Transport Roads & Maritime Services

NSW GOVERNMENT

Test method T1010

Quantitative determination of chlorides in soil

NOVEMBER 2012

Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		Reformatted and Revision Summary Added	D.Dash	June 2001
Ed 2/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as 'RMS'.

The most recent revision to Test method T1010 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1010

Quantitative determination of chlorides in soil

1. Scope

This test method sets out the procedure for the determination of chlorides in soil by titrating a solution of the soluble ions extracted from the soil with silver nitrate using potassium chromate indicator. The method is derived from the method set out in American Society for Testing and Materials Designation D512 Method B.

2. Apparatus

(a) A 2.36 mm woven wire AS sieve.

(b) 1 litre plastic bottle with liquid tight seal.

(c) Filtration Funnel.

(d) Whatman No. 42 filter paper (110 mm diameter).

(e) Filter paper pulp.

(f) Laboratory glassware including burettes, pipettes, casserole or Erlenmeyer flasks, etc.

3. Reagents

(a) Phenolphthalein Indicator Solution (10 g/l).

(b) Potassium Chromate Indicator Solution.
Dissolve 50 g of potassium chromate (K_2Cr0_4) in 100 ml of distilled water and add silver nitrate until a slight red precipitate is produced. Allow to stand, protected from light, for at least 24 hours. Filter to remove the precipitate and dilute to 1 litre with distilled water.

(c) Standard Silver Nitrate Solution (0.025 N).
Crush approximately 5 g of silver nitrate ($AgNO_3$) crystals and dry to constant mass at 40°C. Dissolve 4.2473 ± 0.0002 g in water and dilute to 1 litre.

(d) Sodium Hydroxide Solution.
Dissolve 1 g of sodium hydroxide in water and dilute to 100 ml.

(e) Sulphuric acid

(f) Carefully add 1 volume of concentrated sulphuric acid (1.84 g/ml) with constant stirring to 19 volumes of distilled water.

CAUTION: Sulphuric acid can cause severe burns. Avoid contact with eyes, skin and clothing. Always dilute by carefully adding acid to water - NEVER THE REVERSE. Always wear safety glasses when handling acid.

(g) Ammonium nitrate A.R. Grade.

4. Extraction of Soluble Salts

(a) Weigh out, to the nearest 0.1 g, 200 g of soil passing a 2.36 mm sieve into a suitable 1 litre plastic bottle.

(b) Add exactly 500 ml distilled water and shake well for 1 minute (500 ml is conveniently measured using a volumetric flask).

(c) Allow to stand for 1 hour with occasional shaking, making sure that the soil is well broken up.

(d) After the soil has settled (up to 1 hour after the last shaking) pipette off a 100 ml aliquot for analysis (See Note below). Filter if necessary through a "Whatman" No. 42 filter paper half full of paper pulp.

NOTE: The water layer above the soil should be substantially free from suspended matter, otherwise errors in volume will be introduced. If it is not clear, the addition of 0.2 g of ammonium nitrate after the first shaking is often helpful in settling out difficult soils. A slight turbidity is not harmful in the determination of chlorides. In cases of obstinate turbidity the solution must be clarified by passing it through a millipore filter.

5. Procedure

(a) Place the 100 ml aliquot from *Clause 4 (d)* above into a 250 ml flask.

(b) Add phenolphthalein and adjust the pH to the end-point (pH 8.3) using dilute sulphuric acid (1:19) or sodium hydroxide solution (10 g/l).

(c) If sulphite ion is present add 0.5 ml of 30% hydrogen peroxide and mix for one minute. Adjust the pH as in (b) above.

(d) Add approximately 1 ml of potassium chromate indicator solution and mix.

(e) Titrate with standard silver nitrate solution until the brick-red or pink colour persists throughout the sample.

6. Calculations and Reporting

Calculate the chloride content in the original sample, to the nearest 0.1%, as follows:

$$\text{Chloride Content \%} = V \times M \times \frac{V_1}{V_2} \times \frac{3.5453}{M_2}$$

Where

V	=	Volume of titre (ml)
M	=	Molarity of silver nitrate
V_1	=	Volume of water added to bottle (ml)
V_2	=	Volume of aquilot for titration (ml)
M_2	=	Mass of sample (grams)