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Sodium hydrogen carbonate for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

Austria	Hungary	Poland
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Czechoslovakia	Israel	Switzerland
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Germany	New Zealand	

No Member Body expressed disapproval of the document.

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Sodium hydrogen carbonate for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

1 SCOPE

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of the iron content of sodium hydrogen carbonate for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products having iron contents equal to or greater than 0,1 mg/kg.

3 PRINCIPLE

Reduction of the trivalent iron by hydroxylammonium chloride, followed by the formation of a bivalent iron/1,10-phenanthroline complex in a buffered system. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

4 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N.

4.2 Ammonia solution, ρ approximately 0,91 g/ml, about 25 % (*m/m*) NH_3 solution or approximately 13 N, with a maximum iron content of 0,2 mg/kg.

4.3 Hydroxylammonium chloride ($NH_2OH \cdot HCl$), 10 g/l solution.

4.4 Buffer solution, pH 4,9.

Dissolve 272 g of sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$) in about 500 ml of water. Add 240 ml of glacial acetic acid (ρ approximately 1,05 g/ml, 99 to 100 % (*m/m*) solution or approximately 17,4 N) to the solution and dilute to 1 000 ml.

4.5 Bromine water, saturated at room temperature.

4.6 1,10-Phenanthroline hydrochloride monohydrate ($C_{12}H_8N_2 \cdot HCl \cdot H_2O$), 2,5 g/l solution.

This product may be replaced by 1,10-phenanthroline monohydrate ($C_{12}H_8N_2 \cdot H_2O$).

4.7 Iron standard solution, corresponding to 0,200 g of Fe per litre.

Dissolve 1,404 3 g of ammonium iron(II) sulphate hexahydrate [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$], weighed to the nearest 0,0001 g, in 200 ml of water. Add 20 ml of sulphuric acid, ρ approximately 1,84 g/ml, cool to room temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

4.8 Iron standard solution, corresponding to 0,010 g of Fe per litre.

Transfer 25,0 ml of the iron standard solution (4.7) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use.

1 ml of this standard solution contains 0,010 mg of Fe.

4.9 Methyl orange, 0,5 g/l solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Spectrophotometer, or

5.2 Photoelectric absorptiometer, fitted with filters giving maximum transmission between 500 and 520 nm.

6 PROCEDURE

6.1 Test portion

Weigh, to the nearest 0,1 g, 100 g of the test sample.

6.2 Blank test

Pour 25 ml of water and a volume of the hydrochloric acid solution (4.1) identical to that used to neutralize the test portion (see 6.4.1) into a 600 ml beaker. Add 75 ml of the ammonia solution (4.2), 5 drops of the methyl orange solution (4.9) and then neutralize with the ammonia solution (4.2). Add the hydrochloric acid solution (4.1) drop by drop until the colour changes to red, and then an excess of 2 ml of this acid. Add 5 ml of the bromine water (4.5) to remove the colour of the indicator, boil for 5 min,