SHORT NOTE

Isolation of 2,2‘- Furoin from a crude reaction mixture

Example of a solid sample loading (sample adsorbed to silica gel)

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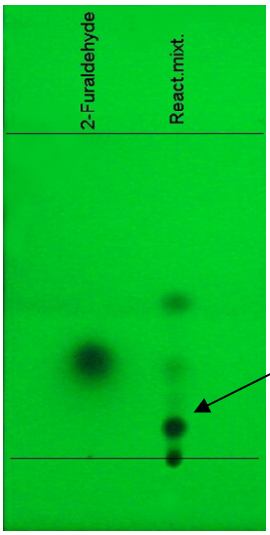
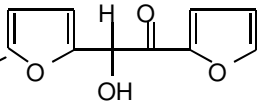
TLC of the crude reaction mixture:

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





Furoin

Sepacore configuration

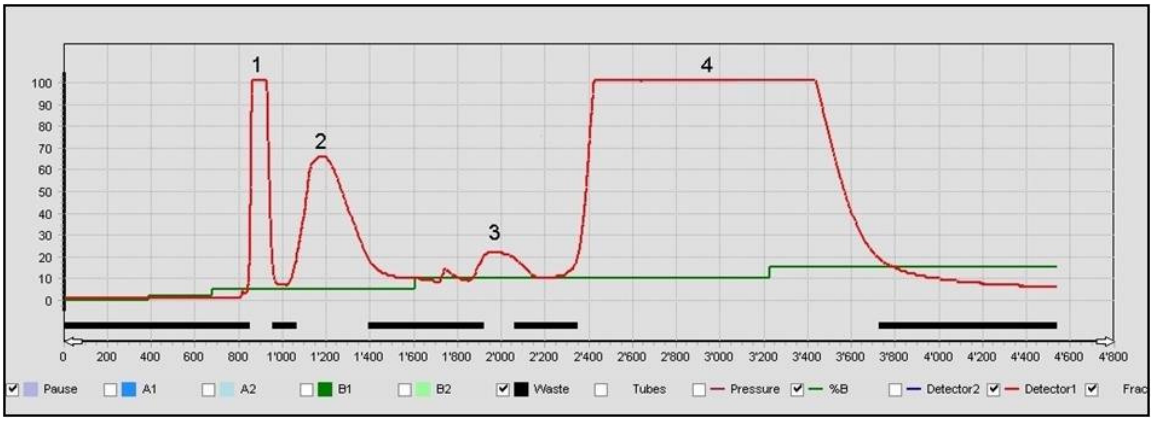
Cartridge 12 x 150mm, prepacked with silica gel 60, 40 – 63 µm
2 Pump Modules C-605
Fraction Collector C-660
Control-Unit C-620 with SepacoreControl software
UV Photometer C-635

Separation conditions

Eluent: n-hexane, n-hexane with 2%, 5%, 10% and 15% ethylcacetate, step gradient (see. chromatogram)
Flow rate: 10 ml/min
Sample dissolved in toluene (solubility in n-hexane with 5% isopropyl ether too low)
Sample: 0.25 g brown, semicrystalline crude product, adsorbed to 0.75 mg silica gel Si60, 40-63 µm

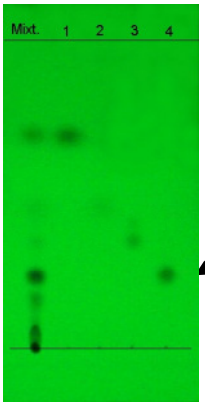
Solid sample application using the Büchi Prep Elut Adaptor

Separation



TLC of the separated fractions

TLC on silica gel 60_{F254}, developed in n-hexane with 20% ethyl acetate, detection UV 254 nm



Furoin

Recovery

Fraction 4: 0.15 g Furoin, slightly yellowish crystals, mp 136.5°C