



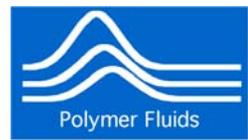
Micro Flow Disc Reactors for continuous flow organic synthesis

Christian H. Hornung, Bart Hallmark, Malcolm R. Mackley

26th April 2007
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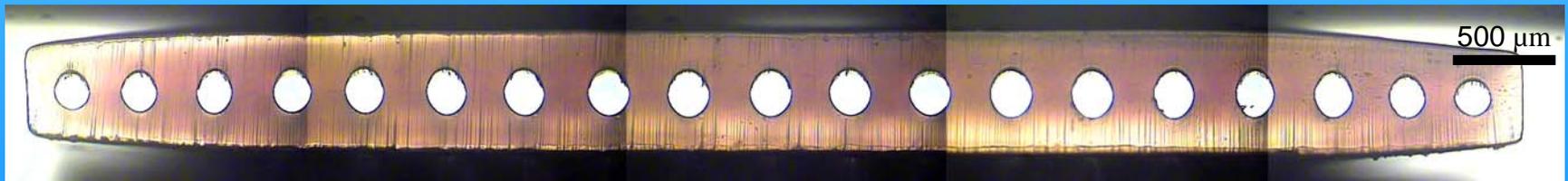
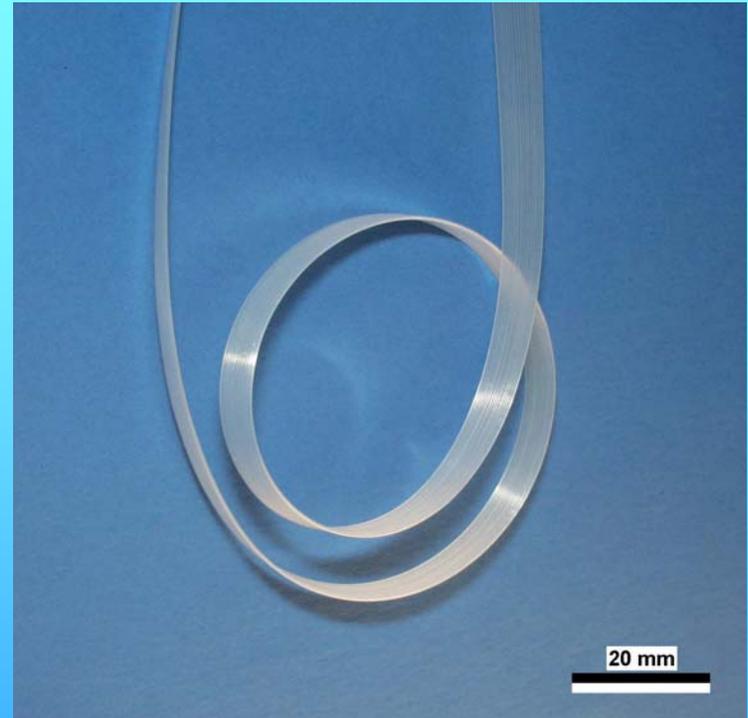


Microcapillary films (MCFs)



Microcapillary Films (MCFs):

MCFs are plastic films containing microcapillary arrays of continuous length. The capillaries have a round to elliptical shape with diameters between 30 and 500 μm . MCFs are manufactured by gas-entrained polymer extrusion.

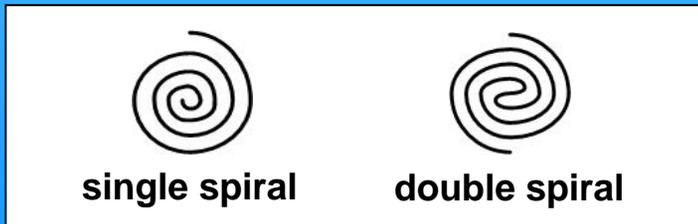
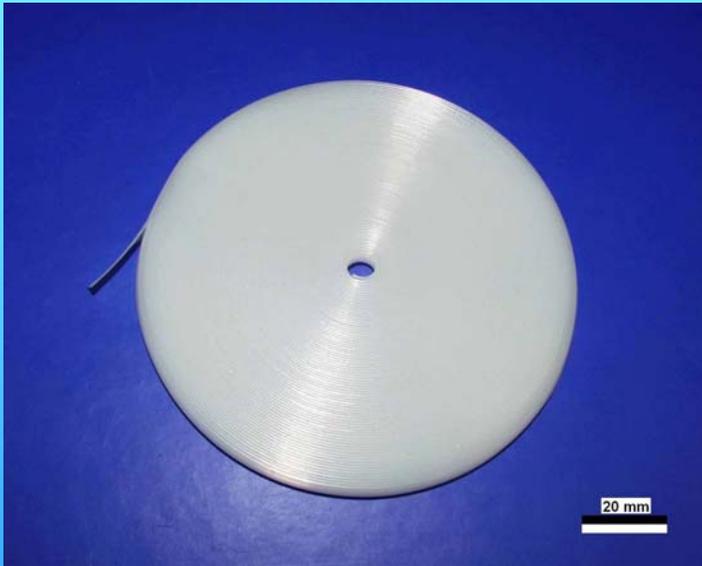




Microcapillary flow disc (MFD)

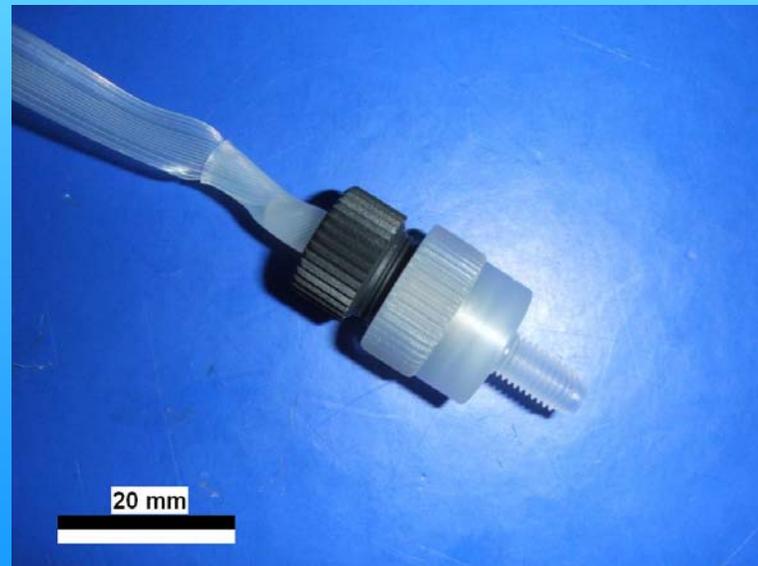


- MCF wound up to form spiral (single or double spiral)
- Heat pressing (at 116 °C for LLDPE) → solid compact disc
- typical length: 5 – 40 m



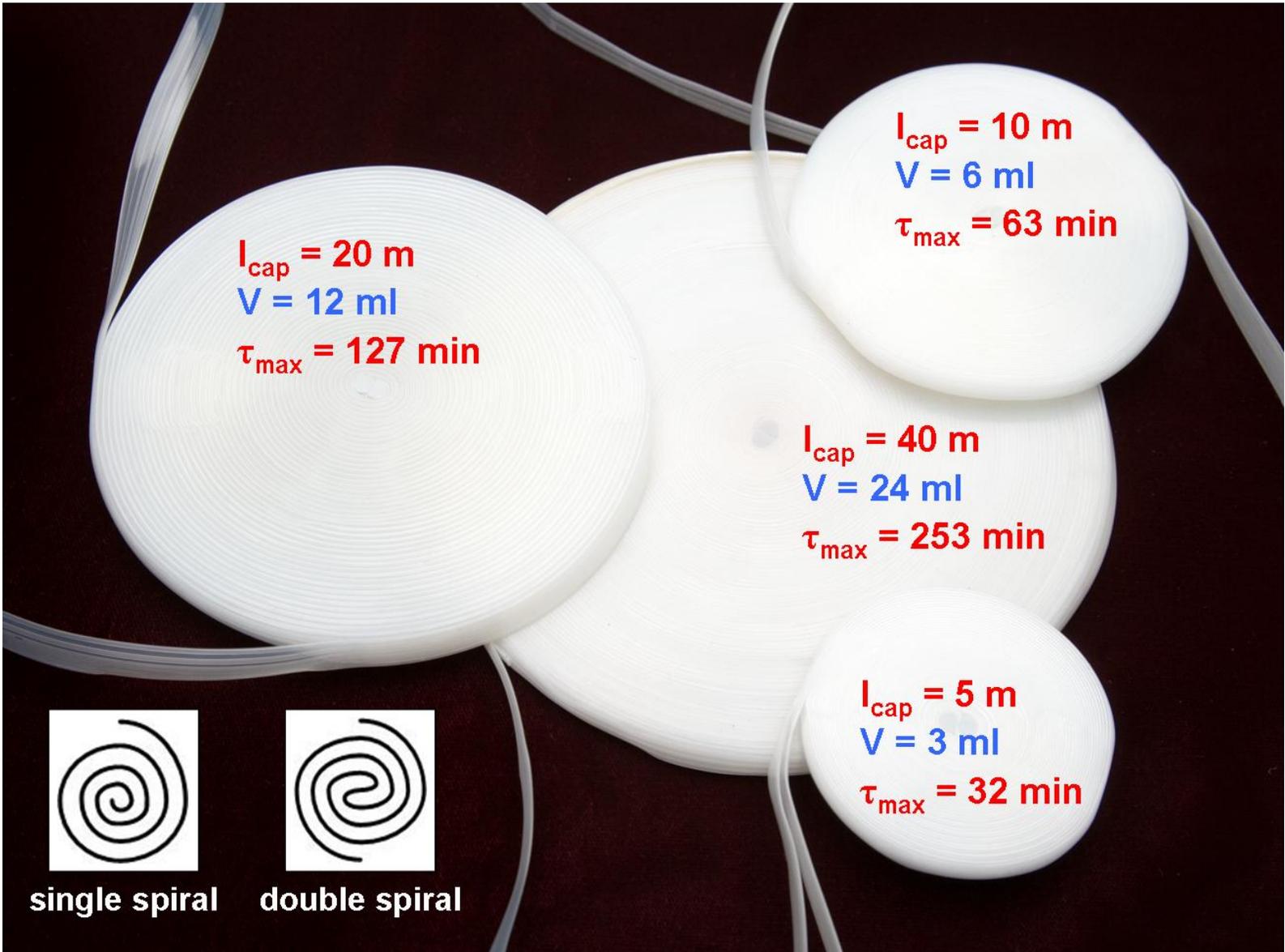
Connectors:

- 1) epoxy resin / HPLC fitting
- 2) cyl. MCF unit / HPLC fitting



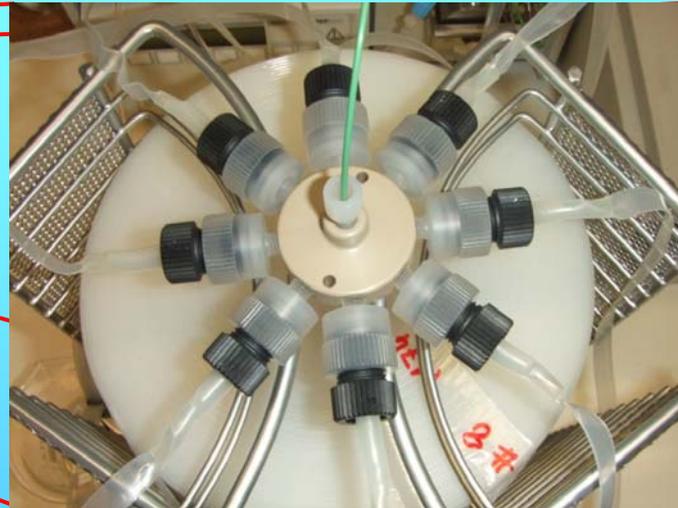
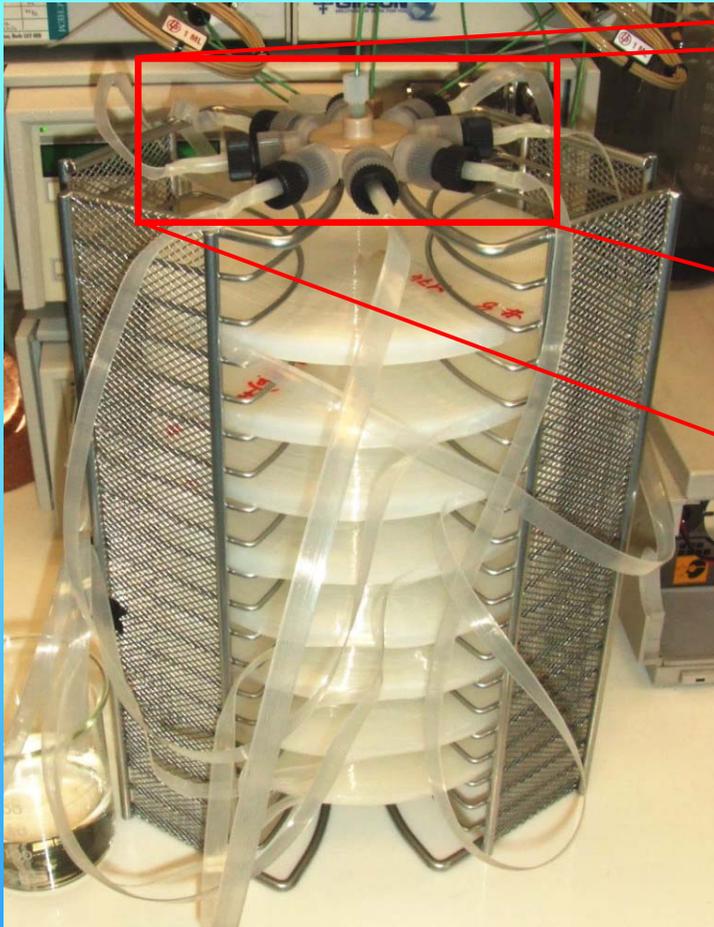


Microcapillary flow disc (MFD)





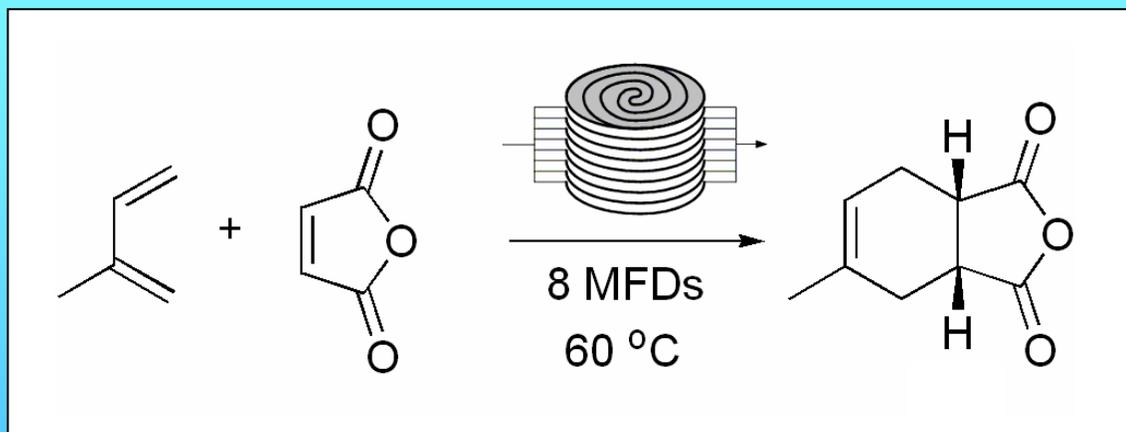
Kilogram-scale reactor



- 8 flow discs running in parallel
- 152 capillaries / length: 40 m
- ➔ total: 6.1 km microchannel
- ➔ $Q = 0.1 \dots 10 \text{ ml/min}$
- ➔ production of g- to kg-quantities



Diels-Alder reaction



Reaction conditions:

Q = 2 – 6 ml/min

solvent: MeCN

τ = 28 - 113 min

T = 60 °C

2 ml/min / 113 min

→ 93 % conv.

→ 1.1 kg/day

4 ml/min / 57 min

→ 93 % conv.

→ 2.2 kg/day

6 ml/min / 28 min

→ 93 % conv.

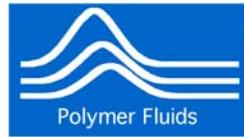
→ 3.9 kg/day



- Variable, low cost, continuous flow microreactor concepts made from MCFs
- Key potential features of MCFs:
 - Flexible film of continuous length
 - Fast heat transfer / good temperature control
 - Optical transparency / microwaveable



Acknowledgements



- Prof. Steven V. Ley †
- Prof. Robert P. Hesketh ‡
- Dr. Ian R. Baxendale †
- Polymer Fluids Group *
- ITC †
- EPSRC



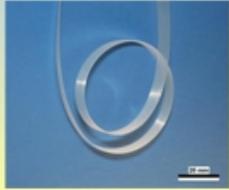
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Microcapillary Reactors (MCRs)



Microcapillary Films (MCFs)

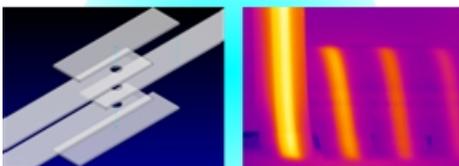


Typical dimensions:
 capillary dia.: 100 – 200 μm
 film width: 5 – 10 mm
 film thickness: 0.4 – 0.8 mm
 no. of cap.: 5 - 20

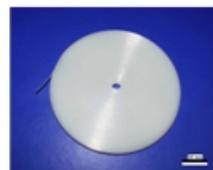
Separation reactor /
 Capillary separator
 → semi-permeable MCF matrix

Cont. flow photo-
 chemical reactor
 → fluoro-polymer MCF

Fast flow reactor
 → for fast exothermic reactions



MCF flow disc



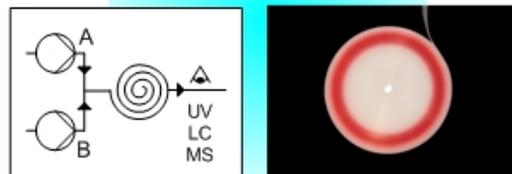
Cont. flow
 microwave reactor
 → fluoro-polymer MCF



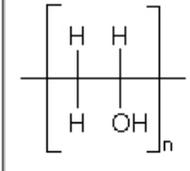
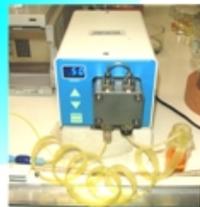
Production scale
 reactor



Disposable lab
 reactor system
 → for organic synthesis / high
 throughput screening

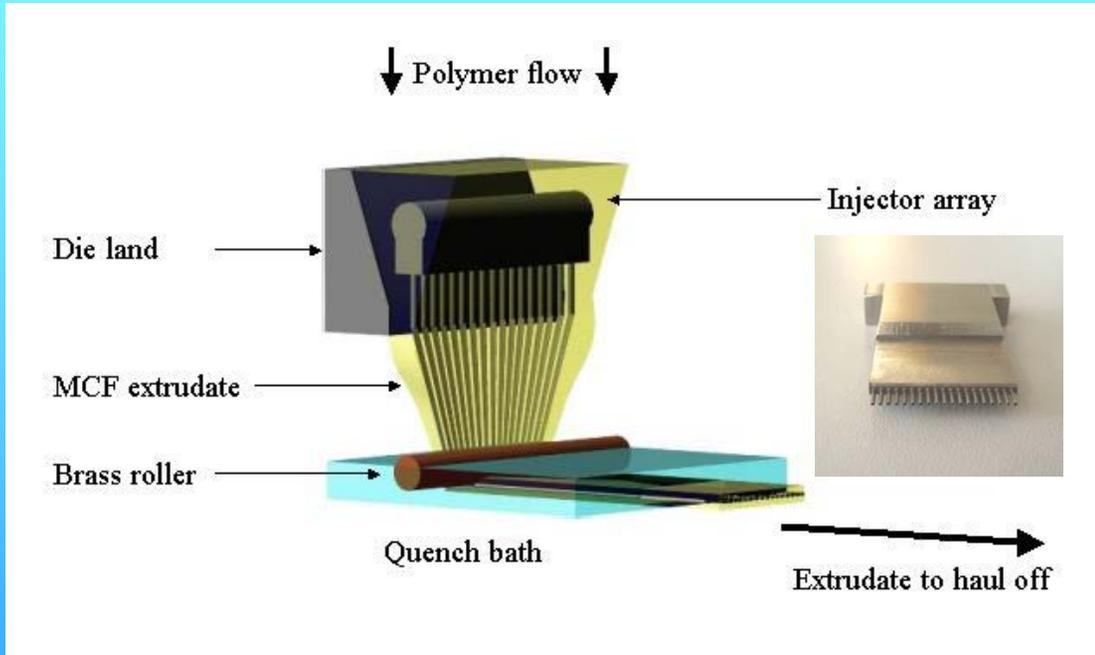


Functionalised wall
 reactor
 → catalytic wall coating
 → enzyme immobilisation



Gas-entrained polymer extrusion:



polymer matrix:

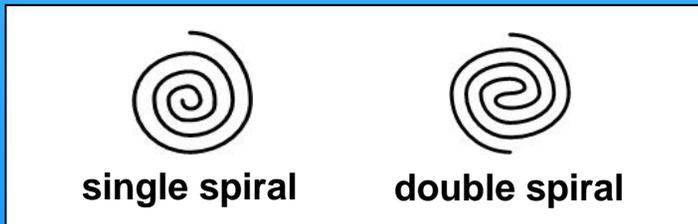
- linear-low-density polyethylene (LLDPE)
- polypropylene (PP)
- Affinity™ polyolefin plastomer (POP)
- polyvinyl alcohol (PVA)
- Santoprene™ thermoplastic vulcanizate (TPV)
- polyurethane (PU)
- polystyrene (PS)

rectangular extrusion die: 13 to 1 aspect ratio
gas injector with up to 19 hypodermic needles



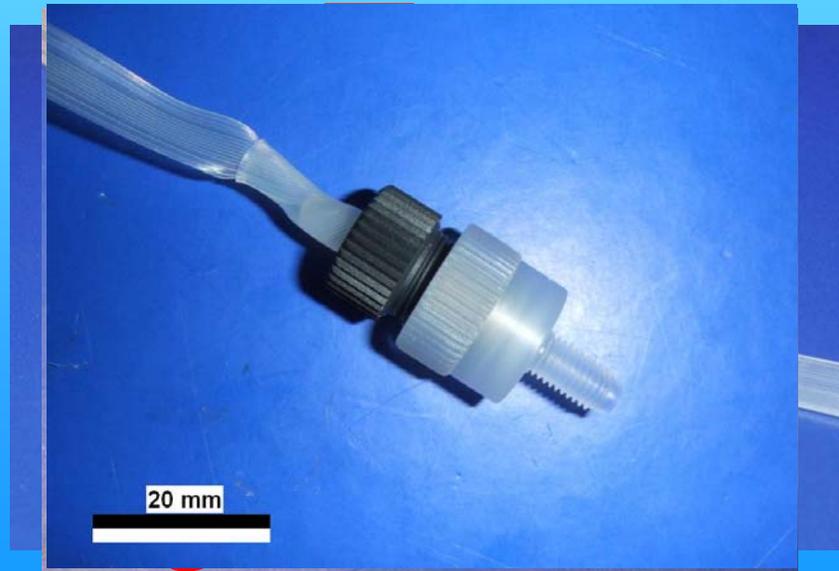
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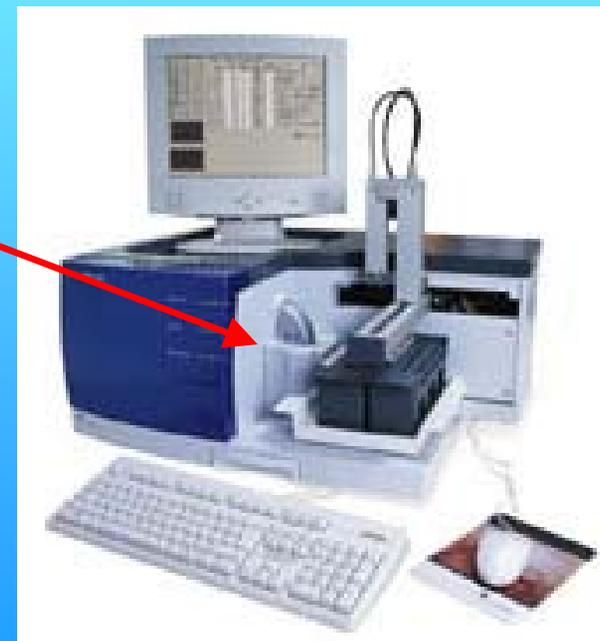


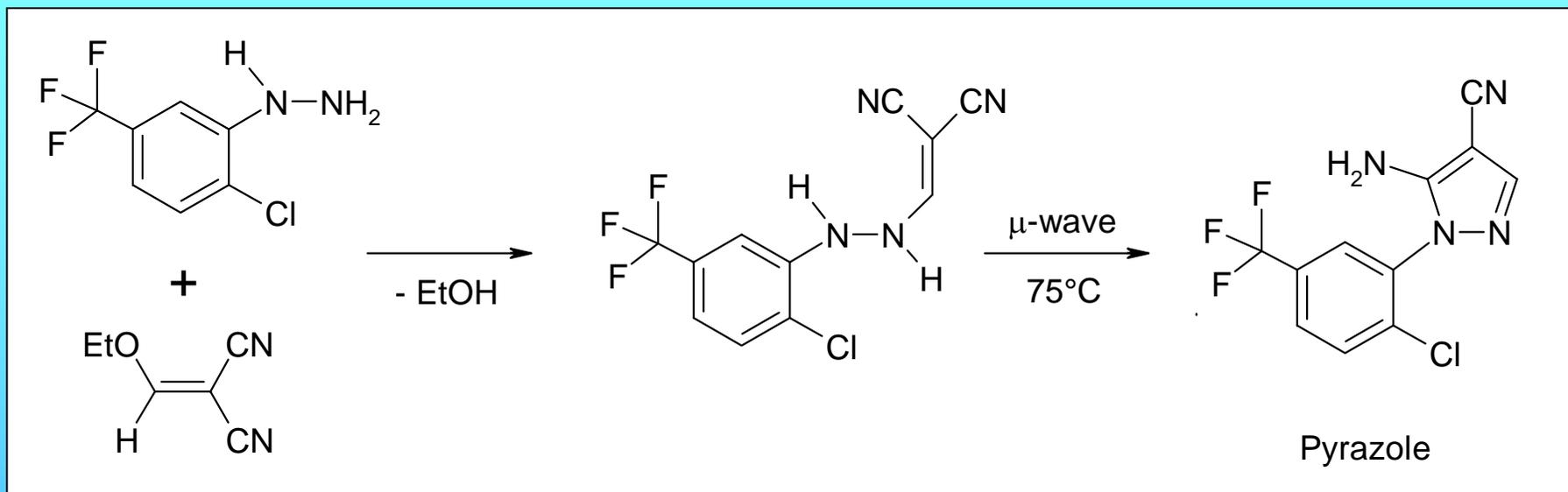


Continuous flow microwave reactor



- microwave synthesizer (Emrys™ Optimizer)
- MCF wound around teflon microwave insert
- 2 cylindrical MCF connectors
- length: 4 m inside microwave cavity (+ 2x 20 cm leads)





Reaction conditions:

$Q = 0.1 - 0.2 \text{ ml/min}$

solvent: MeOH

$\tau = 15 - 30 \text{ min}$

$T = 60 - 75 \text{ }^\circ\text{C}$

15 min / $60 \text{ }^\circ\text{C}$

15 min / $75 \text{ }^\circ\text{C}$

30 min / $75 \text{ }^\circ\text{C}$

→ 39.67 % conv.

→ 64.46 % conv.

→ ~100 % conv.