International Standard 6488

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Tobacco — Determination of water content (Reference method)

Tabac — Détermination de la teneur en eau (Méthode de référence)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6488 was developed by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, and was circulated to the member bodies in January 1980.

It has been approved by the member bodies of the following countries :

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United Kingdom

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Tobacco — Determination of water content (Reference method)

0 Introduction

The method specified in this International Standard is suitable as a reference method for the determination of the water in tobacco, and is the method for the calibration of routine methods (e.g. drying in an oven, electrical methods).

1 Scope and field of application

This International Standard specifies a reference method for the determination of the water content of tobacco by means of distillation using a solvent non-miscible with water.

The method is applicable to unmanufactured tobacco and manufactured tobacco, in particular cut tobacco.

NOTE — For certain manufactured tobaccos, certain types of humectants at high levels can give different results.

2 References

ISO 684, *Laboratory glassware — One-mark pipettes.*

ISO 835, *Laboratory glassware — Graduated pipettes.*[1]

ISO 4797, *Laboratory glassware — Flasks with conical ground joints.*

ISO 4799, *Laboratory glassware — Condensers.*

3 Principle

Entrainment of water of a test portion by means of boiling in a solvent non-miscible with water and separation of the entrained water. Measurement of the volume of water using a graduated tube and calculation of the percentage by mass of water contained in the test portion.

4 Reagents

During the analysis, use only reagents of recognized analytical grade.

4.1 Cyclohexane.

NOTES

1 As the solubility of water in cyclohexane is only 70 μl/l, it can be considered as having no significant effect on the results of the determination.

2 As cyclohexane is dangerous appropriate precautions should be taken during its use.

4.2 Silica gel, with a water-content indicator (blue-gel).

4.3 Anhydrous solid or liquid wetting agent.

NOTE — A sodium arylalkylsulphate is recommended as a liquid wetting agent. A polyethyloxylated lauryl alcohol is recommended as a solid wetting agent.

5 Apparatus

5.1 Balance, capable of weighing 500 g to the nearest 0,01 g.

5.2 Distillation apparatus, consisting of the following items.

NOTE — A suitable apparatus is shown in figure 1.

5.2.1 Round-bottom flask, of capacity 1 000 ml, complying with ISO 4797.

5.2.2 Round-bottom flask, of capacity 50 and 100 ml, complying with ISO 4797.

NOTE — The flask (5.2.2) is necessary in order to close the apparatus when it is not being used.

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[1] At present at the stage of draft. (Revision of ISO/R 835.)

5.2.3 Combined distillation measuring tube, of capacity 12 ml, graduated in 0,1 ml increments.

NOTE — Check the calibration of the combined distillation measuring tube by a standardized method, using one-mark pipettes complying with ISO 648 and/or graduated pipettes complying with ISO 835.

5.2.4 Liebig-West condenser, with a narrow cooling jacket, of length 250 mm, complying with ISO 4799.

5.2.5 Guard tube, filled with blue silica gel (4.2), to prevent the ambient humidity entering the apparatus.

NOTE — The silica gel should be dried before it turns completely pink at a temperature between 100 and 120 °C.

Before the apparatus is assembled, the joints shall be greased slightly with a mineral or siliconized grease.

5.3 Heating device.

Preferably use an explosion-proof electrical heating device.

5.4 Test room.

In view of the nature of the solvent used, a test room fitted with explosion-proof electrical devices is recommended.

6 Procedure

6.1 Preparation of glassware

Carefully clean all the glassware, using a mixture of chromic and sulphuric acids or a suitable detergent, in order to prevent droplets of water adhering to the internal walls of the glassware. Repeat this cleaning operation whenever it is difficult to remove droplets of water from the internal walls. After cleaning, rinse the glassware with ethanol and then dry it.

6.2 Constitution of the test sample, precautions to be taken during its preparation and during the preparation of the test portions

The test sample shall consist of manufactured tobacco or of fragments of leaves whose surface area shall be less than 1 cm^2. In the case of cigarettes, use only the tobacco (after removing the paper, filters, etc.).

Take all precautions necessary to prevent changes in the water content of the test sample during the preparation or during the weighing of the test portion.

6.3 Determination

6.3.1 Preparation and weighing of test portions

Weigh, to the nearest 0,01 g, from the test sample test portions of 50 g.

If the presumed water content is higher than 24 % (*m*/*m*), reduce the size of the test portion until the graduated volume of the measuring tube (5.2.3) is sufficient to contain the water which has been extracted.

NOTE — It is convenient to weigh the tobacco directly into the distilling flask by placing the flask on a balance and taking the tare of the flask into account. There is no objection to weighing a series of test portions one after the other into a series of flasks. After finishing each weighing, close each flask hermetically.

6.3.2 Distillation

Pour 350 ml of the cyclohexane (4.1) into the flask containing the test portion (6.3.1). The cyclohexane (4.1) may be added either immediately after weighing of the test portion or at any subsequent moment, provided that the flask is immediately reclosed and kept in this condition until distillation.

Assemble the apparatus (5.2). Start distillation and set the heating power so that the rate of distillation is 30 ± 5 ml/min. Cooling shall be sufficient for condensation to take place in the lower third of the condenser (5.2.4).

Continue for 180 min, measured from the moment when the first flow of solvent is noted. After this time, proceed as follows :

Slightly incline the apparatus and stop the current of cold water in the condenser twice for a few moments in order to facilitate the collection of droplets of water remaining in the condenser.

During this time, continue to heat so that the area of condensation rises as the drops of water located in the upper part of the condenser are washed down. Stop the distillation by removing the apparatus from the heat. Allow to cool to ambient temperature.

Remove the guard tube (5.2.5), containing the silica gel (4.2). Introduce a twisted wire, previously coated with a thin film of the wetting agent (4.3), into the condenser at the level of the condensation area. Transfer the droplets of water remaining in the graduated tube by washing them downwards three or four times with 10 ml of the cyclohexane (4.1).

6.3.3 Determination of water content

Cool the graduated tube rapidly to ambient temperature. Using a twisted wire, incorporate into the aqueous layer the droplets of water adhering to the internal walls of the tube containing the solvent.

If necessary, add a small quantity of the wetting agent to the twisted wire.

6.3.4 Number of determinations

For a complete analysis, carry out at least two independent determinations under identical conditions.

7 Expression of results

7.1 Readings

Read the volume of water collected in the graduated tube to the nearest 0,05 ml.

Subtract 0,05 ml from this value because of the flattening of the meniscus caused by the addition of the wetting agent. This value of the corrected volume is designated as V.

7.2 Calculation

The water content H, expressed as a percentage by mass, is given by the formula

$$H = \frac{m_1}{m_2} \times 100$$

where

m_1 is the mass, in grams, of volume V of water collected in the graduated tube;

m_2 is the mass, in grams, of the test portion.

NOTE — For the purposes of this International Standard, the mass m_1, expressed in grams, has the same numerical value as the volume V, expressed in millilitres.

Where the mass $m_2 = 50 \pm 0{,}01$ g, the water content $H = 2\ V$.

All the individual results shall be given with a decimal to the nearest 0,1 %.

7.3 Repeatability and reproducibility

7.3.1 Repeatability

The difference between the results of two determinations carried out successively on two test portions that are as identical as possible, by the same operator, using the same apparatus and in the same laboratory, shall not exceed 0,2 % (m/m) of water.

7.3.2 Reproducibility

It is difficult to state the reproducibility of the method with accuracy; nevertheless, experience shows that a difference between the results of two determinations carried out by different operators in different laboratories on test portions that are as identical as possible is acceptable in the order of 0,35 % (m/m) (or less).

8 Test report

The test report shall state the method used and the results obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have affected the results.

The test report shall include all details required for complete identification of the sample.

Dimensions in millimetres and capacity in millilitres

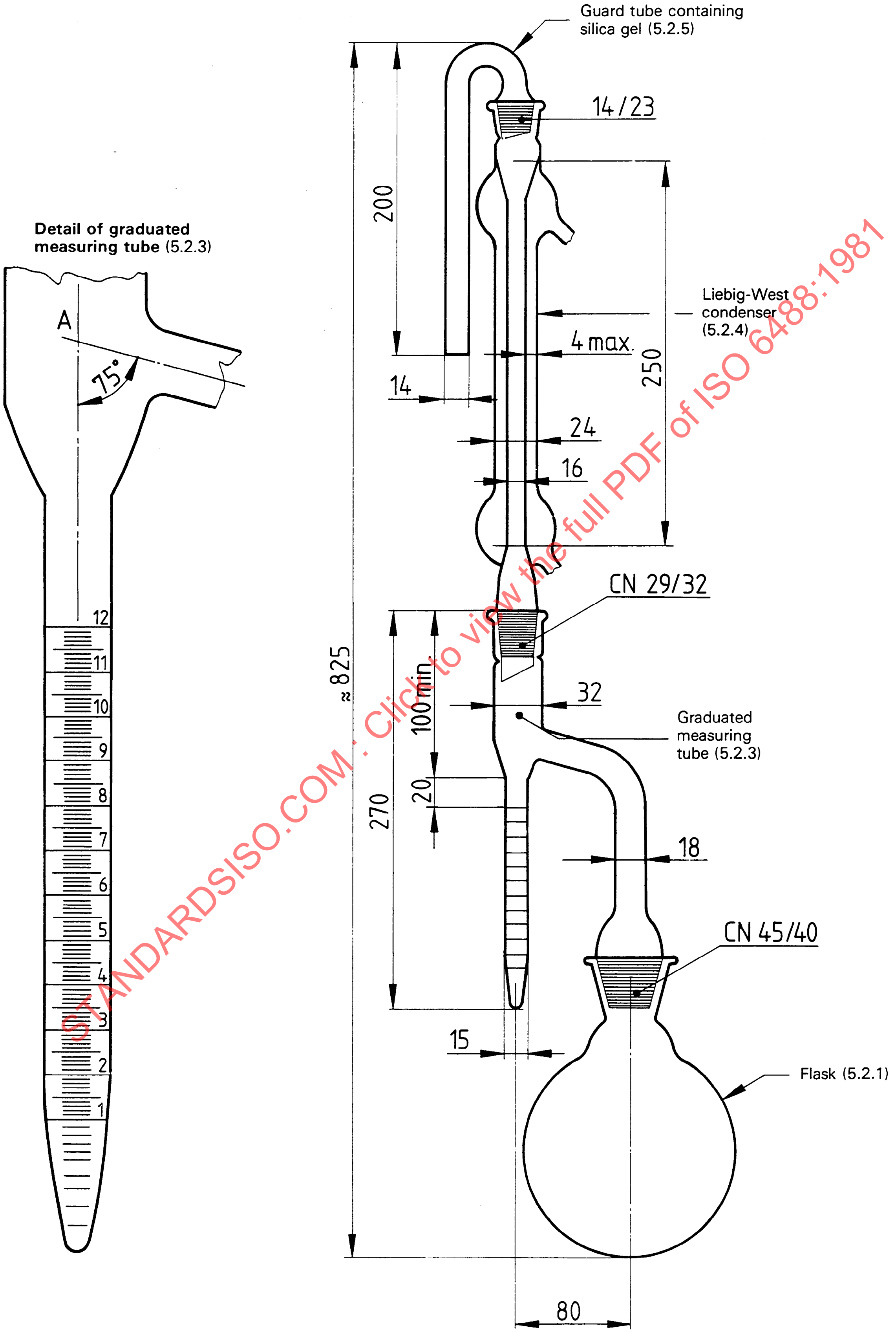

Figure — Example of a suitable distillation apparatus (5.2)