



FRX Application Note

Note Number: 2

Cleaning Microreactors

1 Summary

This application note gives hints and tips on cleaning & unblocking flow reactors, i.e. chips or tube based reactors. It is suggested that the whole document is read before attempting to unblock a reactor.

2 Standard Chip Cleaning

The simplest way to clean (unblocked) chips after use is to leave the chip connected to the system and use the pumps to pass solvent through them.

If reagents are pumped through the FRX pumps (rather than injected using a Reagents Store), simply move the input pipe (with the filter on) and place in a beaker of clean solvent and run the pumps.

If using a Reagents Module and pumping reaction solvent through the FRX pumps, switch the manual valve on the Reagent Store so that the sample loops are bypassed and the solvent is diverted directly to the chip without flushing the loop.

3 Unblocking Chips

From time to time precipitates may form in a reaction and block a chip, however it is almost always possible to unblock a chip. It may be possible to simply change solvents and carry on pumping through the chip, however you may find the following tips useful.



3.1 Recommended method of unblocking

3.1.1 Choose rescue solvent

Virtually all blockages in chips are porous, that is they let *some* flow through, however little. This means that you will almost certainly be able to get a “rescue” solvent to the site of the blockage. Ensure the rescue solvent you intend to pump through will have a high chance of dissolving the solid in the chip. This will be dependant upon the chemistry being performed, but if the nature of the precipitate is unknown, the following solvents/reagents may be a good starting point:

1:1 acetonitrile:water

1:1 dichloromethane:methanol

2-6M NaOH(aq)

3.1.2 Purge rescue solvent up to chip

When unblocking chips, exactly the same method of pumping solvent into the chip can be used as for Standard Chip Cleaning (above). However, to allow the solvent to reach the blockage quicker (which may be important at low flow rates), it may be necessary to purge the pipe up to the chip with rescue solvent. To do this, disconnect the pipe from the chip header and pump a sufficient volume of rescue solvent through.

3.1.3 Reverse direction

Typically, as a precipitate forms, particles may get stuck e.g. on the wall of the chip which will cause other particles to get stuck and so the blockage can turn into a “filter pad”. Simply reversing the direction of flow can help to break up this blockage and allow flow in the reverse direction.

To do this, connect the purged pipe containing rescue solvent to the “output” port in the chip header and connect 2 or 3 spare pieces of pipe to the inputs (depending on the number of inputs to the chip!).



3.1.4 Low flow rate

Depending on the severity of the blockage, it will often be necessary to pump at a low flow rate to avoid over-pressurising the system. To avoid over pressurising the system when unblocking a chip, take the following action

- pump just one channel at a low flow
- ensure the BPR (back pressure regulator) is not in-line
- set the pressure limit on the Pump to 30bar

3.2 Other possibilities

Other techniques that have been used to unblock stubborn chips include:

- Raise the temperature of the chip to assist in solubility
- Pyrolyze the contents of the chip using the heater/cooler and then pump through NaOH(aq) to dissolve contents
- Place chip in an ultrasound bath whilst pumping