

Scope and Application	This test method is adapted from USEPA Method 335.2. It determines total cyanide in drinking water, wastewater, surface water, groundwater, and distillates.
Method Summary	Prior to analysis, off-line manual distillation releases cyanide from cyanide complexes. Cyanide collects in a sodium hydroxide receiver solution. Sodium cyanide converts to cyanogen chloride by reacting with chloramine-T trihydrate at a pH less than 8. The cyanogen chloride then reacts with pyridine-barbituric acid to form a red-colored complex. The absorbance is measured at 570 nm.
Sampling and Holding Time	<p>Collect samples in plastic or glass, and perform sample analyses as soon as possible to eliminate analyte loss.</p> <p>Preserve samples with sodium hydroxide at pH ≥ 12 and store at 4 °C at the time of collection. Perform sample analyses as soon as possible to eliminate analyte loss. Samples can be held for up to 14 days from the time of collection.</p> <p>Treat samples containing hydrogen sulfide using powdered cadmium carbonate.</p> <p>Treat sample containing chlorine using ascorbic acid.</p> <p>NOTE: Sodium arsenite is also used to remove oxidizing agents (0.1 g sodium arsenite per 100 mL of sample).</p>
Contamination and Interferences	<p>Some of the known interferences include sulfur compounds, aldehydes, nitrate-nitrite, and oxidizing agents such as chlorine, thiocyanate, and thiosulfate. Multiple interferences may require analysis of a series of laboratory fortified sample matrices to verify the suitability of the chosen treatment. Distillation reduces some interferences.</p> <p>Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. Treat samples according to the procedure in the <i>DA 3500 Discrete Analyzer Methods Manual</i>.</p> <p>High results may be obtained for samples that 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 HCN. Pretreatment with sulfamic acid minimizes nitrate and nitrite interferences.</p> <p>Oxidizing agents such as chlorine decompose most cyanides during the distillation step. Treat samples according to the procedure in the <i>DA 3500 Discrete Analyzer Methods Manual</i>.</p> <p>Other compatible procedures for removing or suppressing interferences may be used, provided they do not adversely affect overall method performance.</p> <p>Method interferences can be caused by contaminants in DI water, reagents, glassware, and other sample processing apparatus that can bias analyte response.</p> <p>Distillation-based methods that determine cyanide are rarely free of interferences. If interferences are known to be present, use OIA Method 1677 or OI Analytical's Total Cyanide by Amperometric Detection method.</p>

Method Performance

Parameter	Specification
Range	0.05–0.5 mg CN/L
Precision	10.1% RSD at 0.05 mg CN/L 4.9% RSD at 0.10 mg CN/L 2.44% RSD at 0.25 mg CN/L 3.00% RSD at 0.50 mg CN/L
MDL	0.01 mg CN/L
Accuracy	101%
Carryover	0.0492 mg/ CNL

Selected References

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

DA 3500 Discrete Analyzer Methods Manual (PN 323171). Available from OI Analytical, P.O. Box 9010, College Station, TX, 77842-9010.