

Cyanide Methods for Lachat Automated Ion Analyzers

Flow Injection Analysis



10 June 2019

QuikChem[®] Methods List

Use this list to:

- Identify and select analytical methods for your analyte, range, and matrix requirements.
- Locate all current Lachat methods for ion chromatography and flow injection analysis.
- Find methods accepted for USEPA compliance monitoring. These methods have symbols after the method number depending on whether the method is Accepted or Equivalent for NPDES and/or NPDWR reporting. Additional regulatory information can be found in the Regulatory Quick Reference section.
- Find methods with ERA or other external QC included in the support data. These methods have a * after the method number.

Performance Data Specifications

●**Range:** The range quoted in the Lachat methods list is based on the **actual, calibrated range**. The calibrated range is the lowest calibration standard to the highest calibration standard. (A blank is typically included in the calibration but is not included in the method range)

●**MDL:** The MDL (method detection limit) is calculated by the following protocols:
The Student's T number for the number of replicates is multiplied by the standard deviation calculated from those replications.

If **7 replicates** are used: The Student's T value is 3.14.

If **21 replicates** are used: The Student's T value is 2.528.

Example for 21 replicates: $2.528 \times 0.123 = 0.39$ for an MDL

●**Quantitation Limit:** Quantitation limit is typically 3 to 5 times the calculated MDL or 10X the standard deviation of the MDL standard used. Typically, this is the lowest calibration standard in a given method.

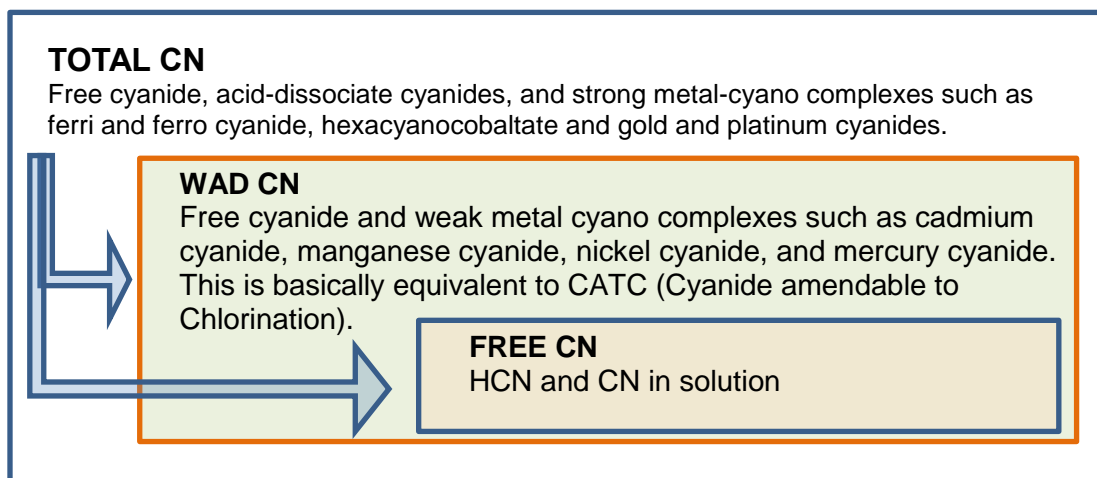
●**Precision:** Stated in the methods as %RSD. %RSD is calculated as follows: $\%RSD = (SD / \text{Mean}) \times 100$

Part Numbers Versus Method Numbers

To convert Method Numbers to part numbers, place an **E** in front of the Method Number.

Introduction

Cyanide is toxic to living things, and even more deadly to aquatic life than it is to humans. Cyanide can enter the environment due to human activities such as gold mining and gold plating. It is also produced by many species of plants such as Cassava (the source of Tapioca among other products).

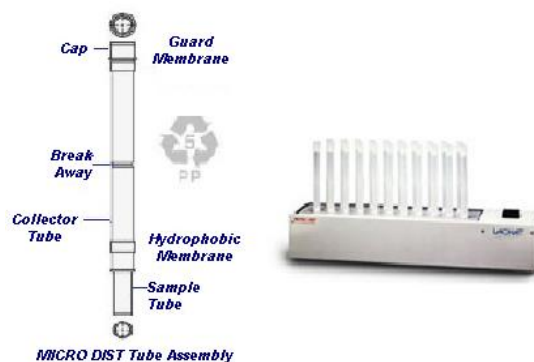


Categories of Cyanide

In assessing the toxicity of water samples, WAD cyanide is often measured, since this category of cyanide includes the toxicologically and environmentally important forms, including free cyanide and moderately and weakly complexed metal-cyanides. For a given sample, the total cyanide level is always greater than or equal to the WAD cyanide level, and likewise, the WAD cyanide level is always greater than or equal to the free cyanide concentration.

Distillation The purpose of distillation is sample clean-up. (In the case of cyanide, there can also be digestion). The distillation process transfers the analyte from the sample into a clean, reproducible solution, to enable more accurate measurement and reduce or eliminate interferences. In some cases, it can also provide some sample concentration. Sample clean-up by distillation has historically been performed in large volume, macro or midi- distillation glassware.

Lachat has two additional options for distillation. The first is a scaled down, off-line distillation (MicroDist). The MicroDist block has 21 positions for distillations of samples and standards, and the distillation takes only 30 minutes! The Microdist Tubes are disposable or recyclable so an added benefit is no glassware to wash.



Lachat's MicroDist

Lachat Instruments also has methods available in which the digestion and distillation are automated as part of the analysis. These methods utilize pH, heat, and for Total Cyanide, UV light. The methods allow the digestion, distillation and analysis of each sample or standard in 4 minutes or less.

Off-line distillation is required for sludge, soil, tissue, and in cases where samples contain particulate matter that must be included in the result.

Automated, in-line digestion methods can be used for water and other liquid samples (Such as filtered soil extracts) that do not contain particulates or in which samples can be filtered prior to the analysis.

Cyanide Analysis Utilizing Colorimetric Cyanide Methods

Colorimetry has been used in the analysis of cyanide for decades. In the US, the chemistries based upon pyridine and barbituric acid are used. In other parts of the world, chemistries based upon isonicotinic acid and 1,3-dimethyl barbituric acid are favored. Lachat Instruments has available methods utilizing these chemistries, covering Free, Total, and WAD Cyanide as well as Thiocyanate. Total cyanide methods can include off-line (macro- or Microdist™ distillations) or in-line digestion/distillation.

For the analysis of total cyanide following an off-line distillation, there are also Ultra High Throughput (120 samples and standards per hour) and Ultra Low Flow methods (reagent volumes similar to Discrete Instruments and 60 samples and standards per hour) available. Both suites of methods were designed to decrease reagent use, and they are compliant with the March 2007 MUR; in addition, the high throughput method is EPA accepted under NPDWR. Ultra High throughput methods allow the analysis of over 100 samples per hour and also offer reduced reagent consumption on a per-sample basis.

Regulatory Quick Reference

These QuikChem® cyanide methods are considered permitted reporting options for the National Pollutant Discharge Elimination (NPDES) and/or the National Primary Drinking Water Regulations (NPDWR) programs of the US Environmental Protection Agency (USEPA). Also listed are those QuikChem® methods that follow ISO standards.

The most recent MUR (Method Update Rule) was signed by the Administrator on April 17, 2012 and published at the CFR on May 18, 2012. There are some changes included that have the potential to positively affect Lachat Customers.

Although the information in the MUR is published in the CFR, states still have primacy. As a result, it is absolutely vital that labs discuss their plans to use any method (including a modified method) with their auditor prior to implementation, to be sure the proposed change or modified method will be accepted. By doing so, the lab will also know in advance what validation will be required in their specific case for implementation

designation in the methods list means the method is EPA accepted for NPDWR, NPDES, or both

^ designation in the methods list means the method is equivalent for NPDES reporting under the MUR.

R designation means the manifold is compliant with RoHS-2. Please check with your salesperson for availability.

Method Number	USEPA NPDES	USEPA NPDWR	USEPA Method
Cyanide			
	NPDES		
10-204-00-1-A#	Accepted	Accepted	
10-204-00-1-B	Equivalent		335.4
10-204-00-1-F	Equivalent		335.4
10-204-00-1-X	Approved	Approved	Promulgated method
10-204-00-1-X2^#	Equivalent	Accepted	

¹ EPA has revised the language at (b)(4)(T) to be **more specific with respect to the use of gas diffusion across a hydrophobic semi-permeable membrane**, to separate the analyte of interest from the sample matrix in place of manual or automated distillation for the analysis of certain analytes.

This is an acceptable change to an approved method for the following analytes: ammonia, **cyanide**, TKN, and total phenolics.

Although information regarding approved and accepted methods is published in the CFR, states still have primacy. As a result, it is important that labs discuss their plans to use **any** method (including promulgated, accepted, equivalent/modified methods) with their auditor **prior** to the method's implementation, to be sure the proposed change or modified method will be accepted. By doing so, the lab will also know in advance what validation will be required in their specific case for implementation.

Heaters:

Standard heater: Has a 175 cm section of 0.032" i.d. (0.8mm) and a 650 cm section of 0.032" i.d. tubing

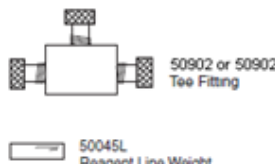

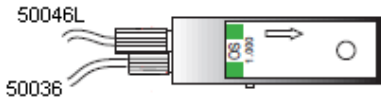
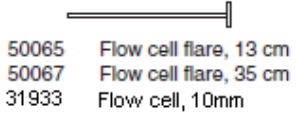

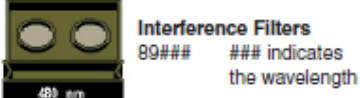
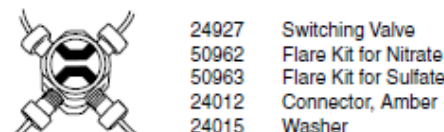
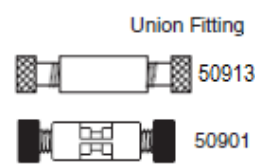
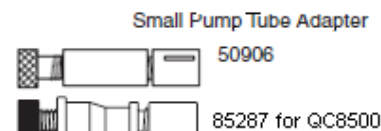
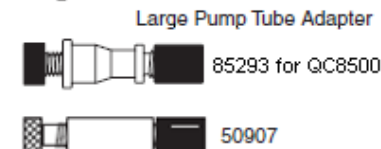
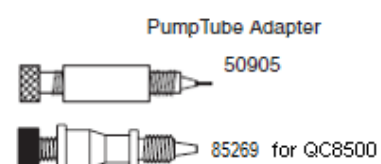
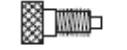


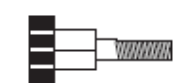

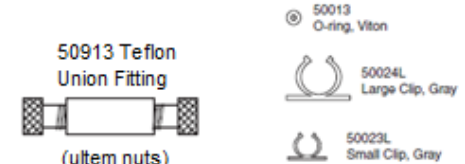
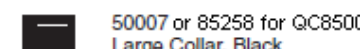
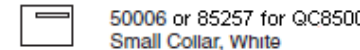



Non-standard heater: Has a different type and/or length of tubing than that listed above. (Controller and heater block are the same; only the tubing is different)

Cyanide

10-204-00-1-A#	0.005 – 0.5	0.0005	mg CN ⁻ /L	Waters	Total Cyanide Macro distillation method; 0.25 M NaOH matrix following distillation; Pyridine/barbituric acid, 570 nm. NPDES / NPDWR Accepted; follows Standard Methods (4500-CN). Requires a standard heater.	29-Nov-07
10-204-00-1-D ^R	0.20 – 10.0	0.003	mg CN ⁻ /L	Waters	Total Cyanide Acetate buffer; 0.25 M NaOH matrix following distillation. Pyridine/ barbituric acid, 570 nm. Requires a standard heater.	18-Sep-03
10-204-00-1-G ^R	2.0 – 500	0.5	µg CN ⁻ /L	Waters	Macro distillation method; 0.25 M NaOH matrix following distillation; pyridine-free reagents (isonicotinic/barbituric acid). 600 nm. Standard heater required.	16-Sep-03
10-204-00-1-H	0.002 – 0.01 0.1 – 5.0	0.00047 0.0138	mg CN ⁻ /L	Waters	Free Cyanide ; isonicotinic/barbituric acid. 600 nm. pyridine-free reagents; can be used w/ 10-204-00-2-G for inline total CN; multi-range method; Requires a standard heater.	7-Jun-06
10-204-00-1-J	25-1000	1.4	µg CN ⁻ /L		Total or WAD CN; Off-line Distillation. Pyridine/pyrazolone method. JIS K0102	31-Oct -14
10-204-00-1-X#	0.005 – 0.50	0.001	mg CN ⁻ /L	Waters	Total Cyanide ; MicroDIST [®] method; 0.25 M NaOH matrix following distillation; Pyridine/barbituric acid, 570 nm. NPDES/NPDWR approved method Requires a standard heater.	29-Nov-07
10-204-00-1-X2# [^]	0.002 – 0.5	0.00038	mg CN ⁻ /L	Waters	Total Cyanide ; MicroDIST [®] method; 0.25 M NaOH matrix following distillation; Pyridine/barbituric acid, 570 nm. <u>Ultra-High Throughput method</u> (>125 samples/hr); NPDES Equivalent / NPDWR Accepted Requires a standard heater.	16-Apr-08
10-204-00-2-C	2 – 100	0.21	µg CN ⁻ /L	Waters	Total Cyanide ; inline method; low-flow method; Pyridine/barbituric acid, 570 nm. Samples w/ particulates not suitable Inline module and Standard heater required.	14-Sep-07
10-204-00-2-D	5 – 500	0.51	µg CN ⁻ /L	Waters	Total Cyanide ; inline method; low-flow method; Pyridine/barbituric acid, 570 nm. Samples w/	19-Sep-07

Method No	Range	MDL	Units	Matrix	Comments	Rev Date
10-204-00-2-E ^R	2 – 100	0.5	µg CN ⁻ /L	Waters	particulates not suitable Inline module and Standard heater required. Total Cyanide ; inline method; low-flow method; lower recovery of ferricyanide; Pyridine/barbituric acid, 570 nm. Samples w/ particulates not suitable Inline module, Standard heater required	3-Dec-08
10-204-00-2-G ^R	0.002 – 0.01 0.1 – 5.0	0.00016 0.015	mg CN ⁻ /L	Waters	Total Cyanide ; inline method; pyridine-free reagents; can be used w/ 10-204-00-2-H for free cyanide; 600 nm multi-range method; Samples w/ particulates not suitable. In-line module and standard heater required.	22-Jun-07
10-204-00-4-B ^R	2.0 – 100	0.16	µg CN ⁻ /L	Waters	WAD Cyanide ; inline method; pyridine-free reagents; Isonicotinic/barbituric acid 600 nm. samples w/ particulates not suitable; MANIFOLD ONLY	27-Jul-07
10-204-00-4B52 ^R					DEDICATED 220V CHANNEL FOR QC8500 Isonicotinic/barbituric acid 600 nm. samples w/ particulates not suitable Requires 2 channels, two heaters, one detector and one valve)	
10-204-00-4-C ^R	2.0 – 100	0.17	µg CN ⁻ /L	Waters	WAD Cyanide ; Low Flow Method. In line distillation method; pyridine-free reagents; Isonicotinic/barbituric acid 600 nm. Samples w/ particulates not suitable; Requires an inline module and a standard heater.	11-Mar-13

Selected Parts. Please note this is NOT a complete listing



28081
Blades for PEEK
Tubing Cutter



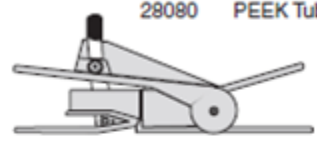
50021
Transmission Tubing, PVC, 0.030" ID



50029
Transmission Tubing, PVC, 0.060" ID



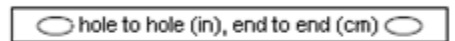
50091 Microloop Tubing, Teflon, 16 cm
50092 Microloop Tubing, Teflon, 12.5 cm



28080 PEEK Tubing Cutter



Teflon Tubing
50927 Manifold, 0.022" ID, Green
50928 Manifold, 0.032" ID
41300L Tefzel Tubing, 0.040" ID x 0.060" OD
30928 Zeus Tubing, 0.032" ID



Coils

Size	Coil Support Only	Wrapped, Teflon 0.022" ID	Wrapped, Teflon 0.032" ID
1" or 4.5 cm	50016L	50981	50916
2" or 7cm	50018L	50982	50918
2.5" or 8 cm	50017	50983	50917
4" or 12cm	50020	50984	50920
8" or 22 cm	50022L	N/A	50922



Alternating Coils 12 cm

Coil Support Only	Wrapped, Teflon 0.022" ID	Wrapped, Teflon 0.032" ID
50039	50985	50921



Pump Tubing

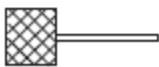
534XX	PVC
544XX	Duraprene
494XX	Silicone
654XX	Acidflex

XX is the number that specifies the pump tube color:

05	Orange-Yellow, 0.020" ID	13	Blue-Blue, 0.065" ID
06	Orange-White, 0.025" ID	14	Green-Green, 0.073" ID
07	Black-Black, 0.030" ID	15	Purple-Purple, 0.081" ID
08	Orange-Orange, 0.035" ID	16	Purple-Black, 0.090" ID
09	White-White, 0.040" ID	17	Purple-Orange, 0.100" ID
10	Red-Red, 0.045" ID	18	Purple-White, 0.110" ID
11	Gray-Gray, 0.051" ID	19	Yellow-Blue, 0.060" ID
12	Yellow-Yellow, 0.056" ID		



Bottles
28193L Glass Bottle, 1000 mL
35102 Glass Bottle, 2000 mL
43915 Glass Bottle, 100 mL (includes cap)



50012
O-ring Remover



28101L
End Plug



50062
Drill Bit

Lachat Instruments Brand Loveland, Colorado USA

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