

Standard Operating Procedure

Paragon Laboratories, Inc.
12649 Richfield Court
Livonia, MI 48150

SOP:

N0051

Revision:

0

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Effective Date:

12/19/06 00:00:00

Sampling Water and Wastewater for Available Cyanide (AvCN) Analysis by EPA Method OIA-1677

1.0 SCOPE AND APPLICATION

1.1 REFERENCE INFORMATION

Discipline:	ENV, TCN, ACN, AvCN, Cyanide
Related Documents:	SOP-A0140, SOP-A0191
Applicable Programs:	Wastewater, Surface Water, Groundwater
Regulatory References:	40CFR136

- 1.2 This procedure provides instruction for collecting surface water or wastewater effluent grab samples to be analyzed for available cyanide (AvCN). It is intended for use by Paragon staff and other individuals responsible for available cyanide sample collection that may require the removal of sulfide interference.

[NOTE: Sulfide can be a negative or positive interferent. If both sulfide ion and cyanide ion are present in the sample, they react to form thiocyanate, which is not detected in the OIA-1677 analytical system for AvCN. (In addition, thiocyanate can form from precipitated lead sulfide, formed during treatment to remove sulfide.) When sulfide reacts in this manner it will cause AvCN results to be biased low. If unreacted sulfide remains in the analyzed sample, however, the OIA-1677 system detects it as AvCN, and results will be biased high.]

2.0 SUMMARY OF METHOD

- 2.1 When sulfide interference is suspected in the source water, two bottles must be submitted for each sample. One bottle will contain the "**raw**" sample preserved with sodium hydroxide. The second bottle will contain the sample after being "**treated**" with lead carbonate (to remove sulfide) and then preserved with sodium hydroxide. The "**treated**" sample is analyzed for AvCN, and the "**raw**" preserved sample is held by the laboratory for possible confirmation analyses.
- 2.2 All sample treatments and preservations **must occur within 15 minutes** of sample collection.

3.0 SAFETY

- 3.1 Bottles labeled as containing "NaOH" contain 1.25 mL of 10N sodium hydroxide (NaOH) per 250 mL. This solution is very caustic. Handle with care and avoid skin contact.
- 3.2 Bottles labeled as containing "Lead Carbonate" contain 0.25g of solid lead(II) carbonate (PbCO₃). Avoid inhalation and skin contact.

4.0 EQUIPMENT AND SUPPLIES

- 4.1 A vacuum pump is required for filtering each sample using the pre-assembled filter unit. Either an electric- or hand-powered vacuum pump must be available.

[NOTE: When requested, Paragon can include a hand vacuum pump with sampling supplies. Contact Paragon for specific arrangements or to check availability prior to sampling.]

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- 4.2 Each sampling kit includes the following items:
- (a) 1 120mL plastic bottle labeled "Raw Sample; NaOH"
 - (b) 1 250mL amber glass bottle labeled "Treated Sample; NaOH"
 - (c) 1 250mL plastic bottle labeled "Lead Carbonate"
 - (d) 1 plastic powder funnel
 - (e) 1 sheet of filter paper
 - (f) 1 250 mL pre-assembled vacuum filter unit containing a 0.45µm SFCA membrane filter (Nalgene #157-0045, or equivalent)

5.0 PROCEDURE

Set out and familiarize yourself with the contents of the sampling kit described in Section 4.2. Open the sealed pouch containing the pre-assembled filter unit. (Disregard the manufacturer's instructions printed on the pouch.) Remove the lid of the filter unit and attach the vacuum pump to the nipple on the side of the unit.

Collect the grab sample only when ready to proceed smoothly through all steps of Section 5.

5.1 **UNTREATED SAMPLE COLLECTION**

Fill the 120mL plastic bottle labeled "Raw Sample; NaOH" up to the bottom of its neck with sample. Do not rinse the bottle or overfill to overflowing. Cap the bottle securely.

5.2 **TREATED SAMPLE COLLECTION (to remove sulfide interference)**

If the sample does not contain visible particulates, begin with Step 5.2.1. If the sample contains visible particulates, begin with Step 5.2.2.

5.2.1 Sample Does Not Contain Particulates

Fill the 250mL plastic bottle labeled "Lead Carbonate" up to the bottom of its neck with sample. Do not rinse the bottle or overfill to overflowing. Proceed immediately to Section 5.2.3.

5.2.2 Sample Contains Particulates

Uncap the 250mL plastic bottle labeled "Lead Carbonate" and set the powder funnel on top of the open bottle. Fold the sheet of filter paper in half; then fold in half again. Hold the folded filter at the point and open to form a funnel shape; with the point down, place inside the powder funnel.

A little at a time, pour sample through the folder filter and plastic powder funnel and into the 250mL plastic bottle labeled "Lead Carbonate". Only filter enough sample to fill the bottle up to the bottom of its neck.

Without touching the inside of the filter, fold the used filter paper and insert into the amber bottle labeled "Treated Sample; NaOH". Proceed immediately to Section 5.2.3.

5.2.3 Filter the Lead Carbonate Treated Sample

Cap the Lead Carbonate bottle and **gently swirl** to mix the sample and PbCO₃ (approximately 30 seconds). Pour the entire mixed sample into the top container of the filter unit, and using the vacuum pump, filter to remove remaining lead carbonate and any precipitated lead sulfide.

When the sample is completely filtered, transfer all of the filtrate in the bottom container of the filter unit into the amber bottle labeled "Treated Sample; NaOH". Cap the bottle securely.

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5.3 RETURNING SAMPLES TO PARAGON

- 5.3.1 Immediately refrigerate all samples or place in a cooler with sufficient ice to begin cooling to between >0°C to 6°C. Samples should be returned to the laboratory promptly to avoid approaching the 14-day maximum holding time for analysis.
- 5.3.2 Please return all plastic powder funnels and unopened preassembled filter units with samples.
- 5.3.3 Discard used filter units and used "Lead Carbonate" bottles.

6.0 REFERENCES

- 6.1 USEPA. 2006. *Code of Federal Regulations*, Title 40, Part 136. Footnote 6 of Table II to 40CFR136.3. Washington: GPO. [Available on the internet at <http://ecfr.gpoaccess.gov>.]
- 6.2 USEPA. Office of Water. 1999. Method OIA-1677, Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry. EPA-821-R-99-013. Washington: USEPA, August. [Also available through ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842-9010.]
- 6.3 USEPA, NERL-CI. 1993. Determination of Total Cyanide by Semi-automated Colorimetry, Method 335.4, Revision 1.0. *Methods for the Determination of Inorganic Substances in Environmental Samples*. EPA/600/R-93/100. Washington: USEPA, August.
- 6.4 USEPA. Office of Solid Waste & Emergency Response. 2004. *Test Methods for Evaluating Solid Waste, Physical / Chemical Methods (SW-846)*. 3rd ed, as amended by Final Updates I, II, IIA, IIB, III, IIIA, and IIIB. Washington: GPO, November. [Also available on the internet at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.]
--Total and Amenable Cyanide: Distillation, Method 9010C, Revision 3, November 2004.
--Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation), Method 9012B, Revision 2, November 2004.
- 6.5 American Public Health Association. 1999. Part 4500-CN, Cyanide. *Standard Methods for the Examination of Water and Wastewater*. On-line version. Washington: APHA.

Revision History

Rev	Description of Change	Originator	Source File	Source SOP	Effective Date
0	Initial Release	JMS-225	SOP-N0051-R0.doc	N0051	12/19/06
1					
2					

Approvals

Primary Authority:	John C. Parmentier, Vice President <hr/> <small>(Name and Title)</small>
Secondary Authority:	John M. Spurr, Environmental Lab Manager <hr/> <small>(Name and Title)</small>

Signed by: jspurr

Full Name: John Spurr

Employee ID: 225

Signed from: ENVIROMGR : PARAGON : jspurr

Signed on: 2006-12-19 11:07:33 GMT -05:00

Server Date/Time: 2006-12-19 11:08:33 GMT -05:00

Authorization Mode: Database Login

Reason Code: SOP Approved for Use

Placed Comment:

Signed by: jcparmentier

Full Name: John Parmentier

Employee ID: 210

Signed from: JCP : PARAGON : jcparmentier

Signed on: 2006-12-19 10:00:31 GMT -05:00

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