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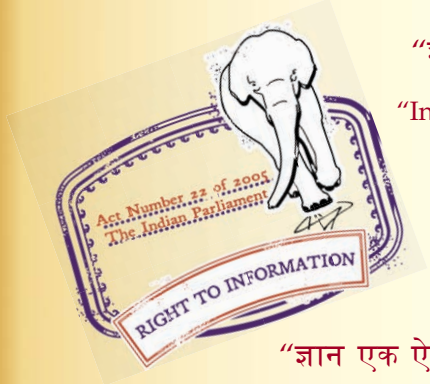
IS 10666 (2009): Cigarette Filters [FAD 4: Tobacco and Tobacco Products]



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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
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"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

तम्बाकू एवं तम्बाकू उत्पाद — सिगरेट फिल्टर — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

TOBACCO AND TOBACCO PRODUCTS — CIGARETTE FILTERS — SPECIFICATION

(First Revision)

ICS 65.160

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 2009

Price Group 8

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Tobacco and Tobacco Products Sectional Committee had been approved by the Food and Agriculture Division Council.

With the growth of indigenous manufacture of cigarettes, the use of filters in the cigarettes is on the increase. It is hoped that this standard would serve the need to regulate the quality of filters that are indigenously produced and thereby enable control on quality of cigarettes that are manufactured.

This standard was originally published in 1983. In this first revision, its provisions are updated to include advances in technology in this area during the last two decades. In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in this field in the country. The figures of the apparatus used for determining various characteristics have also been incorporated in this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

TOBACCO AND TOBACCO PRODUCTS — CIGARETTE FILTERS — SPECIFICATION

(First Revision)

1 SCOPE

This standard specifies the requirements and methods of sampling and test for cigarette filters.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
196 : 1966	Atmospheric condition for testing
4905 : 1968	Methods for random sampling
15795 : 2008	Cigarettes and filter rods – Determination of nominal diameter – Method using a laser beam measuring apparatus

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Butt Length — The length of cigarette remaining after smoking the cigarette, at the moment when smoking is stopped.

3.2 Cigarette Holder — The device for holding the butt end of the cigarette during the smoking procedure.

3.3 Cigarette Position — The position of the cigarette on the smoking machine. In particular it is determined by the angle made by the longitudinal axis of the cigarettes and the horizontal plane when the cigarette is inserted into a cigarette holder in an analytical smoking machine.

3.4 Conditions for Testing and Sample Conditioning — An environment having agreed characteristics (as exactly reproducible as possible) of temperature, relative humidity and pressure, for a specified period of time or, if specified, until the samples or test pieces have reached equilibrium with the conditioning atmosphere, to carry out specified tests in a standard test atmosphere.

3.5 Draw Resistance — Negative pressure which has to be applied to the butt end, under test conditions in order to sustain a volumetric flow of 17.5 ml/s, existing at the butt end, when the cigarette is encapsulated in a measurement device to a depth of 9 mm.

3.6 Filtration Efficiency (FE) — It is the percentage of smoke components retained by a filter as smoke passes through it. FE of a filter is usually measured in terms of its nicotine removal efficiency and/or particulate material removal efficiency and is expressed as percentage. FE depends upon the length, circumference, filtration medium used and pressure drop.

3.7 Free Smoking – The condition that exists when the butt end of the cigarette is completely exposed to the atmosphere between successive puffs, 'a condition that exists in human smoking'.

3.8 Mainstream Smoke — The smoke drawn from the butt end of a lit smoking article during puffing.

3.9 Nominal Diameter of a Cigarette or a Filter — The diameter in millimetres of a metal measuring rod, ground to an accuracy of ±0.005 mm, giving in the same measuring head the same pressure reading (water height h) as the cigarette or filter submitted to the measurement.

NOTE — By convention, it is accepted that the results obtained, define the nominal diameter of cigarettes or filters which may possibly not be perfectly circular.

3.10 Nominal Diameter of a Measuring Head (Dn) — The diameter in millimetre of the metal measuring rod, ground to an accuracy of ±0.005 mm, with which the reading is situation in the centre of the measuring range; this position corresponding to maximum sensitivity.

3.11 Non-ventilated Room — A room in which the speed of the air current in the vicinity of the cigarettes during smoking is not greater than that of a normal convection current.

3.12 Pressure Drop — Static pressure difference between the two ends of the test piece completely encapsulated in a measuring device such that no air can pass through the outer membrane (or wrapping); or a pneumatic circuit when it is traversed by an air flow under steady conditions in which the measured volumetric flow, under standard conditions, at the output end is 17.5 ml/s, at standard conditions as specified in 7.

3.13 Puff Duration — The time in seconds taken from the start to the finish of a puff.

3.14 Puff Frequency — The number of puffs in a given time.

3.15 Puff Number — The number of puffs taken to smoke a cigarette to a specified butt length in a smoking machine.

3.16 Puff Profile — The flow measured directly behind the butt end of the cigarette, and depicted graphically as a function of time.

3.17 Puff Volume — The volume of air and smoke mixture leaving the mouth end of a lit smoking article during one puff.

3.18 Restricted Smoking — The condition that exists when the butt end of the cigarette is closed to the atmosphere between successive puffs, 'a condition that exists in smoking machines'.

3.19 Sidestream Smoke — The smoke which leaves the lit end of the smoking article, especially in between puffs.

3.20 Smoulder Stream Smoke — The smoke which leaves the butt end of a lit smoking article in between puffs (or during the time when the article is not being puffed on).

3.21 Total Dead Volume — The volume of smoke which exists between the butt end of the cigarette and the suction source.

3.22 Ventilated Room — A room having a forced circulation of air.

4 REQUIREMENTS

4.1 Material

4.1.1 *Filter*

The filter shall be made from suitable non-toxic material which may be bonded with non-toxic binder. The filter rods may be made of mono, dual or multiple materials, with or without special/selective absorbing additives. Generally filters are made of viscose staple, acetate tow and/or crimped paper.

4.1.2 *Wrapper*

The filter rod wrapper shall be made of light mass paper of shade/colour and porosity as approved by the purchaser.

4.1.3 *Adhesive*

The adhesives used on the seam of the filter rod wrapper and for glue lines, to bind filter to wrapper, shall be non-toxic.

4.2 Length

4.2.1 The length of the primary filter rods shall be a convenient multiple of the actual length of filter plugs desired to be used on cigarettes by the cigarette manufacturer. The normal trade requirement is 54 to 150 mm.

4.2.2 The variation on filter rod length shall be within ±0.5 mm, or as agreed between manufacturer and supplier.

4.3 Diameter

4.3.1 Since the diameter of filter rods have to be matching with the diameter of cigarettes on which they are to be used by the cigarette manufacturer, it shall be as agreed between the purchaser and the manufacturer. The normal trade requirement is 5.0 to 10.5 mm. It shall be determined as prescribed in IS 15795.

4.3.2 The variation in filter rod diameter shall be within ±0.09 mm.

4.4 Hardness

The hardness of the filter shall be as agreed to between the purchaser and the manufacturer and shall be tested in accordance with method prescribed in Annex A. The normal trade requirement for hardness is 85 to 95 percent.

4.5 Pressure Drop

4.5.1 The pressure drop of filter rods shall be as agreed to between the purchaser and the manufacturer, to suit filtration required. The normal trade requirement is 60 to 350 mm water gauge for a 66 mm filter rod.

4.5.2 The variation in pressure drop of filters shall be within ±15 percent, or as agreed between manufacturer and supplier, when tested in accordance with method prescribed in Annex B.

4.6 Manufacture

4.6.1 *Mass*

The mass of filter rods shall be as agreed to between the cigarette manufacturer and the supplier. The mass variation between the rods shall be not more than ± 6 percent.

4.6.2 *Formation*

The filter rods shall be round and of uniform formation without soft pockets.

4.6.3 *Wrapper*

The wrapper shall be bonded with the filter material and shall be without creases and slackness. The seam shall be firmly bonded.

4.6.4 *Cut-Off*

The filter rods shall have ends which are smooth and cut straight.

4.6.5 *Material Odour*

The filter rods shall be free from odour of the materials employed in making it, since such odour could taint the smoke of cigarettes they are used on.

4.7 Filtration Efficiency

4.7.1 The filtration efficiency, dependent on the type and quality of material used and manner of processing and manufacture of filter rods, shall be as agreed to between the purchaser and the manufacturer. The normal trade requirement is between 15 to 40 percent for 11 mm plug.

4.7.2 The filtration efficiency of the filter rods shall be determined by tests in accordance with method prescribed in Annex C.

5 PACKING AND MARKING

5.1 Packing

The filters should be packed in suitable containers as agreed to between the manufacturer and the purchaser.

5.2 Marking

5.2.1 The following particulars shall be legibly and indelibly marked on each primary as well as bulk container in addition to declarations to be given as required under various statutory Rules and Regulations:

Type of filter,

Number of filters,

Date of manufacture or date code,

Length,

Circumference,

PD, and

Name and address of manufacturer.

5.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING AND CRITERIA FOR CONFORMITY

The sampling and criteria for conformity of the filters shall be done in accordance with Annex D.

7 ATMOSPHERE

7.1 Conditioning Atmosphere

a) Temperature 22 + 1°C, and

b) RH 60 + 3 percent.

7.2 Test Atmosphere

a) Temperature 22 + 2°C, and

b) RH 60 + 5 percent.

ANNEX A
(*Clause* 4.4)

DETERMINATION OF HARDNESS AND RESILIENCE OF FILTERS

A-1 PRINCIPLE AND PROCEDURE

The hardness and resilience of filter rod is tested with the use of a hardness and resilience tester. This tester is designed to measure accurately the deformation in diameter that takes place when a filter rod is subjected to a standard load of 300 g for 15 s, applied through an anvil of 12.7 mm diameter, with vibration, and then the residual deformation after 15 s of removal of the load.

NOTE — Alternatively, a suitable electronic instrument may be used.

A-2 CALCULATIONS

$$\text{Hardness, percentage} = \frac{C - \pi a}{C} \times 100 \text{, and}$$

$$\text{Hardness, percentage} = \frac{C - \pi b}{C} \times 100$$

where

C = circumference of filter rod, in mm;

a = average impression under 300 g load, in mm; and

b = average depression, in mm, after removal of the load.

ANNEX B
(*Clause* 4.5.2)

DETERMINATION OF PRESSURE DROP OF FILTERS

B-1 APPARATUS

B-1.1 The instrument consists essentially of an air compressor, an air filter set to remove water and oil from the compressed air, flow controller and flowmeter, a water manometric column and a suitable holder for the filter rod, connected in line.

B-1.1.1 The specimen holder is so designed that when the filter is inserted into the holder it makes air-tight connection with the instrument without distortion of the rod due to excessive pressure.

NOTE — Alternatively, a suitable electronic instrument may be used.

B-2 PROCEDURE

B-2.1 Condition the test sample and adjust the volumetric air flow on the pressure drop apparatus to the prescribed value of 17.5 ml/s at the standard conditions (*see* 7).

B-2.2 Calibration may be carried out by means of a soap bubble flowmeter or standard pressure drop capillaries that may be provided.

NOTE — Standard pressure drop capillaries, sometimes provided with equipment for quicker calibration, are calibrated at sea level barometric pressure of 1 013 m bar. If the equipment is used at an altitude, the pressure drop value of the standard should be recalibrated, adjusting the flow rate to 17.5 ml/s by means of soap bubble flowmeter.

B-2.3 The position of the specimen during testing may be either horizontal or vertical. On equipments, more commonly used it is horizontal. Where it is vertical it may be mentioned in the test report.

B-2.4 The specimen shall be inserted, by its output end into the holder completely.

B-2.5 In case the paper wrapper on the rod is significantly porous or perforated, the specimen shall be completely inserted into a suitable totally encasing holder provided on the equipment so that no passage of air can be produced through the filter wrapper.

B-2.6 The static pressure differential shall be read on the water gauge manometer when it has steadied.

ANNEX C
(*Clause* 4.7.2)

DETERMINATION OF FILTRATION EFFICIENCY OF FILTERS

C-0 GENERAL

C-0.1 Two methods, direct and indirect, have been prescribed for determination of filtration efficiency in terms of alkaloid retention. A routine analytical cigarette machine as described in Annex E may be used for the test.

NOTE — The methods are applicable to filters attached to cigarettes. The indirect method may be used only if the direct method is not applicable, owing to incomplete recovery of retained nicotine 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.

C-1 PRINCIPLE

C-1.1 Direct Method

This method consists of smoking of the filter cigarettes as per Annex F on a routine analytical cigarette smoking machine, followed by removal of the filter tips from the remaining cigarette butts and submission of the filter tips, after addition of methanol, to steam distillation from acid solution to remove neutral and acid stream — volatile substances. Distillate is discarded.

The residue in the distillation flask is rendered alkaline by addition of strong alkali, and further steam distilled to give nicotine alkaloids. Nicotine alkaloid content is estimated by spectrophotometric measurement of the absorbance of the distillate from the alkaline distillation, and the alkaloid content is calculated as nicotine.

The mainstream smoke condensate from the filter cigarettes is collected, methanolic solution is prepared and its nicotine alkaloid content is determined by distillation in accordance with Annex G.

C-1.2 Indirect Method

This method consist of smoking of the filter cigarettes as per Annex F on a routine analytical cigarette smoking machine, collection of the mainstream smoke condensate, preparation of a methanolic solution of the condensate and determination of its nicotine alkaloid content by distillation in accordance with Annex G.

C-1.2.1 Further the filter material from a second sample of identical filter cigarettes are removed and the remaining tobacco rods are subjected to smoking in accordance with Annex F on a routine analytical cigarette smoking machine. The mainstream smoke condensate is collected, methanolic solution of its condensate is prepared and its nicotine alkaloid content is determined by distillation in accordance with Annex G.

C-2 REAGENTS

C-2.1 Methanol

C-2.2 Sodium Hydroxide, 8 N solution.

C-2.3 Sulphuric Acid, 2 N solution.

C-3 APPARATUS

C-3.1 Conditioning Chamber, *see* 7.

C-3.2 Routine Analytical Cigarette Smoking Machine, complying with the requirement of Annex E.

C-3.3 Steam Distillation Apparatus, consisting of the following:

a) *Distillation Splash Head, see* Fig. 1.

b) *Round Bottom Flask*, 500-ml capacity, short side-neck (*see* Fig. 2).

c) *Jacketed Coil Condenser*, with spherical joint fitting on to the distillation splash head (*see* Fig. 3).

d) *Plug-Type Funnel, see* Fig. 3.

C-3.4 Spectrophotometer, capable of measuring in the range 230 to 290 mm.

C-3.5 Matched Quartz Cells, with an optical path length of 1 cm. The absorbance of the cells shall be equal before and after each measurement; if not, a suitable correction shall be applied.

C-3.6 One-Mark Volumetric Flasks, capacity 250-ml, with ground stoppers of Class A.

C-3.7 One-Mark Pipettes

C-3.8 Glass Funnels, 55 mm diameter.

C-3.9 Filter Paper, fast filtering grade.

C-4 SAMPLING

Form the population of the test batch mentioned in **C-4.1.1** from representative sample of the original population submitted for tests.

NOTE — Cigarettes which show obvious defects should be rejected. Care should be taken to avoid damaging of the cigarettes during handling.

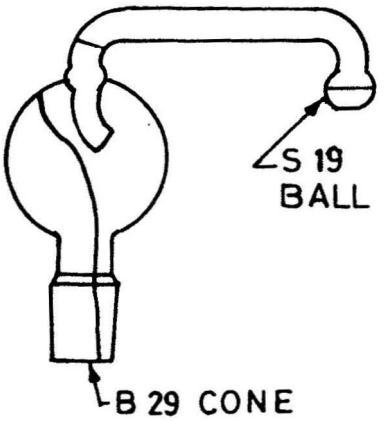


FIG. 1 TYPICAL DISTILLATION SPLASH HEAD

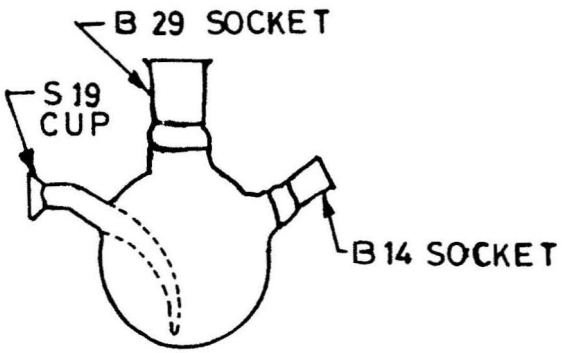


FIG. 2 TYPICAL ROUND BOTTOM FLASK

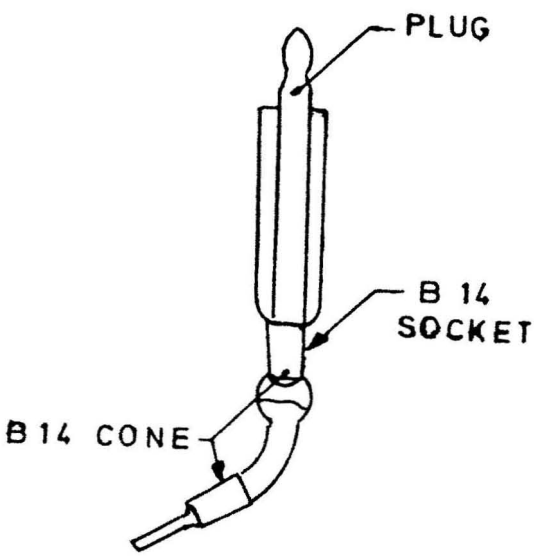


FIG. 3 TYPICAL PLUG TYPE FUNNEL

C-4.1 Procedure

C-4.1.1 *Preparation of Samples*

Two cases are envisaged. The laboratory sample, representative of the population to be studied can be formed,

a) from packets of cigarettes, or

b) from cigarettes in bulk.

C-4.1.1.1 *Laboratory sample formed from packets of cigarettes*

Open the packets and select q cigarettes from each packet. The number q will be chosen in such a way that nq is 200. Sub-sample thus made up is composed of a number of cigarettes which is a multiple of the number of packets n and at least equal to the number of cigarettes necessary for smoking. It is prudent to make up two sub-samples, one of which is reserved for any eventual repetition of the measurements.

C-4.1.1.2 *Laboratory sample formed from the bulk of cigarettes*

If Q number of cigarettes in the bulk is less than 400, calculate the greatest integer in $Q/20$ (integral quotient). Distribute the cigarettes one by one into k lots of 20 cigarettes each and eliminate the remainder. Take two lots at random, of which one represents the final test portion, and the other being reserved for any eventual repetition of the measurements. Prepare the test sample destined for each channel, the cigarettes which comprise them are randomly chosen from the final test portions.

If Q is more than 400, reduce the sample by selecting 400 cigarettes at random. Distribute the cigarettes one by one into two lots of 200 cigarettes; one of the lots represents the final test portion and the other reserved for any future repetition of the measurements.

C-4.1.2 *Selection of Cigarettes*

On the two lots of cigarettes belonging to the prepared laboratory sample (*see* **C-4.1.1**), determine the individual mass accurately up to 0.01 g and nominal diameter (*see* IS 15795). Select 2 sets of 20 cigarettes from the two lots of laboratory samples, falling within ±0.03 mm of the average nominal diameter.

C-5 PROCEDURE

C-5.1 Direct Method

C-5.1.1 *Preparation of Samples*

Selected filter cigarettes prepared in accordance with **C-4**, for the set of smoking runs to be carried

out shall be conditioned in accordance with IS 196 in the conditioning chamber (*see* 7).

C-5.1.2 *Determination*

Smoke the cigarettes according to Annex F, on the routine analytical cigarette smoking machine (*see* **C-3.2**), to the required butt length in one or more smoking runs, collecting the mainstream 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 [*see* **C-3.3**(b)] and add 20 ml of the methanol (*see* **C-2.1**). 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.

C-5.1.2.1 *Determination of alkaloids retained in the cigarette filter*

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 (*see* **C-3.7**) an aliquot part from the methanolic solution and add it to the distillation flask.

Add 10 ml of sulphuric acid solution (*see* **C-2.3**), assemble the distillation apparatus (*see* **C-3.3**) and start the predistillation under acidic 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 (*see* **C-2.2**) and resume the distillation with a 250-ml volumetric flask (*see* **C-3.6**) containing 10 ml of the sulphuric acid (*see* **C-2.3**) 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 Annex G.

C-5.1.2.2 *Determination of alkaloids in the main-stream smoke condensate*

Remove the traps containing the main-stream smoke condensate from the filter cigarettes smoked and place the glass fibre filter discs into a flask containing a small volume of methanol (*see* **C-2.1**). The volume shall be adjusted according to the number of traps and cigarette smoked so that the alkaloids from 2 to 3 cigarettes are contained in the aliquot part of methanolic solution taken for 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 in this solution to determine spectrophotometrically the alkaloids in the mainstream smoke condensate in accordance with Annex G.

C-5.2 Indirect Method

C-5.2.1 *Preparation of Samples*

Select from the laboratory sample prepared in accordance with **C-4** twice the number of filter cigarettes required for the direct method and condition them in accordance with IS 196 in the conditioning chamber (*see* **C-3.1**). 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.

C-5.2.2 *Determination*

C-5.2.2.1 *Determination of alkaloids in the main-stream smoke condensate of the filter cigarettes of sub-sample A*

Smoke the cigarettes of sub-sample A according to Annex F on the routine analytical cigarette smoking machine (*see* **C-3.2**) 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 mainstream 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 (*see* **C-2.1**). Continue the determination in accordance with **C-5.1.2.2**.

C-5.2.2.2 *Determination of alkaloids in the main-stream smoke condensate of the non-filter cigarettes of sub-sample B*

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. Collect the mainstream smoke condensate and prepare a methanolic solution of the smoke condensate as described in **C-5.2.2.1**, separating and adding the

tipping sleeve to the methanolic solution. Determine the alkaloid content of the smoke condensate solution as described in C-5.2.2.1.

C-5.3 Expression of Results

C-5.3.1 *Method of Calculation and Formulae*

Calculation shall be done in accordance with C-5.3.1.1 and C-5.3.1.2.

C-5.3.1.1 Calculate the alkaloid content of the mainstream smoke condensate, expressed in milligrams per cigarette, for each individual smoking run, in accordance with Annex G.

C-5.3.1.2 Calculate the mean alkaloid content of the mainstream smoke condensate per set of smoking runs as the mean of the results obtained for each set of smoking runs in accordance with Annex G.

C-5.3.2 Calculate the mean alkaloid content of the mainstream smoke condensate for the whole test sample as the mean of the results obtained for each smoking run.

C-5.3.3 Calculate the alkaloid content of the smoke condensate retained by the filter, in milligrams per cigarette filter, for each smoking run in accordance with Annex G.

C-5.3.4 Calculate the mean alkaloid content of the smoke condensate retained by the filter, for the whole of the filters of the test sample as the mean of the results obtained for each smoking run.

C-5.3.5 The mean percentage filtration efficiency for nicotine, expressed as the percentage by mass, of all the filters of the test sample is equal to:

a) *Direct Method*

Filtration efficiency, percent $= \frac{F \times 100}{H + F}$

b) *Indirect Method*

Filtration efficiency, percent $= \frac{(S - H) \times 100}{S}$

where

F = mass of nicotine retained by the filter (*see* C-5.1.2.1);

H = mass of nicotine in the mainstream smoke leaving the filter (*see* C-5.1.2.2 and C-5.2.2.1); and

S = mass of nicotine in the mainstream smoke from the cigarette, with filter removed (*see* C-5.2.2.2).

C-5.4 Precision of Results

Express the test results as follows:

Alkaloid content, expressed as nicotine content, of the mainstream smoke condensate, in milligrams per cigarette smoked, to the nearest 0.01 mg for all the filter cigarettes of the test sample.

Alkaloid content, expressed as nicotine content of the smoke condensate retained by the filter, in milligrams per cigarette smoked, to the nearest 0.01 mg for all the filters, of the test sample.

Mean percentage filtration efficiency for nicotine content of the test sample of filters, as a percentage, to the nearest 1 percent. It is desirable to calculate the confidence interval of the mean percentage filtration efficiency for nicotine.

C-6 TEST REPORT

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

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

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

a) Description of the product tested.

b) Sampling procedure, that is,

1) the method of sampling,

2) number and cigarettes in the test sample, and

3) date and place of purchase or sampling.

c) Test conditions in accordance with 7.

d) Test results, expressed in accordance with C-5.4.

1) If obtained by the direct method, the test report shall contain following information:

i) The complete test results;

ii) Alkaloid content F expressed as nicotine content, in milligrams per filter, calculated to the nearest 0.01 mg, giving separate values for each individual smoking run, if available;

iii) Alkaloid content F expressed as nicotine content, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate values for each set of smoking runs;

iv) Alkaloid content H expressed as nicotine content, of the mainstream smoke condensate, in milligrams per cigarette, calculated to the nearest 0.01 mg giving separate values for each individual smoking run, if available;

v) Alkaloid content *H*, expressed as nicotine content of the mainstream smoke condensate, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate values for each set of smoking run;

vi) Filtration efficiency with respect to nicotine as percentage, to the nearest 1 percent separate values for each set of smoking runs; and

vii) Date of the test.

2) If obtained by the indirect method, the test report shall contain following information:

i) The complete test results;

ii) Alkaloid content *H*, expressed as nicotine content, of the mainstream smoke condensate from the filter cigarettes, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate values for each individual smoking run, if available;

iii) Alkaloid content *H*, expressed as nicotine content, of the mainstream smoke condensate from the filter cigarettes, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate value for each set of smoking runs;

iv) Alkaloid content *S*, expressed as nicotine content, of the mainstream smoke condensate from the filterless cigarettes, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate values for each individual smoking run, if available;

v) Alkaloid content *S*, expressed as nicotine content, of the mainstream smoke condensate from the filterless cigarettes, in milligrams per cigarette, calculated to the nearest 0.01 mg, giving separate values for each set of smoking runs;

vi) Filtration efficiencies with respect to nicotine content, expressed as a percentage, calculated to the nearest 1 percent, giving separate values for each set of smoking runs; and

vii) Date of the test.

ANNEX D

(*Clause* 6)

SAMPLING AND CRITERIA FOR CONFORMITY OF CIGARETTE FILTERS

D-1 SCALE OF SAMPLING

D-1.1 Lot

All the bulk containers in a single consignment of the filters constitute a lot.

NOTE — If the consignment is declared to consist of different types of filter rods, the bulk containers belonging to the same kind shall be grouped together to constitute separate lot.

D-1.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements as agreed between the supplier and the user.

D-1.3 Number of bulk containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with Table 1.

D-1.4 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, a random number table (*see* IS 4905) shall be used. If such a table is not available the following procedure shall be adopted:

Arrange all the bulk containers in the lot in systematic manner and starting from any container count 1, 2, 3, ..., etc, up to *r* and so on. Every *r*th bulk container so counted shall be withdrawn to give a sample for the test, where $r = N/n$, N being the number of bulk containers in a lot, and n being the number of bulk containers to be chosen according to Table 1. If r comes out to be a fractional number, its value shall be taken as equal to the integral part of it.

NOTE — In case the bulk container has secondary containers, choose one random secondary container as sample.

Table 1 Number of Containers to be Selected for Sampling

(*Clauses* D-1.3 *and* D-1.4)

Sl No.	Lot Size (*N*)	No. of Bulk Containers to be Selected for Sampling (*n*)
(1)	(2)	(3)
i)	Below 20	2
ii)	21-50	3
iii)	51-200	4
iv)	201-500	5
v)	501-750	6
vi)	751-1 000	8
vii)	1 001 and above	10

D-1.5 From the quantity of bulk containers selected, pick up 200 filter rods at random, representing the total number of bulk containers and divide this quantity into two equal sub-samples (*A* and *B*).

Lot *A* should be used for determination of mass, circumference, pressure drop and hardness and resilience.

Lot *B* should be kept for reference.

D-2 CRITERIA FOR CONFORMITY

A lot of filter rods shall be declared as conforming to the specification when each of the test satisfies the requirements as mentioned in **3**.

ANNEX E
(*Clauses* C-0.1 *and* C-3.2)

ROUTINE ANALYTICAL CIGARETTE SMOKING MACHINE

E-1 SPECIFICATION FOR ROUTINE ANALYTICAL SMOKING MACHINE

E-1.1 The smoking machine shall comply with the standard conditions mentioned below.

E-1.1.1 *Machine Draw Resistance*

The whole flow path between the butt end of the cigarette and the suction source shall offer the least possible draw resistance and the pressure drop shall not exceed 300 Pa (3 m bar).

E-1.1.2 *Puff Duration*

The standard puff duration shall be 2 ± 0.02 s.

E-1.1.3 *Puff Volume*

The standard puff volume shall be 35 ± 0.3 ml. In one puff duration, not less than 95 percent of the puff volume shall leave the butt end of the cigarette.

E-1.1.4 *Puff Frequency*

The standard puff frequency shall be one puff every 60 ± 0.5 s.

E-1.1.5 *Puff Profile*

The puff profile when measured on unlit cigarette shall be bell shaped with a maximum between 0.8 and 1.2 seconds from the start of puffing. The increasing and decreasing parts of the profile shall not have more than one point of inflection each. The maximum flow rate shall lie between 25 and 30 ml/second (*see* **E-8**). The curve may be amputated up to 0.02 seconds from the start (*see* **E-7.2.2**) and Fig. 4.

E-1.1.6 *Restricted Smoking*

An analytical smoking machine shall be a restricted smoker.

E-1.1.7 *Puff Number*

Each individual puff shall be counted and recorded and the puff number rounded off to the nearest one-tenth of a puff on the basis of the puff duration.

E-1.1.8 *Total Dead Volume*

The total dead volume shall be as small as possible and shall not exceed 100 ml.

E-1.1.9 *Butt Length*

The standard butt length in the case of plain cigarettes shall be 23 mm. Filter cigarettes shall be smoked to the following butt lengths:

a) 8 mm over the length of the filter, or

b) 3 mm over the length of the filter overwrap, whichever is greater.

NOTE — The 'filter overwrap' is the external tipping material which joins the tobacco rod to the filter tip.

E-1.1.10 *Cigarette Holders*

The standard cigarette holder shall cover 9 mm, with a standard deviation of (=5 mm, from the butt end of the cigarette and shall be impermeable to smoke components and to air. The standard cigarette holder shall ensure that the leakage between the cigarette and the cigarette holder is not greater than 0.5 percent of the puff volume. The draw resistance of the unlit cigarette shall not significantly increase when it is held in the cigarette holder.

E-1.1.11 *Ambient Conditions*

The difference between the mean puff number of cigarettes smoked in a ventilated room and the mean puff number of cigarettes smoked in a non-ventilated room shall not exceed 3 percent of the mean puff number obtained in the non-ventilated room (with a confidence level of 95 percent). In a non-ventilated room, cigarettes shall not be smoked in their own sidestream smoke.

E-1.1.12 *Cigarette Position*

The angle formed by the longitudinal axis of the cigarette and the horizontal plane shall be as small

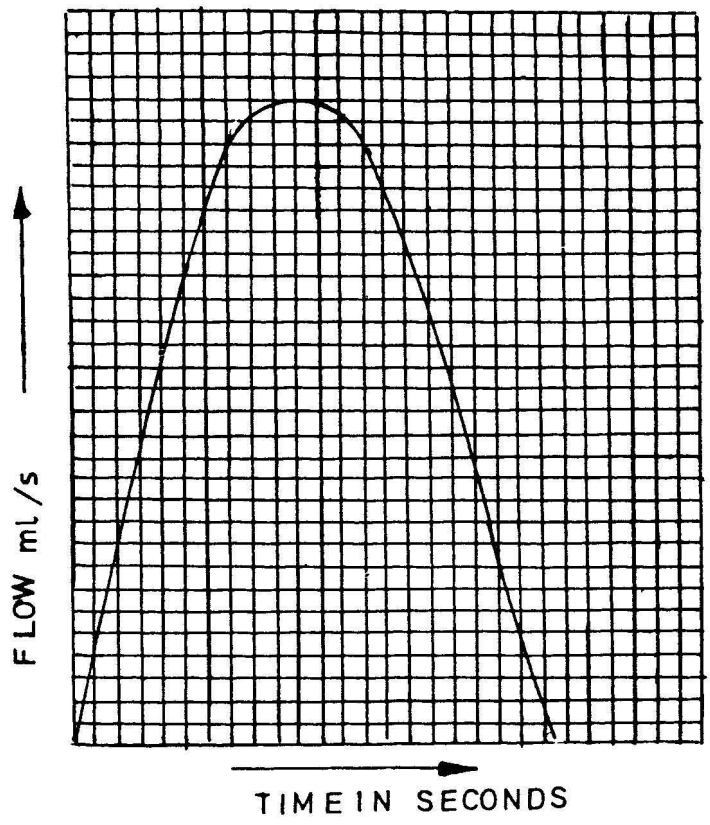


FIG. 4 TYPICAL PUFF PROFILE

as possible, it shall not exceed 10°, if the centre of the butt end is lower than the centre of the other end and 5°, if the centre of the butt end is higher than the centre of the other end.

The ports shall be arranged so that no cigarette influences the burning of any other cigarette.

E-2 . OPERATING PRINCIPLE OF SMOKING MACHINE

E-2.1 The machine shall include a device to draw a fixed volume of air (puff) through a cigarette by applying a difference in pressure between the front (lit) end and the butt end of the cigarette.

E-2.2 The machine shall be a restricted smoker (*see* **E-1.1.6**).

E-2.3 Reliability and Compensation

E-2.3.1 The machine shall contain devices to control the puff volume, the puff duration and the puff frequency.

E-2.3.2 The machine shall possess the mechanical and electrical reliability, necessary to meet the standard conditions regarding these parameters (*see* **E-1.1.2** and **E-1.1.4**) for prolonged periods.

E-2.3.3 The machine shall be capable of sufficient compensation.

When the machine has initially been set to give the standard puff volume of 35 ml without draw resistance, a reduction of not more than 1.5 ml shall be observed when the machine is tested with a draw resistance of 3 000 Pa (30 m bar) and a flow rate of 17.5 ml/s.

NOTE — Compensation is the ability to maintain consistent puff volumes and puff profiles when the pressure drop of the cigarettes changes.

E-2.3.4 The connecting piping between the filter holder (*see* **E-6.1**) and the suction source shall offer the least possible resistance to flow. The draw resistance of the total flow path between the butt end of the cigarette and the suction source shall not exceed 300 Pa (3 m bar) before smoking (*see* **E-1.1**).

E-2.3.5 The total dead volume shall be as small as possible (*see* **E-1.1.8**).

E-2.4 Cigarette Holders and Smoke Traps

E-2.4.1 The machine shall contain devices for holding the cigarette and for trapping on a filter, the smoke produced.

E-2.4.2 The machine shall be equipped to enable the smoke to be trapped on glass fibre filter discs (*see* **E-6.2**) held in the filter holders (*see* **E-6.1**).

E-2.5 The cigarette to be smoked shall be attached to the filter holders (**E-6.1**) by standard cigarette holders (*see* **E-1.1.10**).

E-2.6 The smoking ports to which suction is applied shall be positioned on the machine. The smoking ports shall be designed to hold the filter holders (**E-6.1**) so that the cigarettes are presented in the position (*see* **E-1.1.12**).

The machine shall be designed to prevent losses of smoke constituents between the butt end of the cigarette and the filter holder.

E-2.7 The smoking ports shall be arranged so that the sidestream smoke does not affect cigarettes smoked in adjacent ports (*see* **E-1.1.12**). The distance between the centres of adjacent burning zones shall be at least 50 mm.

E-2.8 Each port shall have its own puff termination device linked to the puff counter (*see* **E-4**) the device may either be,

a) a micro-switch activated by the burning through a cotton thread; or

b) an infra-red detector.

E-2.9 The machine shall be capable of smoking a wide range of cigarettes of different lengths, diameter and cross-sectional shapes while complying with the standard conditions regarding cigarette butt lengths (*see* **E-1.1.9**).

E-2.10 The machine shall be capable of taking one or more clearing puffs after the termination of smoking.

E-3 AMBIENT CONDITIONS

The ambient conditions shall be controlled to ensure that all cigarettes are smoked under identical conditions with regard to ambient air flow. The ambient air flow shall be such that sidestream smoke can be effectively removed without changing the rate of free combustion of the cigarettes during the intervals of time between puffs (*see* **E-1.1.11**).

It is recommended that the linear air speed across the cigarettes should be adjustable to 200 ± 30 m/s.

E-4 PUFF COUNTING

Each port shall have its own puff counter capable of counting to the nearest 0.1 puff (*see* **E-1.1.7**).

E-5 IGNITION

Electrical, gas flame or alcohol flame ignition may be used.

If gas or alcohol flame ignition is used, the flame shall be adjusted so that it does not touch the end of the cigarette before the lighting puff.

NOTE — Electrical ignition is recommended as it has proved satisfactory in practice. If used, electrical lighters shall be hot enough to light the cigarettes at the first attempt. Each lighter should incorporate a locking device to lock it in position once set, that is, 1 mm from the end of the cigarette.

E-6 AUXILIARY EQUIPMENT

E-6.0 Auxiliary equipment shall include the following.

E-6.1 A Filter Holder, made of an airtight and chemically inert material, preferably transparent.

E-6.2 A Filter Disc, of glass fibre material, 1 to 2 mm thick and with a diameter of at least 44 mm. The rough filter surface shall face the oncoming smoke.

E-6.2.1 The filter material shall have the following characteristics:

a) It shall retain at least 99.9 percent of all particles having a diameter equal to or greater than 0.3 micrometers of a dioctylphthalate aerosol at a linear air velocity of 140 mm/s. The draw resistance of the filter assembly shall not exceed 900 Pa (9 m bar). The content of polyacrylate binder shall not exceed 5 percent (*m/m*).

b) The filter assembly shall be capable of quantitatively retaining all the mainstream smoke produced by the cigarettes without loss of crude smoke condensate. In addition, the filter assembly shall be chosen so that the increase in the draw resistance of the assembly does not exceed 250 Pa (2.5 m bar) at a flow rate of 17.5 ml/s when measured after the smoking.

E-6.3 Cigarette holders shall be capable of holding the butt end of the cigarette during smoking. The standard conditions relative to the length of butt covered by this device, the influence of this device on the draw resistance of the cigarette and the air-tightness of the seal are given in **E-1.1.10**. It shall not induce sweating.

A vacuum holder or a labyrinth seal is recommended for attaching cigarettes.

E-6.4 Device for attaching cigarette holders to the machine, be such that the cigarette holders are held rigidly. A screwed fitting or '*O*' ring seal is recommended. Rubber tubing is considered to be unsatisfactory.

E-7 DESCRIPTION OF THE PUFFING MECHANISM OF A PISTON TYPE SMOKING MACHINE

E-7.0 The following description defines an example of use of the piston principle which is a recognized

and proven system but it is not intended to preclude or restrict the future development of smoking machines.

E-7.1 The piston may be:

a) either a conventional piston and crankshaft with connecting rod and small end, *P* (*see* Fig. 5A), or

b) the well-established arrangement in which the cylinder is pivoted at *H* and the piston is connected to the crankshaft (*see* Fig. 5B).

Alternative (b) gives a non-symmetrical profile (*see* Fig. 5B) when measured without a cigarette butt, attains symmetry under smoking conditions. The crankshaft describes a circular path and is driven intermittently by an electric motor with variable speed control.

E-7.2 Special Characteristics

E-7.2.1 *Total Swept Volume*

The total swept volume is the volume of air displaced when the piston passes from the top dead centre to the bottom dead centre. This volume shall be measured immediately at the cylinder inlet.

E-7.2.2 *Puff Volume*

The puff volume is adjusted to 98 ± 1 percent of the total swept volume so as to eliminate the 'skirt' or 'tails' of the puff. To achieve this in relation to the piston movement, the beginning of the puff needs to be retarded and the end advanced, adjustments that can best be achieved by a cam and microswitch assembly or by similar devices.

E-7.3 Design Considerations of Puffing Mechanism

If reasons of symmetry are paramount, *h* should be as large as possible but not less than 10 *r*.

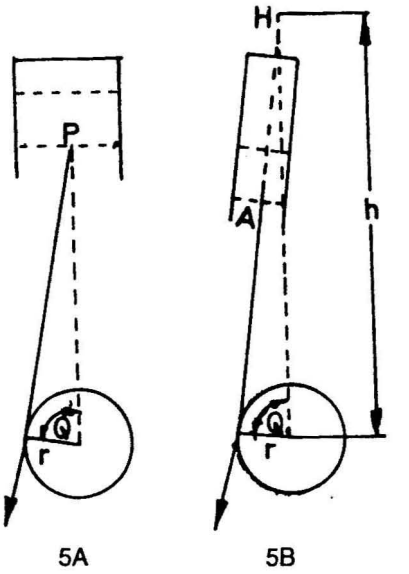
Therefore, in the manufacture of a piston type of smoking machine, the following should apply.

E-7.3.1 The speed of rotation of the shaft shall be constant during puffing, fully adjustable and shall have fine control.

E-7.3.2 The cylinder shall have a bore of 28 ± 1 mm and a stroke that can be varied up to 80 mm. This covers the puff volume, range 0 to approximately 50 ml and gives a bore/stroke ratio of 0.5 at the 35 ml level. This specification may be considered too rigid but it is the one that works very well in practice and gives the recommended puff characteristics.

E-7.3.3 It is desirable that pistons and cylinders should be completely interchangeable.

E-7.3.4 The distance *h* should be greater than 10 *r*.



A = area of curved section,
r = radius of the crank, and
h = distance between the crank and the fist.

FIG. 5 PUFFING MECHANISM

E-7.3.5 The piping between the filter holder and the cylinder should offer the least possible draw resistance (*see* **E-2.3.4**).

E-7.3.6 The dead volume between the cylinder and the filter holder shall be as small as practicable. The total dead volume, including the filter holder, the connecting tubing and the cylinder, when the piston is at top dead centre shall not exceed 100 ml (*see* **E-1.1.8**).

E-7.3.7 In order to ensure that the machine performs according to the specification, incorporation of a mechanism may be necessary to start or stop the piston at a definite point.

E-7.3.8 The puffing mechanism shall have fine control of puff to the nearest 0.1 ml for each port. The suggested range is 2 ml.

E-7.4 Timing Cycle of Solenoid Valve

Each port is fitted with a three-way solenoid valve for the purpose of applying suction to the cigarette.

E-7.4.1 A puffing cycle consists of the following sequence of operations, described below for a piston type smoking machine. Other suitable mechanisms, should nevertheless reproduce exactly the same conditions:

a) With the piston at top dead centre that is when the angle in Fig. 5A and 5B are 0°, the path from piston to cigarette is closed, the path between the piston and the bypass to atmosphere is open.

b) When the angle is between 5° and 10° past top dead centre (to remove leading tail) the path between the piston and the cigarette is open, the path between the piston and the bypass to the atmosphere is closed.

c) When the angle is between 5° and 10° before bottom dead center (to remove trailing tail) the path between the piston and the cigarette is closed, the path between the piston and the bypass to the atmosphere is open.

The vapour phase is then automatically vented to atmosphere as the piston returns to top dead centre.

E-8 DIAGRAMMATIC REPRESENTATION OF A PUFF PROFILE

To illustrate certain definitions and certain standard conditions, the puff profile may be described as follows (*see* Fig. 6).

E-8.1 At time $t = 0$, a pressure difference may be applied to the cigarette. The flow rate ϕ at the butt end of the cigarette varies to give a bell shaped puff profile. The maximum flow rate ϕ m is reached at time t_m. The flow rate then decreases during the puff duration to reach the value ϕ_d at time t_d when the puffing source ceases to apply a pressure difference.

Finally, the flow rate decreases slowly to 0, a value reached at time t_d (end of the puff).

E-8.2 The standard puff profile shall have its maximum so that 25 ml/s $\leq \phi_m \leq$ 30 ml/s at time t_m so that 0.8 s $\leq t_m \leq$ 1.2 s.

The standard puff duration shall be t_d = 2.0 s, and the time t_e shall be limited by the standard puff frequency to t_e = 60 s.

The puff volume v shall be calculated on the basis of the shaded area in Fig. 6, using the formula:

$$V = \int_0^{t_e} \phi(t)\,dt = A + B\int_0^{t_d} \phi(t)\,dt + \int_{t_d}^{t_e} \phi(t)\,dt$$

The standard conditions shall be the following:

$$v = 35\text{ml}$$

$$A = \int_0^{t_d} \phi(t)\,dt \geq 0.95\ v$$

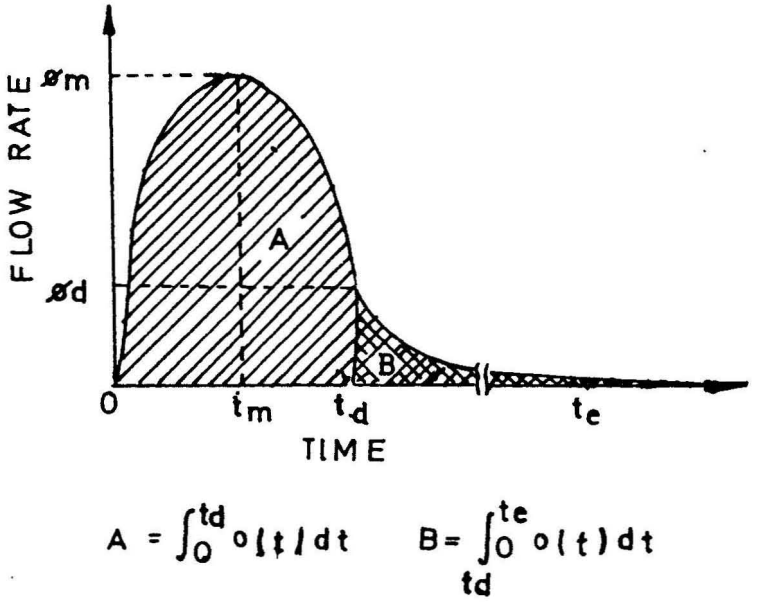


FIG. 6 DIAGRAMATIC REPRESENTATION OF A PUFF PROFILE

ANNEX F

(*Clauses* C-1.1, C-1.2, C-1.2.1, C-5.1.2 *and* C-5.2.2.1)

SMOKING PROCEDURE

F-1 APPARATUS

F-1.1 Standard Automatic Smoking Machine, *see* Annex E.

F-1.2 Soap Bubble Flow Meter, range 0 to 50 ml, graduated in 0.1 ml divisions.

F-2 PROCEDURE

F-2.1 Smoking Plan

Use 20 cigarettes in batches of 5 per each channel, selected as per the sampling procedure (*see* **C-4**).

F-2.2 Butt Markings

The butt length shall be marked on the cigarettes in accordance with **E-1.1.9**.

F-2.3 Preparation of Smoke Traps

For all operations, contamination from the fingers shall be prevented by wearing gloves of a suitable material.

F-2.3.1 *Glass Fibre Filter Discs*

The filter discs shall be inserted in their holders and the holders assembled. If the filter disc material has a rough and smooth side, the rough side should be placed so that it faces towards the cigarettes. After assembly, the filter holders should be examined to confirm that the disc is fitted properly and then exposed to the air in the laboratory overnight.

F-2.4 Setting Up the Smoking Machine — Switch on and allow to warm up on automatic cycling for at least 20 minutes. If draught screens are needed to achieve the standard ambient conditions (*see* **E-1.1.11**) they should be set in place. With the machine warmed up, check that the puff duration and puff frequency on each channel are in accordance with the standard conditions specified in Annex E.

F-2.4.1 *Measurement of Duration of Puff*

A timer working with reference to a crystal controlled oscillator must be used to measure the period of time which elapses between the triggering operations which begin and end a puffing action of the smoking machine.

NOTE — It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of suitable timers and smoking machines available. The accuracy of the timing device must, however, be such as to ensure that the error in the puff duration can be detected.

F-2.4.2 *Checking of Puff Frequency*

A timer must be used to measure the period of time which elapses between the triggering operations which begin successive puffing actions of the smoking machine. This will determine the puff frequency.

NOTE — Although a clockwork stop-watch is sufficiently accurate, it is likely that a crystal oscillator timer will be used. It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of timers and smoking machines available.

Fit the prepared smoking trap or traps and cigarette holders onto the machine. For each channel, attach a resistance equal to the mean draw resistance of the test cigarettes plus 20 mm water gauge and check the puff volume of 35 ± 0.1 ml and read, if necessary (*see* **E-1.1.3**).

F-2.4.3 *Measurement of Puff Volume*

The displacement of the bubble in a soap bubble flow meter gives a direct measurement of puff volume.

NOTE — A suitable indicator must be accurate over a range 0 to 50 ml and shall be graduated in 0.1 ml divisions. It must be connected through a standard resistance (equal to the mean resistance of the cigarettes to be tested plus 20 mm water gauge) to the cigarette holder of the smoking machine channel under test.

Before use on a series of measurements, the instrument must be wetted twice with detergent and then allowed to drain for a period of not less than 30 seconds and not more than 45 s.

The bubble flow meter must contain an aqueous solution of a surface-active agent of adequate concentration.

Repeat determinations must be made until the necessary precision of measurement is obtained.

Measure the temperature and relative humidity of the air in the neighbourhood of the smoking machine and note the atmospheric pressure. Check the ambient conditions (*see* **E-1.1.11**), if there is doubt that air current might be too high.

F-2.5 Smoking Process

Insert the cigarettes into the cigarette holders to the depth standardized in accordance with Annex E and

avoiding any leaks or deformations. Ensure that the cigarettes are positioned according to Annex E; the axis of cigarettes should coincide with the axis of smoke traps.

Adjust the position of the butt mark so that when the burning coal reaches the butt mark, the puff termination device is activated. If the burning through of cotton threads is used to terminate smoking at the butt mark, the cotton should be adjusted so that it just touches the cigarette at the butt mark, without modifying the cigarette positioning.

Zero the puff counters and light the cigarettes at the beginning of the first puff. When the puff mark has been reached, the burning coal must be removed from the cigarette and note the final reading of the puff counters.

New cigarettes should be inserted as soon as possible and the smoking process repeated until the predetermined number of cigarettes, according to the smoking plan, have been smoked into the smoke trap.

ANNEX G

(*Clauses* C-1.1, C-1.2, C-1.2.1, C-5.1.2.1, C-5.1.2.2, C-5.3.1.1, C-5.3.1.2 *and* C-5.3.3)

DETERMINATION OF NICOTINE ALKALOID CONTENT BY DISTILLATION

G-1 PROCEDURE

G-1.0 For complete analysis, two independent determinations shall be carried out under identical conditions.

G-1.1 Distillation

Introduce an aliquot V_A, of the methanolic crude smoke condensate solution (Volume V_K), corresponding to 2 to 3 cigarettes, into the distillation flask. Add 10 ml of the sulphuric acid (*see* **C-2.3**) and about 25 ml of water and start the steam distillation.

Stop distillation when about 100 ml distillate has been collected and discard the distillate. Add slowly 5 ml of sodium hydroxide solution (*see* **C-2.2**) and resume the distillation with a 250-ml volumetric flask containing 10 ml of sulphuric acid, as receiver. Collect 200 to 230 ml of distillate, dilute to the mark with water (Volume V_D) and filter.

The rate of distillation shall be at least 10 to 12 ml of distillate per minute. The volume of the liquid in the distillation flask shall not be allowed to increase during distillation. Auxiliary heating shall be employed, if necessary.

NOTE — The quantities specified in **G-1.1** refer to the type of apparatus used. If other apparatus is used, these quantities may be modified provided that the results obtained are the same.

G-1.2 Determination of Alkaloids in the Distillate

Measure the absorbance of the filtrate at the wavelengths 236 nm, 259 nm and 282 nm against a reference solution of 10 ml of the sulphuric acid (*see* **C-2.3**), diluted to 250 ml with water. If the absorbance at 259 nm exceeds 0.7, dilute Vv ml of the distillate further to V_M ml with the sulphuric acid (**C-2.3**) and measure the absorbance of this solution against a reference solution of the sulphuric acid diluted in the same way.

NOTE — Instead of determining the alkaloids from a crude smoke condensate solution, the determination may be carried out by direct distillation of the alkaloids from glass fibre filters, if these had been used for trapping the smoke condensate. To this effect the appropriate number of filter pads should be introduced into the distillation flask. Add 20 ml of methanol and shake repeatedly. Thereafter the procedure is identical as given above, starting from addition of 10 ml of the sulphuric acid (*see* **C-2.3**).

G-2 EXPRESSION OF RESULTS

G-2.1 Method of Calculation and Formulae

The alkaloid content of the smoke condensate (H), expressed in milligrams per cigarette, for each smoking run is equal to:

$$H = \frac{A \times V_K \times V_D \times V_M}{a \times d \times V_A \times V_V \times Q}$$

where

a = absorptivity (decadic extinction coefficient) of nicotine in 0.05 N sulphuric acid (that is 34.3 at the absorption maximum of 259 nm);

A = corrected absorbance (extinction) calculated from the absorbances measured at 236 nm, 259 nm and 292 nm, that is,

$$A = 1.059 \left[A_{259} \frac{A_{236} + A_{282}}{2} \right]$$

d = optical path length of the cell, in cm;

V_A = aliquot of methanolic smoke condensate solution used for the distillation, in ml;

V_D = volume of distillate from the alkaline distillation, in ml;

V_M = volume to which the aliquot V_V of the distillate was further diluted, in ml;

V_V = aliquot of distillate V_D used for further dilution to V_M ml; and

Q = number of cigarettes smoked into the smoking trap.

G-2.2 Accuracy of Results

Express the alkaloid content of the whole test sample to the nearest 0.1 mg. This content may be expressed to the nearest 0.01 mg if the result accompanied by a statement of the confidence interval of the mean at a probability level of 95 percent.

G-2.3 Test Results

Shall include:

a) nicotine content in milligrams per cigarette smoked, to the nearest 0.01 mg for each individual smoking runs;

b) nicotine content in milligrams per cigarette smoked, to the nearest 0.01 mg for each set of smoking runs;

c) mean nicotine content, in milligrams per cigarette smoked, to the nearest milligrams for the whole test sample; to the nearest 0.01 mg, if the confidence interval is expressed; and

d) date of test.

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Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices: *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14, C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

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