THE EUROPEAN TOBACCO PRODUCT DIRECTIVE AND E-CIGARETTE COMPLIANCE: A TEST METHOD FOR DROP TESTING OF E-LIQUID BOTTLES

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1. Introduction

In early January 2016, the European Union informed on the Draft Commission Implementing Decision technical standards for the refill mechanism of electronic cigarettes. In April, a standard for product performance detailing the requirements for e-liquid bottles was published [1]. Amongst others, this proposed technical standard specifies an e-liquid bottle performance "*that emits no more than 20 drops of refill liquid per minute when placed vertically and subjected to atmospheric pressure alone at 20 °C ± 5 °C*". At this time, no detailed testing protocol for this product requirement has been proposed.

This study was performed in order to assess potential methods and limitations for standardized product testing. Based on the results presented here, we propose a test protocol to assess product compliance.

We present our findings on the influence of external (conditioning and testing temperature) and internal factors (liquid viscosity ranging from 100% vegetable glycerol to 100% propylene glycol and bottle filling height) on the product performance. In addition, several commercially available brands and e-liquid compositions were tested.

2. Experimental Set Up

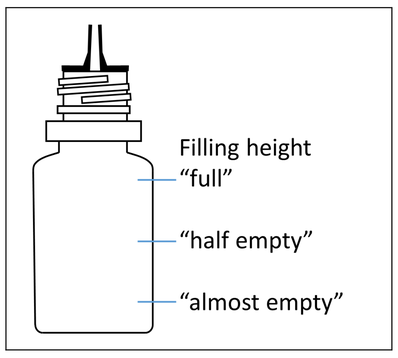
1. Method Development

- blu™ e-liquid bottles were filled with liquids 1 to 6 (see *Table 1*) at three different heights: "full", "half-empty" and "almost empty" (equal by volume of the liquids, and therefore varying weight, see *Figure 1*). Note: nozzles were cleaned before testing.
- Bottles were conditioned in closed, right-side-up position for 1 hour (see *Table 2*).
- For the test, bottles were removed from the climate oven, opened and immediately put onto a rack upside down for one minute.
- During conditioning and testing the bottles were subjected to atmospheric pressure alone.

Table 1. Composition of test liquids 1 to 6. Propylene glycol (PG), vegetable glycerol (VG) and water (H2O), all in %(w/w).

	1	2	3	4	5	6
PG	0	25	50	75	100	90
VG	100	75	50	25	0	0
H2O	0	0	0	0	0	10

Figure 1. Filling heights of e-liquid bottles.



2. Benchmark Test

- Bottles were conditioned and tested at 21±1°C.

Table 2. Conditioning/ test temperatures.

Range for conditioning:	Temperatures during testing:
20°C to 40°C (in 4°C steps)	20°C to 37°C

3. Results

Observations (see *Tables 3, 4 and 5*)

- When $T_{conditioning} < T_{testing}$:
  - Drops are released over the course of 30 to 45 seconds where drip rate decreases over time.
  - Number of drops increases with decreasing viscosity and filling height, with an increasing ΔT.
- When $T_{conditioning} \geq T_{testing}$:
  - Drops still attached to the nozzle retract.

Interpretation

- When $T_{conditioning} < T_{testing}$ and the bottle is turned upside down immediately after removing from climate oven, the air volume in the bottle changes (density adjusts to the new temperature) and by that "squeezing" the liquid out through the nozzle until the air density reaches equilibrium again.
- This effect is greater with increasing gas volume, i.e. decreasing fill height.

Benchmark

- A number of different e-liquid brands/ bottle types (A-E) with various flavour types (1-3) were assessed (n = 3).
- Chemical analysis showed that base liquid composition covered a range of propylene glycol 32% to 68%, vegetable glycerol 19% to 62% and water 0% to 14%.
- The results are shown in *Table 6*. All tested products were found to be compliant with the EU standard.

Table 3. Number of drops released from the bottle during testing. $T_{conditioning}$ = 20°C, $T_{testing}$ = 27°C.

Liquid No.	1	2	3	4	5	6
Full	0					1
Half-full						
Almost empty	3					7

Table 4. Number of drops released from the bottle during testing. $T_{conditioning}$ = 20°C, $T_{testing}$ = 37°C.

Liquid No.	1	2	3	4	5	6
Full	1					4
Half-full						
Almost empty	4					11

Table 5. Number of drops released from the bottle during testing. $T_{conditioning}$ = 40°C, $T_{testing}$ = 27°C.

Liquid No.	1	2	3	4	5	6
Full	0					0
Half-full						
Almost empty	0					0

Table 6. Number of drops released from the bottle during testing. $T_{conditioning}$ = 21±1°C, $T_{testing}$ = 21±1°C. A … E = e-liquid brand/ bottle type, 1 … 3 = flavour types of said brands

Liquid No.	A1	A2	A3	B1	B2	B3	C1	C2	C3	D1	D2	E1
Full	1	0	0	1	0	1	0	0	1	0	2	0
Half-full	2	3	1	2	2	2	1	3	1	0	2	1
Almost empty	2	3	3	2	3	2	2	2	1	2	3	1

4. Test Method Proposal

Based on the requirements described in the EU Commission Implementing Decision (EU) 2016/586 [1], and the results of our findings presented here, the following test method is perfectly suitable and could be proposed for discussion with standardization bodies:

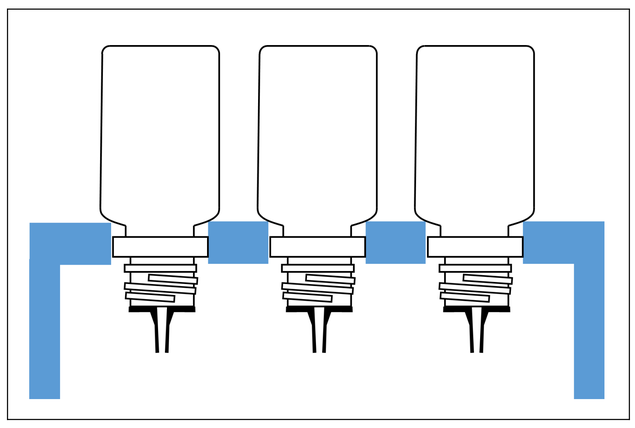
Apparatus

- A rack in which the e-liquid bottles can be placed vertically without subjecting them to any pressure (see *Figure 2*).

Procedure

- Preparation of the samples:
  E-liquid bottles shall be conditioned at 20±5°C.
- Determination of drip rate:
  Bottles are opened and placed vertically onto a rack at 20±5°C and drops are counted for exactly one minute after placing the bottles onto the rack, after which the test is complete.

Figure 2. Proposed apparatus for leakage testing of e-liquid bottles. The bottles are placed onto the rack vertically and are not subject to any other but atmospheric pressure.



5. Conclusions

Fontem Ventures supports sound, evidence-based, reasonable and proportionate regulation of e-cigarettes, as well as the introduction of robust product standards. Both of which should ensure increased consumer confidence in the e-cigarette category.

The proposed Implementing Decision provides a good basis for harmonizing standards across the EU and for protecting e-cigarette users against accidental exposure to e-liquids. At this time, no method for drop testing for e-liquid bottles has been proposed.

Our study was performed in order to assess potential methods and their limitations for standardized product testing.

Here we propose a test protocol to assess EU product compliance. This protocol could be considered for the development of a testing standard, for example by the ISO sub-committee TC126/SC3 dedicated to e-cigarettes and related vaping products.

References

[1] Commission Implementing Decision (EU) 2016/586 on technical standards for the refill mechanism of electronic cigarettes, 14 April 2016