

CN Analyzer Sample Preparation Instructions
Version 1.0
1/17/08

Liquid Samples on the TOC-V:

- 1) Filter sample through a 0.45 um filter.
- 2) Pipette sample into a 40 ml septa-capped vial.

Liquid Samples on the SSM:

- 1) Use only clean sample boats and tweezers to minimize contamination. See the section titled "Sample Boat Cleaning" if boats need to be cleaned. The tip of the tweezers can be heated in a Bunsen burner or furnace to oxidize any attached carbon.
- 2) There are two methods for preparing liquid samples for analysis:
 - i) Weigh the sample directly into a tared sample boat
 - ii) Inject a specific volume of sample directly into a tared sample boat
 - The maximum measurable mass of a liquid sample is 0.5 g for TC and 0.3 g for IC.
 - For best accuracy, use a volume of 100 ul or less for aqueous-based solutions.
 - Samples that are measured by weight will be treated as solid samples by the software (for calculation purposes).
 - For TC analysis, the sample can be impregnated into clean ceramic fiber placed on the bottom of the sample boat (if desired). Ceramic fiber is not used for IC analyses.
- 3) The measurement ranges for the analyses are: TC: 0.1 – 30 mg C, IC: 0.1 – 20 mg C.

Solid Samples:

- 1) Use only clean sample boats and tweezers to minimize contamination. See the section titled "Sample Boat Cleaning" if boats need to be cleaned. The tip of the tweezers can be heated in a Bunsen burner or furnace to oxidize any attached carbon.
- 2) Pulverize and homogenize the sample. The final sample should be able to pass through a 200-mesh sieve.
- 3) Dry the sample.
- 4) Tare a sample boat and weigh the sample directly into the boat. Record the sample weight. Try to spread the sample evenly throughout the boat. The maximum sample weight is 1 g, and the measurement ranges for the analyses are: TC: 0.1 – 30 mg C, IC: 0.1 – 20 mg C.
- 5) Alkaline samples, samples with large particles, and samples with a greater IC content than TOC content cannot be analyzed for IC content. These samples must be pre-treated (usually by reacting with HCl) to remove IC before they can be analyzed for TC content. Ensure that the samples are dried after treatment to remove residual acid.
- 6) When analyzing a sample that may scatter during combustion, cover the sample with clean ceramic fiber.

- 7) Pre-moisten carbonate samples with a small amount of water if they will be analyzed for IC content.
- 8) Sample additives may be required or desired to accelerate combustion or thermal decomposition reactions. For samples containing alkaline metals or alkaline earth metals, tungsten oxide powder or vanadium pentoxide may be spread over the entire sample. Take great care if using vanadium pentoxide; it is a very hazardous chemical! Tungsten oxide may also be used for samples containing phosphorus.
- 9) Use standards (for the calibration curve) that are as similar to the sample(s) as possible.

Sample Boat Cleaning:

- 1) Brush or scrape out as much of the residual sample in the boat as possible.
- 2) Soak the boat in 2M HCl for 10 minutes.
- 3) Wash thoroughly with tap water, rinse with e-pure water, and oven dry.
- 4) For TC analysis, additionally bake the dried boat in a furnace at 900C for 20 minutes.
- 5) New ceramic fiber can also be cleaned by baking in a furnace at 900C for 20 minutes. Used ceramic fiber should be discarded.