Cooperation Centre for Scientific Research Relative to Tobacco

CORESTA Guide N° 25

Technical Guide for Aerosol Collection and Considerations when Testing E-Vapour Product Technologies

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1. Introduction

CORESTA Recommended Method (CRM) N° 81[1] has been developed as a standard to describe the requirements found necessary for the generation and collection of e-vapour aerosol for analytical testing purposes. While this standard can be used as a mechanism for comparison of products based on aerosol deliveries, it by no means represents the only strategy for the collection of e-vapour aerosol. It is recognized that the e-vapour category represents a diverse range of product designs that is rapidly evolving and the subsequent publication of Technical Guide N° 22[3] addresses considerations for alternative puffing regimens that can be used. Whilst CRM N° 81 and Technical Guide N° 22 do not aim to describe analytical procedures, the following list of considerations for potential collection strategies, and combination thereof, does represent some of the typical challenges faced when collecting e-vapour aerosol samples for analytical testing and should be considered in conjunction with the purpose of the testing being conducted.

2. Field of Application

This Technical Guide is applicable for current and emerging e-vapour product devices and technologies.

3. Considerations when Designing Collection Strategies

3.1 Puffing Regimen – Test Conditions

The 55 mL "square wave" puff over 3 seconds in CRM N° 81 was selected for a number of reasons. It can be achieved on all conventional smoking machines without modification, meaning that existing equipment can be used as a routine analytical machine for e-cigarette aerosol generation and collection. This puff profile is also within the range found for human usage of "cigalike" and some tank products, see CORESTA Technical Guide N° 22. This puff profile also yields a flow rate of 18.3 ml/sec which is sufficient to activate a wide range of puff activated devices.

Longer puff durations have the potential to increase the delivery per individual puff within the design limitations of the device. However, increasing the duration beyond the rate at which the e-liquid can saturate the wick in certain devices can have adverse effects (e.g. "dry wicking"). Furthermore, several devices have a safety cut off after a fixed number of seconds. Increasing the duration significantly beyond the 3 seconds has the potential to result in devices not producing aerosol or not being activated for the full duration of the puff.

The puff period of (30 ± 0.5) s, as specified in CRM N° 81, was an agreed upon frequency identified as producing aerosol samples within a reasonable timeframe while reducing the potential of "over-stressing" the device. Reducing the puff period (more puffs in a shorter timeframe) can increase the overall temperature of atomization by reducing the amount of time the atomizer cools between puffs, which can result in increased deliveries. This elevation in temperature can also damage the device and contribute to device failure.

The "square wave" ("constant flow") profile was selected in an attempt to reduce the impact of the activation flow rate threshold (see 3.3) differences between devices; this concern only applies to puff activated systems. Some puff activated devices are at risk of not remaining activated for the full duration of the puff when a "bell-shaped" profile is used due to an insufficient flow rate at the beginning and end of the puff. A "square wave" profile may not be necessary for button activated systems. However, changing from a "square wave" to a "bell-shaped" profile will change the maximum velocity of the aerosol passing through the collection system over the duration of the puff.

Whilst CRM N° 81 provides a robust rationale and platform for a standardized puffing regimen to allow product comparisons, it may be appropriate to operate outside of these parameters for some test protocols. For example, to mimic measured human topography data on a specific device, to test the limitations at which the device can operate, to determine or verify the activation flow rate of a device.

3.2 Collection Segments

In general, e-cigarettes do not have a pre-defined usage session, and therefore, the number of puffs collected (collection segment) for any given analysis is driven in part by the anticipated aerosol delivery and in part by the sensitivity (limits of detection and quantification) of the methodology used in the analysis. In some cases, where no delivery of a compound is expected (non-detected), the collection segment may be increased in an attempt to exceed the compound's detection limit.

The desired collection segment should never exceed the capacity of the collection media. Shorter collection segments can be used to demonstrate consistency of delivery across the life of the device. However, it is recognized that the initial puffs (e.g. first 10 puffs) delivered by some devices may be more variable in yields than the puffs taken once the device has equilibrated to the conditions of use. This should be considered when attempting to collect aerosol from small puffing segments. Depending on the purpose of the test, it may be appropriate, with sufficient evidence, to use the first collection segment as priming puffs and to treat the data differently to those that represent the bulk of usage under normal/equilibrated conditions.

Some devices can demonstrate extremely variable aerosol deliveries and selection of collection segments can influence the variability observed. When assessing a product, it is important to determine if the testing is being performed to identify unit-to-unit variability or within product variability. As an example, 5 replicates of 20 puffs (totaling a 100 puff collection) using a single unit per collection, can show less variability than 5 replicates of 20 puff collection segments using 5 unique units per collection.

Collection segments should be selected in light of the full life of the device. Depending on the desired testing, multiple collection segments may be needed to characterize the delivery from the entire device. When collecting near the end of the device, care must be taken to avoid dry puffing. Dry puffing can occur when liquid supply is insufficient, resulting in elevated coil temperature. For determination of an appropriate final collection segment, see section 3.5 on device depletion.

3.3 Activation System – Puff Activated or Button Activated

Puff activated systems require a minimum flow rate for activation and this flow rate is device dependent. This should be taken into consideration when selecting the puff duration, volume, and profile to be used with puff activated devices (see section 3.1).

Button activated systems present unique challenges with respect to synchronizing the activation of the device with the puffing mechanism. Deliveries can be influenced by factors such as activation prior to puffing (pre-heating) and the overall length of time the unit remains activated during or post-puffing. Whilst it may be appropriate, or even necessary, to include pre-heating whilst testing certain devices, post puffing activation is not recommended as it is likely to damage the heating system of the test sample. Decisions on how to activate may be device dependent, influenced by heating rate, heat transfer, and overall temperatures reached during activation.

Manual activation should be avoided as it introduces increased variability due to human interaction (lack of accuracy and technician-to-technician variability). Automated triggering systems should consider some mechanism for synchronization. If the triggering mechanism is independent of the puffing mechanism, a difference/bias of 0.01 seconds per puff could result in a cumulative difference of a full second for a 100 puff collection.

Activation button sizes, locations, and pressures can present additional challenges for simultaneous determinations of products with different configurations. Due to these complexities, device activation should be verified by an appropriate means such as observed device mass loss or visual aerosol generation.

3.4 Filling and/or Refilling of Tanks

Tank systems come in a variety of shapes and sizes. These tank systems are generally refillable and intended to be re-used.

Most tank systems have either fill levels marked on the tank or require a known volume to be added to the tank. These levels/amounts may not be accurate and can differ based on the device or manufacturer's instructions. Tanks are routinely filled per the manufacturer's instructions, but there may be circumstances to test outside (over-fill or reduced volumes) these recommended levels to gain a better understanding of device performance characteristics, particularly in terms of assessing the emissions from the device when the tank is empty. When testing a product to dryness, one should consider that the heating system (e.g. wick and coil) may have been damaged and that any subsequent emissions are unlikely to be characteristic of the product under 'normal' operating conditions.

Based on the device configuration (wicking material, number of wicks, coil position, etc.), the liquid level may change from the initial filling as the wicking material absorbs the liquid. Consideration should be given to the length of time between filling the tanks and initiating testing, whether the device needs "mixing" (e.g. inversions) to promote saturation of the wick, and if the tank volume should be readjusted once the wick or device has reached an equilibrium (saturation). Sufficient liquid should be used for the desired collection, taking into account any liquid that is absorbed into the tank materials and is no longer available for aerosolization. Care should be taken to ensure that for the device configuration being testing, appropriate wicking is possible under the testing conditions selected (rest time after filling, device orientation, fill level, etc.).

When the purpose of testing requires a tank/device is refilled, the point at which the tank is refilled (e.g. taken to near dryness, 25 % of total volume, device overheat protection etc.) should be defined, with reference to any instructions for use provided with the product, as this may impact the atomizer and influence deliveries for subsequent analysis when the tank is re-used. For a comparison of different devices with different deliveries, refilling both devices at a specified puff count can simplify the testing but may not be reflective of how the device is intended to be used.

Normally, tanks to be re-used are not puffed to full depletion. Therefore, residual liquid remains in the tank which may be slightly different from the liquid initially introduced into the tank due to differences in the distillation rates of the components of the liquid. In general, there are two options: add to the residual amount with fresh liquid or remove the residual liquid before refilling the tank. If the goal of the analysis is to reach a certain number of puffs with a given liquid, refilling until the desired number of puffs is reached is recommended. When switching to test a new liquid, cleaning the tank or utilizing a new, unused tank is recommended. Refillable devices will generally come with recommendations on when a tank should be cleaned and when the component parts, such as the atomizer, should be replaced. Cleaning and maintenance should be done in accordance with the manufacturer's instructions.

3.5 Liquid Depletion

Devices are rarely puffed to dryness, since this will lead to dry puffing of the wick and may cause device failure. Puffing to dryness should be relegated to special circumstances such as determining the total number of viable puffs that can be obtained from a device before dryness is achieved. There are several different parameters which can be used to determine the 'end point' for a device. These include (but are not limited to) a pre-defined total mass loss of the device, a determined mass loss per collection segment as a percentage of the mass loss of the initial collection segment, no visual aerosol generation, or a specified remaining tank level at the completion of a collection segment.

3.6 Battery Charging / Recharging Cycles

Routinely, batteries are fully charged at the initiation of testing. When the testing requires multiple collection segments to be determined, re-charging of the batteries may be required. The number of puffs or segments collected between re-charging is highly product dependent. Again, dependent on the purpose of testing, no re-charging or utilization of a fully charged battery with each collection may be required.

Over time, the capacity of a battery will decrease, and the battery output may be affected. Should exact output from the battery be required, it is recommended that a check of the battery is conducted using electrical testing equipment. It is also good practice to track the amount of use from each battery that has been tested to understand any effect of previous usage and age on the device performance.

3.7 Orientation of Devices During Aerosol Generation

The range of product designs that exists within the category (top coil, bottom coil, dual coil, vertical coil, etc.) means that the orientation of the product during testing may impact the performance of the device, particularly when puffing is being conducted with small amounts of e-liquid remaining in the reservoir.

For "cigalike" devices, if testing is to be conducted with the device in any orientation other than horizontal, this must be specified in the testing plan, along with the rationale for the selected orientation. For tank systems, the orientation required to facilitate appropriate wicking should be specified in the testing plan. Points of consideration when defining the orientation of devices during testing should include the product design (i.e position of wick, coil, size of device, ergonomics, etc.) and how this may affect consumer usage.

It is also necessary to ensure that the aerosol collection mechanism is not compromised by holding the device at an angle from horizontal. An appropriate seal must be maintained between the device and the collection mechanism at the collection port to avoid aerosol condensation outside of the collection mechanism.

3.8 Physical Dimensions of Devices Tested

The e-cigarette category has a vast range of products within it. The range of dimensions is incredibly broad; battery size and shape, tank size, mouthpiece size and shape, and the mass of the device may all require modifications to the port of an aerosol collection system to be made. An alternative strategy could be to fit a connective adapter that allows the device to be connected to the port.

It is necessary to ensure that the aerosol collection mechanism is not compromised by the dimensions of the device or the inclusion of a connective adapter.

- An appropriate seal must be maintained between the device and the aerosol collection system, at the collection port.
- The trapping system should be designed to avoid loss of aerosol to the trapping system, during sample collection.
    - Any insertion or connective materials should be appropriately inert with respect to any chemical analysis being conducted.
    - The ‘dead volume’ between the device and collection media should be minimized.
    - Any changes in direction of the aerosol path between the device and the collection media should be avoided.
- A physical support may be required to hold the device in place to maintain the integrity of the collection system.

3.9 Programmable Parameters of Devices Tested

Many devices contain a user interface that allows the consumer to modify the usage conditions of the device. Typical parameters that can be programmed include the power setting of the battery and the restriction of air flow through the device. These parameters must be specified in the study design and may be selected dependent on the purpose of the study (e.g. intense or non-intense usage conditions). It may be appropriate to use a statistical design of experiment approach if an assessment of the full range of programmable parameters is to be assessed.

Care should be taken when selecting the power (wattage) applied to the coil during puffing. Atomizer coils are typically supplied with a specified wattage range. Exceeding the power rating for the coil may damage the device or lead to device failure and is likely to generate an aerosol with a chemical profile that is not representative of expected usage. In general, as one increases the power setting of a device, the mass of aerosol generated will also increase. This must be considered when selecting the number of puffs in a collection segment.

Once the device settings have been defined, it may be necessary to ascertain an initial understanding of the aerosol yields at each setting. For example, one may wish to reduce the number of puffs in a collection segment as the power increases to ensure the collection media is not saturated or the device is not puffed to dryness (see section 3.2).

3.10 Handling Device Failures (battery malfunction, leakage, etc.)

Many of these devices are susceptible to failures (become non-operational) during testing. It is important when designing a study to recognize this in order to have prescribed action plans for handling and recording these types of failures, which can provide valuable information about the product. This may require additional product (batteries, tanks, cartomizer, liquid, etc.) to be available for testing should these types of failures occur. It is important to recognize potential handling issues which may cause some types of failures, and actions to prevent these types of failures happening should be implemented (e.g. e-liquid leakages to battery contacts). These types of failures should be distinguished from potential product defects causing failures.

In some cases, failures may require tests to be repeated in order to provide the adequate number of replicate observations for statistical analysis. However, this strategy would also be dependent on the amount of product available for testing. It is unlikely that any result generated by a failed device should be included in the statistical analysis of the results. However, the individual replicate data from a failed device may provide other useful information in assessing the overall product performance.

4. Data Collection

It is recommended that the device mass is recorded at the start and end of a collection segment. This allows calculation of the mass of e-liquid aerosolized during collection, enabling the device performance to be assessed against a specification. Additionally, it allows the efficiency (with respect to aerosol mass) of the trapping system (e.g. Cambridge Filter Pad or electrostatic precipitator) to be calculated.

5. References

[1] CORESTA Recommended Method N° 81 - Routine Analytical Machine for E-Cigarette Aerosol Generation and Collection - Definitions and Standard Conditions

[2] CORESTA Guide N° 18 - Sample Handling and Sample Collection of E-Cigarettes and E-Vapour Generating Products

[3] CORESTA Guide N° 22 - Technical Guide for the Selection of Appropriate Intense Vaping Regimes for E-Vapour Devices