SLOVENSKI STANDARD
oSIST prEN ISO 24211:2021

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Hlapni proizvodi - Ugotavljanje deleža izbranih karbonilov v emisijah hlapnih proizvodov (ISO/DIS 24211:2021)

Vapour products - Determination of selected carbonyls in vapour product emissions (ISO/DIS 24211:2021)

Dampfprodukte - Bestimmung von ausgewählten Carbonylen in Emissionen von Dampfprodukten (ISO/DIS 24211:2021)

Produits de vapotage - Dosage de carbonyles sélectionnés dans les émissions de produits de vapotage (ISO/DIS 24211:2021)

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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).

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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*.

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 require 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 (Draft) [3] 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. Therefore, the experimental design parameters [4][5] used to collect the aerosolised vapour should be evaluated and documented for each analysis.

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 and inconsistencies in deliveries of these compounds in emissions from vapour products [6].

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*

ISO 24197, *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 an aerosol trapping system 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
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.

3.3
puff block
finite series of sequential puffs

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) or an impinger in series

3.5
reagent blank
solution that is evaluated to ensure that no contamination is introduced by the reagents

3.6
aerosol blank
port that is attached to an aerosol trapping system that contains no vapour product and is carried through the same collection, preparation and analysis steps as the 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 (as specified in 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 pad is combined with the impinger solution and shaken mechanically for 20 minutes. 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 (LC-UV or LC-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

Use only reagents of recognized analytical grade.

5.1 Acetonitrile, ACN, HPLC grade.

5.2 Ethanol, HPLC grade.

5.3 Phosphoric acid, (H_3PO_4, a mass fraction of 85 %, or a volume fraction of 10 % Aqueous Solution).

5.4 Water, HPLC grade or Deionized or equivalent.

5.5 Pyridine, minimum purity 99 %.

5.6 Formaldehyde-DNPH, minimum purity 99 %.

5.7 Acetaldehyde-DNPH, minimum purity 99 %.

5.8 2,4-Dinitrophenylhydrazine Hydrochloride (DNPH-HCL) or **2,4-Dinitrophenylhydrazine (DNPH)(containing approximately 30 % water)**.

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

5.9.1 10 % H_3PO_4

Prepare by bringing 118 ml of 85 % H_3PO_4 (5.3) to 1 l water. Vendor prepared 10 % H_3PO_4 may be used instead. Store at room temperature.

5.9.2 DNPH Trapping solution, prepared with DNPH-HCL (5.8)

Dissolve 1,0 g DNPH-HCl (5.8) in 500 ml of ACN (5.1), combine with 40 ml of 10 % H_3PO_4 (5.9.1) and bring to 1 l with water. 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.

5.9.3 DNPH Neutralised 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.

5.10 Preparation of Standards

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

5.10.1 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.

5.10.2 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.

5.10.3 Secondary carbonyl standards

Pipette predetermined volumes (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.

5.10.4 Carbonyl working standards

Take appropriate volumes (0,05 ml to 5 ml) of the secondary carbonyl standard (5.10.3) 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.

