

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

- Article 1 (1) Member States shall require that the analyses necessary for...
Article 2 Member States shall bring into force the laws, regulations or...
Article 3 This Directive is addressed to the Member States.

ANNEX I

SCOPE OF THE COMMUNITY METHODS OF ANALYSIS FOR CERTAIN SUGARS INTENDED FOR HUMAN CONSUMPTION

Determination of the loss of mass on drying in: semi-white...

ANNEX II

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

INTRODUCTION

1. Preparation of the sample for analysis
2. Reagents and apparatus
3. Expression of results

METHOD DETERMINATION OF THE LOSS OF MASS ON DRYING

1. Scope and field of application
2. Definition
3. Principle
4. Apparatus
 - 4.1. Analytical balance, accurate to within 0.1 mg.
 - 4.2. Oven, suitably ventilated, thermostatically controlled, and capable of being maintained...
 - 4.3. Metal weighing dish, flat-bottomed, resistant to attack by the samples...
 - 4.4. Desiccator, containing freshly activated silica gel or an equivalent desiccant,...
5. Procedure
 - 5.1. Dry the dish (4.3) to constant weight in the oven...
 - 5.2. Allow the dish to cool in the desiccator (4.4) for...
 - 5.3. Weigh accurately, to the nearest 0.1 mg, approximately 20 to...
 - 5.4. Place the dish in the oven (4.2) at $103 \pm$...
 - 5.5. Allow the dish to cool in a desiccator (4.4) and...
 - 5.6. Replace the dish in the oven at 103 ± 2 ...
 - 5.7. Do not exceed four hours total drying time.
6. Expression of results
 - 6.1. Formula and method of calculation
 - 6.2. Repeatability

METHOD 2 DETERMINATION OF DRY MATTER Vacuum oven method 1. Scope and... Vacuum oven method 1. Scope and field of application The...

1. Scope and field of application
2. Definition
3. Principle
4. Reagents
 - 4.1. Kieselguhr: place in a Buchner funnel and purify by repeated...
5. Apparatus
 - 5.1. Vacuum drying oven, leak tight, thermostatically controlled and equipped with...
 - 5.2. Air-drying train consisting of a glass tower filled with freshly...
 - 5.3. Vacuum pump capable of maintaining the pressure in the oven...
 - 5.4. Metal weighing dish, flat-bottomed, resistant to attack by the samples...
 - 5.5. Glass rod of a length such that it cannot completely...
 - 5.6. Desiccator containing freshly activated dry silica gel, or an equivalent...
 - 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...
 - 6.2. Restore atmospheric pressure in the oven by cautiously increasing the...
 - 6.3. Accurately weigh to the nearest 1 mg approximately 10 g...
 - 6.4. Dilute the test portion with 10 ml of warm water...
 - 6.5. Place the dish containing the test portion and the glass...
 - 6.6. Restore atmospheric pressure in the oven by cautiously increasing the...
 - 6.7. Continue operation (6.5) for a further four hours. Restore atmospheric...
 - 6.8. For the determination of the dry matter in dextrose anhydrous...
7. Expression of results
 - 7.1. Formula and method of calculation
 - 7.2. Repeatability

METHOD 3 DETERMINATION OF TOTAL DRY MATTER (Refractometric method)

1. Scope and...(Refractometric method) 1. Scope and field of application The method...
1. Scope and field of application
2. Definition
3. Principle
4. Apparatus
 - 4.1. Refractometer, accurate to four decimal places, provided with a thermometer...
 - 4.2. Light source consisting of a sodium vapour lamp.
5. Procedure
 - 5.1. If any crystals are present in the sample, redissolve them...
 - 5.2. Measure the refractive index of the sample at 20 °C...
6. Expression and calculation of results
 - 6.1. Calculate the dry matter content from the refractive indices for...
 - 6.2. If the sample was diluted to 1: 1 (m/m) with...
 - 6.3. Repeatability

METHOD 4 MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGARS (Berlin Institute...(Berlin Institute method) Scope and field of application 1. The...

1. The...
 1. Scope and field of application

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1. The method determines the reducing sugar content expressed as invert...
2. Definitions
3. Principle
4. Reagents
 - 4.1. Copper II solution (Muller's solution)
 - 4.2. Acetic acid solution 5 mol/litre.
 - 4.3. Iodine solution 0·01665 mol/litre (i.e. 0·0333 N, 4·2258 g/litre).
 - 4.4. Sodium thiosulphate solution 0·0333 mol/litre.
 - 4.5. Starch solution: to one litre of boiling water add a...
5. Apparatus
 - 5.1. Conical flask, 300 ml; precision burettes and pipettes.
 - 5.2. Water-bath, boiling.
6. Procedure
 - 6.1. Weigh a portion of the sample (10 g or less)...
 - 6.2. Carry out a blank test with water. This is to...
 - 6.3. Carry out a control test under cold conditions with the...
7. Expression of results.
 - 7.1. Formula and method of calculation
 - 7.1.1. The number of ml consumed in the blank test carried...
 - 7.1.2. The number of ml consumed in the cold test with...
 - 7.1.3. A value of 2·0 ml for every 10 g of...
 - 7.2. Repeatability

METHOD 5 MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR (Knight and...(Knight and Allen method) 1. Scope and field of application...

1. Scope and field of application
2. Definition
3. Principle
4. Reagents
 - 4.1. Ethylene diamine tetra-acetic acid solution (disodium salt) (EDTA) 0·0025 mol/litre:...
 - 4.2. Murexide indicator solution: add 0·25 g of murexide to 50...
 - 4.3. Alkaline copper reagent: dissolve 25 g of anhydrous sodium carbonate...
 - 4.4. Standard invert sugar solution: dissolve 23·750 g of pure sucrose...
 - 4.5. Pure sucrose: sample of pure sucrose with an invert sugar...
5. Apparatus
 - 5.1. Test tubes, 150 x 20 mm.
 - 5.2. White porcelain dish.
 - 5.3. Analytical balance, accurate to within 0·1 mg.
6. Procedure
 - 6.1. Dissolve 5 g of sugar sample in 5 ml of...
 - 6.2. Transfer quantitatively the solution in the test tube to the...
 - 6.3. Construct a calibration graph by adding known amounts of invert...
7. Expression of results
 - 7.1. Method of calculation
 - 7.2. When a concentration greater than 0·017 g invert sugar/100 g...
 - 7.3. Repeatability
8. Note

METHOD 6 DETERMINATION OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR OR DEXTROSE...(Luff-Schoorl method) 1. Scope and field of application The method...

1. Scope and field of application
 - 1.1. The reducing sugars content expressed as invert sugar in:
 - 1.2. The reducing sugar content, expressed and calculated (on the dry...
 - 1.3. The reducing sugar content expressed as D-glucose in:
2. Definition
3. Principle
4. Reagents
 - 4.1. Carrez solution I: dissolve 21·95 g of zinc acetate dihydrate...
 - 4.2. Carrez solution II: dissolve 10·6 g of potassium hexacyanoferrate IT...
 - 4.3. Luff-Schoorl reagent: prepare the following solutions:
 - 4.4. Sodium thiosulphate solution, 0·1 mol/litre.
 - 4.5. Starch solution: to one litre of boiling water add a...
 - 4.6. Sulphuric acid, 3 mol/litre.
 - 4.7. Potassium iodide solution, 30% (m/v).
 - 4.8. Pumice chips, boiled in hydrochloric acid, washed free of acid...
 - 4.9. Isopentanol
 - 4.10. Sodium hydroxide, 0·1 mol/litre.
 - 4.11. Hydrochloric acid, 0·1 mol/litre.
 - 4.12. Phenolphthalein solution, 1% (m/v) in ethanol.
5. Apparatus
 - 5.1. Conical flask, 300 ml, fitted with a reflux condenser.
 - 5.2. Stop-watch.
6. Procedure
 - 6.1. Standardization of the Luff-Schoorl reagent (4.3)
 - 6.2. Preparation of the solution
 - 6.3. Titration by the Luff-Schoorl method
7. Expression of results
 - 7.1. Formula and method of calculation
 - 7.2. Repeatability
8. Note

METHOD 7 MEASUREMENT OF REDUCING SUGARS EXPRESSED AS INVERT SUGAR (Lane and...(Lane and Eynon constant volume modification) 1. Scope and field...

1. Scope and field of application
2. Definition
3. Principle
4. Reagents
 - 4.1. Fehling's solution:
 - 4.2. Sodium hydroxide solution, 1 mol/litre.
 - 4.3. Standard invert sugar solution: dissolve 23·750 g of pure sucrose...
 - 4.4. Methylene blue solution, 1 g/100 ml.
5. Apparatus
 - 5.1. Narrow-necked laboratory boiling flasks, 500 ml.
 - 5.2. Burette, 50 ml, with tap and offset tip, graduated to...
 - 5.3. Pipettes graduated at 20, 25 and 50 ml.
 - 5.4. One mark volumetric flasks, 250, 1 000 and 2 000...
 - 5.5. A heating device, suitable for maintaining boiling according to the...
 - 5.6. Stop-watch, indicating to within at least one second.
6. Procedure

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- 6.1. Standardization of Fehling's solution
 - 6.1.1. Pipette 50 ml of solution B (4.1.2) and then 50...
 - 6.1.2. Rinse and fill the burette with 0.25 % (0.25 g/100...
 - 6.1.3. Pipette a 20 ml aliquot of the mixed solutions A...
 - 6.1.4. Heat the flask and contents till boiling and allow to...
 - 6.1.5. Continue the standardization by adding, from the burette, the standard...
 - 6.1.6. The end-point should be reached at the end of three...
- 6.2. Preparation of sample solutions
- 6.3. Preliminary test
 - 6.3.1. A preliminary test must be carried out to ensure that...
 - 6.3.2. If, after the addition of the methylene blue solution, the...
- 6.4. Final analysis of sample solution
 - 6.4.1. Pipette into the boiling flask 20 ml of mixed Fehling's...
 - 6.4.2. Add, from the burette, the observed titre of the sample...
7. Expression of results
 - 7.1. Formula and method of calculation
Note:
 - 7.2. Repeatability
8. Note

METHOD 8 DETERMINATION OF DEXTROSE EQUIVALENT (Lane and Eynon constant) 1. Scope...(Lane and Eynon constant) 1. Scope and field of application...

1. Scope and field of application
2. Definition
 - 2.1. 'Reducing power': the reducing sugar content, determined by the method...
 - 2.2. 'Dextrose equivalent': the reducing power, calculated as a percentage by...
3. Principle
4. Reagents
 - 4.1. Fehling's solution:
 - 4.1.1. Solution A:
 - 4.1.2. Solution B:
Note:
 - 4.1.3. Preparation of the mixed Fehling's solution
Note:
 - 4.2. Anhydrous dextrose (D-glucose) (C₆H₁₂O₆)
 - 4.3. Standard dextrose solution, 0.600 g/100 ml
 - 4.4. Methylene blue solution, 0.1 g/100 ml
5. Apparatus
 - 5.1. Narrow necked laboratory boiling flasks, 250 ml.
 - 5.2. Burette, 50 ml, with tap and offset tip, graduated to...
 - 5.3. One mark pipettes, 25 ml and 50 ml.
 - 5.4. One mark volumetric flasks, 100 and 500 ml.
 - 5.5. A heating device, suitable for maintaining boiling according to the...
 - 5.6. A stop-watch, indicating to at least the nearest second.
6. Procedure
 - 6.1. Standardization of the Fehling's solution
 - 6.1.1. Pipette 25 ml of Fehling's solution (4.1.3) into a clean,...
 - 6.1.2. Fill the burette (5.2) with standard dextrose solution (4.3) and...
 - 6.1.3. Run into the boiling flask (5.1) from the burette 18...

- 6.1.4. Place the boiling flask on the heating device (5.5), previously...
- 6.1.5. When boiling commences, start the stop-watch from zero.
- 6.1.6. Boil the contents of the flask for 120 seconds, as...
- 6.1.7. After boiling has continued for 120 seconds (by the stop-watch)...
- 6.1.8. Repeat 6.1.1 and 6.1.2.
- 6.1.9. Run into the boiling flask (5.1) from the burette a...
- 6.1.10. Repeat 6.1.4, 6.1.5 and 6.1.6.
- 6.1.11. After boiling has continued for 120 seconds (by the stop-watch),...
- 6.1.12. Note the volume (V_0 ml) of standard dextrose solution used...
- 6.1.13. V_0 shall be between 19.0 and 21.0 ml standard dextrose...
- 6.1.14. For the day-to-day standardization of the mixed Fehling's solution, as...
 - Note 1:
 - Note 2:
 - Note 3:
 - Note 4:
- 6.2. Preliminary examination of the prepared sample
 - 6.2.1. Unless the reducing power (2.1) of the prepared sample is...
 - 6.2.2. Prepare a 2% m/v solution of the sample 'Z', having...
 - 6.2.3. As 6.1.2, using the sample solution (6.2.2) in place of...
 - 6.2.4. As 6.1.1.
 - 6.2.5. As 6.1.3, using 10.0 ml sample solution instead of 18.0...
 - 6.2.6. As 6.1.4.
 - 6.2.7. Heat the contents of the flask to boiling. Add 1...
 - 6.2.8. Immediately boiling has started, start the stop-watch (5.6) from zero...
 - 6.2.9. 'Y' must not exceed 50 ml. If it does, increase...
 - 6.2.10. The approximate reducing power of the prepared sample in percent...
- 6.3. Test portion
- 6.4. Test solution
- 6.5. Determination
 - 6.5.1. As 6.1.1.
 - 6.5.2. Fill the burette (5.2) with test solution (6.4) and adjust...
 - 6.5.3. Run into the boiling flask from the burette 18.5 ml...
 - 6.5.4. As 6.1.4.
 - 6.5.5. As 6.1.5.
 - 6.5.6. As 6.1.6.
 - 6.5.7. As 6.1.7, using test solution in place of standard dextrose...
 - 6.5.8. As 6.1.8.
 - 6.5.9. As 6.1.9, using test solution in place of standard dextrose...
 - 6.5.10. As 6.1.10.
 - 6.5.11. As 6.1.11, using test solution in place of standard dextrose...
 - 6.5.12. Note the volume (V_1) of test solution used up to...
 - 6.5.13. V_1 shall be between 19.0 and 21.0 ml test solution....
 - 6.5.14. Carry out two determinations on the same test solution.
- 6.6. Dry matter content
7. Expression of results
 - 7.1. Formulae and method of calculation
 - 7.1.1. Reducing power
 - 7.1.2. Dextrose equivalent

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- 7.1.3. Take as the result the arithmetic mean of the two...
- 7.2. Repeatability

METHOD DETERMINATION OF SULPHATED ASH

1. Scope and field of application
2. Definition
3. Principle
4. Reagents
 - 4.1. Sulphuric acid, dilute solution: slowly and cautiously add 100 ml...
5. Apparatus
 - 5.1. Electric muffle furnace, equipped with a pyrometer and capable of...
 - 5.2. Analytical balance, accurate to 0.1 mg.
 - 5.3. Ashing crucibles, platinum or quartz, of suitable capacity.
 - 5.4. Desiccator, containing freshly activated silica gel or an equivalent desiccant...
6. Procedure
7. Expression
 - 7.1. Formula and method of calculation
 - 7.2. Repeatability
8. Notes
 - 8.1. The sulphuric acid is added in small quantities to prevent...
 - 8.2. Every relevant precaution must be taken during the first carbonization...
 - 8.3. If the sample is difficult to ash completely (i.e. black...

METHOD DETERMINATION OF POLARIZATION

1. Scope and field of application
2. Definition
3. Principle
4. Reagents
 - 4.1. Clarification agent: basic lead acetate solution.
 - 4.2. Diethyl ether
5. Apparatus
 - 5.1. Saccharimeter graduated for the normal weight of 26 g of...
 - 5.2. Light source, consisting of a sodium vapour lamp.
 - 5.3. Precision polarimeter tubes, length 200 mm, error not exceeding \pm ...
 - 5.4. Analytical balance, accurate to within 0.1 mg,
 - 5.5. Individually calibrated 100 ml volumetric flasks stoppered. Flasks with a...
 - 5.6. Water-bath, controlled thermostatically at 20 ± 0.1 oC.
6. Procedure
 - 6.1. Preparation of the solution
 - 6.2. Polarization
 - 6.2.1. Obtain the zero correction of the apparatus.
 - 6.2.2. Filter the sample through a filter paper. Discard the first...
 - 6.2.3. Wash the polarimeter tube by rinsing twice with the sample...
 - 6.2.4. Fill the tube carefully at 20 ± 0.1 oC with...
 - 6.2.5. Read the rotation to within 0.05 oS or 0.02 angular...
7. Expression of results
 - 7.1. Formula and method of calculation
 - 7.2. Repeatability

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- (1) [OJ No L 356, 27. 12. 1973, p. 71.](#)