

Determination of Water-soluble Magnesium Salts (Epsom Salts)

1 Scope

In the case of leathers containing water-soluble calcium or phosphate, a modification (described in 9) is necessary, but otherwise this method is applicable to all types of leather.

2 Definition

For the purposes of this method the following definition applies.

Epsom salt content of leather. The quantity of magnesium salts calculated as magnesium sulphate, $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (Epsom salts), obtained by extraction with water under the conditions described in this method.

3 Principle

In the absence of any interfering calcium and phosphate ions, magnesium is determined in the water-soluble inorganic matter by complexometric titration.

4 Reagents

The following reagents are required.

- (a) Hydrochloric acid reagent solution, 2N.
- (b) Ammonia reagent solution, 2N.
- (c) Sodium hydroxide reagent solution, 2N.
- (d) Buffer solution, pH 10.5, containing 54 g of ammonium chloride and 350 ml of diluted ammonia solution (1 volume of ammonia solution ($d = 0.880$) and 3 volumes of water) per litre.
- (e) disodium dihydrogen ethylenediamine-*NNN'N'*-tetra-acetate (EDTA), 0.01M, standard volumetric solution.

Prepare a standard EDTA, 0.01M, solution by one of the following methods.

- (1) Using EDTA as a volumetric standard. Dry the EDTA at 80 °C for several hours. Accurately weigh the required quantity of 3.722 g of EDTA; dissolve in water and make up to one litre in a volumetric flask, at 20 °C.
- (2) Standardising the EDTA with zinc. Dissolve 0.16 g of pure zinc, weighed accurately, in 200 ml of 2N hydrochloric acid, heating on a water-bath and taking precautions to prevent loss of spray. When solution is complete, transfer to a 250 ml volumetric flask, cool to 20 °C and dilute to the mark.
Pipette 25.0 ml of the zinc solution into a conical flask, dilute with 50 ml of water and add 30 ml of 2N ammonia solution. Add Mordant black 11 indicator mixture until clear red then titrate with a 0.01M EDTA solution until a pure blue colour is obtained (without any red tinge).
1 ml of EDTA solution, exactly 0.01M, is equivalent to 0.6538 mg of zinc. Calculate the factor of the prepared solution as follows.

$$\text{Factor} = \frac{M_z}{6.538b}$$

Where M_z is the mass, in milligrams, of zinc,
and b is the volume, in millilitres, of EDTA, 0.01M, used in the standardisation titration.

NOTE. If closed borosilicate glass or polyethylene vessels are used for storage, the EDTA solution can be kept for a practically unlimited time.

- (f) Mordant black 11* indicator mixture, consisting of 1 part by mass of Mordant black 11 well mixed with 500 parts by mass of sodium chloride (solid). This mixture will keep indefinitely.
- (g) Methyl orange, indicator solution, 0.2%, aqueous.

ammonia nearly disappears. Filter the solution while warm and wash out the beaker and filter with hot water. If calcium is absent, proceed as described in 6.2; if calcium is present, proceed as described in 9.2.

9.2

Elimination of calcium ions

Dissolve the ash in a little 2N hydrochloric acid (if phosphate is absent), or use the filtrate from 9.1. Add 3 ml of ammonium chloride solution (100 g/l) and boil gently. Add drop by drop to the boiling solution, 0.5 ml of ammonium oxalate solution (50 g/l) and follow this by the addition, drop by drop with stirring, of 7.5 ml of dilute ammonia solution (1 volume of ammonia solution ($d = 0.880$) and 2 volumes of water). Allow to stand without further heating for at least 1 hour. Filter and transfer the filtrate and washings to a 500 ml conical flask. Boil the solution for a few minutes and then cool.

Continue as described in 6.2.

10 Test report

The report shall include:

- (a) the results obtained, to 1 decimal place;
- (b) a reference to the method used;
- (c) details of any special circumstances which may have affected the results;
- (d) identification details of the sample.

* Mordant black 11 is also known as 'Erichrome black T' and 'Solochrome black'.

Apparatus

laboratory apparatus is required and, in particular, the following.

- (a) Burette, 50 ml.
- (b) Conical flask, 500 ml.
- (c) Measuring cylinders, 50 ml and 250 ml.
- (d) Thermometer, range 0-100 °C with 1 °C graduation.

Procedure

(calcium and phosphate being absent)

Proceeding as described in SLC 5, obtain the water-soluble inorganic matter and dissolve this, in the crucible, with a little 2N hydrochloric acid, warming gently. Transfer the solution into the 500 ml conical flask, rinsing the crucible into the flask several times with a little 2N hydrochloric acid and water. Neutralise the solution, using methyl orange as indicator, with 2N ammonia solution or 2N sodium hydroxide solution and boil for a few minutes.

Dilute the solution in the conical flask with 150 ml of water, add 20 ml of the buffer solution, adjust to 50 °C and add Mordant black 11 indicator mixture until the solution becomes a clear red. Titrate with 0.01M EDTA solution until the colour changes to pure blue (with no red tinge).

Expression of results

Calculate the following percentage.

Epsom salts ($MgSO_4 \cdot 7H_2O$), percentage by mass, in the leather =

$$\frac{T \times 0.002465 \times 10 \times 100}{M_s}$$

where T is the volume, in millilitres, of EDTA, 0.01M, solution used for titration

and M_s is the mass, in grams, of the original sample of leather.

The calculation is based on the following.

1 ml of EDTA, 0.01M, solution = 0.000403 g of MgO
= 0.002465 g of $MgSO_4 \cdot 7H_2O$.

Repeatability

The results of a duplicate determination calculated on the original mass of leather taken shall not differ by more than 0.2% when expressed as Epsom salts ($MgSO_4 \cdot 7H_2O$), or by more than 0.03% when expressed as magnesium oxide (MgO).

Modified procedures for determination of Epsom salts ($MgSO_4 \cdot 7H_2O$) in the presence of phosphate or calcium ions

In the determination of Epsom salts using the sulphated ash of the water-soluble matter obtained in accordance with SLC 5, it is necessary first to eliminate interfering phosphate or calcium ions (or both together) if their presence has been indicated by preliminary qualitative tests.

Elimination of phosphate ions

Mix the ash in the crucible with 5 ml of 10% (m/m) hydrochloric acid and dissolve under slight heat. Rinse the solution into a 250 ml glass beaker and dilute with water to approximately 50 ml. Then add 3 ml of ammonium chloride solution (100 g/l), a few drops of concentrated nitric acid and a few drops of ferric chloride solution (100 g $FeCl_3$ /l). Make the solution alkaline with ammonia and boil. The precipitate which forms should be quite brown in colour; if it is not, then acidify again with hydrochloric acid, add a few drops of ferric chloride solution and make alkaline again with ammonia.

Repeat this procedure until the formation of a distinctly brown-coloured precipitate occurs. Boil the solution until the smell of