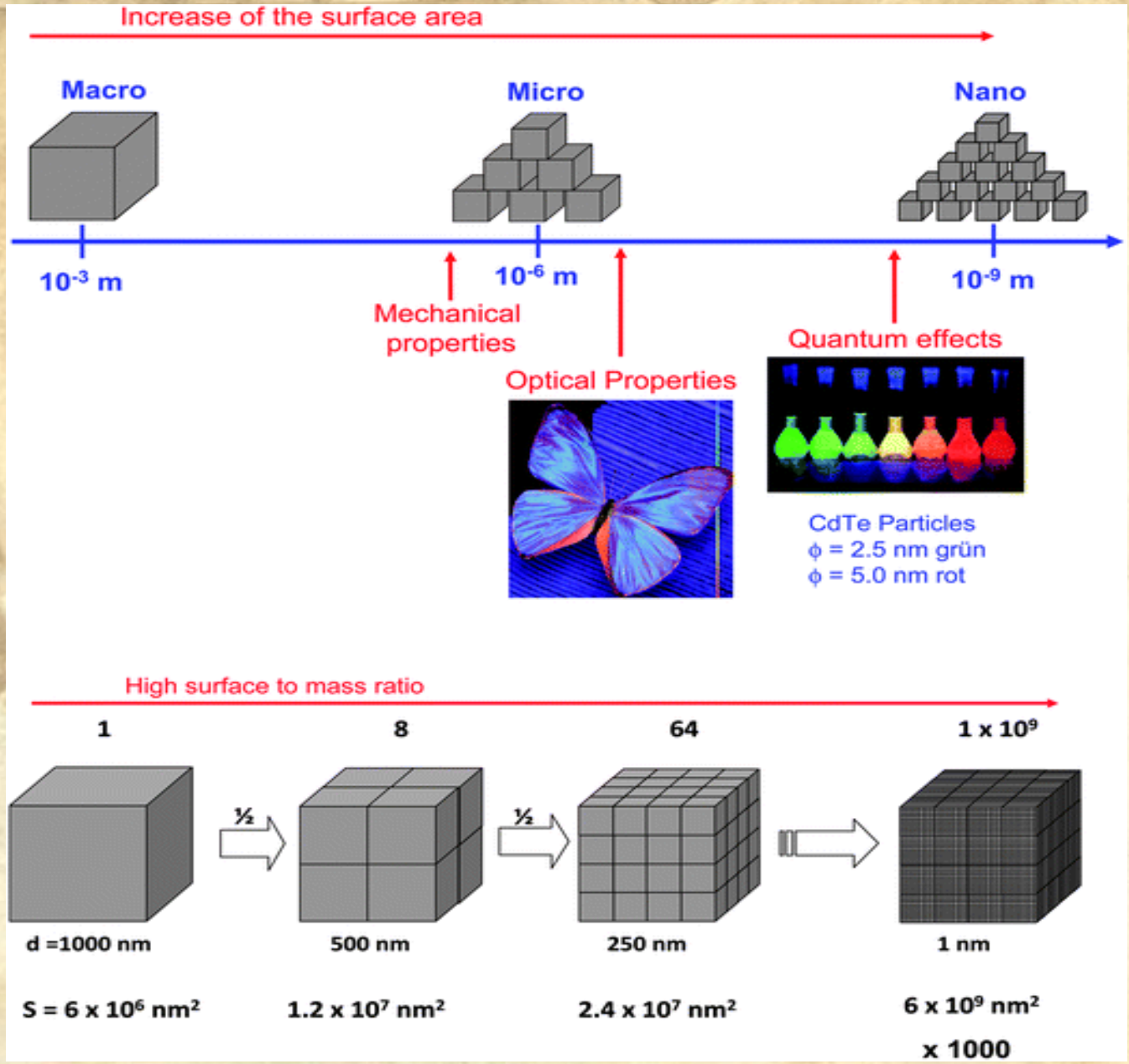
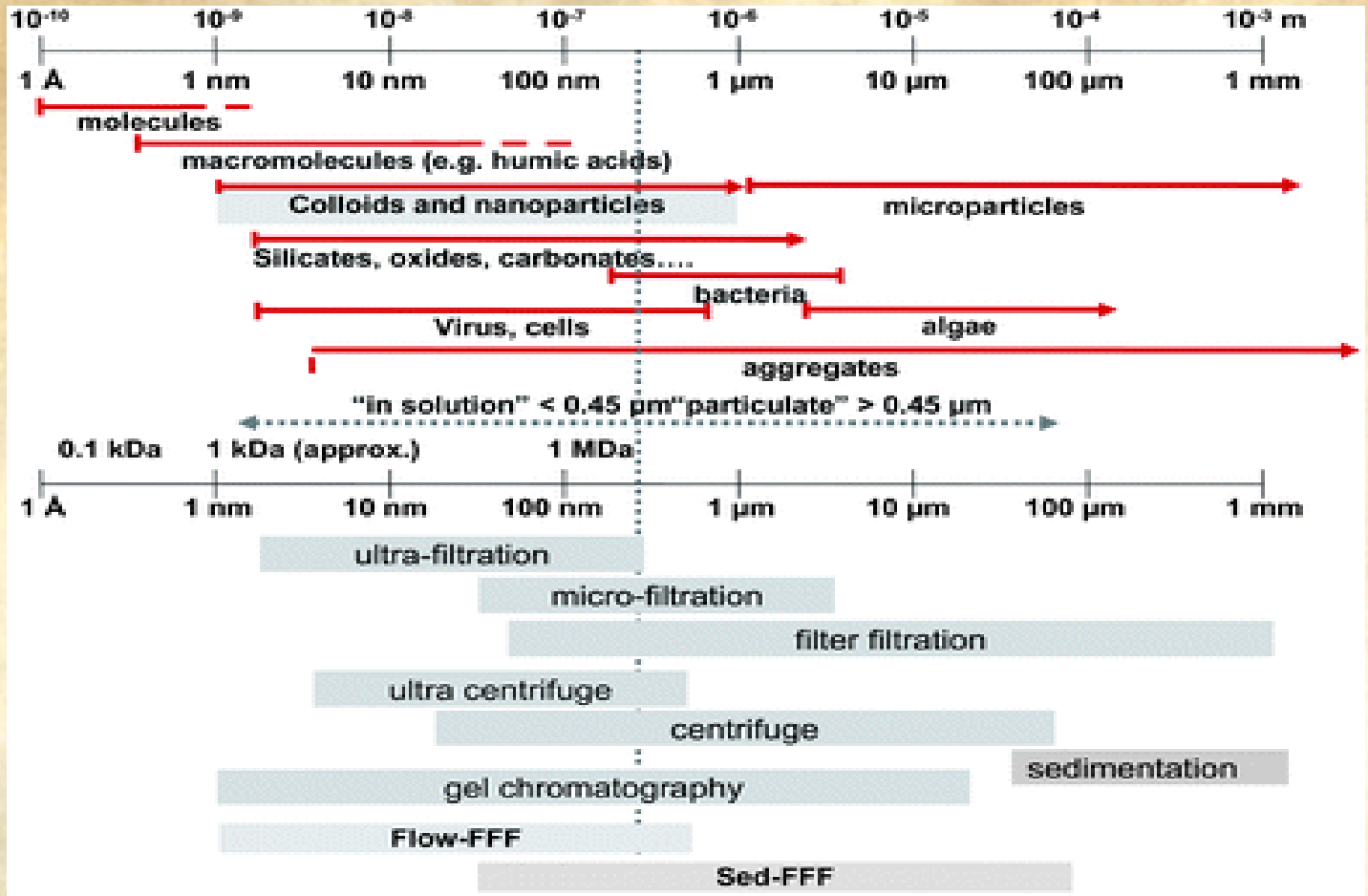
Nano komponensek analitikai mérése

Analitika= elválasztás, identifikálás, kvantifikálás

NÉBiH, 2012.V.22.

BUDAPESTI CORVINUS EGYETEM
Fodor Péter


Increase of the surface area
Macro
Micro
Nano
10-3 m
10-6 m
10-9 m
Mechanical properties
Optical Properties
Quantum effects
CdTe Particles
ϕ = 2.5 nm grün
ϕ = 5.0 nm rot
High surface to mass ratio
1
8
64
1 x 10⁹
½
½
d =1000 nm
500 nm
250 nm
1 nm
S = 6 x 10⁶ nm²
1.2 x 10⁷ nm²
2.4 x 10⁷ nm²
6 x 10⁹ nm²
x 1000


10-10
10-9
10-8
10-7
10-6
10-5
10-4
10-3 m
1 Å
1 nm
10 nm
100 nm
1 µm
10 µm
100 µm
1 mm
molecules
macromolecules (e.g. humic acids)
Colloids and nanoparticles
microparticles
Silicates, oxides, carbonates….
bacteria
Virus, cells
algae
aggregates
"in solution" < 0.45 µm
"particulate" > 0.45 µm
0.1 kDa
1 kDa (approx.)
1 MDa
1 Å
1 nm
10 nm
100 nm
1 µm
10 µm
100 µm
1 mm
ultra-filtration
micro-filtration
filter filtration
ultra centrifuge
centrifuge
sedimentation
gel chromatography
Flow-FFF
Sed-FFF

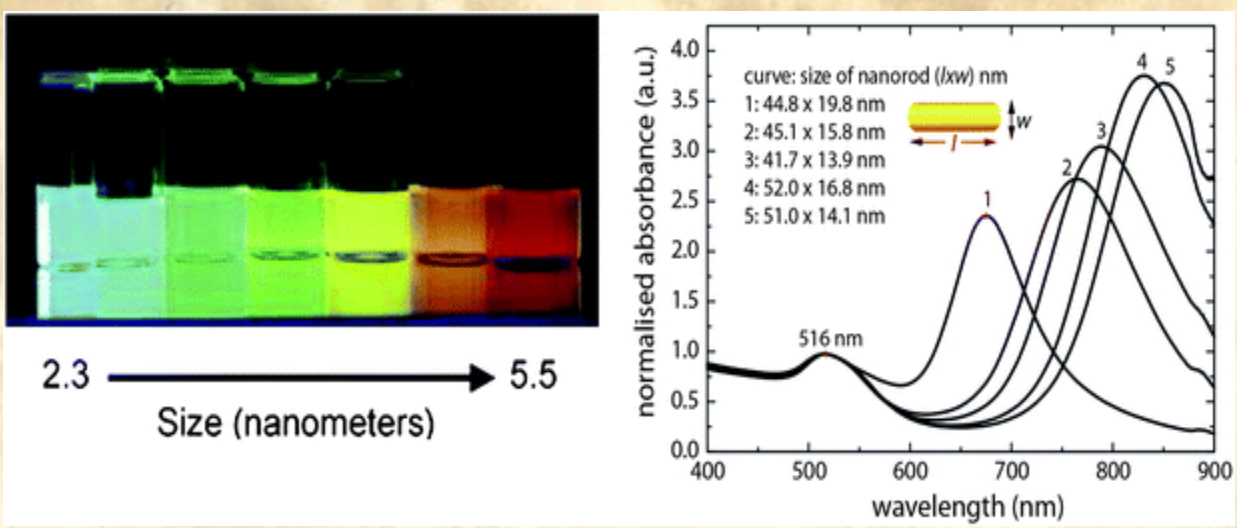


Fig. 3 (a) Fluorescence of CdSe–ZnS core–shell nanoparticles after excitation at 470 nm showing the characteristic red-shift with increasing sizes. Figure reprinted with permission. (b) Optical absorbance spectra of gold nanorods with increasingly red-shifted longitudinal plasmon bands with increasing aspect ratios. The sizes of the particles ascertained from transmission electron microscopy (TWM), responsible for the spectra 1–5 respectively, are indicated.

M.E.Schimpf, K.D.Caldwell and J.C.Gidding: Field Flow Fractination Handbook, Wiley Interscience, 2000.

Térerő által létrehozott Áramló minta Frakcionálása

Térerő:

Fl-FFF Flow Field Flow Fractination

Sd-FFF Sedimentation Field Flow Fractination

El-FFF Electrical Field Flow Fractination

Th-FFF Thermal Field Flow Fractination

Csak Fl-FFF és Sd-FFF –ICP-MS

Stephan Dubascoux, and c.w. : Field-flow fractionation and ICP-MS coupling: History, development and application

J.Anal.At.Spectrom., 2010, 25, 613. 63 references

Petra Krystek and c.w. :Application of plasma spectrometry for the analysis of engineered nanoparticles in suspension and products

J.Anal.At. Spectrom., 2011, 26, 1701. 344 references

Frank von de Kammer and c.w.: Separation and characteriazation of nanoparticles in complex food and environmental samples by field flow fractionation

Trends in Anal. Chemistry, 2011, 30, 425. 63 references, 3 food!!!

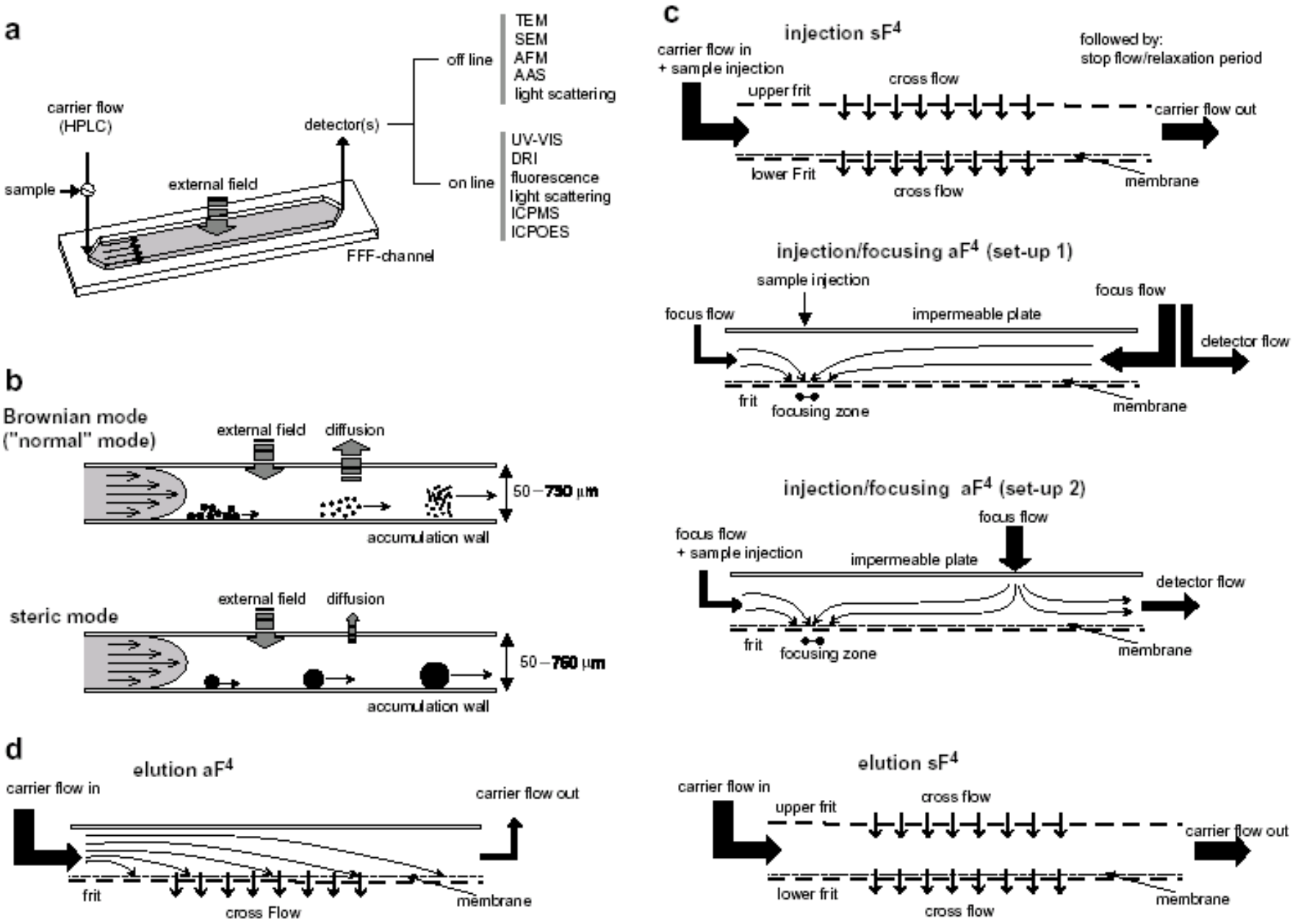
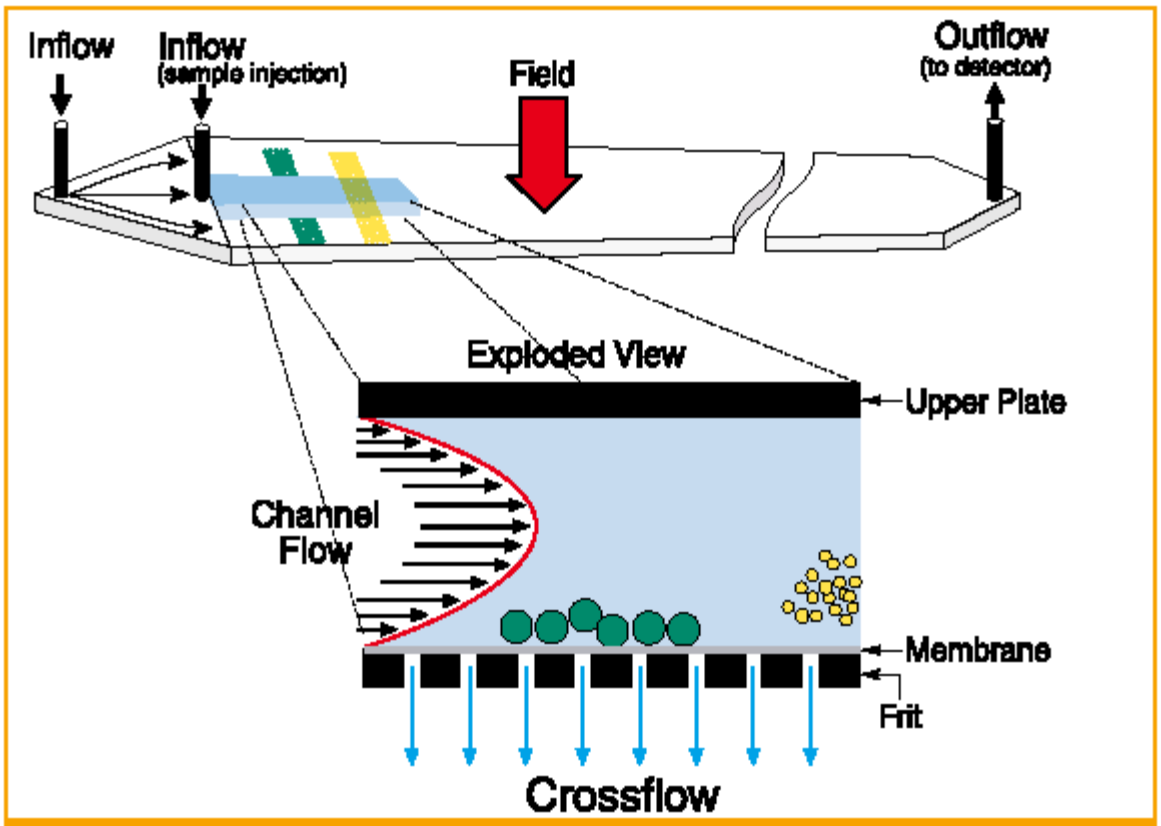


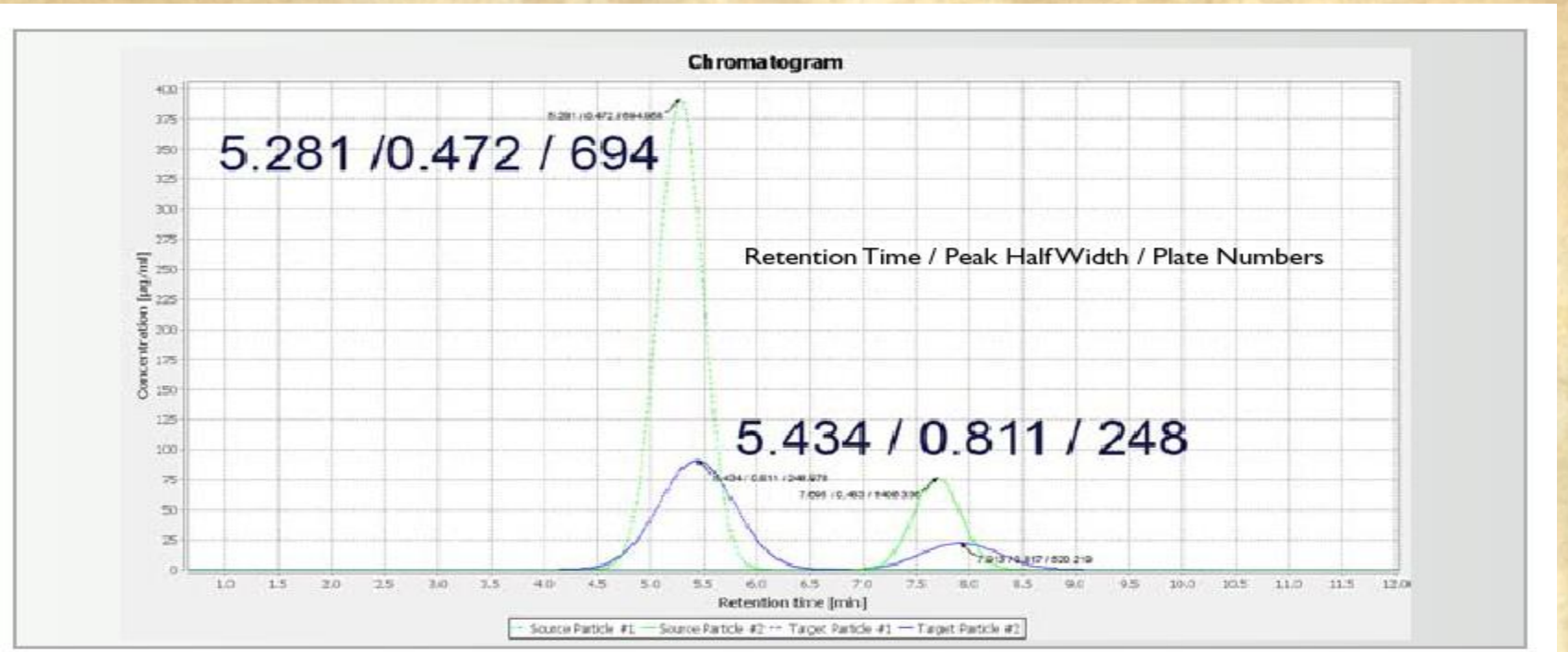
Figure 1. (a) Typical field-flow fractionation (FFF) lay-out with examples of different detectors; (b) lateral cross sections through the FFF channel, presenting the principle of particle-size fractionation for the "normal" Brownian and the steric elution modes; (c) lateral cross section through the FFF channel, describing the injection and focusing procedure for symmetric flow-FFF (sF^4) (top) and symmetric flow-FFF (aF^4) (middle and bottom). Two different set-ups are possible for sample injections in aF^4: independent injection in the focusing flow (middle) and injection with the inlet flow; and, (d) lateral cross section through the FFF channel, showing the elution procedure for sF^4 and aF^4.

Aszimetrikus Flow Filed Flow Fractination



Principle of AF4 separation: the smaller particles elute first as they diffuse further into the parabolic flow stream.

Detektorok: 18 szögben szóró lézer, refraktometriás detektor, dinamikus fényszórásos detektor (abszolút móltömeg és a hidrodinamikai átmérő mérésére)



Fraktogram –elválasztások paraméter (áramlási sebességek, sűrűségek, stb.) függése

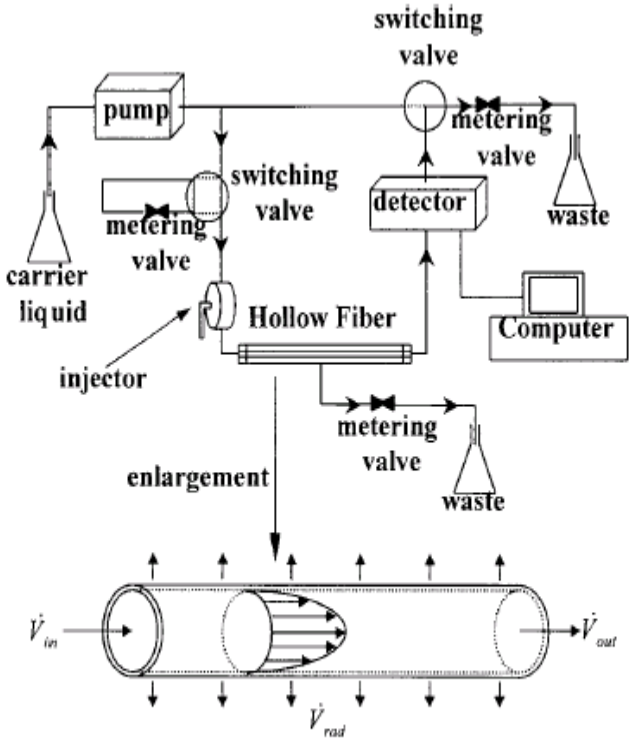


Figure 1. Schematic diagram of hollow fiber flow FFF (HF-FIFFF) system with the enlarged view of fiber representing the flow movement inside the fiber.

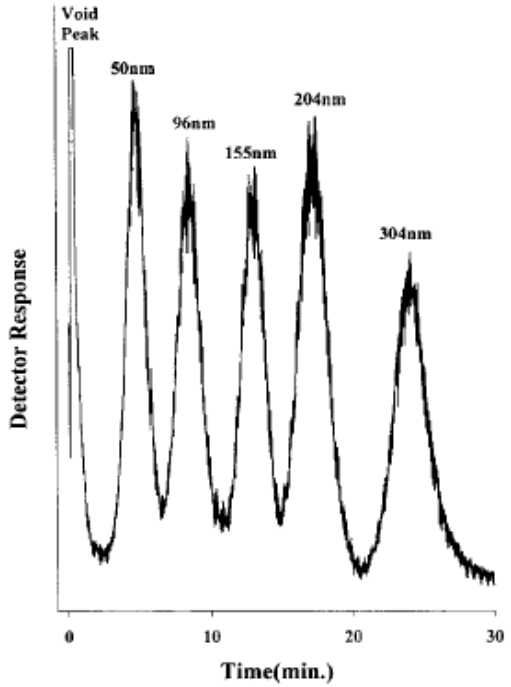
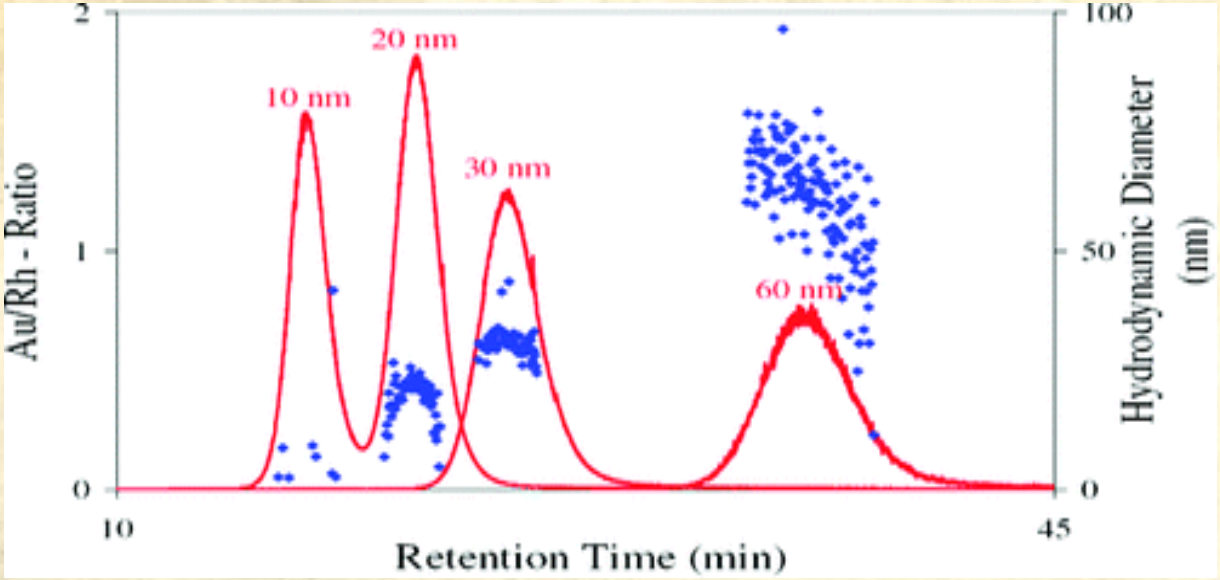


Figure 4. Separation of a polystyrene latex mixture by HF-FIFFF obtained at $\dot{V}_{out}/\dot{V}_{rad}$ = 1.41/0.12.

Quantitative Characterization of Gold Nanoparticles by Field-Flow Fractionation Coupled Online with Light Scattering Detection and Inductively Coupled Plasma Mass Spectrometry

Bjørn Schmidt†‡, Katrin Loeschner‡, Niels Hadrup‡, Alicja Mortensen‡, Jens J. Sloth‡, Christian Bender Koch†, and Erik H. Larsen*‡

‡ National Food Institute, Technical University of Denmark, *Anal. Chem.*, 2011, 83 (7), pp 2461–2468



Au eloszlás 3 Au CRM-ben, majd 10nm, 60nm és a keverék eloszlása patkány intravénás injekciózás után a májban, TMAH oldás.

ICP-MS analitikai problémák:

Higulás-elő-utó koncentrálás

Hordozó oldat kémia-fizika-mikrobiológia (pH-NH_4 NO_3, Na-azid)

Mátrix függőség

Mintaelőkészítés

Recovery!!!!!!!!!

Zavaró hatás –ütközési cella

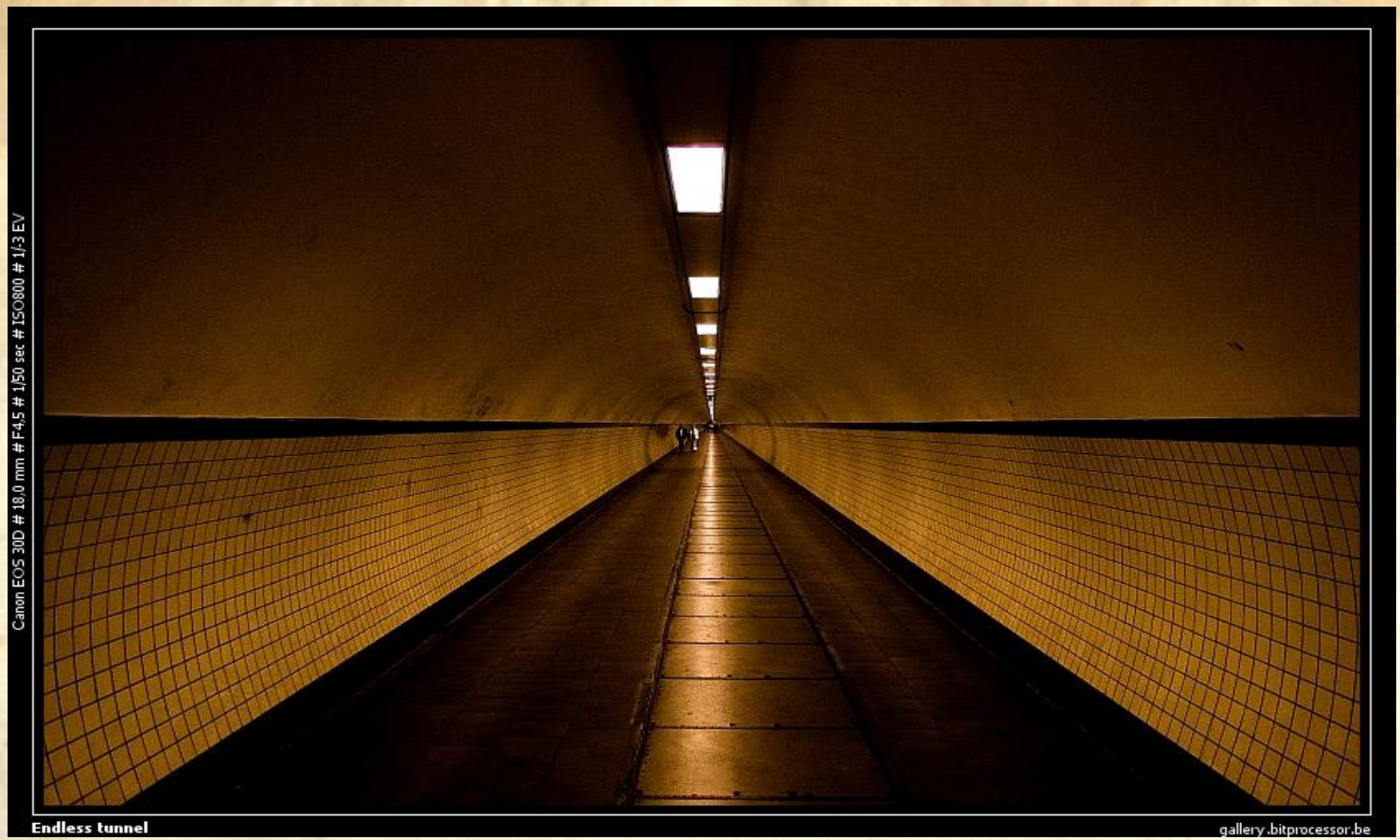
Matrix reference materials

- Silica nanoparticles in soup at two different mass fractions (Fig. 3)
- Silver nanoparticles in meat at two different mass fractions
- Cross-linked gelatine in fruit juice at two different mass fractions
- C_{60} fullerenes in vegetable oil at two different mass fractions

Fig. 3: Blank tomato soup (large jar, right) and soup spiked with silica nanoparticles

NanoLyse: The project

The NanoLyse project is a European collaborative research project which is partly funded by the European Commission under the 7th Framework Programme, contract no. 245162. It is dedicated to the development of analytical methods for detection and characterisation of engineered nanoparticles in food. The NanoLyse consortium comprises 10 universities and research centres from Europe and Canada and is coordinated by RIKILT - Institute of Food Safety (Wageningen UR). The project started in January 2010 and will last for 3 years.


Canon EOS 30D # 18.0 mm # F4.5 # 1/50 sec # ISO800 # 1/3 EV
Endless tunnel
gallery.bitprocessor.be