FFF as a fast and powerful Tool for high-end Characterization of Nano-scale Materials

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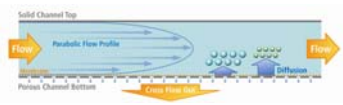
Introduction

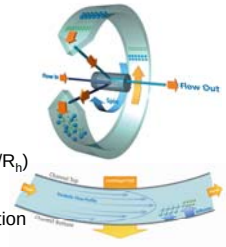
Today, Nano-Technology is one of the most increasing research topics in the world. An optimal knowledge about size, structure and distribution of the nano-scale materials is essential for the application and further development. So far, the necessary analytical techniques could not keep up with the rapid growth of Nano Science. The most methods for characterization of nanoparticles offer no separation or an immense analysis time has to be taken into account. In the last years the Field-Flow Fractionation technology (FFF) was strongly improved. FFF is a flow based method comparable with liquid chromatography which offers the possibility to separate particles with nearly no restrictions in size. The separation is realized in an empty flow channel using an external physical separation field without any stationary phase inside. As a result FFF offers full recovery, maximal resolution and short analysis times. In this poster the performance of two FFF-sub techniques was tested with reference materials of known size. In addition various application examples are given for the successful separation different kinds of organic and inorganic particulates.

Asymmetric Flow FFF (AF4)



- Cross-Flow field for separation
- Particles are forced towards channel bottom (accumulation wall)
- Laminar flow with parabolic flow profile inside the channel
- Diffusion of particles leads to arrangement in layers of different flow velocity
  → Separation according to **Hydrodynamic Diameter** ($D \sim 1/R_h$)
- Cross-Flow gradient of any shape for „tailor made“ separation

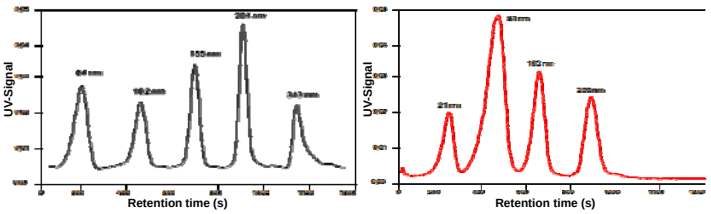
Centrifugal FFF (CF3)



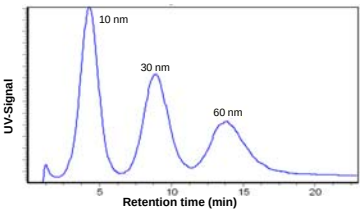
- Centrifugal field for separation (up to 4000 rpm)
- Centrifugal force pushes molecules towards channel bottom
- In addition: Diffusion of particles leads to arrangement in layers of different flow velocity
  → Separation according to **Hydrodynamic Diameter** ($D \sim 1/R_h$) AND according to **Differences in Density**
- Centrifugal gradient of any shape for „tailor made“ separation

Validation of Asymmetric Flow FFF (AF4)

Separation of different Mixtures of Latex Particles with defined Size



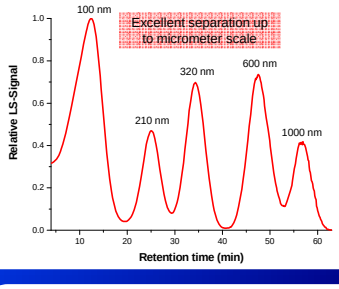
Separation of a Mixture of NIST Gold-Standards



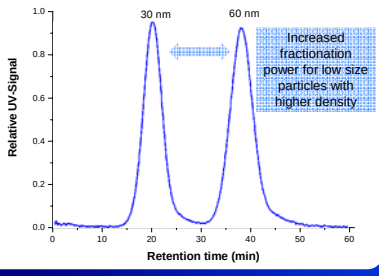
- These fractograms show impressively that AF4 is able to separate nanoparticles over a wide size-range with ultra-high resolution
- The free adjustable Cross-Flow gradient allows to modify the fractionation power
- The separation of different organic and inorganic particles demonstrates the universal applicability of the AF4-method

Validation of Centrifugal FFF (CF3)

Separation of Latex Particles with defined Size



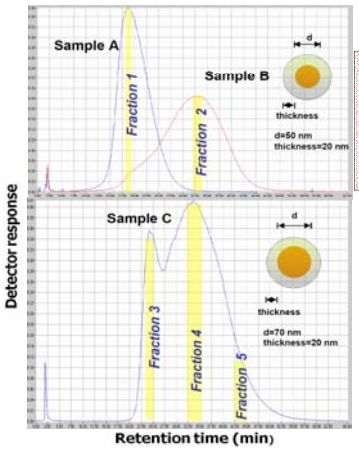
Separation of a Mixture of NIST Gold-Standards



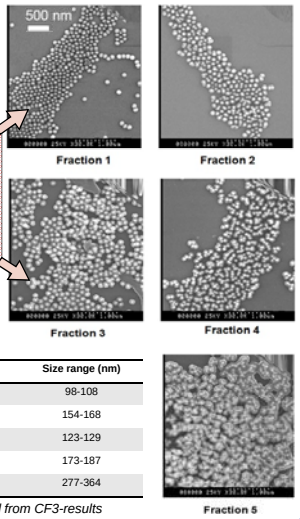
Application of CF3

Gold Nano-Particles with Silica Coating

- *Fractograms obtained by CF3 with UV detection*
- *Sample A is more mono-dispersed with the majority of the particles in the singlet form*
- *Sample B consists mostly of aggregates while sample C contains both*



Electron Microscopy confirms the results obtained by CF3!



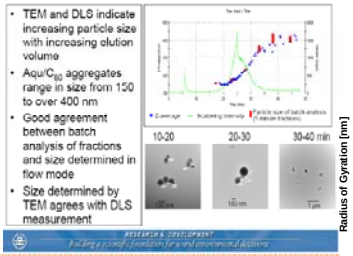
Fraction	Size range (nm)
1	98-108
2	154-168
3	123-129
4	173-187
5	277-364

Size calculated from CF3-results

Application of AF4

Aggregation of Fullerene C60

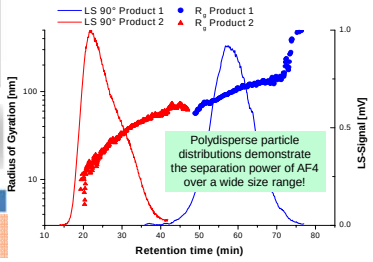
- TEM and DLS indicate increasing particle size with increasing elution volume
- Aqu'C_{60} aggregates range in size from 150 to over 400 nm
- Good agreement between batch analysis of fractions and size determined in flow mode
- Size determined by TEM agrees with DLS measurement



Two independent techniques confirm the optimal separation of the aggregates by AF4

Particles in Window Cleaner

- Nanoparticle coating on glass surface avoids permanent attachment of dust/dirt (Lotus-Effect)
- In this example the knowledge of the exact particle size is essential for the product characteristics and potential harms

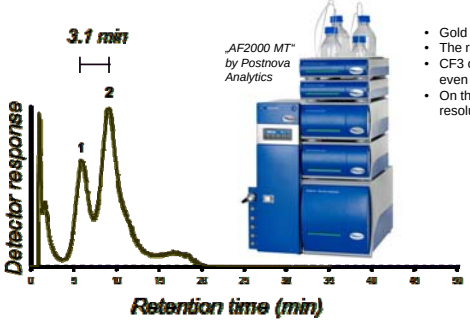


Comparison of AF4 and CF3

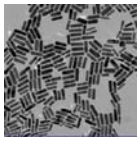
Separation of Gold Rods with different Shape

- Gold rods of different size were separated with both FFF methods and UV detection.
- The results show that both methods can be used for successful Gold particle separation.
- CF3 offers a better resolution for high density particles and the possibility to separate even particles of the same hydrodynamic diameter but different density.
- On the contrary AF4 separates strictly according to hydrodynamic size and offers better resolution for low density particles.

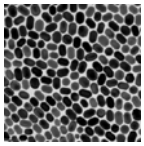
Asymmetric Flow FFF



„AF2000 MT“ by Postnova Analytics



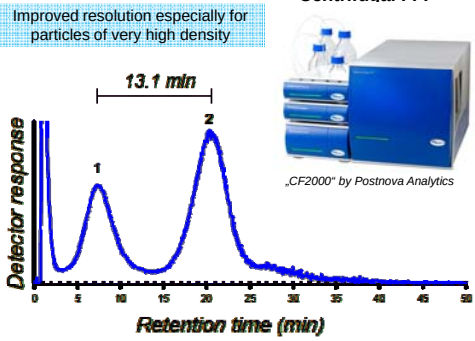
Peak 1 Axial = 10 nm Legth = 35 nm



Peak 2 Axial = 25 nm Legth = 35 nm

Improved resolution especially for particles of very high density

Centrifugal FFF



„CF2000“ by Postnova Analytics

Conclusions

The variety of different nano applications described in this presentation illustrate the universal applicability of FFF. With a single FFF channel sizes ranging from a few nanometers up to several microns can be characterized. The separation can be customized just by simple adjustment of the cross-flow or centrifugal gradient. It was demonstrated that AF4 offers a separation over a broad size-range strictly according to hydrodynamic size. Centrifugal FFF is especially capable for structures of higher density because of the increased resolution which is due to the additional density-influence on the retention. Moreover, this influence parameter allows to separate structures with similar hydrodynamic properties but different densities. It was shown that FFF offers a fast and simple characterization of organic and inorganic nano-scale materials with mono- or poly-disperse size-distribution. FFF is also able to separate polymer materials which is shown in a second poster. As a result it can be summarized that FFF is powerful and universal separation method for nano-materials and polymers, which surpasses the numerous limitations of older traditional methods, such as Microscopy, Chromatography or batch Static and Dynamic Light scattering.