

Australian Standard[®]

AS 5101.3.2—2008

Methods for preparation and testing of stabilized materials

Method 3.2: Lime or cement content of stabilized pavement materials (EDTA method)

1 SCOPE

This Standard sets out the method for determining the lime or cement content of samples of soils, gravel or crushed rock stabilized with lime or cement either in a cured or uncured state by titrating with ethylene diamine tetra-acetic acid disodium salt (EDTA) to determine the soluble calcium ion content (Ca^{2+}).

The method is dependent on determining the amount of calcium ions in the stabilized material, the lime or cement additive and the unstabilized material on a dry basis.

NOTE: With certain clay minerals present in the matrix, low results may be observed. In such cases, more accurate results may be obtained by using a standard addition technique, that is, standards prepared from known masses of lime and pavement material. These techniques are not described in this Standard and the number of this Standard is not applicable if a standard addition technique is used.

2 PRINCIPLE

This method involves steps designed to achieve the following:

- (a) To dissolve the available calcium in weak acid (it assumes that all the soluble calcium comes from the stabilizing agent).
- (b) To remove the iron oxide and aluminium oxide, which would otherwise react with EDTA to give a false high result, as follows:
 - (i) Removing elemental iron and other ferromagnetic minerals and contamination with a magnet.
 - (ii) Baking to coarsen gelatinous silicates which could block filtering.
 - (iii) Making very slightly alkaline to precipitate $\text{Fe}(\text{OH})_3$ and $\text{Al}(\text{OH})_3$ by slow and not excessive addition of ammonia. (Too quick addition may result in gelatinous precipitate.)
 - (iv) Filtering the precipitate.
- (c) To determine the concentration of calcium ions by titration. One mole of Ca^{2+} ions reacts with one mole of EDTA.

3 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS	
1141	Methods for sampling and testing aggregates
1141.2	Method 2: Basic testing equipment
1152	Specification for test sieves

1289 Methods of testing soils for engineering purposes

1289.1.2.1 Method 1.2.1: Sampling and preparation of soils—Disturbed samples—
Standard method

4 APPARATUS

The following apparatus, complying with the relevant provisions of AS 1141.2, is required:

- (a) *Analytical balance*—of sufficient capacity, with a limit of performance of ± 0.005 g or better.
- (b) *Oven*—capable of maintaining a temperature within the range of 105°C to 110°C.
- (c) *Crushing and grinding equipment*.
- (d) *Mortar and pestle*.
- (e) *Sieves*—4.75 mm and 425 μ m sieve in accordance with AS 1152.
- (f) *Magnet*.
- (g) *Laboratory glassware*—50 mL, 100 mL and 1000 mL measuring cylinders, 50 mL burette, 10 mL pipette, 250 mL conical flasks, 500 mL conical flasks, 500 mL beakers, 100 mL volumetric flask, pasteur pipette (medicine dropper), clock-glasses to cover beakers, and glass rods.
- (h) *Hotplate*—to heat between 400°C and 600°C.
- (i) *Water bath*—capable of operating close to 100°C; OR *Steam rings*—an apparatus utilizing steam to transfer heat.
- (j) *pH papers*—range 9 to 13.
- (k) *Paper pulp filtering agent* (lime content only).
- (l) *Filter funnel and stand*.
- (m) *Filter paper*—to fit funnel (Whatman No. 41 or other papers of similar grading).

5 REAGENTS

5.1 General

All reagents shall be analytical grade. Distilled or demineralized water shall be used for the analysis.

5.2 Reagents

The following reagents are required:

- (a) *Ammonium chloride or ammonium nitrate*.
- (b) *Dilute ammonia solution*—9 M made by diluting commercial ammonia solution (25–35%) to approximately 15% (1 part solution to 1 part water); store in a stoppered bottle to prevent carbonation.
NOTE: Ammonia solution can be tested for carbonation by the addition of a little barium chloride, which will cause a precipitate if carbonate is present.
- (c) *Dilute hydrochloric acid* (1:2 hydrochloric acid)—3 to 4 molar (made by diluting 1 part of concentrated acid with 2 parts water).
- (d) *Dilute hydrochloric acid* (1:1 hydrochloric acid)—5 to 6 molar (made by diluting 1 part of concentrated acid with 1 part water).
- (e) *Potassium hydroxide solution*—3 M (made by dissolving 200 g of potassium hydroxide in water and making up to 1 L with water).

- (f) *Solution of EDTA disodium salt*—0.0250 mol/L (usually made up using a commercially certified ampoule).
- (g) *Methyl red indicator*—0.1 g methyl red in 100 mL of 1:1 ammonia solution (see Item (b)).
- (h) *Nitric acid* concentrated 65%.
- (i) *Wash solution of ammonium chloride* (20 g/L)—brought to a pH 7 with ammonia. Ammonium nitrate solution (20 g/L) should be used if there are high concentrations of iron.
- (j) *Mixed indicator*—1.0 g of naphthol green B and 0.2 g of murexide (ammonium purpureate) ground together and mixed with 100 g of sodium chloride.

6 SAMPLES

The samples shall be prepared as follows:

- (a) Obtain a sample of the material, prior to the addition of the stabilizing agent, in accordance with AS 1289.1.2.1.
- (b) Obtain a sample of the stabilized material in accordance with AS 1289.1.2.1.
- (c) Obtain a representative sample of the stabilizing agent.
- (d) If the sample from either Item (a) or (b) is excessively wet, dry the sample, unless otherwise specified, until it is apparently dry. Crush and riffle the sample to obtain suitable subsamples for use in test portions (see Clause 7) to result in the required masses (i.e., 5 g or 0.5 g as appropriate).

7 TEST PORTIONS

7.1 Stabilized material

Prepare stabilized material as follows:

- (a) Dry a representative subsample of the material to constant mass in an oven at 105°C to 110°C.
NOTE: The determination of constant mass should be such that after a further period of drying using the selected method, the loss in mass does not exceed 1% of the total losses of moisture.
- (b) Grind the sample to pass a 425 µm sieve. Remove any iron with a magnet.
- (c) Weigh out approximately 5 g of the ground stabilized material and record the mass (m_1).

7.2 Unstabilized material

Prepare unstabilized materials as follows:

- (a) Dry a representative subsample of the material to constant mass in an oven at 105°C to 110°C.
- (b) Grind the sample to pass a 425 µm sieve. Remove any iron with a magnet.
- (c) Weigh out approximately 5 g of the ground unstabilized material and record the mass (m_2).

7.3 Stabilizing agent

Prepare stabilizing agents as follows:

- (a) Dry a representative subsample of the material to constant mass in an oven at 105°C to 110°C.