Summary:

Prior to analysis, off-line manual distillation releases cyanide from cyanide complexes. Cyanide collects in a sodium hydroxide receiver solution. Reaction with chloramine-T trihydrate at a pH less than 8 converts sodium cyanide to cyanogen chloride. Cyanogen chloride then reacts with pyridine-barbituric acid to form a red-colored complex. The absorbance is measured at 570 nm.

Interferences:

Some known interferences include aldehydes, nitrate-nitrite, and oxidizing agents such as chlorine, thiocyanate, thiosulfate, and sulfide. Multiple interferences may require analyzing a series of laboratory-fortified sample matrices (LFM) to verify suitability of the chosen treatment. Distillation eliminates or reduces some interferences.

Treat samples containing sulfides, which adversely influences results by producing hydrogen sulfide during distillation.

High results can occur if samples contain nitrate or nitrite. During distillation, nitrate and nitrite form nitrous acid that reacts with some organic compounds to form oximes. These oximes decompose under test conditions to generate hydrogen cyanide. Pretreatment with sulfamic acid eliminates nitrate and nitrite interferences.

Treat samples containing oxidizing agents, such as chlorine, which decompose most cyanides.

Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.

Method interferences can be caused by contaminants in reagent water, reagents, glassware, and other sample processing apparatus that can bias analyte response.

Performance Specifications:

Range		5.0 µg/L–500 µg/L
Throughput		30 samples/hour
Precision at	5.0 µg/L	<2% RSD
	50 µg/L	<1% RSD
	500 µg/L	<1% RSD
Method Detection Limit (MDL)		0.5 µg/L
Continuing Calibration Verification Recovery		90–110%

Chemicals:

Acetone, C_3H_6O

Ascorbic acid, $C_6H_6O_6$

Brij®-35, (21% Solution) (PN A21-0110-33)

Chloramine-T trihydrate, $C_7H_7ClNO_2SNa{\bullet}3H_2O$

5-[4-(Dimethylamino)benzylidene]rhodanine, $C_{12}H_{12}N_2OS_2$

Ethylenediamine, anhydrous, $C_2H_8N_2$

Hydrochloric acid, concentrated, HCl

Potassium cyanide, KCN

Potassium hydroxide, KOH

Pyridine, C_5H_5N

Silver nitrate, $AgNO_3$

Sodium hydroxide, NaOH

Sodium phosphate monobasic monohydrate, $NaH_2PO_4{\bullet}H_2O$

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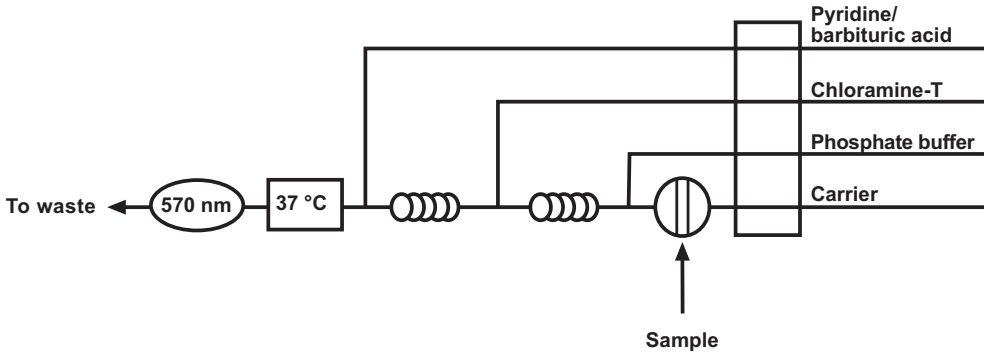
Basic Flow Diagram:



Note: This method complies with USEPA Methods 335.2 and 335.4, or an equivalent method.

This method also applies to determining total cyanide in samples distilled by other methods, such as *Standard Methods* or ASTM D2036, as along as calibration standards are prepared with the same sodium hydroxide concentration as used in the samples.

Selected References:

Standard Test Methods for Cyanides in Water. *Annual Book of ASTM Standards Volume 11.02,* ASTM International; ASTM D2036-98.

Cyanide, Total (Titrimetric; Spectrophotometric). *Methods for Chemical Analysis of Water and Wastewater;* U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, **1984**; EPA-600/4-79-020; Method 335.2.

Determination of Total Cyanide by Semi-Automated Colorimetry. *Methods for Chemical Analysis of Water and Wastewater;* U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory: Cincinnati, OH, **1984**; EPA-600/4-79-020; Method 335.4.

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