First Commission Directive of 26 July 1979 laying down Community methods of analysis for testing certain sugars intended for human consumption (79/796/EEC)

ANNEX II

METHODS OF ANALYSIS TO VERIFY THE COMPOSITION OF CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

METHOD 2**DETERMINATION OF DRY MATTER**Vacuum oven method

1. **Scope and field of application**

The method determines the dry matter content in:

— glucose syrup,

— dried glucose syrup,

— dextrose monohydrate,

— dextrose anhydrous.

2. **Definition**

'The dry matter content': the content of dry matter as determined by the method specified.

3. **Principle**

The dry matter is determined at a temperature of 70 ± 1 ^{o}C using a vacuum oven at a pressure not exceeding 3·3 kPa (34 mbar). The test portions in the case of glucose syrup or dried glucose syrups, are prepared by mixing with water and kieselguhr before drying.

4. **Reagents**

4.1. *Kieselguhr:* place in a Buchner funnel and purify by repeated washings with dilute hydrochloric acid (1 ml of concentrated acid, density at 20 ^{o}C = 1·19 g/ml per litre of water). The treatment is complete when the washings remain definitely acid. Wash with water until the pH value of the filtered water is greater than 4. Dry in an oven at 103 ± 2 ^{o}C and store in an airtight container.

5. **Apparatus**

5.1. *Vacuum drying oven,* leak tight, thermostatically controlled and equipped with a thermometer and a vacuum manometer. The oven design must be such that the heat is rapidly transferred to the weighing dishes placed on the shelves.

5.2. *Air-drying train* consisting of a glass tower filled with freshly activated dry silica gel or an equivalent desiccant containing a water content indicator. This tower is mounted in series with a gas scrubber containing concentrated sulphuric acid connected to the air intake of the oven.

5.3. *Vaccum pump* capable of maintaining the presure in the oven at 3·3 kPa (34 mbar) or less.

5.4. *Metal weighing dish,* flat-bottomed, resistant to attack by the samples and the conditions of test, diameter at least 100 mm, depth at least 300 mm.

5.5. *Glass rod* of a length such that it cannot completely fall into the container.

5.6. *Desiccator* containing freshly activated dry silica gel, or an equivalent desiccant, with a water content indicator.

5.7. *Analytical balance* accurate to within 0.1 mg.

6. **Procedure**

6.1. Pour approximately 30 g of kieselguhr (4.1) into the weighing dish (5.4) equipped with a glass rod (5.5). Place the whole in the oven (5.1) at 70 ± 1 °C and reduce the pressure to 3·3 kPa (34 mbar) or less.

Dry for at least five hours, drawing a slow stream of air into the oven through the drying train. Check the pressure from time to time and correct it if necessary.

6.2. Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the dish together with the glass rod in the desiccator (5.6). Allow to cool and then weigh.

63. Accurately weigh to the nearest 1 mg approximately 10 g of the sample to be analyzed into a 100 ml beaker.

6.4. Dilute the test portion with 10 ml of warm water and transfer the solution quantitatively into the weighing dish, using the glass rod (5.5).

6.5. Place the dish containing the test portion and the glass rod in the oven and reduce the pressure to 3.3 kPa (34 mbar) or less. Dry at 70 ± 1 °C, allowing a slow stream of dry air to pass through the oven.

The drying operation should proceed for 20 hours; the bulk of the loss should occur towards the end of the first day. It will be necessary to keep the vacuum pump working at a preset pressure and allow a slow stream of dry air to enter the oven so as to maintain a pressure of approximately 3·3 kPa (34 mbar) or less during the night.

6.6. Restore atmospheric pressure in the oven by cautiously increasing the intake of dry air. Immediately place the weighing dish and contents in the desiccator. Allow to cool and then weigh to the nearest 1 mg.

6.7. Continue operation (6.5) for a further four hours. Restore atmospheric pressure in the oven and immediately place the dish in the desiccator. Allow to cool and then weigh. Ascertain whether constant mass has been reached. It is considered that constant mass has been satisfactorily attained if the difference between the two weighings of the same dish does not exceed 2 mg. If the difference is greater, repeat operation 6.7.

6.8. For the determination of the dry matter in dextrose anhydrous or dextrose monohydrate samples the use of kieselguhr and water is not required.

7. Expression of results

7.1. *Formula and method of calculation*

The dry matter content, expressed as a percentage by mass of the sample is given by:

$$(m_1 - m_2) \times \frac{100}{m_0}$$

where:

m_0 = the initial mass, in grams, of the test portion,

m_1 = the mass, in grams, of the weighing dish plus the kieselguhr, the glass rod and the residue of the test portion after drying,

m_2 = the mass, in grams, of the weighing dish plus the kieselguhr and the glass rod.

7.2. *Repeatability*

The difference between the results of two determinations when carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0·12 g per 100 g of sample.