Colorado Procedure – Laboratory 2104-21

Standard Method of Test for

Determining the Chloride Ion Content in Water or Water-Soluble Chloride Ion Content in Soil

1. SCOPE

1.1 This method covers the procedures for chloride ion content determination in soil or water.

1.2 Method A describes the procedure for determining the concentration of chloride ions present in water. Method B describes the concentration of water soluble chloride ions present in soil using titration. Method C describes the concentration of water soluble chloride ions present in soil using the X-Ray.

2. REFERENCED DOCUMENTS

2.1 AASHTO Standards:
T291 Determining Water-Soluble Chloride Ion Content in Soil.

2.2 Colorado Procedures:
CP 30 Sampling of Aggregates
CP 32 Reducing Field Samples of Soil and Aggregate to Testing Size.

2.3 Other Procedures:
HACH Method 8207 Silver Nitrate Method (10 to 10,000 mg/L as Cl-).

Note 1: This method was adapted from AASHTO T291 and HACH Method 8207.

3. SIGNIFICANCE AND USE

3.1 This method is capable of detecting chloride concentrations up to 10,000 parts per million in water or 3.00% in soil.

3.2 Iron in excess of 10 mg/L masks the endpoint.

3.3 Orthophosphate in excess of 25 mg/L will precipitate silver.

3.4 Sulfite in excess of 10 mg/L will cause interference. If sulfite interference is suspected, eliminate it by adding three drops of 30% hydrogen peroxide in subsection 5.7 or 7.13.

3.5 If sulfide interference is suspected, eliminate it by adding the contents of one Sulfide Inhibitor Reagent Powder Pillow to approximately 125 mL of sample, mixing for one minute, and then filtering through a folded filter paper prior to subsections 5.3 or 7.9.

3.6 Cyanide, iodide, and bromide interfere directly and while titrating they appear as chloride. No attempt to remove these is made because they are usually present in insignificant quantities compared to chloride.

4. APPARATUS

4.1 Balance that reads a minimum of 500 g, accurate to 0.1 g.

4.2 Oven capable of drying samples at a temperature not exceeding 140°F.

4.3 Sieve – 2.00mm (No. 10) and a pan.

4.4 Sample Splitter – Meeting the requirements of CP 32.

4.5 Pulverizing Apparatus

4.6 HACH digital titrator and delivery tube.

4.7 Chloride 2 Indicator Powder Pillows.

4.8 Silver Nitrate titrate cartridges, 0.2256 N and 1.128 N.

4.9 Sulfide Inhibitor Reagent Powder Pillow.

4.10 Deionized water.

4.11 Graduated cylinder.

4.12 Erlenmeyer Flask.

4.13 X-Ray capable of measuring water soluble chloride ions.

METHOD A
(Chloride Ion Content in Water)

5. PROCEDURE

5.1 Obtain a sample as required by Pipe Materials Selection Policy.

5.2 If the sample exhibits turbidity then filter the sample through a 0.45 micron membrane filter.

5.3 Check the pH with a meter or with phydrion paper. If the pH is in the range of six through eight, proceed to the next step. If the pH is below six, add sodium bicarbonate to adjust the pH to a range of 6 to 8. If the pH is above 8, add nitric acid to adjust the pH to a range of 6 to 8.

Note 2: Determine the amount of acid or base necessary in a separate sample because pH electrodes will introduce chloride into the sample

5.4 Select the sample volume and Silver Nitrate Titration Cartridge corresponding to the expected chloride concentration from *Table 1*.

5.5 Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.

5.6 Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

5.7 Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250 mL Erlenmeyer flask. Dilute to 100 mL with deionized water.

5.8 Add the contents of one Chloride 2 Indicator Powder Pillow and then swirl to mix.

5.9 Place the delivery tube tip into the solution and swirl the flask while titrating with silver nitrate until the solution changes from a yellow to a red-brown color. Record the number of digits required from the digital titrator.

6. CALCULATION

6.1 Use the following formula to calculate the concentration of chloride:

(Digits Required) x (Digit Multiplier) = mg/L or parts-per-million (ppm)

METHOD B
(Chloride Ion Content in Soil, Titration)

7. PROCEDURE

7.1 Obtain a sample according to CP 24 or as required by Pipe Materials Selection Policy.

7.2 Dry the sample to a constant weight at a temperature not exceeding 140°F.

7.3 Process the material over a No. 10 sieve being careful to dislodge any material adhering to the aggregate particles and avoid breaking down the natural size of the particles.

7.4 Split the processed soil into a 100 g sample and place it into a sealable sample cup.

7.5 Add 300 mL of distilled water (3:1 dilution).

7.6 Seal the container and shake vigorously for 20 +/- 1 seconds.

7.7 Let the sample sit at room temperature for 1 hour and then repeat the shaking process and let the sample sit for another hour.

7.8 If the sample exhibits turbidity after the completion of Subsection 7.7 then filter the sample through a 0.45 micron membrane filter.

7.9 Check the pH with a meter or with phydrion paper. If the pH is in the range of six through eight, proceed to the next step. If the pH is below six, add sodium bicarbonate to adjust the pH to a range of 6 to 8. If the pH is above 8, add nitric acid to adjust the pH to a range of 6 to 8.

Note 4: Determine the amount of acid or base necessary in a separate sample because pH electrodes will introduce chloride into the sample

7.10 Select the sample volume and Silver Nitrate Titration Cartridge corresponding to the expected chloride concentration from *Table 1*.

7.11 Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.

7.12 Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

7.13 Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250mL Erlenmeyer flask. Dilute to 100-mL with deionized water.

7.14 Add the contents of one Chloride 2 Indicator Powder Pillow and then swirl to mix.

7.15 Place the delivery tube tip into the solution and swirl the flask while titrating with silver nitrate until the solution changes from a yellow to a red-brown color. Record the number of digits required from the digital titrator.

8. CALCULATION

8.1 Use the following formula to calculate the concentration of chloride:

(Digits Required) x (Digit Multiplier) x 3 = mg/L or parts-per-million (ppm) Chloride)

Note 5: Multiplying by 3 takes into account the 3:1 dilution of the soil in water.

8.2 Divide the ppm obtained from the calculation and divide by 10,000 to obtain the percent chloride in soil by mass.

Table 1: **Concentrations**

Range (mg/L as Cl-)	Sample Volume (mL)	Titration Cartridge (N AgNO3)	Digit Multiplier
10-40	100	0.2256	0.1
25-100	40	0.2256	0.25
100-400	50	1.128	1.0
250-1000	20	1.128	2.5
1000-4000	5	1.128	10.0
2500-10000	2	1.128	25.0

METHOD C
(Chloride Ion Content in Soil, X-Ray)

5. PROCEDURE

5.1 Obtain sample according to CP 24 or as required by the Pipe Material Selection Policy.

5.2 Dry the sample to constant weight at a temperature not exceeding 60°C (140°F).

5.3 Process the material over a #4 sieve being careful to dislodge any material adhering to the aggregate particles and avoid breaking down the natural size of the particles.

5.4 Process the minus #4 material using a rubber coated pestle until it passes the #40 sieve being careful to dislodge any material adhering to the aggregate particles and avoid breaking down the natural size of the particles. Repeat until no additional minus #40 material is produced.

5.5 Prior to obtaining a test sample, ensure sample is mixed so that uniformity of the sample is achieved. Obtain a representative 25g test sample and place it in a clean, 500 ml flask.

5.6 Add 250 ml of distilled water to the flask. Mix thoroughly, by shaking, using sufficient effort so that no material is left on the bottom of the flask (this is the 1st 10:1 dilution). Seal the flask with a rubber stopper and let the sample sit undisturbed for a minimum of 16 hours. Maintain the temperature of the solution @ 140°F (+/- 5°F).

5.7 After completion of the soaking period if the sample exhibits turbidity (cloudiness) filter the solution through a #42 Whatman filter paper until a clear sample is attained.

5.8 Use a pipet to place 10 ml of solution into sample cell. Insert sample cell into x-ray, cover, and take the reading according to manufacturer's instructions. Record reading.

Note 6: X-ray is capable of determining both water soluble sulfate and chloride simultaneously.

6. CALCULATION

6.1 Multiply reading by the total dilution of 10. This will give the parts per million.

6.2 Divide the parts-per-million (ppm) obtained in Subsection 6.1 by 10,000 to obtain the percent chloride in soil by mass.