Title: Dilution of Samples in Canisters		Copy No: ##
SOP No.: 7.02/3.1/S	Effective Date: September 11, 2013	Location: ###

QSM Approval: _____

Dilution of Samples in Canisters

1. Scope

This Standard Operating Procedure provides instructions for dilution of samples in canisters. For optimal analysis when using the Entech preconcentrator samples are not diluted to pressures in excess of 25 psi. In addition, for these procedures to remain valid the final canister pressure must not exceed the inlet pressure of the clean air supply.

2. Procedure ideal for only a few dilutions performed on the same day

2.1 Check the Methane Heat pyrometer gauge on the clean air generator. The gauge should indicate a temperature of 300°C ± 20°C. A red light above the gauge should be cycling on and off. Seek assistance if this temperature reading is not observed.

2.2 Connect the sample canister to the **pressure gauge/ SS-4H valve/ 3-way valve assembly (GVA)** using a 9/16 in. wrench. If not already attached, connect the tubing from the outlet of the preferred mass flow controller to the common port of the 3-way valve using a 7/16 in. wrench, and turn the 3-way valve to the vent position. Connect the SS-4H valve branch of the GVA to one of the manifold lines on the manually-operated vacuum apparatus. Ensure that the other manifold lines are capped securely.

2.3 Raise a dewar over the vacuum apparatus cold trap, clamp securely in place, and fill the dewar slowly with liquid nitrogen. Switch on the power bar, this turns on the high vacuum pump, which is connected to the outlet of the cold trap. **CAUTION: Prior to use ensure the water level in the trap is low enough that there is no risk of clogging the trap outlet when the water freezes. This vacuum pump must ALWAYS be on whenever the cold trap is immersed in liquid nitrogen. Failure to observe these precautions could, under certain circumstances, result in an eventual explosion.**

2.4 Ensure the reading of mass flow controller (mfc) output is set to desired rate. Approximately 223 for a reading that corresponds to an actual flow rate of approximately 250 mL/min or use the higher rate mfc that is set approximately to 1000ml/min. (Accurate measurement of this flow rate is not necessary). Clean,

humidified air will pass through the mfc, thus it is necessary that prior to use it is ensured that the humidified air canister contains de-ionized H_2O.

2.5 Attach the sample canister to the open 9/16 in. port. With the canister valve closed, the dilution air flow vented through the 3-way valve, and with the SS-4H valve on the GVA open, raise the vacuum toggle valve on the vacuum apparatus. This evacuates the manifold line and the GVA.

2.6 Close the vacuum toggle valve, and raise the clean air supply toggle valve to pressurize the GVA with clean air. Repeat this evacuation / pressurization process several times to ensure that all traces of lab air are evacuated from the GVA**. CAUTION: Never have both toggle valves on the vacuum apparatus open at the same time, this will place a strain on the clean air supply and vacuum pump.**

2.7 With the pressure gauge on the GVA reading at minimum (usually very close to -28.5 in. Hg on this gauge), close the SS-4H valve to isolate the GVA from the vacuum apparatus. As a precaution against possible error at a subsequent step, it is recommended that the vacuum toggle valve also be closed at this point, and that the vacuum apparatus is pressurized to a maximum of 20 psi. Observe the pressure gauge for at least one minute monitoring for a rise in pressure, which is an indication of an air leak. If a leak is observed tighten, re-attach, or replace connections.

2.8 If no leak is observed, open the sample canister valve briefly (two seconds should be sufficient to achieve equilibrium pressure in the GVA). Read the gauge pressure as precisely as possible. Record the initial sample pressure in the Sample Dilution Log Book along with all canister and sample information.

2.9 Close the canister valve. Open the SS-4H valve on the GVA and flush/evacuate the line several times as in step **2.6**. Pressurize the line to slightly above 0 psi. Close the SS-4H valve. Disconnect the canister from the GVA.

2.10 Replace it with a fully-evacuated canister of the same volume. This will serve as a volume calibration canister. Repeat steps **2.5** to **2.7** to flush and leak check the GVA.

2.11 Open the calibration canister valve, there must be no pressure rise observed (i.e. the canister must be under full vacuum). **Note: if the calibration canister is NOT fully-evacuated, open the vacuum toggle valve, open the SS-4H valve on the**

GVA, and evacuate the canister until the GVA gauge reading remains at the minimum for 15 minutes. Close the SS-4H valve and repeat step 2.7 before proceeding.

2.12 With the calibration canister at full vacuum, the GVA isolated from the vacuum apparatus, and the 3-way valve in the vent position, open the canister valve. Simultaneously turn the 3-way valve to the fill position and start the stopwatch.

2.13 Allow the canister to fill with air. Record the time at which the canister has filled to exactly the pressure previously recorded for the sample. (Most samples are diluted by a factor of two. Confirmation of subsequent sample dilution accuracy is enhanced if the calibration canister is allowed to fill for twice the time necessary to reach the sample pressure reading. The calibration canister pressure at 2X sample volume is recorded and compared later to the final pressure of the diluted sample. Agreement within ± 0.2 psig is expected).

2.14 Re-evacuate the calibration canister and disconnect it from the GVA using the procedure in step **2.9**. Re-attach the sample canister and repeat steps **2.5** to **2.7**. **Ensure that the SS-4H valve is closed before proceeding further.**

2.15 Open the sample canister valve. Simultaneously turn the 3-way valve to the fill position while starting the stopwatch.

2.16 Allow the canister to fill with dilution air for exactly the time required to achieve the desired dilution factor, and then return the 3-way valve to the vent position. Close the canister valve, and record the dilution time and the final canister pressure in the log book. Label the canister **clearly** with the sample dilution factor, i.e. 2.00× the original concentration. .

2.17 Repeat step **2.9**. Turn off the high vacuum pump power bar and remove the liquid nitrogen dewar from the cold trap. Store excess liquid nitrogen safely in storage dewars.

3. Procedure ideal for several dilutions performed on the same day

3.1 Begin with steps **2.1** to **2.7**. In step **2.2**, substitute the sample canister with a calibration canister (a fully evacuated canister of the same volume as the sample canisters).

3.2 For this method the **flow rate must be measured accurately**. Attach the vent end of the 3-way valve to a flow calibrator. Using the calibrator while the 3-way valve is in the vent position record several readings of the flow rate and calculate the average value.

3.3 Repeat steps **2.11** and **2.12**. Record the pressure at several time points covering the range of the dilution, with a minimum of five sets of data points. Fill to a maximum of 20 psi, turn the 3-way valve to the vent position. Re-evacuate the calibration canister and disconnect it from the GVA using the procedure in step **2.9**.

3.4 Create a calibration curve by plotting the Pressure versus Volume data. Volume is calculated by multiplying the average flow rate by the time it took to fill the canister to a given pressure. The data should have an r^2 value $\geq$0.980. Determine the equation of the linear trendline. **Note: this equation is only valid for the vacuum apparatus used and for dilutions performed on the same day, unless a minimum of three data points recorded at a later date fit on the previous curve within r^2 value $\geq$0.980**.

3.5 Attach the sample canister to the GVA. Repeat steps **2.5** to **2.8**.

3.6 Once the sample's initial pressure is recorded, a dilution time can be determined using the linear equation generated from the calibration curve.

The linear equation is:

$$P=mV+B$$

Where: P=Pressure
m= slope of linear equation
V= Volume
B= Y-intercept

Thus, dilution time is calculated as:

$$D_{time}=\frac{(P_{int}-B)}{m\times F}$$

Where: P_{int} = initial canister pressure
F= average flow rate as determined in step **3.2**
D_{time}=Time to dilute a sample to double the initial volume (i.e 1:1 or 2.0×)

For a 3× dilution factor, multiply the dilution time by 2 to give one part sample and two parts humid air. If preferred, the canister can be filled to the calculated expected final pressure instead of determining a dilution time. The equation is as follows:

$$P_{fin} = (m \times V_{int} \times D_f) + B$$

Where: P_{fin} =expected final pressure
m= slope of linear equation
V_{int} = initial canister volume
D_f =dilution factor
B=Y-intercept

3.7 Simultaneously turn the 3-way valve to the fill position and start the stopwatch. Allow the canister to fill with air for the exact time required to achieve the desired dilution factor or to the desired pressure and then immediately turn the 3-way valve to the vent position. Once complete calculate the actual dilution factor. Ensure the sample is clearly labeled with the dilution factor (i.e. 2.00×) and that the dilution time, dilution factor and final pressure are recorded in the sample dilution handbook.

3.8 With the sample canister valve closed repeat step **2.17** to remove the canister and shutdown the pump.

4. Revisions

Sep 2011:

- Deleted past revisions
- Corrected labeling of NUPRO valve to SS-4H
- Changed procedure headings
- Modified procedures to add detail, maximize clarity, delete outdated information, add safety information and ensure that they reflect current practice
- Where sections were reordered, references were changed accordingly
- Section 3.6: Included equations used for dilution calculations

Aug 2013:

- Removed instruction to open canister valve as it is open at this point

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