INTERNATIONAL STANDARD ISO 4388

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Tobacco and tobacco products — Determination of retention of coloured part of smoke condensate by cigarette filters — Direct spectrophotometric method

Tabac et produits du tabac — Détermination de la rétention de la partie colorée du condensat de fumée par les filtres de cigarettes — Méthode spectrophotométrique directe

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 4388 was developed by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, and was circulated to the member bodies in September 1976.

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

Australia	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Korea, Rep. of	Switzerland
Czechoslovakia	Mexico	United Kingdom
Egypt, Arab Rep. of	Netherlands	Yugoslavia
France	New Zealand	
Germany	Poland	

No member body expressed disapproval of the document.

Printed in Switzerland

Tobacco and tobacco products — Determination of retention of coloured part of smoke condensate by cigarette filters — Direct spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a direct spectrophotometric method for the determination at 450 nm of the retention by cigarette filters of the coloured part of smoke condensate.

The method is applicable to filter cigarettes. It is not applicable to cigarettes with coloured filters or to cigarettes with filters containing special additives, for example alkaline additives or active absorbents such as charcoal.

NOTE — The method is an empirical, quick and practical method relating only to coloured components. The results should not be interpreted with respect to any particular constituent of the smoke condensate.

2 REFERENCES

ISO 3308, *Tobacco and tobacco products — Routine analytical cigarette-smoking machine — Definitions, standard conditions and auxiliary equipment.*

ISO 3402, *Tobacco and tobacco products — Atmospheres for conditioning and testing.*

ISO . . ., *Tobacco and tobacco products — Cigarettes — Machine smoking and determination of crude and dry smoke condensate.* [1)]

ISO . . ., *Tobacco and tobacco products — Cigarettes — Sampling.* [1)]

3 DEFINITION

smoke condensate retention index (R_c) of a cigarette filter : The ratio, expressed as a percentage by mass, of the smoke condensate retained by the filter to that entering the filter.

$$R_c = \frac{F_c}{S_c} \times 100 = \frac{F_c}{F_c + H_c} \times 100$$

where

F_c is the mass of smoke condensate retained by the filter;

S_c is the mass of smoke condensate entering the filter;

H_c is the mass of smoke condensate in the main-stream smoke.

4 PRINCIPLE

Smoking of the filter cigarettes according to ISO . . ., and dissolution in methanol of the crude smoke condensate. Removal of the filter material from the remaining tobacco butts, and dissolution in methanol of the smoke condensate retained by the filters.

Comparison of the colour intensities of the two methanolic solutions by spectrophotometric measurement of the absorbances at 450 nm and calculation of the retention index from the ratio of the absorbances.

The absorbances are directly proportional to the masses of smoke condensate (F_c and H_c), assuming that the retention of the coloured components is not selective.

5 REAGENT

Methanol, of analytical reagent quality, at least 99,5 % (*V*/*V*).

6 APPARATUS

Usual laboratory apparatus not otherwise specified and the following items :

6.1 Conditioning enclosure, regulated in accordance with the requirements of ISO 3402.

6.2 Routine analytical cigarette-smoking machine, complying with the requirements of ISO 3308.

6.3 Spectrophotometer, suitable for measurements at a wavelength of 450 nm.

6.4 Matched glass cells, having an optical path length of 1 cm and, if necessary, 0,5 and/or 2 cm.

The absorbance of the cells shall be equal before and after each measurement.

1) In preparation.

6.5 Narrow-necked boiling flasks, of capacity 100 or 125 ml, with ground glass stoppers, complying with ISO 1773.

6.6 One-mark pipettes, complying with ISO 648.

6.7 Centrifuge, capable of producing a relative centrifugal acceleration of 25 g[1].

7 SAMPLING

Carry out sampling in accordance with the method specified in ISO . . .

8 PROCEDURE

8.1 Conditioning of test sample

Keep the test sample in the conditioning enclosure (6.1) until constant mass is attained.

8.2 Test portion

Take, as the test portion, 20 filter cigarettes from the conditioned test sample.

8.3 Determination

8.3.1 *Smoking of the cigarettes*

Using the routine analytical cigarette-smoking machine (6.2), smoke the 20 filter cigarettes of the test portion (8.2) according to ISO . . . into four smoke traps so as to collect the main-stream smoke condensate from five cigarettes on the glass fibre filter disc in each trap.

Extinguish the butts by separating the burning zone, remove the filter plugs and free them from adhering tipping and plug wrap material and from tobacco.

8.3.2 *Preparation of methanolic smoke condensate solution*

Combine the 10 cigarette filter plugs from two of the channels of the smoking machine and drop them into 100 ml of the methanol (clause 5) in a flask (6.5). Do the same with the filter plugs from two other channels. Swirl the contents of each flask and allow them to stand in the dark for about 24 h.

Remove the four glass fibre filter discs from the smoke traps and prepare from each disc a solution of the smoke condensate in 100 ml of the methanol in a flask (6.5).

Swirl the contents of each flask and allow them to stand in the dark for the same time as the methanolic solutions obtained from the cigarette filter plugs.

Transfer, by means of a pipette (6.6), 5 to 10 ml from each of the smoke condensate solutions, prepared as described above, to the centrifuge (6.7) and centrifuge for approximately 1 min to remove suspended matter.

8.3.3 *Spectrophotometric measurement*

Using the spectrophotometer (6.3), measure the optical absorbances of the clear supernatant smoke condensate solutions at 450 nm against a methanol blank.

Matched glass cells with an optical path length of 1 cm (6.4) are generally appropriate. (See 10.3.)

In this way obtain two absorbance values (A_F) for the solutions from the cigarette filter plugs and four absorbance values (A_H) for the solutions from the glass fibre filter discs.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

The smoke condensate retention index R_c of the cigarette filter, expressed as a percentage by mass, for each pair of smoking machine channels is given by the formula

$$R_c = \frac{F_c}{F_c + H_c} \times 100 = \frac{A_F/l_1}{A_F/l_1 + A_H/l_2} \times 100$$

where

A_F is the absorbance of the combined filter plug extracts, for one pair of channels, measured in cells of optical path length l_1 cm;

A_H is the sum of the absorbances of the extracts from the glass fibre filter discs corresponding to the same pair of channels, measured in cells of optical path length l_2 cm.

NOTE — The calculation remains unchanged if the method described in 10.3.2 is used. If the method described in 10.3.3 is used, the appropriate dilution factor must be taken into account.

9.2 Precision of results

Express the smoke condensate retention index R_c, as a percentage by mass, to the nearest 1 %, as separate values for each pair of smoking machine channels.

10 NOTES ON PROCEDURE

10.1 Separation of the butts and extraction of the filter plugs with methanol shall be carried out with the minimum delay after each smoking run (see 8.3.2).

10.2 The absorbance at the chosen wavelength of a methanolic extract of glass fibre filter disc blanks, of cellulose acetate and/or paper cigarette filters, should be the same as that of the methanol blank.

1) $1\,g = 9{,}81\ m \cdot s^{-2}$

10.3 In order to obtain optimum accuracy, the absorbance values for the test samples should lie between 0,2 and 0,7. Generally, with a cell of optical path length 1 cm, the undiluted methanolic solutions can be used.

10.3.1 If the absorbance is less than 0,2, a cell of longer path length may be used. Similarly if the absorbance is greater than 0,7, a cell of shorter path length may be used. If the possibility of using a longer or shorter path length is excluded because of the construction of the spectrophotometer, then one of the methods described in 10.3.2 or 10.3.3 may be used.

10.3.2 If the absorbance is less than 0,2, select a wavelength shorter than 450 nm but not less than 350 nm. Performing the determination at another wavelength influences only the level of absorbance and does not affect the accuracy of the determination.

Once the wavelength has been set for a particular determination, this setting should not be altered until the determination has been finished.

10.3.3 If the absorbance is greater than 0,7, adjust the concentration by diluting an aliquot of the sample solution to a measured volume.

11 TEST REPORT

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

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

The test report shall, in particular, include the following items of information :

a) Description of the product tested;

b) Sampling procedure;

   1) Method of sampling;

   2) Number of cigarettes comprising the test sample;

   3) Date and place of purchase or sampling;

c) Test conditions, in accordance with ISO 3402;

d) Test results expressed in accordance with 9.2;

e) Date of test.