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INTERNATIONAL STANDARD ISO 3401

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Tobacco and tobacco products — Determination of alkaloid retention by filters of cigarettes

Tabac et produits du tabac — Détermination de la rétention des alcaloïdes par les filtres des cigarettes

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Tobacco and tobacco products — Determination of alkaloid retention by filters of cigarettes

1 SCOPE AND FIELD OF APPLICATION

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

— the direct method;

— the indirect method.

The methods are applicable to filter cigarettes. Generally the direct method is used. The indirect method shall be used only if the direct method 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.

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

2 REFERENCES

ISO 3308, *Tobacco and tobacco products — Routine analytical cigarette-smoking machine — Definitions, standard conditions and auxiliary equipment.* [1]

ISO 3400, *Tobacco and tobacco products — Determination of alkaloids in cigarette smoke condensates — Spectrophotometric method.*

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

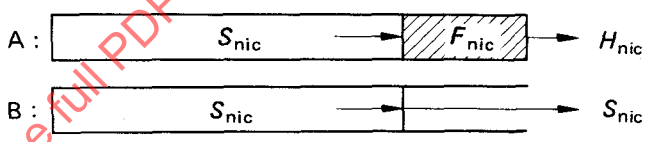
ISO . . ., *Tobacco and tobacco products — Cigarettes — Sampling.* [2]

ISO . . ., *Tobacco and tobacco products — Cigarettes — Machine smoking and determination of crude and dry smoke condensate.* [2]

3 DEFINITION

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 main-stream 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 main-stream smoke;

S_{nic} is the mass of alkaloids entering the filter (in case A above).

1) At present at the stage of draft.

2) In preparation.

7 SAMPLING

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

8 PROCEDURE

8.1 Direct method

8.1.1 *Preparation of the sample*

Select from the laboratory sample prepared in accordance with clause 7 the required number of filter cigarettes for the set of smoking runs to be carried out and condition them in accordance with ISO 3402 in the conditioning enclosure (6.1).

8.1.2 *Determination*

Smoke the cigarettes according to ISO . . . on the routine analytical cigarette-smoking machine (6.2) to the required butt length in one or more smoking runs, collecting the main-stream smoke condensate, in a trap, for each smoking run. As soon as each cigarette has been smoked to the required butt length, extinguish it. At the end of each smoking run, remove the filter tips, carefully free them from any adhering tobacco, and cut them open. Place the filter tips in a distillation flask (6.3.2) and add 20 ml of the methanol (5.1). For each subsequent smoking run, collect the filter tips, treat them in the same way and add them to the distillation flask.

NOTE — The separation of the butts and the extraction of the filter tips with methanol shall be carried out with the minimum of delay after the smoking of each cigarette. If it is not possible to submit the filter tips directly to distillation, extract the filter tips with methanol and make up to a definite final volume. Take an aliquot part of the methanolic solution for subsequent distillations.

8.1.2.1 Determination of alkaloids retained in the cigarette filters (F_{nic})

In the case of direct distillation, shake the mixture of filter tips and methanol in the distillation flask several times. In the case of a prior extraction, take by pipette (6.7) an aliquot part from the methanolic solution and add it to the distillation flask.

Add 10 ml of sulphuric acid solution (5.3), assemble the distillation apparatus (6.3) and start the predistillation under acid conditions. Adjust the rate of distillation to give at least 10 ml of distillate per minute. Do not allow the volume of the liquid in the distillation flask to increase during distillation; use auxiliary heating if necessary.

Stop the distillation when about 100 ml have been collected and discard the distillate. Add slowly 5 ml of the sodium hydroxide solution (5.2) and resume the distillation with a 250 ml volumetric flask (6.6) containing 10 ml of the sulphuric acid (5.3) as receiver. Collect 220 to 230 ml of distillate, dilute to the mark with distilled water, mix and filter, if necessary. Use this solution to determine spectrophotometrically the alkaloids retained in the filter, in accordance with ISO 3400.

8.1.2.2 Determination of alkaloids in the main-stream smoke condensate (H_{nic})

Remove the traps containing the main-stream smoke condensate from the filter cigarettes smoked and introduce the glass fibre filter discs into a flask containing a suitable volume of methanol (5.1). The volume shall be adjusted according to the number of traps and cigarettes smoked so that the alkaloids from 2 to 3 cigarettes are contained in the aliquot part of methanolic solution taken for the distillation, ideally 20 ml. Wipe the inside of the trap and any connecting tubes with a half filter disc and add it to the flask. Close the flask and let it stand overnight. Use this solution to determine spectrophotometrically the alkaloids in the main-stream smoke condensate in accordance with ISO 3400.

8.2 Indirect method

8.2.1 *Preparation of the sample*

Select from the laboratory sample prepared in accordance with clause 7 twice the number of filter cigarettes required for the direct method and condition them in accordance with ISO 3402 in the conditioning enclosure (6.1). Separate the total prepared sample into two identical sub-samples, A and B.

From sub-sample B remove the filter material, leaving the tipping sleeve in place on the cigarettes. If the tipping sleeve has to be removed, replace it with a new tipping sleeve of the same length as the original tipping on the cigarette.

8.2.2 *Determination*

8.2.2.1 Determination of alkaloids in the main-stream smoke condensate of the filter cigarettes of sub-sample A (H_{nic})

Smoke the cigarettes of sub-sample A according to ISO . . . on the routine analytical cigarette-smoking machine (6.2) to the required butt length in one or more smoking runs (N being the number of smoking runs or the number of traps used in one set of smoking runs) and collect the main-stream smoke condensate in a trap for each smoking run.

Remove the traps and introduce the glass fibre filter discs into a flask containing a suitable volume of methanol (5.1). Continue the determination in accordance with 8.1.2.2.

8.2.2.2 Determination of alkaloids in the main-stream smoke condensate of the non-filter cigarettes of sub-sample B (S_{nic})

Smoke the cigarettes of sub-sample B in the same way as those of sub-sample A, ensuring that the length of the cigarette projecting from the cigarette holder of the smoking machine is the same in both sub-samples and that the length of tobacco rod smoked is the same for both sub-samples.