CORESTA RECOMMENDED METHOD N° 13

DETERMINATION OF ALKALOID RETENTION BY CIGARETTE FILTERS

(September 1968)

1. SCOPE

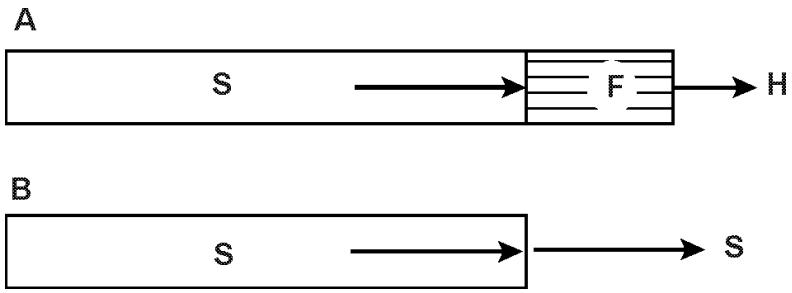
This method is applicable to filter cigarettes.

It measures only the retention of tobacco alkaloid, expressed as nicotine, by the filter. The retention of other substances present in the main smoke stream is not necessarily related to the alkaloid retention.

2. DEFINITIONS

2.1. The alkaloid retention R by a filter is defined as the percentage of the total alkaloid entering the filter that is retained by the filter :

$$R = \frac{F}{S} \times 100 \text{ per cent}$$



2.2. The retention may be determined *directly* by measuring the amount of alkaloid retained by the filter and the amount in the main smoke stream leaving the filter :

$$R = \frac{F}{H + F} \times 100 = \frac{F}{S} \times 100$$

where :

F = alkaloid retained by the filter.

S = alkaloid entering the filter (in case A above).

H = alkaloid in the main smoke stream.

2.3. Retention may also be determined *indirectly* by measuring the difference in alkaloid content of the main smoke streams from a cigarette with filter (A) and from one with filter removed (B). :

$$R = \frac{S - H}{S} \times 100 = \frac{F}{S} \times 100$$

where :

H = alkaloid in the main smoke stream from the cigarette with filter (A).

S = alkaloid in the main smoke stream from the cigarette with the filter removed (B).

Note: The indirect method should be used only when the direct method is not applicable because of incomplete recovery of the retained alkaloid (*e.g.* charcoal filters).

3. PRINCIPLE OF THE METHOD

3.1. *Direct method*

The filter cigarettes are smoked according to CORESTA Recommended Method N° 10. The filter tips are then removed from the remaining tobacco stubs, and the amount of alkaloid in them is determined spectrophotometrically on a steam distillate. The amount of alkaloid in the main smoke stream condensate from these cigarettes is determined according to CORESTA Recommended Method N° 12.

3.2. *Indirect method*

3.2.1. The filter cigarettes (A) are smoked according to CORESTA Recommended Method N° 10 and the alkaloid in the main smoke stream condensate is determined according to CORESTA Recommended Method N° 12.

3.2.2. From a second sample of identical cigarettes the filter tips are removed and the tobacco rods remaining (B) are smoked according to CORESTA Recommended Method N° 10. The alkaloid in the main smoke stream condensate is determined according to CORESTA Recommended Method N° 12.

4. SAMPLING

The sampling method shall be appropriate for the nature of the test and the product tested. A detailed recommended for sampling procedure is in preparation.

5. APPARATUS

5.1. Steam distillation apparatus consisting of the following single parts (or any other apparatus giving the same results).

5.1.1. Round bottom flask, 500 ml, short centre neck and side necks (see figure 1).

5.1.2. Distillation splash head (see figure 2).

5.1.3. Jacketed coil condenser with spherical joint fitting 5.1.2.

5.1.4. Plug-type funnel for sodium hydroxide addition (see figure 3).

5.2. Spectrophotometer, covering the wavelength range 230-290 nm.

5.3. Quartz cells, path length 10 mm.

5.4. Volumetric flasks, 250 ml, narrow neck type with ground stopper.

5.5. Pipettes, analytical.

5.6. Funnels, 55 mm diameter.

5.7. Filter paper.

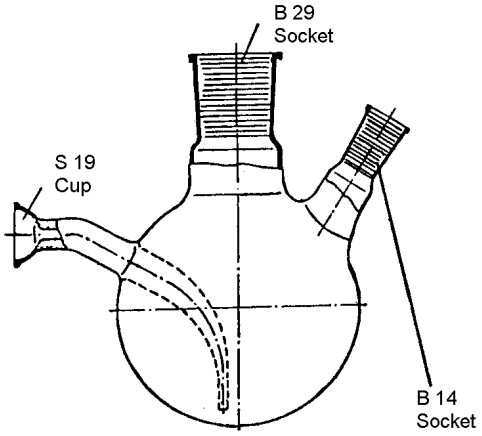


Figure 1

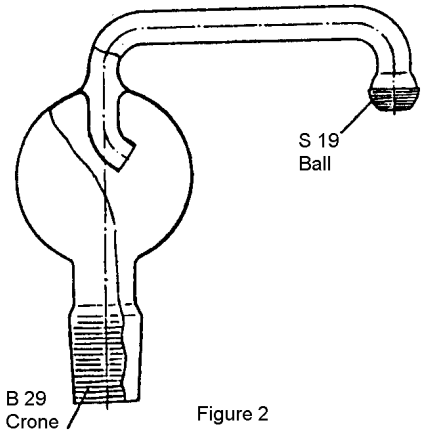


Figure 2

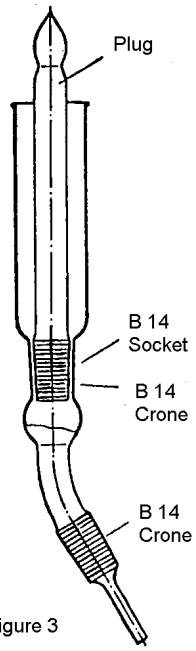


Figure 3

6. REAGENTS

Analytical grade reagents should be used.

6.1. 8 N Sodium hydroxide solution.

6.2. 2 N Sulphuric acid.

6.3. 0.05 N Sulphuric acid.

6.4. Methanol.

7. DIRECT METHOD FOR THE DETERMINATION OF ALKALOID RETENTION

7.1. *Sample preparation*

Smoke the filter cigarettes according to CORESTA Recommended Method N° 10. Extinguish the stubs, remove the filter plugs and free them from any adhering tobacco.

7.2. *Procedure*

Make two independent determinations on different days.

Cut open the filter plugs from one smoking run according to CORESTA Recommended Method N° 10 (5 to 20 plugs), place them in the distillation flask and add 20 ml methanol. Shake repeatedly. Add 5 ml 2 N sulphuric acid, assemble the distillation apparatus ans start the pre-distillation under acid conditions. Keep the volume in the distillation flask constant by auxiliary heating if necessary. Stop the distillation when about 100 ml has been collected and discard the distillate.

Add 10 ml 8 N sodium hydroxide and resume the distillation with a 250 ml volumetric flask containing 15 ml 2 N sulphuric acid as receiver. Collect 220-230 ml of distillate, make up to the mark with distilled water (volume VD = 250 ml), mix, and filter.

Measure the absorbance of the filtrate at 236 (A_{236}), 259 (A_{259}), and 282 (A_{282}) nm against a reference solution of 15 ml 2 N sulphuric acid diluted to 250 ml with distilled water.

If the absorbance at 259 nm exceeds 0.7, dilute V_V ml of the filtered distillate further to V_M ml with 0.05 N sulphuric acid and measure the absorbance of this solution against a sulphuric acid reference diluted in the same way.

7.3. *Calculation*

7.3.1. Calculate the retained alkaloid F_{nic}, as mg nicotine, per filter :

$$F_{nic} = \frac{A \cdot V_D \cdot V_M}{a \cdot d \cdot V_V \cdot n} \qquad A = 1.059 \left(A_{259} - \frac{A_{236} + A_{282}}{2}\right)$$

where :

a = absorptivity (decadic extinction coefficient) of nicotine in 0.05 N sulphuric acid, *i.e.* 34.3 at 259 nm.

A = corrected absorbance (extinction), cf. CORESTA Recommended Method N° 20 (formula 2).

d = optical path length, cm.

n = number of filter plugs taken for distillation.

V_D = volume of distillate from the alkaline distillation.

V_V = aliquot of distillate taken for further dilution to V_M.

V_M = volume to which the aliquot V_V of the distillate was further diluted.

The difference between determinations on two single smoking runs should agree within 10 per cent of the smallest value. If not, further determinations should be made until this requirement is fulfilled.

7.3.2. Calculate the percentage alkaloid retention R_{nic} by the filter :

$$R_{nic} = \frac{F_{nic}}{H_{nic} + F_{nic}} \text{ x } 100$$

where :

F_{nic} = nicotine content in mg per filter plug.

H_{nic} = nicotine content of the main smoke stream condensate from the filter cigarettes in mg per cigarette as determined according to CORESTA Recommended Method N° 12.

The difference between two single determinations should agree within 3 per cent retention. If not, further determinations in pairs should be made until this requirement is fulfilled.

8. INDIRECT METHOD FOR THE DETERMINATION OF ALKALOID RETENTION

8.1. *Sample preparation*

Use a test sample of filter cigarettes selected according to CORESTA Recommended Method N° 10.

Prepare identical subsamples A and B of the test sample.

8.1.1. The cigarettes A are the selected but otherwise unmodified filter cigarettes.

8.1.2. Remove the filter plugs from cigarette subsample B leaving the filter tipping material in place on the cigarettes. If the tipping material must be removed replace with a new tipping forming a hollow sleeve on the end of the cigarette.

8.2. *Procedure*

8.2.1. Smoke the filter cigarettes A according to CORESTA Recommended Method N° 10 and determine the main smoke stream alkaloid content, H_{nic}, according to CORESTA Recommended Method N° 12.

8.2.2. Smoke the filterless cigarettes B according to CORESTA Recommended Method N° 10 ensuring that the length projecting is the same and the length of cigarette smoked is the same as for cigarettes A. Add the tipping to the methanolic solution of smoke condensate and determine the main smoke stream alkaloid content, S_{nic}, according to CORESTA Recommended Method N° 12.

8.3. *Calculation*

Calculate the percentage alkaloid retention R_{nic} by the filter from :

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

where :

H_{nic} = nicotine content of the main smoke stream condensate from the filter-cigarettes in mg per cigarette (as determined according to CORESTA Recommended Method N° 12).

S_{nic} = nicotine content of the main smoke stream condensate from the filterless cigarettes in mg per cigarette (as determined according to CORESTA Recommended Method N° 12) .

The difference between two single determinations should agree within 5 per cent retention. If not, further determinations in pairs should be made until this requirement is fulfilled.

9. REPORT

The report on the results should include reference to this Recommended Method and :

9.1. Description of the product tested, cf. CORESTA Recommended Method N° 10, paragraph 10.1.

9.2. Sampling procedure :

a) sampling method ;

b) number of samples ;

c) date and place of purchase.

9.3. Test conditions, cf. CORESTA Recommended Method N° 10.

9.4. Test results.

9.4.1. If obtained by the direct method :

a) as stated in CORESTA Recommended Method N° 10, paragraph 10.4 ;

b) nicotine content F_{nic} in mg per filter plug to the nearest 0.01 mg ; separate values for each smoking run ;

c) nicotine content H_{nic} of the main smoke stream condensate in mg per cigarette to the nearest 0.01 mg ; separate values for each smoking run ;

d) alkaloid retention R_{nic} in per cent to the nearest one per cent ;

e) date of test.

9.4.2. If obtained by the indirect method :

a) as stated in CORESTA Recommended Method N° 10, paragraph 10.4 ;

b) nicotine content H_{nic} of the main smoke stream condensate from the filter cigarettes in mg per cigarette to the nearest 0.01 mg ; separate values for each smoking run ;

c) nicotine content S_{nic} of the main smoke stream condensate from the filterless cigarettes in mg per cigarette to the nearest 0.01 mg ; separate values for each smoking run ;

d) alkaloid retention R_{nic} in per cent to the nearest one per cent ;

e) date of test.