STANDARD OPERATING PROCEDURES (SOP)
"Ammonium Analysis by Salicylate Method using the Lachat Flow-injection System"
MED-SOP-CHA031-SAL TJ 4-2015
Revision 0

Prepared by: Terri M Jicha Date: 04/15/15
Revised by:
Reviewed by: Anne M Cotter
Branch Chief: _____ Date: 4/15/15

Approved by:
Quality Assurance Manager: _____ Date: 4/23/2015

U.S. Environmental Protection Agency
National Health and Environmental Effects Research Laboratory
Mid-continent Ecology Division

Contents

PROCEDURAL SECTION

1.0 SCOPE AND APPLICATION

1.1 LACHAT QuikChem AE Instrument Manual- (QuikChem Method 10-107-06-2-C) -Ammonia (Salicylate) in Waters, stored with the Lachat Instrument.

1.2 The appropriate working range is 0.02-2.00mg-N (as NH_4)/L

1.3 This method can be used for filtered water samples or sediment KCl extracts

1.4 This method replaces MED-SOP-CHA012, using phenolate.

2.0 SUMMARY OF METHOD

2.1 Ammonium heated with salicylate and hypochlorite in an alkaline phosphate buffer produces an emerald green color which is proportional to the ammonium concentration. Ammonium concentration is detected using a 660nm filter.

2.2 Definitions: DIW: deionized water

2.3 Health and Safety Warnings: Safety glasses, a lab coat and nitrile gloves must be worn at all times.

2.4 Interferences: Calcium and magnesium will interfere by forming precipitate. EDTA is added to the buffer to prevent this interference.

3.0 PERSONNEL QUALIFICATIONS

3.1 Proper training in the use, care and troubleshooting techniques is required by the instrument manager before using the Lachat.

3.2 Trained personnel are listed in the Lachat instrument logbook.

4.0 MATERIALS AND PROCEDURES

4.1 <u>Materials</u>

4.1.1 13x 100mm borosilicate test tubes

4.1.2 Lachat QuickChem 10-107-06-2-C Ammonia manifold with a 150cm for water or 15cm for KCl extracts sample loop and 100cm back pressure loop.

4.1.3 ASX-520 XYZ autosampler by CETAC Technologies.

4.2 Reagents and Standards

4.2.1 All reagents and standards are to be prepared with DIW of 10 megohm or better as indicted by reading on deionized water system meter.

4.2.2 Reagent 1: Buffer
To a 1L beaker, add 482.5g DIW + 15g NaOH + 33.5g $Na_2HPO_4 \cdot 7H_2O$ + 12.5g $Na_2EDTA \cdot 2H_2O$ (ethylenediaminetetraacetic acid disodium salt dihydrate). Stir to dissolve. Can be kept at room temperature. Degas with Helium.

4.2.3 Reagent 2: Salicylate-Nitroprusside Color Reagent
To a 1L beaker, add 908g DIW + 144g Sodium Salicylate $[C_6H_4(OH)(COO)Na]$ + 3.5g Sodium Nitroferricyanide dihydrate (NaNitroprusside) $[Na_2Fe(CN)5NO \cdot 2H_2O$. Stir to dissolve. Refrigerate. Prepare fresh **weekly**. Degas with Helium.

4.2.4 Reagent 3: Hypochlorite
To a 1L beaker, add 936g DIW + 64g (5.25%) NaClO *or* 56g (6.0%) NaClO
Make fresh **daily.**

4.2.5 Reagent 4: Carrier
1L DIW degassed with Helium. Make fresh **daily.**

4.2.6 Stock Standard: 1000mg/L Ammonium
Dissolve 1.910g Ammonium Chloride (NH_4Cl) dried for one hour at 105°C in DIW to 500mL final volume.

4.2.7 Working Calibration Standards: Prepare standard over the range of analysis. For the working range of 0-2.00 mg-N (as NH_4)/L the following standards should be use:

2.00 1.00 0.50 0.25 0.10 0.05 0.02 0.00 mg/L

4.2.8 Quality Control Standard: A second source standard should be prepared within the expected sample concentration range.

4.3 Procedure

4.3.1 Water samples are prepared by filtering collected water through a 0.45um membrane filter and store frozen until analysis within 6 months of collection. KCl extract samples are filtered through 2V pleated Whatman filters and stored frozen until analysis within 6 months of collection.

4.3.2 Prepare standards and reagent according to the above recipes.

4.3.3 Degas all reagents except the hypochlorite and standards by bubbling helium gas thru a disposable glass pipette into the reagent for 5 minutes minimum. Adjust the flow just slow enough to prevent blowing the reagent out of its container.

4.4 Lachat setup and analysis

4.4.1 Turn on instrument and software 15 minutes prior to starting analysis to allow for the heater block to warm up to 60°C.

4.4.2 Place reagent lines in appropriate reagents and start pump. Be sure to check that all reagents are flowing smoothly. Allow to run through system for minimum of 5 minutes.

4.4.3 Pour samples in tubes in racks and record on bench sheets. Note: that while the instrument is set up to handle 4 trays of 90. It is best to restrict batches to 2 racks of 90.

4.4.4 Open the NH4water_YYYY_method.omn or NH4Kcl_YYYY_Method.omn, enter samples and cup numbers in the sample table.

4.4.5 Press the **Start** button when ready to run.

4.4.6 When run is complete, place reagent lines in DIW water and rinse through for 10 minutes, then pump air for another 10 minutes to dry the lines.

4.5 Data calculations

4.5.1 Lachat software yields results directly as mg/L NH_4-N. Occasionally it is appropriate to split a curve for greater sensitivity. If this is required the data can be exported to an excel spreadsheet where the analyst can recalculate the standard curve equations by regression and recalculate the sample concentrations using the peak areas based on the new curve equation.

5.0 QUALITY CONTROL AND QUALITY ASSURANCE SECTION

5.1 Laboratory replicates and spiked samples should be prepared for at least 5% of samples to be analyzed.

5.2 Duplicate field samples and blanks should be collected for at least 5% of the total number collected.

5.3 Check standards and/or QC samples should be analyzed at least 5% of the total number of samples analyzed.

5.4 Method blanks and lab fortified blanks (LFB) should be ran a minimum of 2 per batch.

5.5 Standard curve is automatically calculated by the software when the set of working Calibration Standards are ran as sample type "Calibration" in the sample table. A standard curve should be ran daily or at a minimum with each batch for fresh reagents. A set of Working Calibration Standards should be run at the end

of each run as "unknown" samples to verify calibration. Should instrument drift occur this set of standards could be recalculate the standard curve equations by regression and recalculate the sample concentrations using the peak areas based on the new curve equation.

6.0 REFERENCES

6.1 Lachat Instruments Quik Chem Method No. 10-107-06-2-C "Ammonia in surface water, wastewater." August 1992

6.2 Lachat QuikChem 8000 Operating Manual