BS 5711 : Part 14 : 1979

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British Standard Methods of

Sampling and test for glycerol

Part 14. Limit test for organic chloride

Méthodes d'échantillonnage et d'essais pour le glycérol
Partie 14. Essai limite de chlorure organique

Verfahren für die Probenahme und Prüfung von Glyzerin
Teil 14. Grenzprüfung für organisches Chlorid

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 turbidimetric limit test for organic chloride in chemically pure glycerol.

2. References

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

3. Principle

Hydrolysis of organic chloride in a test portion, development of turbidity with acidified silver nitrate solution and visual comparison of test and control solutions.

4. Reagents

The reagents 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 *Morpholine*

4.2 *Nitric acid,* concentrated, 70 % (*m/m*) (16N).

4.3 *Silver nitrate,* 0.01N standard volumetric solution.

4.4 *Hydrochloric acid,* 0.01N standard volumetric solution.

5. Apparatus

5.1 *Nessler cylinders*, 50 ml, complying with the requirements of BS 612.

5.2 *Flasks*, round bottom, 100 ml, complying with the requirements of BS 2734 and fitted with ground glass necks complying with the requirements of BS 572.

5.3 *Condenser,* water-cooled, complying with the requirements of BS 3787, with a conical joint to fit the flask (**5.2**).

6. Sampling

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

7. Procedure

Weigh, to the nearest 0.01 g, 5 g of the sample into a dry flask (**5.2**), add 15 ml of the morpholine (**4.1**), fit the reflux condenser (**5.3**) and boil the mixture gently for 3 h in a fume cupboard. Cool, rinse the condenser with 10 ml of water, collecting the rinsings in the flask, remove the condenser and cautiously acidify the solution with the nitric acid (**4.2**). Cool, transfer the mixture to a Nessler cylinder (**5.1**), add 5 ml of the silver nitrate solution (**4.3**), dilute to the mark with water and mix thoroughly. To a second Nessler cylinder add 10 ml of water and 15 ml of the morpholine (**4.1**), cautiously acidify with the nitric acid, cool, add 4 ml of the hydrochloric acid solution (**4.4**) and 5 ml of the silver nitrate solution (**4.3**), dilute to the mark and mix. This is the matching solution corresponding to 28 mg/kg of chloride.

Compare the turbidity of the solutions visually.

NOTE. It is advisable that all tests should be carried out in duplicate.

8. Test report

8.1 Report whether the test solution is more or less turbid than the matching solution.

8.2 The test report shall include the following particulars:

(a) the result and method of expression used;

(b) a reference to this British Standard;

(c) any unusual features noticed during the test;

(d) any operation not included in this British Standard or regarded as optional.