

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

QSM Approval: _____

Canister Sample Analysis Using the Entech Preconcentrator

1.0 Scope

- 1.1 This Standard Operating Procedure provides instructions for operating the Entech 7000 and 7001 preconcentrators and 7016 CA autosampler to perform a sequence of canister blank, sample, and standard analyses according to VOC method requirements.
- 1.2 This Standard Operating Procedure also provides instructions for operating the Agilent MS ChemStation software to perform a sequence VOC analyses.

2.0 Procedure

2.1 Sequence Construction

Analysis sequences are constructed as follows:

- Air Blank
- Air Blank
- Daily Calibration OR Initial Calibration standard (CANMIX) runs
- Air Blank
- Samples
- Repeat of one sample

2.2 Loading the Autosampler

The Entech 7016 CA autosampler rack can accommodate 16 canisters. Port numbers are assigned from left to right, top to bottom. Port #1 is used as a dedicated line for CANMIX and Port #2 is used as a dedicated line for a humidified air inlet. (The LOW LEVEL calibration standard has its own preconcentrator inlets).

- To load the Entech preconcentrator software, use the desktop shortcut. It will have one of the following labels, “NT7000” or “7100” After loading the preconcentrator configuration file, the software will perform a “System Check” to verify that valves and heaters are functioning normally. (The preconcentrator cannot be operated if it fails the

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

“**System Check**”). Following this startup check, the “**7100 Sequence Table**” window will open.

- Upon initial startup, initiate the bake cycle by clicking on the “**Bake**” button in the Entech Sequence Table. Note that on some instruments this initial bake will need to be manually stopped by pressing “**OK**” in a series of three pop-up boxes.
- Click on the “**VIEW**” button to open a “**Microscale Purge & Trap**” window which graphically displays real-time status of all preconcentrator parameters throughout the analysis sequence.
- Click on the “**OPEN**” button to display a list of sequence files, select the appropriate sequence to flush the sample lines and click “**OK**”. A sequence table is opened, with each line representing a different autosampler port #, beginning at “**3**”, and ending at “**16**”. If the sequence to be run includes less than 14 samples, click on the line for any port # that will not be needed, and remove it from the table by clicking on “**CUT**”. Click on the first line in the sequence (the line will turn blue) to select port #3 as the sequence starting point.
- Click on the “**DATA**” button and enter the desired “**Flush**” time (in seconds) in the “**Pres. time**” box and the number of cycles in the “**Cycles**” box. Remove any value that may appear in the “**Evac. Time**” box. Click on the check sign or OK button to save the entered value(s).
- Ensure that all required autosampler lines are open, and that sample canisters are pre-arranged in the order in which they will be attached to the autosampler.
- Click on the “**FLUSH**” button then click “**GO**” in the pop up “**System Flush**” window. Beginning with line “**3**”, each autosampler line will be sequentially backflushed with helium for the time selected earlier. After backflushing of a line is complete, immediately attach a canister to finger-tightness then snug up the Swagelok fitting with a 9/16” wrench. **DO NOT OVERTIGHTEN**. When all lines have been backflushed, click on “**EXIT**”. Proceed to leak check the lines.

2.3 Leak Check

- Click on the “**DATA**” button. Under “**Leak Check (Auto)**”, ensure that “**Use Vacuum**” switch is toggled on. In the appropriate boxes, verify, or enter, values of “**30**” seconds for evacuation, “**10**” seconds for pressure equilibration, and “**30**” seconds to monitor for pressure rise. Click on the check sign or OK button to save these values.

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

- Ensure that all sample canister valves are closed. Using the appropriate sequence file, ensure that the cursor is positioned to begin the leak check procedure at autosampler port #3. Click the “LEAK” button, and click the “GO” button in the pop-up “Leak Check” window. Beginning with “3”, selected autosampler ports will be sequentially leak checked according to the program entered earlier.
- Leak check pressure values are displayed in real time as the sequence proceeds, but it is not necessary to watch these values on the monitor. When all lines have been leak checked, click on “EXIT”.
- Open **Windows Explorer**. Locate the “Smart” directory, and display all files. Click on the “lcr” file that matches the leak check sequence (the “.lcr” extension may not be displayed). Double click to open the file as a WordPad document. Scroll to the bottom of the report file for the most recent report. The leak check report includes the date and time that the sequence was run, followed by a port-by-port listing of initial pressure (at the end of the pressure equilibration stage), final pressure (30 seconds later), and a leak rate, expressed as psi/min. Unless authorized by the laboratory supervisor, only **A LEAK RATE OF LESS THAN OR EQUAL TO 0.2 psi/min. IS ACCEPTABLE**. The analyst must take note of the initial and final pressure values in comparison to previous leak checks. If they are all found to have increased at a similar rate, this may indicate a problem with the diaphragm pump and should be reported to the laboratory supervisor. If the pressures for one port are higher than for the others, this is an indication that there may be a problem with that sample line
- For any port(s) which exceed 0.2 psi/min, re-tighten the Swagelok fitting and ensure that the canister valve is tightly closed. “CUT” all but the affected line(s) from the “Sequence Table”, and repeat the leak check, as described above. When all autosampler lines have been successfully leak-checked, proceed to build the table for the analysis sequence.

2.4 Entering the Entech Sequence

- In the “Sequence Table” window, click on “OPEN” and select a recent, previous analysis sequence file.
- “CUT” table lines for any standard or blank runs which may not be required, and “CUT” lines for any autosampler ports which are not connected to sample canisters.

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

- Ensure that the value in the “**Internal Std. Vol.**” Box is “**50**” mL. Edit if necessary.
- Place the cursor (mouse click) on the first table line that requires editing.
- Edit the “**Sample Name**” box to insert the appropriate information. **For CANMIX standards, enter a standard identifier. For samples, enter the sample identification code found on the canister tag. The canister identification number may also be added. CLICK THE “REPLC” (i.e. replace) BUTTON TO EFFECT THE CHANGE.** Proceed to edit the next box, or if no other changes are required, move the cursor to the next line requiring editing.
- The value in the “**Sample Inlet #**” box is always “**1**” (for autosampler #1), **EXCEPT for the LOW LEVEL calibration standard. Sample Inlet “2” is used for this standard ONLY.** To edit this box, click the “**INLET 2 AUTO**” button, and click “**REPLC**”.
- If necessary, edit the “**Auto Pos #**” box by clicking on the button for the desired autosampler port # (blue “**ASP AUTO**” buttons numbered 1 through 16), and clicking “**REPLC**”.
- Edit the “**Sample Vol.**” box, if necessary, by entering the desired volume and clicking “**REPLC**”.
- The number in the “**Cal. Std. Vol.**” Box should be “**0**” for all runs if necessary, enter “**0**” and click “**REPLC**”.
- Edit the “**Method**” box as described above, if necessary. Make sure that the sequence specifies the **CURRENT** method.
- When all editing changes are complete, review the sequence for possible errors, then “**SAVE**” the sequence table file as the run date.

2.5 Entering the Agilent ChemStation Sequence

- From the “**Start**” menu select “**GC_MS Instrument #1**” and select it again from the sub-menu to load the sample analysis software or use the desktop shortcut. **Load the method “STDBY”, if this method does not load automatically on startup.**
- Under “**View**”, select “**Instrument Control**”.
- Under “**Sequence**”, select “**Load Sequence**”. A list of previous sequence files will pop up. Select a previous sequence for editing and click “**Select**”.

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

- Under “**Sequence**”, select “**Edit Sequence**”. The sequence table will be displayed.
- Enter requested information into the appropriate boxes. The run “**Type**” should be “**Sample**”, for all runs.
- Under “**Vial**”, enter the autosampler port#.
- Under “**Data File**”, enter the appropriate sample, blank, or calibration standard identification code. For the repeat sample, use the same file name as the original run, but with an appended “**R**” or replace the numbers in the code with “**DUP**”.
- In the “**Sample**” and “**Comment/Keyword String**” boxes, enter the sampling date, site name or code, canister information number as well as any relevant comments about the sample.
- Under “**Method**”, enter the name of the CURRENT data acquisition method. All SIM mode acquisition methods are named “**VOCSIM_**”, followed by a number. (Any time the acquisition method is modified it is saved under the same name, but with an incrementally higher number appended).
- When editing is done, ensure that the sequence matches the Entech sequence and that an extra sample called “**STANDBY**” is placed at the end to return the GC/MSD to “**STANDBY**” method when the sequence is complete, then click “**OK**”.
- Under “**Sequence**”, select “**Run Sequence**”. In the pop-up box, ensure that “**Full Method**” is selected.
- Enter operator initials and “**Data File Path**” in the appropriate boxes. Enter the date that the sequence will be started after C:\MSDCHEM\1\DATA\. Be sure to include the day, month and year. Click “**OK**”.
- Under “**Sequence**”, “**Save Sequence**” as the run date, including the day, month and year.
- Under “**Sequence**”, select “**Print Sequence**” and then click on the circle for “**Brief Format**” to print a hardcopy of the sequence table. Or, print a copy of the “**Sequence Log**” that is generated along with the sample data files in a sequence. File this hardcopy in the MSD Sequence Log Book.

2.6 Starting the Entech Sequence

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Page:
5 of 7

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

- Fill the CANMIX standard, internal standard and LOW LEVEL standard canisters to the desired pressure from their reservoirs, if necessary. Open all sample canister valves. Open the valve on the liquid nitrogen tank connected to the preconcentrator. (The system consumes very close to one 169 L tank per sequence).
- Ensure that the “**Sequence Table**” cursor is set to the first line in the analysis sequence, and click “**GO**” to start the sequence running.

2.7 Starting the Agilent ChemStation Sequence

- Under “**Sequence**”, select “**Run Sequence**”. Press the “**Run Sequence**” button approximately 13 minutes after starting the Entech sequence, at the point when the module 3 temperature “**Setpt**” reads “**OFF**”.

2.8 Following Completion of the Sequence

- Close all canister valves and initiate the bake cycle by clicking on the “**Bake**” button in the Entech Sequence Table.
- Each analysis run generates a detailed QA report from the Entech, which lists temperatures, pressures, volumes, and valve positions at each stage of the concentration procedure. These reports must be checked in a timely manner to ensure that any issues are identified as soon as possible. To review the QA reports, go to the “**Sequence Table**” window. Check the date in the “**QA/QC Report**” box, and edit if necessary. The QA file name format is “**R_YYMMDD**”. Click on the “**QAQC**” button. A pop-up box will appear with a listing of QA reports for all runs for the date selected. Select and open a report by double-clicking on the line for any run of interest. A new box will appear with the report for that run. To close the report, double-click anywhere in the report box. Select the report for another run from the displayed listing, or click the “**QAQC**” button to exit from QA reports. **If problems did occur, note or describe them in the Sequence Logbook.** Note that these files may also be accessed via Windows Explorer and found in the Smart folder in the name format described above.
- To close the Entech software, select “**Exit**” in the “**Microscale Purge & Trap**” window, then click on the “**EXIT**” button in the “**Sequence Table**” window.
- Examine chromatograms for all runs for obvious problems (e.g. shifted or poorly resolved peaks, particularly at the start of the chromatogram).

Title: Canister Sampling Analysis Using the Entech Preconcentrator		Copy No: ##
SOP No.: 7.3/3.1/S	Effective Date: September 11, 2013	Location: ###

- Process the data files for internal standard results as described in the Standard Operating Procedure 7.6/*.*S.
- If samples were run successfully (i.e satisfy method criteria), highlight the appropriate instrument code on the canisters' Sample Identification Labels in the designated colour (green for "MSD" or "Non-Polar" and orange for "Polar") to indicate completion of the GC/MS run.

3. Revisions

- Sep 2011:
- Replaced "HP" with Agilent and updated software menu item names
 - Fixed typographical errors and menu selections that were no longer relevant
 - Changed reference point for starting GC/MS sequence and reordered steps to reflect current practice and make instructions clearer
 - Made SOP more generic
 - Removed requirement to print hardcopies of chromatograms
 - Included ability to use available desktop shortcuts
 - Removed revisions from 2003
- Aug 2013:
- Section 2.2 Corrected formatting error and added instructions on how to manually stop the bake cycle
 - Section 2.8 Added a line to indicate that Entech QA/QC Reports must be checked

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