DETERMINATION OF TOTAL KJELDAHL NITROGEN IN WATER BY GAS DIFFUSION SEGMENTED FLOW ANALYSIS

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1. SCOPE AND APPLICATION

1.1. This SOP is applicable to the determination of Total Kjeldahl Nitrogen (TKN) in surface, ground, effluents, influents and saline water. The applicable range for this method is from the method detection limit (MDL), 0.030 mg N/L, to 4.0 mg N/L. The range may be extended by dilution. The practical quantitation limit (PQL) is 0.10 mg N/L.

1.2. Tests covered by this SOP are W-TKN and W-TKN-F.

2. SUMMARY OF THE METHOD

2.1. This SOP is based upon EPA Method 351.2, Rev. 2.0 (1993) and OI Analytical Method 327152 utilizing gas diffusion. For method modifications, see Table 4 in the OI Analytical method. The volume of sample digested for this method is 10 mL. For the calibration range, see section 5.3.3 below

2.2. Prior to analysis, digestion converts free ammonia and organic nitrogen compounds to ammonium sulfate (DEP SOP NU-097).

2.3. The sample pH is raised to a pH of >11. The ammonia molecules generated at this pH pass through a gas diffusion membrane and are absorbed into an alkaline hypochlorite solution to form chloramine. The chloramine reacts with salicylate to form indophenol blue in an amount that is proportional to the ammonia concentration. Sodium nitroferricyanide intensifies the blue color. The absorbance is measured at 660 nm.

2.4. Interferences: Heavy metals in the sample may interfere by precipitating. Complexation with citrate should minimize this interference. High nitrate concentrations (>10 mg N/L) interfere with the TKN preparation procedure. The interference can be either negative or positive. For any samples with a known nitrate concentration of 10 mg N/L or higher the TKN result should be qualified with a J qualifier or the sample diluted sufficiently to decrease the nitrate concentration to below 10 mg N/L PRIOR to digestion.

3. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

3.1. Samples are preserved at collection with sulfuric acid to a final concentration of 0.2 % (v/v), shipped on ice and stored at ≤ 6 ºC (not frozen).

3.2. Samples must be analyzed within 28 days of collection date in order for data to be reported unqualified. Samples analyzed greater than 28 days after collection date are reported with the Q qualifier.

3.3. Refer to DEP SOP NU-097 for preparation procedure.

4. EQUIPMENT AND SUPPLIES

4.1. Instrumentation – OI Analytical Flow Solution® 3100 Automated Chemistry Analyzer for Segmented Flow Analysis.

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4.1.1. Precision Pump with 8 channels.

4.1.2. Autosampler 3360.

4.1.3. Expanded Range™ (ER) Photometric Detector with 5 mm path length flowcell and 660 nm optical filter.

4.1.4. Heater Module.

4.1.5. Data acquisition system (PC or notebook PC) with WinFLOW™ software, part number 327060 Flow Solution® 3100.

4.1.6. In-line Gas Diffusion Cartridge (PN 327152) and membranes (A001520).

4.1.7. N_2 pillow.

4.1.8. 5510 Branson Sonicator (see Section 5.2.1.).

4.2. Glassware and pipettes (volumetric flasks and measuring cylinders are Class A):

4.2.1. Variable Eppendorf type pipettes (100 µL-1000 µL, 10 µL-100 µL, 1 mL-10 mL)

4.2.2. 100 mL, 200 mL, 500 mL and 1 L volumetric flasks for working standards and reagent preparation.

4.2.3. 1- 4 L container for reagent water.

4.2.4. 1- 1 L amber Nalgene bottle for salicylate reagent.

4.2.5. 3- 1 L Nalgene bottles for complexing agent, startup solution, and 10 N NaOH.

4.2.6. Millipore glass fiber prefilters, cat # APFF040700.

4.2.7. 1- 500 mL bottle for 1M sodium hydroxide solution.

4.2.8. 1- 100 mL and 1- 250 mL graduated cylinder.

4.2.9. Beakers.

4.2.10. Spatulas for weighing out reagents.

4.2.11. 1 L Filtration apparatus.

4.2.12. Disposable culture tubes, borosilicate glass 3x100 mm cat #14-961-27.

4.2.13. Disposable syringes with 0.45 µm filter tips (Whatman Puradisc PP cat. # 05-713-404A).

5. REAGENTS AND STANDARDS (SEE DEP SOPS NU-041 and NU-042)

Note: Prior to using the chemicals in this analysis, read their MSDS and the safety notes found in section 9 of this SOP.

Note: The volumes prepared for the reagents and standards may be altered by the analyst as necessary. The final concentrations of the constituents of a standard or reagent must not be changed.

Note: All reagent and standard preparations must be entered in the Laboratory Information

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Management System (LIMS) 'Standard Preparation Tracker' module. Unique ID numbers for each standard are documented in the prep worksheet for digestion (see DEP SOP NU-097). The Certificate of Analysis for the standard reference material is kept in a binder in C307.

Note: Unique ID numbers for each analysis reagent are generated in the LIMS module and documented in the Reagent and Standard Logsheet located on the network at: \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\REAGENT_PREP\TKN-OI\. This sheet needs to be updated, printed, and submitted as part of the analysis folder. If available, a Certificate of Analysis for each purchased reagent is also maintained in the \REAGENT_PREP\TKN-OI\ folder. All containers holding standards or reagents must be labeled with their unique ID and expiration date.

5.1. Chemicals:

5.1.1. Ammonium Chloride, NH_4Cl

5.1.2. Brij-35, 30 % solution (Ricca cat. #1145)

5.1.3. Sodium citrate dihydrate, $C_6H_8O_7Na_3 \cdot 2H_2O$ (FW 294.1)

5.1.4. Sodium salicylate, $NaC_7H_5O_3$ (FW 160.1)

5.1.5. Sodium Hydroxide, NaOH

5.1.6. Sodium hypochlorite, 8.25 % available chlorine (household bleach, concentration of available chlorine in commercial product can change, see 5.2.7.), NaOCl (FW 74.44)

5.1.7. Sodium Nitroferricyanide (sodium nitroprusside), $Na_2Fe(CN)_5NO*2H_2O$

5.1.8. Sulfuric Acid, concentrated, H_2SO_4

5.1.9. Standard Reference Material (ALTQC) from Environmental Research Associates (ERA).

5.1.10. Glycine, $C_2H_5NO_2$

5.1.11. β-Glycerophosphoric Acid, Disodium Salt, Pentahydrate, $C_3H_7Na_2O_6P*5H_2O$

5.2. Reagents:

5.2.1. **Reagent Water:** DI water or degassed DI water. Degassing is only necessary when air spikes create problems in the detector baseline. Degas DI water using vacuum filtration. The filtration apparatus is set up in a sonicator bath and the filtrate is sonicated for about 15 minutes. After preparing degassed reagent water, store it in a tightly-sealed container to protect it from reabsorbing atmospheric gases.

5.2.2. **Startup solution (1 L):** Add 1.1 mL of 30 % Brij-35 (5.1.2.) to approximately 900 mL of reagent water (5.2.1.) in a 1 L volumetric flask. Make the volume up to the mark with reagent water and mix gently (final concentration 0.03 % Brij-35). Holding time, 14 days.

5.2.3. **10 N Sodium Hydroxide Solution (1 L):** While stirring, carefully add

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400 g of sodium hydroxide (5.1.5.) to approximately 500 mL of reagent water in a 1 L volumetric flask. Allow the solution to cool to room temperature. Make the volume up to the mark with reagent water and mix well. Holding time, 42 days.

5.2.4. **1 M Sodium Hydroxide Solution (500 mL):** Add 50 mL of 10 N Sodium Hydroxide Solution (5.2.3.) to a 500 mL volumetric flask containing 300 mL of reagent water. Dilute with reagent water to the mark and mix well. Holding time, 42 days.

WARNING: Mixing sodium hydroxide with water produces a great of amount of heat. Take appropriate precautions.

5.2.5. **Stock Salicylate/Nitroferricyanide Solution (1 L):** While stirring, carefully add 250 mL of 10 N sodium hydroxide solution (5.2.3.) to approximately 700 mL of reagent water (5.2.1.) in a 1 L volumetric flask. Cool the solution to room temperature. Add 150 g of sodium salicylate (5.1.4.) and 0.3 g of sodium nitroferricyanide (5.1.7.) and stir until dissolved. Dilute to 1 L with reagent water and mix well.

IMPORTANT: Filter solution before use. **Store in an amber PE bottle and refrigerate.** Holding time, 42 days.

5.2.6. **Working Salicylate Reagent (200 mL):** Add 1.4 mL of 30 % Brij-35 solution (5.1.2.) to 200 mL of stock salicylate/nitroferricyanide solution (5.2.5.) and mix gently (final concentration 0.2 % Brij-35). Prepare fresh solution daily.

5.2.7. **Working Hypochlorite Solution (1 L):** Add 1 mL of 1 M NaOH (5.2.4) to 800 mL of reagent water (5.2.1.) and mix well. Add sodium hypochlorite (5.1.6.) to a final concentration of 0.01 % (for example, 1.5 mL of 8.25 % available chlorine) to the solution. Dilute to 1 L with reagent water and mix well. Prepare fresh solution daily.

5.2.8. **Complexing Reagent (1 L):** While stirring, add 112 g of sodium citrate (5.1.3.) to 200 mL of reagent water (5.2.1.) in a 1 L volumetric flask. Add 320 mL of 10 N sodium hydroxide solution (5.2.3.) to the mixture. Cool the solution to room temperature. Dilute to 1 L with reagent water and mix well**.** Holding time, 42 days. **IMPORTANT**: Filter solution before use.

5.3. Standards and spiking solution:

5.3.1. **TKN stock, 1000 mg N/L:** Dry at least 4 g of NH_4Cl (5.1.1.) for 12-24 hours at 105 ^oC. Dissolve 3.819 g of dried NH_4Cl in 800 mL of DI water contained in a 1 L volumetric flask. Dilute to volume using reagent water (5.2.1.) and invert 12 times. Store under refrigeration. This standard is also used for NH_3 analysis (see DEP SOP NU-095). Holding time, 180 Days.

5.3.2. **TKN intermediate, 10 mg N/L:** To a 200 mL volumetric flask containing about 50 mL of reagent water (5.2.1.), add 2 mL of the 1000

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mg N/L NH_3 stock (5.3.1.). Dilute with reagent water to volume. Prepare this standard each day that samples are digested.

5.3.3. **TKN working calibration standards (CAL STD), continuing calibration verification (CCV), practical quantitation limit (PQL) standard (PQCK2), and blanks (method blank (MBLK), continuing calibration blank (CCB), and additional blanks (CAR)):** Prepare standards by pipetting specified amounts of the TKN intermediate (10 mg N/L, 5.3.2.) into volumetric flasks according to the table below. Acidify with concentrated sulfuric acid (5.1.8.). Use reagent water (5.2.1.) to make up the final volume. Invert each standard 12 times. Prepare these standards each day that samples are digested. Digest the standards according to DEP SOP NU-097. Holding time of the standards, 7 days.

Standard Concentration (mg N/L)	QC standard	Final Volume (mL)	Volume of H_2SO_4 (mL)	Volume of Intermediate (mL)
4.0	CAL STD	100	0.2	40
2.0	CAL STD	100	0.2	20
1.0	CAL STD, CCV	100	0.2	10
0.20	CAL STD	100	0.2	2.0
0.10	CAL STD PQCK2	100	0.2	1.0
0.080	CAL STD	100	0.2	0.80
0.050	CAL STD	100	0.2	0.50
0	MBLK, CCB, CAR	2000	4.0	--

5.3.4. **ALTQC standard (ERAQCX):** The supplier for the standard reference material (currently Environmental Resource Associates) differs from the supplier of the calibration standards. This quality control standard (5.1.9) is prepared from the ampule according to the manufacturer's instructions. For the concentration and expiration date of the ampule, see the manufacturer's certificate. The holding time of the prepared stock standard is 180 days. This ALTQC stock solution may be diluted appropriately so that the working solution's concentration falls within the calibration range. The holding time of the working solution is 30 days. (The X in the designation ERAQC is a digit incremented each time an ampule with a new lot # is used.)

5.3.5. **TKN/TP Spiking Stock Solution (TKN, 150 mg N/L, TP, 50 mg P/L)**: Dry approx. 1.1 g glycine (5.1.10.) overnight at 60 ^{O}C. Add 0.804 g glycine and 0.4941 g β-glycerophosphoric acid (5.1.11.) to a 1 L volumetric flask containing approx. 400 mL DI water. Bring to volume with DI water. Invert 12 times. Store in refrigerator. Holding time of the prepared standard, 90 days. This solution is used to prepare laboratory fortified blanks (LFB), matrix spikes (MS) and matrix spike duplicates

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(MSD). This solution is also used for the analysis of TKN and TP in soils (DEP SOPS NU-081and NU-084, respectively).

Note: **Glycine:** C2H5NO2, formula weight: 75.07 g/mol. Molecular weight factor = 14.0067/75.07 = 0.1866.

β-**glycerophosphoric acid:** C3O11H17PNa2, formula weight: 306.11 g/mol. Molecular weight factor = 30.9738/306.11 = 0.1012.

6. SAMPLE ANALYSIS AND QUANTITATION (SEE DEP SOP NU-033)

6.1. Perform any maintenance e.g. replacing pump tubing, membrane, or lamp as needed. Record all maintenance procedures in the instrument maintenance book.

6.2. Preparation:

6.2.1. Select digested trays to be analyzed, and vortex samples and standards thoroughly.

6.2.2. Remove reagents from the refrigerator. Check the expiration dates on all standards and reagents. Discard expired standards and/or reagents.

6.3. Instrument Setup:

6.3.1. Turn on autosampler, pump and detector.

6.3.2. Double-click the Winflow icon on the computer desktop to start software.

6.3.3. Clamp down pump tubing (reagents, nitrogen, and debubbler).

6.3.4. Turn on nitrogen pillow, 1 full turn.

6.3.5. Connect **all** reagent pump tubes to the reagent bottle containing the startup solution (5.2.2.). Start the pump at 40 % allowing the solutions to flow through the entire system (at least 10 minutes). Connect all three waste lines (sample, reagent, and debubbler) to sink waste reservoir.

6.3.6. The temperature is set at 50 °C. Once the heater reaches 50 °C verify that the baseline is stable.

6.3.7. Make sure that the flowcell is purged of all bubbles and the flow is stable and free from surging.

6.3.8. Leave the salicylate line in the startup solution while connecting all other reagents to their respective bottles allowing them to flow for at least five minutes. Then connect the salicylate line to its reagent bottle and pump the salicylate/nitroferricyanide solution through the system (at least 10 minutes).

6.3.9. When the system has stabilized, run the pump at least 10–15 minutes and verify again that there are no bubbles in the flowcell. Obtain a stable baseline at 660 nm and autozero the baseline before beginning the analysis. Connect the sample waste line to a hazardous waste bottle.

6.3.10. Load the sampler tray with calibrants, blanks, samples, and QC samples according to the digestion sheet. Identify the batches and their QC

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samples. Make sure there is a method blank (MBLK), LFB (laboratory fortified blank), matrix spike, and a matrix spike duplicate or a sample duplicate digested for every batch of 10 samples.

6.3.11. Start and finish every run with a CCB (5.3.3.) and a CCV (5.3.3.). After these initial standards add your PQL (PQCK2 (5.3.3.)) and ALTQC (5.3.4.). Analyze a CCB (5.3.3.) and a CCV (5.3.3.) every 10 cups throughout the entire run.

6.4. Analysis setup in the Winflow program:

6.4.1. Click on the box called sample table located in the center of the screen. It will show the last table used.

6.4.2. On the menu bar: select 'File' and 'open'. Select the Nutrients drive from the drop down box, then the OI Analytical folder, then the TKN subfolder. Then, select: Template.TBL. This template includes the setup for the weighted linear calibration. All standards are analyzed in triplicate.

6.4.3. Start with cup 101 and enter sample ID's in the 'Name' column. Include the tray # as a suffix for the ALTQC standard (e.g. ERAQC13_4721). For the 'Type' column, enter U for samples and RB's for baseline corrections. Type RB's after every ten samples.

A typical run sequence includes:

Sync – high standard (5.3.3.)
Blank – to check for carryover (5.3.3.)
Calibrants – run in triplicate (5.3.3.)
CCV/CCB – these checks are run every ten cups (5.3.3.) and at the end of the run
PQCK2 (5.3.3.)
ERAQCX_(dilution factor) – (5.3.4.) the SRM is diluted within the calibration range
Batched samples, including an MBLK_F, an LFB, and a set of duplicate spikes per batch

6.4.4. Save the template as YYMMDDT# for SFA Instrument 1 or YYMMDDK# for SFA Instrument 2 (e.g. 140929T1). The file should be saved on the Nutrients drive at \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Nutrients\OI-Analytical\TKN\ in the current year's sub-folder.

6.4.5. Go to 'File' and select 'print' to obtain the benchsheet. Close the sample table box.

6.5 Start the analytical run:

6.5.1. Click on the box called 'Collect Data' located in the center of the screen.

6.5.2. Enter your initials for 'Operator Name' and 'Operator ID' and select OK.

6.5.3. In the 'Run Setup' box check that your sample table name and your

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method are correct and select OK.

6.5.4. Specify the file name for the results report, using the same convention as for the sample table (6.4.4.) (e.g. YYMMDDT#.RST)

6.5.5. Click on the play button to view your baseline. Check for bubbles in the flow cell (6.3.7.). After your baseline is stable, zero it out (6.3.9.).

6.5.6. Start the run by clicking on the fast forward button.

6.5.7. After the run is finished print the peak table, calibration, and the peak plot. To print the run report go to 'view' in the menu bar, select 'reports', and print report 'by peak'.

6.6. Quantitation/Calculations:

6.6.1. The instrument reports results for TKN analysis in ppb. The results are converted to mg N/L when uploaded into the QC Manager program. Transfer the prep batch ID's assigned in the electronic prep worksheet to the QC Manager file.

6.6.2. The QC Manager calculates recoveries for spikes and QC standards as well as relative percent differences (RPD) for duplicate samples and spikes. For general calculations including RPDs and spike recoveries see Section 5.9 of the FDEP Quality Manual.

6.6.3. The working calibration standards are prepared according to section 5.3.3. The correlation coefficient for the weighted linear calibration is calculated by the instrument software. Recoveries for the calibrants are calculated according to Section 5.9 of the FDEP Quality Manual.

7. DATA ARCHIVAL

7.1. The raw data is archived in paper format. A report folder is created for each analytical run. Occasionally more than one run may be in one folder. The folder is labeled with the test(s) (W-TKN, W-TKN-F), filename/s (YYMMDDT# or YYMMDDK# (where T is for SFA 2, K is for SFA 1, and the # indicates the 1st, 2nd etc. run of the day), and initials of the analyst.

7.2. Each report folder should contain the following items - the run report(s), the data logger temperature report, bench sheet(s), Reagent and Standard Logsheet including the digestion prep worksheet ID, and a copy of the data and QC assessments uploaded from QC Manager to LIMS at the time of test authorization.

7.3. The report folders are on file with the nutrients and general chemistry supervisory personnel. Periodically, the files are moved to the State Records Center for long time storage according to DEP SOP LB-006.

7.4. OI SFA instrument data excel transfer files for upload to QC manager are stored in electronic format in the current year's subfolder located at

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8. QUALITY CONTROL/DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES (SEE DEP SOPS NU-038 AND NU-043)

8.1. For method performance and acceptance criteria for QC measures and corrective action for handling out-of-control or unacceptable data refer to DEP SOPs NU-038 and NU-043.

8.2. A continuing calibration verification, CCV (5.3.3.) and a CCB (5.3.3.) are run every 10 cups and at the beginning and end of each run. An MBLK is analyzed for every batch of 10 samples. Acceptance limits for the recoveries of the check standard are 90-110 %. Acceptance limits for the CCB's and MBLK's are < 0.030 and > -0.030 mg N/L.

8.3. One certified standard reference material, ALTQC (5.3.4.), is analyzed on every analytical run. The supplier for the QC stock standard differs from the supplier of the calibration standards. Acceptance limits for the recovery of the ALTQC are 90-110 %.

8.4. One practical quantitation limit level standard, PQCK2 (5.3.3.), is analyzed on every analytical run. Acceptance limits for the recovery of the PQCK2 standard are 70-130 %.

8.5. An LFB, a matrix spike, and a matrix spike duplicate or a sample in duplicate should be analyzed for every batch of 10 samples. Acceptance limits for the recoveries of the LFB's and matrix spikes are 90-110 %. The sample/matrix spike reproducibility should be less than 20 % RPD for results over the practical quantitation limit (PQL).

8.6. Linear Calibration Range (LCR): See the FDEP Quality Manual Appendix B, Section 7.0, 'Protocol for Determining the Test Method Range of Applicability'. The correlation coefficient of the weighted linear calibration must be equal to or larger than 0.995. Recoveries for calibration standards above the PQL must be 90-110 %.

8.7. Every day samples are analyzed a new calibration must be performed at the beginning of the analysis and the LCR must be verified (see 8.6.). Consecutive runs on one day may use the same calibration.

9. SAFETY/HAZARDOUS WASTE MANAGEMENT/POLLUTION PREVENTION

9.1. Review the Laboratory Health and Safety Plan (accessible on the intranet at https://www.fldepnet.org/content/dear/bureau-laboratories-safety-documents).

9.2. Safety glasses, gloves and lab coats should be worn at all times whilst handling samples and reagents.

9.3. Read the MSDS information for safe reagent and sample handling.

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9.4. All digested standard waste, digested sample waste, and the sample waste line outflow is collected in a container labeled 'Hazardous Waste'. The waste should be disposed as acidic metals waste.

10. REFERENCES/DEFINITIONS/VALIDATION DATA

For definitions see section 3 of the FDEP Quality Manual. All abbreviations used throughout the document are defined at least once in the document usually at the first time of use. Method validation data is on file with the supervisor. A limit of detection (LOD) verification is performed annually and documented in the maintenance logbook.

10.1. EPA method 351.2, Rev. 2.0 (1993).

10.2. OI Analytical Method 327152 and User Manuals.

10.3. Quality Manual, Bureau of Laboratories, FDEP.

10.4. Laboratory Health and Safety Plan.

10.5. DEP SOP NU-033, Summary of Sample Analysis Procedures for the General Chemistry and Nutrients Workgroups.

10.6. DEP SOP NU-038, Nutrients and General Chemistry: Quality Assurance and Quality Control.

10.7. DEP SOP NU-041, Reagent Preparation Procedure.

10.8. DEP SOP NU-042, Standard Preparation Procedure.

10.9. DEP SOP NU-043, Nutrients and General Chemistry Quality Assurance and Data Authorization Guidelines

10.10. DEP SOP NU-081, Determination of Total Kjeldahl Nitrogen in Soils Using the Westco SmartChem Discrete Analyzer.

10.11. DEP SOP NU-084, Determination of Total Phosphorus in Soils Using the AQ2 Discrete Analyzer.

10.12. DEP SOP NU-092, Determination of Total Kjeldahl Nitrogen in Water Using the Seal AQ2 Discrete Analyzer.

10.13. DEP SOP NU-095, Determination of Ammonia in Water by Gas Diffusion Segmented Flow Analysis.

10.14. DEP SOP NU-097, Preparation of Waters for Total Kjeldahl Nitrogen Analysis (Copper catalyst, Low Volume)

10.15. DEP SOP LB-006, Standard Operating Procedure for Records Maintenance and Storage.

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Appendix of Significant Changes

10/30/2013 Documentation protocol for standards prepared daily was added to the note in section 5. The standard preparation procedures in 5.3.2 and 5.3.3 were modified.

04/01/2014 Started use of 30 % Brij solution (from Ricca) instead of 21 % (from OI Analytical). Final Brij concentration was not changed.

10/07/2015 Data archival information was clarified in Section 7.

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