

# Application Note 15



## DETERMINATION OF INORGANIC CARBON IN BLACK LIQUORS



Figure 1: CM140 Total Inorganic Carbon (TIC) Analyzer

#### PRINCIPLES OF OPERATION

Black liquor samples are acidified with acid to release CO<sub>2</sub>. Carbon dioxide-free air sweeps the evolved CO2 from the heated reaction chamber through an air-cooled condenser, scrubbers and into the CO2 Coulometer where it is measured by automatic, coulometric titration.

### **PROCEDURE**

1. Assemble the apparatus as described in the Instruction Manual with the following addition.

Install the auxiliary scrubber between the sample column exit and sidearm of the postscrubber. Fill the auxiliary scrubber and the post-scrubber with a silver nitrate (AgNO<sub>3</sub>) or silver sulfate (AgSO<sub>4</sub>) solution.

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- 2. Determine the blank and run a standard to confirm proper operation of the system.
- 3. Charge the sample flask with a known weight of the black liquor sample. The sample size should be selected to contain 1 3 mg of carbon.
  - The sample can be weighed directly into the sample flask or into porcelain, platinum, alumina or similar type sample containers. The sample container is then placed into the sample flask.
- 4. Attach the sample flask to the apparatus. Allow approximately one (1) minute for the system to purge itself of CO<sub>2</sub>.
- 5. Move the sample flask into position over the heater, pump acid into the reaction chamber, and start the coulometer.
- 6. When all of the CO<sub>2</sub> is evolved and titrated, the CM5017 automatically detects the endpoint, ends the analysis, saves results on a USB Flash Drive and/or prints the result to the printer\*.
- (\*) Endpoint determination and result calculations are performed automatically based on user selectable settings entered into the CM5017 Carbon Coulometer.

#### **RESULTS**

When samples contain over 1 mg of C, the titration accuracy is +/-0.1% relative. Overall accuracy is typically +/-0.3% relative. When sample availability or volume limits the amount of CO<sub>2</sub> evolved to small amounts, the accuracy is generally better than 1 µg C.

Analysis times are typically 5 - 7 minutes. Poor miscibility of the sample with the acid can extend the analysis time. A 50/50 mixture of kerosene and iso-propanol can be added to the sample to improve miscibility.

For information about the instrument's capabilities for specific types of samples, contact the UIC, Inc. or UIC Europe.

Contact us for more information: