

Application Note 20



DETERMINATION OF BICARBONATE AND CARBONATE IN CONSUMER PRODUCTS



Figure 1: CM140 Total Inorganic Carbon (TIC) Analyzer

PRINCIPLES OF OPERATION

Samples are heated to 150 °C to release CO₂ from the bicarbonate portion of the sample. A carbon dioxide-free carrier gas sweeps the evolved CO2 from the heated reaction flask into the Carbon Coulometer. The Carbon Coulometer automatically titrates the evolved CO₂. After the CO₂ from the bicarbonate has been titrated, the sample is then treated with acid to release CO₂ from the carbonate portion of the sample. This CO₂ is then swept into the coulometer and titrated.

PROCEDURE

- 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.

Contact us for more information:

UIC Europe +351 910-908-188 zjovanovic@uic-europe.com www.uic-europe.com



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- 3. Charge the sample tube with a known weight of sample and attach the tube to the apparatus. Sample size ideally should be selected to contain 1000 μ g 3000 μ g carbon.
- 4. Allow approximately one minute for the system to purge itself of atmospheric CO₂ after attaching the sample tube.
- 5. Move the sample tube into position in the heater block and start the coulometer.
- 6. When all the CO₂ from bicarbonates is evolved and titrated (recognized by a stable coulometer display) record the display value and calculate the results.
- 7. Without removing sample tube from the heater block, introduce acid into the reaction flask, approximately 10 ml.
- 8. When all CO₂ from carbonates is evolved and titrated, the CM5017 automatically detects the endpoint, ends the analysis and prints the result to a USB Flash Drive and/or 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 1000 μ g C, the titration accuracy is better than +/- 0.15 % relative. Overall accuracy is typically +/- 0.3 % relative. When sample availability limits the amount of CO₂ evolved to small amounts, the accuracy is generally better than 1 μ g C.

Total analysis times are typically 15 to 20 minutes. For some samples, the reaction time with either the heat or the acid is very slow, thus extending the analysis time. A major advantage of the Carbon Coulometer is that no chemical calibration is required, also the analysis completion can be seen, avoiding low results due to incomplete analysis times or wasted time due to overly long analysis times. Other advantages include the easy addition or modification of scrubbers, the ability to use different acids and the ease of using wetting/emulsifying agents and indicators in the acid.

Bicarbonate (sample heated to 150 °C): $2NaHCO_3 \xrightarrow{\Delta} Na_2CO_3 + CO_2 + H_2O$ Carbonate (added acid to sample): $Na_2CO_3 + 2H^+ \rightarrow CO_2 + H_2O + 2Na^+$

% NaHCO₃ =
$$\frac{2 \cdot (\mu \text{g C from heating}) \frac{\text{FWNaHCO_3}}{\text{FWC}}}{\mu \text{g Sample}} \cdot 100 = \frac{2 \cdot (\mu \text{g C from heating}) \cdot 6.99}{\mu \text{g Sample}} \cdot 100$$

% Na₂CO₃ = $\frac{(\mu \text{g C from acid} - \mu \text{g C from heating}) \frac{\text{FWNa_2CO_3}}{\text{FWC}}}{\mu \text{g Sample}} \cdot 100 =$
= $\frac{(\mu \text{g C from acid} - \mu \text{g C from heating}) \cdot 8.82}{\mu \text{g Sample}} \cdot 100$

ADDITIONAL INFORMATION

For information about the instruments capabilities for specific types of samples, or to have a sample analysis completed, contact the UIC, Inc. Applications Laboratory or UIC Europe.

Contact us for more information:

UIC Inc. +1 815-744-4477 sales@uicinc.com www.uicinc.com