Supplementary Information

Design and development of a 3D-printed back-pressure regulator

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In this communication, we describe the novel design and preparation of a back-pressure regulator that can be used in flow chemistry applications. Using low cost components that can be readily sourced, a low-cost 3D printer and freeware design software, we developed, and 3D printed a back-pressure regulator that is simple to assemble and resistant to blocking. This device can be used to maintain the pressure of a fluidic system between the pump head to the back-pressure regulator and allows the collector or collection vessel to be at atmospheric pressure. Ensuring control of pressure within the fluidic system is essential for maintaining consistent flow rates in flow chemistry set ups.

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S1. Design and Development of a back-pressure regulator (BPR)

S1.1. Design Software

The face plates of the back-pressure regulator were designed using the web-based freeware design program - Tinkercad (www.tinkercad.com) which can export models in .STL file format for use with a 3D printer.

S1.2. Device Design

It was important that in the design of the 3D printed back-pressure regulator that we considered the limitations of the method of additive manufacturing. The parts are built in additive layers and therefore from the bottom upwards. For this reason, the design was made in two parts or separate face plates. To avoid blocking and to cope with the potential for suspended particulates we preferred to opt for a diaphragm type system. We could then take advantage of the design in being two face plates and effectively sandwich the PTFE membrane diaphragm between the two plates. Another reason for designing the BPR in two parts, each as a separate face plate, was so that we could insert into the print hex nuts of the appropriate size and thread ready for the fittings to be installed. We could pause the print to allow us to insert the hex nut at the point where the print reached the top of the walls of the nut enclosure, and then resume the print to seal in the nut. Effectively this allows us to insert a single pause into each print. (See S2. Material and 3D Printer Settings)

Polypropylene was chosen for its properties and compatibility. Polypropylene (PP) has reasonable resistance to common solvents[1] and the area of the face plate that would be in contact with the solvent was minimised in the design. The PTFE membrane would also provide a protective layer as well as separate liquid from gas in the assembled BPR. The dimensions of the printer table will allow the building of up to 9 of the same plate simultaneously.

Integral to the design was the embedding of nuts that have the appropriate threads for the standard fittings that are available for the fluidic path and gas distribution (see S2.2. Additional Components). Liquid face plate (see Figure 1 and 2) accommodated two ¼-28 UNF stainless steel hex nuts. These fittings are suitable for both 1/8th and 1/16th OD tubing in common use in flow chemistry and compatible with most vendors. 2 rectangular slots were designed to connect the membrane face to the liquid input and output which would also be interchangeable due to the symmetrical design. These were designed to be sufficiently wide enough to reduce the occurrence of blocking in the BPR itself.

Gas face plate (see Figure 3 and 4) was designed to accommodate a single M5 nut which would provide the thread for a 4 mm push-fit straight adapter M5 that would connect via 4 mm OD tubing to a regulated compressed air or nitrogen supply. 4 mm OD tubing was chosen due its compatibility with pneumatic fittings available from several suppliers. The gas face plate features a recess for seating of a 9.2 mm ID silicone O-ring and a shallow domed recess to allow the diaphragm to be pushed away from the liquid face plate by the liquid back pressure. It was envisaged that the domed recess in the gas face plate would allow for small particulates to pass and exit the BPR, reducing the chances of blocking.

S1.3. CAD Diagrams

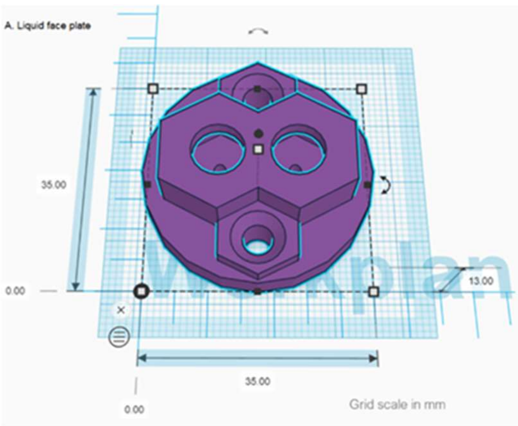

Figure 1. CAD diagram of back-pressure regulator. A; Liquid face plate

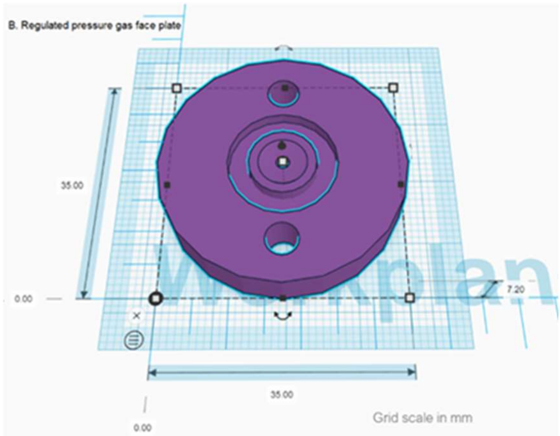

Figure 3. CAD diagram of back-pressure regulator. B; Gas face plate

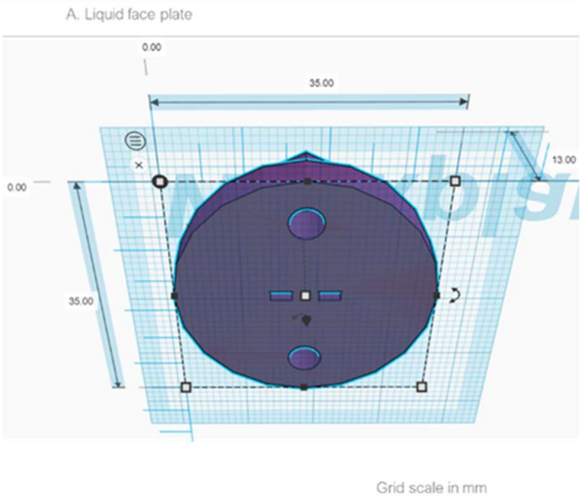

Figure 2. CAD diagram of back-pressure regulator. A; Liquid face plate (underside showing slots)

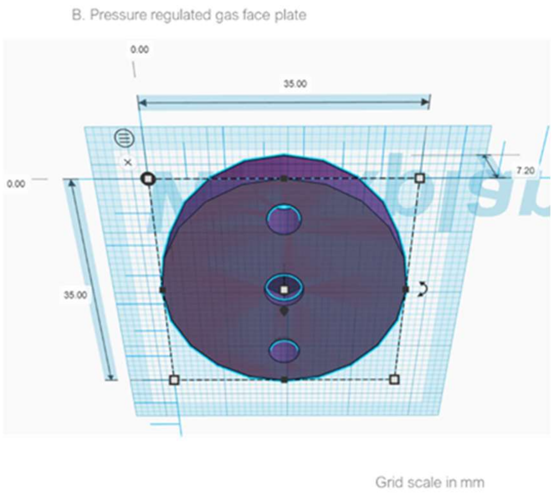

Figure 4. CAD diagram of back-pressure regulator. B; Gas face plate (underside showing port for 4 mm push-fit straight adapter M5)

S2. Material and 3D Printer Settings

Print material was supplied by Ultimaker and is 2.85mm in diameter and comes on a 500g spool. Settings were as per the manufacturer's instructions.

Settings:

Printing temperature 205°C
Build plate temperature 85°C
Print speed 25 mm/s
Fan speed 20%

Quality:

Layer height 0.1 mm
Wall thickness 1.14 mm
Top / bottom thickness 1 mm
Infill density 100%
Infill Pattern Octet

Build plate adhesion:

Prime blob enabled
Adhesion type – brim
PP adhesion sheet applied to glass print bed

Pausing Procedure:

1. Choose Extensions, Post Processing, Modify G-code
2. Choose Add a script, Pause at height
3. Set the required height (or layer number) and then close

S2.1. STL Files

STL files that can be used to 3D print the Vernalis BPR can be found at https://github.com/vernalis/3Dprint_files

S2.2. Additional Components

Additional components can be easily sourced from several reputable suppliers. Suppliers and supplier catalogue numbers are merely illustrative, and prices quoted are that of January 2020.

Item	Supplier	Catalogue number	Price £
2 x ¼ 28 UNF hex nut (Price of 100-pack)	Amazon	B000H6KD86	29.19
2 x M4 (4mm x 25mm) Hex Socket Countersunk Machine Screw (Bolt) - Steel (Price for pack of 20)	Amazon	B075SY4L9J	5.49
2 x Wing Nuts ZINC Plated (M4) (Price for pack of 20)	Amazon	B07MCDLVPH	5.20
1 x M5 (5mm) Hex Nut - Steel (Price for pack of 40)	Amazon	B07BVX8QQ5	5.49
1 x PTFE film 50 mm x 100 mm x 0.1 mm	Amazon	B07LGVKK4K	8.99
1 x Straight Pneumatic Push to Quick Connect Fittings M5 Male x 4mm Tube OD (Price for pack of 8)	Amazon	B07KGN3HYZ	6.12
1 x Ace O-rings, silicone I.D. 9.2 mm, wall 2.6 mm (Price for pack of 12)	Merck Sigma Aldrich	Z504157	17.60

S3. Assembly

Using one face from one of the BPRs as a template, mark and cut out the shape of the diaphragm from 0.1 mm thick PTFE sheet. Mark out the position for the holes for the M4 bolts to pass through the membrane and then cut out using a 4 mm hollow punch. Similarly, cut out a gasket layer again marking the position for the bolt holes and cut out the centre using a 9 mm hollow punch.

The BPR should be assembled in the order shown in Figure 5

1. Insert 2 x M4 bolts through the liquid face plate and use as a guide for positioning the subsequent layers

2. Place PTFE diaphragm

3. Place PTFE gasket

4. Place Gas face plate with silicone O-ring installed in the seating.

The plates are then held in place by 2 x M4 stainless steel wing nuts which are then lightly secured to apply a small amount of compression to the silicone O-ring onto gasket and diaphragm

5. Screw the straight pneumatic push fit 4 mm OD male M5 adapter into the gas face plate port to finger tightness

6. Install the fittings for liquid input and output into the liquid face plate and the gas face plate. Connect the push fit adapter to a regulated gas supply using 4 mm ID tubing (Figure 6)

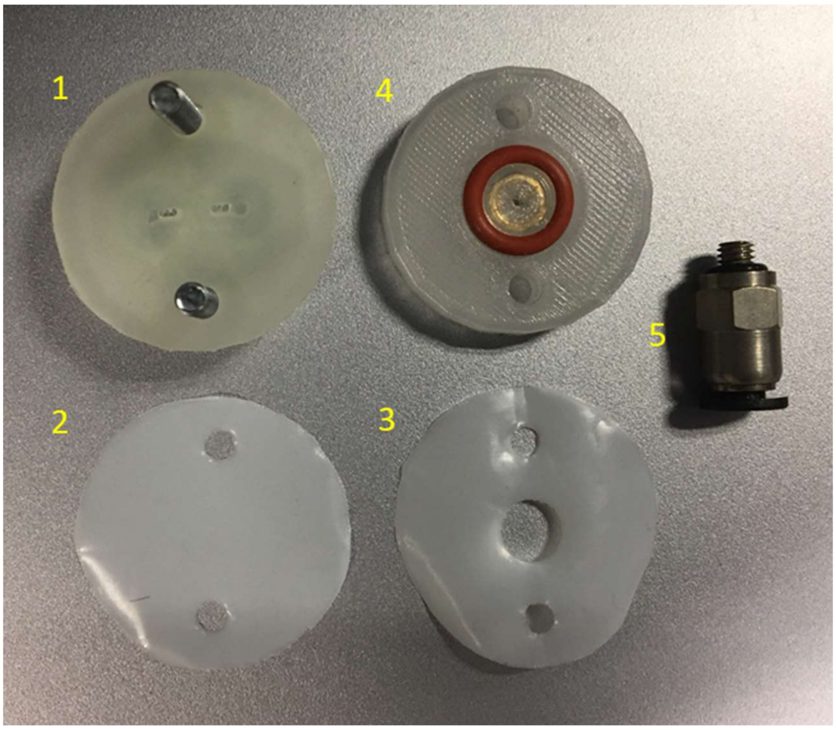

Figure 5. Parts and assembly of the back-pressure regulator

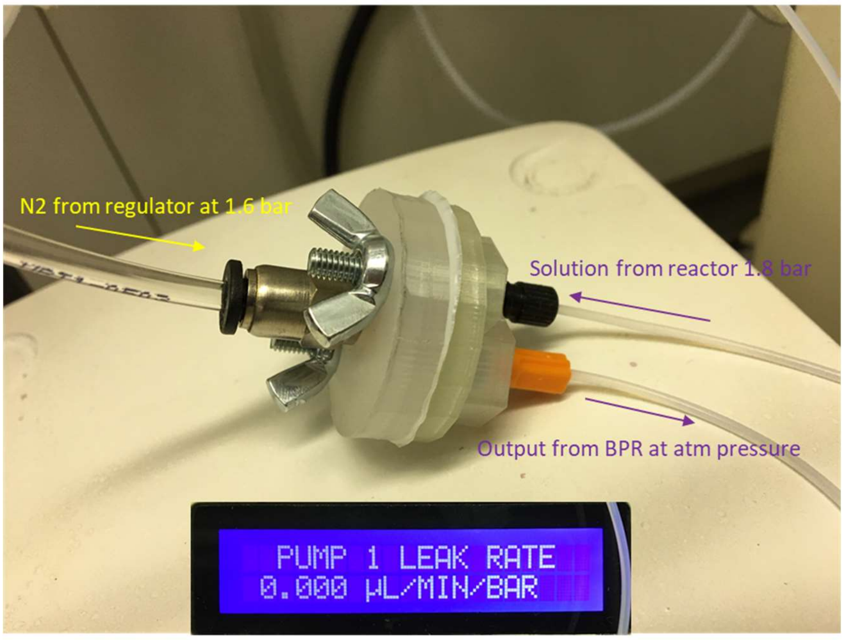

Figure 6. Leak rate testing the BPR

S3.1. Testing

The BPR was tested for leaks and performance using tetrahydrofuran and acetonitrile as the solvents. We tested the BPR within a typical operating range, gas pressures of up to 2 bar and flow rates up to 3 mL min^{-1} however we did not establish upper limits for either parameter.

Prior to using the leak test feature on the Syrris Asia Pump Module we pumped acetonitrile at 3 mL min^{-1} through the BPR with the nitrogen gas pressure set at 1.6 bar. The pump head pressure was recorded at 1.8 bar and this was maintained for 30 minutes. The pump was stopped, and a standard leak test performed which detected no loss of pressure in the fluidic system behind the back-pressure regulator (Figure 6).

S4. References and Notes

1. Kitson, P., Glatzel, S., Chen, W. et al. 3D printing of versatile reactionware for chemical synthesis. Nat Protoc 11, 920–936 (2016) doi:10.1038/nprot.2016.041

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