060/2010
Sepacore™

SHORT NOTE

Cleaning up of α- Methylstyrene by flash chromatography

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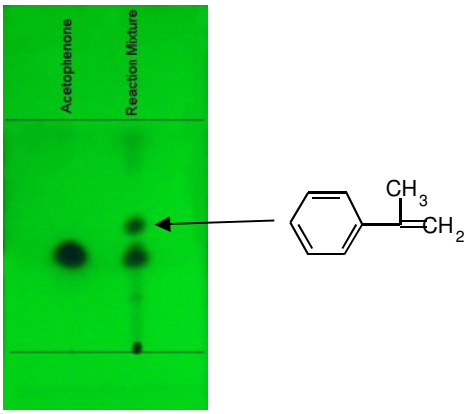
Assessment: The isolation of α-Methylstyrene from the crude reaction mixture seems easy. However the reaction mixture contains some dimethyl sulfoxide and the oily wetting agent from the sodium hydride. Both of them are not UV-active and therefore in the TLC invisible.

TLC of the crude reaction mixture:

TLC on silica gel $Si60_{F254}$, developed in n-hexane/ethyl acetate 90:10, detection UV 254 nm



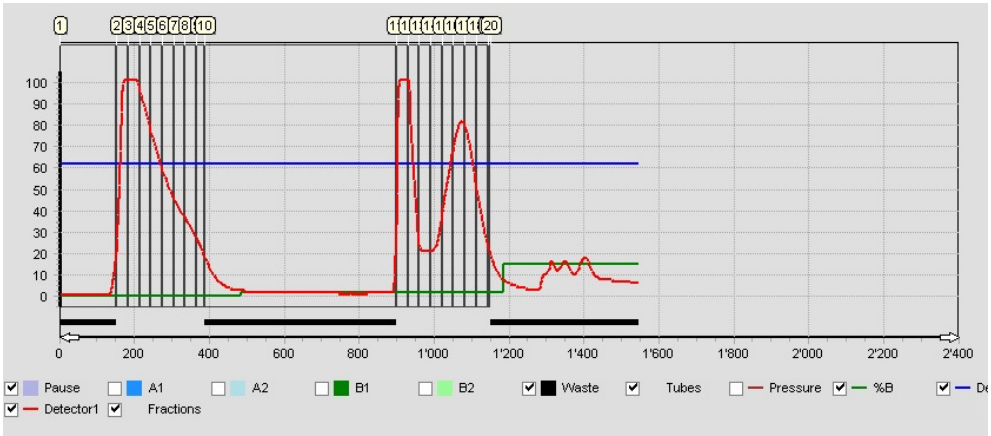
Sepacore configuration

Cartridge 40 x 150mm, prepacked with silica gel 60, 40 – 63 µm
2 Pump Modules C-605
Fraction Collector C-660
Control-Unit C-620 with SepacoreControl software
UV Photometer C-635

Separation conditions

Eluent: n-hexane with 0%, 2% und 15% ethyl acetate, step gradient
Flow rate: 100ml/min
Sample: 3 g crude mixture, dissolved in toluene
Injection volume: 4.5 ml (although the mixture is liquid, the sample is not fully miscible with n-hexane and must be dissolved in toluene and injected directly onto the cartridge).

Separation

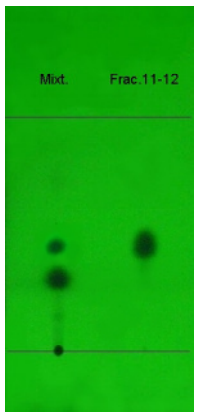


Fractions

2–10: mainly toluene and wetting agent
11–12: α-Methylstyrene
14–18: Acetophenone (reactant)

TLC of the separated fractions

TLC on silica gel 60_{F254}, developed in n-hexane-ethyl acetate 9:1, detection UV 254 nm



Recovery

Fraction 11 - 12: 0.4 g colourless oily liquid