INTERNATIONAL STANDARD

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Cigarettes — Determination of alkaloid retention by the filters — Spectrometric method

Cigarettes — Détermination de la rétention des alcaloïdes par les filtres — Méthode spectrométrique

Reference number
ISO 3401:1991(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 3401 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This second edition cancels and replaces the first edition (ISO 3401:1977), which has been technically revised.

Cigarettes — Determination of alkaloid retention by the filters — Spectrometric method

1 Scope

This International Standard specifies two methods for the spectrometric determination of alkaloid retention by filters of cigarettes:

— the direct method;

— the indirect method.

The methods are applicable to filter cigarettes. The direct method shall be used unless it is not applicable owing to incomplete recovery of the retained alkaloids from the filter material (for example, with some types of charcoal filters). The indirect method is not applicable to cigarettes with perforated or porous filter tipping wraps. This International Standard is not applicable in the case of filters having an irreversible nicotine retention and equipped with perforated or porous wrapping.

NOTE 1   These methods determine the retention only of alkaloids of tobacco smoke, expressed as nicotine. The retention of other substances present in the mainstream smoke is not necessarily related to the alkaloid retention.

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 3400:1989, *Cigarettes — Determination of alkaloids in smoke condensates — Spectrometric method.*

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.*
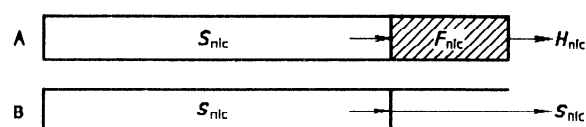
ISO 8243:1991, *Cigarettes — Sampling.*

3 Definition

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

3.1 alkaloid retention index R_{nic} of a cigarette filter: The ratio, expressed as a percentage by mass, of the alkaloids retained by the filter to those entering the filter:

$$R_{nic} = \frac{F_{nic}}{S_{nic}} \times 100$$

The alkaloid retention index may be determined as follows:

a) *Directly*, by measurement of the mass of alkaloids retained by the filter and of the mass of alkaloids in the mainstream smoke leaving the filter:

$$R_{nic} = \frac{F_{nic}}{H_{nic} + F_{nic}} \times 100 = \frac{F_{nic}}{S_{nic}} \times 100$$

where

F_{nic} is the mass of alkaloids retained by the filter;

H_{nic} is the mass of alkaloids in the mainstream smoke leaving the filter;

S_{nic} is the mass of alkaloids entering the filter [see (A) above].

b) *Indirectly*, by measurement of the difference between the mass of alkaloids contained in the mainstream smoke from a cigarette with filter (A) and of the corresponding mass from another cigarette with filter material removed (B), the smoked length of which is the same as that of the filter cigarette (A):

$$R_{nic} = \frac{S_{nic} - H_{nic}}{S_{nic}} \times 100 = \frac{F_{nic}}{S_{nic}} \times 100$$

where

H_{nic} is the mass of alkaloids in the mainstream smoke from the cigarette with filter (A):

S_{nic} is the mass of alkaloids in the mainstream smoke from the cigarette with the filter material removed (B).

4 Principle

4.1 Direct method

Smoking of the filter cigarettes (A), in accordance with ISO 4387 on a routine analytical cigarette-smoking machine complying with the requirements of ISO 3308, and removal of the filter tips from the cigarette butts remaining; subjection of the filter tips, after addition of methanol, to steam distillation from acid solution to remove neutral and acid steam-volatile substances, and discarding of the distillate.

Rendering of the residue in the distillation chamber alkaline by addition of alkali, and steam distillation of the alkaloids; estimation of the alkaloid content by spectrometric measurement of the absorbance of the distillate from the alkaline distillation, and calculation of the alkaloid content as nicotine.

Collection of the mainstream smoke condensate from the filter cigarettes (A), preparation of a methanolic solution of the condensate and determination of its alkaloid content by distillation in accordance with ISO 3400.

4.2 Indirect method

4.2.1 Smoking of the filter cigarettes (A) in accordance with ISO 4387 on a routine analytical cigarette-smoking machine complying with the requirements of ISO 3308, collection of the mainstream smoke condensate, preparation of a methanolic solution of the condensate and determination of its alkaloid content by distillation in accordance with ISO 3400.

4.2.2 Removal of the filter material from a second sample of identical filter cigarettes (A), smoking of the remaining tobacco rods (B) in accordance with ISO 4387 on a routine analytical cigarette-smoking machine complying with the requirements of ISO 3308, collection of the mainstream smoke condensate, preparation of a methanolic solution of the condensate and determination of its alkaloid content by distillation in accordance with ISO 3400.

5 Reagents

Use only reagents of recognized analytical grade and distilled water or water of at least equivalent purity.

5.1 Methanol.

5.2 Sodium hydroxide, solution, $c(NaOH) = 8$ mol/l.

5.3 Sulfuric acid, solution, $c(H_2SO_4) = 1$ mol/l.

5.4 Sulfuric acid, solution, $c(H_2SO_4) = 0,025$ mol/l.

5.5 Nicotine, minimum purity 98 %.

6 Apparatus

Usual laboratory apparatus 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, with glass fibre filter smoke trap (see ISO 4387).

6.3 Steam distillation apparatus, consisting of the following parts:

6.3.1 Distillation chamber.

A cylindrical, vertically mounted distillation chamber of about 50 ml to 100 ml capacity, which has a steam inlet at its base.

It shall be possible to heat the chamber in order to maintain a constant liquid level during the distillation.

6.3.2 Distillation splash head.

6.3.3 Jacketed coil condenser, with spherical joint fitting on to the distillation splash head (6.3.2).

6.3.4 Plug-type funnel, or other system for addition of sodium hydroxide solution and, if required, the filter tips.

6.3.5 Testing of the distillation apparatus.

Test the system in accordance with the indicated procedure (ISO 3400) with pure nicotine solution (5.5) at the maximum expected level. Recovery shall be at least 98 % of the theoretical value. If not, optimize by modification of the distillation rate. For routine tests it is possible to use nicotine salt calibrated against pure nicotine (5.5).

NOTE 2   The diagrams of apparatus currently used (figures 1 to 3) are given as examples. Other apparatus may also be used provided that the results obtained are the same.

6.4 Spectrometer, covering a wavelength range from 230 nm to 290 nm.

6.5 Quartz cells, having an optical path length of 1 cm, or identical matched cells in the case of a single beam apparatus.

The absorbance of the cells shall be equal before and after each measurement; if not, a suitable correction shall be applied.

6.6 Volumetric flasks, of capacity 250 ml, with ground stoppers.

6.7 One-mark pipettes, of capacities 5 ml, 10 ml or 25 ml.

6.8 Glass funnels, of diameter about 55 mm.
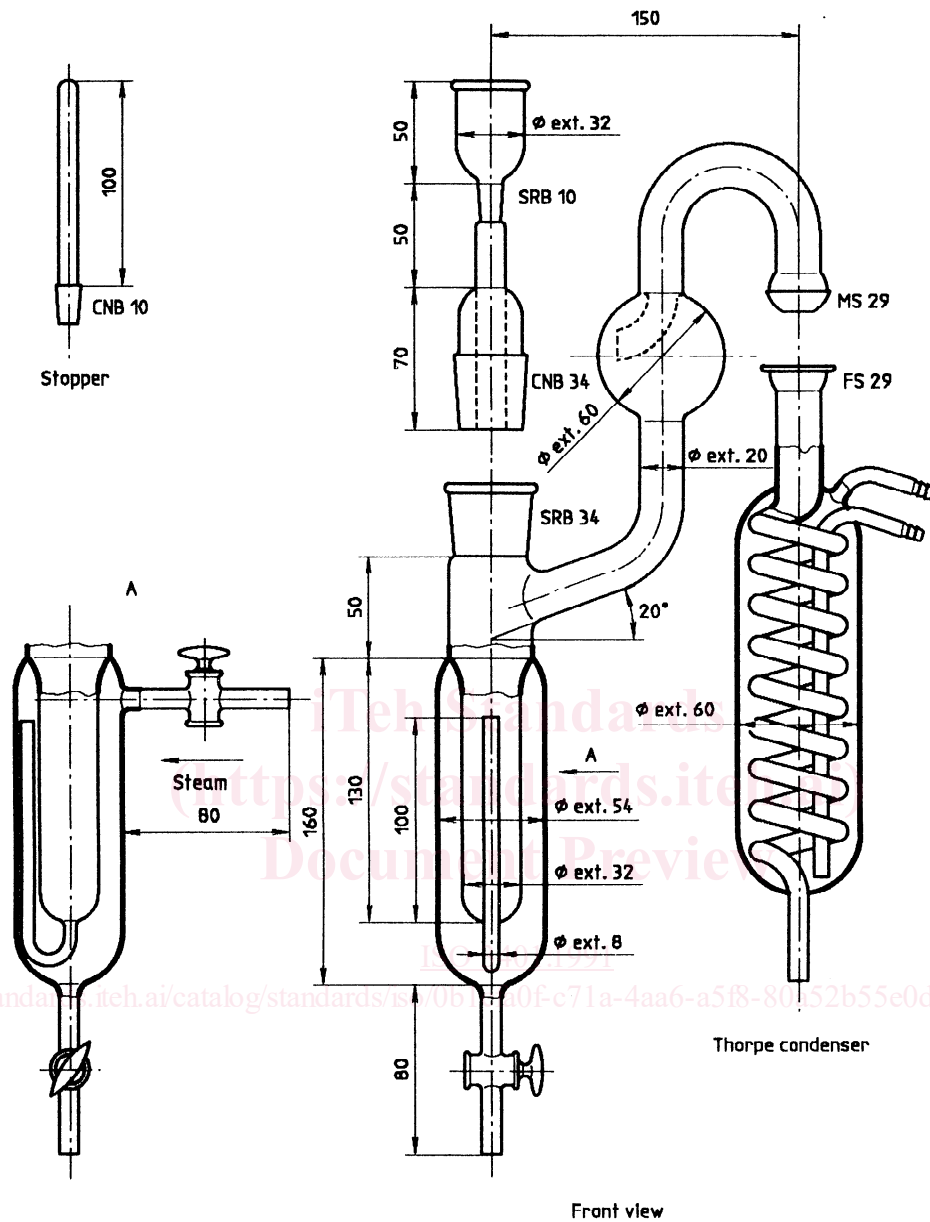
6.9 Filter paper, fast filtering grade.

7 Sampling

Carry out sampling in accordance with the method specified in ISO 8243.

Dimensions in millimetres

NOTES

1 All glass is medium wall borosilicate.

2 Stopcocks: 4 mm bore PTFE.

Figure 1 — Example of apparatus currently in use