

Cooperation Centre for Scientific Research Relative to Tobacco

CORESTA Guide N° 18

Technical Guide for Sample Handling and Sample Collection of E-Cigarettes and E-Vapour Generating Products

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E-Vapour Sub-Group



CORESTA TECHNICAL GUIDE N° 18

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Technical Guide for Sample Handling and Sample Collection of E-Cigarettes and E-Vapour Generating Products

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1. INTRODUCTION

This Technical Guide for the sample handling and sample collection of e-cigarettes and e-vapour products has been developed by the CORESTA E-Vapour Sub-Group to provide guidance to the industry and independent testing laboratories.

2. FIELD OF APPLICATION

This Technical Guide is applicable for the sample handling of e-cigarettes and e-vapour products before analysis and also includes guidance for sample aerosol collection.

3. TERMS AND DEFINITIONS

Tank-based e-cigarettes: Devices with an e-liquid reservoir separate from the atomizer. Many of these devices are designed to be refilled by the end user.

Cartomizer type devices: Devices with an e-liquid, wick and atomizer contained in a single cartridge. In general, these devices are not designed to be refillable.

Refill bottles: Bottles containing e-liquid preparation to be used in tank-based e-cigarettes.

4. PURPOSE

The objective of this guide is to describe the sample handling process after the sample has reached the laboratory and also to recommend general guidance for sample aerosol collection. It is difficult to recommend procedures for sample handling that will address every possible situation. The objective of sample handling is to minimize sample deterioration and to reduce variability in analytical results. For example, the elapsed time between sampling and testing will affect sampling and handling parameters as well as analytical results.

Other considerations are:

- Amount needed for all analyses including amounts for retests.
- Appropriate storage of samples before and during analysis. Storage containers should be selected to prevent potential contamination or uptake of water from the testing environment. Zip top plastic or Mylar bags have been found to be suitable for sample storage.
- Short-term or long-term storage of samples.
- Removal of e-liquid from pre-filled devices.

5. SAMPLING

It is recommended that after the samples have been collected, they shall be packed securely with adequate protection against damage, stored at ambient temperature and sent to the laboratory by the most expeditious means available. The amount of each sample needed is related to the requested analysis.

6. SAMPLE PREPARATION AND ANALYSIS

6.1 Sample Storage

After arrival at the laboratory, but before analysis, it is recommended that the samples be stored in zip top bags or other air-tight containers (or original finished good package) to reduce the possibility of water uptake from the testing environment. Appropriateness of the storage container should be verified by the testing facility before use. It is recommended that samples are stored under ambient conditions, but cartomizers and e-liquids may be stored at below -10 °C (typically -20 °C is standard freezer temperature) when long term storage is required. If samples are stored below 0 °C a pre-test (visual check) shall be performed to ensure that all compounds which could have precipitated are re-dissolved when e-liquid is brought back to ambient temperature. If not possible to verify (non-transparent container) it is recommended to store at +4 °C. It is recommended to perform the pre-tests with a reduced number of samples prior to storing the entire batch of samples. To reduce the uptake of water, storage in a desiccator when bringing samples to ambient temperature is recommended.

6.2 E-liquid extraction preparation

Use the following procedure to remove e-liquids from pre-filled cartomizer devices:

- Remove all packaging and applicable endcaps and discard. Remove the mouthpiece with a pair of needle-nose pliers.
- Place tweezers all the way to the bottom of the cartridge and twist the cartridge several times to free the substrate from the cartridge.
- Unwrap the substrate and place in the barrel of a disposable syringe. Re-insert the plunger and press down on the syringe plunger to force the liquid into a non-hygroscopic, inert, sealable container appropriate for storage time and storage conditions, e.g. a glass vial with Teflon or plastic-lined cap.
- Storage and handling of the e-liquid is dependent upon the intended analysis. However, many e-liquid compositions contain relatively volatile flavours and/or hygroscopic ingredients such as glycerol. Thus, samples should be stored or processed as quickly as possible in a manner that avoids prolonged exposure to the atmosphere.

Note: Some tanks/cartomizer may be sealed or tamper resistant. Liquid may be removed by centrifugation. Plastic tanks may be cut open using a small "PVC" pipe cutter. Depending on the design, it is also possible to extract the liquid using a syringe equipped with a needle (in this case, if the sampling is intended for metals analyses, absence of contamination from the needle should be verified, e.g., by performing blank analysis). Alternative procedures may be used to remove the e-liquid depending on the tanks/cartomizer design. All above-mentioned techniques can be applied with minor adaptation to pre-filled e-cigarettes depending on their design.

6.3 Sample conditioning prior to aerosol generation and collection

Prior to using any e-cigarette for testing it is recommended to equilibrate the e-liquid to the temperature of the testing laboratory atmosphere. It is known that e-liquid viscosity might be impacted by the temperature and thus modify the flow through the wick leading to variable aerosol mass determinations. However, due to its composition (propylene glycol and glycerol) e-liquids are hygroscopic and subject to water uptake from ambient moisture. Temperature equilibration should therefore be done in closed packs/containers.

- Prior to aerosol generation, samples shall be temperature equilibrated by storing them in the aerosol testing laboratory environment for a minimum of 12 h (or overnight). Samples shall be kept in the original sealed packaging to minimize water uptake.
- The temperature and relative humidity of the test and conditioning atmosphere shall be kept within the following limits as described in CRM 81:
 - Temperature: ± 2 °C.
 - Relative humidity: \pm 5 %.
- When aerosol collection cannot be performed during the same day, e.g., achieving full depletion of the cartomizer, samples shall be stored in sealed containers and kept in the testing laboratory environment. During storage, samples shall be orientated to avoid drying of the wick. In general, during storage devices should be stored upright.

6.4 Aerosol collection

The following section provides general guidance for collection of aerosol from e-cigarettes.

- It is recommended that batteries be fully charged before beginning the collection of aerosol.
- Cartomizer type devices are generally designed to be depleted with a single battery charge. It is recommended that only one battery charge is used per cartridge. Depleting cartomizer samples with more than one battery charge may lead to depletion of the liquid supply and elevated levels of thermal degradation products in the aerosol. Device yields measured at different points across the life of the cartridge can be used to inform a decision on the appropriate number of battery charges per cartridge. However, due to the rapid change of technology in e-cigarette it is recommended to refer to the product specifications.
- Aerosol should be generated using a collection system capable of meeting the requirements described in CRM 81 using a pre-set puff volume and puff duration.
- Devices that require filling, e.g., tank-based e-cigarettes, should be filled before analysis according to the manufacturer's instructions. Some devices require priming of the wick before use by adding a few drops of e-liquid and allowing it to become saturated before filling the tank and commencing analysis.
- Laboratory-filled e-cigarettes should be allowed to equilibrate after adding the liquid, i.e., the liquid must be allowed to saturate the wick and coil before any testing.
- Laboratory-filled e-cigarettes samples should be completely filled before analysis and refilled when the liquid level drops below 50 %.
- During active puffing, e-cigarettes and e-vapour products should be oriented in either the horizontal plane or with the mouth end elevated, for example at 45 degrees.

Note: In order to reduce artificial overheating, it is recommended that devices are allowed to rest in between collection of samples and that rapid and prolonged puffing is avoided. For devices that are puffed in the horizontal plane, it is recommended that devices are placed in the vertical position with the mouthpiece facing upwards between puff blocks to allow for liquid to transfer to the wicking system.

7. Bibliography

- CORESTA Recommended Method No. 81: Routine Analytical Machine for E-Cigarette Aerosol Generation and Collection Definitions and Standard Conditions, June 2015
- ISO 20768:2018 : Vapour products Routine analytical vaping machine Definitions and standard conditions, September 2018