COMMISSION

FIRST COMMISSION DIRECTIVE

of 13 November 1979

laying down Community methods of analysis for testing coffee extracts and chicory extracts

(79/1066/EEC)

THE COMMISSION OF THE EUROPEAN COMMUNITIES,

Having regard to the Treaty establishing the European Economic Community,

Having regard to Council Directive 77/436/EEC of 27 June 1977 on the approximation of the laws of the Member States relating to coffee extracts and chicory extracts [1], and in particular Article 8 thereof,

Whereas under Article 8 of Directive 77/436/EEC, the composition and characteristics of coffee extracts and chicory extract are required to be checked according to Community methods of analysis;

Whereas a first series of methods for which studies are completed should now be adopted;

Whereas the measures provided for in this Directive are in accordance with the opinion of the Standing Committee on Foodstuffs,

HAS ADOPTED THIS DIRECTIVE:

Article 1

Member States shall take all measures necessary to ensure that the analyses necessary for the verification of the criteria set out in Annex I are carried out in accordance with the methods described in Annex II.

Article 2

Member States shall bring into force the laws, regulations and administrative provisions necessary to comply with this Directive within 18 months of its notification. They shall forthwith inform the Commission thereof.

Article 3

This Directive is addressed to the Member States.

Done at Brussels, 13 November 1979.

For the Commission

Étienne DAVIGNON

Member of the Commission

[1] OJ No L 172, 12. 7. 1977, p. 20.

ANNEX I

SCOPE OF THE FIRST COMMUNITY METHODS OF ANALYSIS DIRECTIVE FOR COFFEE EXTRACTS AND CHICORY EXTRACTS

I. General provisions.

II. Determination of the caffeine content in decaffeinated coffee extracts using Annex II, method 1.

III. Determination of the dry matter content in dried coffee extract and dried chicory extract, soluble coffee and chicory, and instant coffee and chicory, using Annex II, method 2.

IV. Determination of the dry matter content in liquid coffee extract, liquid chicory extract, coffee extract paste and chicory extract paste using Annex II, method 3.

ANNEX II

METHODS OF ANALYSIS RELATING TO THE COMPOSITION OF COFFEE EXTRACTS AND CHICORY EXTRACTS

GENERAL PROVISIONS

1. PREPARATION OF THE ANALYSIS SAMPLE

1.1. General

The mass of the sample presented to the laboratory for analysis shall be at least 50 g.

1.2. Preparation of the sample for analysis in the laboratory

1.2.1. Mixing

The sample for analysis shall always be mixed thoroughly before any test portion is weighed out.

1.2.1.1. Samples in powder or paste form shall be removed from the container, any lumps broken down, the sample mixed in an appropriate manner and placed in a suitable container.

1.2.1.2. Samples in liquid form shall be mixed by stirring.

1.3. Containers

The sample shall always be kept in an airtight and moisture-tight container.

2. REAGENTS

2.1. Water

2.1.1. Wherever mention is made to water for solution, dilution or washing purposes, distilled water, or demineralized water of at least equivalent purity, shall be used.

2.1.2. Wherever reference is made to 'solution' or 'dilution' without further indication, 'solution in water' or 'dilution with water' is meant.

2.2. Chemicals

All chemicals used shall be of recognized analytical reagent quality except where otherwise specified.

3. EQUIPMENT

3.1. Lists of equipment

The lists of equipment contain only those items with a specialized use and items with a particular specification.

3.2. Analytical balance

Analytical balance means a balance capable of weighing to at least 0.1 mg.

4. EXPRESSION OF RESULTS

4.1. Results

The result stated in the analytical report is the mean value obtained from at least two determinations, the repeatability of which is satisfactory.

4.2. Calculation of percentage

Except where otherwise specified, the result shall be calculated as a percentage by mass of the

4.3. **Number of significant figures**

The result shall not contain more significant figures than are justified by the precision of the method of analysis used.

5. TEST REPORT

The test report shall identify the method of analysis used as well as the results obtained. In addition, it shall mention all details of procedure, not specified in the method of analysis, or which are optional, as well as any circumstances that may have influenced the results obtained. The test report shall give all the information necessary for the complete identification of the sample.

METHOD 1: DETERMINATION OF CAFFEINE CONTENT

1. SCOPE AND FIELD OF APPLICATION

This method determines the caffeine content of decaffeinated coffee extracts.

2. DEFINITION

Caffeine content: the content of caffeine as determined by the method specified.

3. PRINCIPLE

Caffeine is extracted from a test portion of the sample in an ammoniacal medium. It is then successively purified with diethyl ether on two chromatographic columns, the first in an alkaline medium and the second in an acid medium. The caffeine is then eluted from the column by chloroform and determined spectrophotometrically.

4. REAGENTS

4.1. Sulphuric acid, 2 M solution.

4.2. Sodium hydroxide, 2 M solution.

4.3. Celite 545 or equivalent.

4.4. Ammonia solution, approximately 4 M (prepare by adding 1 volume of concentrated ammonia solution, p_{20} ca. 0.9 g/ml to 2 volumes of water).

4.5. Diethyl ether, pure or repurified by chromatography on a column of basic aluminium oxide of activity grade 1.

Pass 800 ml of diethyl ether through a column filled with 100 g of aluminium oxide. The diethyl ether thus purified, shall be kept in dark bottles until used.

(Diethyl ether, recently distilled and free of peroxides, can be used instead of diethyl ether purified by chromatography.) Saturate the diethyl ether with water.

4.6. Caffeine (1,3,7-trimethyl-2,6-dihydroxypurine), pure, anhydrous ($C_8H_{10}N_4O_2$).

4.7. Chloroform, pure or repurified by chromatography according to the method specified in 4.5 and saturated with water.

5. APPARATUS

5.1. Chromatographic columns (see figure 1), approximately 250 mm long, 21 mm internal diameter (column I) and 17 mm internal diameter (column II), fitted with stopcocks.

5.2. Ultra-violet spectrophotometer: The spectrophotometer shall be accurate to within 0.004 absorbance unit within the range used.

5.3. Silica cells with 10 mm optical path length.

5.4. Usual laboratory equipment, including

5.4.1. Water bath, boiling.

5.4.2. One-mark volumetric flasks, 50 ml, 100 ml and 1 000 ml, complying with ISO 1042.

5.4.3. One-mark pipettes, 2 ml and 5 ml; complying with ISO 648.

5.4.4. Analytical balance.

6. PROCEDURE

6.1. Preparation of the test portion

Weigh, to the nearest 0.1 mg, about 0.5 g of dried coffee extract sample, between 0.5 and 0.7 g of coffee extract paste sample or between 0.8 and 3.2 g of liquid coffee extract sample. (These last two weights should be chosen to give test portions containing approximately 0.5 g of dried coffee extract.) Transfer sample to a 100 ml beaker add 5 ml of ammonia solution (4.4) and warm for two minutes on the boiling water bath (5.4.1). Add 6 g of Celite (4.3) and mix carefully.

6.2. Filling of the columns

6.2.1. *Column I* (alkaline column)

Layer A. Mix carefully, by kneading with a flexible spatula blade, 3 g of Celite (4.3) and 2 ml of the sodium hydroxide solution (4.2), until homogeneous (see note below). A slightly wet powder will be obtained. Transfer this powder, in small portions (of approximately 2 g), into a chromatographic column (5.1), the lower part of which is packed with a wad of cotton wool or glass wool. Tamp down the mixture after each addition, without excessive force, using a glass rod, one end of which is flattened to the internal diameter of the column, until a perfectly homogeneous and compact layer is obtained. A small wad of cotton wool or glass wool may be placed on the top of layer A.

Note: Column packing material may be prepared in bulk quantities in advance and stored in closed containers.

Layer B. Transfer the Celite-sample mixture (6.1) into the column upon layer A. Dry the sample beaker twice with portions of about 1 g of Celite (4.3) transferring this Celite into the column. Tamp down to obtain a homogeneous layer and place a wad of cotton wool or glass wool on the top of layer B.

6.2.2. *Column II* (acid column)

Place in a second column, the lower part of which is packed with a wad of cotton wool or glass wool, 3 g of Celite (4.3) and 3 ml of the sulphuric acid solution (4.1), carefully mixed as directed for layer A in 6.2.1 (see the note in that sub-clause). Place a wad of cotton wool or glass wool on the top of the layer to prevent damage to the top of the column packing.

6.3. Chromatography

Mount the columns one above the other so that the effluent from column I can drip directly into column II. Pass 150 ml of diethyl ether (4.5) through the two columns. Keep open the stopcock in column I. Adjust the stopcock of column II so that a quantity of supernatant liquid remains above the column packing. Remove column I. Pass 50 ml of diethyl ether (4.5) through column II, using the initial portion to wash the tip of column I and passing this portion also into column II. Discard the effluent from column II.

Note: Used diethyl ether may be recovered by shaking it with iron (II) sulphate.

Pass a stream of air through column II with the stopcock open (for example by using an inflated rubber blower), until no more diethyl ether drips from the column and the air flow from the stopcock carries only a faint smell of diethyl ether (see note below). Elute column II with 45 to 50 ml of chloroform (4.7). Collect the eluate in a 50 ml volumetric flask (5.4.2), make up to volume with chloroform (4.7) and mix carefully.