

## ***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 CO<sub>2</sub> from the heated reaction chamber through an air cooled condenser, scrubbers and into the CO<sub>2</sub> 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 the fritted sample (silver) scrubber. Fill the auxiliary scrubber and the sample (silver) scrubber with a silver nitrate (AgNO<sub>3</sub>) or silver sulfate (AgSO<sub>4</sub>) solution.
2. Determine the blank and run a standard to confirm proper operation of the system.

3. Charge the sample tube 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 tube or into porcelain, platinum, alumina or similar type sample containers. The sample container is then placed into the sample tube.

4. Attach the sample tube to the apparatus. Allow approximately one (1) minute for the system to purge itself of CO<sub>2</sub>.
5. Move the sample tube 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 CM5014 automatically detects the endpoint, ends the analysis and prints the result to the printer and/or diskette. \*

*(\*) – Endpoint determination and result calculations are performed automatically based on user selectable settings entered into the CM5014 CO<sub>2</sub> Coulometer.*

## **RESULTS**

When samples contain over 1 mg 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 ug 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.