BS 3247:2011

Incorporating Corrigendum No. 1

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Specification for salt for spreading on highways for winter maintenance

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Date	Text affected
31 July 2013	C1. In Table 1 in the row Marine salt, Coarse, 6.3 mm has been changed to 10 mm. See Foreword.

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Foreword

Publishing information

This British Standard is published by BSI Standards Limited, under licence from The British Standards Institution and came into effect on 28 February 2011. It was prepared by Technical Committee B/510, *Road materials*. A list of organizations represented on this committee can be obtained on request to its secretary.

Supersession

This British Standard supersedes BS 3247:1991, which is withdrawn.

Information about this document

A variety of spreading machines of suitable performance exist and are covered by BS 1622, which also gives advice on minimizing the environmental impact of the use of salt on highways. The purpose of BS 3247 is to specify a material which will enable such machines to give good results, especially after the salt has been stored.

In this revision of this British Standard, one of the main changes concerns the grading requirements for rock salt, with a fine grade which applies to most European salt and the retention of a coarse grade to allow a satisfactory spread on wide carriageways.

Further information on the use and storage of salt for roads can be found in "Well-maintained highways: code of practice for highway maintenance management" [1].

All testing for chemical composition is now carried out on a smaller subsample to decrease dissolution time, and to improve homogeneity, repeatability and reproducibility. Grinding of samples can further reduce dissolution time but is not essential. The drying operation has been revised to remove any hazards associated with industrial methylated spirits. This standard provides Rapid (sulfate as calcium sulfate) and General (total sulfate) methods for soluble sulfate determinations (Annex I and Annex J respectively).

A few hundred tonnes of urea are used per annum on highways in place of salt in order to protect some structures from the corrosive effects of salt. Where urea is used, special arrangements will need to be agreed between the user and the supplier.

The previous edition of this British Standard did not contain, or define, a reproducible test for the chemical analysis of salt. The assessment of the soluble sulfate content of salt can be laborious. A rapid method for determining sulfate content as calcium sulfate content has been introduced to address this (Annex I).

The start and finish of text introduced or altered by Corrigendum No. 1:2013 is indicated in the text by tags C1⟩ ⟨C1.

WARNING. This British Standard calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage.

Use of this document

It has been assumed in the preparation of this British Standard that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its requirements are expressed in sentences in which the principal auxiliary verb is "shall".

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

1 Scope

This British Standard specifies the essential properties of salt for spreading on highways for winter maintenance and includes tests for some of these properties.

Marking requirements are also specified.

The types of salt which are used for winter road maintenance are described in Annex A. Some important considerations affecting the grade of salt to be chosen for particular purposes are given in Annex B. Information on how salt works as a de-icing agent is included in Annex C.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 410-1, *Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth*

BS 5898:1980, *Specification for principles of design and construction of volumetric glassware for laboratory use*

BS EN ISO 10012, *Measurement management systems – Requirements for measurement processes and measuring equipment*

BS EN 932-1:1997, *Tests for general properties of aggregates – Part 1: Methods for sampling*

BS EN 933-1:1997, *Tests for geometrical properties of aggregates – Part 1: Determination of particle size distribution – Sieving method*

3 Terms and definitions

For the purposes of this British Standard, the following terms and definitions apply.

3.1 friable
capable of being easily crumbled between the fingers of the hand and restored to the original grading

4 Storing quality

The salt shall contain the minimum amount of anti-caking additive necessary to ensure that the bulk remains loose after open storage for 18 months in an unprotected stockpile at least 1 m high with sides inclined at the angle of repose of the salt in a loose and usable condition. In these conditions, any non-friable crust formed shall not exceed 75 mm in thickness.

NOTE An important consideration in choosing a site for a stockpile is its potential effect on the environment. Stockpiles should preferably be on well-drained sites and precautions should be taken to ensure that the salt does not constitute a nuisance to human beings or a hazard to animals (including fish) or plant life.

5 Moisture content

When sampled in accordance with Annex D and tested in accordance with Annex E, the moisture content of the salt on delivery shall be no greater than 4% (by mass – m/m).

NOTE Low moisture content salt may be provided following discussion between supplier and purchaser.

6 Grading

When sampled in accordance with Annex D and tested in accordance with Annex E and Annex F, the salt shall conform to Table 1. Salt shall be graded as either coarse or fine according to the results of the sieve analysis (see Annex F).

NOTE 1 Conformity to the requirements for grading ensures the suitability of the salt for spreading for the treatment of snow and ice but does not, of itself, ensure free-running or non-caking properties.

NOTE 2 Spreaders conforming to BS 1622 should be adjusted and calibrated when changing the grade of salt.

The grading for marine salt shall be similar to that specified in Table 1 for rock salt wherever possible and shall conform to the grading for marine salt (coarse).

Table 1 **Grading of salt**

Type and grade of salt		**BS 410-1 test sieve**	**Percentage (m/m) passing test sieve**
Rock Salt	Coarse	10 mm	100
		6.3 mm	75 to 95
		2.36 mm	30 to 70
		300 µm	0 to 20
	Fine	6.3 mm	100
		2.36 mm	30 to 80
		300 µm	0 to 20
Marine salt	Coarse	[C1⟩ 10 mm ⟨C1]	100
		1.18 mm	0 to 80
		150 µm	0 to 10
Vacuum salt	Fine	1.18 mm	100
		150 µm	0 to 30

7 Chemical composition

When sampled in accordance with Annex D and tested in accordance with Annex E, Annex G, Annex H and Annex I or Annex J using reagents of analytical reagent quality, the chemical composition of the salt by dry mass shall be as follows.

a) Soluble chloride compounds [including sodium chloride ($NaCl$), potassium chloride (KCl), magnesium chloride ($MgCl_2$), calcium chloride ($CaCl_2$), etc. expressed as sodium chloride]: not less than 90%.

b) Soluble sulfate compounds [expressed as calcium sulfate ($CaSO_4$)]: not more than 2.5%

c) Material insoluble in water at (20 ±2) °C: not more than 7.5%.

NOTE In addition to the above requirements, no substances should be present in such an amount as to be a hazard to human beings, animals (including fish) or plant life under normal conditions of use of the salt.

8 Marking

The following information shall be marked on the salt packaging or on associated documentation:

a) the words “Salt for highway winter maintenance”;

b) the type of salt (see Annex A);

c) the grade of salt, i.e. coarse or fine;

d) the number and date of this British Standard, i.e. BS 3247:2011 1).

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1) Marking BS 3247:2011 on or in relation to a product represents a manufacturer’s declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is therefore solely the claimant’s responsibility. Such a declaration is not to be confused with third party certification of conformity.

Annex A (informative) Types of salt used for winter road maintenance

A.1 Rock salt

This is salt derived by mining natural salt beds. It contains associated impurities such as calcium sulfate, potassium chloride, magnesium chloride and marl. In appearance it consists of white to reddish brown crystals crushed to various degrees of fineness.

A.2 Vacuum salt

This is a pure salt prepared by the evaporation under vacuum of brine. It is available in fine and coarse grades.

A.3 Marine salt

This is salt derived by solar evaporation of sea water and thus is imported from countries with a warm climate. It contains impurities found in sea water. These are principally calcium sulfate and magnesium chloride, with a small amount of insoluble material. In appearance, this salt consists of off-white irregular crystals of variable size.

Annex B (informative) Particle size and effectiveness

B.1 Salt is more effective when used to stop ice forming if it is spread before the advent of ice or snow because it prevents snow and ice from hard packing. There is no ideal particle size for all uses and the factors governing the choice of salt grading as specified in Clause **6** might vary according to the purpose of the spreading to be carried out and the machine in which it will be used.

B.2 Grading of salt is important as there is a general relationship between it, the height of distribution above the road surface and the width of spread.

B.3 If the salt particles are too small then, with machines conforming to BS 1622 with a height of distribution of 300 mm to 350 mm above the road surface, the particle velocity is often not sufficient to give a very wide spread. Thus, fine salt might not be appropriate for use in one pass on wide roads with these machines.

B.4 Similarly, if large particles are present, too wide a spread can be obtained, particularly if the point of distribution is high. Considerable wastage on the verge can occur on narrow roads and damage might be caused to the road user.

B.5 With fast-moving spreading machines, a considerable slipstream is produced. The effect of this slipstream is greatest on fine particles and least on large particles.

B.6 The rate of solution of the salt on thin ice film is important if quick action is desirable. Fine salt has the advantage of dissolving quickly.

B.7 Where more than 50 mm of snow is present, it should be ploughed. Snow stops salt from the spreading machines reaching its normal width of spread. If any snow is present, only the larger particles will tend to sink through the snow to the road surfaces. If they are large enough, they will reach the road surface before they dissolve and help to prevent hard packing of snow. Traffic will speed up this process, depending on its frequency.

Annex C (informative) How salt works as a de-icing agent

C.1 General

Sodium chloride salt, in common with many other inorganic and organic compounds, dissolves in water and has the effect of both raising the boiling point and depressing the freezing point of that water. The magnitude of the effect is based upon the molecular weight of the compound, the degree of dissociation and the concentration.

C.2 Use of sodium chloride

Sodium chloride is particularly useful for road de-icing as it is relatively soluble in cold water, relatively inexpensive and readily available. Also, as a low molecular weight compound, it has more effect, weight for weight, than other salts of higher molecular weight. Other inorganic salts have been used and also a few organic compounds but none have been able to compete with the use of sodium chloride for cost effectiveness.

C.3 Road traffic action

Road traffic action is an important variable as pressure alone from a vehicle on any ice will cause it to melt but without salt, this will tend to rapidly re-freeze. Any salt particles or solution adhering to the track or surface of the vehicle tyres will reduce this re-freezing and might also be transported to another location by the tyre acting as a secondary distributor.

C.4 Depression of freezing point

C.4.1 General

To demonstrate the principle of freezing point depression calculations, the following assumptions and examples are supplied.

- Degree of dissociation is 75%.
- Cryoscopic constant H_2O is 18.6 °C.
- 1 mm layer of ice/water per m^2.
- Equivalent to 1 000 mL/m^2.
- Salt spreading rate of 10 g/m^2.
- Equivalent to 1g/100 mL.
- Molecular weight (M_W) of NaCl is 58.442 8.

C.4.2 Example 1: 100% NaCl (i.e. pure salt)

58.442 8 g NaCl in 100 mL water reduces freezing point by 18.6 °C.

Therefore, 1 g NaCl in 100 mL water reduces freezing point by

$$\frac{(18.6 \times 1 \times 0.75)^{\circ}\mathrm{C}}{58.442\,8}.$$

Equivalent to: 0.23 °C.

C.4.3 Example 2: 97% NaCl and 3% insoluble content

58.442 8 g NaCl in 100 mL water reduces freezing point by 18.6 °C.

Therefore 1 g NaCl in 100 mL water reduces freezing point by

$$\frac{(18.6 \times 1 \times 0.75)^{\circ}\mathrm{C}}{58.442\,8}.$$

1 g road salt contains 0.97 g NaCl.

Therefore 0.97g NaCl in 100 mL water reduces freezing point by

$$\frac{(18.6 \times 0.97 \times 0.75)^{\circ}\mathrm{C}}{58.442\,8}.$$

Equivalent to: 0.23 °C.

Annex D (normative) Sampling for laboratory testing

D.1 This Annex describes the procedure for sampling salt for laboratory testing.

D.2 The sample to be used for the test (the laboratory sample) shall be taken in accordance with BS EN 932-1:1997, Clause **8.**

D.3 Subdivide the laboratory sample in accordance with BS EN 932-1:1997, Clause **9,** to obtain a representative sample weighing not less than 500 g for drying and subsequent testing.

NOTE Guidance on sampling and testing aggregates is given in BS EN 932-1.

Annex E (normative) Drying and determination of moisture content

E.1 Principle

The sample is dried in an oven to constant mass at a chosen temperature ±2 °C between 110 °C and 150 °C and the moisture content is determined.

NOTE 1 Removing some of the water of crystallisation of gypsum reduces the solubility of calcium sulfate, but it is increased in saline solutions.

NOTE 2 Hot air oven, microwave drying or infra-red moisture determination is much quicker, easier and safer than handling methylated spirits.

E.2 Apparatus

E.2.1 *Balance*, capable of weighing up to 1 200 g, accurate to 0.01 g. The balance shall be regularly calibrated in accordance with BS EN ISO 10012.

E.2.2 *Sample container or weighing bottle,* capable of being heated to a temperature of (150 ±2) °C without changing mass.

E.2.3 *Fan assisted electric oven*, with natural draught, capable of being controlled at a temperature of 110 °C to 150 °C.

NOTE Check this temperature with a thermometer mounted so that the bulb is in close proximity to the weighing bottle used during the test.

E.2.4 *Desiccator*, containing self-indicating silica gel, phosphorus pentoxide or a molecular sieve, of sufficient size to take the sample container (**E.2.2**).

E.3 Procedure

E.3.1 Place the sample obtained in accordance with Annex D in a suitable container (**E.2.2**) which has been pre-weighed.

E.3.2 Weigh and record the mass of the sample and container to the nearest 0.1 g.

E.3.3 Transfer the container to a well controlled and ventilated oven (**E.2.3**), preheated to a chosen temperature ±2 °C between 110 °C and 150 °C and leave until a constant mass to within 0.1% is obtained on successive weighings (**E.3.4** and **E.3.5**).

E.3.4 After drying, allow the sample to cool in the desiccator (**E.2.4**).

E.3.5 Weigh and record the mass of the container and dry sample to the nearest 0.01 g.

E.4 Calculation and expression of result

Calculate the moisture content, c, (in percentage by dry mass) of the sample from the following equation:

$$c = \frac{100(M_1 - M_2)}{M_2 - M_3}$$

where

M_1 is the mass of the sample and container before drying (in g);

M_2 is the mass of the sample and container after drying (in g);

M_3 is the mass of the container (in g).

Express the moisture content of the sample to the nearest 0.1%

NOTE Alternatively, use a calibrated moisture analyser in the target temperature range or the method detailed in BS 7319-2.

Annex F (normative) Grading by sieve analysis

F.1 Apparatus

F.1.1 *Sample divider*, of the size appropriate to the maximum particle size, or alternatively, a flat shovel and a clean flat hard horizontal surface, e.g. a metal tray for use in quartering.

NOTE A suitable divider is the riffle box illustrated in BS EN 932-1:1997, Figure A.8.

F.1.2 *Balance*, capable of weighing up to 2 kg accurate to 0.1 g. The balance shall be calibrated every six months in accordance with BS EN ISO 10012.

F.1.3 *Test sieves*, of the sizes and apertures appropriate to the type and grade of salt being tested (see Table 1), conforming to BS 410-1 and with the appropriate lid and receiver.

F.1.4 *Mechanical sieve shaker* (optional).

F.1.5 *Sieve brush.*

F.2 Procedure

F.2.1 From a dry sample obtained in accordance with **E.3**, obtain a representative subsample weighing not less than 250 g in accordance with BS EN 932-1:1997, Clause **9**.

F.2.2 Weigh and record the mass of the subsample to the nearest 0.1 g.

F.2.3 Carry out a sieve analysis in accordance with BS EN 933-1:1997, **7.2**, using the sieves given in Table 1.

Annex G (normative) Determination of matter insoluble in water

G.1 General

This Annex describes the procedure for determining the amount of matter insoluble in water and the preparation of a stock solution for further chemical analysis (see Annex H and Annex I or Annex J).

G.2 Principle

A subsample of salt is dissolved in distilled or deionized water. The solution is filtered into a volumetric flask with the filter paper washed to remove all traces of chlorides. The undissolved matter on the filter paper is dried to determine the insoluble matter content with the solution in the volumetric flask being used for further chemical analysis (see Annex H and Annex I or Annex J).

NOTE Taking 100 g into 500 mL of water is approaching the solubility limit of sodium chloride in water, i.e. approximately 20%. This means that the solubility time is at a maximum and it is possible for salt to remain undissolved within a shorter time span.

By taking a smaller mass of salt and by increasing the volume of water, the dissolution time can be minimized. 100 g of sample will provide around 5 g of insoluble material which can be resolved on a two place balance to 0.2%.

Further reducing the mass of sample taken will reduce the accuracy of the determination, e.g. 20 g sample will yield around 1 g insoluble which can be resolved to 1% relative.

G.3 Apparatus

G.3.1 *Sample divider*, of the type given in **F.1.1**.

G.3.2 *Balance*, capable of weighing up to 1200 g accurate to 0.01 g. The balance shall be regularly calibrated in accordance with BS EN ISO 10012.

G.3.3 *Sample container*, capable of being heated to a temperature of (105 ±5) °C.

G.3.4 *Desiccator*, containing self-indicating silica gel.

G.3.5 *Glass beaker*, of 1000 mL capacity.

G.3.6 *Wash bottle*, containing distilled or deionized water.

G.3.7 *Magnetic stirrer*, with a glass- or plastics-coated magnetic follower bar.

G.3.8 *Filter funnel.*

G.3.9 *Filter paper*, medium grade, of the appropriate size.

G.3.10 *Volumetric flask*, of 1 000 mL capacity, and conforming to grade B of BS 5898:1980.

G.3.11 *Well ventilated oven*, capable of maintaining a temperature of (105 ±5) °C.

G.4 Reagents

G.4.1 Silver nitrate solution, ($AgNO_3$) of concentration 0.1 mol/L.

G.5 Procedure

G.5.1 From a dry sample obtained in accordance with **E.3**, obtain a representative subsample weighing not less than 20 g in accordance with BS EN 932-1:1997, Clause **9**.

G.5.2 Weigh a subsample of (20 ±0.01) g into a glass beaker (**G.3.5**) and add 500 mL of distilled or deionized water (**G.3.6**).

G.5.3 Stir for (10 ±1) min at a temperature of (20 ±2) °C using a magnetic stirrer (**G.3.7**).

G.5.4 Filter the solution through a dry pre-weighed medium grade filter paper (**G.3.9**) placed in the filter funnel (**G.3.8**) in turn placed in the neck of the volumetric flask (**G.3.10**), rinsing with distilled or deionized water to remove all traces of chlorides. Three discrete rinses (where the filter runs dry each time) will usually be sufficient to remove all traces of chloride. If necessary, check for the absence of chlorides by allowing a drop of filtrate to pass through the filter paper into a small quantity of silver nitrate solution (which will turn milky if chlorides are present).

G.5.5 Take the rinsed filter paper and insoluble material in the sample container (**G.3.3**) and dry in a well ventilated oven (**G.3.11**) at (105 ±5) °C to a constant mass within 0.1% and allow to cool in a desiccator (**G.3.4**).

G.5.6 Add distilled or deionized water to the volumetric flask (**G.3.10**) and make up to 1 000 mL for further chemical analysis.

G.5.7 Weigh and record the mass of the dry filter paper and insoluble material to the nearest 0.01 g.

G.6 Calculation and expression of result

Calculate the matter insoluble in water, I, (in percentage by dry mass) of the sample from the following equation:

$$I = M_4 - M_5$$

where

M_4 is the mass of the filter paper and dried sample (in g);

M_5 is the mass of the filter paper (in g).

Express the matter insoluble in water to the nearest 0.1%.

Annex H (normative) Determination of chloride content

H.1 General

This Annex describes the procedure for determining the chloride content of the salt sample.

NOTE All chlorides present are expressed in terms of, and reported as, the sodium chloride content.

H.2 Principle

A solution of the salt is titrated with a solution of silver nitrate using potassium chromate as an indicator.

H.3 Apparatus

H.3.1 *Pipette*, of 50 mL capacity, and conforming to grade B of BS 5898:1980.

H.3.2 *Volumetric flask*, of 100 mL capacity, and conforming to grade B of BS 5898:1980.

H.3.3 *Wash bottle*, containing distilled or deionized water.

H.3.4 *Pipette*, of 25 mL capacity and conforming to grade B of BS 5898:1980.

H.3.5 *Two conical flasks*, of 250 mL capacity.

H.3.6 *Pipette*, of 1 mL capacity, and conforming to grade B of BS 5898:1980.

H.3.7 *Burette*, of 50 mL capacity, and conforming to grade B of BS 5898:1980.

H.4 Reagents

H.4.1 General

Reagents of recognized analytical quality and only distilled water or deionized water shall be used.

H.4.2 Required reagents

H.4.2.1 Potassium chromate (K_2CrO_4) aqueous solution.

H.4.2.2 Silver nitrate solution, ($AgNO_3$) of concentration 0.100 mol/L, prepared by drying about 20 g of silver nitrate at a temperature of (105 ±5) °C for 1 h to 2 h and allowing to cool in a desiccator. Weigh out 16.987 g of the dried silver nitrate and dissolve in distilled water, diluting to 1 000 mL in a volumetric flask.

H.4.2.3 Alternatively, use an ampoule of silver nitrate solution made up with distilled water to 1 000 mL in a volumetric flask to give 0.100 mol/L solution or an already prepared silver nitrate solution of 0.100 mol/L analytical reagent (AR) grade.

H.4.2.4 Store the solution in an amber colour glass reagent bottle and protect from prolonged exposure to sunlight.

H.4.2.5 The shelf life of the silver nitrate solution shall be 3 months.

H.4.2.6 Stock solution for chemical analysis (**G.5.6**).

H.5 Procedure

H.5.1 Take 50 mL of the stock solution (**H.4.2.3**) by pipette (**H.3.1**) diluting to 100 mL in a volumetric flask (**H.3.3** and **H.3.2**). Take two 25 mL aliquots of the diluted solution by pipette (**H.3.4**) and transfer to conical flasks (**H.3.5**). Add 1 mL by pipette (**H.3.6**) of a 5% aqueous solution of potassium chromate (**H.4.2.1**) as an indicator to each aliquot. Titrate with 0.100 mol/L silver nitrate solution (**H.4.2.2**) using a burette (**H.3.7**) until the colour changes permanently from yellow to red-brown.

H.5.2 Note the volume of silver nitrate solution added to each of the aliquots. If the duplicate titrations vary by more than 0.2 mL, repeat the test using two new aliquots.

H.6 Calculation and expression of result

Calculate the chloride content expressed as sodium chloride, S, (in percentage by dry mass) from the following equation:

$$S = 2.338\ V$$

where

V is the mean volume of 0.100 mol/L silver nitrate solution added to the aliquots of diluted solution.

Express the sodium chloride content to the nearest 0.1%.

NOTE Alternatively, it is possible to use the method detailed in BS 7319-1.

Annex I (normative) Rapid determination of soluble sulfate compounds

I.1 General

This Annex describes the procedure for determining the amount of soluble calcium compounds present in the salt sample.

NOTE All soluble calcium compounds are expressed in terms of, and reported as, the calcium sulfate content.

I.2 Principle (Rapid method)

It is assumed that all soluble sulfate compounds will be associated with calcium. Soluble calcium is determined titrimetrically with ethylenediaminetetraacetic acid (EDTA) solution.

NOTE Sulfate determination is relatively time consuming and semi-skilled work involving the preparation and handling of toxic compounds (barium chloride) whereas dissolved calcium determinations are relatively simple and less hazardous. Calcium sulfate is relatively easily dissolved in saline solution and if a quantity less than the solubility limit is taken, it can be assumed that it is completely soluble. If soluble sulfate salts other than calcium sulfate are likely to be present, e.g. magnesium sulfate, the General method (Annex J) will provide a more accurate result. The General method can be used to validate the Rapid method results periodically.

I.3 Apparatus

I.3.1 *Pipette*, of 25 mL capacity, and conforming to grade B of BS 5898:1980.

I.3.2 *Beakers*, of 400 mL capacity.

I.3.3 *Burette*, of 50 mL capacity and conforming to grade B of BS 5898:1980.

I.3.4 *Scoop* of 0.5 g capacity.

I.3.5 *Pipette*, of 5 mL capacity, and conforming to grade B of BS 5898:1980.

I.3.6 *Wash bottle* containing distilled or deionized water.

I.4 Reagents

I.4.1 Hydroxynaphtol blue di-sodium salt indicator powder or tablets.

I.4.2 Standard calcium solution. Weigh 2.5 gm of analytical reagent (AR) grade calcium carbonate into a 600 mL beaker. Add (10 ±1) mL distilled water and 25.0 mL 1 N hydrochloric acid. Warm on a hot plate to promote solution and to expel any carbon dioxide in the solution. Cool and transfer with washings into a 1 000 mL volumetric flask. Make up to the mark with distilled water and shake well. This solution is such that 1 mL equals 1 mg calcium.

I.4.3 Stock solution for chemical analysis (**G.5.6**).

I.4.4 2 mol Sodium hydroxide.

I.4.5 0.02 mol EDTA.

I.5 Standardization procedure

I.5.1 Stock EDTA solution

Pipette 25 mL of standard calcium solution (**I.4.2**) into a clean 400 mL beaker (**I.3.2**). Add (25 ±2) mL distilled water, 5 mL 2 mol sodium hydroxide (**I.4.4**) and a scoop of indicator (**I.4.1**). Titrate with EDTA against a white background until the end point is observed; i.e. a colour change from red to blue.

Once the titration is complete, read off the titre result from the burette. This result is equivalent to 25 mg of Ca. Therefore, 1 mL of EDTA = 25 mg Ca divided by the actual titre. Calculate the volume of water or weight of EDTA required to adjust the strength of the EDTA solution to 1 mL EDTA = 1.648 mg Ca.

I.5.2 Dilute EDTA solution

Pipette 5 mL standard calcium solution (**I.4.2**) into a clean 400 mL beaker. Add 95 mL distilled water, 10 mL 2 mol sodium hydroxide (**I.4.4**) and a scoop of indicator (**I.4.1**). Titrate the standard calcium solution with EDTA against a white background until the end point is observed. The titre will normally be around 30 mL, therefore 1 mL of EDTA is equivalent to 5 mg divided by the actual titre (mL).

This will give a standardization factor which can be then be used to calculate calcium and magnesium parts per million (ppm) (see **I.6**).

I.5.3 Deionized water background level

If deionized water is used, at the start of each series of determinations, check the background level of calcium. Measure 100 mL deionized water into a clean 400 mL beaker, add 10 mL 2 mol sodium hydroxide

(**I.4.4**) and a scoop of indicator (**I.4.1**). Titrate with the dilute EDTA solution (**I.5.2**) against a white background until the end point is reached. Call this titre "T_{bl}".

I.5.4 Procedure

Measure 100 mL of the stock solution (**G.5.6**) into a clean 400 mL beaker, add 10 mL 2 mol sodium hydroxide (**I.4.4**) and a scoop of indicator (**I.4.1**) and titrate with the dilute EDTA solution (**I.5.2**) until the end point is observed. Call this titre "T_S".

I.6 Calculation of result

Calculate the percentage of Ca as follows.

- 100 mL stock solution contains 20 mg of sample.
- Therefore, a 20 mg sample contains ($T_S - T_{bl}$) × standardization factor (**I.5.2**)
- Therefore, the percentage of Ca = ($T_S - T_{bl}$) × standardization factor.

Convert the percentage of Ca to percentage of $CaSO_4$ using the following equation:

$$\% \ CaSO_4 = \% \ Ca \times 16.983.$$

Annex J (normative) General determination of soluble sulfate compounds

J.1 General

This Annex describes the procedure for determining the amount of soluble sulfate compounds present in the salt sample.

NOTE All soluble sulfate compounds are expressed in terms of, and reported as, the calcium sulfate content.

J.2 Principle

Soluble sulphates are determined gravimetrically by precipitation with barium chloride solution.

J.3 Apparatus

J.3.1 *Pipette*, of 50 mL capacity, conforming to grade B of BS 5898:1980.

J.3.2 *Two glass beakers*, heat-resistant, of 500 mL capacity.

J.3.3 *Wash bottle*, containing distilled or deionized water.

J.3.4 *Paper indicator strips*, with a range of pH 6 to pH 8.

*NOTE Alternatively, methyl orange indicator solution (**J.4.2.2**) may be used.*

J.3.5 *Pipette*, of 1 mL capacity, conforming to grade B of BS 5898:1980.

J.3.6 *Hot plate*, thermostatically controlled, capable of heating the solutions in **J.4** to near boiling.

J.3.7 *Fume cupboard.*

J.3.8 *Ignition crucible*, with porous porcelain filter.

J.3.9 *Well ventilated oven*, capable of maintaining a temperature of (105 ±5) °C.

J.3.10 *Desiccator*, containing self-indicating silica gel.

J.3.11 *Balance*, capable of weighing up to 100 g accurate to 0.001 g. The balance shall be regularly calibrated in accordance with BS EN ISO 10012.

J.4 Reagents

J.4.1 General

Reagents of recognized analytical quality and only distilled or deionized water shall be used.

J.4.2 Required reagents

J.4.2.1 Hydrochloric acid, (HCl) 5 mol/L, prepared by diluting 410 mL of hydrochloric acid density 1.18 mL to 1 000 mL.

CAUTION. Always add acid to water (on no account add water to acid) and always use acids in a fume cupboard.

J.4.2.2 Methyl orange indicator solution.

*NOTE This may be used in place of paper indicator strips (**J.3.4**).*

J.4.2.3 Barium chloride solution, ($BaCl_2$), approximately 5% (m/m) prepared by dissolving 5 g of barium chloride in distilled or deionized water and making up to 100 mL in a volumetric flask.

J.4.2.4 Silver nitrate solution, ($AgNO_3$) of concentration 0.1 mol/L.

J.4.2.5 Stock solution for chemical analysis (**G.5.6**).

J.5 Procedure

J.5.1 Take duplicate 50 mL portions of the prepared stock solution (**J.4.2.5**) by pipette (**J.3.1**) and transfer to glass beakers (**J.3.2**).

J.5.2 Dilute to 100 mL with distilled or deionized water and neutralize with 5 mol/L hydrochloric acid (**J.4.2.1**) using paper indicator strips (**J.3.4**) or a few drops of methyl orange indicator (**J.4.2.2**) to determine the amount to be added. Add 1 mL of acid in excess using a pipette (**J.3.5**).

J.5.3 Heat to near boiling on a hot plate (**J.3.6**) and add warm 5% (m/m) barium chloride solution (**J.4.2.3**) dropwise, whilst stirring, until precipitate ceases to form. Add 1 mL of the barium chloride solution in excess, allow to cool to room temperature, and filter through a previously ignited and weighed porous porcelain filter and ignition crucible (**J.3.8**). Wash the crucible thoroughly with hot distilled or deionized water until no chloride can be detected in the washings when they are tested with silver nitrate solution (**J.4.2.4**).

J.5.4 Dry the crucibles at (105 ±5) °C for approximately 1 hr in a well ventilated oven (**J.3.9**).

J.5.5 Allow the crucibles to cool in a desiccator (**J.3.10**) and weigh and record the mass of the crucibles and precipitate to the nearest 0.001 g.

J.5.6 Calculate the soluble sulfate content in accordance with **J.6**.

J.6 Calculation and expression of result

J.6.1 Calculate the soluble sulfate content, X, (in percentage by dry mass) from the following equation

$$X = 58.3\ (M_6 - M_7)$$

where

M_6 is the mass of the crucible and precipitate (in g);

M_7 is the mass of the crucible (in g).

J.6.2 Calculate the result for both test portions to the nearest 0.1%. If the individual results differ by more than 0.1%, repeat the test.

Bibliography

Standards publications

For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

BS 1622, *Specification for spreaders for winter maintenance*

BS 7319-1, *Analysis of sodium chloride for industrial use – Part 1: Method for determination of sodium chloride content*

BS 7319-2, *Analysis of sodium chloride for industrial use – Part 2: Method for determination of moisture content*

Other publications

[1] ROADS LIAISON GROUP. *Well-maintained highways: code of practice for highway maintenance management*. The Stationery Office, 2005. ISBN 978 0 11552 643 5 (2)

Further reading

HIGHWAYS AGENCY. *Routine and Winter Service Code*. Highways Agency, 2009. (Available from www.standardsforhighways.co.uk.)

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(2) Referred to in the foreword only.

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