

DETERMINATION OF CARBON IN SULFUR



Figure 1: Model CM120 TC/TOC Analyzer

PRINCIPLES OF OPERATION

Sulfur is burned in the combustion apparatus converting the sulfur to SO_2 and SO_3 and carbon to CO_2 . The SO_2 and SO_3 are removed from the oxygen carrier gas by reacting with the barium chromate combustion catalyst/scrubber forming barium sulfate. The CO_2 is swept through further post scrubbers to remove other acidic combustion gases and into the CO_2 Coulometer where it is automatically measured by coulometric titration.

INSTRUMENT SET-UP

1. The Combustion Apparatus is assembled and operated according to the regular instructions for solid samples.
2. Fill the coulometer cell using just enough solution so the light beam passes through the solution without any interference from the stir bar or the stirring vortex (approximately 50ml). A small piece of cardboard or some other non-metallic material can be placed under the cell to raise the cell assembly so the stir bar is just under the light beam. The lower volume of cell solution will help to improve sensitivity.
3. Allow the coulometer cell and the combustion system to stabilize.

NOTE: In some cases, filling the cell the evening before use may improve stability, although care must be taken to keep an excess of potassium iodide (KI) in the anode compartment and to avoid silver deposits from accumulating on the platinum electrode.

PROCEDURE

1. Determine the blank and run a standard to confirm proper operation of the system.
2. Weigh the sample into a platinum boat. No more than 50-100 mg of sample should be used. Larger samples may burn too violently and can rapidly deplete the barium chromate's scrubbing capabilities.

NOTE: The barium chromate theoretically has a capacity to remove over 6 g of sulfur with an actual efficiency of approximately 30 to 60%.

3. Place the sample into the ladle and place the ladle into the unheated portion of the combustion tube. Replace the breech block cap and allow the system to purge for approximately one (1) minute.

NOTE: Great care must be taken to avoid contamination of the boats and the ladle. Precautions, such as wearing gloves, are recommended.

4. Slowly move the sample just into the combustion zone. Allow the sulfur to burn off before moving the ladle all the way into the heated zone.
5. When all of the CO₂ 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 CO₂ Coulometer.*