

METHOD #: 335.3 (Issued 1978)
TITLE: Cyanide, Total (Colorimetric, Automated UV)
ANALYTE: CN Cyanide
INSTRUMENTATION: Autoanalyzer
STORET No. 00720

1.0 Scope and Application

- 1.1 This method is applicable to the determination of cyanide in drinking and surface waters, domestic and industrial wastes
- 1.2 The applicable range is 5 to 500 µg/L

2.0 Summary of Methods

- 2.1 The cyanide as hydrocyanic acid (HCN), is released from cyanide complexes by means of UV digestion and distillation. Cyanides are converted to cyanogen chloride by reactions with chloramine-T which subsequently reacts with pyridine and barbituric acid to give a red-colored complex

3.0 Sample Handling and Preservation

- 3.1 The sample should be collected in plastic bottles of 1 liter or larger size. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers
- 3.2 Samples must be preserved with 2 mL of 10 N sodium hydroxide per liter of sample (pH > or = 12) at the time of collection
- 3.3 Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C.
- 3.4 Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume

4.0 Interferences

- 4.1 Thiocyanates are a positive interference. During the UV digestion thiocyanates are decomposed to cyanide
- 4.2 Sulfides adversely affect the colorimetric procedure. If a drop of the sample on lead acetate test paper indicates the presence of sulfide, treat 25 mL more of the stabilized sample (pH > or = 12) than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of

the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

5.0 Apparatus

5.1 Technicon Auto Analyzer

- 5.1.1 Sample
- 5.1.2 Manifold with UV digester
- 5.1.3 Proportioning pump
- 5.1.4 Heating bath with distillation coil
- 5.1.5 Distillation head.
- 5.1.6 Colorimeter equipped with a 15 mm flowcell and 570 nm filter.
- 5.1.7 Recorder

6.0 Reagents

- 6.1 Distillation reagent: Carefully add 250 mL of 85% phosphoric acid and 50 mL of hypophosphorus acid to 700 mL of distilled water, mix and dilute to one liter with distilled water
- 6.2 Phosphate buffer, pH 5.2: Dissolve 13.6 g of potassium dihydrogen phosphate and 0.28 g of disodium phosphate in 900 mL of distilled water and dilute to one liter
- 6.3 Chloramine-T: Dissolve 2.0 g of chloramine-T in 500 mL of distilled water
- 6.4 Pyridine barbituric acid reagent: Place 15 g of barbituric acid in a one liter beaker. Wash the sides of the beaker with about 100 mL of distilled water. Add 75 mL of pyridine and mix. Add 15 mL of conc. HCl and mix. Dilute to about 900 mL with distilled water and mix until all the barbituric acid has dissolved. Transfer the solution to a one liter flask and dilute to the mark
- 6.5 Sodium hydroxide, 1 N: Dissolve 40 g of NaOH in 500 mL of distilled water and dilute to one liter
- 6.6 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 900 mL of distilled water and mix. Dilute to one liter. Standardize with 0.0192 N AgNO₃ to appropriate concentration. 1 mL = 1 mg CN
- 6.7 All working standards should contain 2 mL of 1 N NaOH (6.5) per 100 mL

7.0 Procedure

- 7.1 Set up the manifold as shown in Figure 1 in a hood or a well-ventilated area
- 7.2 Set temperature of the heating bath at 150°C
- 7.3 Allow colorimeter and recorder to warm up for 30 minutes. Run a baseline with all reagents, feeding distilled water through the sample line
- 7.4 Place appropriate standards in the sampler in order of decreasing concentration. Complete loading of sampler tray with unknown samples.
- 7.5 When the baseline becomes steady begin the analyses

8.0 Calculation

8.1 Prepare standard curve by plotting peak heights of standards against concentration values. Compute concentrations of samples by comparing sample peak heights with standards

9.0 Precision and Accuracy

9.1 Precision and accuracy data are not available at this time

Bibliography

1. Technicon AutoAnalyzer II Methodology, Industrial Method No. 315-74 WCUV digestion and distillation, Technicon Industrial Systems, Tarrytown, N.Y., 10591, (1974).
2. Goulden, P. D., Afghan, B. K. and Brooksbank, P., Anal. Chem 44, 1845 (1972)

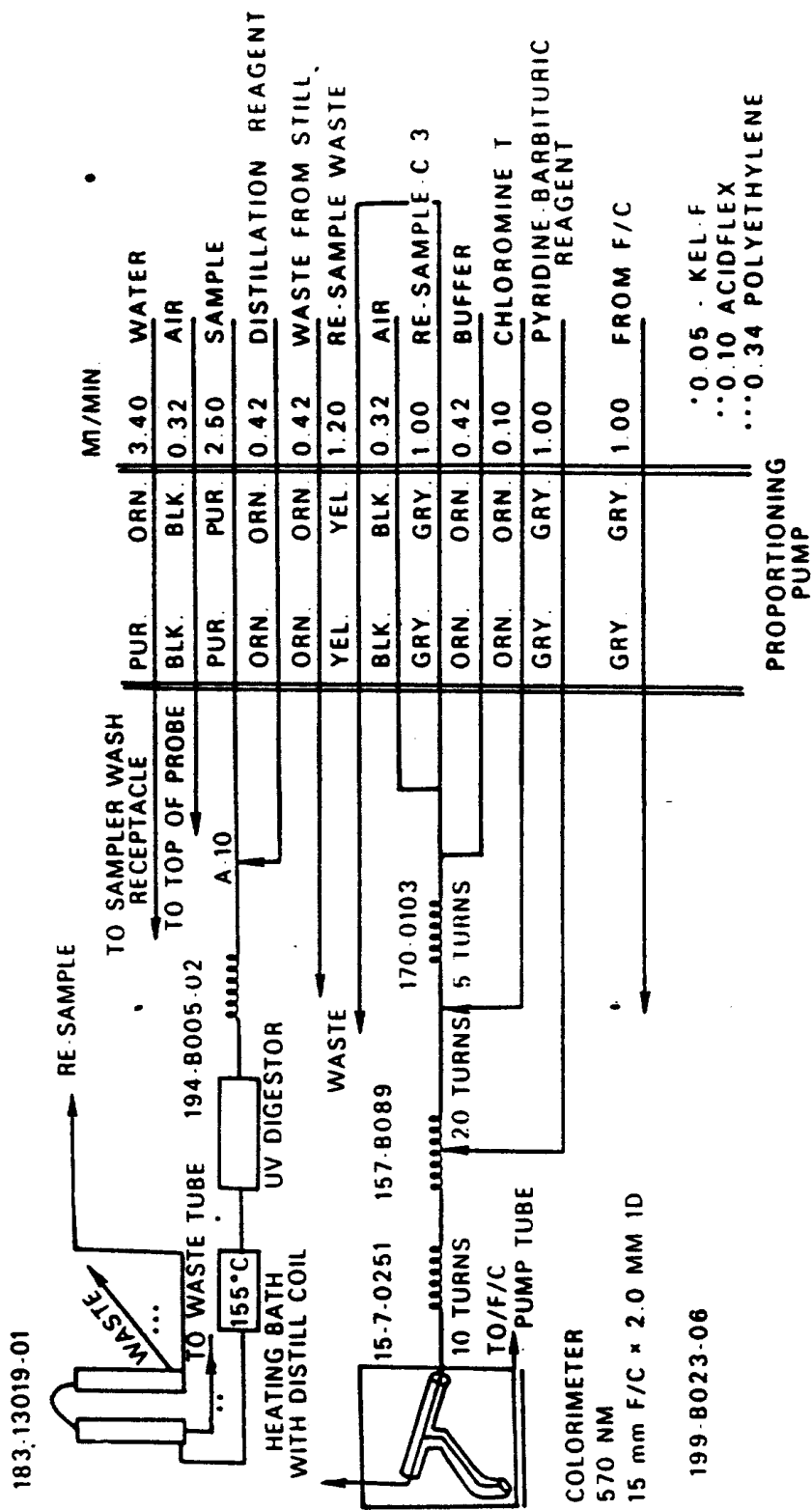


FIGURE 1. CYANIDE MANIFOLD AA11