INTERNATIONAL STANDARD

ISO 4388

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Cigarettes — Determination of the smoke condensate retention index of a filter — Direct spectrometric method

Cigarettes — Détermination de l'indice de rétention du condensat de fumée d'un filtre — Méthode spectrométrique directe

Reference number
ISO 4388:1991(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4388 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This second edition cancels and replaces the first edition (ISO 4388:1977), of which it constitutes a technical revision.

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Cigarettes — Determination of the smoke condensate retention index of a filter — Direct spectrometric method

1 Scope

This International Standard specifies a direct spectrometric method for the determination at 450 nm of the smoke condensate retention index of a cigarette filter.

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 absorbents such as active charcoal.

This empirical, quick and practical method relates only to smoke condensate. The results should not be interpreted with respect to any particular constituent of the smoke condensate.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3308:1991, *Routine analytical cigarette-smoking machine — Definitions and standard conditions.*

ISO 3402:1991, *Tobacco and tobacco products — Atmosphere for conditioning and testing.*

ISO 4387:1991, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine.*

ISO 8243:1991, *Cigarettes — Sampling.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 smoke condensate retention index (R_c) of a cigarette filter: The ratio, expressed as a percentage by mass, of the crude smoke condensate retained by the filter to that entering the filter (see figure 1):

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

where

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

S_c is the mass of crude smoke condensate entering the filter;

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

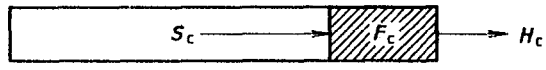

Figure 1

4 Principle

Smoking of filter cigarettes in accordance with ISO 4387. Dissolution in methanol of the crude smoke condensate from the main-stream smoke. Removal of the filter material from the remaining tobacco butts; dissolution in methanol of the crude smoke condensate retained by the filters.

Determination of the absorbances at 450 nm of the two methanolic solutions by spectrometric measurement and calculation of the retention index from

the ratio of the absorbances. The absorbances are directly proportional to the masses of crude smoke condensate (F_c and H_c).

5 Reagent

5.1 Methanol, having a water content lower than 0,05 % (m/m).

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 and ISO 4387.

6.3 Spectrometer, suitable for carrying out measurements at a wavelength of 450 nm.

6.4 Matched quartz cells, having an optical path length of 10 mm, if necessary, 5 mm and/or 20 mm.

6.5 Volumetric flasks, of capacities 100 ml or 125 ml, with ground glass stoppers.

6.6 One-mark pipettes, of capacities 5 ml and 10 ml.

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 8243.

8 Procedure

8.1 Conditioning of the test sample

Keep the test sample in the conditioning enclosure (6.1) until it attains constant mass.

8.2 Test portion

Take, as the test portion, 20 filter cigarettes from the conditioned test sample (8.1).

1) $1\ g = 9{,}81\ ms^{-2}$

8.3 Determination

8.3.1 Smoking of the cigarettes

Using the routine analytical cigarette-smoking machine (6.2), smoke the 20 filter cigarettes constituting the test portion (8.2) in accordance with ISO 4387.

NOTE 1 The number of cigarettes smoked per trap depends on the diameter of the glass-fibre filter and the yield of crude smoke condensate of the cigarette.

For a 44 mm smoke trap this number is in general 5.

For a 92 mm smoke trap this number is in general 20.

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

8.3.2 Preparation of methanolic crude smoke condensate solution

8.3.2.1 Combine the cigarette filter plugs from one channel of the smoking machine and put them into 100 ml of methanol (5.1) in a volumetric flask (6.5). Do the same with the filter plugs from the other channels. Stopper the flasks, swirl the contents of each flask and allow them to stand in the dark for about 24 h.

8.3.2.2 Remove the glass-fibre filter discs from the smoke traps and prepare from each disc a solution of the crude condensate in 100 ml of methanol (5.1) in a volumetric flask (6.5).

Stopper the flasks, 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 (8.3.2.1).

8.3.2.3 Transfer, by means of a pipette (6.6), 5 ml to 10 ml of each of the crude smoke condensate solutions, prepared as described in 8.3.2.1 and 8.3.2.2, to the centrifuge (6.7) and centrifuge for approximately 1 min to remove suspended matter.

8.3.3 Measurement

Check that the absorbance of each cell (6.4) is the same before and after each measurement.

Matched quartz cells with an optical path length of 10 mm (6.4) are generally suitable for the spectrometric measurement, but see 10.3.

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

Denote the absorbance values for the solutions from the cigarette filter plugs as A_F and the absorbance values for the solutions from the glass-fibre filter discs as A_H.

9 Expression of results

The smoke condensate retention index, R_c, of a cigarette filter, expressed as a percentage by mass, for each smoking machine channel 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 extract for one channel, measured in a cell of optical path length l_1, in millimetres;

A_H is the absorbance of the extract from the glass-fibre filter disc corresponding to the same channel, measured in a cell of optical path length l_2, in millimetres.

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 shall be taken into account.

For the cigarettes of each channel of the smoking machine, express the smoke condensate retention index, R_c, as a percentage by mass, to the nearest 1 %.

10 Notes on procedure

10.1 Separation of the butts and extraction of the filter plugs with methanol (8.3.2) shall be carried out with the minimum delay after each smoking run (8.3.1).

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.

10.3 In order to obtain optimum accuracy, the absorbance values for the test solutions should lie between 0,1 and 0,7. Generally, with a cell of optical path length of 10 mm, the undiluted methanolic solutions can be used.

10.3.1 If the absorbance is less than 0,1, 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 spectrometer, 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 0,1, select a wavelength shorter than 450 nm but not less than 350 nm.

NOTE 2 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 completed.

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.

NOTE 3 This is nearly always the case when smoking 20 cigarettes onto a 92 mm filter disc, where a dilution of the condensate solutions to a four times greater volume is recommended.

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) characteristic data about the cigarettes;

b) sampling procedure, including

   1) method of sampling,

   2) number of cigarettes comprising the test sample, and

   3) date and place of sampling;

c) test and conditioning atmosphere, in accordance with ISO 3402;

d) test results expressed in accordance with clause 9;

e) date of the test.

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Descriptors: tobacco, cigarettes, cigarette filters, smoke, condensates, chemical analysis, determination, retention index, spectrometric method.

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