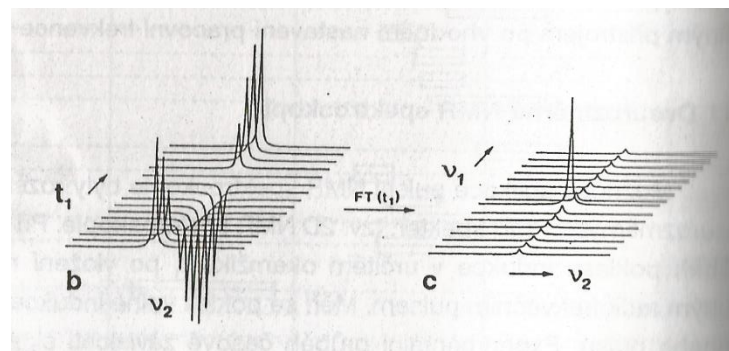


PRINCIP NMR

Experimentální uspořádání



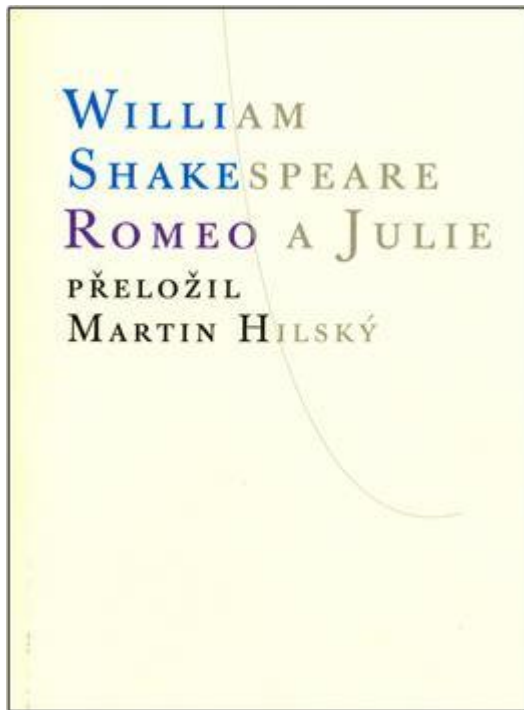
Fourierova transformace



NMR

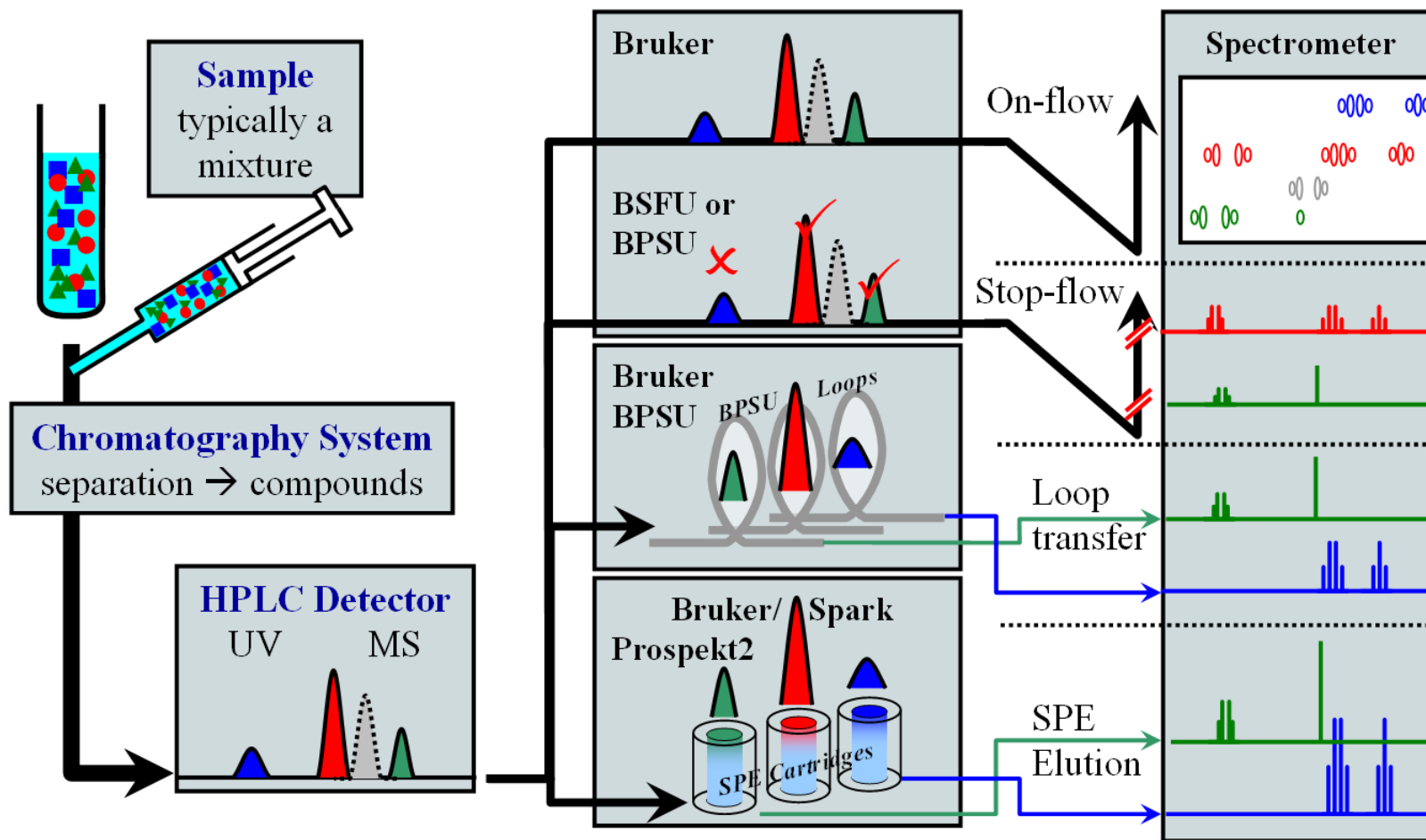


LC-NMR SPOJENÍ



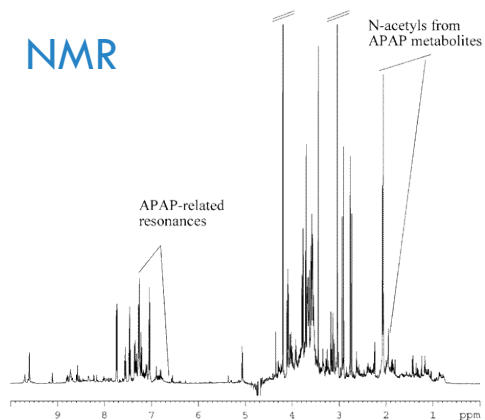
LC-NMR

Measurements - Schematic view



ON-FLOW LC-NMR

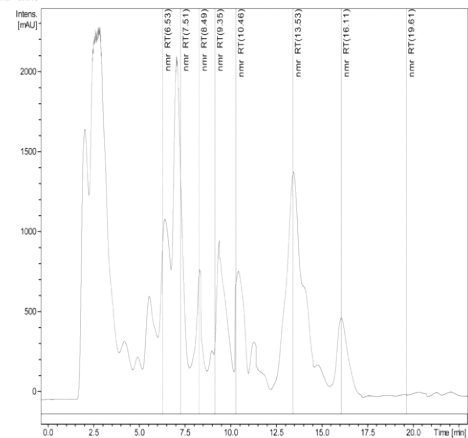
NMR



UV

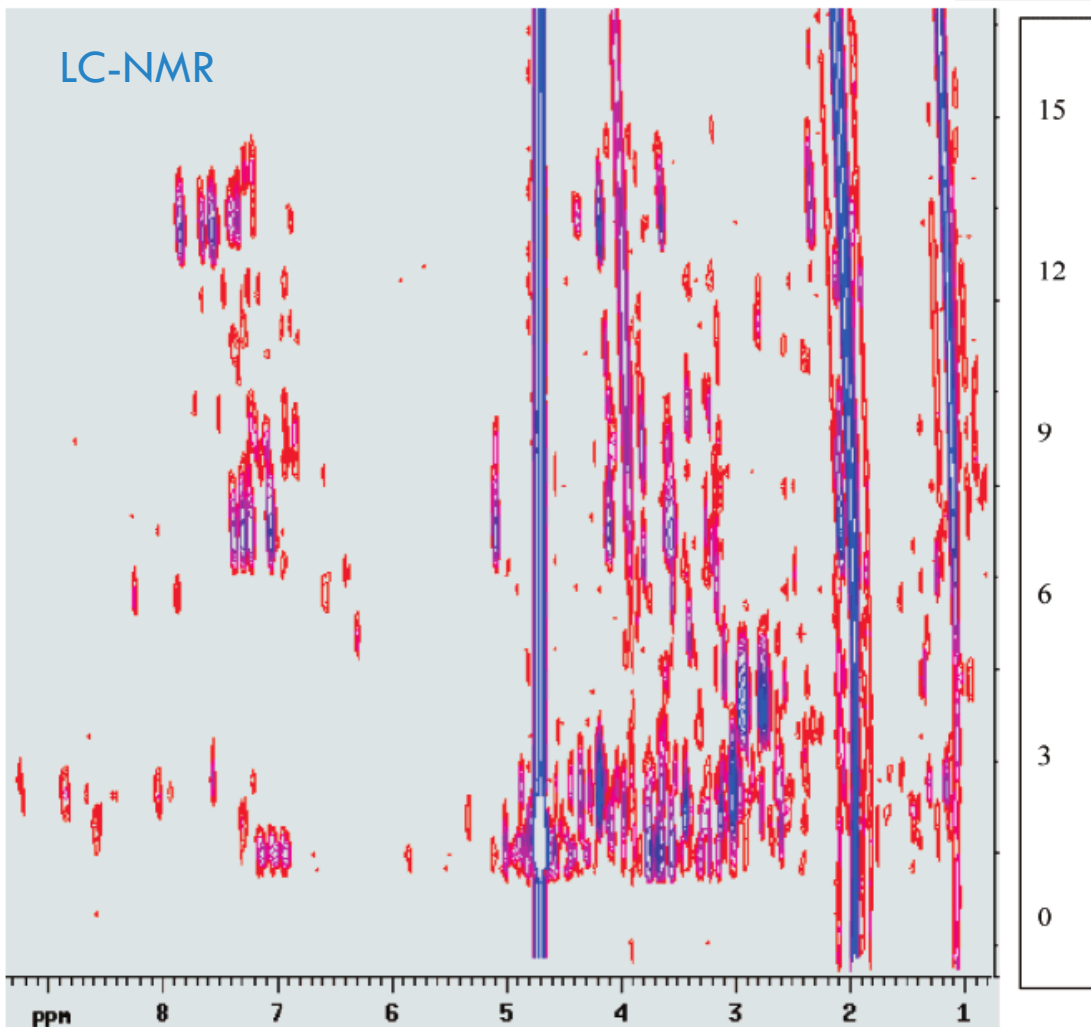
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DAD (210)



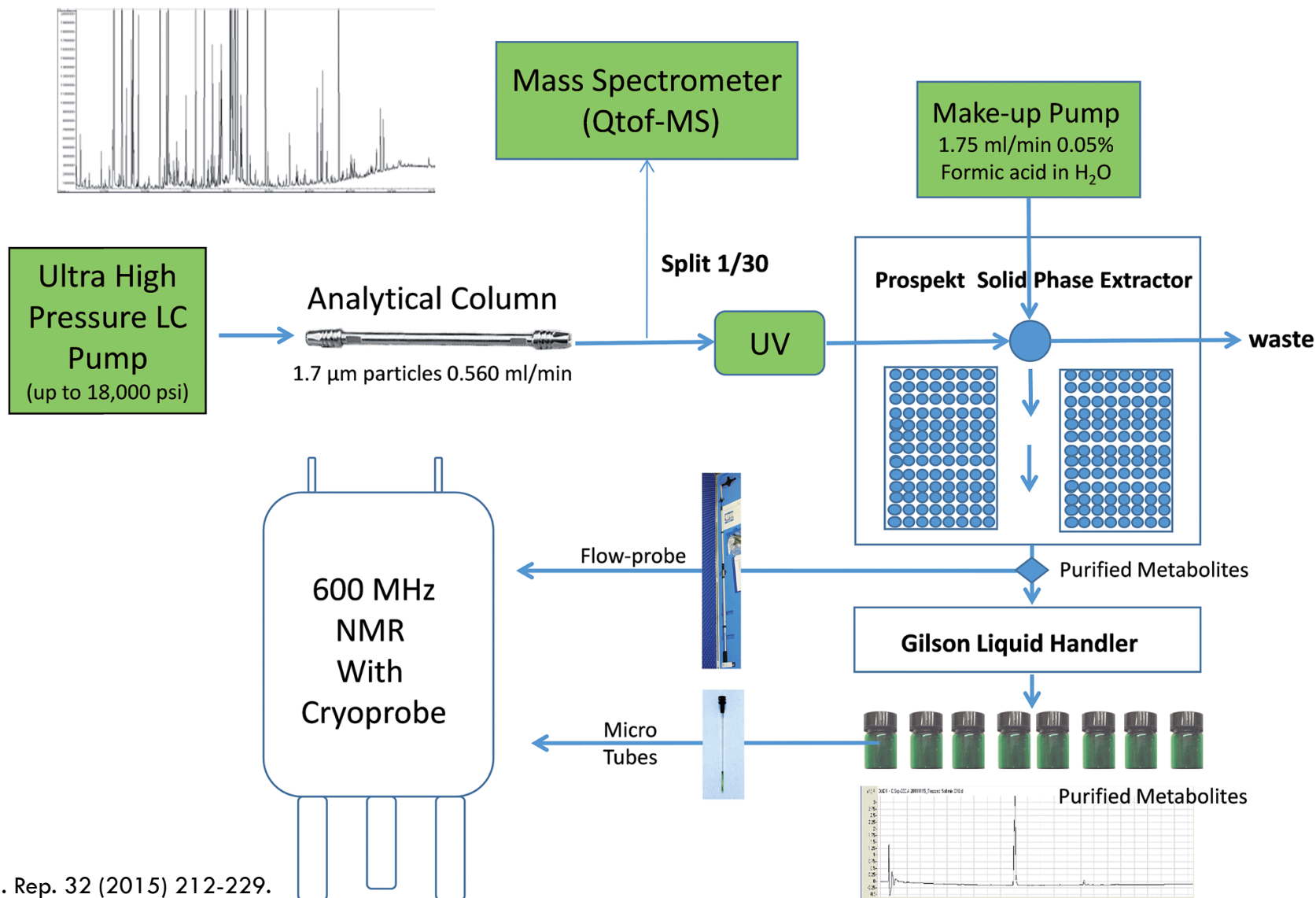
Tr (min)

LC-NMR

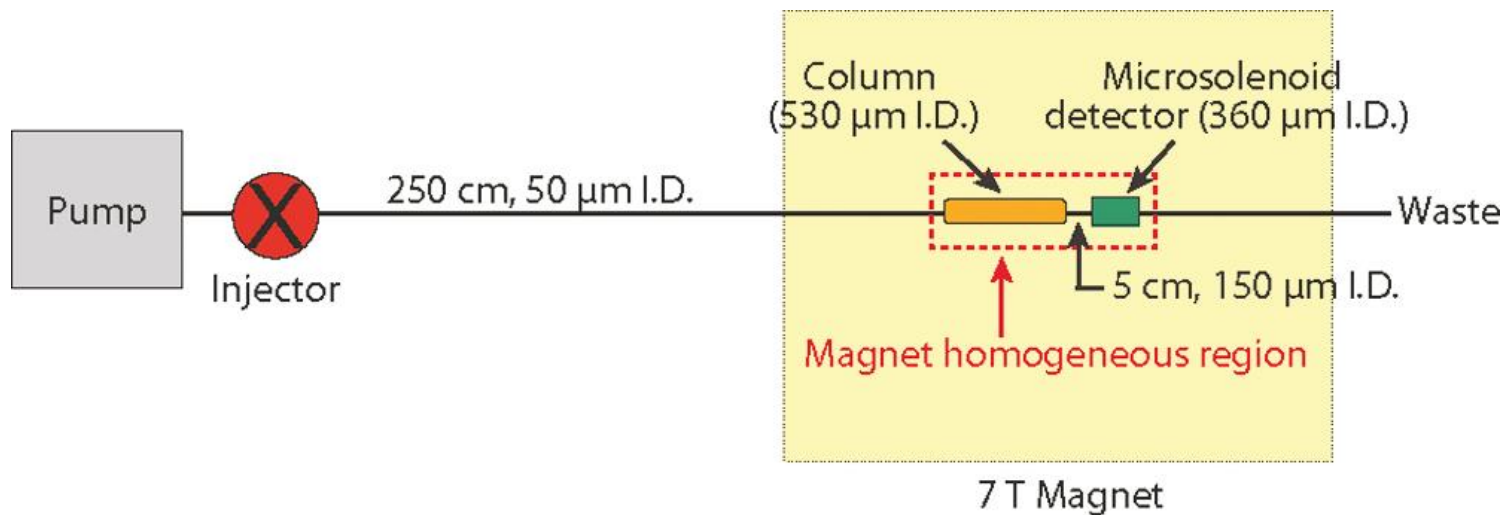
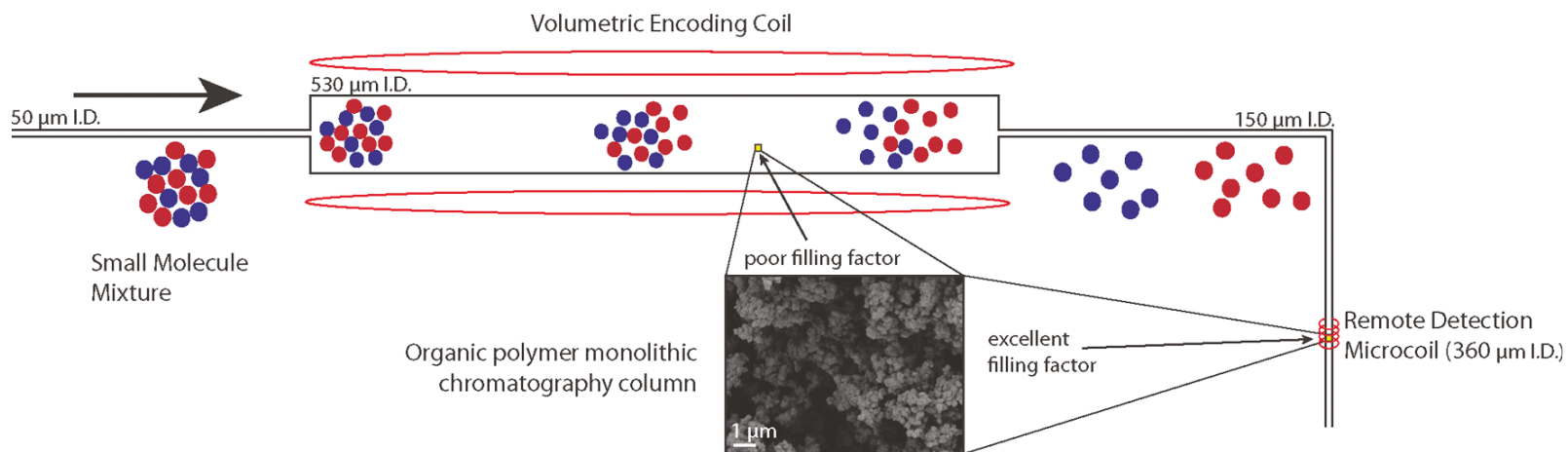


Moč

UHPLC-UV-MS-SPE-NMR

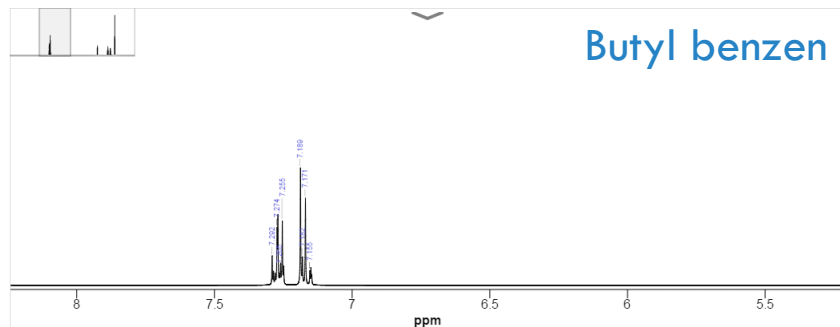
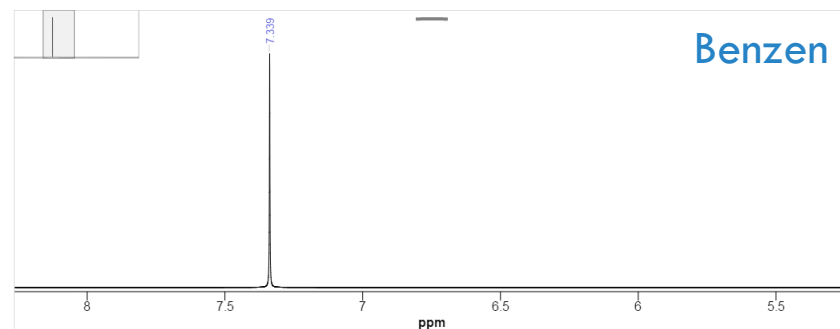
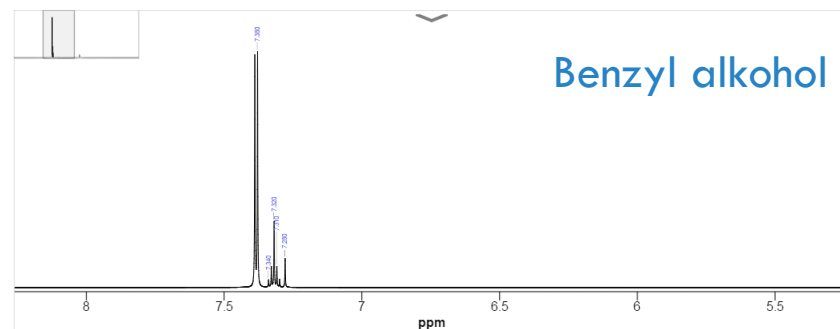
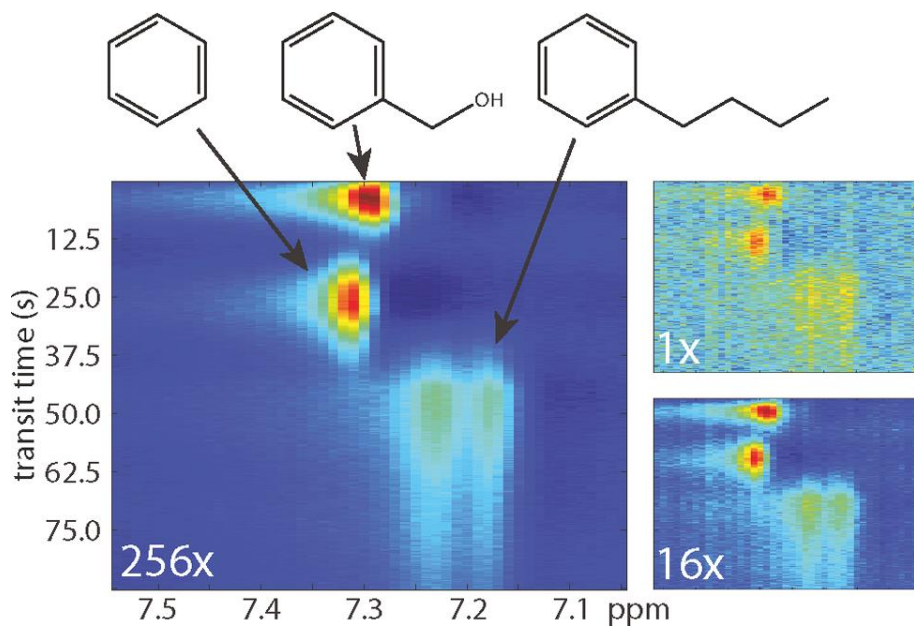


LC-NMR (REMOTE DETECTION)

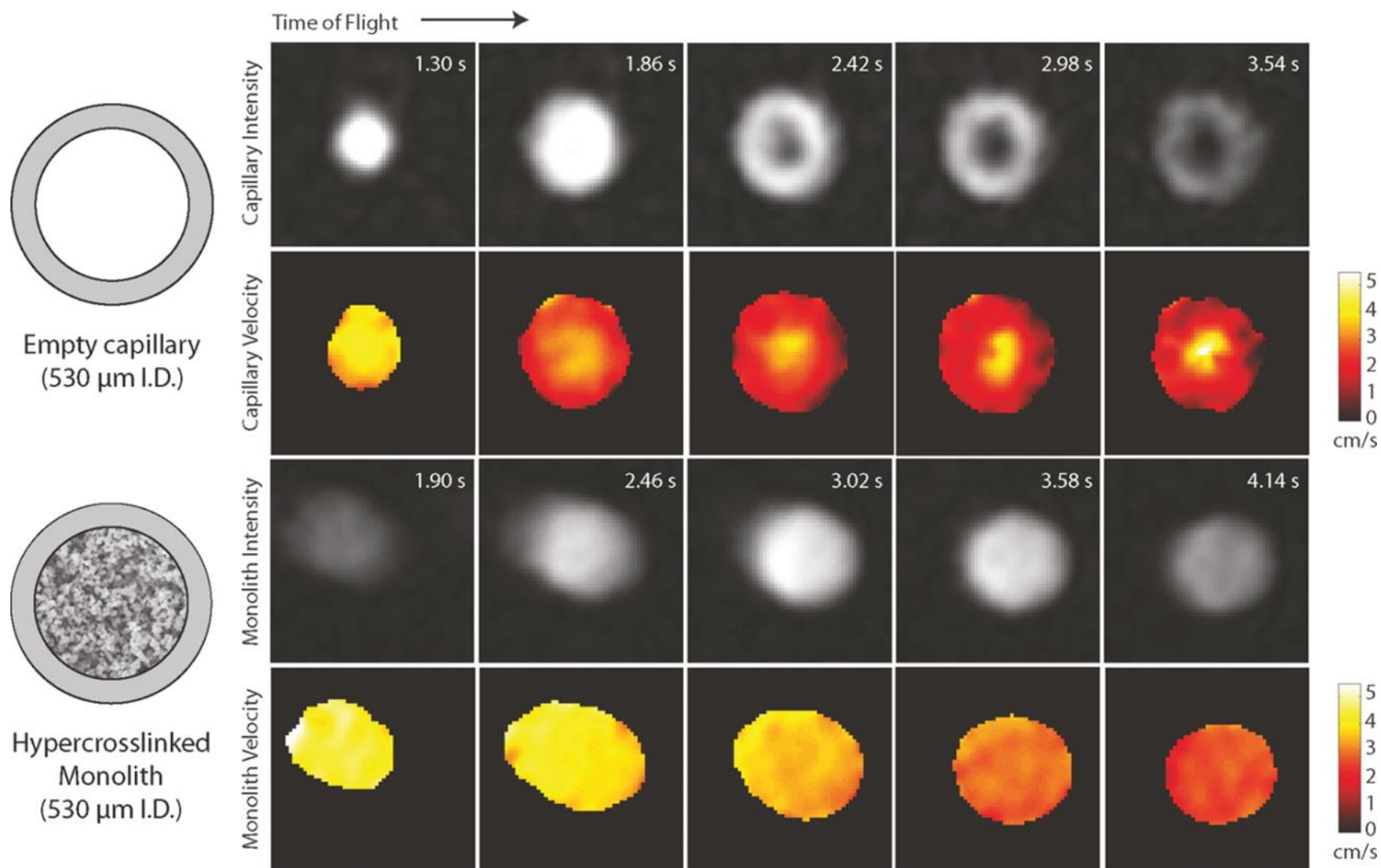


LC-NMR (REMOTE DETECTION)

Superpozice signálu



STUDIUM PROFILU TOKU MOBILNÍ FÁZE





*“Správně vidíme jen svým srdcem.
Co je důležité, je očím neviditelné.”*

Antoine de Saint-Exupéry