

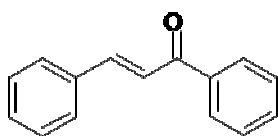
Isolation of Benzylideneacetophenone from a crude reaction mixture

Example for scale-up

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TLC of the crude reaction mixture:

TLC on silica gel Si60_{F254}, developed in n-hexane/ethyl acetate 19:1, detection UV 254nm



Sepacore configuration

Cartridge 12 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: see below

Flow rate: 10 ml/min

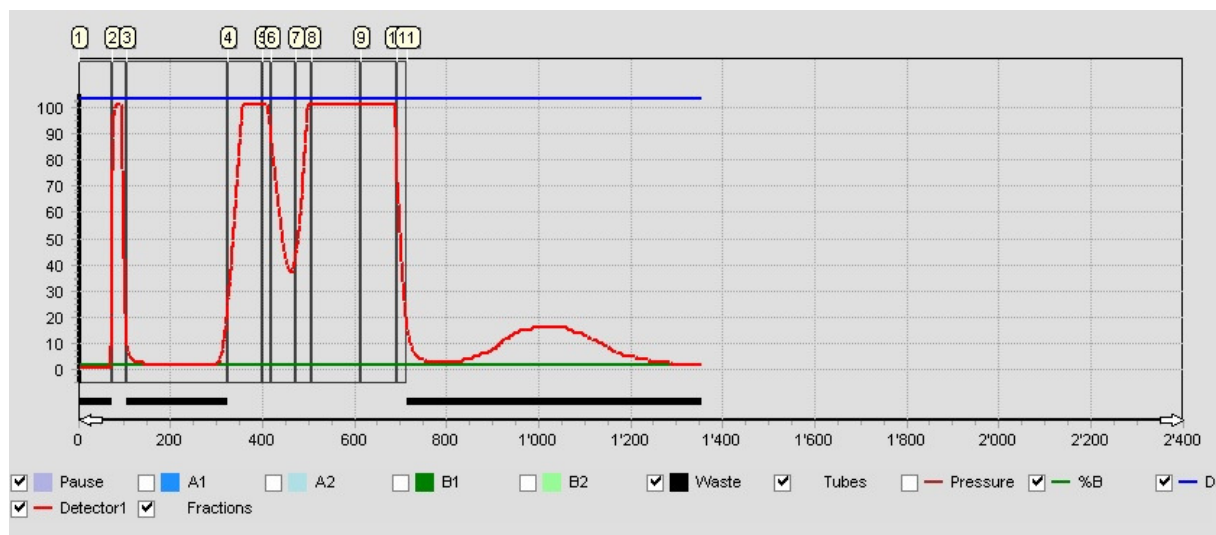
100 mg crude mixture, dissolved in n-hexane and some drops of toluene (solubility of the sample in pure n-hexane too low)

Loading: approx. 0.4 ml

1. Test runs

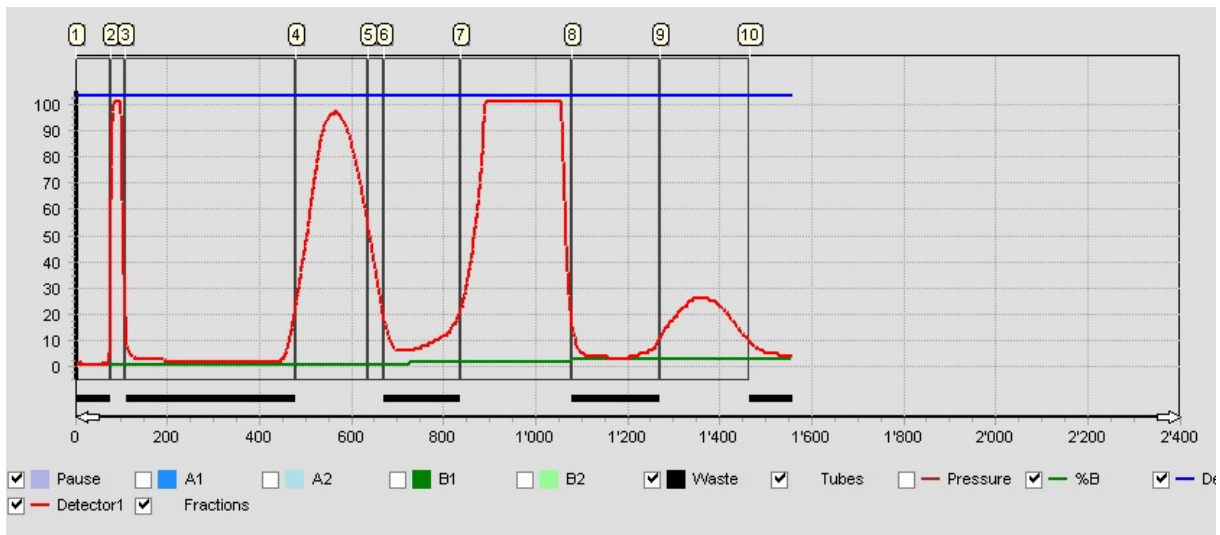
Separation 1

Eluent: n-hexane with 2% ethyl acetate, isocratic



Separation 2

Eluent: n-hexane with 1%, 2% und 3% ethyl acetate, step gradient. Each step was initiated after a component was completely eluted (at the end of a peak)



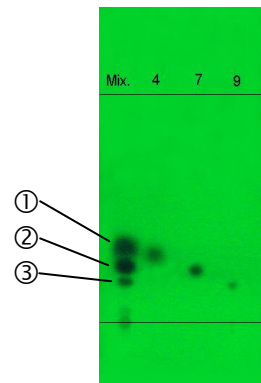
TLC check

TLC on silica gel Si60_{F254}, developed in n-hexane/ethyl acetate 19:1, detection: UV 254nm

- ① = Benzaldehyde
- ② = Benzylideneacetophenone
- ③ = Benzophenone

Recovery

Fraction 7: 65 mg



2. Scale-up

Calculation of the scale-up factor

Cartridge 12 x 150mm: cross sectional area = 1.13 cm²

Cartridge 40 x 150mm: cross sectional area = 12.56 cm²

Scale-up-factor = 12.56 cm² / 1.13 cm² = 11.1 ≈ 10

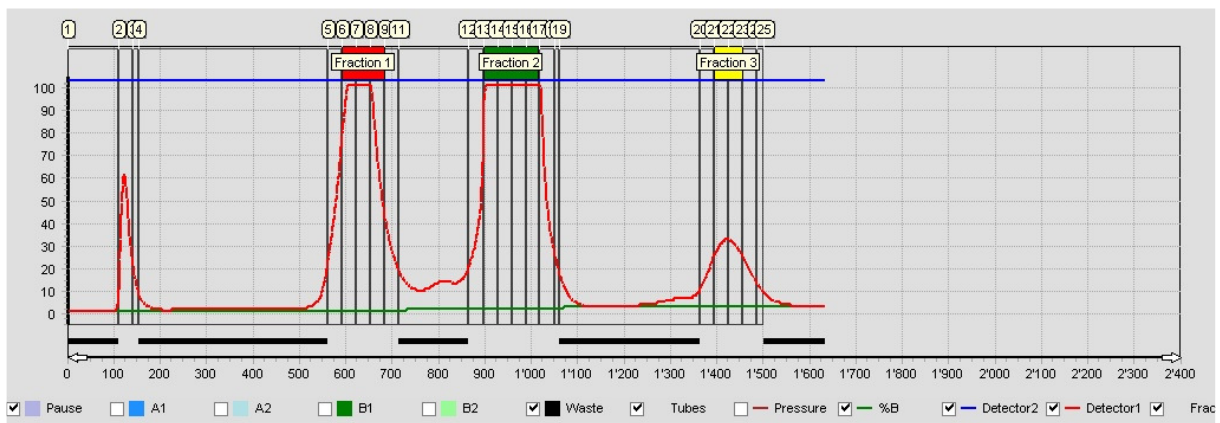
Separation conditions

Eluent: n-hexane with 1%, 2% and 3% ethyl acetate, step gradient.

Sample: 1 g crude mixture, dissolved in n-hexane/toluene 7:3 (solubility of the sample in n-hexane too low, toluene is eluted as front peak)

Injection volume: 2 ml

Scale-up separation



TLC-Check

TLC on silica gel Si60_{F254},
developed in n-hexane/ethyl acetate 19:1,
detection: UV 254nm

Recovery

Fraction 13 – 16: 512 mg crystalline product, mp 57 °C

