

DETERMINATION OF ORGANIC OXYGEN



Figure 1: Model CM122 Total Oxygen Analyzer

PRINCIPLES OF OPERATION

The method for the determination of organic oxygen is similar to one originally described by Schutze and developed by Zimmerman and Unterzaucher. Samples are pyrolyzed in a stream of nitrogen or helium. The pyrolyzed products are swept over carbon at 1120°C, converting all of the oxygen to carbon monoxide, CO. After all of the acidic gases are scrubbed from the stream, the carbon monoxide is oxidized to CO₂. The carbon dioxide is then swept to the CO₂ Coulometer where it is automatically measured by coulometric titration.

APPLICATIONS

The method is generally applicable to organic oxygen. Inorganic forms of oxygen are determined if the components are reduced or oxygen displaced at 1120°C. Oxygen in refractory metal oxides will not be determined. Reducible oxides will be determined if the sample contains sufficient carbon to reduce the oxide or if a reducing or displacing agent is added to the sample. (See Kapron and Brandt, *Analytical Chemistry* 33:1762, 1961 and Mertz, *Analytica Chemica Acta* 50:305, 1970.) Fluorine causes a positive interference by displacing oxygen in the pyrolysis tube.

PROCEDURES

1. Assemble and prepare the components for operation as described in the Instruction Manuals.
2. Determine the blank and run a standard to confirm proper operation of the complete system.
3. Weigh the sample into a platinum boat.
4. Place the boat in a cool ladle.
5. Insert the ladle into the unheated end of the combustion tube.
6. Place the breech block cap on loosely and increase the carrier gas flow to 80 cc/minute to back sweep the system for 30 to 60 seconds.
7. Decrease the gas flow to 20 cc/minute and tighten the cap. Begin the analysis by pressing "Enter" on the CM5014 CO₂ Coulometer and move the ladle into the furnace zone.
8. After four (4) minutes, increase the gas flow to 80 cc/minute.
9. After three (3) additional minutes, the result is printed to the printer and/or diskette and the ladle is withdrawn from the furnace. * (NOTE: The coulometer can be set to calculate micrograms oxygen for this analysis. See the CO₂ Coulometer Instruction Manual for details.)
10. Allow the ladle to cool before proceeding to the next analysis.

() – Endpoint determination and result calculations are performed automatically based on user selectable settings entered into the CM5014 CO₂ Coulometer.*

RESULTS

Oxygen may be determined in the range of 0.5 to 100% with an absolute accuracy of +/-0.3 to 0.5%. The lower level is limited by the variability of the instrument blank and may be lower with some sample types or alternate procedures. Typical sample weights are 2 to 10 mg and analysis times are under ten (10) minutes.

Some actual test results on standards are given below:

<u>Compound</u>	<u>% Oxygen Theoretical</u>	<u>% Oxygen Found</u>	<u># of Repetitions</u>	<u>Range</u>
Benzoic Acid	26.21	26.18	11	+/-0.33
Stearic Acid	11.25	11.27	11	+/-0.19
Triphenylmethane	0.00	0.08	6	+/-0.05