Villanova University  
Villanova Urban Stormwater Partnership  
Watersheds Laboratory

Standard Operating Procedure – VUSP – P  
Chloride, USEPA by Discrete Analysis  
Method Easy Chloride 325.2-01  
(Colorimetric, Automated, Ferricyanide)

Author: _____________________________________________________  
Water Resources Laboratory Technician – Sonali Joshi

Approved: ___________________________________________________  
VUSP Director Robert G. Traver

Revised from Easy Chem Methodology  
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Systea Scientific, LLC  
900 Jorie Blvd., Suite #35  
Oak Brook, Illinois 60523
Distribution

Sonali Joshi – Laboratory Technician
Laboratory Copy, maintained by Quality Assurance Officer

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<thead>
<tr>
<th>Revision</th>
<th>Date</th>
<th>Responsible Person</th>
<th>Description of Change</th>
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<td>Mar 2008</td>
<td>Mary Ellen Dukart</td>
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<td>1</td>
<td>Oct 2011</td>
<td>Sonali Joshi</td>
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The following laboratory staff have read and been trained on this Manual. A copy of this page will be maintained in the Laboratory. Training is good for one year.

<table>
<thead>
<tr>
<th>Signature</th>
<th>Name / Position</th>
<th>Date</th>
<th>Trainer</th>
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<tbody>
<tr>
<td></td>
<td>Robert G. Traver Director</td>
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<td>Bridget Wadzuk PI</td>
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<td>John Komlos</td>
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<td>Quality Assurance Officer</td>
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CONTENTS

1. Scope and Application ................................................................. 4

2. Summary of Method… ................................................................. 4

3. Definitions................................................................................. 4

4. Interferences.............................................................................. 4

5. Safety......................................................................................... 5

6. Equipment and Supplies ............................................................ 5

7. Reagents and Standards.............................................................. 5

8. Sample Collection, Preservation, and Storage ............................... 8

9. Quality Control (USEPA Guidelines). ........................................... 8

10. Calibration and Standardization ................................................. 13

11. Procedure ............................................................................... 14

12. Data Analysis and Calculations ................................................. 15

13. Pollution Prevention ................................................................. 16

14. Waste Management ................................................................. 16

15. References .............................................................................. 16

16. Tables, Diagrams, and Validation Data ........................................ 16

17. Definitions .............................................................................. 17

18. Consumables .......................................................................... 18

19. Procedures for Mercury Precipitation ....................................... 19
EasyChem Methodology

Chloride, USEPA by Discrete Analysis

Method EASY Chloride 325.2-01

(Colorimetric, Automated, Ferricyanide)

1.0 Scope and Application

1.1 This method is used for the determination of Chloride in drinking water, surface, and saline waters, as well as domestic and industrial wastes according to USEPA Method 325.2 (Reference 16.1).

1.2 This method is for use in the Environmental Protection Agency’s data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response Compensation and liability Act, NELAC, and the Safe Drinking Water Act.

1.3 The Method Detection Limit (MDL) of this method is 1mg/L Chloride. The applicable range of the method is 1-200 mg/L Chloride. The range may be extended to higher or lower concentrations by sample dilutions, sample to reagent ratio changes, and larger or smaller flow cells.

1.4 This method is performance based. The laboratory is permitted to omit any step or modify any procedure (e.g., to overcome interferences, to lower the cost of measurements), provided that all performance requirements in this method are met.

1.5 Each laboratory must demonstrate the ability to generate acceptable results using procedure in Section 9.0.

1.6 CASRN Chloride 7723-14-0

2.0 Summary of Method

2.1 The thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms a highly colored ferric complex of which the intensity is proportional to the chloride concentration. The colored complex is measured at 480nm.
2.2 See section 17.0 for complete method information.

3.0 Definitions
   Definitions for terms used in this method are provided in Section 17.0.

4.0 Interferences
   4.1 There are no significant interferences.
   4.2 Filter turbid samples before determination.

5.0 Safety
   5.1 The toxicity or carcinogenicity of each analyte or reagent has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this be made available to the analyst.
   5.2 The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be available to all personnel involved in the analyses. Additional information on laboratory safety can be found in References 16.3-16.5.
   5.3 The following chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate MSDS before handling.
      5.3.1 Ferric Nitrate Fe(NO₃)₃·9H₂O (FW 404.00)
      5.3.2 Mercuric Thiocyanate Hg(SCN)₂ (FW 316.75)
      5.3.3 Nitric Acid, concentrated HNO₃ (FW 63.02)
      5.3.4 Thioacetamide CH₃CSNH₂ (FW 75.13)

6.0 Equipment and Supplies
   6.1 Equipment
      6.1.1 EasyChem Analyzer
      6.1.2 Computer for data collection
6.1.3 Printer

6.1.4 List of consumables used in this method are provided in Section 18.0.

7.0 Reagents and Standards Unless otherwise specified all chemicals should be of ACS grade or equivalent.

7.1 List of Raw Materials Safety Classification

7.1.1 Ferric Nitrate Fe(NO$_3$)$_3$·9H$_2$O (FW 404.00) harmful

7.1.2 Mercuric Thiocyanate Hg(SCN)$_2$ (FW 316.75) toxic

7.1.3 Nitric Acid, concentrated HNO$_3$ (FW 63.02) caustic

7.1.4 Thioacetamide CH$_3$CSNH$_2$ (FW 75.13) toxic

7.1.5 Methanol CH$_3$OH (FW 32.04) flammable

7.1.6 Sodium Chloride NaCl (FW 58.44) --

7.2 Reagents Unless otherwise specified all chemicals should be of ACS grade or equivalent.

7.2.1 Stock Mercuric Thiocyanate Solution (100ml)

Mercuric Thiocyanate 0.417g
Methanol to 100mL
Dissolve 0.417g of mercuric thiocyanate in approximately 50mL of methanol contained in a 100mL volumetric flask. Dilute the solution to 100mL with methanol and mix well.

**Caution:** Mercuric thiocyanate is a poison. Do not breathe dust, and wash hands thoroughly after handling. Methanol is flammable. Ingestion may be fatal or cause blindness. Avoid prolonged breathing of vapor. Use both materials only with adequate ventilation. Weigh these in the hood.

7.2.2 Stock Ferric Nitrate Solution (100ml)

Ferric Nitrate 20.2 g
Nitric Acid, Concentrated 3.15mL
DI water to 100mL
Dissolve 20.2g of ferric nitrate in approximately 50mL of deionized water contained in a
100mL volumetric flask. Carefully add 3.15mL of nitric acid. Dilute the solution to
100mL with deionized water and mix well.

7.2.3 Working Chloride Color Reagent (100ml)

Stock Mercuric Thiocyanate Solution 15mL
Stock Ferric Nitrate Solution 15mL
DI water to 100mL
Add 15ml of Stock Mercuric Thiocyanate Solution and 15ml of Stock
Ferric Nitrate Solution to approximately 50mL of deionized water
contained in a 100mL volumetric flask. Dilute the solution to 100mL with
deionized water and mix well.

7.3 Standards

7.3.1 Stock Calibrant 10,000 mg/L (100mL)

Sodium Chloride, Dried at 140°C 1.6484g
DI water to 100mL
Dissolve 1.6484g of dry sodium chloride in approximately 80mL of
deionized water contained in a 100mL volumetric flask. Dilute the
solution to 100mL with deionized water and mix well.

7.3.2 Intermediate Calibrant 1000mg/L Chloride (100mL)

10,000 mg/L Stock Standard 10mL
DI water to 100mL
Dilute 10mL of 10,000 mg/L Stock Standard in a 100mL volumetric flask
with DI water.

7.3.3 Working Standard Solution 100mg/L (100ml)

1000 mg/L Intermediate Calibrant 10mL
DI water to 100mL
Dilute 10mL of 1000 mg/L Intermediate Calibrant in a 100mL volumetric
flask with DI water.

7.3.4 Working calibrants may be prepared to cover the desired range by adding
the appropriate amount of working standard solutions (Section 7.3.3) to
100 mL volumetric flasks that contain approximately 80 mL DI water.
Dilute the solutions to 100 mL with DI water. Prepare working calibration
standards daily. See Table below for specific directions.

7.3.5 The following formula can be used to calculate the amount of stock (or
working) calibrant to be used.
\[ C_1V_1 = C_2V_2 \]

where:

\[ C_1 = \text{desired concentration (in mg/L) of working calibrant to be prepared} \]
\[ V_1 = \text{final volume (in mL) of working calibrant to be prepared (generally 100mL)} \]
\[ C_2 = \text{concentration (in mg/L) of stock (or working) calibrant} \]
\[ V_2 = \text{volume (in mL) of stock (or working) calibrant to be used} \]

Rearranging the equation to solve for \( V_2 \) yields:

\[ V_2 = \frac{C_1V_1}{C_2} \]

For example, to prepare a 1.0mg/L working calibrant from a 1000mg/L stock calibrant, use 0.1mL (100µL) of the stock calibrant in 100mL final volume.

\[ V_2 = \frac{(1.0\text{mg/L})(100\text{mL})}{1000\text{mg/L}} \]
\[ V_2 = 0.1\text{mL} \]

Add this amount of stock calibrant to the volumetric flask and then dilute to volume with the sampler wash solution.

Standard curves in desired ranges can be derived from the formula above or the tables below.

The following table provides the quantity of secondary standard necessary to prepare working standards of the specified concentration.

<table>
<thead>
<tr>
<th>Working Calibration Standard Concentration (mg/L) (100mL)</th>
<th>Standard Solution Volume Added to Volumetric Flask</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working Standard</td>
<td>Working Standard</td>
</tr>
</tbody>
</table>

SOP-P-8
8.0 Sample Collection Preservation and Storage

8.1 No special requirements.

8.2 Samples may be held for 28 days.

9.0 Quality Assurance/Quality Control

9.0.1 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis. The procedures and criteria for analysis of a blank are described in Section 9.1.

9.0.2 Analysis of calibrant is required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis. The procedures and criteria for analysis of a blank are described in Section 9.2.

9.0.3 Accompanying QC for the determination of chloride is required per analytical batch. An analytical batch is a set of samples analyzed at the same time, to a maximum of 10 samples. Each analytical batch of 10 or fewer samples must be accompanied by a calibrant and a laboratory blank in the batch. If greater than 10 samples are analyzed at one time, the samples must be separated into analytical batches of 10 or fewer samples.

9.1 Laboratory Blanks (LB)—Laboratory blanks are analyzed to demonstrate freedom from contamination.
9.1.1 Analyze DI water blank initially and with each analytical batch. The blank must be subjected to the exact same procedural steps as the samples.

9.1.2 If analyte is detected in the blank at a concentration greater than the lowest calibration standard, analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination.

9.2 Calibration Verification—Verify calibration of the analytical equipment before and after each analytical batch of 10 or fewer measurements. This can be accomplished by analyzing at least one lower range calibration standard and verifying that it is within +/-25% of the true value. (The concentration of the calibration verification depends on the calibration range being used.) Failure to attain recoveries within the acceptance criteria requires recalibration of the analysis system.

10.0 Calibration and Standardization

10.1 Working Standard should be prepared as required to cover range of analysis at a minimum of four standards and a blank. If analyzing samples not dissolved in pure DI water, dilute the working standards with sample matrix solution.

10.2 Calibration should include a minimum of 4 standards and a blank. However, the blank should be disabled after the run is completed. The linear correlation coefficient \( r^2 \) should be greater than 0.995.

10.3 Prepare standard curve by plotting instrument response against concentration readings. A calibration curve may be fitted to the calibration standards response data using the computer. Acceptance or control limits should be established using the difference between the measured reading of the calibration standards and the “true” value concentration.

11.0 Procedure

11.1 Instrument Configuration

11.1.1 Configure the EasyChem Discrete Analyzer according to the Operator’s Manual, including computer, communication cables, etc.

11.1.2 Power On the analyzer using the power switch located on back, bottom left corner.

11.1.3 Select EasyChem icon to open data acquisition software. When the system has been properly initialized a message box will display “Analyzer Ready”.

11.1.4 Fill the wash water bottle with DI water; insert the wash tube into the bottle.
11.1.5 Close the peristaltic pump located on the right side of the analyzer, by pressing the pump plate upwards and screwing the plate knob.

11.1.6 Empty waste container.

**Note:** Mercuric thiocyanate is a poison. The mercury in the waste can be precipitated as mercuric sulfide to reduce the volume for disposal. Refer to Section 19.0.

11.1.7 Initiate filters auto zero function insuring all values fall into specified ranges.

11.1.8 Fill position 18-reagent container with reagent water for the dilution of standards (if necessary).

11.1.9 Perform water test as detailed in EasyChem Discrete Analyzer Operator’s Manual to insure proper instrument functionality. All values should be consistently very low otherwise follow instruction detailed in Operator’s Manual.

11.1.10 Fill reagent 1 container as detailed in Section 7.0 and place is position specified in Chloride Method.

11.1.11 Place all required standards (calibrants, QC, LCS, etc) into sample tray. Input information required by the data acquisition software such as concentrations, replicates.

11.1.12 Calibrate instrument by analyzing standards. The data acquisition system will then associate the concentrations with the instrument OD readings for each standard.

11.1.13 After an approved calibration has been obtained load the required samples into the sampler tray and perform analysis.

### 11.2 Method Parameters

<table>
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<th>Standard Sensitivity</th>
<th>10 mm Path Length Flowcell</th>
<th>1 to 200 ppm Chloride</th>
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<tr>
<td>High Sensitivity</td>
<td>20 mm Path Length Flowcell</td>
<td>0.10 to 10 ppm Chloride</td>
</tr>
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</table>

The flow cell in use in the lab is 10mm path length flowcell.
12.0 Data Analysis and Calculations

12.1 Prepare a calibration curve by plotting instrument readings (OD) against standard concentration. Compute sample concentration by comparing sample response with the standard curve.

12.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

13.0 Pollution Prevention

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, recycling is the next best option.

13.2 The quantity of chemicals and standards purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent and standard preparation volumes should reflect anticipated usage and reagent stability.

13.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult “Less is Better: Laboratory Chemical Management for Waste Reduction,” available from the American Chemical Society’s Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington DC, 20036, 1-202-872-4477.

14.0 Waste Management

14.1 It is the laboratory’s responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14.2 For further information on waste management, consult ”The Waste Management Manual for laboratory Personnel,” and “Less is Better: Laboratory Chemical Management for Waste Reduction,” both available from the American
15.0 References


15.8 Technicon Industrial Method Number 99-70W, Chloride in Water and Wastewater, Technicon Instruments Corporation, Tarrytown, New York.

16.0 Table, Diagrams, and Validation Data

16.1 Method parameters for Chloride (1-200mg/L)
16.2  Calibration parameters for Chloride (1-200mg/L)
16.3 Quality Control parameters for Chloride (1-200mg/L)
17.0 The definition and purposes below are specific to this method, but have been conformed to common usage as much as possible.

17.1 **Calibration Blank (CB):** A volume of reagent water in the same matrix as the calibration standards, but without any contamination from the method analyte.

17.2 **Calibration Standard (CAL):** A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

17.3 **Clean Water Act (CWA):** An act to provide for water pollution control in the Public Health Services of the Federal Security Agency and in the Federal Works Agency and for other purposes as specified in the Federal Water Pollution Control Act.
17.4 **Chemical Abstracts Service Registry Number (CASRN):** The largest substance identification system in existence. When a chemical substance, newly encountered in the literature, is processed by CAS, its molecular structure diagram, systematic chemical name, molecular formula, and other identifying information are added to the Registry and it is assigned a unique CAS Registry Number.

17.5 **Comprehensive Environmental Response Compensation Liability Act (CERCLA):** The 1980 Superfund statute established liability to the U.S. Government for damage to natural resources over which the U.S. has sovereign rights and requires the President to designate Federal officials to act as trustees for natural resources.

17.6 **Deionized Water (DI Water):** The vast majority of dissolved impurities in modern water supplies are ions such as calcium, sodium, chlorides, etc. The deionization process removes ions from water via ion exchange.

17.7 **Environmental Protection Agency (EPA):** The mission of the Environmental Protection Agency is to protect human health and the environment.

17.8 **Instrument Precision Check (IPC):** Also known as a Continuing Check Value (CCV) used to evaluate the performance of the instrument throughout the analysis.

17.9 **Linear Calibration Range (LCR):** The concentration range over, which the instrument response is linear.

17.10 **Laboratory Reagent Blank (LRB):** An aliquot of reagent water or other blank matrices that is digested exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or other apparatus.

17.11 **Method Detection Limit (MDL):** The EPA defines MDL as “the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero.” This procedure is outlined in 40 CFR 136. Prepare a solution of the analyte that is three to five times the estimated detection. Test this solution seven or more times, then determine the standard deviation of the data set. The method detection limit is calculated according to the formula: MDL = Student’s (t) value x the standard deviation.

17.12 **Matrix Spike (MS):** Also known as a Laboratory Spiked Blank (LSB). An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control,
and whether the laboratory is capable of making accurate and precise measurements.

17.13 **Matrix Spike Duplicate (MSD):** Also known as a Laboratory Spiked Sample Matrix (LSM). An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

17.14 **Material Safety Data Sheet (MSDS):** Written information provided by vendors concerning a chemical’s toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

17.15 **National Institute of Standards and Technology (NIST):** Founded in 1901, NIST is a non-regulatory federal agency within the U.S. Commerce Department's Technology Administration. NIST’s mission is to develop and promote measurement, standards, and technology to enhance productivity, facilitate trade, and improve the quality of life.

17.16 **Occupational Safety and Health Administration (OSHA):** OSHA’s mission is to assure the safety and health of America’s workers by setting and enforcing standards; providing training, outreach, and education; establishing partnerships; and encouraging continual improvement in workplace safety and health.

17.17 **Quality Control Sample (QCS):** Also known as a Laboratory Control Sample (LCS). A solution of method analytes of known concentrations that are used to verify stock standard solutions or spike an aliquot of MS or MSD. The QCS is obtained from a source external to the laboratory and different from the source of the calibration standards. It is used to check laboratory performance with externally prepared test materials.

17.18 **Resource Conservation and Recovery Act (RCRA):** RCRA gave EPA the authority to control hazardous waste from the "cradle-to-grave." This includes the generation, transportation, treatment, storage, and disposal of hazardous waste. RCRA also set forth a framework for the management of non-hazardous wastes.

17.19 **Safe Water Drinking Act (SWDA):** The Office of Ground Water and Drinking Water (OGWDW), together with states, tribes, and its many partners, protects public health by ensuring safe drinking water and protecting ground water. OGWDW, along with EPA’s ten regional drinking water programs, oversees implementation of the Safe Drinking Water Act, which is the national law safeguarding tap water in America.

17.20 **Stock Standard Solution (SSS):** A concentrated solution containing one or more method analytes prepared in the laboratory using reference materials or purchased from a reputable commercial source.
18.0 Consumables used for this method.

<table>
<thead>
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<th>P/N</th>
<th>Description</th>
<th>Sales unit</th>
<th>Estimated consumption</th>
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<tr>
<td>CUP-EASY-01</td>
<td>SAMPLE CUPS 2.0 ml</td>
<td>Pk/500</td>
<td>500 tests</td>
</tr>
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<td>E/F25</td>
<td>FILTERS - SAMPLE PROBE CLEANER</td>
<td>Pk/25</td>
<td>1 pk per month</td>
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<td>E/R18</td>
<td>REAGENT BOTTLES</td>
<td>Pk/18</td>
<td>1 pk 12 months</td>
</tr>
<tr>
<td>E/RZ24</td>
<td>REACTION CUVETTES</td>
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<tr>
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<td>Pk/25</td>
<td>1 pk per month</td>
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<td>1 yearly</td>
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<tr>
<td>E/K02</td>
<td>PROBES KIT</td>
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<td>SOURCE LAMP ASSEMBLY</td>
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<td>SILICONE TUBE 2 x 4 mm</td>
<td>meter</td>
<td>5m per year</td>
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<td>EASY-FLOW-10</td>
<td>EASYCHEM 10MM FLOWCELL</td>
<td>EA</td>
<td>1 yearly</td>
</tr>
</tbody>
</table>

19.0 Procedure for Mercury Precipitation

19.1 Reagent for Mercury Precipitation

Thioacetamide 13% Solution (100mL)

Thioacetamide 13g
DI water to 100mL

Dissolve 13g of thioacetamide in approximately 75mL of deionized water contained in a 100mL volumetric flask. Dilute the solution to 100mL with deionized water and mix well.

19.2 Procedure For Mercury Precipitation

19.2.1 Add 20mL of 13% thioacetamide per liter of chloride waste

19.2.2 Mix the solution well and allow it to stand under a ventilation hood for 24 hours. The container should be capped. A precipitate of mercuric sulfide will form.

19.2.3 Filter the solution under the hood through filter paper.

19.2.4 Discard the filtrate as normal non toxic waste.

19.2.5 Store the mercuric sulfide residue in a glass container until it can be disposed of properly.

**Note:** Sulfur dioxide is produced in this reaction. This procedure must be performed with adequate ventilation.
<table>
<thead>
<tr>
<th>Watersheds Laboratory</th>
<th>Chloride – Easy Chem</th>
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<tbody>
<tr>
<td>VU Stormwater Partnership</td>
<td>SOP – VUSP –P</td>
</tr>
<tr>
<td>Villanova University</td>
<td>Date Oct 2011</td>
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</tbody>
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