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British Standard Methods of

Sampling and test for glycerol

Part 11. Determination of propane-1,3-diol content: gas chromatographic method

Méthodes d'échantillonnage et d'essais pour le glycérol
Partie 11. Dosage du propane-1,3-diol, méthode chromatographie gazeuse

Verfahren für die Probenahme und Prüfung für Glyzerin
Teil 11. Bestimmung des Trimethylenglykolgehaltes — Gaschromatographiemethode

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.

0. Introduction

As propane-1,3-diol and glycerol differ in polarity and have widely different vapour pressures at a given temperature, they are easily and directly separated by gas chromatography.

A column packing which minimizes tailing is necessary and, for this purpose, either a polar stationary phase on an inert support or a porous polymer packing may be used.

1. Scope and field of application

This British Standard describes a method for the determination of propane-1,3-diol in soap lye crude and hydrolyser crude glycerols.

2. References

The titles of the standards publications referred to in this standard are listed on the inside back cover.

3. Principle

Separation of propane-1,3-diol and glycerol by gas chromatography.

4. Materials

4.1 Carrier and auxiliary gases

4.1.1 *Carrier gas.* Nitrogen, argon and helium are all suitable.

4.1.2 *Auxiliary gases.* Hydrogen and air are required for use with a flame ionization detector.

4.2 Reagents. The reagents used shall be of a recognized analytical grade.

4.2.1 *Ethanol*, absolute, complying with the requirements of BS 507 or *industrial methylated spirits*, complying with the requirements of BS 3591.

4.2.2 *Dodecan-1-ol*, or *diethylene glycol*, internal standard.

4.2.3 *Propane-1,3-diol*, calibration standard.

4.3 Column packing materials

4.3.1 *Stationary phase.* Polyethylene glycol polymers of average molar mass 20 000 or similar materials are suitable*. Polyesters such as polyethyleneglycol adipate (PEGA), have been used but are less satisfactory.

4.3.2 *Support*, diatomaceous earth, particle size 85 μm to 106 μm.

NOTE. A porous polymer may be used as an alternative to a stationary phase on a support. Chromosorb 102, for example, has been found suitable.

5. Apparatus

5.1 *Gas chromatographic apparatus.* A flame ionization detector is preferred. Care should be taken that, under the conditions of the analysis, both the detector and the amplifier are operating within their linear range.

5.1.1 *Injection heater.* A method of heating the injection zone to a temperature above that of the column shall be available. It is recommended that a glass liner should be used.

5.2 *Micro-pipette* or *micro syringe*, complying with the requirements of BS 1428 : Part D5.

5.3 *Column and packing.*

5.3.1 *Column.* The column may be of glass or metal. For

*Carbowax 20 M and Carbowax 20 M/TPA have been found suitable.

rapid analyses, a column 25 cm to 30 cm long is suitable but, for the maintenance of adequate resolution over an extended period, a column length of at least 1 m is recommended.

5.3.2 *Packing.* Either a packing consisting of 9 parts by mass of the support (**4.3.2**) and 1 part by mass of the stationary phase (**4.3.1**) or the porous polymer (see note to **4.3.2**).

NOTE. The porous polymer is more suitable for repeated measurements on soap lye glycerol or other glycerols with high water contents.

5.3.3 *Preparation of the packed column.* Any suitable method of packing may be used. The final column should have an efficiency of about 200 theoretical plates, measured using the internal standard at the temperature of operation.

Test the column with an approximately 0.5 % (*m/m*) solution of propane-1,3-diol in ethanol and ensure that, when using a test portion size and amplifier attenuation sufficient to give a propane-1,3-diol peak height of about 50 % full scale, there is no overlap between the ethanol peak and the propane-1,3-diol peak.

6. Sampling

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

7. Procedure

It is not practicable to provide complete details of the operating procedure for every type of instrument which might be used, but the following generalizations may be made.

7.1 Injector temperature: 175 °C to 250 °C. The temperature should be slightly higher than the column temperature.

7.2 Column temperature: 120 °C to 200 °C. Higher temperatures give shorter analysis times, but may result in the solvent or internal standard peak overlapping the propane-1,3-diol peak.

7.3 Calibration. Carry out a preliminary experiment to determine the constant of proportionality, K, between the internal standard and the propane-1,3-diol. Calculate the value of K from the formula

$$\frac{m_i \times A_e}{m_e \times A_i}$$

where

A_i is the height or area of the propan-1,3-diol peak (mm or mm^2)

A_e is the height or area of the internal standard peak (mm or mm^2)

m_i is the mass of propane-1,3-diol in the mixture (g)

m_e is the mass of internal standard in the mixture (g).

7.4 Test

7.4.1 *Preparation of the test portion.* Weigh, to the nearest 0.001 g, about 10 g of the sample into a suitable container. Add 0.2 to 0.5 g of the internal standard (**4.2.2**), and re-weigh to the nearest 0.001 g. The internal standard should be chosen so that the internal standard and propane-1,3-diol peaks are of comparable size on the chromatogram. If necessary, the quantities taken may be varied from those recommended above.

Add an equal volume of the ethanol or industrial methylated spirits (**4.2.1**) and mix until homogeneous.

7.4.2 *Sample injection.* Inject a suitable test portion (e.g. 0.1 µl) with the pipette (**5.2**) and adjust the attenuation so that the heights of the internal standard and the propane-1,3-diol peaks lie between 20 % and 80 % of full scale. It is not necessary to adjust the attenuation to keep the glycerol peak on scale.

7.4.3 *Typical relative retention volumes.* As a guide to the interpretation of the chromatogram, the following relative retention volumes have been determined.

	PEGA 170 °C	Carbowax 20 M 200 °C	Chromosorb 102 250 °C
Propane-1,3-diol	0.15	0.15	0.47
Dodecan-1-ol	0.28	0.29	–
Diethylene glycol	0.34	–	0.25
Glycerol	1.00	1.00	1.00

7.4.4 *Measurement.* Measure the height or area of the propane-1,3-diol and internal standard peaks by the method used in the calculation of the response factor.

8. Calculation

Calculate the propane-1,3-diol content, as a percentage by mass, from the formula

$$\frac{100 \times m_e \times A_i \times K}{m \times A_E}$$

where:

m_e is the mass of internal standard added to the test portion (g)

m is the mass of the test portion taken (g)

A_i is the height or area of the propane-1,3-diol peak (mm or mm^2)

A_E is the height or area of the internal standard peak (mm or mm^2)

K is the constant of proportionality (**7.3**).

9. Test report

The test report shall include the following particulars:

(a) a reference to this British Standard;

(b) the result and the method of expression used;

(c) any unusual features noticed during the determination:

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