

AUSTROADS TEST METHOD ATM 710

Chloride Content of Soil

Commentary

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**Source**

This method was developed in-house by Queensland Department of Main Roads with reference to the procedures described in A Textbook of Quantitative Inorganic Analysis including Elementary Instrumental Analysis (3rd Edition) – Arthur I. Vogel: Section III, 24 – Determination of Chlorides.

**Scope**

This method describes the procedure for determining the chloride content of select backfill material intended for use in reinforced earth structures. Chloride content is determined by water extraction and titrimetric analysis of the chloride ions using silver nitrate solution.

**Further Development**

None Proposed

# References

None.

# Equipment

The following apparatus is required:

1. Drying oven of suitable capacity, having a temperature of 105 – 110°C and complying with AS 1289.0.
2. Balance of suitable capacity, with a resolution of at least 0.1 g and with a limit of performance within the range of ± 0.5 g.
3. Balance of suitable capacity, with a resolution of at least 0.0001 g and with a limit of performance within the range of ± 0.0005 g.
4. Furnace, capable of maintaining a temperature of 250°C.
5. Hotplate, capable of maintaining a temperature of 150-200°C.
6. pH meter or pH indicator paper.
7. Volumetric flasks, of 1000 mL and 100 mL capacity.
8. Conical flasks, of 125 mL capacity.
9. Measuring cylinders, of 1000 mL capacity, 100 mL capacity (capable of being stoppered) and 50 mL capacity.
10. Burette, of 25 mL capacity graduated in 0.1 mL divisions.
11. Pipettes, bulb type of 50 mL and 25 mL capacity.
12. Pasteur pipettes.
13. Pipette filler.
14. Desiccator, containing silica gel desiccant or equivalent.
15. Glass filter funnel.
16. Beakers, of 1500 mL, 600 mL and 250 mL capacity.
17. Container, mixing container of 10 litre capacity fitted with an airtight lid.
18. Magnetic stirrer and stirring bar.
19. Centrifuge.
20. Storage bottles (amber glass), of 1000 mL capacity.
21. Storage bottles, of 100 mL capacity.

# Materials

The following materials are required:

1. Distilled water or equivalent (for example, reverse osmosis water).
2. Filter paper, Whatman No. 542 or Whatman No. 541 depending on particle size in sample.

# Reagents

All reagents are analytical reagent grade, and the following are required (Notes 10.(a) and 10.(b)):

1. Standard sodium chloride solution
	* Heat the sodium chloride in a furnace maintained at 250°C for 2 hours and then cool in the desiccator. Dissolve 1.6485 ± 0.0001 g of dry sodium chloride in distilled water (or equivalent).
	* Transfer the solution to a 1000 mL volumetric flask and make up to the mark with distilled water (or equivalent). (1 mL of this solution equals 1 mg of chloride).
2. Silver nitrate solution
	* Dissolve 4.8 g of silver nitrate in distilled water (or equivalent).
	* Transfer the solution to a 1000 mL volumetric flask and make up to the mark with distilled water (or equivalent).
	* Transfer the solution to a 1000 mL amber glass storage bottle and store in a dark cupboard (Note 10.(c).
3. Potassium chromate indicator solution
	* Dissolve 5 g of potassium chromate in 80 mL of distilled water (or equivalent). While stirring, add dropwise the silver nitrate solution until a permanent red precipitate is produced.
	* Filter the solution and dilute the filtrate by making up to the mark in a 100 mL volumetric flask with distilled water (or equivalent). Transfer the solution to a 100 mL storage bottle.
4. Phenolphthalein indicator solution
	* Dissolve 0.5 g of phenolphthalein in 50 mL of 95% ethanol in a beaker and then slowly add 50 mL of distilled water (or equivalent), with constant stirring.
	* Filter the solution and then transfer to a 100 mL storage bottle.
5. Nitric acid solution
	* Decant 60 mL of distilled water (or equivalent) into a 100 mL measuring cylinder and slowly add 10 mL of concentrated nitric acid. Make up to the 100 mL mark with distilled water (or equivalent). Stopper the cylinder when not in use.
6. Sodium hydroxide solution
	* Dissolve 1 g of sodium hydroxide in distilled water (or equivalent).
	* Transfer the solution to a 100 mL measuring cylinder and make up to 100 mL with distilled water (or equivalent). Stopper the cylinder when not in use.
7. Calcium carbonate.
8. Ammonium nitrate

# Standardisation of Silver Nitrate Solution

The following procedure shall be performed in duplicate:

1. Pipette a 25 mL aliquot of standard sodium chloride solution into a 125 mL conical flask.
2. Add approximately 1 mL of potassium chromate indicator solution to the flask by pasteur pipette.
3. Stir the solution using a magnetic stirring bar and titrate with silver nitrate solution to the first permanent red-brown colour change.
4. Record the volume of the silver nitrate solution titrated to the nearest 0.1 mL.
5. Calculate the average volume of the duplicate titrations and record to the nearest 0.1 Ml (V).

# Sample Preparation

The specimen shall be prepared as follows:

1. Prepare a representative subsample of approximately 1000 g passing the 26.5 mm sieve as detailed in Test Method Q101, Subsection 6.4 for fine fraction subsamples.
2. Dry the subsample in an oven maintained at 105 to 110°C.
3. Weigh the subsample into the mixing container and record the mass to the nearest 0.1 g (m).
4. Add 1000 mL of distilled water to the container by measuring cylinder.
5. Fit the lid to the container and shake by hand for 2 minutes and then allow to stand for 45 minutes.
6. Shake the container for a further 2 minutes and then allow to stand for 15 minutes.
7. Carefully decant the extract solution from the container, centrifuge to separate fines and then filter.

# Procedure

The procedure shall be as follows:

1. Pipette 100 mL (Vs) of the sample extract into a 250 mL beaker.
2. Check the neutrality of the solution using pH indicator paper or a pH meter.
	* If the solution is acidic, neutralise by adding a minimal quantity of calcium carbonate (approximately 0.1 g is usually sufficient) and allowing the solution to stand for a few minutes.
	* If the solution is alkaline, neutralise by adding 4 to 5 drops of phenolphthalein indicator solution and nitric acid solution dropwise until the colour changes from pink to just colourless.
3. Add approximately 1 mL of the potassium chromate indicator to the solution and titrate with the silver nitrate solution to the first permanent red-brown colour change. For lengthy titrations, add additional 1 mL increments of potassium chromate indicator for every 10-15 mL of silver nitrate solution used.
4. Record the volume of silver nitrate solution titrated to the nearest 0.1 mL (Vt).
5. Perform a blank titration substituting 100 mL of distilled water (or equivalent) for the sample extract, and repeating Steps 8.1 to 8.5. Record the volume of the blank titration to the nearest 0.1 mL (Vb).

# Equation

1. Calculate the chloride content of the sample for each run of the procedure to the nearest 1 mg/kg as follows:

$$C=\frac{25\left(V\_{t}-V\_{b}\right)×10^{6}}{V×V\_{s}×m}$$

|  |  |  |  |
| --- | --- | --- | --- |
| where | C | = | chloride content of sample (mg/kg) |
|  | Vt | = | volume of silver nitrate solution titrated against sample aliquot extract (mL) |
|  | Vb | = | volume of silver nitrate solution titrated against blank (mL) |
|  | V | = | average volume of silver nitrate solution titrated during standardisation (mL) |
|  | Vs | = | volume of sample extract (mL) |
|  | m | = | mass of subsample (g) |

1. Calculate the average of the two results and record as the chloride content of the sample to the nearest 1 mg/kg.

# Test Report

Report the chloride content of the sample to the nearest 10 mg/kg.

# Notes on Method

1. Before handling chemicals and preparing reagents, the operator should consult the relevant SDS.
2. Unless otherwise indicated, all reagents are to conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
3. Silver nitrate should be handled carefully to avoid any spillage of the solution which readily leaves black stains on bench tops and so on. Should staining occur, it can be removed using a reagent prepared by dissolving 75 g of each of thiourea and citric acid in 1 L of distilled water (or equivalent). This reagent is stable indefinitely.

Amendment Record

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| --- | --- | --- | --- |
| **Amendment no.** | **Clauses amended** | Action | Date |
| - | New test method | New | December 2022 |
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| **Key** |  |
| Format | Change in format |
| Substitution | Old clause removed and replaced with new clause |
| New | Insertion of new clause |
| Removed | Old clauses removed |