UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-TOC-1601, Rev. 3
Effective Date: 08/29/89

CARBON DETERMINATION- I. R. DETECTION

Approved by:

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Analytical and Process Chemistry

Part I

1.0 PURPOSE

To determine total inorganic carbon (TIC), total organic carbon (TOC) and total carbon (TC) in solutions and slurries (nonradioactive and radioactive).

2.0 SCOPE

This method may be used on a large variety of samples ranging from waste water to melter feed. A working range of 0.1 to 10,000 ppm allows the testing of most samples directly.

3.0 DISCUSSION

3.1 Total Inorganic Carbon (TIC) is determined by the measurement of carbon dioxide released by acidification of a sample. As pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. This carbon dioxide is purged from solution, concentrated by trapping, then desorbed and carried into a non-dispersive infrared analyzer (NDIR) which has been calibrated to directly display the mass of carbon dioxide detected. This mass is equivalent to the mass of TIC in the sample. Concentration of TIC is calculated by dividing this mass by the sample volume.

3.2 Total Organic Carbon (TOC) is determined by the measurement of carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged on

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TIC, sodium persulfate ($Na_2S_2O_8$), a strong oxidizer, is added. This oxidant quickly reacts with organic carbon in the sample at 100°C to form carbon dioxide. When the oxidation reaction is complete, the carbon dioxide is purged from the solution, concentrated by trapping, and detected as described for TIC. The resulting carbon mass in the form of carbon dioxide is equivalent to the mass of organic carbon originally in the sample.

3.3 Total Carbon (TC) is the sum of TOC and TIC in the sample.

4.0 REFERENCES

4.1 O. I. Corporation Model 700 Carbon Determinator manual

Part II

5.0 EQUIPMENT

5.1 O. I. Corporation Model 700 Carbon Determinator

5.2 High Purity Nitrogen - adjust flow to levels indicated on flow gauges. (Refer to operating manual)[1]

6.0 REAGENTS AND STANDARDS

6.1 Sodium Persulfate (100 g/L): Prepare a 100 g/L solution of sodium persulfate by dissolving 100 g $Na_2S_2O_8$ into reagent water (1 litre total volume). Stirring may be necessary but do not heat. Transfer a portion of this solution to the appropriate reagent bottle provided with the instrument. Place the lid on the reagent bottle, but DO NOT tighten. Place the bottle in a microwave oven and heat until the solution just comes to a boil. Immediately tighten the lid and immerse in water to cool. This procedure purifies the $Na_2S_2O_8$ solution by reducing (but not eliminating) TOC content of reagent water added during solution of the crystals. The cooled solution should then be purged with inert gas for several minutes to remove any CO_2 from oxidation of organics (the Model 700 provides reagent bottle purge lines). This has a shelf life of 6 months.

6.2 Phosphoric Acid (5 percent vol/vol): OI Part #110-080 for 85 percent acid.. Prepare a 5 percent by volume solution of phosphoric acid by adding 59 mL of ACS reagent grade 85 percent H_3PO_4 to reagent water (1 litre total volume). This has a self life of 1 year.

All standards are to be made using RO water.

(1) These gauges are preset and should not require an adjustment.

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6.3 Calibration stock solution (1000 ppm C-potassium biphthalate) 2.125g/1000 mL (previously dried to constant mass at 110°C).

Shelf life 6 months.

Calibration Working Standard (50 ppm C) made from stock solution

Shelf life 2 months.

QC Standard (50 ppm C) sucrose 0.1186 g/L

Shelf life 2 months.

7.0 SAFETY PRECAUTIONS

Standard laboratory safety practices should be followed. (ACP 7.2)

8.0 RECORDS

8.1 All measurement data and sample identification shall be recorded on the worksheet (Attachment A). The final result will be recorded on the analytical request sheet. Per PRD 5.0

9.0 CALIBRATION AND CONTROL

9.1 A quality control sample is run for each batch of samples of 12 or less. If the results are out of tolerance, the instrument is recalibrated and the run repeated

9.2 Record quality control sample result in the QA log book and on the quality control chart. Per ACP 8.1

9.3 Record the calibration constants in log book when calibration is done.

10.0 PROCEDURE

10.1 Check reagents (fill reagent bottles if low).

10.2 Check drain bottle

10.3 Turn on nitrogen valve (at left of instrument).

10.4 Turn on power switch.

- check gas flow
- leak test (at least once a week)
- adjust IR output to 1 to 10 MV (if necessary)

10.5 Syringe injection:

10.5.1 Set system configuration (Selection #1).

A. Sample loop - disabled.

B. Sample loop valve - disabled.

C. Auto-run analysis - disabled.

10.5.2 Calibration

A. Run blanks to determine clean system.

B. Run calibration standard (50 ppm C-KHP) three times.

1. Press run/stop.

2 Inject 0.40 mL.

3. Press run/stop.

C. Take average millivolt readings from the printer.

D. Display "Calibration Constants".

1. Enter appropriate analysis desired.

2. Leave blank values as indicated.

3. Enter standard mass= (ppm x mL injected).

4. Enter average millivolts from calibration.

5. Scaling factor should be approximately 0.05.

10.5.3 Select "Normal ppm C" display.

10.5.4 Prepare samples by diluting weighted aliquots of original samples with RO in glass volumetrics, so that final carbon concentrations are within the calibration range of the instrument.

10.5.5 Press Run/Stop button and inject 0.40 mL of sample into the instrument when instructed on the display.

10.6 To turn off instrument.

10.6.1 Turn off power switch on the instrument.

10.6.2 Turn off nitrogen (using valve on left).

10.7 For further information consult the Model 700 O. I. Corporation Carbon Determinator Manual.

11.0 CALCULATIONS

11.1 ppm C = ppm C injected sample X D.F.

D.F. = Dilution Factor = $\frac{\text{sample aliquot weight}}{\text{total weight of solution}}$

12.0 ATTACHMENTS

12.1 ATTACHMENT A Carbon Worksheet

ATTACHMENT A

CARBON WORKSHEET

SAMPLE NAME _____ LOG NUMBER _____

SPECIAL INSTRUCTIONS _____

_____

_____

Carbon Analyzer Used: cold model hot model
(circle one)

SAMPLE ID							
WT OF ALIQUOT							
TOTAL WT OF SOLUTION							
DF							
INITIAL TIC							
INITIAL TOC							
INITIAL TC							
FINAL TIC							
FINAL TOC							
FINAL TC							

ANALYST: _____ DATE _____

APPROVED: _____ DATE _____