

British Standard Methods of

Sampling and test for glycerol

Part 16. Determination of iron content

Méthodes d'échantillonnage d'essais pour le glycérol
Partie 16. Dosage du fer

Verfahren für die Probenahme und Prüfung von Glycerin
Teil 16. Bestimmung des Eisengehaltes

NOTE. It is recommended that this Part of BS 5711 be read in conjunction with the general information given in BS 5711 : Part 0 'General introduction', issued separately.

1. Scope and field of application

This British Standard describes a photometric method for the determination of the iron content of technical glycerol.

2. References

The titles of the standards publications referred to in this standard are listed overleaf.

3. Principle

Dissolution of sulphated ash residues, formation of a thioglycollate complex and photometric measurement of the absorbance of the complex.

4. Reagents

The reagents used shall be of recognized analytical grade. Distilled water complying with the requirements of BS 3978 or water of at least equal purity shall be used.

4.1 Potassium hydrogen sulphate, fused.

4.2 Thioglycollic acid, 10 % (m/m) reagent solution. Dilute thioglycollic acid, 90 % (m/m), 1 + 9.

WARNING. Take great care not to allow concentrated thioglycollic acid to come into contact with the skin as it causes painful wounds.

4.3 Trisodium citrate dihydrate, 300 g/l reagent solution.

4.4 Hydrochloric acid, concentrated, 36 % (m/m), 11N.

4.5 Ammonia, 7N reagent solution. Dilute concentrated ammonia solution, 35 % (m/m), (18N), 1 + 1.5.

4.6 Standard iron solutions:

Solution A. Dissolve 0.7023 g of pure ammonium iron (II) sulphate hexahydrate ($\text{FeSO}_4 \cdot (\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}$) in water containing 10 ml of sulphuric acid solution,

50 % (m/m), in a 1000 ml one-mark volumetric flask, mix well and dilute to the mark with water.

1 ml $\equiv$ 100 μg Fe.

Solution B. Transfer 25.0 ml of solution A to a 250 ml one-mark volumetric flask and dilute to the mark with water.

1 ml $\equiv$ 10 μg Fe.

5. Apparatus

5.1 Volumetric flasks, one-mark, 50 ml capacity, with ground glass neck, complying with the requirements of BS 1792.

5.2 Spectrophotometer, adjusted to read at 530 nm or a *photoelectric absorptiometer* with a green filter transmitting from about 500 nm to 545 nm with maximum transmission at 530 nm. Cells, all glass, of such a size that the absorbance of the solution under test lies between 0.2 and 0.8.

5.3 Nessler cylinders, 50 ml capacity, complying with the requirements of BS 612.

6. Sampling

Prepare the laboratory sample as described in Part 2 of this British Standard.

7. Procedure

7.1 Preparation of the calibration curve. Dissolve 2 g of potassium hydrogen sulphate in 10 ml of water and 5 ml of the hydrochloric acid solution (4.4). Add amounts of the standard iron solution (4.6) equivalent to 0, 25, 50, 75, 100 and 125 μg of iron (Fe), and proceed as described for the test solution in 7.2. Measure the absorbances in turn against