International Standard 6463

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Animal and vegetable fats and oils — Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT) — Gas-liquid chromatographic method

Corps gras d'origines animale et végétale — Dosage du butylhydroxyanisol (BHA) et du butylhydroxytoluène (BHT) — Méthode par chromatographie en phase gazeuse

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6463 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in March 1981.

It has been approved by the member bodies of the following countries :

Australia	India	Romania
Austria	Iran	South Africa, Rep. of
Canada	Iraq	Sri Lanka
Chile	Israel	Tanzania
Czechoslovakia	Italy	Thailand
Dominican Republic	Kenya	United Kingdom
Egypt, Arab Rep. of	Korea, Rep. of	USA
Ethiopia	Mexico	USSR
France	Netherlands	Yugoslavia
Germany, F. R.	New Zealand	
Hungary	Portugal	

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Animal and vegetable fats and oils — Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT) — Gas-liquid chromatographic method

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]

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.