
**Vapour products — Determination of
selected carbonyls in vapour product
emissions**

*Produits de vapotage — Dosage de carbonyles sélectionnés dans les
émissions de produits de vapotage*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 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

In many countries, regulation of vapour products requires reporting for carbonyl compounds in emissions. Therefore, there is a necessity to have an International Standard in place to get reliable/comparable data for selected carbonyls in vapour product emissions.

The method in this document is based upon the CORESTA recommended method CRM 96^[1] which was written on the basis of the results obtained in an interlaboratory study conducted in 2019 involving 11 laboratories.

Carbonyl compounds are known to be derived from the thermal degradation of the base ingredients of the e-liquid formulations. The experimental design parameters ^{[2],[3]} used to collect the aerosolised vapour should be evaluated and documented for each analysis.

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Vapour products — Determination of selected carbonyls in vapour product emissions

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices, and determine the applicability of any other restrictions prior to use.

1 Scope

This document specifies a method for the determination of the amount of selected carbonyl compounds (formaldehyde and acetaldehyde) as their 2,4-dinitrophenylhydrazones in vapour product emissions using reversed phase liquid chromatography coupled with ultraviolet or diode array detector (LC-UV or LC-DAD).

This document does not include the analysis of other carbonyl compounds, such as acrolein and crotonaldehyde, due to previous work indicated issues associated with stability of these compounds in the e-liquid solutions that were used to evaluate method performance^[4].

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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 24197:—¹⁾, *Vapour products — Determination of e-liquid vaporised mass and aerosol collected mass*

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.

1) Under preparation. Stage at the date of publication: ISO/FDIS 24197:2022.

3.2
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.

[SOURCE: ISO 24197:—, 3.3]

3.3
puff block

finite series of sequential puffs as defined by the user or 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.

3.4
aerosol trapping system

system for collecting the aerosol from vapour products

Note 1 to entry: For this method the aerosol trapping system consists of a filter trap (pad + holder) and an impinger in series.

3.5
reagent blank

solution that is evaluated to check the level of contamination introduced by the reagents

3.6
aerosol blank

sample from a port that is attached to an *aerosol trapping system* (3.4) that contains no vapour product and is carried through the same collection, preparation and analysis steps as the test samples

4 Principle

The vapour product emissions are generated and collected on a vaping machine according to ISO 20768. The trapping system that is used to trap carbonyls consists of a pad holder containing a glass fibre filter pad according to ISO 24197, in series with an impinger containing an acidified solution of 2,4-dinitrophenylhydrazine (DNPH) in 1:1 acetonitrile:water. Post-vaping, the glass fibre filter pad is combined with the impinger solution and shaken mechanically for 20 min. An aliquot of the sample extract is subsequently neutralised with pyridine and analysed by reversed phase liquid chromatography with ultra violet or diode array detector (RPLC-UV or RPLC-DAD). The carbonyl content in the vapour product emissions is calculated based on an external calibration curve containing the prederivatized DNPH carbonyl compounds. Results are expressed as the weight of carbonyl per puff, per aerosol collected mass (ACM) or per puff block as warranted.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade.

NOTE Alternative reagents can be used provided the suitability and equivalence is verified.

5.1.1 Acetonitrile, ACN, HPLC grade.

5.1.2 Ethanol, HPLC grade.

5.1.3 Phosphoric acid, (H₃PO₄, a mass fraction of 85 %, or a volume fraction of 10 % aqueous solution).

5.1.4 Water, grade 1 according to ISO 3696 or equivalent.

5.1.5 Pyridine, minimum purity 99 %.

5.1.6 Formaldehyde-DNPH, minimum purity 99 %.

5.1.7 Acetaldehyde-DNPH, minimum purity 99 %.

5.1.8 2,4-Dinitrophenylhydrazine hydrochloride (DNPH-HCL) or 2,4-dinitrophenylhydrazine (DNPH)(containing approximately 30 % water).

5.2 Solution preparation

5.2.1 General

Prepare appropriately proportioned amounts of the solutions listed below. All solutions shall be equilibrated to room temperature prior to use. Use graduated cylinders and calibrated pipettes to combine components.

5.2.2 10 % H₃PO₄

Prepare by bringing 118 ml of 85 % H₃PO₄ (5.1.3) to 1 l water (5.1.4). Vendor prepared 10 % H₃PO₄ may be used instead. Store at room temperature.

5.2.3 DNPH trapping solution, prepared with DNPH-HCL (5.1.8)

Dissolve 1,0 g DNPH-HCL (5.1.8) in 500 ml of ACN (5.1.1), combine with 40 ml of 10 % H₃PO₄ (5.2.2) and bring to 1 l with water (5.1.4). Mix solution to ensure all the DNPH-HCL dissolves and no crystals remain. Solution should be prepared fresh weekly, stored at room temperature and protected from light.

NOTE Alternative preparation of trapping solution with DNPH containing 30 % water is provided in [Annex A](#).

5.2.4 Neutralised DNPH trapping solution (if dilutions are required)

Transfer 50 ml of DNPH trapping solution to a suitable size glass bottle and add 2,5 ml of pyridine. Mix solution thoroughly.

5.3 Preparation of standards

5.3.1 General

All solutions shall be equilibrated to room temperature prior to use.

5.3.2 HPLC calibration standards and working solutions

The calibration should cover the concentration range of interest. [Annex B](#) provides a suitable concentration range that can be used for the analysis, however, it can be adjusted depending on the level of carbonyls detected in the samples. The user shall ensure the low calibration standard has a sufficient signal to noise ratio for accurate quantitation (≥10:1) and that the calibration curve is linear.

5.3.3 Primary carbonyl standards

Weigh the hydrazones as described in [Annex B](#) into individual 25 ml volumetric flasks and dissolve in acetonitrile. Record the concentrations of the free aldehyde equivalents in µg/ml.

5.3.4 Secondary carbonyl standards

Pipette predetermined volumes (see [Annex B](#)) of each primary hydrazone standard into a 25 ml volumetric flask and dilute to the mark with acetonitrile.

NOTE Stock solutions of the individual DNPH derivatized carbonyls can be purchased at the required levels.

5.3.5 Carbonyl working standards

Take appropriate volumes (0,05 ml to 5 ml) of the secondary carbonyl standard ([5.3.4](#)) and dilute to 10 ml with acetonitrile to prepare calibration standards with approximate carbonyl concentrations (see [Annex B](#)).

Transfer to auto-sampler vials and cap.

Stability and storage time should be evaluated by the laboratory.

6 Apparatus

Usual laboratory apparatus and, in particular, the following items.

6.1 HPLC system, equipped with UV and/or DAD detector and a suitable data handling system.

6.2 HPLC columns.

- Disposable guard column: Reversed phase (RP) C18, and
- Analytical column: Reversed phase (RP) C18: 4,6 mm x 15 cm, 1,8 µm, or 2,5 µm or equivalent.

6.3 Vaping machine.

The aerosol is generated on a vaping machine, which follows the specifications specified in ISO 20768.

The standard conditions for puff duration and the puff profile are specified in ISO 20768; other parameters may also be used.

6.4 Aerosol trapping system.

6.4.1 44 mm glass fibre filter pads.

6.4.2 Filter pad holder.

6.4.3 Impingers for trapping emissions from vapour products.

6.4.4 Stem: Bubbler insert.

NOTE A coarse fritted tip impinger has been shown to be fit for purpose, alternative bubble insert can be used if demonstrated that the trapping efficiency is equivalent.

6.5 Analytical balance, minimum of 1 mg, which can be read to the nearest 0,1 mg.

6.6 Syringe filter (PTFE: 0,45 µm) and disposable syringe.

6.7 Autosampler vials, caps and PTFE faced septa.

7 Procedure

7.1 Preparation of test samples

All the vapour products to be tested shall be stored according to ISO 20768. Vapour products with rechargeable batteries shall be fully charged before the test. The aerosols are generated on a vaping machine which follows the specifications specified in ISO 20768.

7.2 Glass fibre filter pads handling

Glass fibre filter pads shall be stored in the test atmosphere for a minimum of 24 h prior to determination of pre-testing weights.

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of a proper material (powder free). Between operations, a glass fibre filter pad holder cap, if available, may be installed to prevent water loss or uptake. Glass fibre filter pads shall be processed as quickly after collection as is feasible to prevent uptake or loss of water.

7.3 Aerosol collection and sample preparation

The tested vapour product shall be operated in accordance with the manufacturer recommendations. Samples shall be collected under the conditions specified in ISO 20768. The vaping machine shall be set up to collect aerosol onto the glass fibre filter pad connected in series with an impinger ([Figure 1](#)).

Add 35 ml of DNPH trapping solution ([5.2.3](#)) to a separate impinger with an insert for each sample to be collected.

Assemble trapping system of the vaping machine in the following order (see [Figure 1](#)):

- pre-weighed 44 mm filter pad/holder;
- impinger;
- optional backup filter pad (not included in analysis);
- vaping machine.

Prior to collecting aerosol samples, the trapping system should be evaluated to ensure there are no leaks in the system and to ensure the puff volume is correct.

- Leak check: To determine whether a leak has occurred in the vaping machine impinger setup, use a leak tester.
- Puff volume check: Check and adjust the puff volume drawn by the vaping machine at all channels at the product insertion end of the port with the filter pad/holder and impinger and in line.