Homebuilt Cost-Effective Nitrogen Blowdown Evaporator

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Contents

Bill of Materials

Material	*Qty.*	*Vendor*	*Part number*	*Cost*
Acrylic sheet, 12 in. × 24 in. × 1/4 in.	4	McMaster-Carr	8560K355	$126.2
2×12 gas manifold	2		6527N1	$104.36
Plug w/hex drive, 1/4 NPT male	1		6367N14	$0.53
Plug w/hex drive, 1/8 NPT male	2		44605K231	$0.50
1/4 in. I.D. barb × 1/4 NPT male hose fitting	1		5218K686	$0.57
1/16 in. I.D. barb × 1/8 NPT male hose fitting, 10 pk.	3		5117K91	$45.00
304 stainless steel pipe with 1/4 NPT male threads, 3 in.	1		4830K135	$4.86
3/8 in. O.D. × 1/4 in. I.D. polyurethane tubing, 10’	1		5648K71	$13.10
1/8 in. O.D. × 1/16 in. I.D. LLDPE tubing, 100’	1		5181K15	$8.00
O-ring, Neoprene, -006*, 100 pk.	1		94115K006	$3.60
Weld-On 4 Acrylic Adhesive, 1 pt.	1	Amazon	B0096T6P1Y	$28.95
Pasteur pipette, 9 in., glass	22	Fisher Scientific	16-678-6B**	$4.40†
BM100 water bath, Yamato Scientific America, Inc.	1		50-189-8142	$430
			Total	$770.07

Note: Prices listed here are public list prices as of this writing and do not reflect discounts available through institutional agreements.

*-006 O-ring has a nominal I.D. of 1/8 in., nominal O.D. of 1/4 in.

**Discontinued; P/N 13-678-20D is the manufacturer’s recommended replacement

†This price is based on the cost per each of the manufacturer's recommended replacement, assuming that most users looking to build this system will already have this ubiquitous material in their laboratory or be able to acquire it from a colleague. If not, the cost will be higher, as this is only sold as a high-count box.

Assembly

Verify water bath construction

The thermometer holders in the water bath we used are nominally located at the “12 o’clock” and “3 o’clock” positions. However, we have observed that the tolerance of these parts may be greater than the files we include can accommodate. We advise that you first construct a prototype of the tube support (parts 1 and 2)

from cardboard or other inexpensive material and verify that it fits in your water bath before committing to cutting the parts from acrylic. If necessary, move the thermometer holes in these parts as well as parts 3 and 4 to accommodate your water bath before cutting the acrylic version.

Cut acrylic parts using CO_2 laser

1. Determine the proper settings to cut and engrave acrylic using your laser cutter. Set up the laser so that black markings are cut and the green & magenta markings are engraved. (Change the coloring by using a vector graphics editing software if required by your laser's programming.)
    a. Using a ULS VersaLASER 3.60 100 W CO_2 laser cutter with the High Power Density 2.0 cutting lens, we found that 100% power, 2% speed, 1000 ppi was best for cutting and while 100% power, 50% speed, 1000 ppi was best for engraving.
2. Remove any backing from the acrylic before cutting on at least the side to be engraved. If this is not done, tiny pieces of backing will remain in the engraved words and the depth of the engraving will be diminished as the laser has to first burn away the backing.
3. Cut all parts as designed. Cut one copy of all part files, with the possible exception of part 5 – if using a single leg version of the file, cut three copies. Select the versions of parts 1 and 2 for the type of sample tubes you will be using or design a new version as appropriate.

Assemble acrylic parts using solvent cement

Follow the manufacturer's instructions for use of the solvent cement and work in a fume hood. Work atop a sheet of aluminum foil and support the parts being glued above the work surface so that they do not adhere to it. (Setting the parts atop a 250 mL beaker or similarly sized object works well for this purpose.) In some cases, it will be necessary to apply solvent cement prior to assembly; in other cases, it will be necessary to dry fit the parts before applying solvent cement through gaps. Carefully consider the approach before gluing, as once the glue begins to set the acrylic cannot be separated except by sawing through the bond. Allow each glued part to cure overnight before continuing to assemble the next connected part.

1. Wash all parts to remove residue from manufacturing and laser cutting. We have found that a liberal application of foaming hand soap is sufficient for this purpose and caution against the use of solvents which can craze and crack the acrylic. Wear gloves during this process to avoid leaving finger oils on the clean acrylic. Rinse thoroughly to remove soap residue and dry with paper towels.
2. Assemble the tube support:
   For 15 mL or 50 mL plastic tubes:
    a. Glue part 2b *step ring* on top of part 1a *tube support*. The engraved line around part 2b should align with the outer edge of part 1a and the engraved line around part 1a should align with the inner edge of part 2b.
    b. Glue part 1b *rest ring* on top of part 2b *step ring*. The engraved line around part 1b should align with the outer edge of part 2b.

   For 15 mL glass tubes:
    a. Assemble one copy of part 1a *tube support* with parts 1b *rest ring* and 2b *step ring* as above. This assembly is the *upper support*.
    b. Glue four copies of part 1c *lower support standoff* to one copy of part 1a. Place the *upper support* on top while the parts cure to hold them in the appropriate place. This assembly is the *lower support*.
    c. Glue the *lower support* to the *upper support* by the tab-and-slot connection. See **Figure S-1** for a view of the completed assembly.
3. Assemble the pipette support:

a. Glue the four copies of part 4b *pipette support standoff* by their tabs into the four slots on part 3 *pipette support bottom*. Place part 4a *pipette support top* on top while the parts cure to hold them in the appropriate place.
b. Glue the assembly of parts 3 and 4b to part 4a by the tab-and-slot connection.
c. Glue the three copies of part 5 *pipette support leg* by their tabs into the slots on part 4a. Use part 6 *baseplate* to align the legs properly. A second pair of hands is very useful at this step in holding everything steady as you dry fit the parts prior to the application of acrylic cement. **Do not glue the legs to the base.**

Figure S-1. Views of the assembled 15 mL glass tube support. (A) The assembled support alone. (B) The assembled support, elevated off the surface, with a tube in evaporation position, illustrating how the tube rests on the bottom of the bath.

Assemble the apparatus

1. Assemble the gas manifold. *You may carry out this step while acrylic assemblies are curing.*
   Note: The threading on the male fittings is longer than the depth of the female fittings. Do not attempt to tighten the connections until the threaded portion of the male fitting is fully inside the female fitting.
   a. Connect the two manifold blocks using the 3 in. 1/4 NPT male threaded pipe.
   b. Place the 1/4 NPT male plug in the open end of one of the manifold blocks. Place the 1/4 NPT male threaded hose barb in the open end of the other manifold block.
   c. Place 1/8 NPT male threaded hose barbs in 22 of the 24 openings along the manifold blocks. Place 1/8 NPT male threaded plugs in the other two openings. (If you are using a different number of channels, adjust the number of plugs and hose barbs accordingly.)
   d. Cut an appropriate length of 1/4 in. I.D. polyurethane tubing so that it can reach from the operating position of the NBE apparatus to the gas source. Attach it to the large hose barb at the end of the manifold.
2. Attach the assembled gas manifold to the assembled pipette support stand using zip ties through the holes on one leg. Pick whichever leg is closest to the gas supply.
3. Place a glass Pasteur pipette in each hole in the pipette support that corresponds to a sample position that will be used. If you find that any of the holes in the upper level of the pipette support are too tight of a fit, we have found that smoothing out the hole with a 5/16 in. drill bit suffices to remove any imperfections from the cutting process.
4. Plumb the gas manifold to the Pasteur pipettes.

a. Affix one end of your coil of 1/8 in. O.D. × 1/16 in. I.D. tubing to a barb on the gas manifold.
b. Play out enough tubing to reach a pipette, then add two inches for slack and make a mark.
c. Cut the tubing at the mark using a tubing cutter or, if you do not have one, by laying it on a flat surface and cutting with a safety razor blade.
d. Attach an O-ring and push it ~ 1 in. up the tube.
e. Gently push the O-ring into the end of the Pasteur pipette until it is completely seated inside. A pair of blunt-tipped tweezers is useful in this process. Be careful to apply force only directly along the pipette's long axis, otherwise you may break the pipette. Wear cut-resistant gloves while performing this step.

5. Assemble the complete system in a fume hood.
   a. Place part 6 *base* on the floor of the fume hood.
   b. Place the water bath atop part 6, roughly centered from left to right and a little towards the rear. Place the assembled tube support inside the water bath and insert the thermometer.
   c. Gently lift the pipette support high enough to clear the thermometer, then slowly bring it down until the tabs on the base of the legs rest in the slots. Pay attention to the position of the pipettes within the holes on the tube support and move the water bath as necessary to ensure proper alignment. A second pair of hands is useful in this step.
   d. Connect the nitrogen supply line to a nitrogen source.
   e. Remove the pipette support and set it to the side or rear of the water bath, as determined by the availability of space.
   f. Fill the water bath to an appropriate level and proceed to operation.

Setup notes

Insertion of the O-rings into the Pasteur pipettes is best accomplished with the aid of a fine blunt object, such as blunt-tipped tweezers or a microspatula. Care must be taken to avoid applying too much pressure to the wall of the pipette to avoid shattering. Cut-resistant gloves should be worn any time this procedure is carried out.

In assembling the gas manifold, we avoided the use of Teflon plumber's tape to avoid introducing PFAS contamination into the apparatus. However, this can lead to gas leaks from the connection points in the manifold. We were unable to find a satisfactory PFAS-free pipe thread sealant and have opted to accept the increased gas usage rather than risk the apparatus becoming a point of contamination for our analyses. Users who build this apparatus for analyses other than PFAS may wish to seal the gas connections with Teflon plumber's tape to minimize gas losses.

The fill level of the water bath should be marked at a point below the level of the nitrogen nozzles. If the water bath is filled too high the nozzles will become submerged. This causes splashing of the bath water and contaminates the nozzles.

LC-MS/MS

Analytical conditions

The conditions for LC-MS/MS analysis of the selected PFAS were chosen based on EPA Method 537.1 and adhered as closely as possible to the details given therein.[1] The analytical column was a Waters Atlantis dC18 HPLC column (186001301) with a Waters VanGuard precolumn (186007662 & 186007949). **Table S-1** gives details of the gradient elution program. Source conditions were chosen to avoid in-source collision-induced dissociation of the precursor ions and are given in **Table S-2**. Fragmentor and collision energy settings were

optimized for the MS/MS transitions given in EPA Method 537.1 using *Agilent MassHunter Optimizer* under isocratic flow injection analysis conditions corresponding to the solvent composition at the column exit at the analyte's retention time. A cell accelerator voltage of 5 V was used for all analytes; no crosstalk was observed. The MS/MS transitions monitored, optimization solvent conditions, and the optimized fragmentor and collision energy settings are given in **Table S-3**. The observed response factors for each analyte f_i, determined by dividing the measured peak area by the analyte concentration for each calibration standard, are also given.

Table S-1. Gradient elution program specified in EPA Method 537.1.[1]

Time (min)	*% 20 mM NH_4OAc (aq)*	*% methanol*
0	60.0	40.0
1.0	60.0	40.0
25.0	10.0	90.0
32.0*	10.0	90.0
32.1	60.0	40.0
37.0	60.0	40.0

The remaining time beyond 32.0 min represents a reset to initial conditions; all method analytes have eluted by this time. We set the MS module to stop collecting data at this time to reduce data file size.

Table S-2. MS source parameters.

Parameter	*Value*
Gas Temp	250 °C
Gas Flow	5 L/min
Nebulizer	30 psi
Sheath Gas Temp	350 °C
Sheath Gas Flow	11 L/min
Capillary	2500 V
Nozzle Voltage	0 V

Table S-3. MS/MS transitions and optimized transition-specific MS/MS parameters

Analyte	*CAS number*	*MRM transition*	*%A / %B* at elution*	*Fragmentor (V)*	*Collision energy (eV)*	*f_i (cts × min ÷ nM)*
PFOS	1763-23-1	498.9 → 79.9	25/75	170	96	12.8 ± 0.6
PFHxS	355-46-4	398.9 → 79.9	33/67	140	52	8.3 ± 0.4
PFBS	375-73-5	298.9 → 80.0	46/54	120	40	11.8 ± 0.7

A = 20 mM NH_4OAc (aq); B = methanol

Concentration data

Table S-4. Measured concentration and calculated concentration factors for all three analytes in the unconcentrated standard (n = 1) and concentrated samples (n = 5).

Analyte	*Measured concentration in unconcentrated stock (nM)*	*Measured concentration in concentrated samples (nM)*	*Calculated concentration factor*
PFOS	5.2	41 ± 1	7.9 ± 0.2
PFHxS	5.2	41 ± 1	7.9 ± 0.2
PFBS	5.0	40 ± 1	8.0 ± 0.2

Assembly video

The supplementary video provides a visualization of the assembly process modeled in *Autodesk Inventor*. Most parts are shown from assembly of the individual parts through completion of the entire apparatus. Tubing is not shown as an appropriate tubing model was not available.

References

(1) Shoemaker, J. A.; Tettenhorst, D. R.; Grimmett, P. E.; Boutin, B. K. *Method 537.1 Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS) Version 1.0*; 2018.