DETERMINATION OF LEAKAGE IN E-VAPING PRODUCTS

No: 6

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Introduction and Discussion

An important quality aspect of e-vaping product is the absence of liquid leakage from the cartridge. Therefore it is highly important to verify the cartridge integrity using a reliable method.
Methods to test for occurrence of leakage under exaggerated conditions of stress can be useful, e.g. during the development of new products to ensure that leakage does not occur when new products are introduced in the market.
Various external conditions could influence product leakage and should be considered from the design phase, to the manufacturing and use of the product.

Pressure | Temperature | Movement

We propose a method that can look at the influence of these factors on leakage in different e-cigarette models.

Currently AFNOR standard (AFNOR XP D90-300-1) is the best known standard for leakage testing of e-vaping consumables. According to this standard, the absence of leakage of an e-vaping products is verified by placing the consumable on an absorbent paper, upside-down vertically and horizontally, for a minimum of 6 hours per position. The verification must be conducted at a controlled temperature of 20 °C ± 5°C.

6 hrs

&

6 hrs

Check leakage on filter paper

OK NOK

Different products tested with AFNOR test (AFNOR XP D90-300-1- [TPD Art. 20 §3 (g)] and results as follow;

Type	No of Samples	Leakage (yes) Vertical	Leakage (yes) Horizontal
Disposable	12	0	0
Cartomizer	36	0	0
Tank	36	0	0

Table 1: AFNOR results with different products

Objective

Pressure — Consumer may carry e-vaping products in a plane or a product may be transferred by plane which is close to 800 mbar

Temperature — Product may be carried by consumer and exposed to body temperature or direct sunlight

Movement — Product is exposed to mechanical stress during its use. Transferring from manufacturing center to point of sale

Objective: Develop best quantitative/visual method to determine product leakage robustness in a sequence of dynamic conditions

PMI Proposed Methods

The e-vaping consumable leakage testing method proposed by PMI R&D addresses product leakage robustness in a sequence of dynamic conditions: temperature, movement and pressure are considered as variables that can influence leakage.

Vacuum

Shaking table

Heating

The e-vaping cartridge weight is recorded individually and inserted into a glass tube within one single "qualified" testing instrument (Buchi Syncore) with multiple glass tube holders. On a pre-defined time (1 hr.) and sequence the above conditions are applied. Afterwards the cartridges are removed from the glass tubes, externally dried with a clean towel and re-weighed. The leakage is quantified by the difference in weight before and after.

Daily verification for Buchi instrument is executed by placing two vials (leaking and non-leaking) to represent products which have leakage or no leakage. If leaking vials give >200 mg and other no leakage, test can be initiated

Study Design & Evaluation Criteria

1.

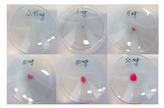
Design no	Method
A1	AFNOR Method (6 hrs vertical + 6 hrs horizontal)
A2-A8	PMI Proposed Method (apply different conditions and weigh samples before & after)

Design No	Vacuum (mbar)	Temperature (C)	Movement (yes / no)	Number of brands	Number of replica
A-1	AP	RT	No	7	12
A-2	800	RT	No		
A-3	800	RT	Yes		
A-4	800	42*	Yes		
A-5	800	42*	No		
A-6	AP	RT	Yes		
A-7	AP	42*	No		
A-8	AP	42*	Yes		

*AP: Atmospheric pressure , RT: Room temperature (22 +/- 2 C), *: 42 C in glass chamber ~36.5 C in product*

Table 2: PMI Method Study Design

Different brands of Cartomizer, Tank and Disposable system e-vaping products selected



Representation of leakages (visual)

2. Visual inspection during Aerosol collection
-Select extreme conditions (A-4) and AFNOR (A-1) conditions and run aerosol collection method.
-Observe leakage on pH paper visually for 70 samples

Evaluation of leakage pH paper on support during aerosol collection

Results

In total; 588 samples tested with PMI Method in Buchi Syncore, 84 samples with AFNOR method
No leakage observed with AFNOR method. Table 3 shows leakage with PMI method

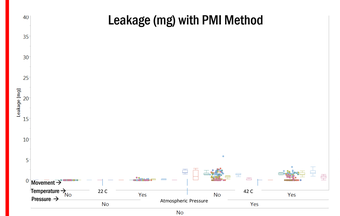
Leakage (mg)	PMI Method (588 replica)	AFNOR (84 replica)
0<x<0.15 (No leakage)	49%	0
0.15<x<1	21%	0
1< x <5	20%	0
5< x <10	0%	0
10< x <20	1%	0
20< x <50	5%	0
>50	4%	0

Table 3: PMI Method Leakage Results

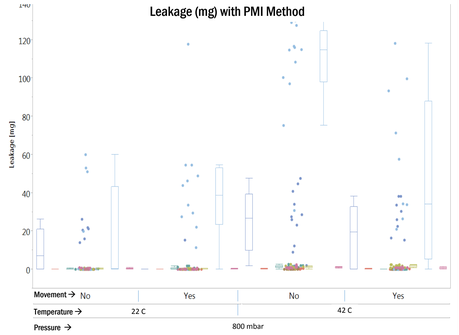
70 samples were inspected during aerosol generation.

%	Leakage after aerosol collection
7.5 mg	0
15 mg	0
130 mg	0

Table 4: Leakage after aerosol collection



No pressure + Temperature + Movement



Pressure + Temperature + Movement

Summary of Results & Conclusions

Results (AFNOR)
None of products (84) demonstrated leakage when tested with AFNOR method

Results (PMI Aerosol Collection)
None of products (70) leaked during aerosol collection is tested with PMI Method

Results (PMI Method-Pressure, Temperature, Movement)
No leakage observed in disposable products (96)
49% of screened products showed no leakage.
41 % of screened products show low levels of leakage, (less than 5 mg).
10% of screened product is leaking between 5 mg and 50 mg

Conclusions
The Buchi Syncore instrument provides an ideal apparatus that can be easily adapted to test for leakage in e-cigarette samples.
Applying negative pressure (800 mbar abs) is the condition to which is most likely to lead to leakage.
Temperature exposure (42 C) also contributes to leakage and is therefore recommended in combination with negative pressure.
Movement did not contribute to leakage for the samples investigated.
This PMI proposed method is able to simultaneously reproduce realistic conditions of products and to discriminate sample quality between batches.

Next steps/recommendations:
Optimize settings for pressure, temperature and exposure time
Use PMI method (Pressure, Temperature, Movement) during product development stage and batch control.

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