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Identification and Assay for Nicotine in Electronic Cigarettes

1. Scope

This method is applicable to the identification and determination of nicotine in electronic cigarettes.

2. Method

After extraction, nicotine is identified and determined by gas chromatography/mass spectrometry (GC/MS).

2.1. Equipments

2.1.1. Gas chromatograph/mass spectrometer

2.1.1.1. Ionization source: Electron ionization

2.1.1.2. Column: HP-5 MS, 0.25 μm film thickness, 0.25 mm i.d. × 30 m; or an equivalent product.

2.1.2. Ultrasonicator

2.2. Chemicals

Methanol, LC grade;
Propylene glycol, GR grade;
Nicotine, reference standard.

2.3. Apparatus

2.3.1. Volumetric flask: 10 mL and 100 mL.

2.3.2. Centrifuge tube: 10 mL.

2.3.3. Membrane filter: 0.45 μm, Nylon.

2.4. 10% Propylene glycol/methanol

Dilute 10 g of propylene glycol with methanol to 100 mL.

2.5. Standard solution preparation

Accurately weigh equivalent 10 mg of nicotine reference standard to a 100-mL volumetric flask, dissolve and dilute with 10% propylene glycol/methanol to volume as the stock solution. When to use, dilute appropriate volume of the stock solution with 10% propylene glycol/methanol to obtain 0.5-50 μg/mL as the standard solutions.

2.6. Sample solution preparation

Transfer 1 g of the well-mixed sample, accurately weighted to a 10-mL volumetric flask, add 8 mL of 10% propylene glycol/methanol and ultrasonicate for 10 min. Dilute with 10% propylene glycol/methanol to volume, and filter with a membrane filter. Take the filtrate as sample solution.

2.7. Identification and Quantitation

Accurately inject 1 μL of the sample solution and the standard solutions into the GC/MS separately, and operate according to the following conditions. Identify nicotine based on the retention time and the relative ion intensities. Calculate the amount of nicotine in the sample by the following formula:

$$\text{The amount of nicotine in the sample (ppm)} = \frac{C \times V}{M}$$

where:

C: the concentration of nicotine in the sample solution calculated by the standard curve (μg/mL)

V: the make up volume of sample (mL)

M: the weight of sample (g)

GC/MS operating conditions:

Column: HP-5 MS, 0.25 μm film thickness, 0.25 mm i.d. × 30 m.

Oven temperature program:

initial temperature: 100°C, hold for 4 min;

temperature gradient rate: 10°C/min;

final temperature: 280°C, hold for 1 min.

Injector temperature: 260°C.

Injection mode: split, ratio 5:1

Injection volume: 1 μL.

Carrier gas and flow rate: Helium, 1.0 mL/min.

Source temperature: 280°C.

Ionization mode: EI, 70 eV.

Ionization temperature: 230°C.

Quadruple temperature: 150°C.

MS scan range: *m*/*z* 40~500.

MS detection mode: Full scan, *m*/*z* 40~500; detected ions are as follows.

Analyte	Quantitation ion (*m*/*z*)	Qualitation ion (*m*/*z*)
Nicotine	84	133, 161, 162

Notes:

1. Relative ion intensities are calculated by peak areas of qualitation ions divided by peak areas of quantitation ion (≤100%). Maximum permitted tolerances for relative ion intensities by GC/MS are as follows:

Relative ion intensity (% of base peak)	Tolerance (%)
> 50	± 10
> 20~50	± 15
> 10~20	± 20
≤ 10	± 50

2. All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

Remark

1. The limit of quantitation (LOQ) for nicotine is 5 ppm.
2. Further validation shall be done when interference compounds appear in samples.