

# **Guideline for Measurement Method of Harmful Ingredients in Cigarette-type Smoking Craving Suppressant**

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**Korea Food & Drug Administration**

## **I. Introduction**

Due to the recent increased interest in smoking cessation, social interest for a cigarette-type smoking craving suppressant is getting increased as well. The smoking craving suppressant is literally the agent used for purpose of decreasing wanting to smoke which is managed as a quasi-drug. The KFDA reviews the safety, efficacy, standards, and analytical methods by item to grant approval. Regarding the cigarette-type smoking craving suppressants, the criteria are set for harmful ingredients including nicotine, tar, carbon monoxide, etc. and the products are managed. For tar and carbon monoxide, they should not exceed 10 mg per cigarette, respectively, and nicotine should not be detected.

Therefore, upon the test methods for ‘carbon monoxide’ and ‘nicotine’ in ‘Guideline for Measurement Method of Harmful Ingredients of Cigarette-type Smoking Craving Suppressant’ published in 2011, test methods for ‘tar’ are added and revised. The revised guideline will be provided to regulatory agency and manufacturers as reference.

## **II. Measurement of Nicotine in Smoke Condensate of Cigarette-type Agent - Gas Chromatography**

### **1. Application Scope**

This guideline regulates the method of measuring nicotine content in smoke condensate of cigarette-type smoking craving suppressant using gas chromatography.

### **2. Principle**

Dissolve the smoke condensates collected from the main stream smoke of smoking craving suppressant in the solvent in which internal standards are included. The content of nicotine contained in a certain amount of

solution is measured by gas chromatography, which is able to calculate a total nicotine content in smoke condensates.

### **3. Reagent**

#### **3.1 Internal Standard**

N-heptadecane or quinaldine (at least 99% of purity), carvone, n-octadecane or other internal standards should be used after purity is evaluated, and they should be used after measuring whether they are co-eluted with other ingredients included in smoke condensates. The peak areas of the internal standards contained in the sample should be recorded consistently. If consistency is not shown, it must be identified whether there are peaks that are eluted at the same time as the peak of the internal standards among the peaks eluted from the smoke extracts when smoke samples are extracted and analyzed with the extracted solution that does not contain internal standards.

#### **3.2 Extracting Solvent**

As propan-2-ol (1.0 mg/mL of the maximum water content) containing internal standards (3.1) with adequate concentration, the domain of 0.2 mg/mL ~ 0.5 mg/mL is generally good. With regard to the solvents stored in a laboratory which cannot control temperature, use it after controlling the temperature of the solvent to  $22 \pm 2^{\circ}\text{C}$ .

### **4. Test Method**

With the given number of cigarette-type smoking craving suppressants, dissolve 20 mL of the smoke condensates obtained by smoking it an automatic smoking device equipped with glass fiber filter collector in a 44 mm glass fiber (discs) or a certain amount of extracting solvent applicable to 50 mL in a 92 mm glass fiber, and use it as the test solution. Here, check whether glass fiber sinks in the solvent sufficiently. Dissolve the separate nicotine standard preparation in extracting solvent, and prepare 4 and more standard solutions with concentration ranged from 0.02 mg/mL ~ 2.0 mg/mL. Avoid direct sunlight, and store the test and standard solutions

at 0°C ~ 4°C. The temperature of the solvents and solutions stored at low temperature should be adjusted to  $22 \pm 2^\circ\text{C}$  before use. Test 2  $\mu\text{l}$  each of test and standard solutions according to gas chromatography, and use the calibration curve obtained in the peak of the standard solution to calculate the content of nicotine in the test solution. Calculate the content of nicotine in smoke condensates from nicotine concentration in the test solution, and convert the combusted sample volume to express the amount of nicotine per cigarette in mg unit.

### <Operation Condition>

Detector: flame ion detector

Column: With internal aperture 2 mm ~ 4 mm and length 1.5m ~ 2 m, the column should be made of glass, but inactive stainless steel or the one made of nickel can also be used. Stationary Phase: The size of silanized supporting materials treated with acid is from 150  $\mu\text{m}$  (100 mesh) to 190  $\mu\text{m}$  (80 mesh), added by 10% PEG 20000 and 2% potassium hydroxide)

Column temperature: 170°C(isothermal)

Inlet temperature: 250°C

Detector temperature: 250°C

Transporting gas: Helium or nitrogen

Flow rate: 30 mL/min

System suitability

The performance of system: Solvent peak, internal standard peak, nicotine peak, and peak of other smoke ingredients, especially neopatadine (depending on situations, the neopatadine peak may occur in nicotine peak tail part.) To allow peak to come out well, set the condition to have 6 ~ 8 minutes of analysis time.

### **III. Measurement of Carbon Monoxide in Smoke of Cigarette-type Agents - NDIR**

#### **1. Application Scope**

This specification regulates the method of measuring carbon monoxide in vapor of cigarette-type smoking craving suppressant.

#### **2. Principle**

Collect vapor phase in cigarette smoke when cigarette is smoked, use non-dispersive infrared analyser (NDIR) adjusted by carbon monoxide to measure carbon monoxide, and calculate the volume of carbon monoxide per cigarette.

#### **3. Equipment**

**3.1 Vapor phase collection system:** The instrument to fix in one or more channels of automatic smoking equipment. Using this collecting system, it must be possible to store all vapor phases (emitted to atmosphere in general) in a container which was emptied in advance for consecutive collection of samples through NDIR analyser. The collecting system should not bother the operation of the automatic smoking equipment and the measurement of total particular matters and nicotine. The non-permeability of the gas collection instrument for vapor phase ingredients should be identified by using the vapor phase including 4 ~ 6% (v/v) carbon monoxide. The concentration of carbon monoxide should be measured immediately after filling the gas collecting instrument which was emptied in advance. Even if 2 hours and more are passed after the collection, the concentration of carbon monoxide in vapor phase of the collected instrument should not be different by not more than 0.2% compared to the value obtained from the initial measurement. When bag is used as the gas collection instrument, the size of the bag should be big enough, so that the final pressure of the bag after the collection should not exceed the atmospheric pressure.

The volume of the bag should not be twice bigger than the volume of gas being collected under atmospheric pressure. Practically, when vapor phase is collected from 5 cigarettes, the bag with 3 L volume is needed, and when vapor phase is collected from 20 cigarettes, the bag with 10 L volume is needed.

**3.2 Non-dispersive infrared (NDIR) analyser:** It should be selective for measurement of monoxide in vapor or gas, and adjusted. NDIR analysers which are produced by a number of manufacturers can be used, but the analyser used should have 0 ~ 10% (V/V) of operation range as the range of carbon monoxide concentration range, and the speed of sample collection should be within the range of 0.5 ~ 2 L/min, if possible. The analyser should show 1% precision compared to full scale, 1% linearity, and 0.2% repeatability under certain temperature and certain pressure condition. The sensitivity for 10% (V/V) carbon dioxide of the analyser should not exceed the sensitivity for 0.05 (V/V) carbon monoxide. The sensitivity for 2(V/V) vapor of the analyser for carbon dioxide should not exceed the sensitivity for 0.05 (V/V) carbon monoxide.

**3.3 Ignition Instrument:** The ignition instrument without flame must be used. Ignite the cigarette in the initial attempt without contacting an (electric) lighter to a cigarette or burning it in advance.

**3.4 Pressure gauge:** The one that can measure atmospheric pressure up to 0.1 kPa.

**3.5 Thermometer:** The one that can measure temperature up to 0.2°C.

**3.6 Standard mixed gas:** Make-up gases in addition to nitrogen, that is, helium, etc. should not be used because they make the sensitivity of detection of carbon monoxide different. NDIR analyser should be adjusted by using at least 3 and more standard mixed gases of which concentrations are accurately known with relative error within 2%. With regard to the range of carbon monoxide concentration of standard mixed gases, the expected samples should include concentrations so that calibration curve extrapolation should not be needed. Generally, the standard gas in which carbon monoxide is mixed at 1, 3 and 5% (V/V) level in nitrogen is adequate.

## **4. Test Method**

### **4.1 Conditioning**

Condition cigarette samples at mean temperature  $22(\pm 1)^{\circ}\text{C}$ , mean relative humidity 60% for 48 hours, and identify whether equilibrium is made appropriately.

The atmosphere of the laboratory in which smoking is implemented should maintain temperature  $22 \pm 2^{\circ}\text{C}$  and relative humidity  $60 \pm 5\%$ . Keep the conditioned test samples in a sealed container (big enough to contain test pieces), and take out immediately before smoking.

### **4.2 Correction of NDIR Analyser**

Warm up the device according to the direction of the manufacturer, and use air to clean the device, and then adjust the calibration to 0.

Fill vapor phase collection container which was emptied in advance with the standard mixed gas with carbon monoxide concentration 5% (V/V), empty it, and then fill it with the same gas again. The temperature and pressure of the gas filled in the container should be same as the surrounding temperature and pressure. Inject the gas of the collection container into the measuring cell using a sample collection pump in the system, but maintain the pressure for 5 ~ 10 seconds to have equilibrium in the analyser. When certain values are obtained, record the values read in the concentration measuring instrument in the analyser.

Repeat the procedures described above in at least 2 and more other standard mixed gases. If the difference of carbon monoxide concentrations between the observed and expected values is greater than 0.2% (V/V), be careful with the linearity of the instrument.

Use the standard gases to reconfirm the device at least once a week. The calibration curve should be linear in the range reported in 3.2.

Use the standard gas in which about 5% (V/V) carbon monoxide is included to check the calibration curve before the actual measurement. If carbon monoxide concentration difference between the observed and expected values is greater than 0.2% (V/V), repeat the entire correction.

### 4.3 Smoking and Collection of Vapor Phase

The vapor phase collection instrument should be cleaned using surrounding air, and then completely emptied before smoking procedure begins. There should not be decompressed status in the collection instrument before smoking begins, and the flow toward reverse direction due to the decompression should not occur as well.

Smoke the testing cigarette in the automated smoking instrument equipped with glass fiber filter collector, and after smoking is completed, remove cigarette butt. Conduct 5 blank smokings per trap.

### 4.4 Measurement of Volume Concentration of Carbon Monoxide

Check the calibration curve in the analyser, and then inject the vapor phase in the measuring cell in the analyzer under the same condition as the surrounding temperature and pressure conditions. Read the concentration of carbon dioxide displayed in the analyser.

## 5. Calculation of Results

Calculation of mean volume of carbon monoxide per cigarette. The mean volume of carbon monoxide per cigarette should be calculated by using formula (1).

$$V_{as} = \frac{C \times V \times N \times p \times T_0}{S \times 100 \times p_0 \times (t + T_0)} \dots\dots\dots (1)$$

Here,  $V_{as}$ : Mean volume of carbon monoxide per cigarette(mL)

C: Reference percentage of measured volume of carbon monoxide

V: Smoking volume (mL)

N: Number of measured smoking of sample including blank smoking

P: Surrounding pressure (kPa)

$P_0$ : Standard atmospheric pressure (kPa)

S: Number of cigarettes smoked

$T_0$ : Temperature of triple point of water (K)

t: Surround temperature, and its unit is Celsius



V = 35mL, when substituting the values of P<sub>0</sub>(101.3 kPa) and T<sub>0</sub>(273K) which were rounded off to the nearest integer, formula(1) will be as follows.

$$V_{as} = 0.9432 \times \frac{C \times N \times p}{S \times (t + 273)} \dots\dots\dots(2)$$

The mean mass of carbon monoxide per cigarette should be calculated using formula (3).

$$m_{cig} = \frac{C \times V \times N \times p \times T_0 \times M_{CO}}{S \times 100 \times p_0 \times (t + 273) \times V_m} \dots\dots\dots(3)$$

Here, m<sub>cig</sub>: mean mass of carbon monoxide per cigarette(mg)

M<sub>CO</sub>: mol mass of carbon monoxide(g/mol)

V<sub>m</sub>: mol volume of abnormal gas(L/mol)

V = 35mL, when substituting the values of P<sub>0</sub>(101.3 kPa) and T<sub>0</sub>(273K) which were rounded off to the nearest integer, formula(1) will be as follows.

$$m_{cig} = 1.179 \times \frac{C \times N \times p}{S \times (t + 273)} \dots\dots\dots(4)$$

## IV. Tar Test Method-Measuring Method of Total Particular Matter and Tar in Cigarette-type Agent

### 1. Application Scope

This test method regulates the method of measuring tar existing in the collected smoke after igniting cigarette-type smoking craving suppressant using an automated smoking equipment.

### 2. Principle

Smoke cigarette-type smoking craving suppressant in an automated smoking equipment which collects total particulate matter [hereinafter TPM, the part of the main stream smoke collected by smoke collector expressed in mg unit per cigarette] using glass fiber filter collector. The mass of the collected TPM is determined by the principle of gravity. Extract the TPM from the collector, and measure water content and nicotine content using gas chromatography. By excluding the amount of water and nicotine from the amount of TPM, calculate the amount of tar, and express in mg per cigarette.

### **3. Equipment**

#### **3.1 Automated Smoking Equipment for Analysis**

#### **3.2 Soap-bubble flow meter**

A measuring instrument of 35 mL with gradation unit of 0.1 mL. Its accuracy is  $\pm 0.2$  mL

#### **3.3 Instrument to measure puff duration and cycle**

#### **3.4 Analytical Scale** Can measure by 0.1 mg

**3.5 Sealed container which can be conditioned** Can maintain condition of temperature  $22 \pm 1^{\circ}\text{C}$  and relative humidity  $60 \pm 3\%$ .

#### **3.6 Instrument to measure length** Can measure up to 0.5 mm.

#### **3.7 Instrument to measure diameter**

**3.8 Smoke Collector Sealing Device** A stopper should be made of the material that is not absorptive, and it should be chemically non-active material.

### **4. Test Method**

#### **4.1 Preparation of Sample**

To prepare the situation where samples are damaged, additional tests are required, or selection is needed according to mass or resistance of suction (or other variables) if necessary, prepare sufficient samples of which test results are reliable.

## **4.2 Marking Butt Length**

**4.2.1 Standard Butt Length<sup>1)</sup>** The standard butt length marked in cigarette-type smoking craving suppressant should be the longest one out of the 2 given lengths in the following.

- ① Filter length<sup>2)</sup> + 8 mm or ② overwrap<sup>3)</sup> length + 3 mm

**4.2.2 Measuring Filter Length** The length of filter defined in 4.2.1 means the average value measured by using a measuring instrument for length of 10 cigarette-type smoking craving suppressants prepared as samples. The average should be marked up to 0.5 mm, and if necessary, they may be measured with 10 and more cigarette-type smoking craving suppressants.

**4.2.3 Measuring Overwrap Length** The length of overwrap defined in 4.2.1 means the average value measured by using a measuring instrument for length of 10 cigarette-type smoking craving suppressants prepared as samples. The average should be marked up to 0.5 mm, and if necessary, they may be measured with 10 and more cigarette-type smoking craving suppressant.

**4.2.4 Marking Butt Length before Conditioning** Depending on the type of cigarette-type smoking craving suppressant, draw a line of standard butt length using a marker with a soft tip with accuracy of 0.5 mm unit from the tip of the mouth. While marking the length of the butt, be careful not to damage the cigarette-type smoking craving suppressant. If cigarette-type smoking craving suppressant are damaged or holes are made while marking it, or defects are found, discard all of them and replace with other samples. If the cigarette is smoked by the automated smoking equipment in which butt length is set in advance, it is not necessary to mark the butt length in cigarette-type smoking craving suppressant themselves.

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1) The butt length is the length of a cigarette-type smoking craving suppressant not burned and remained when smoking is stopped

2) The butt length is the length of a cigarette-type smoking craving suppressant not burned and remained when smoking is stopped

3) Overwrap means all wrappers applied to the tip of mouth of cigarette-type smoking craving suppressant

**4.3 Selection of Cigarette -type Smoking Craving Suppressant** If selection according to mass or suction resistance (or other variables) is required, the selection should not be considered as the way to decrease the number of cigarette-type smoking craving suppressant being smoked.

**4.4 Conditioning** Condition all samples in the environment conditions controlled to maintain temperature  $22 \pm 1^{\circ}\text{C}$  and relative humidity  $60 \pm 3\%$  for at least 48 hours and up to 10 days, and identify whether equilibrium is made appropriately. For any reasons, if the cigarette-type smoking craving suppressant should be stored for more than 10 days before conditioning, it should be stored in original packaging or a container in which enough quantity of samples can be contained and not ventilated. The experiment environment of the laboratory in which smoking experiment is conducted should maintain temperature  $22 \pm 2^{\circ}\text{C}$  and relative humidity  $60 \pm 5\%$ . The atmospheric pressure should be measured, and if the range deviates  $86 \sim 106 \text{ kPa}$ , it should be marked in the study report. The locations of smoking and condition are not adjacent each other or not with the same environment, move the samples contained in a sealed container to the location of smoking.

#### **4.5 Preliminary Test before Smoking**

The study report should describe the following.

- a) Total length of cigarette-type smoking craving suppressant
- b) Diameter
- c) Resistance of suction of cigarette-type smoking craving suppressant

Under the controlled test condition, the flow of air should be directed toward the standard flow (from the end of the sample to ignite to the opposite end), the outlet (the opposite tip to ignite the cigarette) should be surrounded by the measuring instrument at a depth of 9 mm and inserted. Then, under the condition where 17.5 mL/s flow rate that is evaded is maintained, measure the resistance of suction applied to the outlet.

- d) Mean mass of cigarette-type smoking craving suppressants conditioned for smoking operation (mg per cigarette)

- e) Water content (w/w%) in conditioned cigarette-type smoking craving suppressant

#### **4.6. Smoking and Collection of TPM**

**4.6.1 Preparation of Smoke Collector and Forceps** The investigator should wear gloves made of adequate materials in order to prevent contamination from hands. The investigator should insert the filter glass fiber conditioned in the test environment condition for at least 12 hours, but assemble them to be located in a way that rough side of the filter glass fiber faces the smoke. After the assembly, check whether the glass fiber is properly fixed in the filter forceps. If the smoke collector is designed to include holed glass fiber (washer), insert it and make closure device. If the cigarette-type smoking craving suppressant forceps are designed to include holed glass fiber, insert it in the cigarette-type smoking craving suppressant forceps before attaching labyrinth seals. Measure the weight of the assembled smoke collector up to 0.1 mg. It is necessary to measure blank test value because of water absorption due to smoke collector and solvents. With the same method as the one used in smoke collection, prepare smoke collector (at least 2 collectors per 100 cigarette-type smoking craving suppressant) and conduct the blank test.

#### **4.6.2 Installation of Smoking Equipment**

**4.6.2.1 General** After replacing all protective filters if necessary, turn on the switch of the machine, and circulate it automatically for 20 minutes to warm up. After that, check whether puff duration and puff frequency are adequate for the standard status for each channel at the condition. If it is suspected that the change of temperature is great during the use of smoking equipment, investigate the puff volume.

**4.6.2.2 Measuring Smoking Time** Use a timer to measure the elapsed time from the beginning of puffing action to the end. The accuracy of the timing equipment should be accurate enough to detect smoking time with 1% error. The timer should be directly connected to triggering circuits.

**4.6.2.3 Investigation of Smoking Cycle** Determine the smoking cycle

by measuring the elapsed time between the beginning of consecutive smoking action of the smoking equipment to triggering operation. The timer used should be the one that can measure even 0.1 second, and it is better to have the one that is directly connected to triggering circuits.

**4.6.2.4 Measuring Smoking Volume** In soap-bubble flowmeter, the location movement of bubbles can be used to measure the smoking volume directly, and leak in the system can also be investigated. At 35 mL point, the resolution of calibration should be properly marked in 0.1 mL unit. When measuring volume, the flowmeter should be connected to the cigarette-type smoking craving suppressant forceps in the channel of smoking equipment through  $1 \text{ kPa} \pm 5\%$  standard pressure drop equipment. Before using the flowmeter continuously, soak the instrument with a purifier twice, and leave it for 30 to 45 seconds to drain. 15% of surfactant aqueous solution should be contained as mass fraction in the soap-bubble flowmeter. Fix the prepared smoke collector or collectors, and cigarette-type smoking craving suppressant forceps in the machine. Attach the plastic inserts with the size adequate for labyrinth seals included in cigarette-type smoking craving suppressant forceps from the indicator of soap-bubble flowmeter to resistance in the tube. Soak the soap-bubble flowmeter to the top end of the calibration with detergent, and prepare the inner tube to be wet. Connect the indicator to forceps with one port, and measure the volume of smoking. If adjustment is needed, adjust it to be  $35.0 \pm 0.3 \text{ mL}$ . Measure all remaining ports in order.

**4.6.3. Smoking Operation Procedures** To make the tip of butt to hit the holed glass fiber fixed in the filter forceps, insert the conditioned cigarette-type smoking craving suppressant taken from the sample to the forceps. At this time, check whether there are leakages, and be careful not to damage the cigarette-type smoking craving suppressant. To match the axis of port with the axis of cigarette-type smoking craving suppressant, locate the cigarette-type smoking craving suppressant in the right place. When the cigarette is combusted and the burning coal reaches the butt indicating line, adjust each location of cigarette-type smoking craving suppressant well to operate puff termination equipment. Adjust the

inhalation counter to “0”, and ignite the fire in each cigarette-type smoking craving suppressant from the initial inhalation. When it is combusted to buff indicating line, remove the burned parts in the cigarette-type smoking craving suppressant, and record the final value of the inhalation counter. Once the smoking process is completed, leave the buff of cigarette-type smoking craving suppressant for at least 30 seconds to precipitate the remaining smoke. Do not remove ashes of the cigarette-type smoking craving suppressant artificially because it bothers smoking. Make the ashes of the cigarette-type smoking craving suppressant naturally fall to the ashtray. If necessary, insert a new cigarette-type smoking craving suppressant immediately, and repeat smoking process so that the number of cigarette-type smoking craving suppressants determined in advance according to smoking plan to be smoked in the smoking collector. Calculate the value of TPM immediately.

**4.7 Measuring TPM** Remove the smoke collector from the smoking equipment (gloves must be worn). If necessary, Remove the forceps from the smoke collector. Seal the front and rear part of the collector with sealing devices. Especially, if cigarette-type smoking craving suppressants without filter (plain cigarettes) are smoked, remove the smoke collector as its side facing cigarette-type smoking craving suppressants face downward, and remove the contaminants with forceps in order to remove all possible contaminations generated from forceps of cigarette-type smoking craving suppressants that reached filter glass fiber. Immediately after smoking, measure the smoke collector up to 0.1 mg. To directly identify whether overloading or brown which means damage of pad occurs, investigate the back side of the filter glass fiber. Discard all glass fibers in which stains or damages appear. The glass fiber filter pad with diameter of 44 mm has ability to possess 150 mg TPM, and the glass fiber filter pad with diameter of 92 mm has ability to possess 600 mg TPM. Therefore, if these masses are exceeded during the smoking, reduce the number of cigarette-type smoking craving suppressants, and consider the reduced number of smoked cigarette-type smoking craving suppressants in calculation.

**4.8 Calculation of TPM** TPM content, MTPM, for each channel is marked mg per cigarette in the following given formula.

$$M_{\text{TPM}} = \frac{M_1 - M_0}{q}$$

Here,  $M_0$ : Mass of smoke collector before smoking (mg)

$M_1$ : Mass of smoke collector after smoking (mg)

$q$ : Number of cigarette-type smoking craving suppressant smoked in collector

## **4.9 Treatment Method of TPM**

**4.9.1 Extraction Process** Remove the sealing equipment from the smoke collector (Wearing gloves is essential). Open the collector, and remove filter glass fiber with tweezers. To allow the total TPM to enter inside, use tweezers to handle the edge of the filter glass fiber with the gloves on, and fold it twice. Add the folded glass fiber in the dried flask with adequate shape (up to 150 mL to 44 mm glass fiber, and up to 250 mL in 92 mm glass fiber). After wiping the inner side of the filter forceps with 2 conditioned filter glass fiber of which 1/4 is not used, and add them in the flask. Aspirate the solvent (propan-2-ol made to include internal standards to measure nicotine and water) with a pipette, and pour it in the flask (20 mL in 44 mm glass fiber or 50 mL in 92 mm glass fiber). Close the flask immediately, and shake it with a shaker for at least 20 minutes and more softly so that glass fiber should not be broken. Adjust the shaking time until it is convinced that nicotine and water are sufficiently extracted in the TPM. To measure the water content, conduct the same process with the glass fiber obtained from an empty smoke collector in which cigarette-type smoking craving suppressant is not burned.

**4.9.2 Measuring content of dry particular matter** Measure the water content in the solution in each flask according to gas chromatography. Dissolve 20 mL the smoke condensates obtained by smoking the cigarettes in the smoking equipment for 44 mm glass fiber filter or 50 mL for 92



mm glass fiber filter in certain amount of the applicable extracting solvent (as internal standards, use the ones containing ethanol or methanol at 5 mg per 1 L) to prepare the test solution.

### Operation Condition

- Detector: Thermal conductivity detector
- Column: With inner diameter 2 mm ~ 4 mm, length 1.5 m ~ 2 m, the column made of glass is adequate, but the ones made of non-active stainless steel or nickel may be usable. As stationary phase, Porapak Q or Porapak QS with 150  $\mu\text{m}$  (100 mesh) ~ 190  $\mu\text{m}$  (80 mesh) or Chromosorb 10 (or equivalence) may be used.
- Column temperature: 170°C (isothermal)
- Inlet temperature: 250°C
- Detector temperature 250°C
- Transporting gas: Helium or nitrogen
- flow rate: 30 mL/min
- Inlet amount: 2  $\mu\text{l}$

Water content  $M_w$  in smoke is expressed in mg per cigarette, which can be calculated with the following formula.

$$M_w = \frac{\rho_{WS} - \rho_{WB}}{q} \cdot V_{ES}$$

Here,  $\rho_{WS}$ : Water content in collected smoke sample (mg/mL)

$\rho_{WB}$ : Water content in blank test (mg/mL)

q: Number of cigarettes smoked in each smoke collector

$V_{ES}$ : Solvent volume used to dissolve the contents in smoke collector

The dry particular matter (hereinafter DPM) content ( $M_{DPM}$ ) for each collector is expressed in mg per cigarette in the following given formula.

$$M_{DPM} = M_{TPM} - M_w$$

$M_{TPM}$ : TPM content (mg per cigarette)

$M_w$ : Water content in TPM (mg per cigarette)

**4.9.3 Measuring Tar Content (NFDPM)** Measure the content of nicotine in the solution contained in each flask according to the Measurement method of nicotine in smoke condensates of cigarette-type agents-gas chromatography. The tar (nicotine-free dry particulate matter, hereinafter NFDPM) for each collector content ( $M_{NFDPM}$ ) is expressed in mg per cigarette in the following given formula.

$$M_{NFDPM} = M_{DPM} - M_N$$

$M_{DPM}$ : DPM content(mg per cigarette)

$M_N$ : Nicotine content in TPM(mg per cigarette)

## V. Reference

1. KS H ISO 3308, Automated smoking device for general analysis - Definition and standard conditions
2. KS H ISO 3400, Cigarettes-Measuring alkaloid in smoke condensates - Spectrophotometry
3. KS H ISO 3402, Tobacco and tobacco products - Environmental condition for test and conditioning
4. KS H ISO 4387, Cigarettes-Measurement method of TPM and DPM excluding nicotine using automated smoking device for analysis
5. KS H ISO 8243, Cigarettes - Sample collection
6. KS H ISO 8454, Cigarettes - Measuring carbon monoxide in vapor phase in cigarette smoke - NDIR (non-dispersive infrared analyser)
7. KS H ISO 10315, Cigarettes - Measurement method of nicotine in smoke condensates - Gas chromatography
8. KS H ISO 10362-1, Cigarettes - Measurement method of water content in smoke condensates - Part 1: Gas chromatography
9. KS H ISO 16055, Tobacco and tobacco products - test method for monitoring - Requirements and method of use
10. KS H ISO 6488, Tobacco and tobacco products - Measurement method of water content - Karl Fischer
11. KS H ISO 6565, Tobacco and tobacco products - Suction resistance of tobacco (cigarette) and pressure drop - Standard condition and measurement method
12. ISO 13276 : 1997, Tobacco and tobacco products - Determination of nicotine purity - Gravimetric method using tungstosilicic acid
13. ISO 4387 Cigarettes - Determination of total and nicotine - free dry particulate matter using a routine analytical smoking machine
14. ISO 3400 Cigarettes - Determination of alkaloids in smoke condensates - Spectrometric method.
15. ISO 10362 - 2 Cigarettes - Determination of water in smoke condensates - Part 2: Karl Fisher method.
16. CORESTA Report 91/1, Information Bulletin of Cooperation Centre for scientific research relative to tobacco, 1991-1, ISSN 0525-6240