Ring-Opening Metathesis Polymerization and Electron-Beam Curing Derived Monolithic Materials: Versatile Materials and Formats

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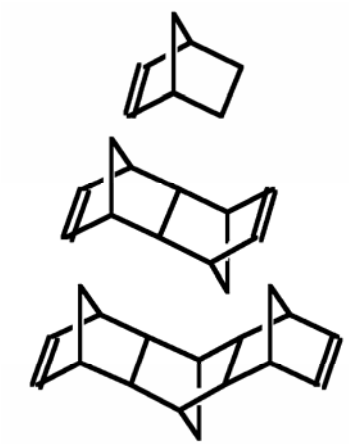
Synthesis of Monolithic Supports via ROMP

1. monomers

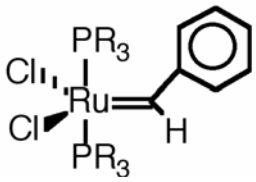


$(NBE\text{-}CH_2\text{-}O)_3\text{-}Si\text{-}Me$

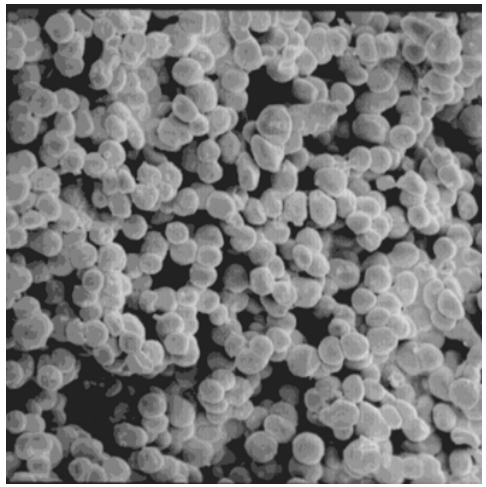
2. solvents

binary solvent systems
toluene ("good" polymer solvent)
2-propanol ("bad" polymer solvent)

3. initiator/modulator

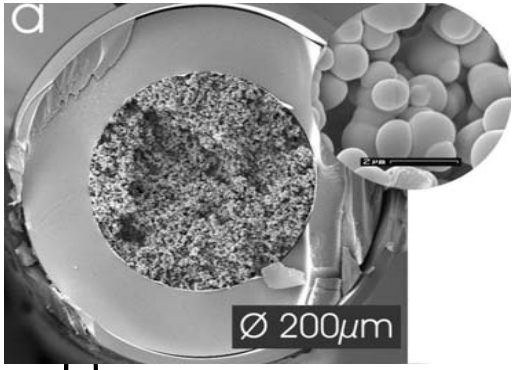


PR_3

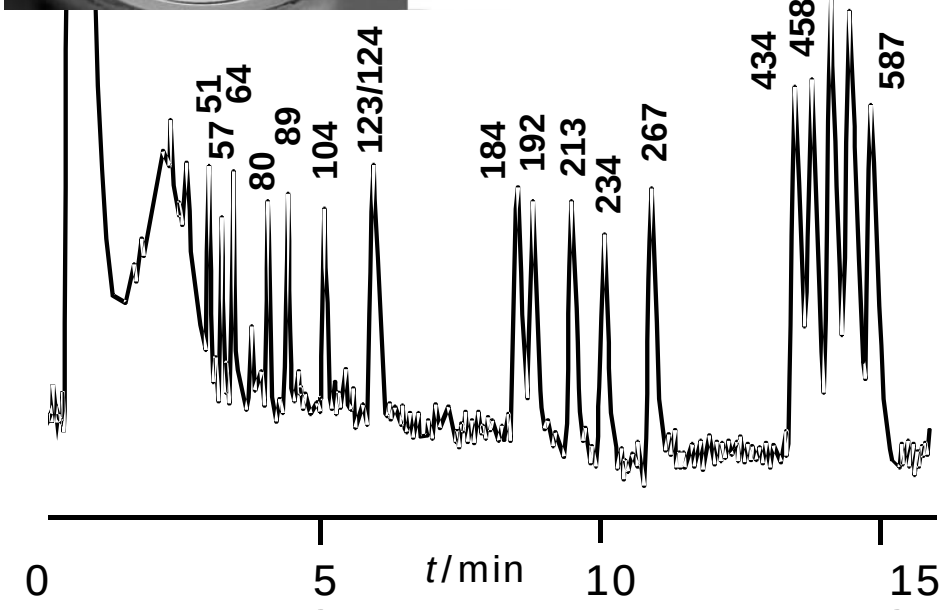


microglobule diameter: $1 < d_p < 30\ \mu m$
specific surface: $10 < \sigma < 100\ m^2$
residual Ru-content << 10 ppm
(essential for bioseparations!)
porosity: $0.3 < V_p < 0.5\ mL$; $0 < \varepsilon_z < 50\ \%$
pore size distribution --> application oriented synthesis

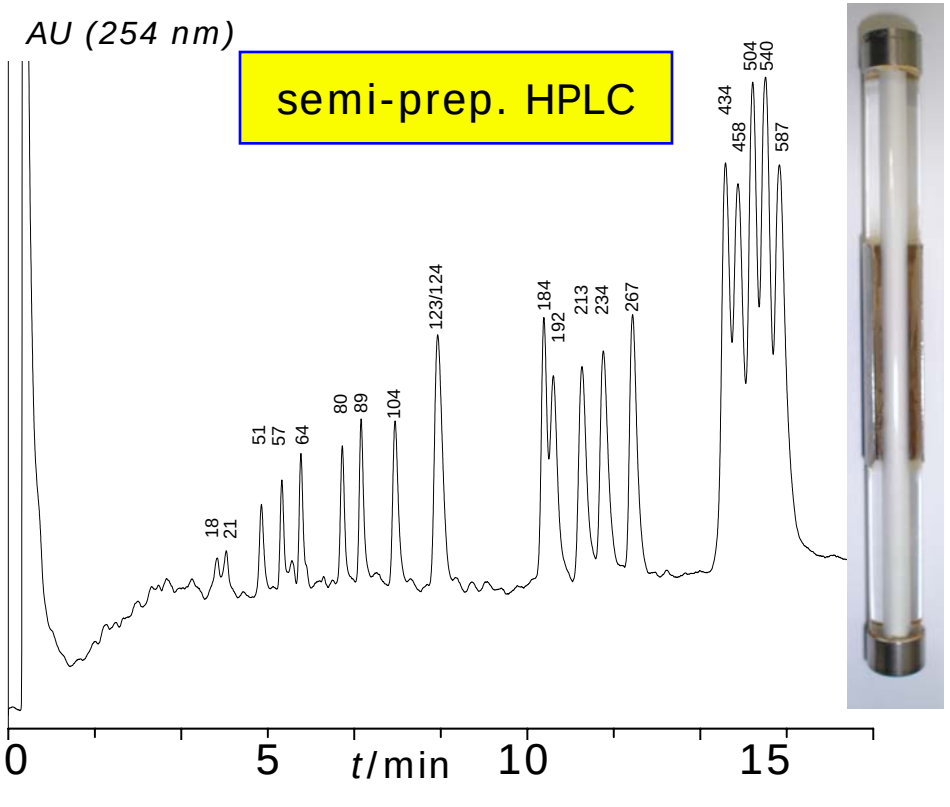
Separation of ds-DNA Fragments



µ-HPLC



semi-prep. HPLC



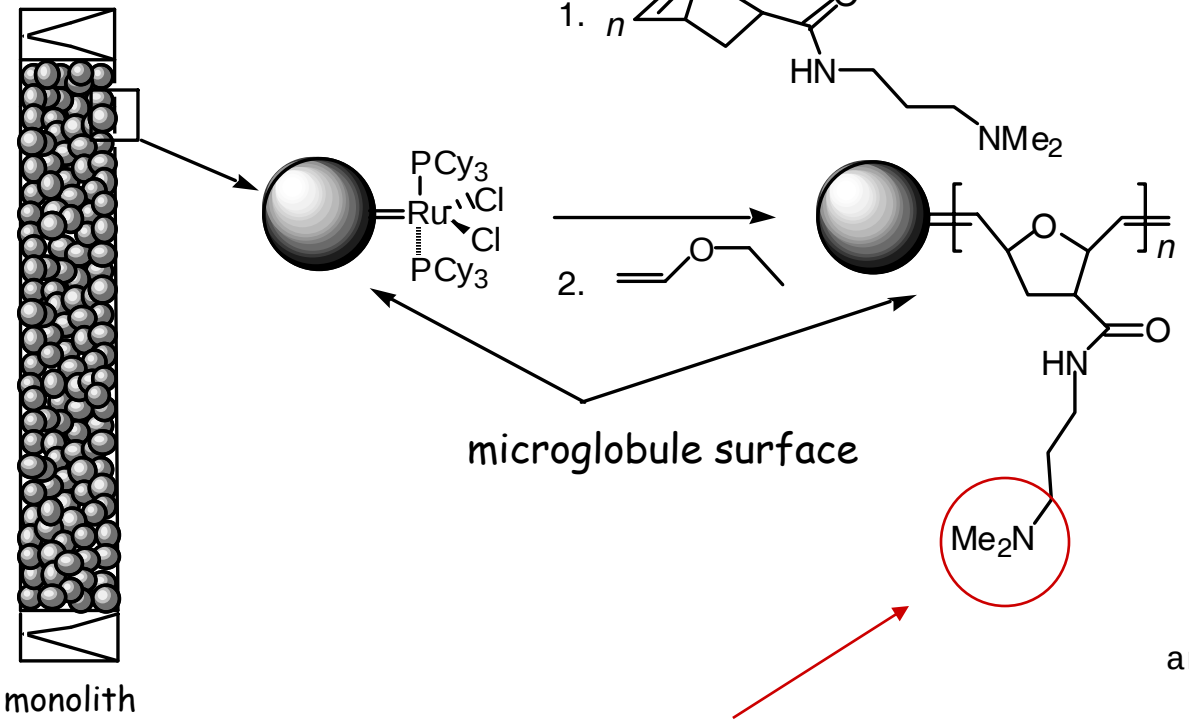
analytes: pBR322 HaeIII (12 ng)

monolith (0.2 x 60 mm); mobile phase 4 -10% acetonitrile in 1 min, 10 – 16% ACN in 14 min in 0.1 M $Et_3NH^+OAc^-$ pH 7.0; 1% MeOH; flow: 3 µL/min.

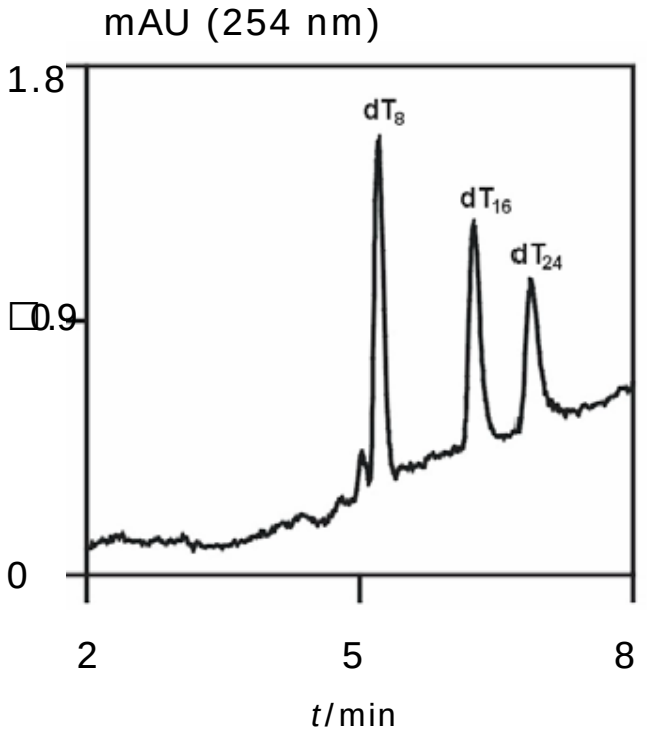
monolith (3 x 100 mm); mobile phase A: 100 % $Et_3NH^+OAc^-$ pH 7, mobile phase B: 100% $Et_3NH^+OAc^-$ pH 7 + 40% ACN. gradient: 10 – 60 % B in 15 min, 4 % glycerol, flow = 2.0 mL/min, T = 50 °C, inj. vol. = 0.75 µg.

J. Chromatogr. A (2002) **959**, 121; *Anal. Chem*. (2002) **74**, 6080.

Separation of Oligonucleotides on Surface-Derivatized Monoliths via Ion-Exchange



quaternized by mobile phase!



monolith (60 x 0.2 mm), gradient:
ammonium acetate (20 mM)-20 % AcCN,
pH 5 to Tris-HCL (20 mM)-20 % AcCN,
pH 8, salt gradient from 0 - 1 M NaCl
in 10 min, 4.3 µl/min. sample dT_{12}, dT_{16}, dT_{24}.

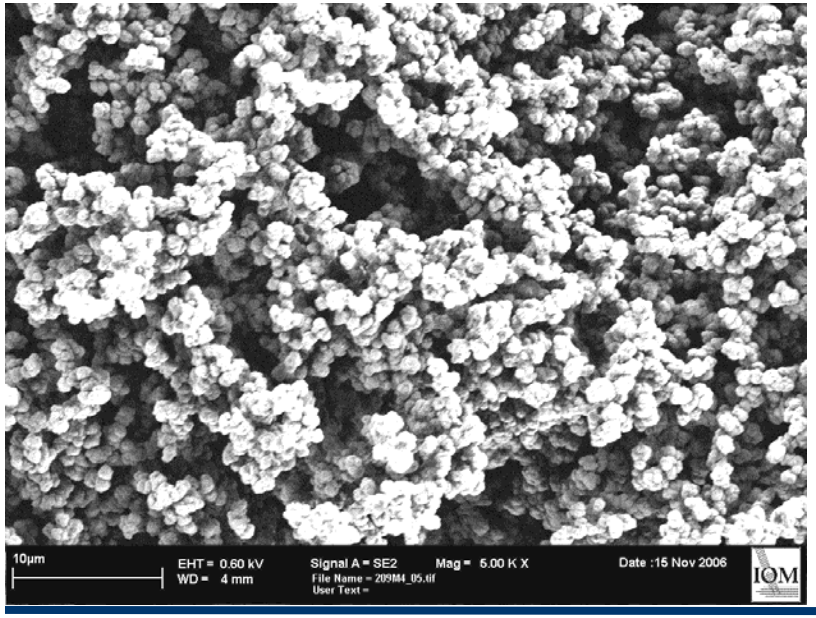
K. Eder, C. Huber, M. R. Buchmeiser, *Macromol. Rapid Commun*. in press (2007)

Monolithic Supports Prepared via EB Curing

Source: 10 MeV electron accelerator
Dose: 20-30 kGy
(pulsed to dissipate heat)

monomers: (meth)acrylates
crosslinkers: (meth)acrylates
porogens: 2-PrOH, dodecanol, toluene,...

specific surface area (σ): < 30 m^2
total porosity (ε_t): < 91% (!!!)



100 x 3 mm i.d.

480 x 70 mm i.d.