CAUTIONS **CE440**

Capsules

Use Aluminum capsules only for easy to burn samples. Be very very careful not to drop the Aluminum capsule onto the quartz combustion tube. The Aluminum with eat into the quartz.

Use Tin capsules only with Nickel sleeves. Crimp the capsules shut with the tweezers or pliers. Insert the capsule (with one end flattened by the seal) into a Nickel sleeve.

Weighing

Weigh very very carefully. Your results will only be as accurate as your ability to weigh. DO NOT push down on the hang down.

Tare (zero) each and every capsule. They look similar but are not similar in the "microgram" world. Their weight can vary by as much as a few 100 micrograms.

Let the tare and the actual weighing step settle before taking a reading. This should take about 20 seconds.

We recommend a sample weight around 2000 micrograms.

FillTime

This is FT on the printer output. FT is the metric we use to ensure that the Helium regulator is stable. FT should be between 20 and 50 secs and not vary by more than +/3 seconds between samples. It is the actual time needed to reach 1500 mmHg in the mixing volume.

Inserting samples on a Manual system

Be very careful to follow the sample insertion script carefully.

Step 1 is to remove the entrance plug and then click OK.

Step 2 involves removing the ladle (the quartz rod holding sample), dumping the spent sleeve, popping on a new sample, inserting the ladle and then replacing the entrance plug. Then click OK.

Step 3 (around 35 secs into the run) is to insert the ladle into the furnace.

It is EXTREMELY important not to wait when prompted for step 3. Doing so will lead to poor analysis results.

The ladle can be withdrawn from the furnace anytime after the FT stage, that is the stage where the A and D valves are finished pulsing. This is the stage where you can hear the valves going click, clack, click. You can remove the ladle to let it cool after this stage. Do not open the entrance plug at this point. Simply withdraw the ladle from the furnace into the front end of the combustion tube. This allows it to cool before the next run.

It is also important not to tip (rotate) the ladle when it is in the furnace. The dumped capsule may damage the combustion tube. I find it helpful to practice moving the ladle in and out with the magnet without a capsule present to practice. One can quickly get the hang of it.

Conditioning

We will often refer to "conditioning". This is running an unweighed sample or two (you can weigh them if you wish but there is no need to) before an actual sample or calibration in order to saturate the internals of the 440 with water vapor and CO2 gas. This will give you more accurate results when running your samples.

The conditioner should be of the same composition and weight as the samples for best results. For example, the conditioning step before a calibration with acetanilide (weight around 2000) should best be 2000 micrograms of acet. There is no need to weight the sample as 1000 – 4000 would be just fine for the conditioning step. Just eyeball the weight. This becomes quite easy with some weighing experience.

Reserved Sample ID

The following sample ID's are reserved for the 440 software.

BLANK: Indicates to the 440 that you are running a BLANK, be it an EMPTY blank (no capsule or sleeve) or a CAPSULE blank (capsule and sleeve)

Always enter a weight of 0 for a blank.

STD1-5: Indicates that the material in the capsule is to be used to calibrate the system. STD1 is acetanilide for example.

Enter the actual sample weight.

COND: Not strictly reserved but used to indicate a Conditioner run by EAI personnel.

Enter a weight of 2000.

In addition, if you enter a sample weight of 100, your results will be printed in absolute micrograms rather than percentage.

Other helpful hints

* NEVER, NEVER run out of Helium.

* Do NOT hard pack the Scrubbers, Traps or Combustion tube.

* DO pack the copper wire into the Reduction tube as hard as you can.

* You MUST use Nickel sleeves if you use tin capsules.

* When running a MANUAL system, make sure you inject the sample as soon as prompted to do so. The prompt comes about 35 seconds into the run. You can remove the ladle after the fill time end (after the click-click-click).

* Hand purge valves on Automated (SHA) system: Do not over tighten the hand purge valves in the system. They use a Teflon ball as a seal and over tightening crushes the ball, causing it to leak at a later date.

* End Connector: Always use two (2) tube nut wrenches when removing or tightening the end connector. Steady the end connector with one tube wrench while turning the tube nut with another. Failure to do so will flex the weld joint at the connecting stainless tube and eventually break the seal, leading to a leak.