
Your Partner in Polymer Analytics



AK Polymeranalytik 22.3.2019

Dr. Robert Brüll
Fraunhofer-Institute for Structural Durability and System Reliability LBF
www.lbf.fraunhofer.de

Process↔Structure↔Property Relationships



rezeipe

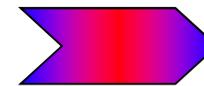


process

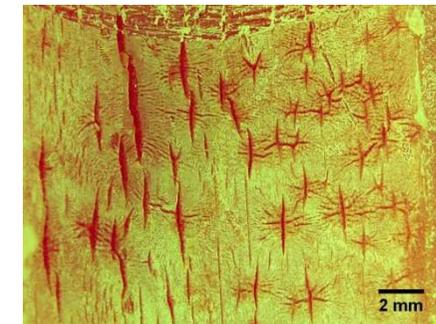
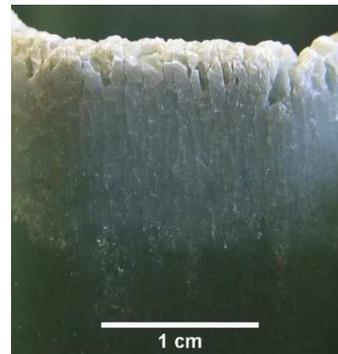


mechanical
properties

long term
properties



Failure



Seite 2

Focus areas: By technique

Separations

- Chromatography (LC)
- Field Flow Fractionation (FFF)
- CRYSTAF, TREF, CEF

Thermal

- DSC
- TGA
- Pyrolysis GC-MS

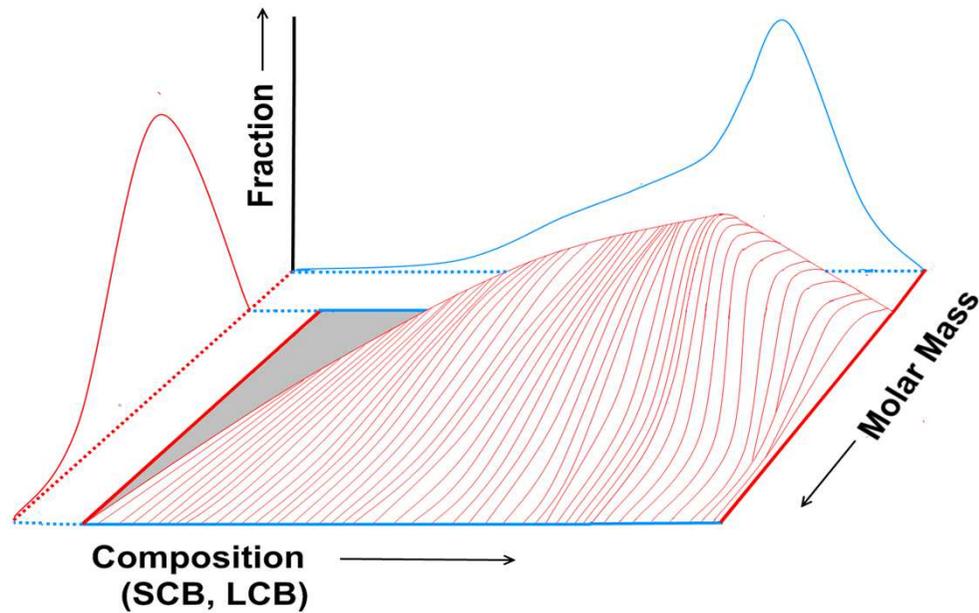
Spectroscopy

- Infrared
- Raman
- NMR

Imaging

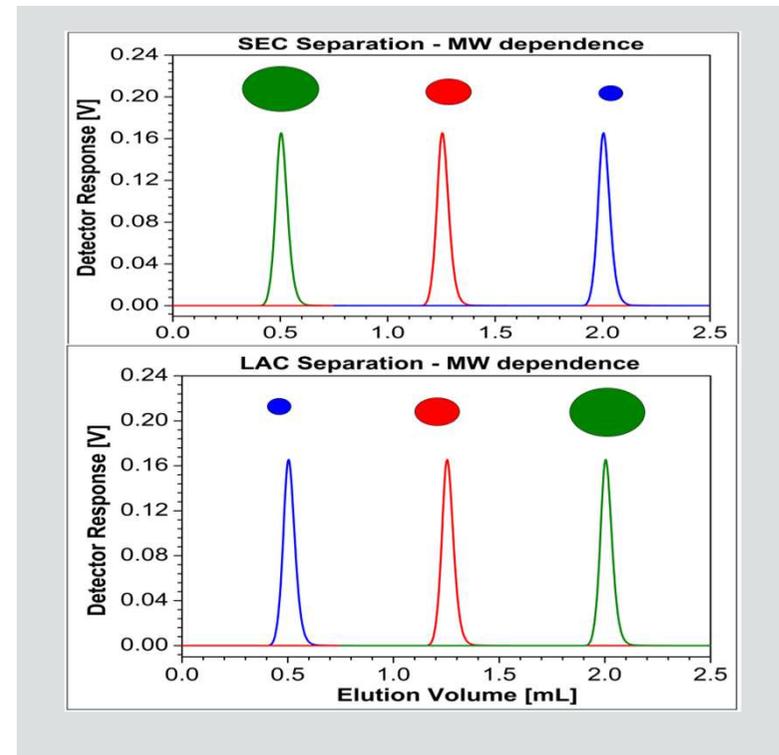
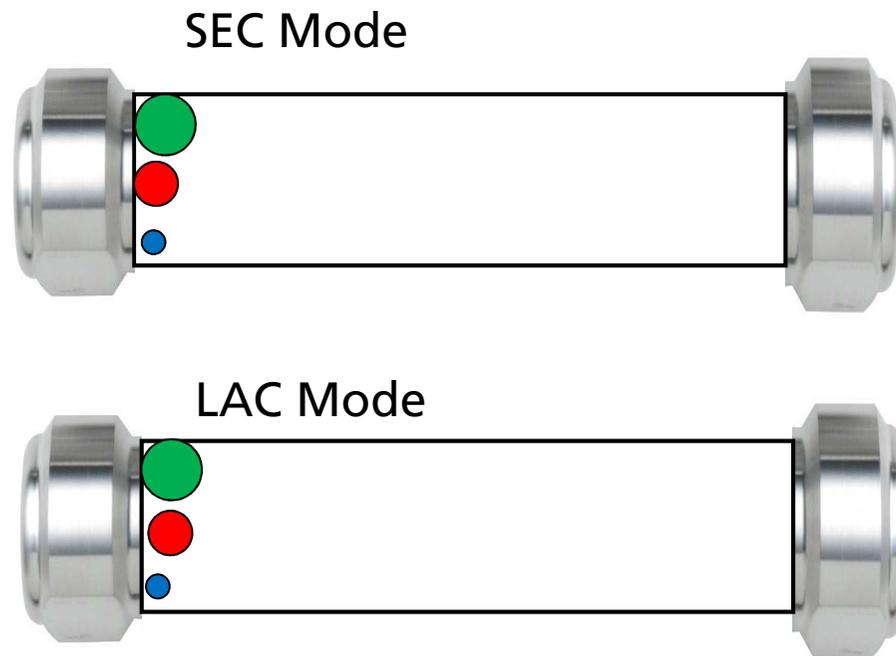
- Infrared-microscopy
- Raman-microscopy
- Polarized light microscopy

Separation techniques



- Multidimensional Techniques are essential to analyze the chemical heterogeneity

Separation modes in liquid chromatography



Multidimensional HT 2D-LC



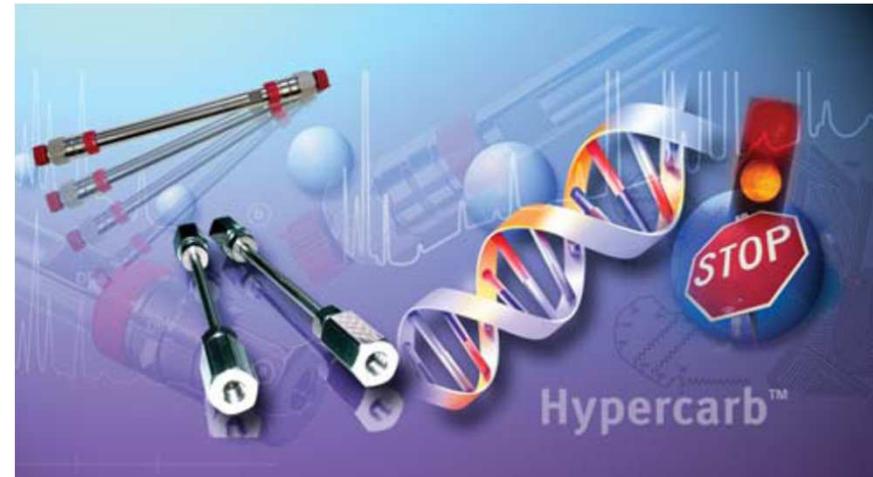
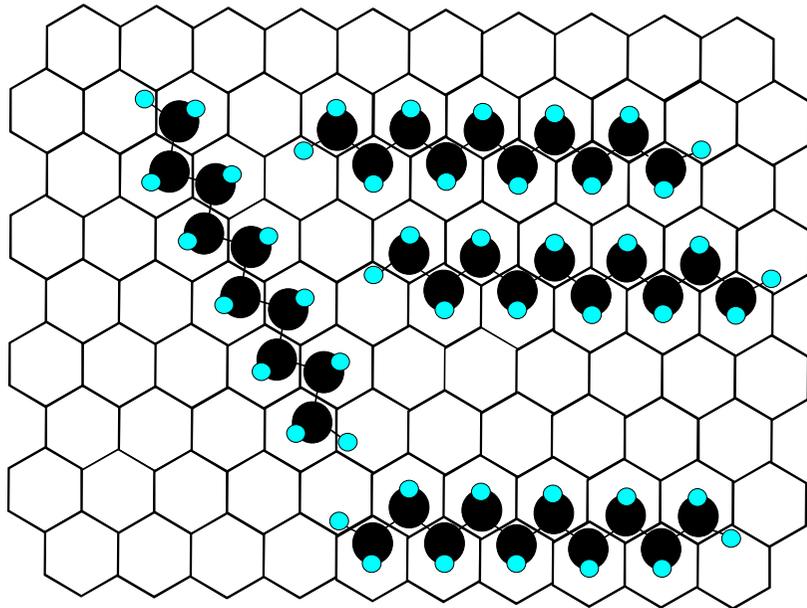
Sample Preparation

**Heatable
Separation
unit(s)**

Detection

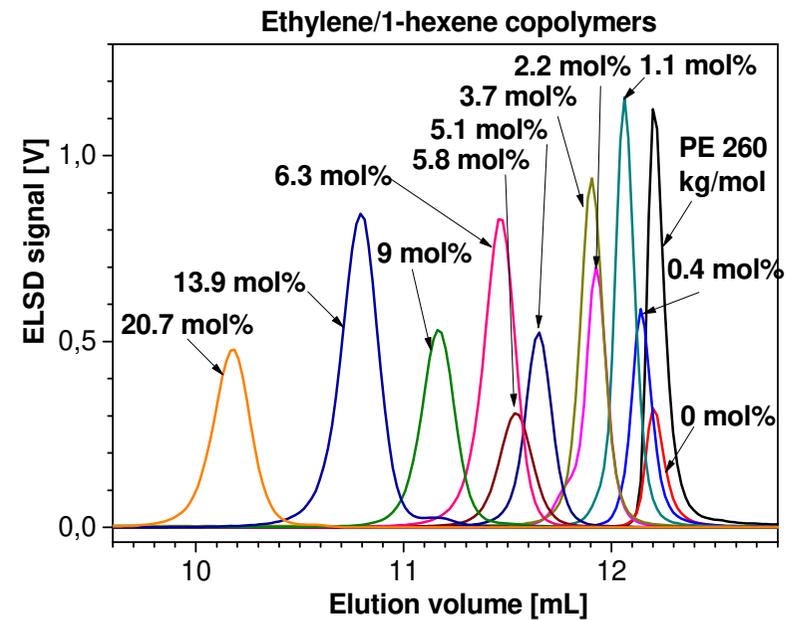
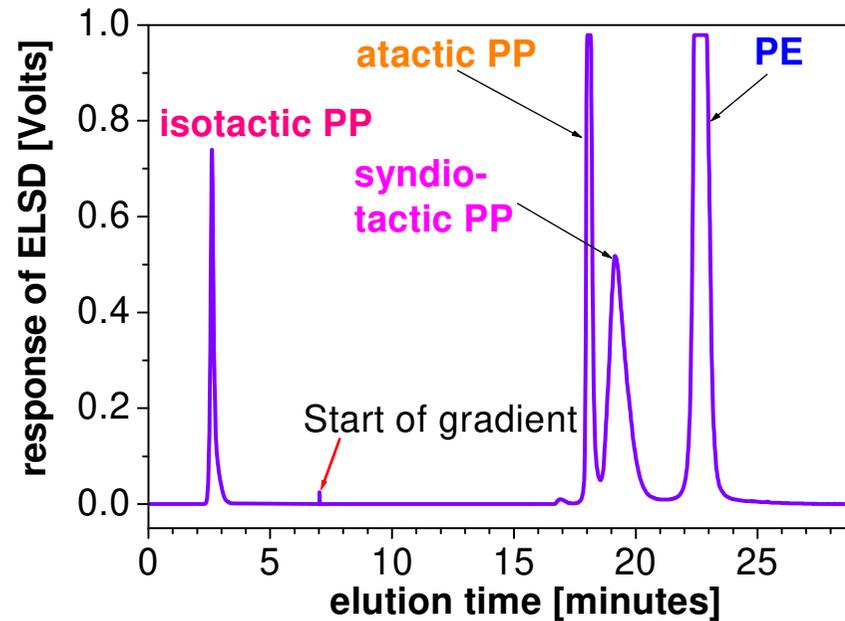
**IR
Raman
ELSD
NMR**

Graphite – A magic stationary Phase



- Absolutely flat surface
- Interactions with the analyte through the full range of electronic interactions

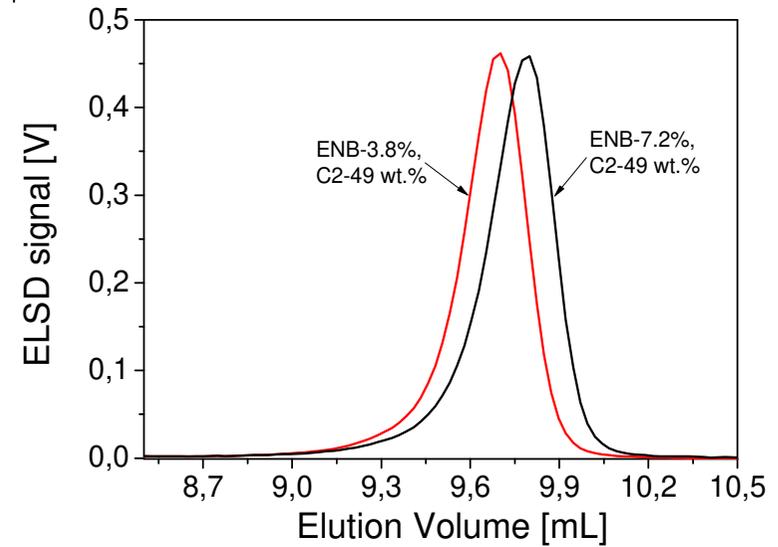
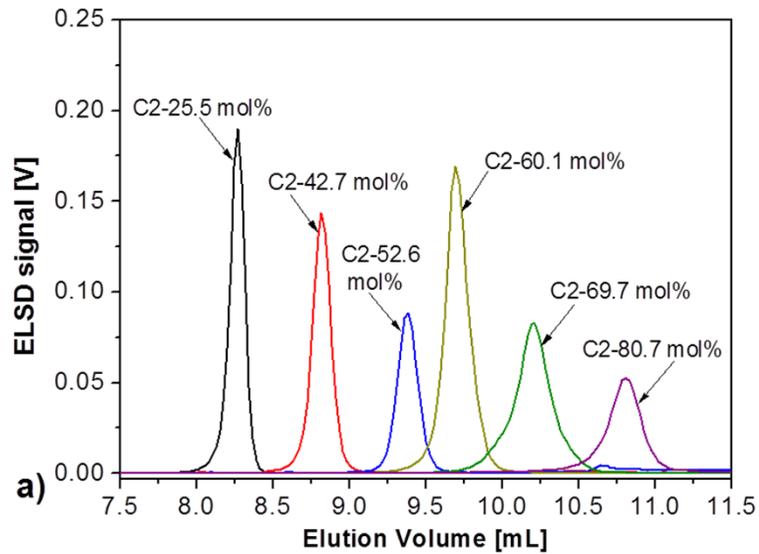
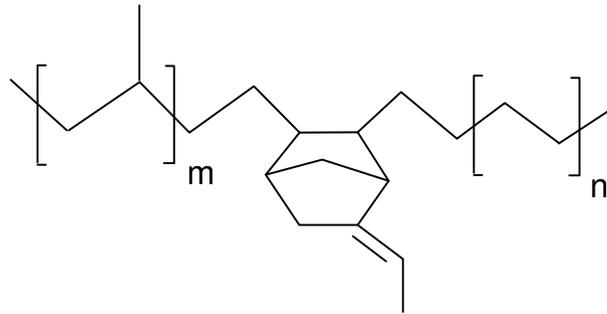
Interactive Chromatography up to 200 °C



- Highly selective interactions according to composition and microstructure become possible

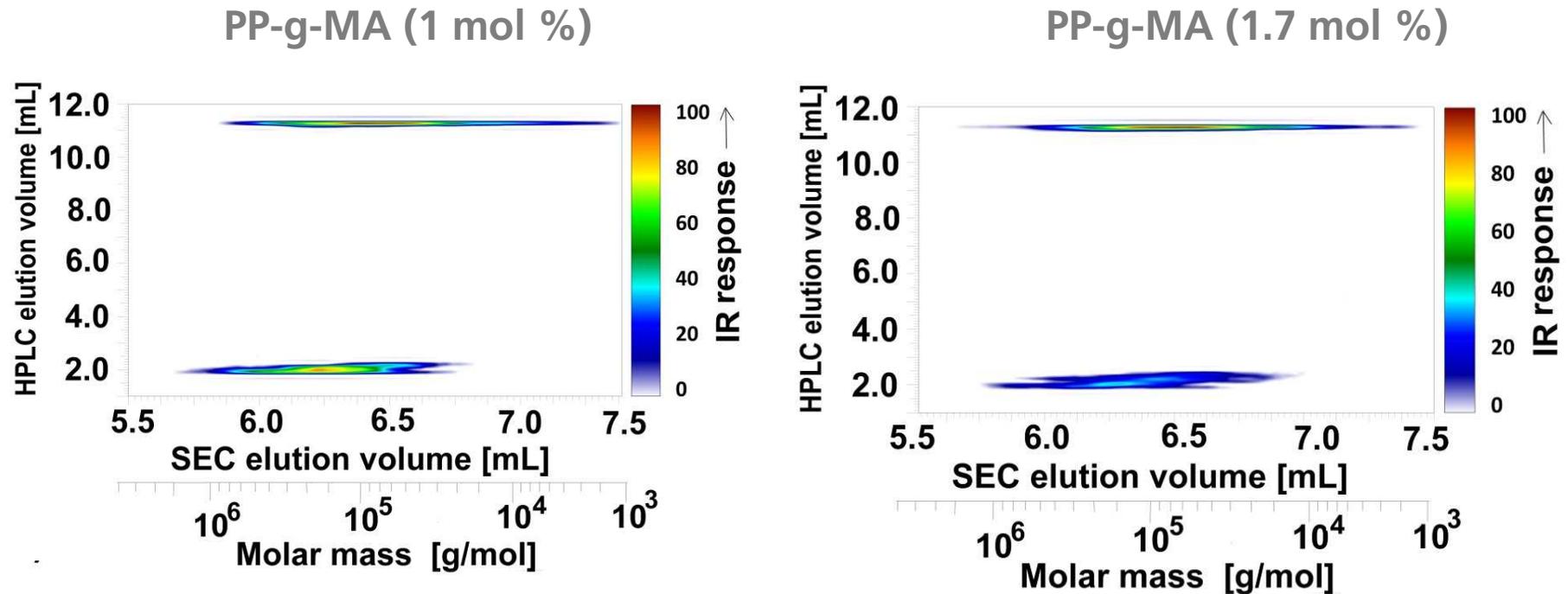
Stationary phase: Hypercarb™, Mobile phase: decanol → TCB

EPDM



- Separation according to ethylene content and diene content are possible

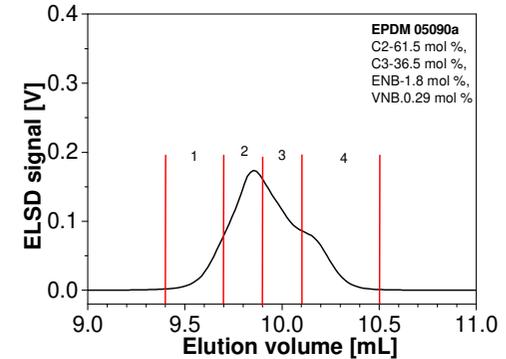
HT 2D-LC-IR of Grafted Polyolefins



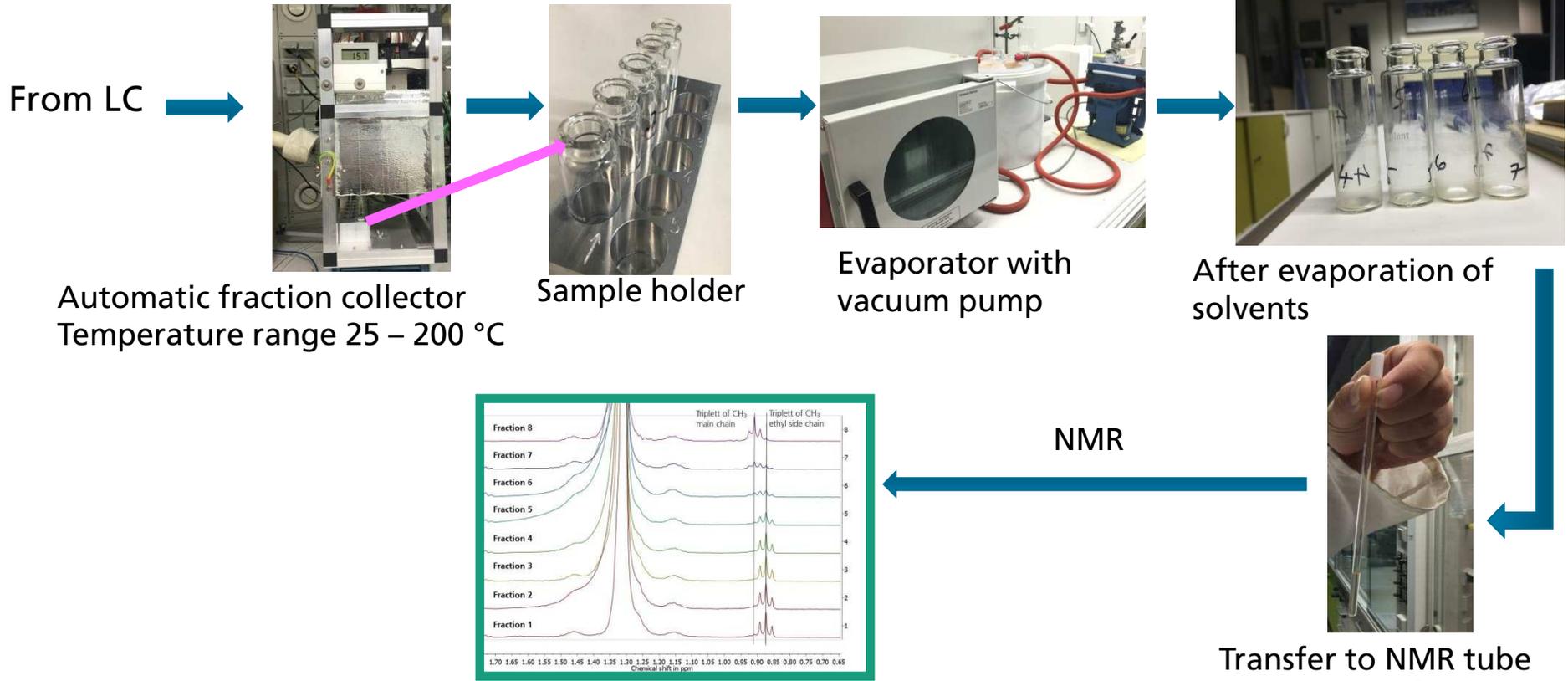
- Graft products can be separated from non-grafted material
- The molar mass distribution of each component can be determined
- The graft heterogeneity becomes visible

Steps for LC → NMR, FTIR and UV/RI (off-line)

Sample → LC-ELSD →



Replace ELSD by portable automatic fraction collector



Automatic fraction collector
Temperature range 25 – 200 °C

Sample holder

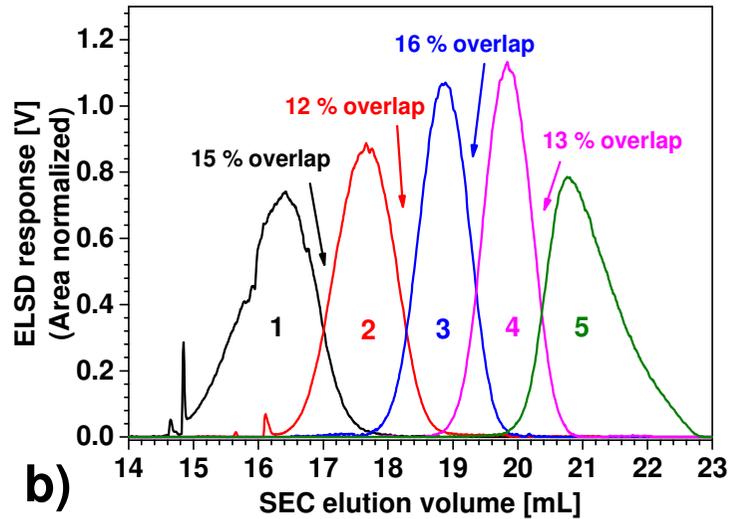
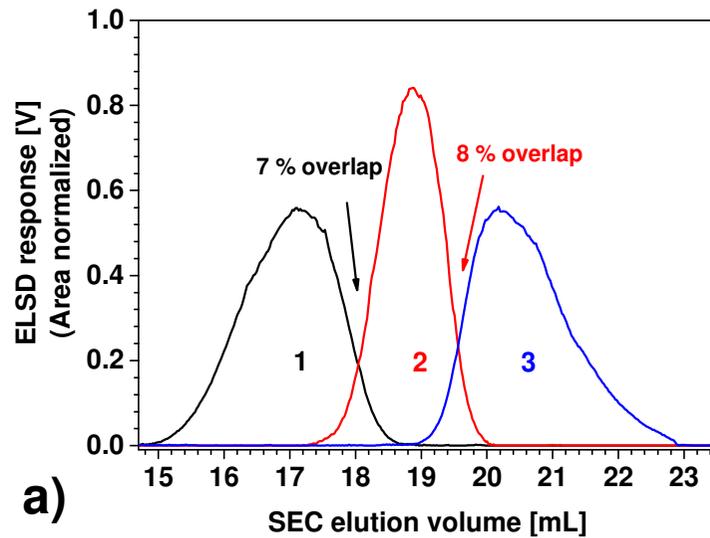
Evaporator with vacuum pump

After evaporation of solvents

Transfer to NMR tube

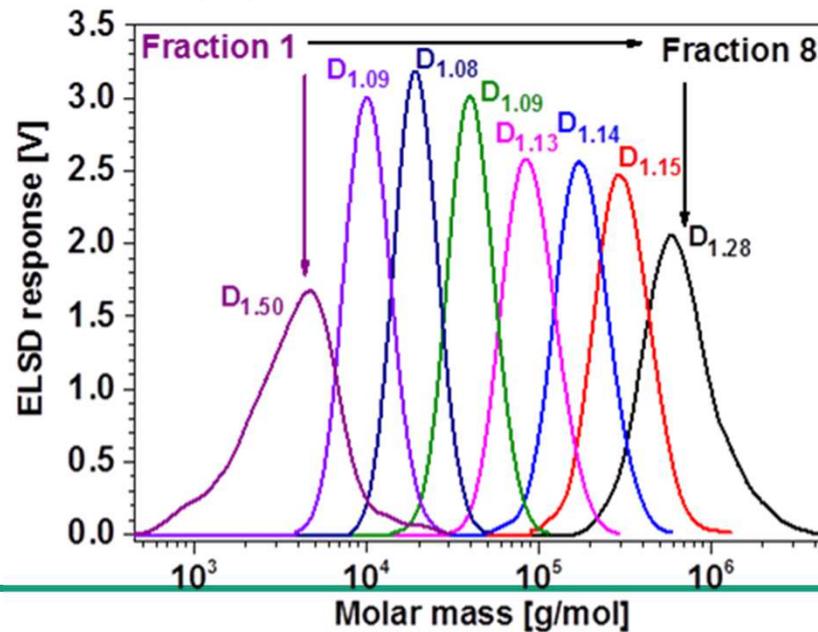
NMR

Preparative LC



3 fractions

5 fractions

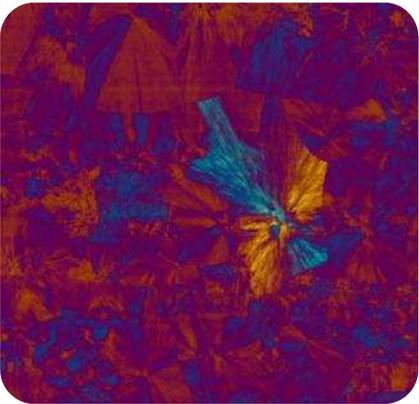


8 fractions

The world of imaging at LBF

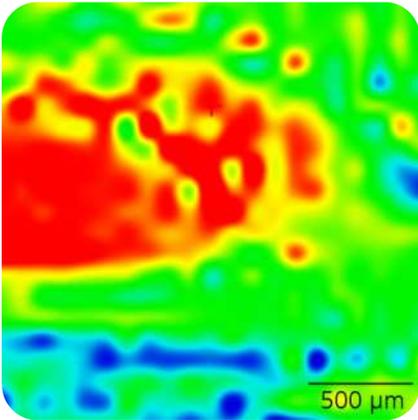
Polarized Light

- Morphology
 - Crystallinity
 - Orientation
 - Polymorphism



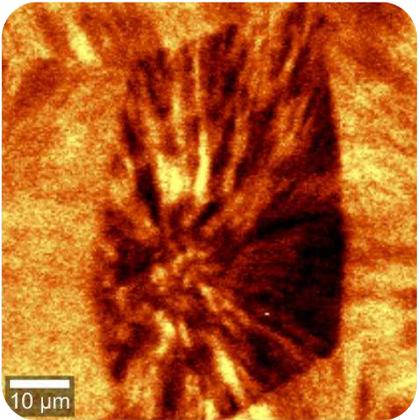
Infrared

- Morphology
 - Crystallinity
 - Orientation
- Additive
 - Content
 - Distribution



Raman

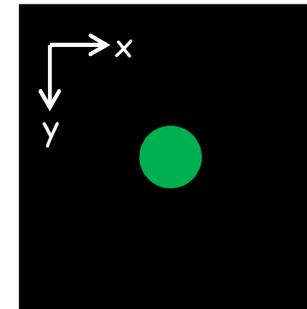
- Morphology
 - Crystallinity
 - Orientation
 - Polymorphism
- Additive
 - Identification
 - Distribution



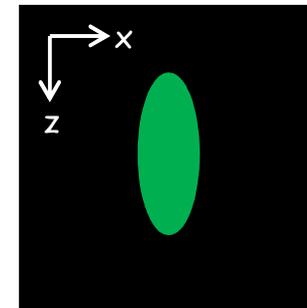
Confocal Raman microscopy (CRM)



Confocal Volume



266 nm



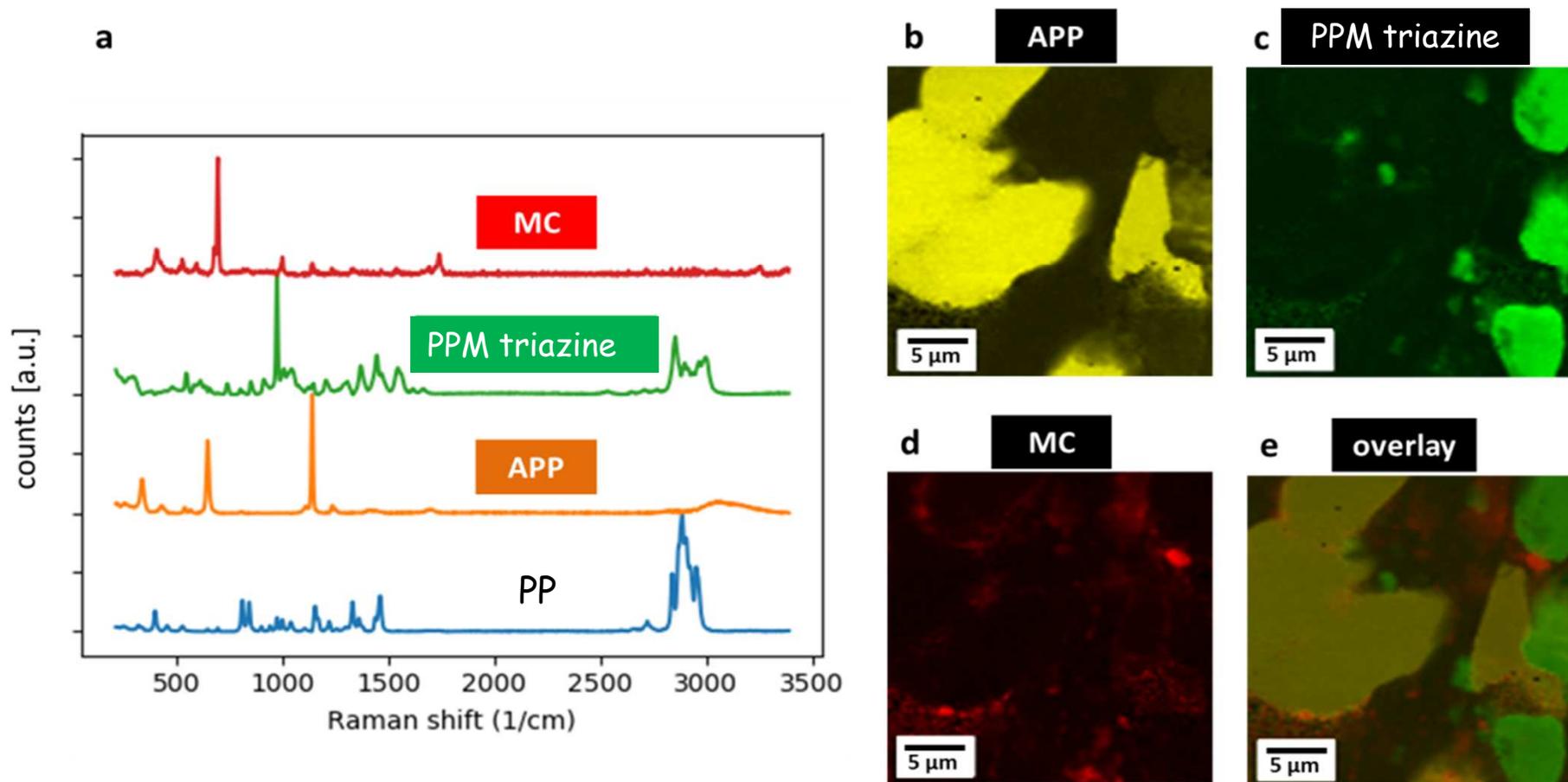
850 nm

Alpha 500 Confocal Raman Microscopy, WITec, Germany

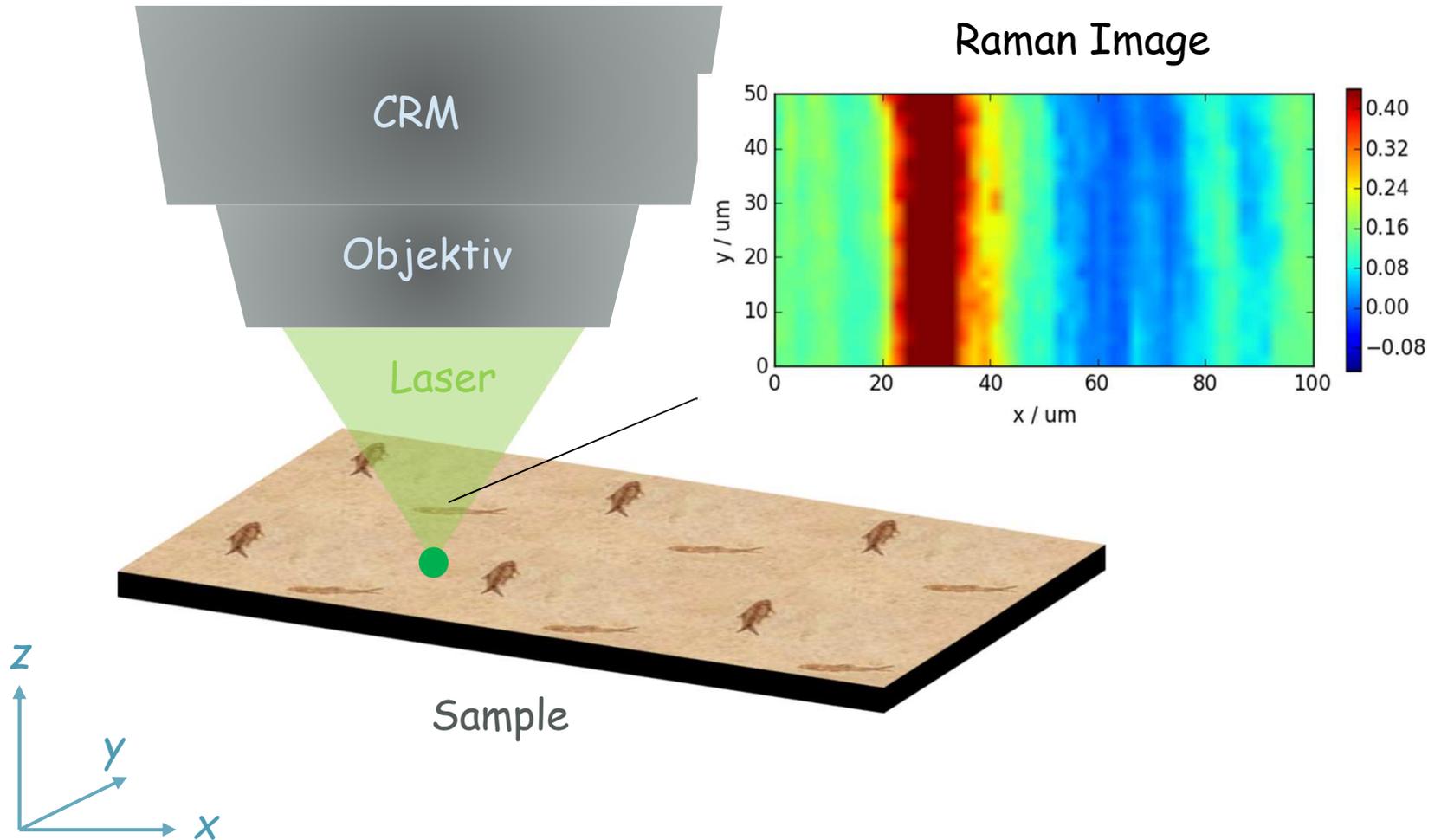
Flame Retarded PP (FR PP)

Cross section of FR PP

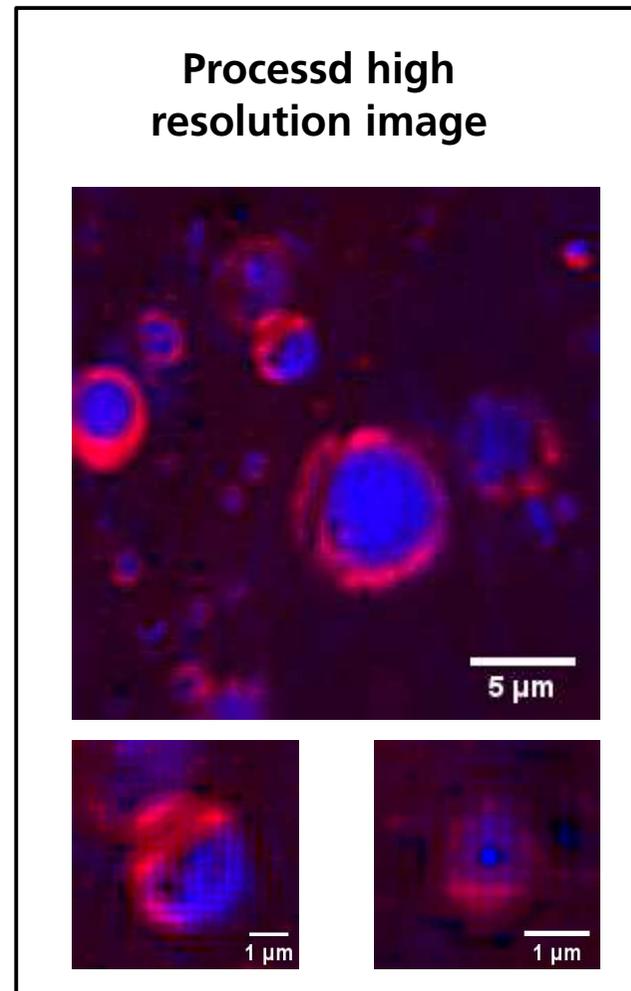
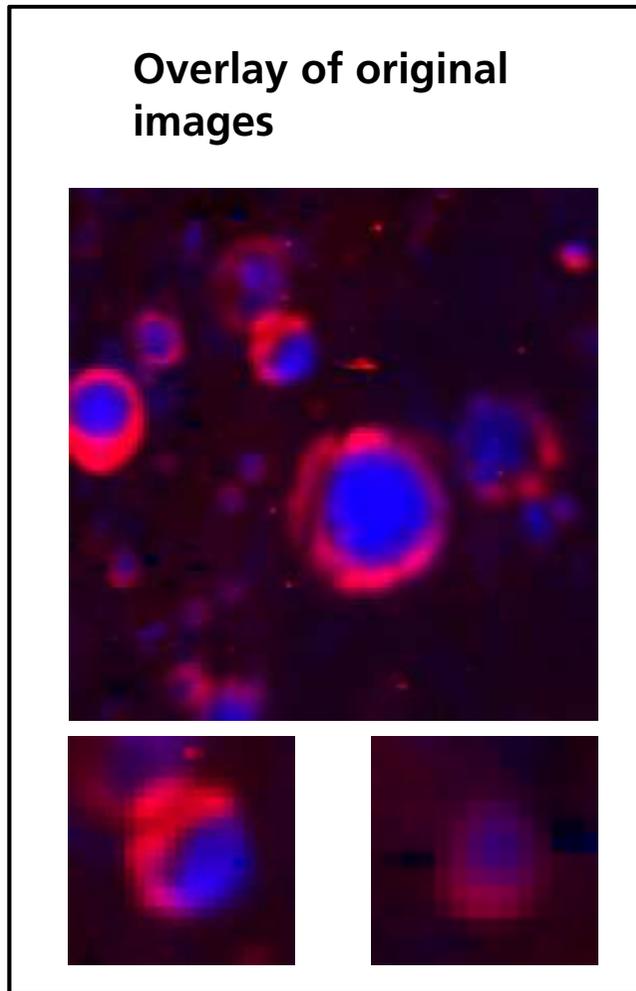
Raman-image of von $25 \times 25 \mu\text{m}^2$ with 100×100 pixels



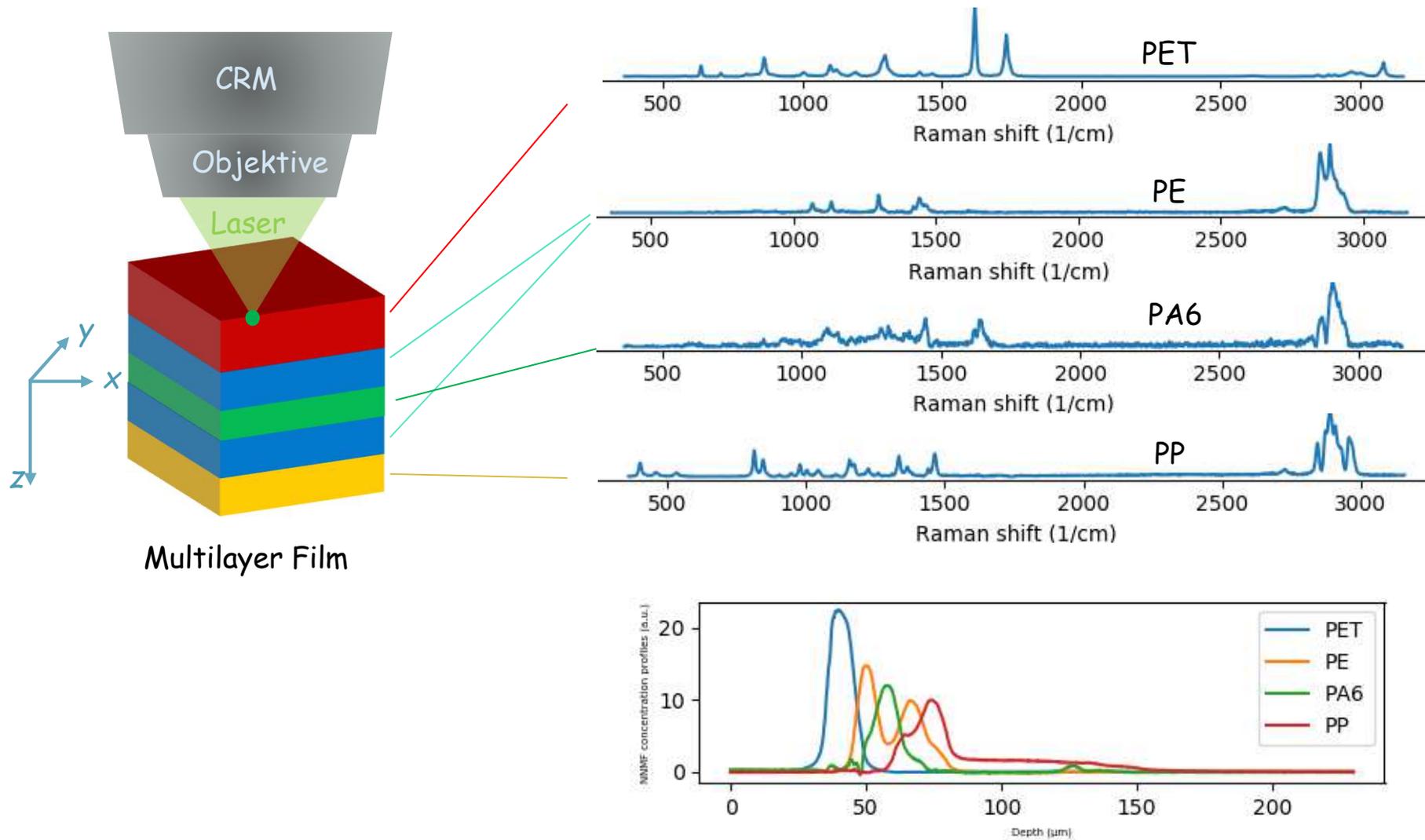
How does CRM work?



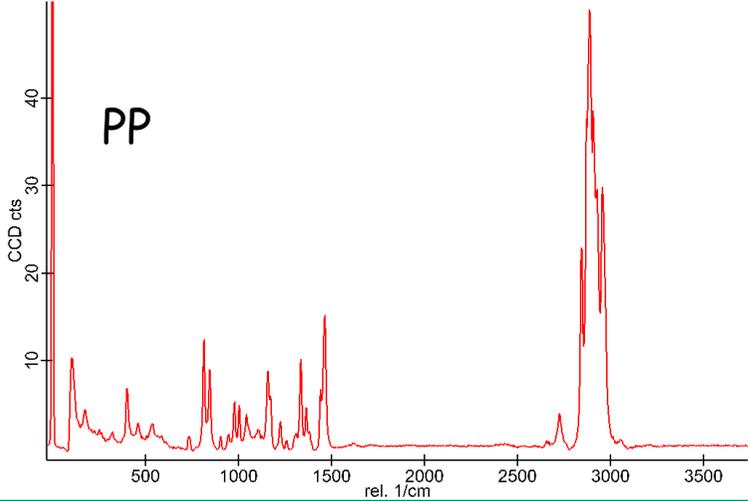
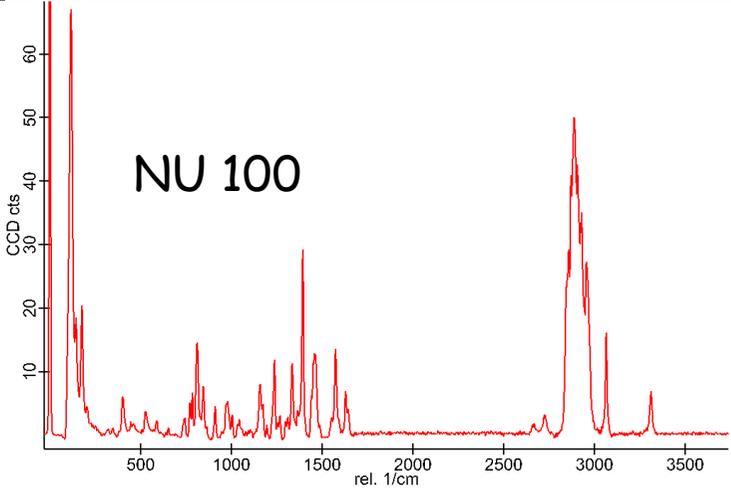
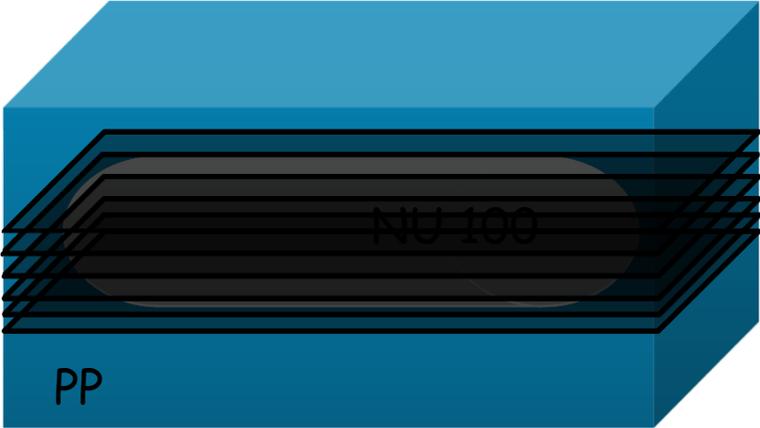
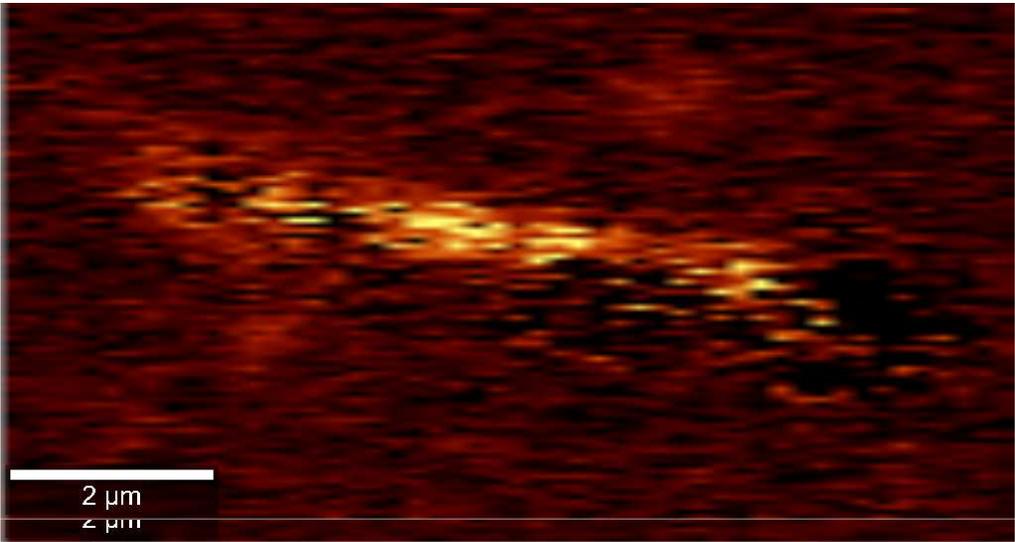
High Resolution CRM of Core-Shell Particles in POM



CRM: Depth Profiling



Volume mapping of additives

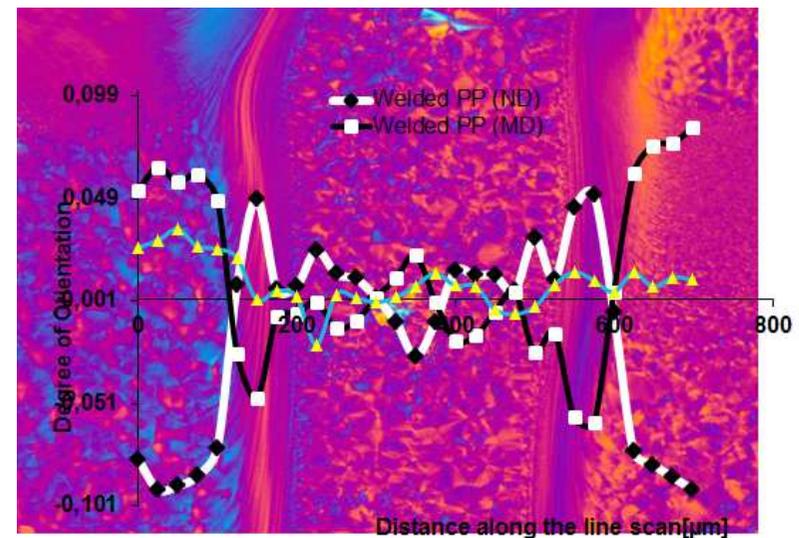
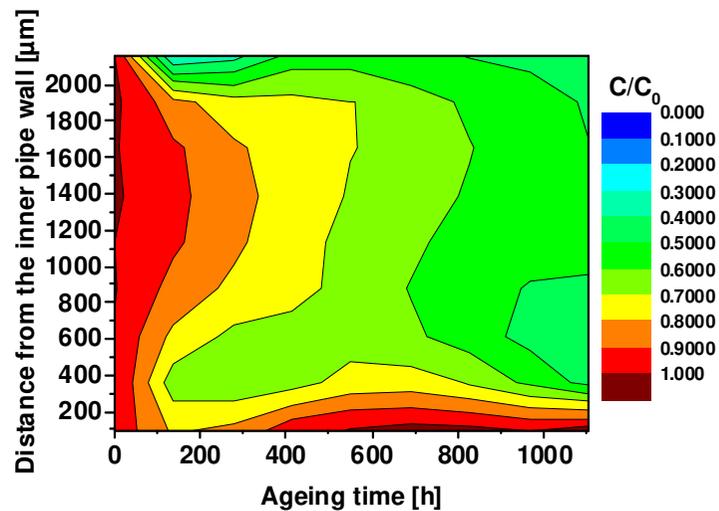


Infrared Imaging

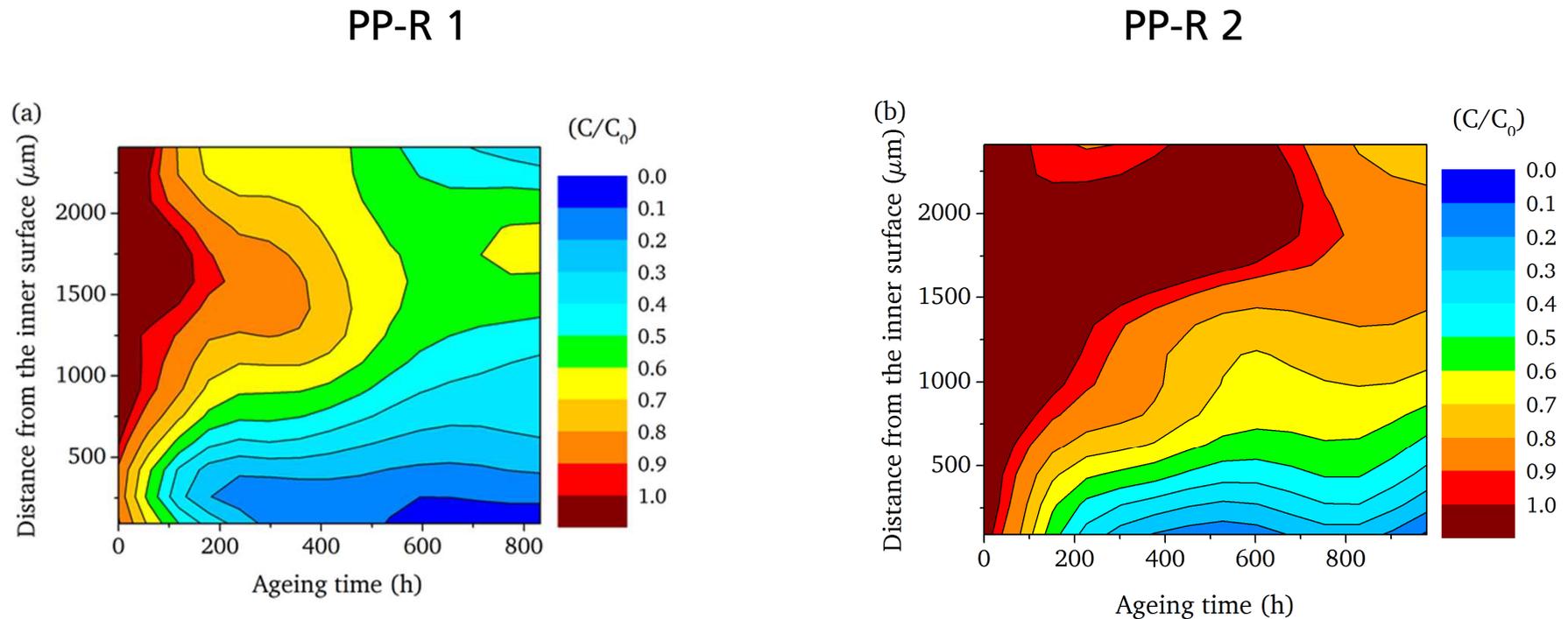
- μ FTIR:
 - Line Scan, Area Map
- Applications
 - Additive distributions
 - Time and space resolved
 - Morphology distributions



Chimassorb 944



Effect of Chlorinated Water on iPP-R Pipes



Chlorine (4 mg/l), 95 °C

- The time and space dependent concentration of stabilizers can be mapped

■ What can we do for you?

Robert.bruell@lbf.fraunhofer.de

Ingo.alig@lbf.fraunhofer.de

Frank.malz@lbf.fraunhofer.de