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DETERMINATION OF CAFFEINE IN TEA BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY AND A MODIFIED DIGESTION PROCEDURE

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SUMMARY

A more rapid and efficient method of sample preparation was needed for caffeine determination than the ones currently available. A variation of the existing AOAC method for coffee was developed using a Tecator digestion block normally used in determining nitrogen. Replicate samples were analyzed varying the digestion time and MgO content. The analysis parameters yielding the best results were chosen for the final method.

INTRODUCTION

Caffeine is one of the most important components in tea. A fast and accurate method is needed for its determination on a large number of samples. No sample preparation method is available which is suitable for the concentration range under consideration, and which can be applied to a variety of sample types[1–3]. This paper is a report on a modification of the AOAC digestion method for caffeine in coffee[4]. This is combined with a chromatographic system involving a dual pump set up to simplify mobile phase handling, an autosampler to facilitate injections and reduce labor, and a data processor to handle calculations. The chromatographic conditions are adapted from a Varian technical bulletin[5]. Analyses were performed on samples in a variety of matrices with caffeine levels ranging from 0.05% to 5.00%. The caffeine limit of detection was 50 ppm. The standard range of linearity was 0–250 μg/ml (the range checked). The standard deviation was 0.059.

EXPERIMENTAL

Reagents

Caffeine was obtained from Eastman (Rochester, NY, U.S.A.), 8-chlorotheophylline (internal standard) from Aldrich (Metuchen, NJ, U.S.A.), magnesium oxide (heavy, U.S.P.) and glacial acetic acid (HPLC grade) from J. T. Baker (Phillipsburg, NJ, U.S.A.) and acetonitrile (UV cutoff 190 nm for LC) from Waters Assoc. (Milford, MA, U.S.A.).

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Standards

Internal standard. Dissolve 100 mg of 8-chlorotheophylline in 1 l of water. This requires stirring a long time at room temperature.

Caffeine standard. Dissolve 100 mg of caffeine in 1 l of water. Make standards equivalent to 5, 10 and 20 μg/ml each with 10 μg/ml internal standard.

Preparation of sample

A digestion unit normally used for nitrogen analyses supplied by Tecator is used to prepare 20 samples at a time. Weigh 1–2 g solid sample or 5 g liquid sample into a 250-ml Tecator digestion tube. Add 2 g heavy MgO or twice the weight of solids content of the sample, whichever is greater. Add 100 ml of water with an automatic pipet. Weigh tube and coantents and record weight. Boil on Tecator block for 1 h. Cool tubes and bring back to original weight with water. Filter the extract through Whatman No. 1 filter paper and then filter approximately 20 ml through a 0.45-μm filter disc. Dilute samples by transferring a suitable aliquot to a 50-ml volumetric flask containing 5 ml 8-chlorotheophylline stock solution.

Chromatography

A Beckman high-performance liquid-chromatography (HPLC) instrument is used. This consists of a Model 500 Autosampler with a 50-μl sample loop, a Model 153 analytical UV detector at a fixed wavelength of 254 nm, two Model 110A pumps mixing and delivering de-gassed 1% acetic acid at a rate of 1.2 ml/min and 100% acetonitrile at a rate of 0.3 ml/min. The separation is achieved on a Varian MCH-10 reversed-phase column (25 cm × 2 mm), equipped with a 4 cm × 2 mm universal guard column. The data are processed with a Hewlett-Packard 85 data handler with Nelson Analytical interface and software.

RESULTS AND DISCUSSION

The results are presented in Tables I and II. By varying the digestion time and amount of MgO, the existing AOAC digestion procedure was modified so that 20 samples could be digested in 1 h. HPLC conditions were set up to yield an analysis time of 10 min. By employing an autosampler to facilitate injections, a dual-pump

TABLE I

MODIFIED DIGESTION PROCEDURE *VS.* AOAC PROCEDURE

Sample	*Amount of MgO (g)*	*Caffeine (%)*		
		Time on block (h)		*AOAC*
		1	*2*	
Black tea leaf	1	2.74, 2.68	2.60, 2.66	
	2	2.72, 2.68	2.69, 2.68	2.71, 2.76, 2.74, 2.78
Tea extract	2	0.91, 0.91	0.90, 0.89	
	5	0.93, 0.90	0.91, 0.91	0.90, 0.92, 0.87, 0.90

TABLE II

RESULTS OF FOUR DUPLICATE ANALYSES OF A BLACK TEA LEAF SAMPLE

Caffeine (%)			
Day 1	*Day 2*	*Day 3*	*Day 4*
2.99, 2.87	2.90, 2.82	2.83, 2.91	2.89, 2.96

Mean = 2.90
Standard deviation = 0.059

system to simplify mobile phase handling, an internal standard for more precise calculations and better control over injection volume, and a data processor to handle calculations, many more samples may be run in a shorter time than could be accomplished previously. One can easily more than double the output of samples.

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REFERENCES

1 S. Ashoor, G. Seperich, W. Moante and J. Welty, *J. Ass. Offic. Anal. Chem.*, 66 (1983) 606–609.
2 S. H. Wang, *Cienc. Prat.*, 4, No. 2 (1980) 85–91.
3 A. Ferrara, P. Reale, M. Grazia Calaminici, T. Iaccarino, *Boll. Chem. Unione Ital. Lab. Prov., Parte Sci.*, 33, No. S1 (1980) 55–60.
4 *Official Methods of Analysis*, Association of Official Analytical Chemists, Washington, DC, 13th ed., 1980, p. 236, method No. 15.051.
5 F. Klink, *Varian Instruments at Work*, No. 97, Varian, Walnut Creek, CA.