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General Lab SOP

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ABSTRACT

General Lab SOP.

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ABSTRACT

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Vial flushing

When collecting atmospheric gas samples prepare vials by flushing with carrier grade Helium for at least one minute.

1. Cap vials using red butyl septa (Grace).
2. Place 26G 5/8 or 1/2 inch vent needle off-center, through septa.
3. Open stopcocks on vial flushing manifold. Only open as many stopcocks as vials to be flushed (max. 4). Allow helium to flow through inlet needles for 10-15 seconds before mounting vials on inlet needles.
4. Place vial onto 27G 5/8 or 1/2 inch inlet needle on vial flushing manifold. The vials will hang freely once mounted on the inlet need.

Start timer.

1. After flushing for at least one minute, remove vials from manifold one at a time. Close vacant stopcock, to maintain flow through remaining vials. Remove vent needle. Repeat for remaining vials.

Change inlet and vent needles approximately once a week or after 100 flushing cycles.

Serum bottle sampling

125ml serum bottles are used to perform incubations of different types. Samples are collected from these bottles periodically to be run on an autosampler system (Sparky or Sparky Jr.) or by direct injection into a sample loop (Speedy).When sampling, use the appropriate volume syringe for the sample to be collected: Usually a 10ml syringe for 5 or 10ml sample. Use a clean stopcock and needle. A stopcock is needed only when injecting the sample directly into sample loop (Speedy).

Materials:

10 ml syringe
25G 5/8 inch needle
stopcock (if needed)

1. If samples are to be direct injected into the sample loop, place stopcock on the end of the sample syringe, otherwise continue without a stopcock.
2. Flush syringe with air 3 to 5 times.
3. Place needle on syringe and vent syringe down to sample volume (usually 5 or 10 ml).
4. Inject air into serum bottle. Without removing the syringe, mix the gas in the bottle by repeatedly filling the syringe **up to the sample volume** and then emptying syringe into the bottle. Repeat this mixing process 3 to 5 times before extracting the final sample.
5. When extracting the sample pull out the exact sample volume only. If present, close stopcock before removing needle from bottle.
6. Inject sample into He flushed vial or directly into sample loop, as the case may be.
7. Change the needle after 20 to 30 sampling cycles.

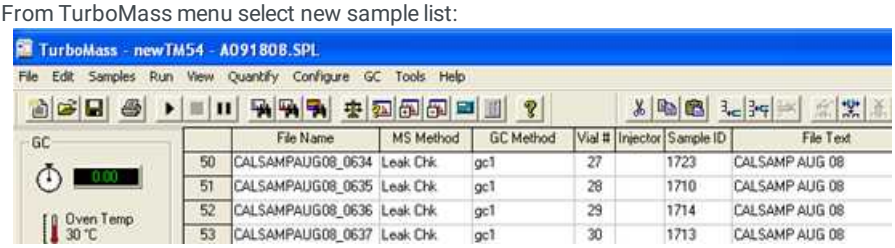
Run samples on the Clarus600 GCMS with HP7694 headspace autosampler (Sparky)

Setting up a sample run requires four main steps:

1. Create a TurboMass sample list.
2. Create a TotalChrom sequence.
3. Load and setup HP7694 headspace autosampler (Sparky)
4. Start the sample run.

How to create a TurboMass sample list

From TurboMass menu select new sample list:



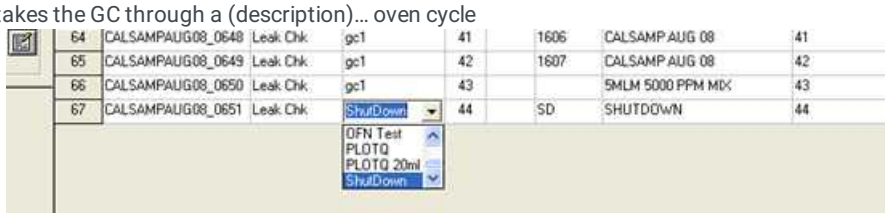
1. Input a unique File Name with a numeric index. This column will autoincrement.
2. Select the MS Method. The current MS Method is **ms method**.
3. Select the GC Method. The current GC Method is **gc method**.
4. Input autosampler carousel position in the Vial # column.
5. Input sample vial ID into the Sample ID column.
6. Type a short description for each sample in File Text, for example CALSAMP AUG 08 or 5000PPM MIX 5ML.
7. Save the Sample List with a distinctive name such as A091808, indicating the first sample run for September 18, 2008.

The first sample of a run is usually a new He flushed vial.

Include one or more check standards approximately every 10-15 sample vials.

The last sample in a run is a MINIBAKEout or SHUTDOWN sample. Change the GC Method to MINIBAKE or Shutdown by double-

clicking the corresponding cell, and selecting the method from the dropdown menu. The Shutdown method turns off the MS and takes the GC through a (description)… oven cycle

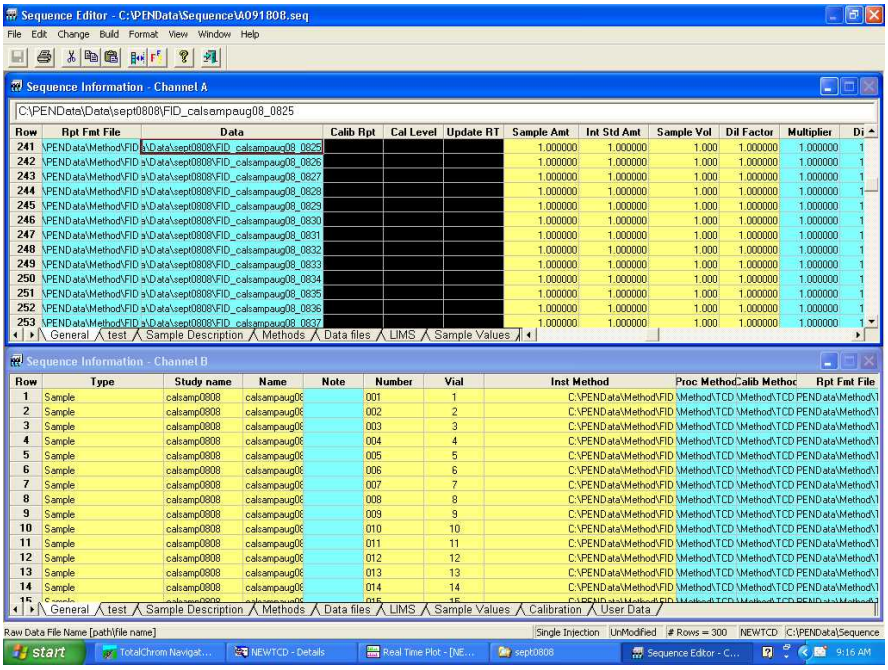


How to create a TotalChrom sequence

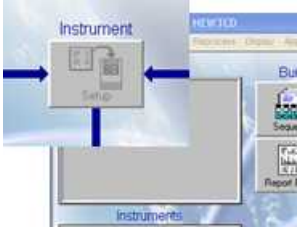
Use a previous sequence as a template for the new sequence to avoid having to input all the sequence information each time. Only the sequence name, and information regarding filenames and sample identification need to be changed for any given run.

Once the template sequence is open:

1. Delete all rows except the first from the window Sequence Information – Channel A (FID). TotalChrom will delete the corresponding rows for Channel B (TCD).
2. Save As…the same filename as the TM Sample List.
3. Edit Data information for the first sample so that it matches the name of the first sample in the TM Sample List. Add the prefix **FID_** to the filename in the Channel A window.



4. Repeat the previous step for the first sample in the Channel B window. Add the prefix **TCD_** to the filename in the Channel B window.
5. Add sample names to the sequence with CTRL-A. There must be at least as many samples in the TC Sequence as there are in the TM Sample List.
6. Save the Sequence. Check that the data path exists otherwise create the corresponding directory using Windows Explorer.
7. In the TotalChrom Navigator click on or select Instrument Setup.



When the TC Status window shows “Active”, TC is ready to run samples.

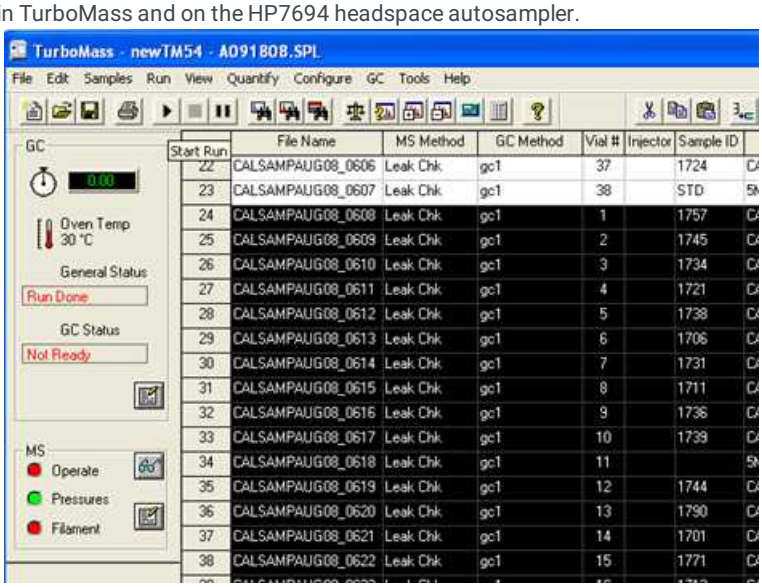
How to load and setup HP7694 headspace autosampler (Sparky)

1. Place vials in appropriate carousel positions according to TM sample list.
2. Set Vial Parameters: First and last vial to the corresponding first and last vial positions in the carousel.
3.
4. Set vial parameters to correct first and last vial position. Use the 7694 headspace keypad buttons to select first and last vial positions.

5. Key in the correct number using the numeric keypad and press Enter.

How to start the sample run

1. The sample run must be started in TurboMass and on the HP7694 headspace autosampler.
2. Highlight the samples to be run.

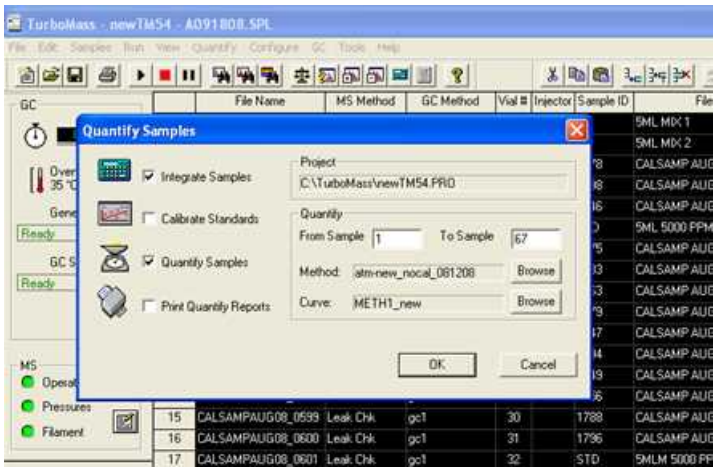


3. Press Play and click OK.
4. When TurboMass shows General Status Ready and GC Status Ready
5. Go to the HP7694 and begin the sample run.
6. The autosampler will reposition the carousel if necessary, and load the first vial.
7. A sample run will generate three data sets, one for each detector. These need to be output to a text file individually.

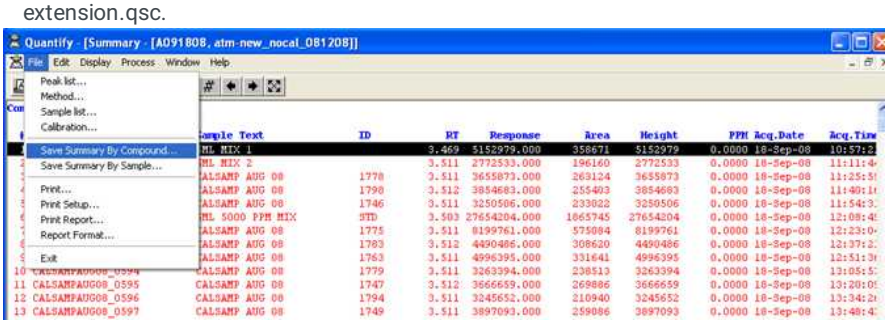
Quantify TurboMass Sample List

1. Highlight the samples in the sample list to be processed.
2. Select Process from the Quantify menu.
3. The current quantify method and curve are **atm-new_nocal_081208** and **METH1_new**.

4. Click OK.



5. Click on the Quantify window.
6. Select Save Summary By Compound from the File menu. The summary file will be stored in the TurboMass\newTM54 with extension.qsc.



Channel A and B must be summarized separately.

1. Select Summarize from menu or click on Summarize button.
2. Click on Summarize by Sequence button
3. Select sequence file
4. Set Starting and Ending row
5. Check box Load files from Channel A (Channel B) to summarize FID (TCD) results.
6. Select Report format file: FIDSummary.sum (TCDSummary.sum)
7. Check box Load component list from method.
8. Click OK to begin summary process.