BS 684 : Section 2.36 : 1983
ISO 6463-1982

[BHA and BHT]

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British Standard Methods of analysis of

Fats and fatty oils

Part 2. Other methods

Section 2.36 Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT)

[ISO title: Animal and vegetable fats and oils – Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT) – Gas-liquid chromatographic method]

Méthodes d'analyse des corps gras
Partie 2. Autres méthodes
Section 2.36 Dosage du butylhydroxyanisol (BHA) et du butylhydroxytoluène (BHT)

Verfahren zur Analyse von Fetten und Fettölen
Teil 2. Andere Verfahren
Abschnitt 2.36 Bestimmung von Butylhydroxyanisol (BHA) und Butylhydroxytoluol (BHT)

IMPORTANT NOTE. It is essential that this Section be read in conjunction with the information in BS 684 : Part 0 'General Introduction' which is published separately.

National foreword

This Section of BS 684 was prepared under the direction of the Food and Agriculture Standards Committee and is identical with ISO 6463-1982 'Animal and vegetable fats and oils – Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT) – Gas-liquid chromatographic method' published by the International Organization for Standardization (ISO).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words 'International Standard' appear, referring to this standard, they should be read as 'British Standard'.

Cross-reference

International standard	Corresponding British Standard
ISO 5558-1982	BS 684 Methods of analysis of fats and fatty oils Section 2.33 : 1983 Detection and identification of antioxidants (Identical)

Additional information. With reference to the note to clause **1**, the method is not now considered suitable for the determination of TBHQ. There is a proposal to amend the text of the international standard.

With regard to **7.2.2.2** it is sometimes useful to use an additional solution containing 20 µg of antioxidant per millilitre.

With regard to **7.2.3** it is recommended that a graph is always plotted. If the graph is a straight line, the mean value of K can be calculated.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

British Standards Institution

1 Scope and field of application

This International Standard specifies a gas-liquid chromatographic method for the determination of butylhydroxyanisole (*tert*-butyl-4-methoxyphenol) (BHA) and butylhydroxytoluene (2,6-di-*tert*-butyl-4-methylphenol) (BHT), used as antioxidants, in animal and vegetable fats and oils.

NOTE — The method also permits quantitative determination of tertiobutylhydroquinone (TBHQ).

2 Reference

ISO 5558, *Animal and vegetable fats and oils — Detection and identification of antioxidants — Thin-layer chromatographic method.*

3 Principle

Dissolution of the fat or oil in a suitable solvent, direct injection into a gas chromatograph, and use of the internal standard method of calibration.

4 Reagents

4.1 Carrier gas : an inert gas (such as nitrogen, helium or argon), carefully dried and containing less than 10 mg of oxygen per kilogram.

4.2 Auxiliary gases :

— hydrogen, minimum purity 99,9 %, free from organic compounds;

— air or oxygen, free from organic compounds.

4.3 Dichloromethane or, failing this, **carbon disulphide**, containing no impurities which could interfere with the determination of BHA or BHT by gas chromatography.

WARNING — Dichloromethane and carbon disulphide are toxic. In addition, carbon disulphide is very volatile and explosive and particular care must be exercised in using it.

4.4 Methyl undecanoate, minimum purity 99 %.

4.5 Butylhydroxyanisole, minimum purity 98 %.

4.6 Butylhydroxytoluene, minimum purity 98 %.

5 Apparatus

Usual laboratory equipment, and in particular :

5.1 Gas chromatograph, with a **flame ionization detector** and **recorder,** comprising :

5.1.1 Injection device, together with one of the following systems to retain the non-volatile fats and oils :

a) a pre-column packed with siliconized glass wool or glass beads;

b) a sleeve lined with siliconized glass wool placed in the injector (only in the case of a horizontal injector).

5.1.2 Column, made of stainless steel or glass, permitting good separation of BHA and BHT, of length about 2 m and 2 to 4 mm in internal diameter, packed, for example, with 10 % methylpolysiloxanes[1] on acid-washed, silylated brick dust.[2]

5.2 Volumetric flasks, of capacities 10, 20 and 100 ml.

5.3 Graduated pipettes, of capacities 1 and 2 ml.

5.4 Analytical balance.

6 Detection

See ISO 5558.

7 Procedure

7.1 Setting up the apparatus

7.1.1 Injection device

— Temperature : 250 °C

The sleeve or pre-column (5.1.1) shall be removed after each day of analysis and conditioned overnight at the test temperature.

NOTE — Verify the proper condition of the sleeve or pre-column by passing, from time to time, a fat or oil of known composition through the chromatograph.

7.1.2 Oven and column

— Temperature under isothermal conditions : 160 °C

— Flow rate of carrier gas : optimum value to be established by the operator.

Before first use, condition the filled columns for 24 h at 220 °C with the carrier gas flowing.

7.1.3 Detector

— Temperature : 250 °C

— Flow rate of auxiliary gases :

hydrogen : approximately 20 ml/min

air or oxygen : according to the manufacturer's instructions.

1) DC 200 [of kinematic viscosity 1,25 m^2/s (12 500 cSt)] is suitable.

2) Gas/Chrom Q, of particle size 150 to 180 µm (80 to 100 mesh) is suitable.