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INTRODUCTION TO MICROFLUIDICS

8 - 26 August 2005

Part B - Microreactors

Topic 4: Chemical Reaction and Microdevices
Topic 5: The Mixing Problem
Topic 6: Reaction Case Studies

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Topic 4. Chemical Reaction and Microdevices

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ICTP Summer School
Introduction to Microfluidics
Trieste, Italy
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Overview of the lectures

- Types of chemical reaction
- Why microreactors?
- Control of chemical reaction
- Some examples
- The mixing problem (Topic 5)
- Case studies (Topic 6):
  - EK slug flow reactor
  - High throughput ionic liquid reactor
  - Towards distillation and reactive distillation

Types of Chemical Reaction

Chemical reactions fall into a wide range of types according to:

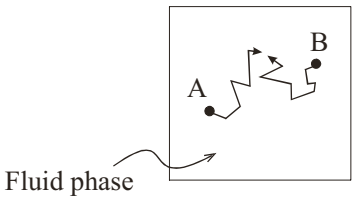
• the phases of matter involved and which chemical species can occupy each of the phases

• how many reactions are involved

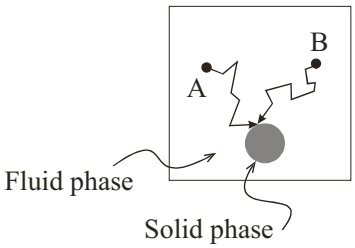
• the orders of the reaction rates

Phases involved in the reaction

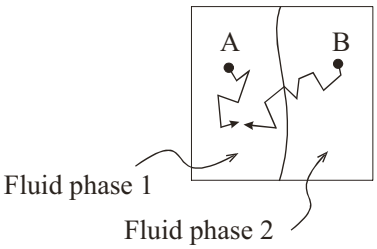
Homogeneous reaction
Reagent and product molecules are all in a single fluid phase (either gas or liquid)



Heterogeneous reaction
Reaction of molecules in a fluid phase requires the presence of a solid material (catalyst)



Biphasic reaction
Reaction involves two separate phases, either gas/liquid or liquid/liquid (immiscible)



Number of reactions

Single reaction: e.g. A + B _ C

Multiple reactions: e.g.
1) A + B _ C
2) B + D _ E
3) A + C _ F

Multiple reactions are divided into:

Series reactions
The product of one reaction is one of the reactants of another (i.e. reactions 3 consumes the product species C of reaction 1)

Parallel reactions
Two reactions consume the same reagent (e.g. 1 and 2 above consume species B)

Order of reactions

Each reaction follows a rate law expressing the rate of consumption or production of a species involved in the reaction. For example, the rate for a species A, R_A, might be expressed as

$$R_A = kC_AC_B$$

where k is the rate coefficient which depends on temperature ; C_A and C_B are concentrations of species A and B.

The ***order of reaction*** is the exponent of the concentrations on which the rate depends. The above reaction is 'first order' in either A or B and 'second order' overall.

Order of reactions (continued)

When two or more reactions compete for the same species, the order of reaction can be used to determine which dominates the conversion of the common species.

At high concentrations, the rate with higher order tends to dominate conversion of the common species.

At low concentrations, the lower order reaction dominates.

Thus, controlling the concentrations of the reactants can be used to optimise the amount of the reactants converted to ***desired product***.

Why use microdevices for chemical reaction?

• Generally, a chemical reaction is important as a way of producing a particular product molecule.

• The efficiency with which the raw materials of the reaction (the reactants) can be converted into the desired products depends on the reaction conditions (temperature, concentration).

• Using a microflow device should allow far greater control of the temperature and composition histories of the reacting fluid elements.

Reaction control

What is the ideal reactor?

- Spatially homogeneous conditions at any given time – all parts of the reaction mixture are at the same uniform conditions (implies also uniformity of material residence time).
- Temperature and concentrations can be varied in time to optimise the reaction.
- Separation of species to remove product, residual or intermediate species from the mixture.

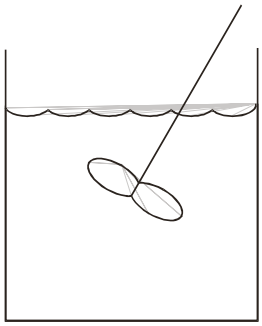
Let us consider the case of reactor types on a conventional (macroscopic) scale:

1) Batch reactor
2) Well-stirred reactor
3) Tubular reactor

Batch reactor

Reagents are added to a vessel and allowed to react in time.

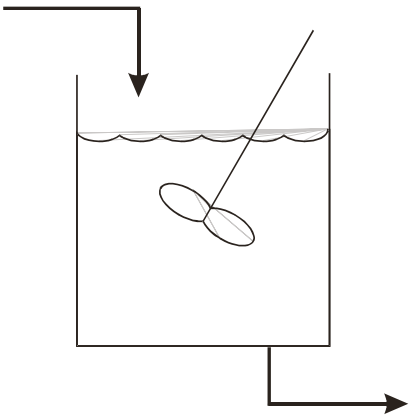
Possible to heat or cool and to change composition by addition of new material in time.



• Non-uniform conditions during the initial period of mixing (mixing time L/V must be small).
• Also, non-uniformity present when reaction time scale is comparable to or smaller than the mixing time.
• Addition of material possible; separation of species not.

Well stirred reactor

Reagents are added continuously to a vessel and allowed to react in for the residence time.



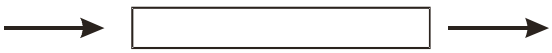
Reactor operates in a steady state.

Inlet composition can vary with time.

- Non-uniformity present when residence time scale is comparable to or smaller than the mixing time.
- Addition of material possible; separation of species not.
- There will be a distribution of residence time: different elements of entering material experience different reaction times.

Tubular reactor

Reagents are added continuously to a conduit and allowed to react during the residence time.



Inlet composition can vary with time.

- The comments made in relation to the well-stirred reactor all apply.
- Mixing though is dependent on the flow state (turbulent or laminar flow) and conduit geometry presence or absence of ribs or baffles.

Challenges for microreactor approaches

Reaction control requires some fundamental issues to be resolved:

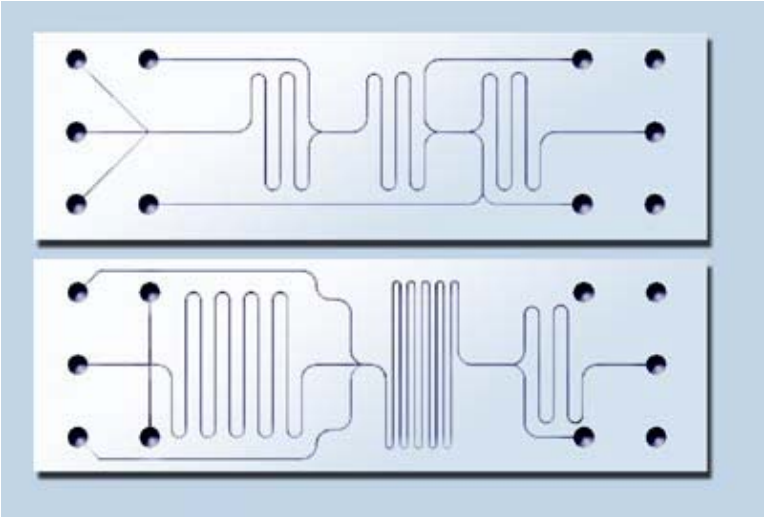
- Rapid and complete mixing must be achieved
- Subsequent mixing with secondary streams to alter composition
- Thermal control to impose a temperature history
- Accessing the result – analysis or product separation
- Avoiding a broad distribution of residence time

Examples of microreactors

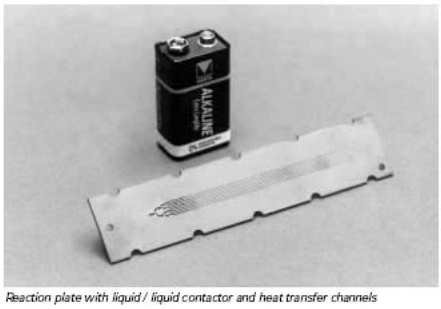
Examples in following slides:

1) Microchannel (tubular reactor)
2) Two-phase reactor of Song
3) Biphasic reactor prospects
4) Quake PDMS integrated devices

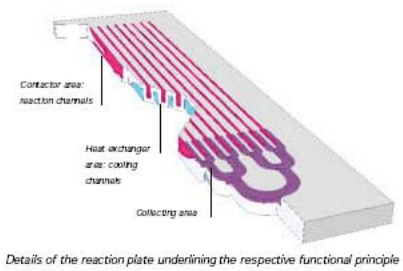
Simple channel reactor
(Micronit, Netherlands)



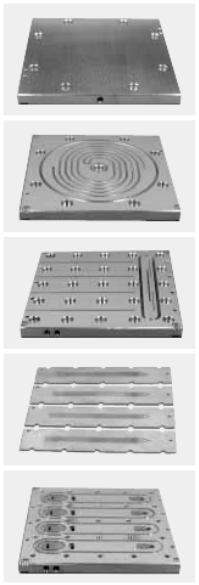
Liquid microreactor, IMM, Mainz, Germany



Reaction plate with liquid / liquid contactor and heat transfer channels



Details of the reaction plate underlining the respective functional principle



Dispersion and residence time distribution

Residence time distribution, short time

For an axisymmetric channel and negligible diffusion

Probability density function $f = \frac{1}{Q}\frac{dQ}{d\tau}$, where $\int_{\tau_{\min}}^{\infty} f d\tau = 1$

$\tau = \frac{L}{u}$ is the residence time with minimum being $\tau_{\min} = \frac{L}{2V}$

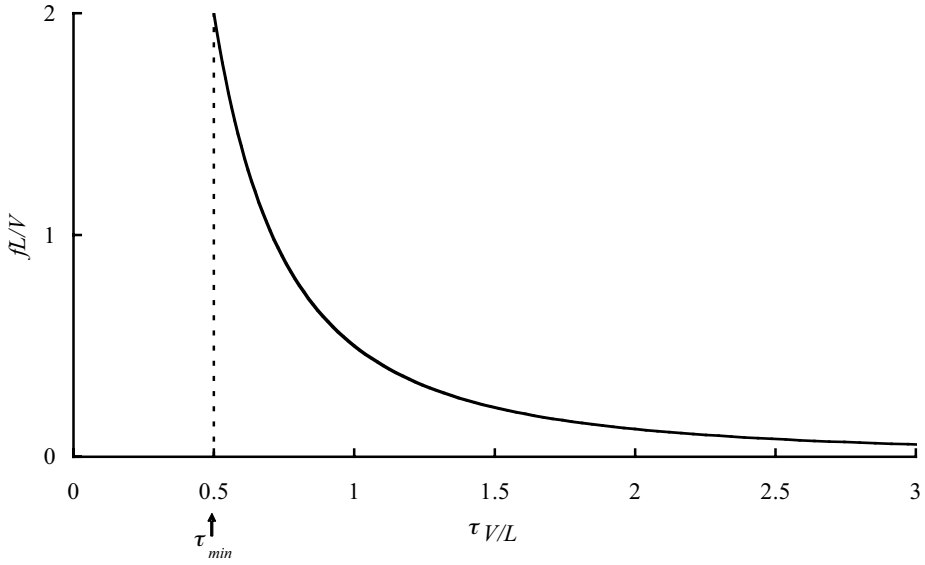
f can be determined from these relations using the known velocity profile for developed flow:

$$u = 2V\left(1 - \frac{4r^2}{D^2}\right)$$

Which gives, in non-dimensional form:

$$\frac{fL}{V} = \frac{1}{2}\left(\frac{L}{V\tau}\right)^2$$

Residence time distribution (Axisymmetric channel, no diffusion)



Residence time distribution, long time

At long times, diffusion acts to reduce to dispersion of fluid.

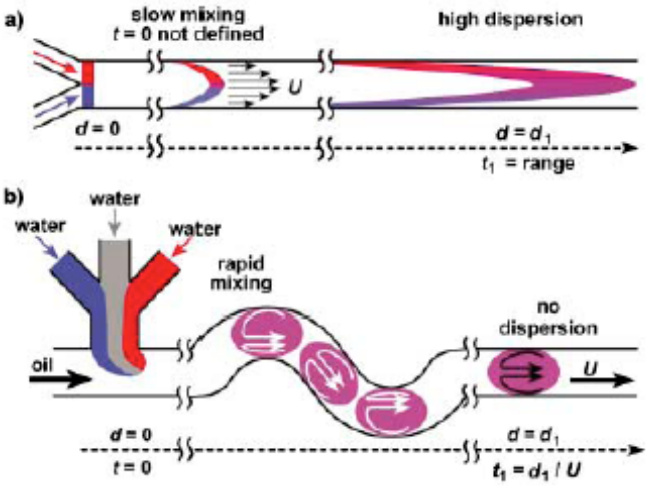
Molecules in the slow fluid near the walls diffuse into faster outer fluid so it can 'keep up' better with the central fluid; molecules in the fast core of the flow diffuse into slower fluid and thereby do not 'run away' so much.

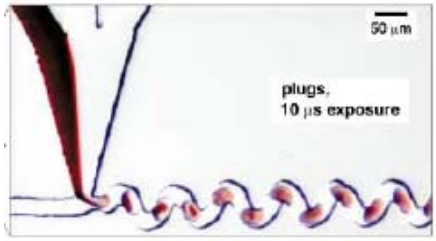
Taylor (1953) showed that an initial section of fluid will have a Gaussian distribution of concentration along the axial direction after flowing a mean distance L downstream. The standard deviation of the position of the original fluid from L given by

$$\sigma_x = \left\{ \frac{d^2 VL}{96D} \right\}^{1/2} \qquad \text{for} \quad \frac{L}{V} >> \frac{d^2}{28.88D}$$

Immiscible liquid drops as reactors

Song et al., 2003, Angew. Chem. Int. Ed., 43, 767-772.

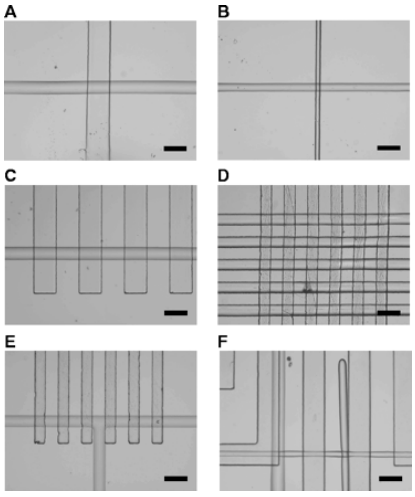


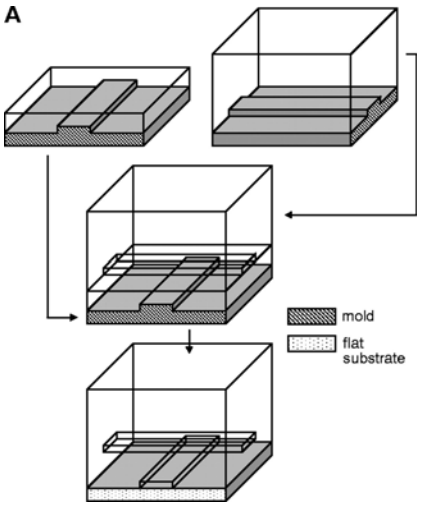


Quake's group (CIT, Stanford)

Complex chips fabricated using moulded PDMS

pneumatic valve close off channels

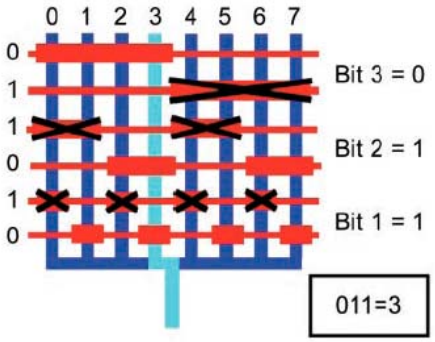




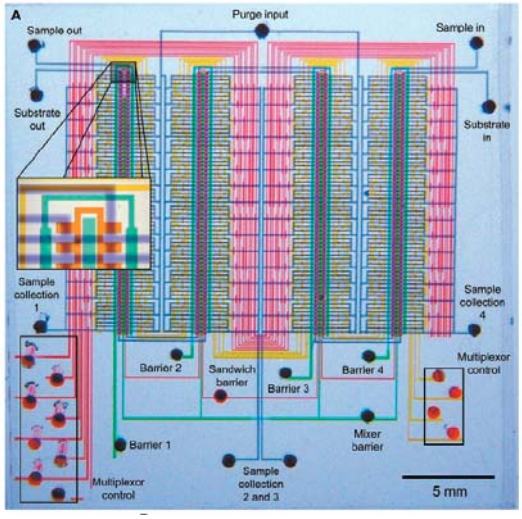
Unger et al., 2000, Science, 288, 113-116.

Control

‘Multiplexor’

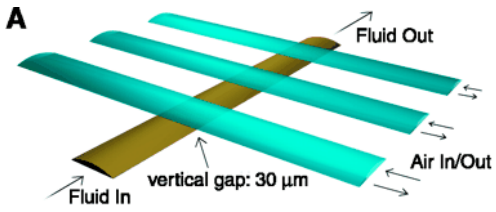


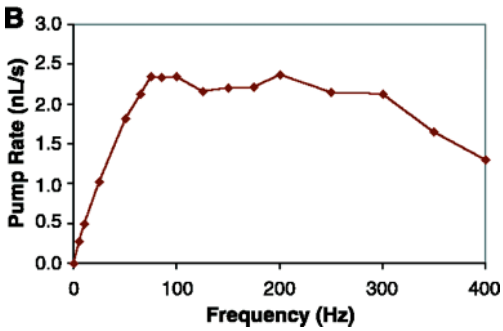
Example device



Todd et al., 2002, Science, 298, 580-584.

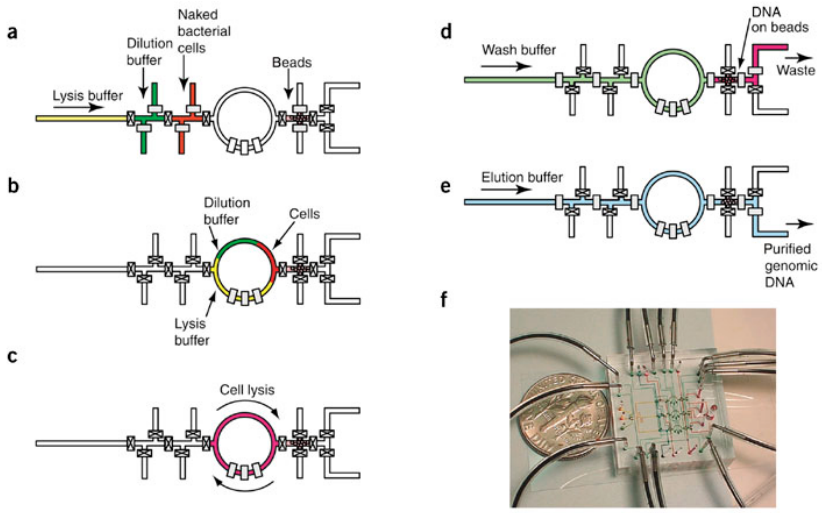
Pump (peristaltic)





Unger et al., 2000, Science, 288, 113-116.

Mixer (‘race track’)



Hong and Quake, 2003, Nature Biotechnology, 21, 1179-1183.

Summary

Simple channel reactor

• Mixing dependent on diffusion (slow)

• Large residence time distribution (Electrokinetic flow could be used to eliminate this problem)

Integrated PDMS approach

• Mixing and sequencing

• Pressure limited by soft material

• Chemical compatibility with PDMS

Immiscible liquid drop approach

• Single phase reaction must be compatible with carrier phase

• Biphasic reactions possible

Topic 5. The Mixing Problem

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Outline

- Background
- Measure of mixing
- Types of mixers
- Channel mixer
- Direct mixers
- Alternating flow mixer
- Folding flow mixer
- Folding network mixer

Background

- Liquid mixing in microscale flows is surprisingly difficult
- Reynolds number is usually low, Peclet number high
- Many different approaches have been proposed
- Which one is best?

One has two requirements:

- Mixture uniformity
- Time allowed for mixing

Criterion of comparison:

- Chip space required
- Cost/complexity/practicality
- Pressure drop

A measure of mixing

Relative rms deviation: $\sigma_Y \equiv \frac{\sqrt{\overline{(Y-\bar{Y})^2}}}{\bar{Y}}$

- Y is concentration variable (e.g. mass fraction or molar concentration)
- Over bar notation indicates averaging operation

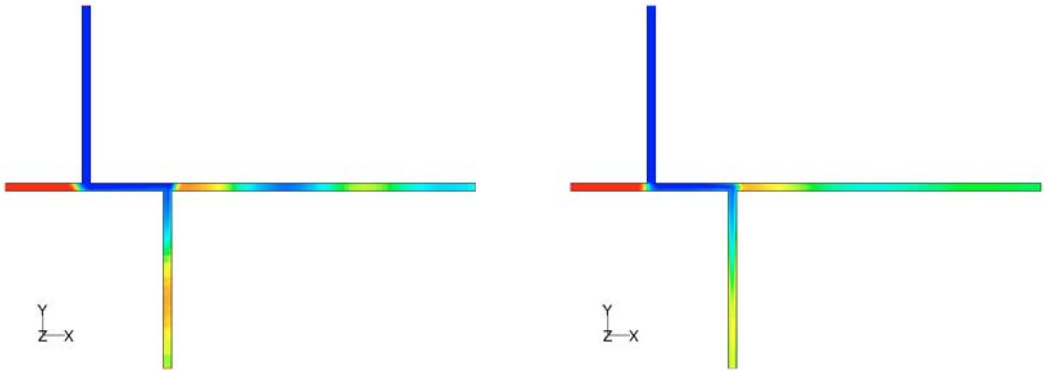
Averaging can be either volume or flux weighted
Typically carried out over a flow section surface S

Volume average: $\bar{Y} = \frac{1}{S}\int_S Y dS$

Flux average: $\bar{Y} = \frac{1}{Q}\int_S uY dS$ where $Q = \int_S u dS$

(Volume averages will be assumed.)

Difficulty of mixing– some examples to illustrate the problem



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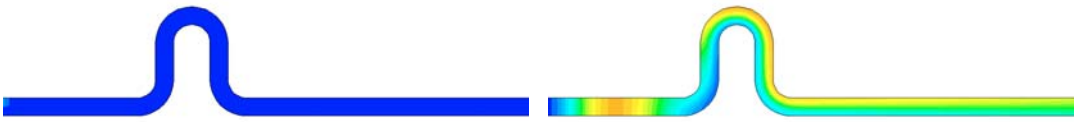
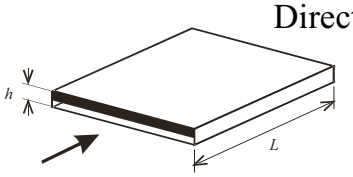
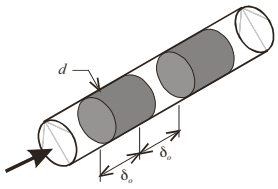


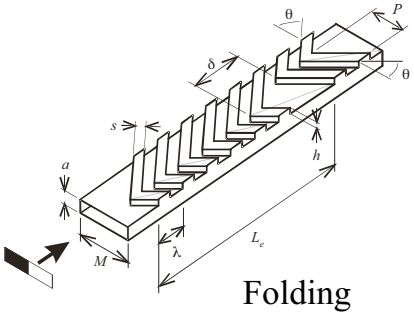
Mixing at low Reynolds number

Direct

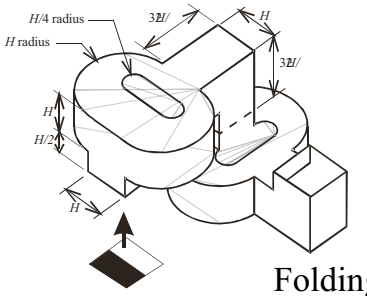




Alternating
Flow
Mixer (AFM)

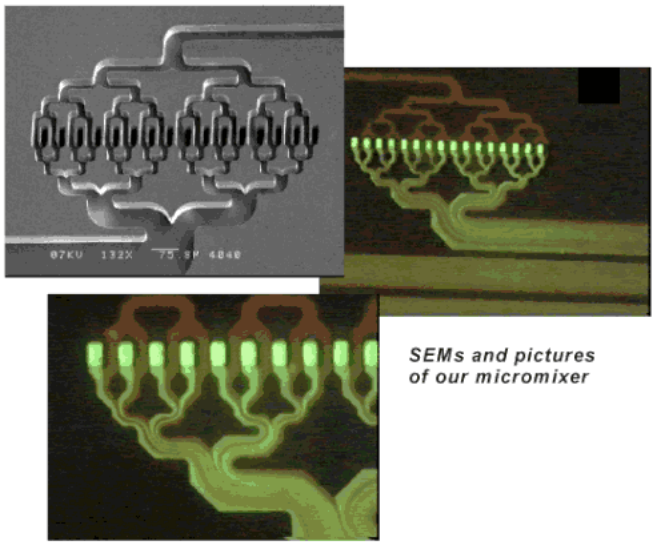


Folding
Flow
Mixer (FFM)



Folding
Network
Mixer (FNM)

Example of the ‘direct’ method



Striation thickness 20 μm
(size of smallest channels)

Mixing time
up to 10 min for low
diffusivity liquids

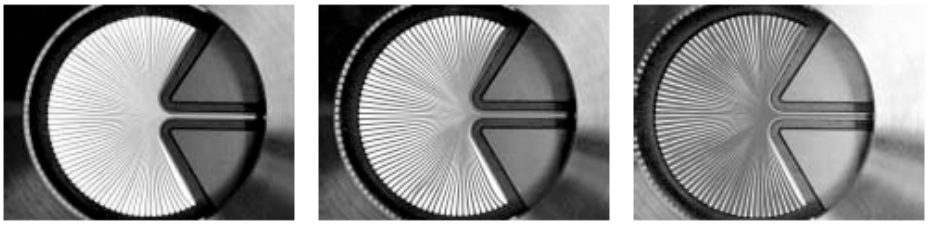
$$\tau_W = W^2/D$$

Small geometric tolerances
are critical to achieving
uniform mixing

May be prone to clogging

(Bessoth, de Mello and Manz, Anal. Comm., 1999)

Another example:
'Superfocus' mixer, IMM, Mainz, Germany

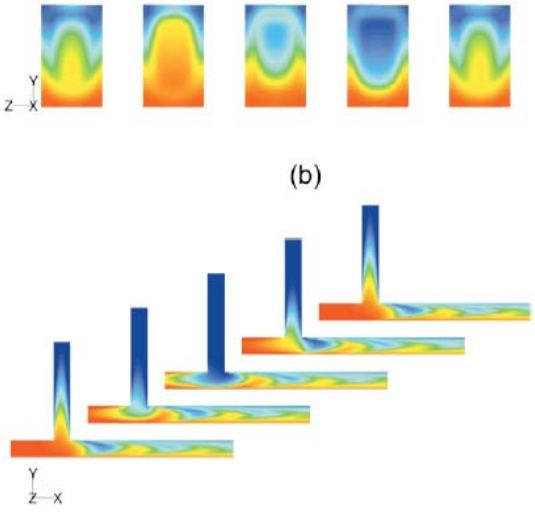


- Images show different relative flow rates for the two liquids
- Liquids can be mixed in milliseconds (excluding time spent in the focusing section!)
- Important that the fluids are injected uniformly side by side (diagram 'a' shows uniformity over depth; 'b' does not)

(a) (b)

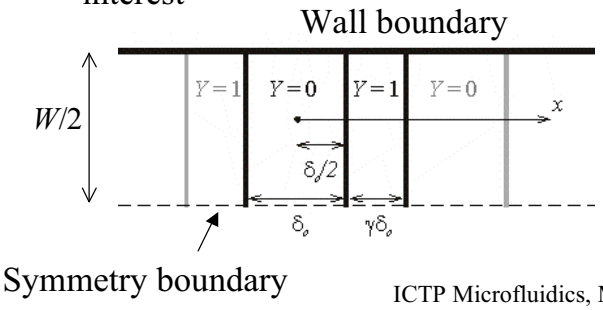
Alternating flow mixing (AFM)

Glasgow and Aubry, 2003, Lab on a Chip, 3, 114-120



Alternating flow mixing (AFM)

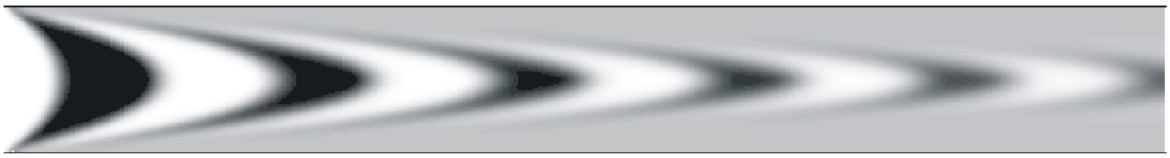
MacInnes et al. (2005) investigated this type of mixing in detail
• Idealised inlet used (with lateral uniformity of fluid distribution)
• Transformation from spatial development of mixing to temporal development necessary to allow accurate solutions
• Wide range of parameters explored in the computations: axisymmetric and planar channels, slug length, slug length ratio and the Peclet number
• rms deviation from perfect mixture determined and a correlation model derived to represent it over entire range of parameter values of interest



(MacInnes, Chen and Allen, Chem. Eng. Sci., 2005)

Time-evolving analogue

Spatial evolution of mixing



x_1 x_2 x_3 x_4

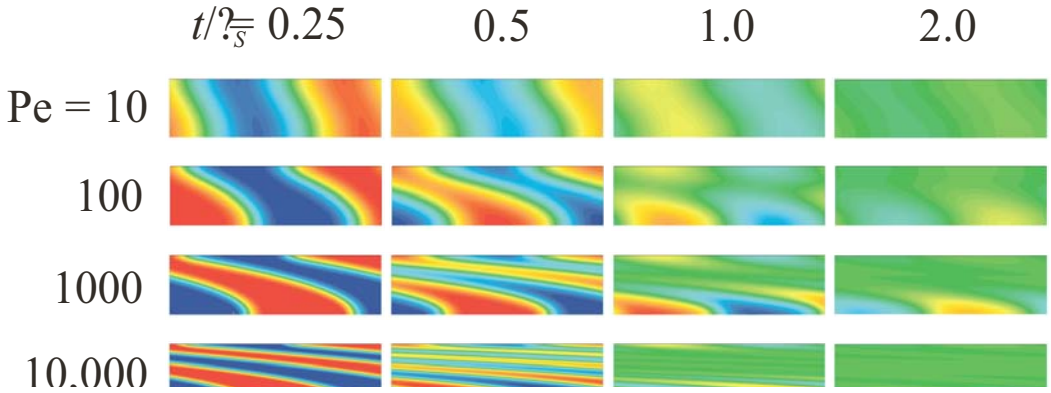
Temporal evolution of mixing

t_1 t_2 t_3 t_4

Transformation between temporal and spatial cases using: $x = V_{\max} t$

Computed concentration contours

Results should scale with the 'strain-rate' mixing time $\frac{\tau_S}{\tau_W} = \left(\frac{3}{32} \frac{(1+\gamma)\delta_o / W}{\mathrm{Pe}} \right)^{2/3}$

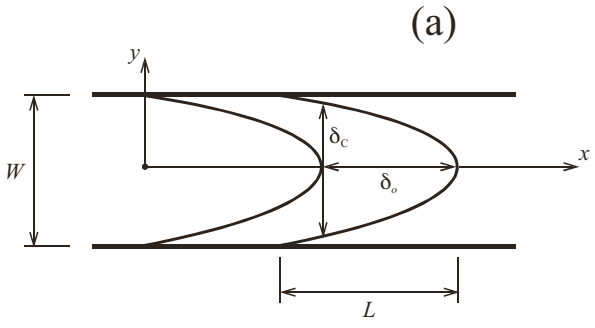


Mixing time scaling is $\tau_\sigma \propto \mathrm{Pe}^{-2/3}$ where $\mathrm{Pe} = \frac{VW}{D}$

Computed concentration contours

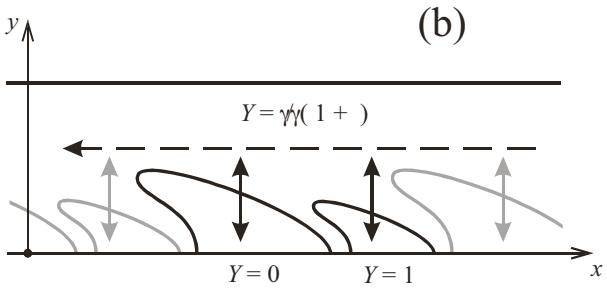
Final stage of mixing depends on:

• width of the unmixed core and the rms deviation of the core at the end of the strain-rate mixing time

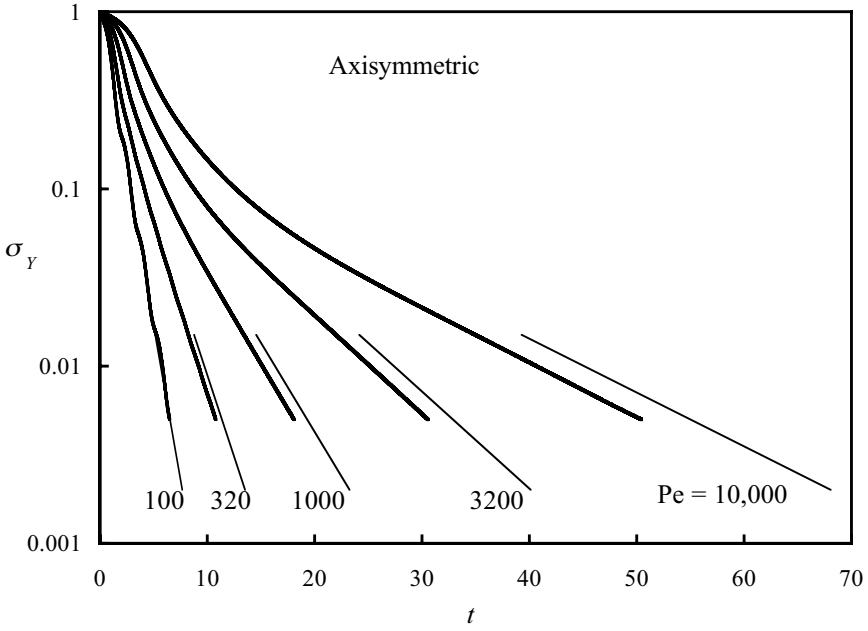


• involves a process of diffusion between the core and the slower fluid adjacent to the core

Mixing time scaling: $\tau_\sigma \propto \mathrm{Pe}^{-1/2}$



Computed decay of mixing deviation



Pressure drop required (axisymmetric channel)

Correlation model of MacInnes et al. (2005):

$$\sigma_Y = 0.6\sigma_{Yo}\exp\left\{-2.33\left(\frac{\tau_\sigma - \tau_S}{\tau_M}\right)\right\} \qquad \text{(core mixing stage)}$$

$$\frac{\tau_S}{\tau_W} = \left(\frac{3}{16}\frac{\delta_o}{W}\frac{1}{\mathrm{Pe}}\right)^{2/3} \qquad \frac{\tau_M}{\tau_W} = \frac{1}{32}\frac{W}{\delta_o}\frac{1}{\mathrm{Pe}}\left\{1-\sqrt{1+\sqrt{128\left(\frac{\delta_o}{W}\right)^3\mathrm{Pe}}}\right\}^2$$

$$\sigma_{Yo} = \frac{2^{3/2}}{\pi}\frac{\delta_C}{W}\exp\left\{-\pi^2\frac{\tau_S}{\tau_W}\left(\frac{W}{\delta_o}\right)^2\right\} \qquad \frac{\delta_C}{W} = \sqrt{\frac{1}{2}\frac{\delta_o}{W}\frac{\tau_W}{\tau_S}\frac{1}{\mathrm{Pe}}}$$

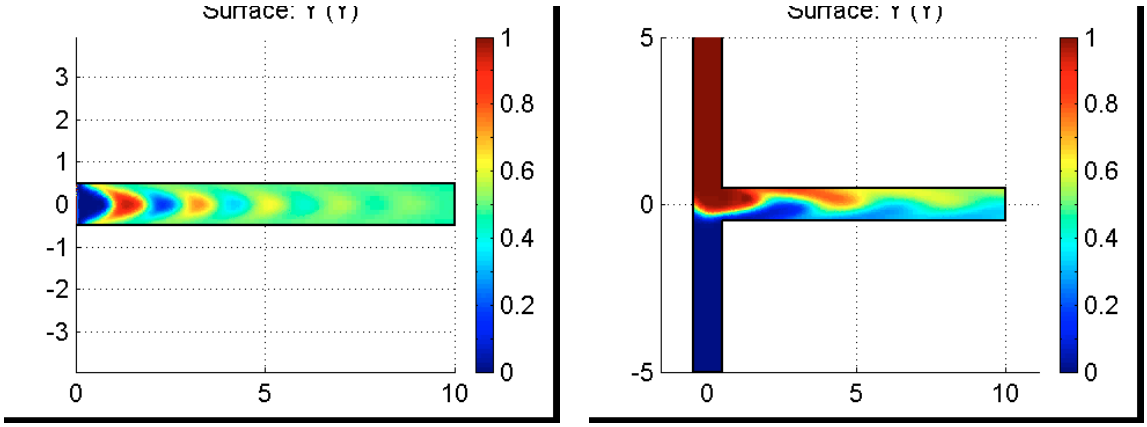
Can be solved for Pe in terms of specified σ_Y , τ_σ/τ_W and δ_o/W .

Then non-dimensional pressure drop is determined: $\dfrac{\Delta p \tau_W}{\mu} = 64\mathrm{Pe}^2\dfrac{\tau_\sigma}{\tau_W}$

Mixing will be worse for practical conditions

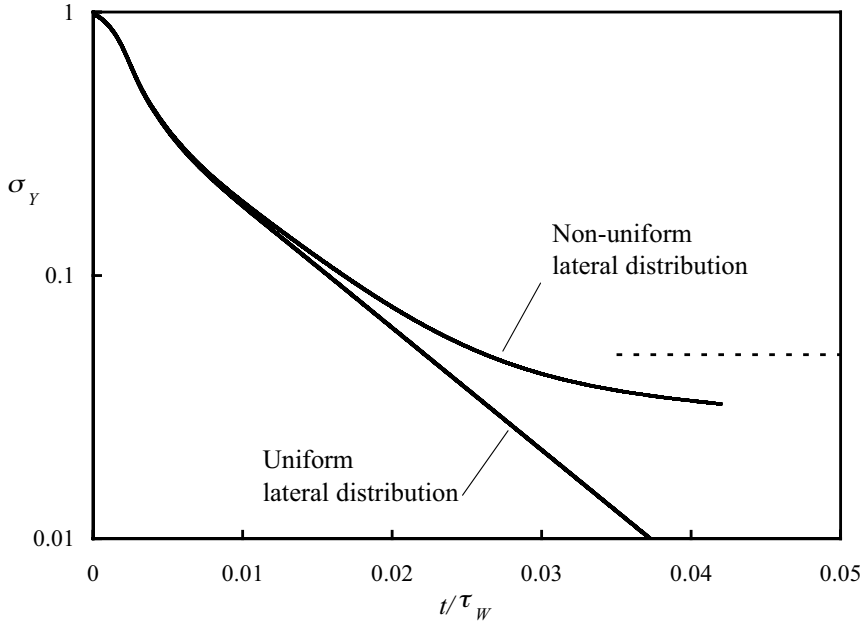
Ideal with uniform lateral injection

Non- uniform lateral injection



(Unpublished computations)

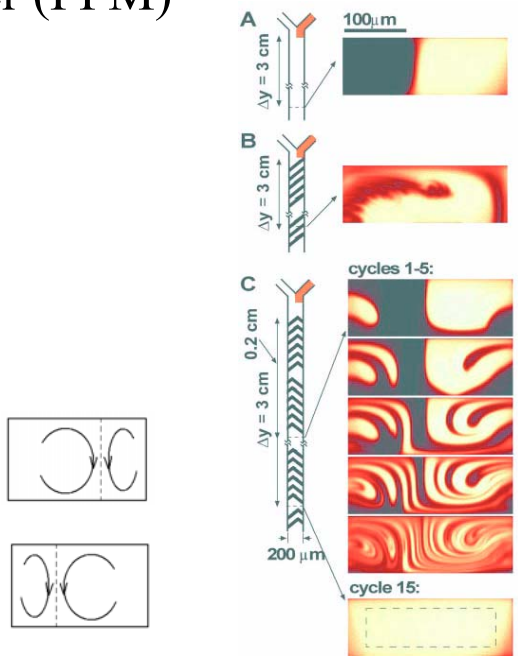
Result of an initial 5% lateral non-uniformity



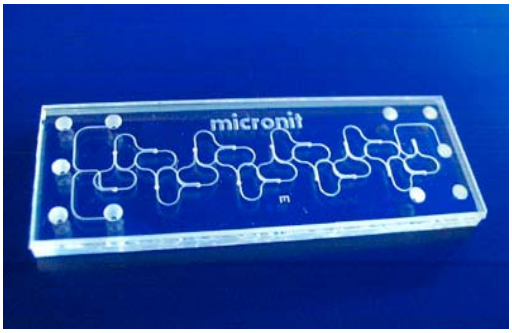
Folding flow mixer (FFM)

Staggered Herringbone Mixer (SHM)
(Stroock et al., 2002)

Rib patterns produce cycles of
alternating recirculation flows:

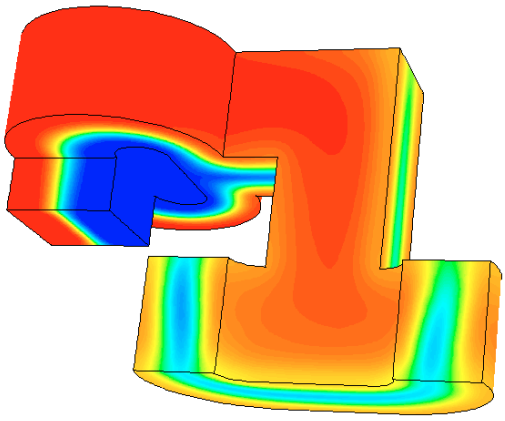


Folding network mixer (FNM)



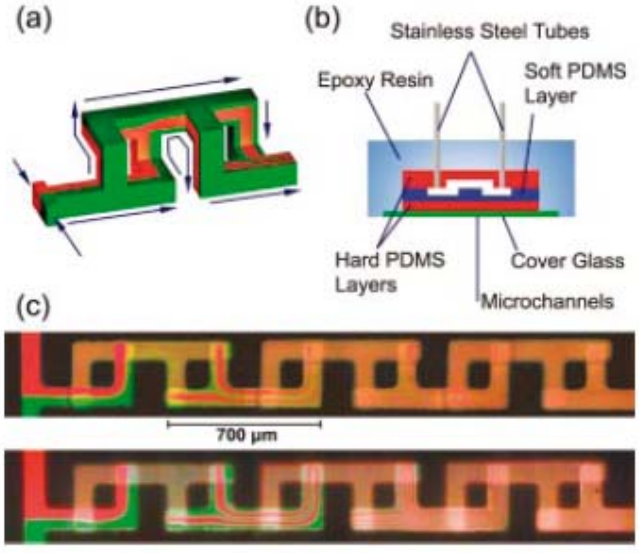
Uses a sequence of (1) dividing, (2) turning
and (3) stacking layers

Folding network mixer (FNM)



Concentration contours plotted on the channel wall.
(MacInnes and Allen, 2005, WCCE)

Folding network mixer (FNM)



Chen and Meiners, 2004, Applied Physics Letters, 84, 2193-2195

Determining performance of either FFM or FNM

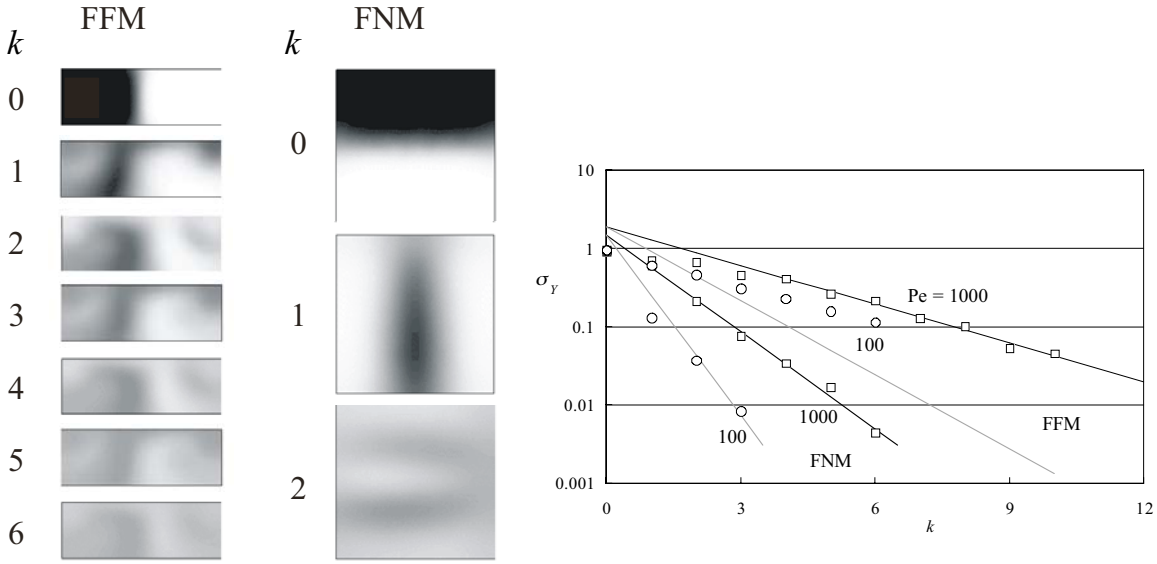
1) CFD computations at low Peclet number (100 and 1000)

2) Scale model to extend results to high Peclet number:

$$\frac{\sigma_Y}{\sigma_{Yo}} = \exp\left\{-\frac{\alpha k}{\ln(\mathrm{Pe}) - \ln(L_e/W)}\right\} \qquad (\ \tau_\sigma \propto \mathrm{Pe}^{-1}\ )$$

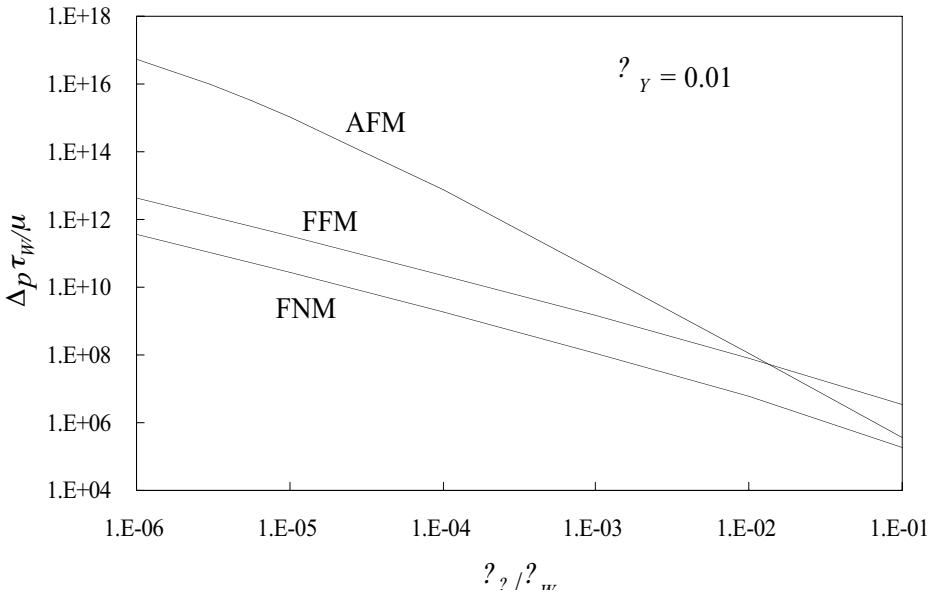
Non-dimensional pressure drop: $\dfrac{\Delta p \tau_W}{\mu} = \beta \dfrac{L_e}{W}\dfrac{\tau_W}{\tau_\sigma} k_\sigma^2$

Scaling to large Peclet number



Concentration contour plots

Pressure drop to produce a given reduction in mixing time τ_σ/τ_W



An example case with some numbers!

Example:

100 μm channel

$\sigma_Y = 0.01$ and $\tau_\sigma = 10$ ms

μ = 0.03 Pa s, $D = 10^{-11}$ m^2/s

FNM Δp = 6 bar

FFM Δp = 90 bar

Direct mixing requires:

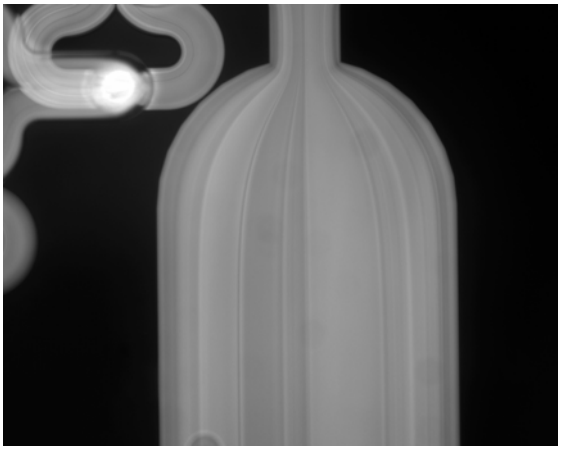
$W = 300$ nm

Application: reaction test chip design

$\Delta p = 4$ bar $\quad \tau_\sigma = 100$ ms

$\sigma_Y = 7\%$

Fabrication: Micronit Microfluidics bv



Conclusion

- Successful design of liquid mixing channels for microflow devices requires great care.
- Folding network mixers appear to be most effective for the common low Re mixing regime of microflows.
- A combination of full solution of governing equations (CFD) has been used with simpler modelling to quantify performance of the folding-type mixers.
- High Reynolds number, but still laminar, flows (say in the range $50 < Re < 1000$) remain to be considered. Here one can take advantage of upstream-downstream recirculation flow.

Topic 6. Reaction Case Studies

Jordan MacInnes
Chemical and Process Engineering
University of Sheffield

ICTP Summer School
Introduction to Microfluidics
Trieste, Italy
August 8-26, 2005

Outline

1) High throughput test reactor
2) Electrokinetic slug-flow reactor
3) Distillation and reaction

1. High Throughput Test Reactor

Project aim: automated testing of reaction in ionic liquid solvents.
• Reduced in reaction times
• Improved selectivity of desired components

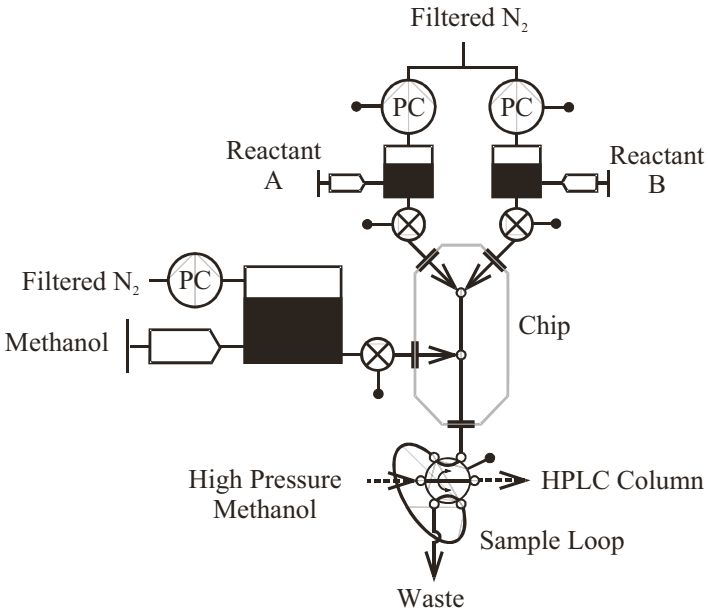
Ionic liquids are room temperature molten salts.
• Zero vapour pressure
• Many different anions and cations are possible and these can be mixed – allowing adjustment to suit a particular reaction

Initial aim is to develop a test reactor system
• Accurate composition measurement requires off chip HPLC analysis

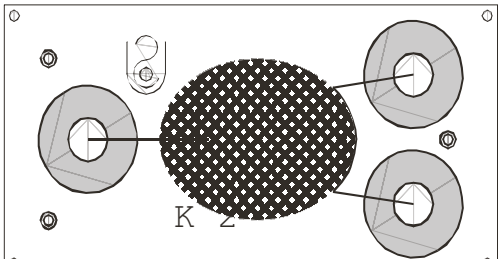
Principal requirements:
• Automated testing with variation of mixture composition and reaction time (possible linking with a search algorithm)
• Reaction must take place under spatially homogeneous conditions
• Accurate measurement of reactant and product species (HPLC)

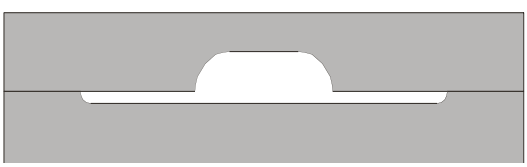
Technical issues:
• Rapid mixing must be achieved (determines precision with which the start time of chemical interaction is known)
• Stopping the flow must be achieved so mixture remains spatially uniform
• Rapid quenching of reaction (by dilution) must be possible
• Sample integrity must be maintained in moving the reacted material from the reactor to the HPLC sample loop
• Ionic liquids have high viscosity and very low diffusivity

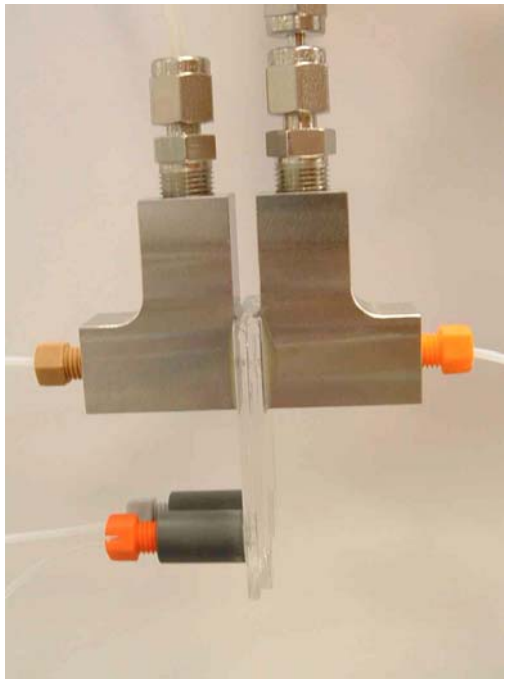
Reaction Test System – Concept



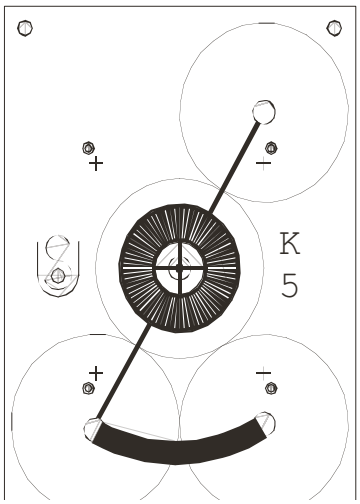
Chip designs that did not work

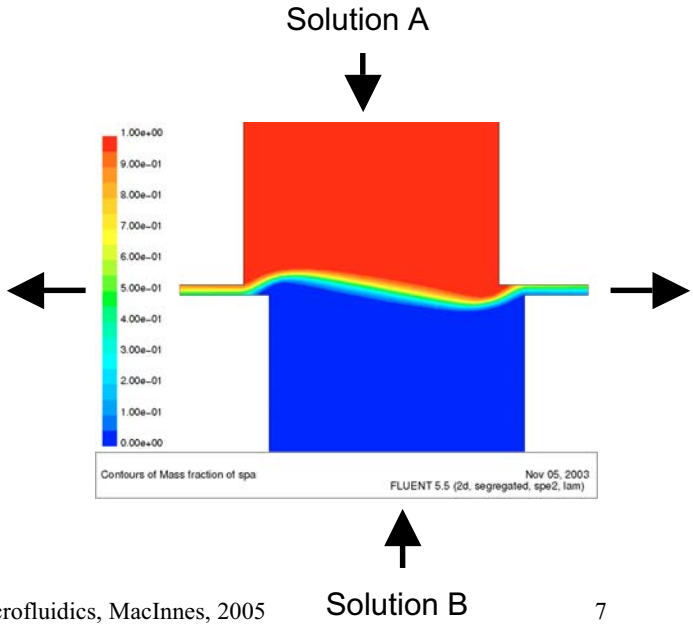




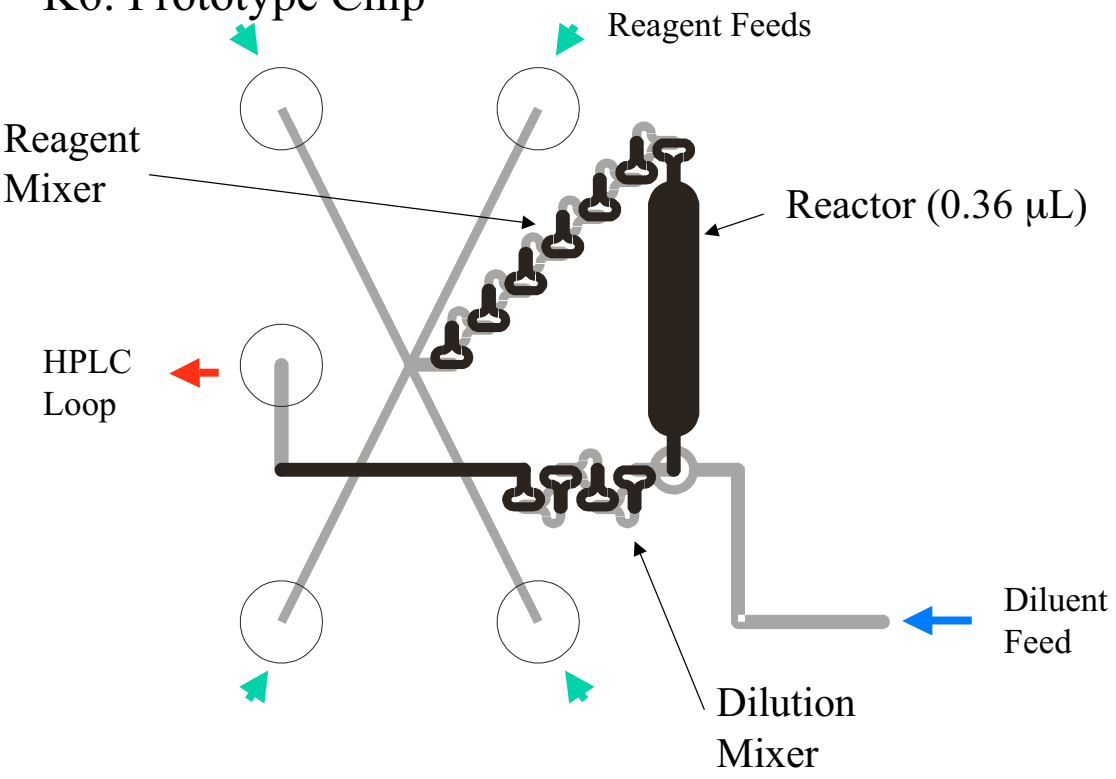


Chip designs that did not work

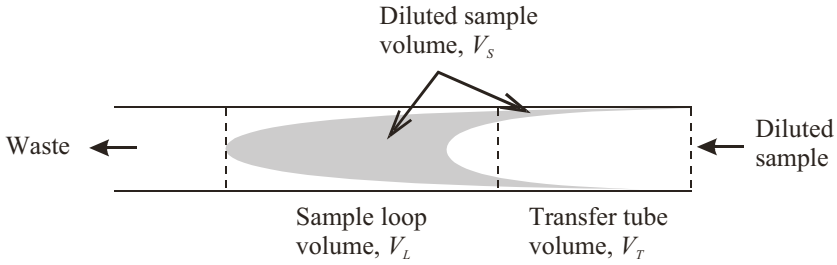




K6: Prototype Chip



Simulation of sample capture



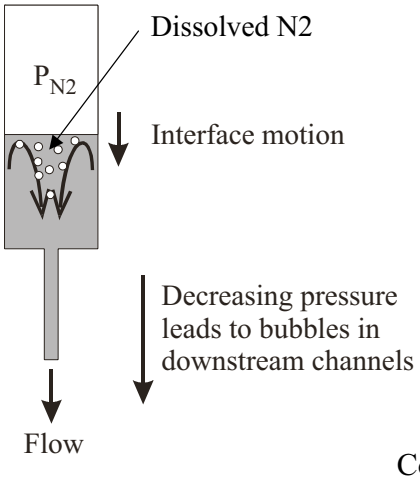
Approximate model for transport of quenched sample to loop
- Uniform diluted sample emerging from the dilution mixer
- Series of different-sized tubes replaced by a single 800 μm dia. tube
- Developed flow (no effect from tube size transitions)

Maximum fraction collected of ejected reactor material given by:

$$f = \frac{V_L(V_L + 2V_T)}{(V_L + V_T)(V_L + V_T + 2V_S)}$$

Provided the sample loop volume is relatively large f is near unity.

N_2 saturation in the reservoirs

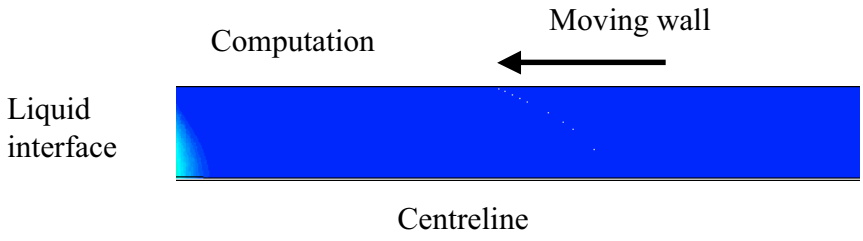


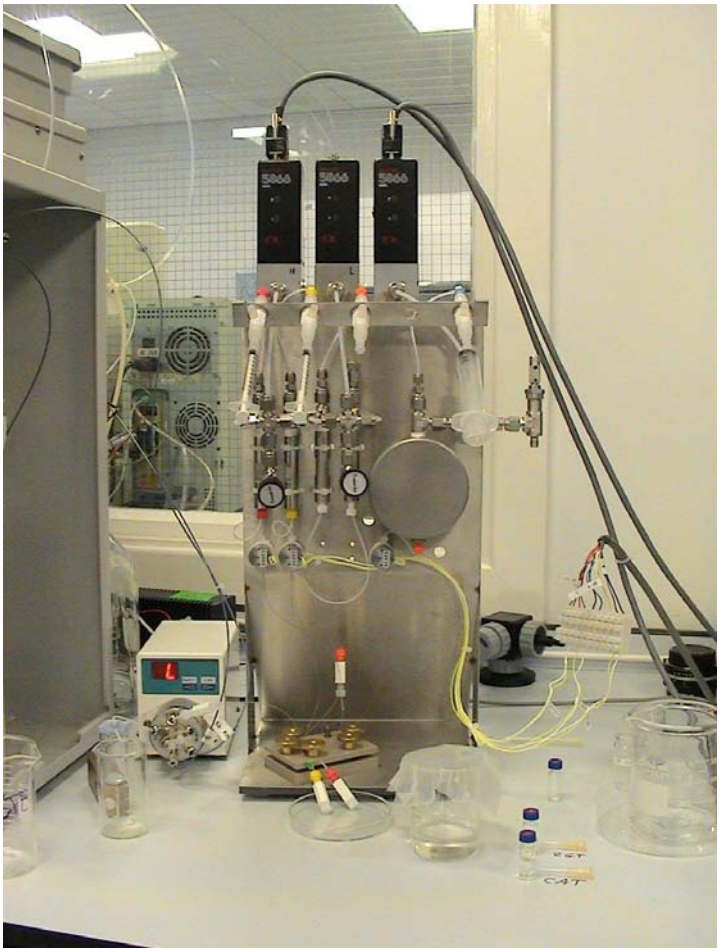
Saturated region becomes accentuated when flow from the reservoir occurs

Worst case is steady flow from reservoir

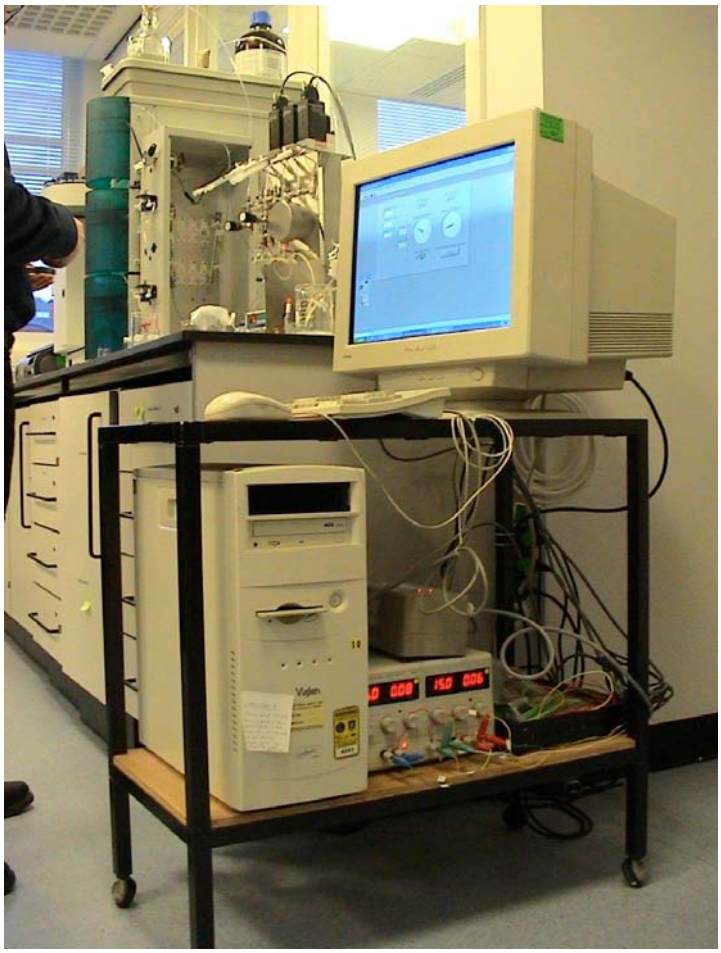
Experiment and computation give similar value for arrival of saturated liquid at the reservoir bottom (outlet).

- 70% of full reagent reservoir can be used
- 50% of diluent reservoir can be used



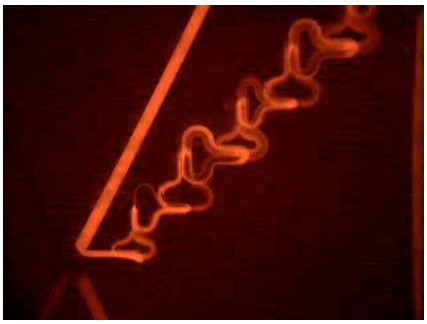


11

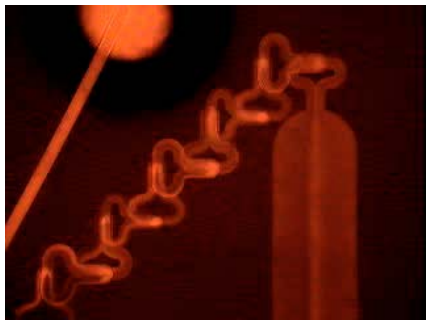


12

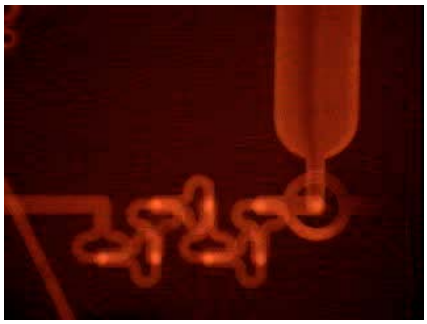
Fluorescent dye (Rhodamine B) visualisations



Reagent mixing

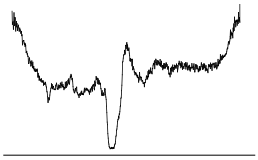


Reaction chamber



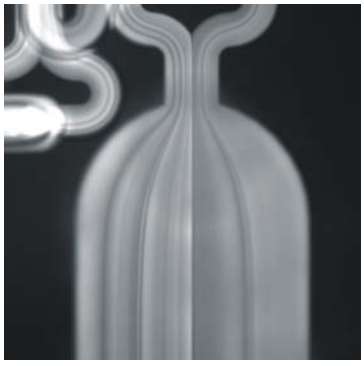
Ejection and Dilution

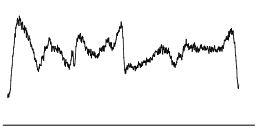
K6 #2



$\sigma_Y = 30\%$

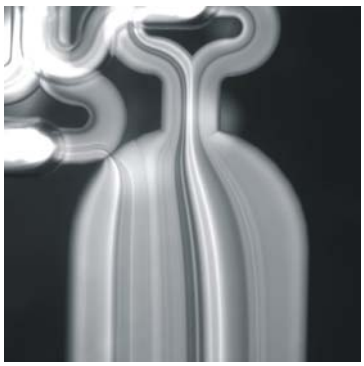
K6 #5

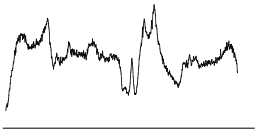




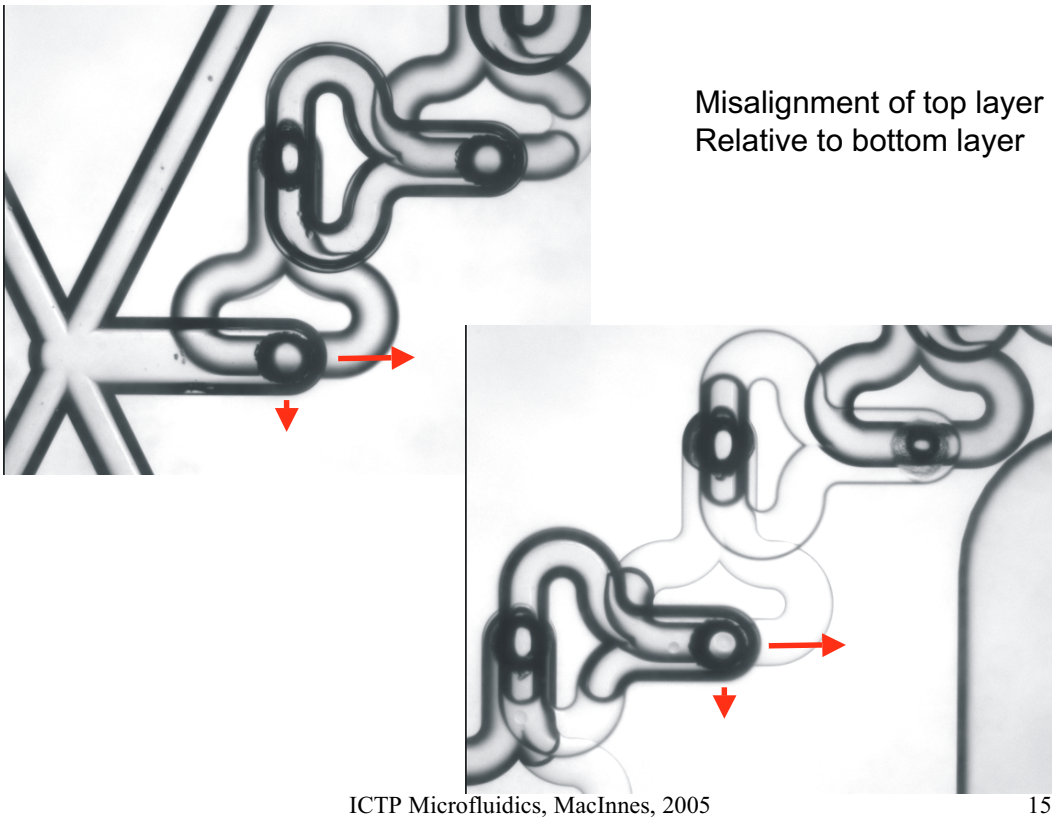
$\sigma_Y = 17\%$

K6 #6



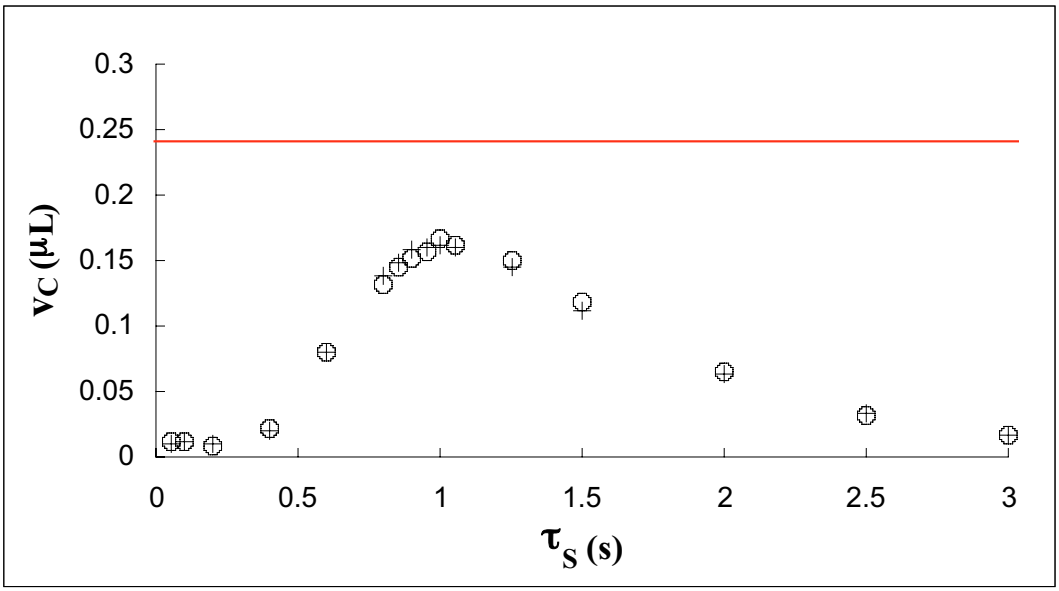


$\sigma_Y = 24\%$

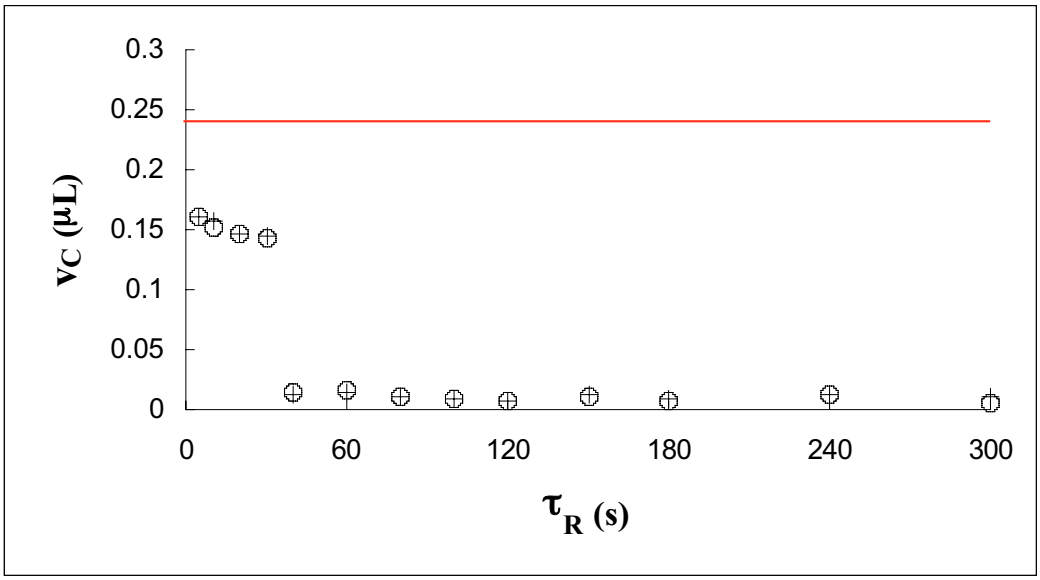


Misalignment of top layer
Relative to bottom layer

Determining loop switching time and maximum sample captured

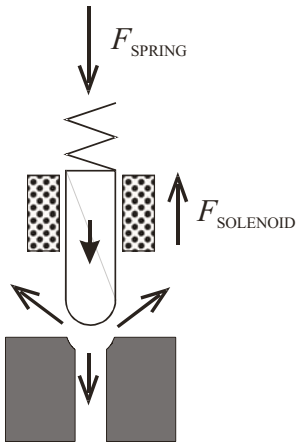


Dependence of ejected volume on reaction time

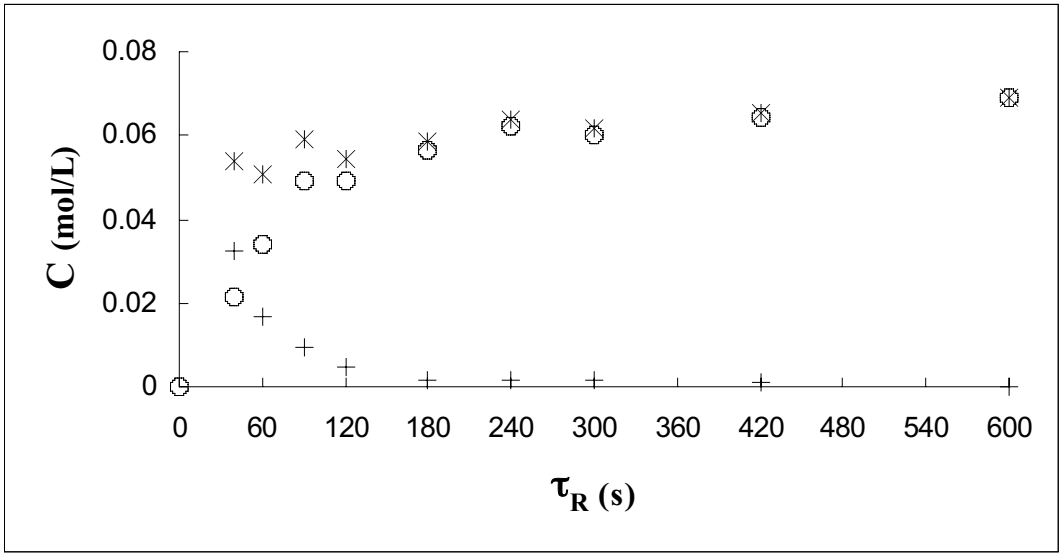


Valve dynamics

- The sample volume captured varies depending on how long the valve has been allowed to close prior to opening for ejection.
- For long times the valve pintle has settled to within a small distance of the seat (leaving less than perhaps a few micron gap).
- This behaviour is believed to be related to the resistance of filling the very narrow gap between the pintle and the valve seat.
- May be necessary to find more appropriate types of valves, e.g. ‘sliding’ valves.



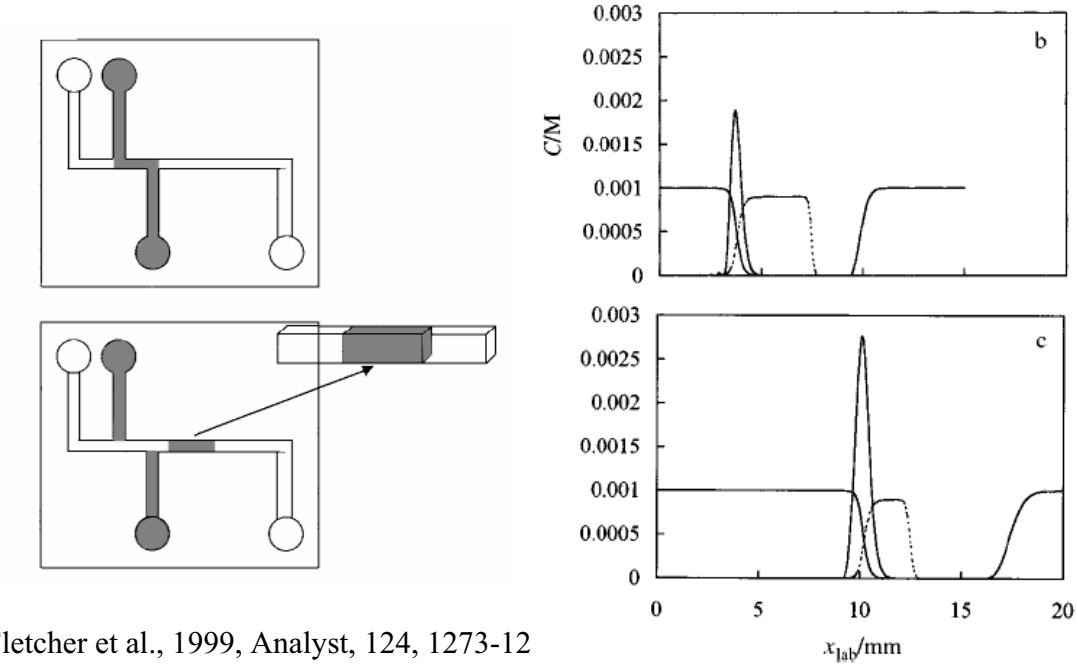
Example results (reaction NBA + DHF _ THQ)
Measured NBA (+) reactant, THQ (o) product and
NBA + THQ (*) against reaction time.



Summary

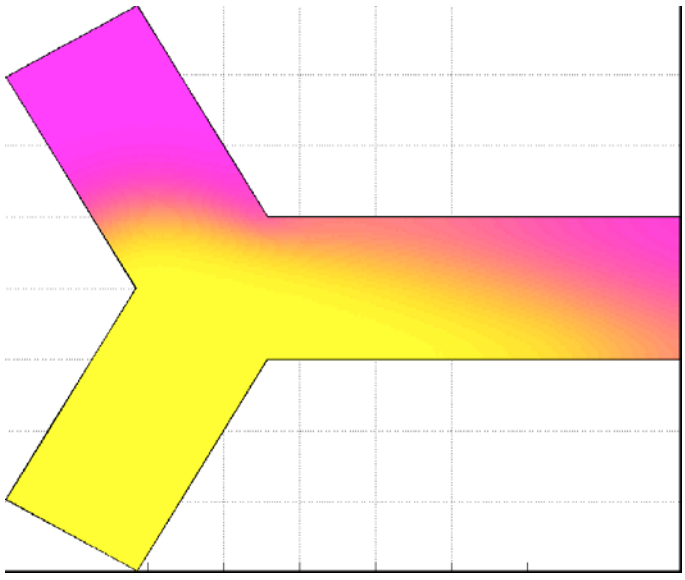
- Mixing has proved a crucial step in developing the successful prototype test reactor
- The device has been operated for long periods of time with no difficulties
- A duplicate device has been delivered to the leading ionic liquids chemistry group of K. R. Seddon at Queen's University Belfast (QUILL)
- Further refinements are continuing, but the method holds much promise

Electrokinetic Slug Flow Reactor

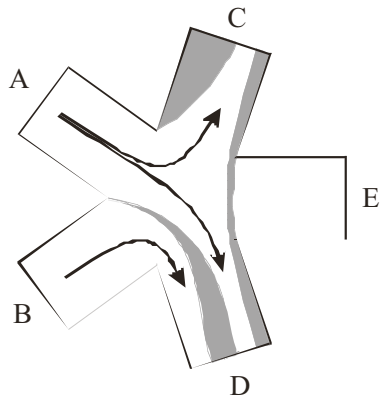


Fletcher et al., 1999, Analyst, 124, 1273-12

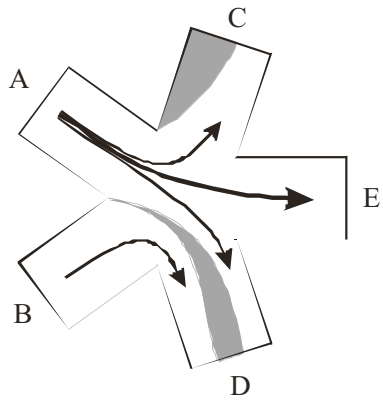
Y junction, uniform electrical properties



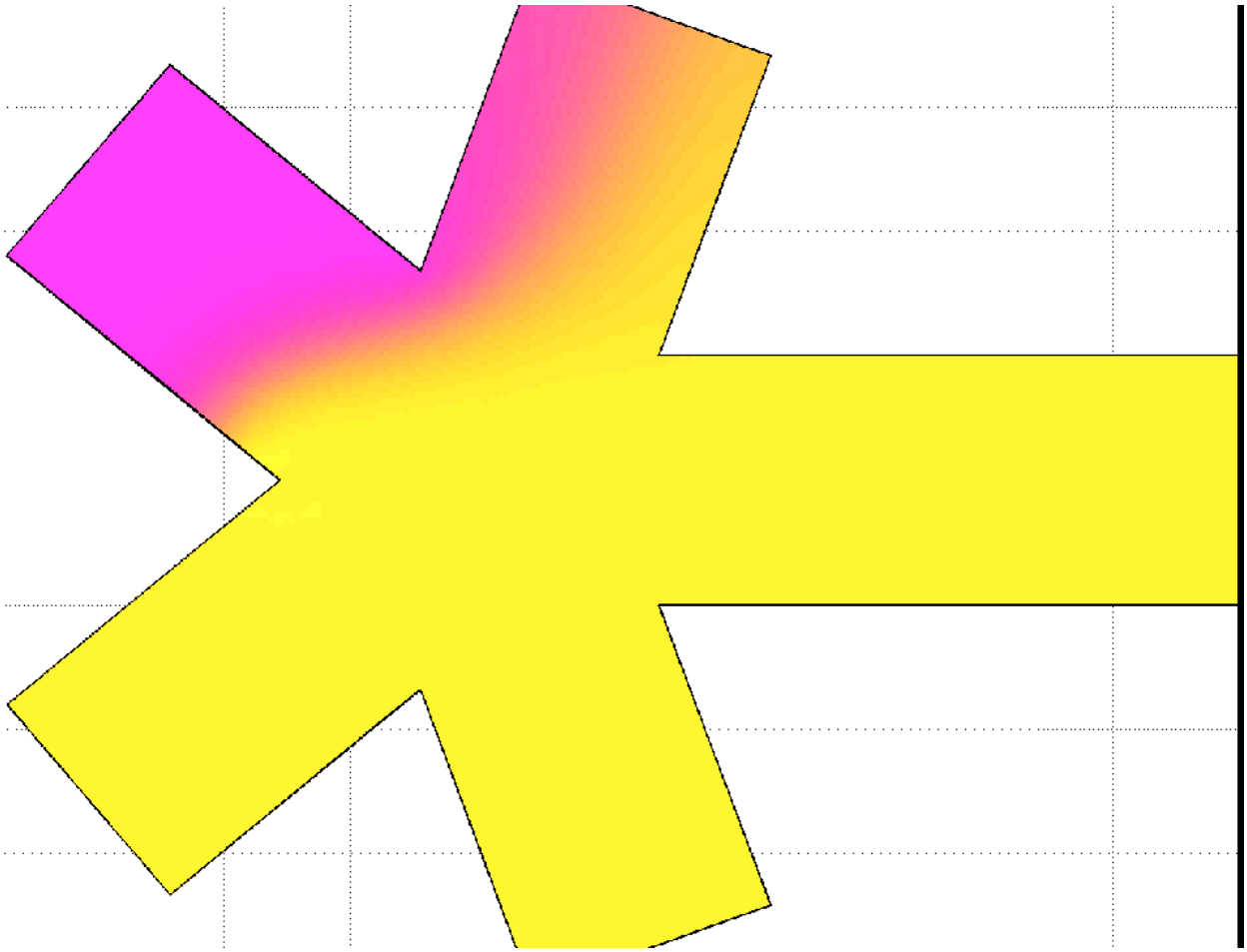
Star Junction



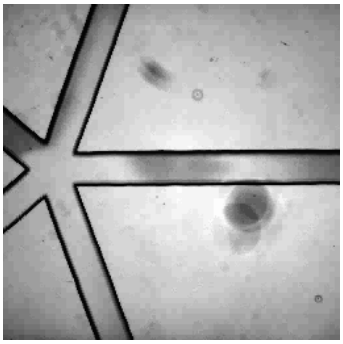
Preparation step



Injection step

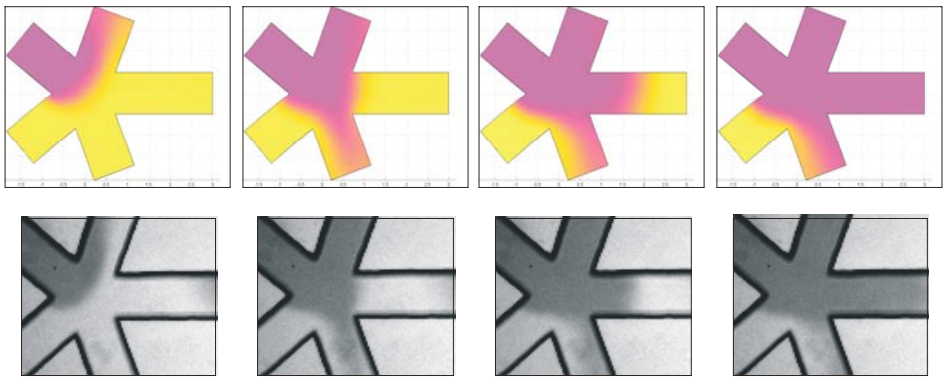


Star junction experiment



Qualitative comparison with experiment

Preparation step Injection step



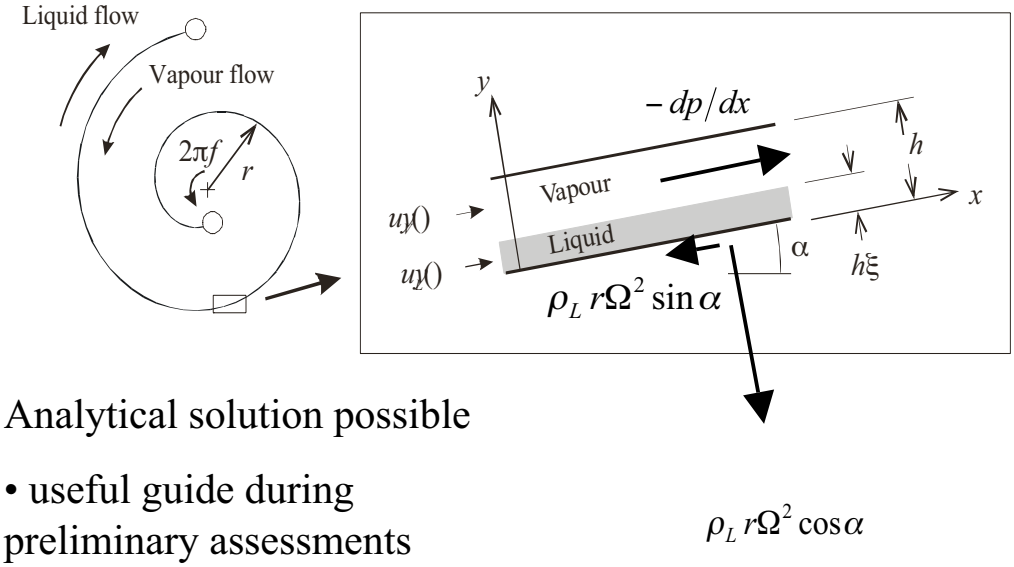
Summary

- Near-perfect slugs are possible with the star junction
- This could have an impact on a range of analysis applications in addition to facilitating the slug type reactor
- A further use may be as a way to form sharp interfaces for studying effects of property non-uniformity on electrokinetic flow
- The work illustrates the power of the computational modelling approach in electrokinetic flows

Distillation (and Reaction)

- Phase contacting refers to organised interaction of one phase with another phase so mass transfer between the two can take place
- Examples include absorption, extraction, stripping and distillation
- Each is a way of performing a separation of one or more species from other species
- Phase contacting in microchannels should allow more effective and precisely controlled mass transfer
- The obstacle to controlling phase interaction at small scales is the dominance of surface forces
- In conventional mass transfer applications gravity is the usual agent used to organise the phases spatially
- At small scales centrifugal body force can be used

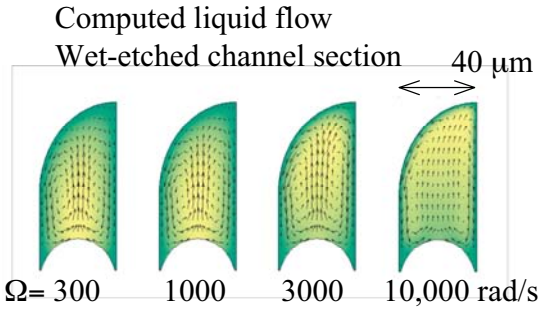
Flow in a spiral planar gap



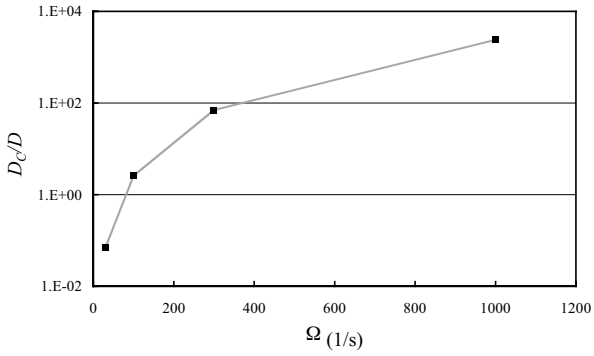
Analytical solution possible

• useful guide during preliminary assessments

Coriolis effect in 2-D channel sections

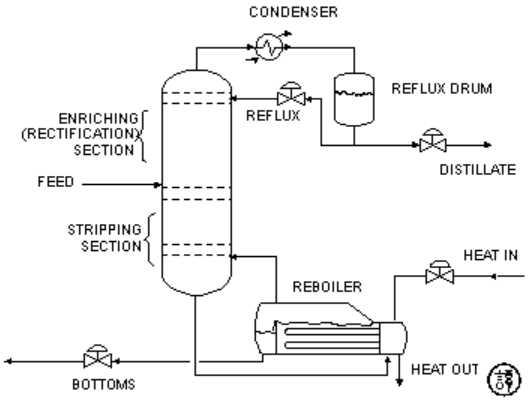


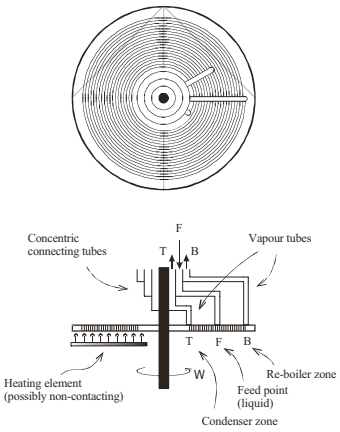
Secondary motions do not increase friction factor below about 10,000 RPM



Secondary motions may increase mass and energy transfer rates by 2 to 3 orders of magnitude

Towards distillation on a chip





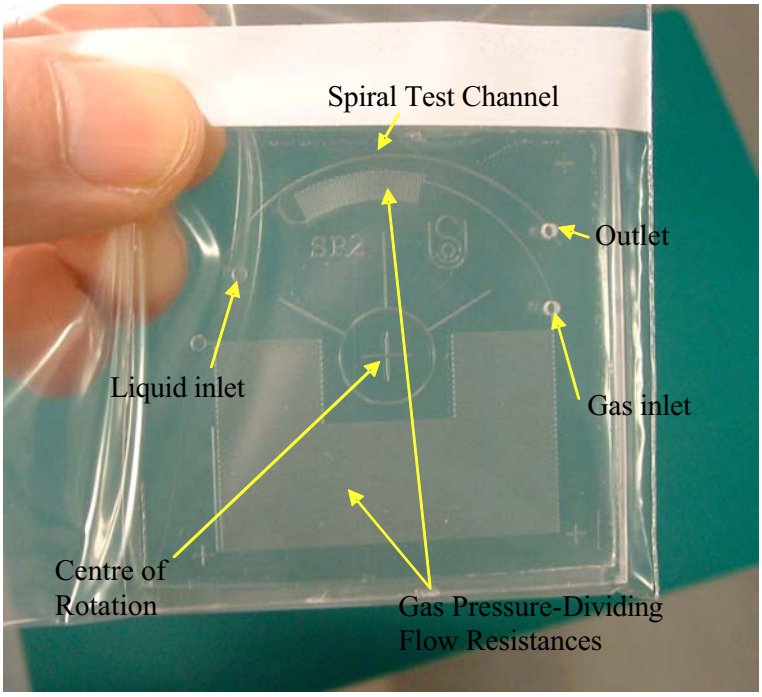
(M. T. Tham, "Distillation", 1997
http://lorien.ncl.ac.uk/ming/distil/distil0.htm)

Applications include:
- Production of fragrances and flavours
- Analytical testing of chiral molecules
- Water purification in gravity-free environments

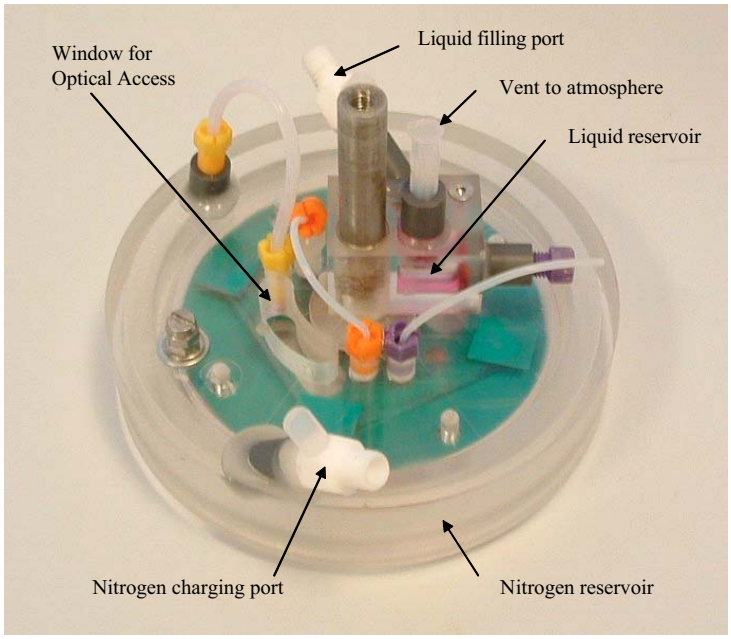
Stage 1 experiment: the chip

- N_2/water system
- Contacting flow stability
- Flow control
- No significant mass or heat transfer

Primary measurement is interface position



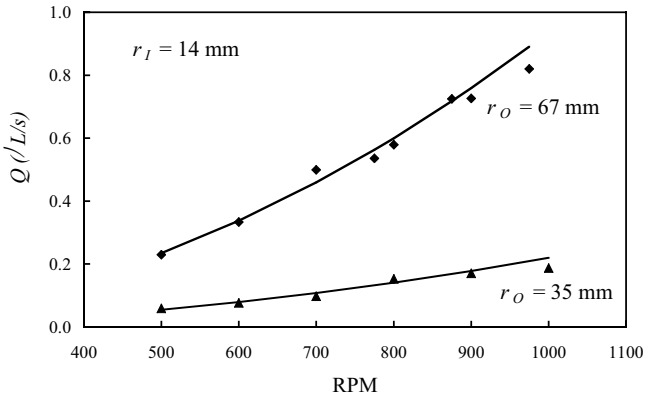
Stage 1 experiment: the apparatus

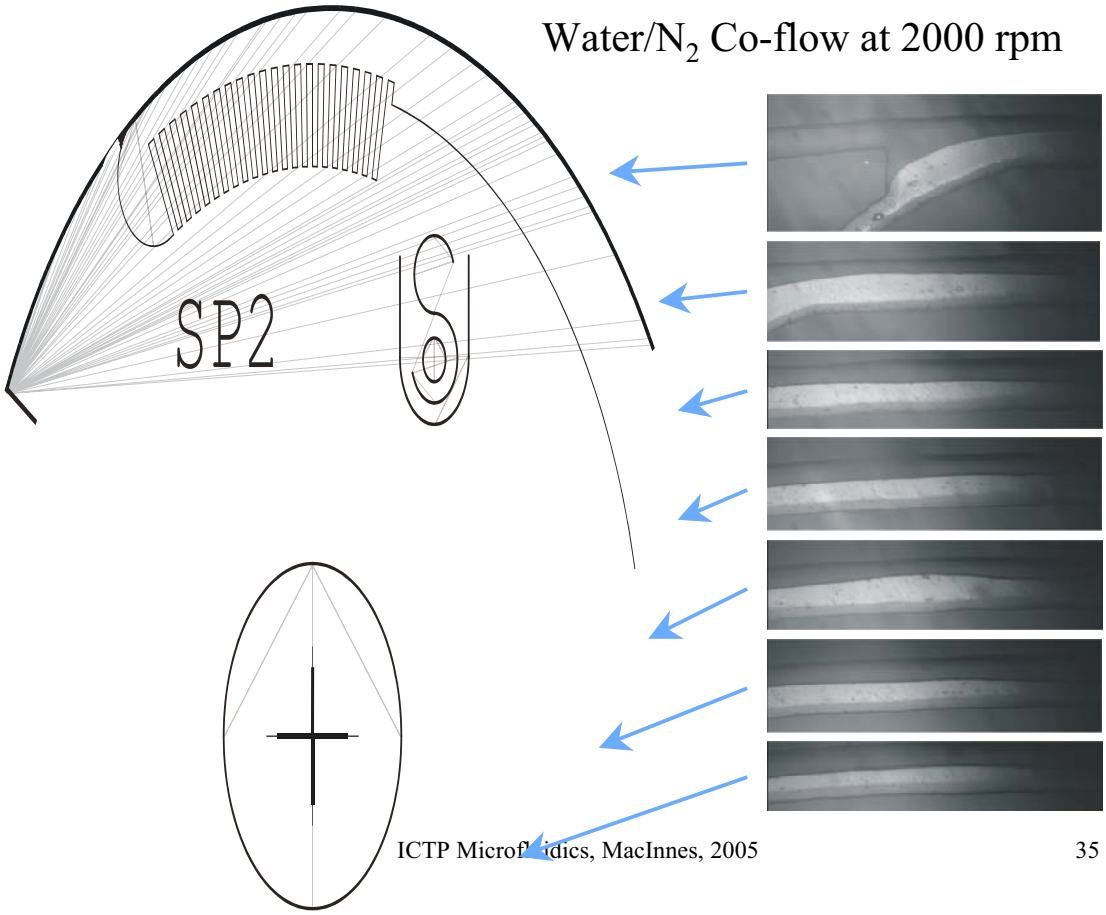


Single-phase tests at low RPM

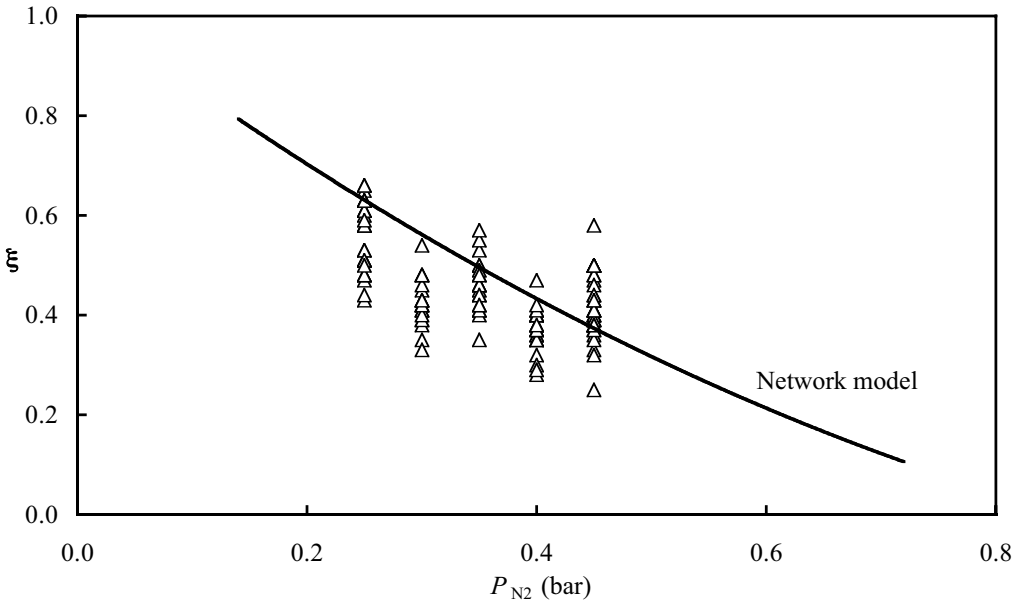
Rotation term must be added to the flow rate law:

$$? \; ? p \; ? \; \frac{?? \; ^2}{2} ? r^2 \; ? \; \frac{f?? s}{R^2} Q$$





Comparison with simple network model



Development of a comprehensive modelling

Modelling completed:

• Network modelling (0D) of the flow – supporting device design
• 2D CFD computation of developed flow in liquid a rotating channel – assessment of Coriolis effects
• Various back-of-the-envelope estimations of heat and mass transfer – supporting distillation device design (stage 2)
• 3D CFD computation of temperature distribution in the stage 2 device – supporting device design

Envisaged:

• Network modelling of composition and energy, 1D since Y and T vary along contacting channel – advanced device design
• 2D CFD computations to determine heat and mass transfer coefficients – supports 1D network modelling
• 3D two-phase flow computations of junction region – supports design of the junctions

Summary

- Rotating spiral channels promise large improvements in separation precision over existing phase contacting approaches
- Applications include chemical analysis (e.g. chiral molecules), fragrance and flavour processing (thermally labile) and water processing in gravity-free environment
- Considerable work is required in the areas of heat and flow control, mass exchange with the device (for continuous production)
- Stage 2 experiment: a distillation apparatus is currently being fabricated
- Can be applied to reactive distillation

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