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Vapour products — Determination of e-liquid vaporised mass and aerosol collected mass

Produits de vapotage — Détermination de la masse de e-liquide vaporisé et de la masse d'aérosol collecté

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

Foreword

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This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, Subcommittee SC 3, *Vape and vapour products,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 437, *Electronic cigarettes and related e-liquids,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Technical investigation of vapour products requires determining aerosol collected mass (ACM) and e-liquid vaporized mass (EVM) measurements in emissions. Therefore, there is a necessity to have an International Standard in place to get reliable/comparable data on ACM and EVM in electronic cigarette emissions.

The method in this document is based upon the CORESTA recommended method (CRM) 84,[1] which was written on the basis of the results obtained from interlaboratory studies conducted in 2015[2] and 2019[3] involving 18 and 11 laboratories, respectively.

This document has been developed to describe the procedures used to measure the amount of ACM and EVM in the aerosol from vapour products utilizing a gravimetric method. The experimental design parameters[4][5] used to collect the aerosolised vapour should be evaluated and documented for each analysis.

Vapour products — Determination of e-liquid vaporised mass and aerosol collected mass

1 Scope

This document specifies a method of measurement of the masses of e-liquid vaporised and the aerosol collected from vapour product(s).

It does not specify the vapour product(s), the vapour product(s) operational settings or, e-liquid to be used.

NOTE Application of this document can be required as a preliminary step for subsequent analyses.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 20768, *Vapour products — Routine analytical vaping machine — Definitions and standard conditions*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
aerosol collected mass
ACM
mass of aerosol collected on a glass fibre filter pad resulting from the operation of a vapour product by a routine analytical vaping machine after a defined number of puffs

Note 1 to entry: Routine analytical vaping machine is covered by ISO 20768.

3.2
puff block
finite series of sequential puffs as defined by the test request

EXAMPLE Puff block 1: puffs 1 to 50, puff block 2: puffs 51 to 100, puff block 3: puffs 101 to 150

3.3
e-liquid vaporised mass
EVM
mass of e-liquid transferred from the vapour product to the aerosol

Note 1 to entry: The term “vapour product mass loss” or “mass loss” refers to the e-liquid vaporised mass.

3.4
glass fibre filter pad overload
physical capacity of the glass fibre filter pad exceeded, which leads to a situation where the (volume of) aerosol condensate is no longer retained by the glass fibre filter pad (trap) or can be introduced into the glass fibre filter pad (be trapped)

Note 1 to entry: The glass fibre filter pad overloading is further described in Annex A.

4 Principle

Aerosol is generated from vapour products by using a routine analytical vaping machine according to ISO 20768. The vapour products generate an aerosol from the e-liquid, which allows the gravimetric measurement of e-liquid vaporised mass and the aerosol collected mass, respectively.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Vaping machine, in accordance with ISO 20768.

5.2 Analytical balance, be precise at a minimum of 1 mg with a display precision of 0,1 mg.

5.3 Soap bubble flow meter, suitable to confirm the required puff volume.

5.4 Glass fibre filter pad, with specifications according to Annex B.

6 Procedure

6.1 General

EVM is calculated from the vapour product mass loss due to aerosol generations, while ACM is calculated from the aerosol generated using a fixed number of puffs and puff blocks following standard operational settings and environmental conditions in accordance with ISO 20768.

Prior to vaping, each aerosol collection system used should be checked to ensure that there are no leaks and that the correct puff volume is obtained. Both leak and puff volume checks should be performed according to the vaping machine manufacturer's recommendations. The amount of aerosol generated depends on the nature of the vapour product, operational settings and the e-liquid. It is highly recommended to perform a trapping efficiency study to verify the glass fibre filter pad has not been overloaded. Trapping efficiency is described in more detail in Annex A.

6.2 Preparation

6.2.1 Vapour products

All the vapour products to be tested should be stored at room temperature. Vapour products with rechargeable batteries shall be fully charged before the test and be operated in accordance with the manufacturer recommendations.

6.2.2 Filter pad handling

The handling of the filter pad shall be done according to the following laboratory practises.

— Wear proper gloves (powder free) for all operations to prevent contamination.

— Filter pads shall be stored in the target atmosphere of the test conditions for a minimum of 24 h prior to use.

— Prepared filter pad holders shall be weighed and mass recorded (see 6.4, m_i).

— If the filter pad holder is not used immediately, install a filter pad holder cap to prevent water loss or uptake.

6.2.3 Aerosol trapping

The number of puffs or puff blocks are dependent on the test device performance and/or test requirements and shall also take into consideration the avoidance of glass fibre filter pad overloading.

6.2.4 Puff volume check

Prior to vaping, each vaping channel shall be checked to ensure the correct puff volume is obtained. Volume checks shall be performed according to the vaping machine manufacturer's recommendations.

6.2.5 Handling of aerosol samples

If aerosol samples are not analysed immediately after vaping, install a filter pad holder cap to prevent water loss or uptake.

6.3 Determination of e-liquid vaporised mass (EVM)

Step 1: To perform the test, the vapour product shall be operated in accordance with the manufacturer's recommendations.

Step 2: The initial measurement shall be carried out on the whole vapour product filled or pre-filled with e-liquid (e.g. tank or pod with power source, but without mouth end protective cap). Measure and record the mass (m_i) of the vapour product, then attach it to the corresponding port of the vaping machine.

Step 3: Generate a sufficient number of puffs to provide enough mass loss to be measured gravimetrically with sufficient accuracy.

Step 4: Disconnect the vapour product from the vaping machine.

Step 5: The final measurement of the vapour product's mass (m_f) shall be carried out on the whole vapour product (e.g. tank or pod with power source, but without mouth end protective cap).

The e-liquid vaporised mass is calculated by Formula (1).

$$m_{EVM} = m_i - m_f \quad (1)$$

where

m_{EVM} is the e-liquid vaporised mass (EVM), in milligram;

m_i is the vapour product mass before aerosol generation, in milligram;

m_f is the vapour product mass after aerosol generation, in milligram.

6.4 Determination of aerosol collected mass (ACM)

Step 1: To perform the test, the vapour product shall be operated in accordance with the manufacturer recommendations.

Step 2: The initial measurement shall be carried out on the aerosol collection system. Connect the pre-measured and recorded (m_i) glass fibre filter pad holder including the glass fibre filter pad to the vaping machine by using trapping seal if necessary.

Step 3: Generate a sufficient number of puffs to provide enough mass to be measured gravimetrically with sufficient accuracy

Step 4: Disconnect the aerosol collection system from the vaping machine.

Step 5: Measure the final mass (m_f) of the glass fibre filter holder.

The aerosol collected mass is calculated by Formula (2).

$$m_{ACM} = m_f - m_i \quad (2)$$

where

m_{ACM} is the aerosol collected mass (ACM), in milligram;

m_i is the glass fibre filter holder mass before aerosol generation, in milligram;

m_f is the glass fibre filter holder mass after aerosol generation, in milligram.

6.5 Specific mass determination of puff blocks

The mass of vapour product or glass fibre filter holder before next aerosol collection shall be verified.

The e-liquid vaporised mass shall be measured on each puff block. The m_E is defined by the following Formula (3) and Formula (4).

$$m_{EVM,t} = \sum_{k=1}^{n} m_{EVM,k} \quad (3)$$

$$m_{EVM,n} = m_{i,n} - m_{f,n} \quad (4)$$

where

$m_{EVM,t}$ is the e-liquid vaporised mass (EVM) for all puff blocks, in milligram;

$m_{EVM,n}$ is the e-liquid vaporised mass for one puff block, in milligram;

k is the number of puff block;

n is the numbering of the puff block or puffs;

$m_{i,n}$ is the vapour product mass before aerosol generation of puff block n, in milligram;

$m_{f,n}$ is the vapour product mass after aerosol generation of puff block n, in milligram.

If reservoir is not refilled between puff blocks: $m_{f,n} = m_{i,n+1}$

If reservoir is refilled between puff blocks: $m_{f,n} \neq m_{i,n+1}$

The aerosol collected mass shall be measured on each puff block. The aerosol collected mass is defined by the following Formula (5) and Formula (6).

$$m_{ACM,t} = \sum_{k=1}^{n} m_{ACM,k} \quad (5)$$

$$m_{\mathrm{ACM},n} = m_{\mathrm{f},n} - m_{\mathrm{i},n} \quad (6)$$

where

$m_{\mathrm{ACM,t}}$ is the aerosol collected mass (ACM) for all puff blocks, in milligram;

$m_{\mathrm{ACM},n}$ is the aerosol collected mass for one puff block, in milligram;

k is the number of puff blocks;

n is the numbering of the puff blocks or puffs;

$m_{\mathrm{i,n}}$ is the glass fibre filter holder mass before aerosol generation of puff block n, in milligram;

$m_{\mathrm{f,n}}$ is the glass fibre filter holder mass after aerosol generation of puff block n, in milligram.

If absence of overloading of the aerosol trap: $m_{\mathrm{f},n} = m_{\mathrm{i},n+1}$

Standard deviation and mean value between puff blocks shall be calculated and reported.

7 Repeatability and reproducibility

7.1 General

The repeatability and reproducibility data given in this document has been generated with the devices stated below. Other devices with higher performance variances can influence the data variability.

7.2 Results from the interlaboratory study 2015 (Study 1)

An international collaborative study was conducted in 2015, involving 18 laboratories and four commercial cigalike type, rechargeable vapour products and one study control (e-liquid sample).[2] Aerosol from each device was collected in three puff blocks (10 puffs, then 20 puffs, then 50 puffs) using CORESTA recommended method N°81,[6] in which the specified vaping regime is identical to that specified in ISO 20768. The ACM and EVM for each sample were calculated for 80 puffs. Each laboratory provided 5 to 8 replicate determinations for each sample. The statistical evaluation was performed according to ISO 5725-5[7] and ISO 13528[8] and is presented in Table 1.

Table 1 — Repeatability and reproducibility limits (mg/80 puffs) for study 1

Sample code	Analyte	Mean value	Repeatability limit	Reproducibility limit	as a percentage of mean value	
		mg/collection	r	R	r %	R %
A	m_{ACM}	148	42	69	28,60	46,60
B	m_{ACM}	127	43	72	33,50	56,70
C	m_{ACM}	109	43	51	39,40	47,20
D	m_{ACM}	142	48	58	33,90	40,50
A	m_{EVM}	137	40	74	28,97	53,88
B	m_{EVM}	120	42	72	34,80	60,07

Key

A nicotine (mass fraction) 0 %; glycerin:propylene glycol = 70:30

B nicotine (mass fraction) 2,4 %; glycerin:propylene glycol = 70:30

C nicotine (mass fraction) 5,4 %; glycerin:propylene glycol = 70:30

D nicotine (mass fraction) 2,4 %; glycerin:propylene glycol = 100:00

Table 1 *(continued)*

Sample code	Analyte	Mean value mg/collection	Repeatability limit r	Reproducibility limit R	as a percentage of mean value r % R %	
C	m_{EVM}	105	43	51	40,71	48,49
D	m_{EVM}	136	48	55	34,99	40,19

Key

A nicotine (mass fraction) 0 %; glycerin:propylene glycol = 70:30

B nicotine (mass fraction) 2,4 %; glycerin:propylene glycol = 70:30

C nicotine (mass fraction) 5,4 %; glycerin:propylene glycol = 70:30

D nicotine (mass fraction) 2,4 %; glycerin:propylene glycol = 100:00

7.3 Results from the interlaboratory study 2019 (Study 2)

An international collaborative study was conducted in 2019, involving 11 laboratories.[3] This study was conducted for the evaluation of the Aspire Nautilus™ Tank[1)] with a 1,8 Ω tank coil and Evolv™ power unit[2)] for potential as a Reference Device. Three study e-liquid samples were tested utilizing the prescribed tank and power unit combination by each laboratory. Aerosol was collected in three puff blocks (25 puffs for each puff block) for each sample using CORESTA recommended method N°81,[6] in which the specified vaping regime is identical to that specified in ISO 20768. The ACM and EVM for each sample were calculated for 75 puffs. Each laboratory provided 3 replicate determinations for each sample. The statistical evaluation was performed according to ISO 5725-2[9] and ISO/TR 22971[10] and is presented in Table 2.

Table 2 — Repeatability and reproducibility limits (mg/75 puffs) for study 2

Sample code	Analyte	Mean value mg/collection	Repeatability limit r	Reproducibility limit R	as a percentage of mean value r % R %	
A	m_{ACM}	731	67	204	9,20	27,80
B	m_{ACM}	705	77	275	10,90	39,00
C	m_{ACM}	691	118	264	17,10	38,20
A	m_{EVM}	719	62	190	8,60	26,40
B	m_{EVM}	696	82	269	11,80	38,60
C	m_{EVM}	683	122	254	17,90	37,20

Key

A unflavored

B tobacco

C tobacco/menthol

8 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this document, or regarded as optional, as well as any deviations that can

1) Aspire Nautilus™ is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

2) Evolv™ is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

influence the results. The test report shall include all details required for complete identification of the sample. If appropriate, the information given below in a) to c) shall be recorded.

a) Characteristic data about the vapour product:

All details necessary for the identification of the vapour products and e-liquid vaporised shall be given. In the case of commercial vapour products this should include:

— name of the manufacturer and country of manufacture;

— product name;

— batch/lot number (of the product sampled that day);

— nicotine concentration (if available);

— temperature setting (if available);

— coil resistance (if available);

— wattage (if available);

— battery power (if available);

— mouthpiece configuration;

— air flow configuration;

b) Description of the test:

— reference to this document;

— date of test;

— sampling method description;

— type of vaping machine used;

— type of aerosol trapping system used (if used);

— number of puffs per puff block;

— initial vapour product mass;

— testing angle;

— if deviated from ISO 20768, report vaping regime, room temperature (in degrees Celsius) and relative humidity (in percent) during testing;

c) Test results:

The expression of the laboratory data depends on the purpose for which the data are required, and the level of laboratory precision. General base units for all mass measurements are reported in milligrams. Additional details should include the following:

— m_{EVM} per puff or per number of puffs. Result reported in mg to two significant figures;

— $m_{EVM,t}$; in case of several puff blocks, mass of e-liquid vaporised total and the mean value;

— m_{ACM} per puff or per number of puffs. Result reported in mg to two significant figures;

— $m_{ACM,t}$; in case of several puff blocks, aerosol collected mass total;

— n (number of puff blocks conducted);

— *N* (number of puffs in each puff block).