

APPENDIX "L"

11/29/94

Method to Determine Total Organic Carbon for
Offset Lithographic Solutions

a. Applicability and Principle.

1. Applicability. This method is applicable for the determination of organic carbon in diluted offset lithographic solutions.
2. Principle. Organic carbon in a sample is converted to carbon dioxide (CO₂) by catalytic combustion or wet chemical oxidation. The CO₂ formed can be measured directly by an infrared detector or converted to methane (CH₄) and measured by a flame ionization detector. The amount of CO₂ or CH₄ is directly proportional to the concentration of carbonaceous material in the sample.

b. Sensitivity and Interferences.

1. Sensitivity. The method is most applicable to measurement of organic carbon above 1 mg/L.
2. Interferences. All distilled water used in making and/or diluting the samples must be acidified with concentrate H₃PO₄ (1 mL of H₃PO₄/1 L of water) and purged with inert gas (He, N₂...) for at least 30 minutes. Inject this water into the Total Organic Carbon analyzer and determine the total concentration (ppm C) of the blank. This method is sufficient for removing most interferences due to inorganic carbon in the water. Do not purge the sample with an inert gas since purging may result in the loss of volatile organic substances.

c. Apparatus.

1. Blender. Waring-type or similar, for blending or homogenizing samples.
2. Total Organic Analyzer. An analyzer capable of measuring carbonaceous material in liquid samples. Consideration should be given to the types of samples to be analyzed, the expected concentration range, and the forms of carbon to be measured.
3. Volumetric Flasks and Volumetric Pipets. For preparing standard solutions and the lithographic solutions.
4. Glass Bottles. For sample collection and storage.

d. Reagents.

1. Water. Distilled water used in preparation of standards and for dilution of samples should be ultra-pure to reduce the carbon concentration of the blank. Carbon dioxide-free, double-distilled water is recommended. Ion-exchanged waters are not recommended because of the possibilities of contamination with organic materials from the resins.
2. Potassium Hydrogen Phthalate, Stock Solution. 1000 mg carbon/L. Dissolve 0.2128 g of potassium hydrogen phthalate (Primary Standard Grade) in distilled water and dilute to 100.0 mL.
3. Potassium Hydrogen Phthalate, Standard Solutions. Prepare standard solutions from the stock solution by dilution with distilled water.
4. Blank Solution. Use the same distilled water (or similar quality water) used for the preparation of the standard solutions.

e. Sample Preparation.

1. Prepare the offset lithographic solutions according to the manufacturer's directions.
2. Dilute the offset lithographic solutions with H₂O to be within the calibrated range of the instrument before analyzing. Dilutions of 1 to 100 or greater may be necessary before lithographic solutions can be analyzed.

f. Procedure.

1. Follow the instrument manufacturer's instructions for calibration, procedure, and calculations.
2. Calibrate using at least 3 standards. The set of calibration standards should consist of one below the expected concentration, one above the expected concentration, and one approximately at the expected concentration.
3. Calculate and report the results as mg C/g sample.

g. Precision and Accuracy.

1. Precision and accuracy determinations for diluted offset lithographic solutions were determined by the method of standard addition. Two analysts in one laboratory analyzed spiked and unspiked diluted solutions. The results are

contained in citation 4 of the Bibliography.

2. Precision and accuracy determinations for diluted lithographic cleaning solutions were determined by the method of standard addition. One analyst in one laboratory analyzed spiked and unspiked diluted solutions. The results are contained in citation 4 of the Bibliography.

h. Bibliography.

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D 2574-79, p. 469 (1976).
2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 532, Method 505, (1975).
3. Method 415.1, Methods for Chemical Analysis of Water and Wastes, Environmental Monitoring and Support Laboratory, USEPA, Cincinnati, OH 45268, EPA 600/4-79-020.
4. Evaluation of Method 415.1 for Offset Lithographic Solutions. September, 1992.