

Report n° 14/282

Analysis of e-liquid and e-cigarettes
Vaporing test results

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Study for :	INSMOKE
Attention of :	Mr MEILE
Customer request nr :	Agreement dated 29.04.14
Certechem request nr:	T277
Certechem quotation nr :	OP/14/322/TP

Issue date : **30 May 2014**

Scientific Approval,

Quality Approval,

1. Introduction

The purpose of this study, ordered by Mr Meile from InSmoke is to evaluate the nature and concentration of aldehydes/ketones and metals in the exhaust air of e-cigarettes. The methodology of this vaping trial has been developed by Certech.

The e-cigarettes have been received the 14/04/14, the e-liquid the 03/04/14. References of the samples are summarized in table 1.

2. Materials and methods

2.1 Materials and samples

The samples were received the 03 and 14 April 2014. The references are presented in Table 1.

Your reference	Our reference
Stardust (without nicotine)	404E004
InSmoke Reevo Mini-S	405E006
RB1632 InSmoke Shisha Apple Crush	405E007

Table 1: Sample references

2.2 Methods

The testing protocol is described in the figure 1 below.

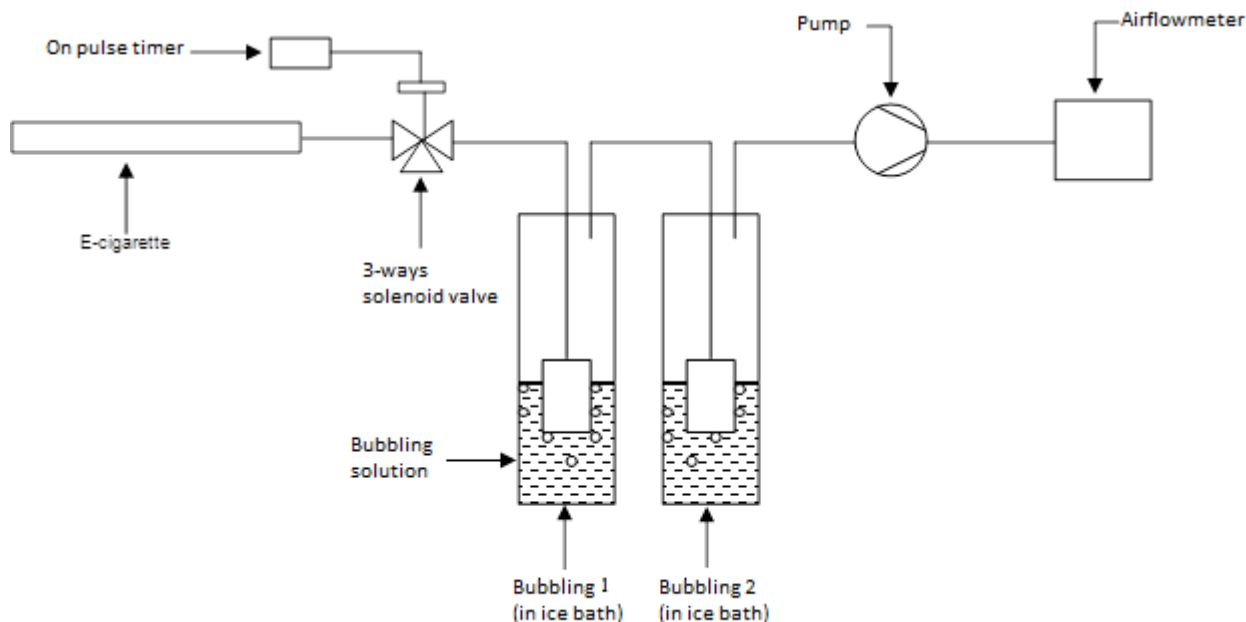


Schéma 1. Set-up of the vaping test protocol

In this set-up, the electronic cigarette (cartridge full of e-liquid) is connected to a 3-ways valve. The connection between the e-cigarette and the valve is made of silicone (< 1cm). The valve is made of stainless steel and Téflon® and is connected to a timer programmed to generate a 3-seconds-puff every 30 seconds.

The aerosol generated is trapped in a first bubbler placed in an ice bath. The connection between the valve and the first bubbler is made of Téflon® (<1cm). The bubbling solution is selected in function of the compounds to analyse. A second bubbler is also connected to ensure the trapping of the compounds which would not have been trapped in the first one. At the outlet of the second bubbler, a pump is connected, set at an airflow rate of around 1000mL/min (measured precisely by a bubble flowmeter).

Practically, a constant airflow is applied by the pump. The valve combined with the timer ensures an air intake through the e-cigarette only during 3 sec., pure air being drawn during the following 30 seconds.

The air of the laboratory is controlled by a conditioning air unit, allowing the generation of pure air by mean of activated carbon filter, regulated in terms of temperature and humidity. The air flow rate in the laboratory is very high to reach an important air exchange rate.

Finally, a blank analysis is performed with the same set-up but without any e-cigarette to evaluate the background concentrations.

2.3 Aldehydes/ketones analysis in the exhaust air

The bubbling has been performed in a solution containing a derivatisation agent, the DNPH (dinitrophénylhydrazine) in order to form stable derivatives. The analysis* has been realised by HPLC-UV with external calibration.

* *Standard list: Formaldehyde, acetaldehyde, acetone, acroleine, propionaldehyde, crotonaldehyde, butyraldehyde, benzaldehyde, isovaleraldehyde, valeraldehyde, o-tolualdehyde, m+p-tolualdehyde, hexaldéhyde +2,5-dimethylbenzaldéhyde.*

2.4 Metals analysis

The bubbling has been performed in an acid solution (miliQ water acidified with 1% nitric acid at 65%). The analysis** has been realised by ICP-MS in a partner lab certified ISO 17025 for this measurement.

** *Standard list : Calcium (Ca), Magnesium (Mg), Natrium (Na), Potassium (K), Bore (B), Aluminium (Al), Vanadium (V), Chrome (Cr), Iron (Fe), Cobalt (Co), Manganèse (Mn), Nickel (Ni), Cuivre (Cu), Zinc (Zn), Arsenic (As), Silicium (Si), Sélénium (Se), Strontium (Sr)⁽¹⁾, Molybdène (Mo), Argent (Ag)⁽¹⁾, Etain (Sn), Cadmium (Cd), Antimoine (Sb), Barium (Ba), Plomb (Pb), Uranium (U)⁽¹⁾, Beryllium (Be)⁽¹⁾, Mercure (Hg), Phosphore total (PO4tot).*

⁽¹⁾ compounds out of the scope of the ISO 17025 accreditation of the partner laboratory.

3. Results

3.1 Aldehydes/ketones

The table 2 hereafter presents the parameters of the tests:

- Columns 1 and 2 mention the references of the samples
- Column 3 indicates the day of the test
- Column 4 is for the consumption of e-liquid during the test. It has been calculating by weighing the sample before and after the test
- Column 5 indicates the volume of each puff. This volume is calculated from the air intake rate measured during the test and the length of the puff (3 seconds)
- Column 6 mentions the duration of the whole test (in min.)
- Column 7 mentions the total amount of puffs, calculated from the duration of the test and the test conditions (one 3-seconds-puff every 30 seconds).

Table 2. Experimental parameters for the quantification of carbonyl compounds in the exhaust air

Samples references		Date of the test	Amount of e-liquid used (g)	Volume per puff (ml)	Duration of the test (min)	Amount of puffs
InSmoke Reevo Mini-S + e-liquid stardust	405E006 + 404E004	14/05/2014	0,15	50	55	100
RB1632 InSmoke Shisha Apple Crush	405E007	14/05/2014	0,22	50	55	100
Blank aldehydes	/	14/05/2014	NA	50	55	100

Table 3 presents the results obtained after analysis of the bubblers by HPLC-UV according to the list of compounds mentioned in point 2 of the report. Results are expressed in $\mu\text{g}/100$ puffs.

Table 3. Results of aldehydes/ketones analysis in the aerosol (in μg for 100 puffs)

Results expressed in μg for 100 puffs	e-Cigarette	Blank	InSmoke Reevo Mini-S 405E006	RB1632 InSmoke Shisha Apple Crush 405E007
	Liquid		Stardust 404E004	-
	Number of puffs	100	100	100
	formaldehyde	<0,7	2,3	18,5
	acetaldehyde	ND	1,5	3,4
	acetone	<0,7	<0,7	8,3
	acrolein	ND	10,6	6,2
	propionaldehyde	2	2,2	13,5
	crotonaldehyde	ND	ND	16
	butyraldehyde	ND	ND	ND
	benzaldehyde	ND	ND	26,1

	isovaleraldehyde	ND	ND	ND
	valeraldehyde	ND	ND	ND
	o-tolualdehyde	ND	ND	3,2
	m+p-tolualdehyde	ND	ND	ND
	hexaldehyde + 2,5-dimethylbenzaldehyde	<0,7	1,5	1,5
	Detection limit	0,2	0,2	0,2

Values obtained for the blank test are reported in the table and not subtracted to the sample results.

3.2 Metals

The table 4 hereafter presents the parameters of the tests:

- Columns 1 and 2 mention the references of the samples
- Column 3 indicates the day of the test
- Column 4 is for the consumption of e-liquid during the test. It has been calculating by weighing the sample before and after the test
- Column 5 indicates the volume of each puff. This volume is calculated from the air intake rate measured during the test and the length of the puff (3 seconds)
- Column 6 mentions the duration of the whole test (in min.)
- Column 7 mentions the total amount of puffs, calculated from the duration of the test and the test conditions (one 3-seconds puff every 30 seconds).

Table 4. Experimental parameters for the quantification of metals in the exhaust air

Samples references		Date of the test	Amount of e-liquid used (g)	Volume per puff (ml)	Duration of the test (min)	Amount of puffs
InSmoke Reevo Mini-S + e-liquid stardust	405E006 + 404E004	13/05/2014	0,18	50	55	100
RB1632 InSmoke Shisha Apple Crush	405E007	13/05/2014	0,28	50	55	100
Blank aldehydes	/	13/05/2014	NA	50	55	100

The table 5 presents the results obtained after analysis of the bubblers by ICP-MS according to the list of compounds listed in point 2. Results are expressed in µg for 100 puffs.

Values obtained for the blank test are reported in the table and not subtracted to the sample results.

Tableau 5. Results obtained for the metals analysis (in µg for 100 puffs)

			BLANK	InSmoke Reevo Mini-S 405E006	RB1632 InSmoke Shisha Apple Crush 405E007
				Stardust 404E004	-
Number of puffs			100	100	100
Air intake through the e-cigarette (L)			5	5	5
E-liquid consumption (g)			/	0,18	0,28
Quantity in µg / 100 puffs					
Mercure	Hg	µg	ND (<0,005)	ND (<0,005)	ND (<0,005)
Uranium	U	µg	ND (<0,003)	ND (<0,003)	ND (<0,003)
Calcicum	Ca	µg	ND (<200)	ND (<200)	ND (<200)
Magnésium	Mg	µg	ND (<10)	ND (<10)	20
Sodium	Na	µg	30	ND (<20)	ND (<20)
Potassium	K	µg	ND (<25)	ND (<25)	ND (<25)
Silice	Si	µg	ND (<10)	10	20
Aluminium	Al	µg	1,8	1,7	1,8
Bore	B	µg	0,5	1,5	0,6
Fer	Fe	µg	2,4	2,4	2,8
Manganèse	Mn	µg	0,3	0,2	0,4
Cuivre	Cu	µg	0,3	0,2	0,3
Zinc	Zn	µg	24,4	9,2	30,8
Etain	Sn	µg	0,3	0,2	0,2
Cobalt	Co	µg	ND (<0,1)	ND (<0,1)	ND (<0,1)
Baryum	Ba	µg	1,3	1,2	1,3
Argent	Ag	µg	ND (<0,05)	ND (<0,05)	ND (<0,05)
Arsenic	As	µg	<0,1	<0,1	<0,1
Beryllium	Be	µg	ND (<0,01)	ND (<0,01)	ND (<0,01)
Cadmium	Cd	µg	ND (<0,005)	<0,1	ND (<0,005)
Chrome	Cr	µg	ND (<0,05)	ND (<0,05)	ND (<0,05)
Nickel	Ni	µg	0,1	0,1	0,1
Plomb	Pb	µg	0,5	0,3	0,6
Antimoine	Sb	µg	<0,1	<0,1	0,1
Selenium	Se	µg	ND (<0,03)	ND (<0,03)	ND (<0,03)
Vanadium	V	µg	ND (<0,03)	ND (<0,03)	ND (<0,03)
Strontium	Sr	µg	ND (<0,3)	ND (<0,3)	ND (<0,3)
Molybdène	Mo	µg	0,1	<0,1	0,1
Phosphate totaux	PO4	µg	ND (<10)	ND (<10)	ND (<10)

Annexe A. Conditions analytiques

Conditions analytiques pour les analyses par GC-MS

GC-MS ANA 23.03

GC : HP 6890

MS : HP 5973

Colonne : Zebron ZB-WAX (60m x 0,25mm x 0,5 μ m)

Gaz vecteur: He 6.0

Conditions analytiques pour les analyses par HPLC-UV

HPLC: Waters Alliance 2690

Colonne : Waters Symmetry[®] C18

Détecteur : Waters PDA 996, λ = 360 nm