CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Determination of Phenolic Antioxidants (Preservatives)

Purpose

Determination of phenolic antioxidants (preservatives) 2- and 3-tert-butyl-4-hydroxyanisole (BHA), 3,5-di-tert-butyl-4-hydroxytoluene (BHT), and tert-butyl-hydroquinone (TBHQ) in edible oil and commercial food products.

Phenolic antioxidants are frequently used in products containing fats and oils to retard their oxidation (rancidity), and to improve shelf-life. Some major antioxidants are BHA, BHT, and TBHQ.

Existing Methods

Predominatly TLC and GC. The latter method exhibits adequate detection limits but requires extensive sample-handling and derivatization. LCUV and LCF methods generally do not have adequate detection limits.

Reference

Determination of Phenolic Antioxidants in Edible Oil by High-Performance Liquid Chromatography with Amperometric Detector, Y. Kitada, Y. Ueda, M. Yamamoto, K. Shinomiya, and H. Nakazawa, J. Liq. Chromatog. 8(1985) 47-57.

Conditions

Electrode: Glassy Carbon
Potential: +1.0 V vs. Ag/AgCl
Column: Diasil CN, 10 µm, 150 x 4 mm (Nihon Kuromato-Kogyo, Co., Ltd. Tokyo, Japan)
Temperature: 40°C
Mobile Phase: acetonitrile: 0.05 M sodium dihydrogen phosphate, pH 3.0, 30:70 (v/v). Flow rate of 1.0 mL/min.
Detection Limit: BHA, 1.0 ng; BHT, 4.0 ng; TBHQ, 1.0 ng injected

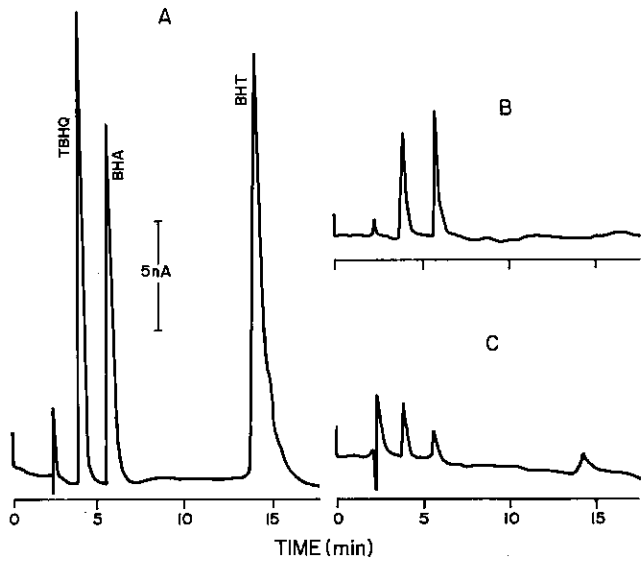

Figure 1. Comparison of response with different detectors. A) electrochemical, potential +1.0 V vs. Ag/AgCl., 40 nA F.S.; B) fluorescence, Ex. 285 nm, Em. 315 nm, L/2; C) ultraviolet, 285 nm, 0.005 AUFS. Amount injected: TBHQ, 5 ng; BHA, 5 ng; BHT, 20 ng. Redrawn from Kitada, et al., J. Liq. Chromatogr. 7(1984) 2031.

Sample Preparation

A continuous liquid-liquid partition technique is described. Recoveries of the antioxidants were greater than 90%. A simpler extraction for oils, foods, and cosmetics was used by King, et al. (Reference 1), also with greater than 90% recoveries.

Note

Some other antioxidants are n-propyl gallate (PG) and nordihydroguaiaretic acid (NGDA). Phenolic additives such as the homologous esters of 4-hydroxybenzoic acid, the parabens, have been

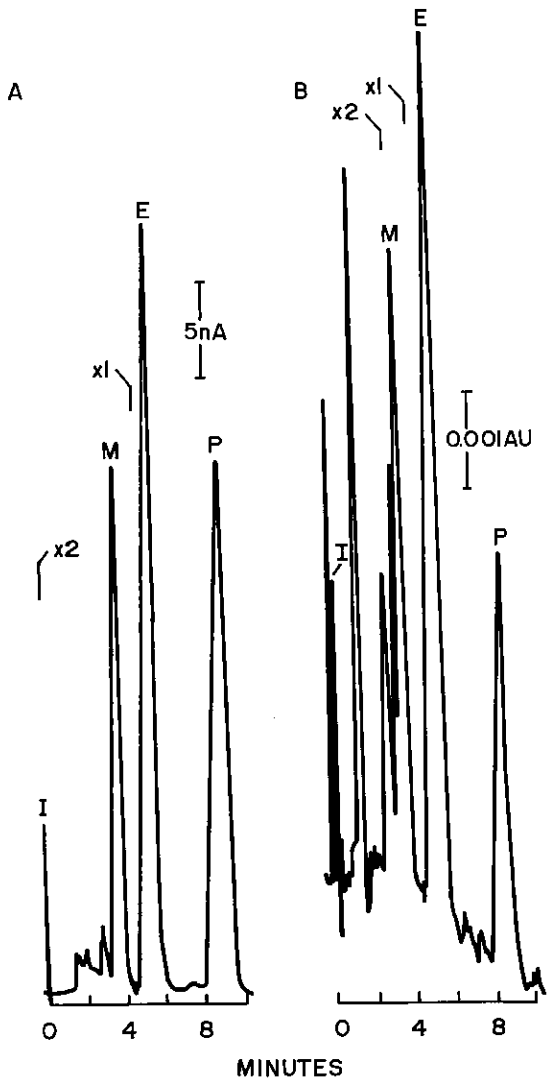

Figure 2. Comparison of amperometric and ultraviolet detection response. Sample: extract of 1-year-old cosmetic containing MPHB (M) and PPHB (P), with EPHB (E) as internal standard. Redrawn from King, et al., Assoc. Off. Anal. Chem. 63(1980) 13 (Reference 1).

added to various products to inhibit the growth of microorganisms. One such product is cosmetics that may be used near the eye, since bacterial contamination can be a potential health hazard. The esters commonly used are methyl-, propyl-, and butyl-p-hydroxybenzoate, MPHB, PPHB, and BPHB respectively. See Figure 2 for the detection of the parabens. This reference (Reference 1) also reports on the determination of the antioxidants PG, NDGA as well as BHA, BHT, and TBHQ.

Related References

1. W.P. King, K.T. Joseph, and P.T. Kissinger, J. Assoc. Off. Anal. Chem. 63(1980) 137.

2. A.N. Masoud, and Y.N. Cha, J. High, Resolut. Chromatog. Chromatog. Commun. 5(1982) 299.

3. Note, the determination of antioxidants presented in this report can be duplicated utilizing a BAS 400 or BAS 200 Problem Solver.