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Report n° 15/614

Analysis of e-liquid and evaluation of volatile organic compounds produced by e-cigarette

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Study for :	**Firstswissventures**
Attention of :	**Rolf Schuler**
Customer request nr :	**Agreement to the quotation dated 22.09.15**
Certech request nr:	**U586**
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Scientific Approval,

Quality Approval,

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Executive summary

This study is relating to the request coming from Firstswissventures AG for the analyses of one e-liquid and the corresponding aerosols generated with the electronic cigarette.

The analysis of propylene glycol and glycerine in the sample showed a proportion of approximately 50/50 for the sample Sparrow.

The concentration of nicotine in the sample Sparrow is 18 mg/ml.

The VOCs detected in the aerosol are propylene glycol, glycerine and nicotine.

Some aldehydes/ketones compounds were detected in the aerosol: formaldehyde, acetaldehyde, acetone, acrolein, propionaldehyde

1. Introduction

This study is relating to the request coming from Firstswissventures AG for the analyses of one e-liquid and the corresponding aerosols generated with the electronic cigarette.

2. Materials and methods

Materials and samples

Samples were delivered on 1st October 2015. References are mentioned in the table 1.

Table 1. Sample references

Samples	Nicotine	Certech references
Sparrow	Yes	510E006

Methods

Analysis of e-liquid

The liquid was sampled from the cartridge. Then, the aliquot was diluted with methanol before analysis by gas chromatography and mass spectrometry (GC-MS). The following compounds were quantified with their specific external standards: propylene glycol, glycerine and nicotine.

Analysis of vapour emitted by the electronic cigarette

Vapours and aerosols were generated using a home-made system customized to simulate puffing conditions of real EC users. Puff duration of 3 seconds and intervals between puffs of 30 seconds were chosen according to a former literature study. A fully charged battery was used.

The system is presented on figure 2 with the e-cigarette connected to the trapping device via an electronic valve. The trapping device consists of two impingers containing 100 ml of a bubbling solution. Bubbling solution is methanol in case of VOC analysis and a solution of DNPH (2,4-dinitrophenylhydrazine) in acetonitrile in case of aldehydes/ketones analysis.

Two pumps are connected to the device. A first one (pump 1) calibrated at around 1 l/min is connected to the device to ensure a sufficient airflow rate for the activation of the e-cigarette. The pump 1 is working continuously. Using the valve, air is pumped discontinuously through the trapping device and through the e-cigarette for 3 seconds every 30 seconds via pump 2, calibrated at around 500 mL/min. During the 30-seconds intervals, the air pumped through the trapping device is pure air. For each 3-seconds puff, the volume of air pumped is around 25 ml.

All connections are as short as possible to avoid adsorption and condensation of e-liquid in the connection.

Blank sample was collected using the same sampling line without any e-cigarette.

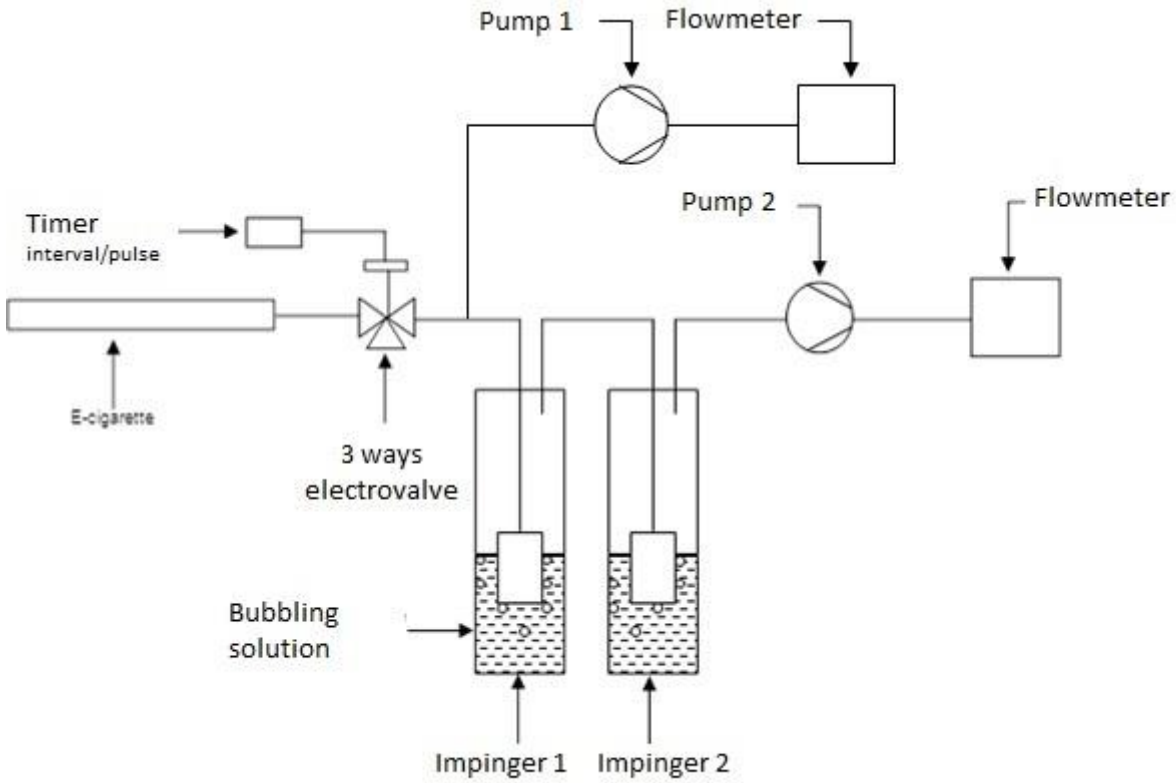

Figure 1. Diagram of the device used to evaluate VOC in the aerosol of e-cigarette.

VOC analysis of the e-cigarette aerosols

For VOC analysis, two impingers filled with 100 ml of methanol were used. Both impingers were placed in an ice bath. After testing, bubbling solutions were adjusted to 100 ml with methanol (to balance evaporation phenomenon during the test). Both bubbling solutions were analyzed separately by gas chromatography and mass spectrometry. The second trapping device ensured the complete trapping of VOC present in the EC vapour.

Table 2 summarizes parameters of the test. Test was stopped after 100 puffs (before total consumption of the e-liquid and/or of the battery).

After puffing session, the solutions were analyzed by GC-MS.

Table 2. Protocol parameters for the analysis of VOC in EC vapour

Samples	Date of the test	Amount of e-liquid consumed during the test (mg)	Volume per puff (ml)	Duration (min)	Number of puff	Total volume of vapour (L)
510E006 Sparrow	15/10/15	47	25,7	55	100	2,57

Aldehydes/ketones analysis of the e-cigarette aerosols

The aldehydes/ketones compounds emitted by the e-cigarettes were analysed according to the same protocol as VOC compounds. The difference is the bubbling solution: 2,4-dinitrophenylhydrazine (DNPH) in acetonitrile instead of methanol. Here again, two impingers were used to ensure complete trapping of volatiles compounds.

Table 3 summarizes the parameters of the test. The test was stopped after 100 puffs (before total consumption of the e-liquid and/or of the battery).

After puffing session, the solutions were analyzed by HPLC-UV.

Table 3. Protocol parameters for the analysis of aldehydes in EC vapour

Samples	Date of the test	Amount of e-liquid consumed during the test (mg)	Volume per puff (ml)	Duration (min)	Number of puff	Total volume of vapour (L)
510E006 Sparrow	15/10/15	61,9	26,0	55	100	2,6

3. Results

3.1. Analysis of e-liquid

Results obtained for propylene glycol, glycerine and nicotine are presented in table 4.

Table 4. Propylene glycol (PG), glycerine (GLY) and nicotine concentrations in the e-liquid

Sample	PG (mg/ml)	GLYC (mg/ml)	Nicotine (mg/ml)
510E006 Sparrow	375	323	18

No other compound was detected after GC-MS analysis of the e-liquid.

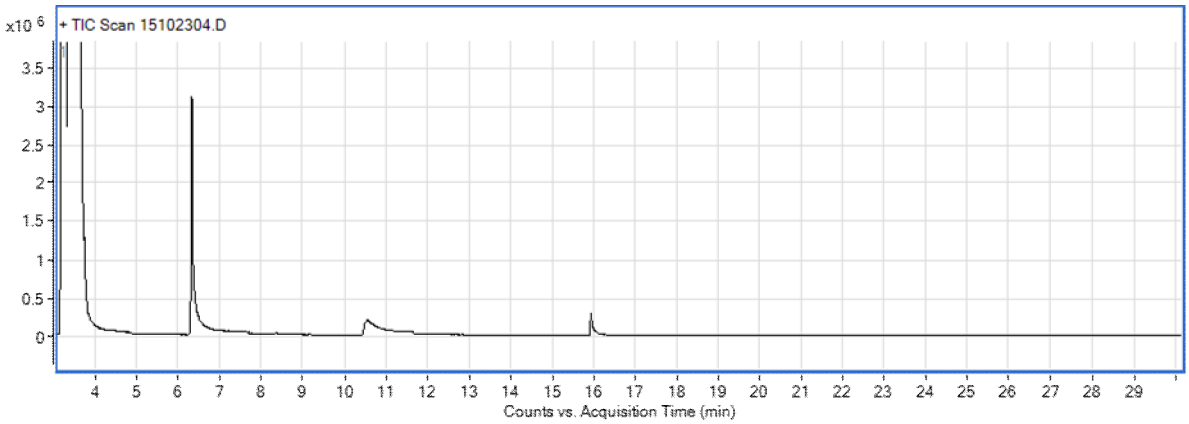

Figure 2. Chromatogram of the e-liquid analysis

3.2. Analysis of volatile organic compounds in the aerosol produced by e-cigarette

Table 5 presents the results obtained for propylene glycol, glycerine and nicotine in the vapour phase.

Table 5. Results obtained for PG, glycerine and nicotine ï sample 510E006 Sparrow

510E006 Sparrow	Total (mg in both impingers)	µg/puff	µg/m³
Propylene glycol	5,7	57,3	22,9
Glycerine	<109	<109	<43,4
Nicotine	0,2	2,4	0,9

No other compounds was detected after the GC-MS analysis of the impingers after puffing sessions. The detection limit is 10,9 mg or 109 µg/puff (due to high dilution during the sampling in impingers and due also to the vapour volume going through the impingers (2,5L)).

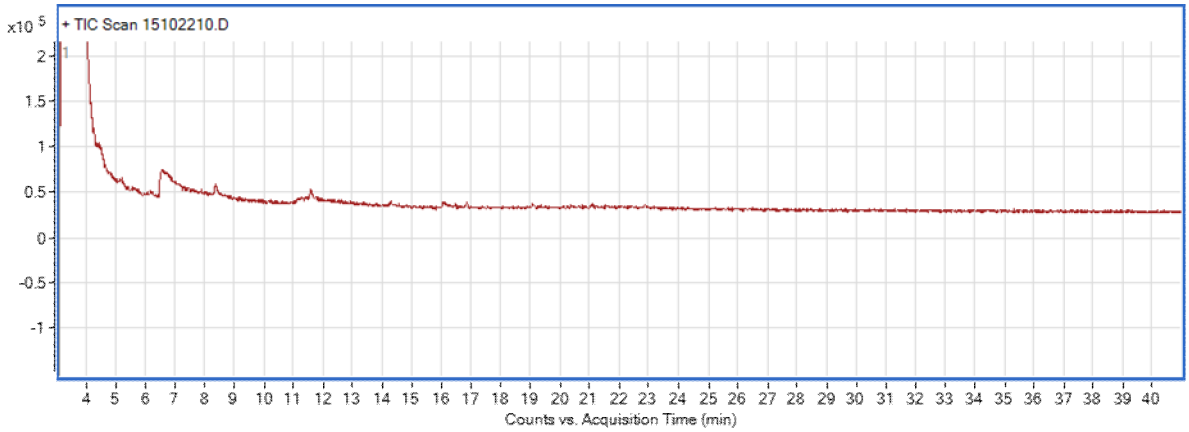

Figure 3. Chromatogram of the GC-MS analysis after 100 puffs

3.3. Analysis of aldehydes/ketones compounds in the aerosol produced by e-cigarette

Table 6 presents the results obtained for aldehydes/ketones analyses in the vapour phase of the electronic cigarette. Aldehydes/ketones concentrations detected in the blank sample were not subtracted to the final results.

Table 6. Results for aldehydes/ketones – Sample 510E006 Sparrow

Sample 510E006 Sparrow	BLANK	µg / both impingers	ng/puff	µg/m^3
formaldehyde	<0,5	<0,5	<5	<192
acetaldehyde	ND	0,8	8	308
acetone	ND	<0,5	<5	<192
acrolein	ND	1,0	10	385
propionaldehyde	ND	<0,5	<5	<192
crotonaldehyde	ND	ND	ND	ND
butyraldehyde	ND	ND	ND	ND
benzaldehyde	ND	ND	ND	ND
isovaleraldehyde	ND	ND	ND	ND
valeraldehyde	ND	ND	ND	ND
o-tolualdehyde	ND	ND	ND	ND
m+p-tolualdehyde	ND	ND	ND	ND
hexaldehyde + 2,5-dimethylbenzaldehyde	ND	ND	ND	ND
Detection limit	0,2	0,2	2	64

4. Conclusion

This study is relating to the request coming from Firstswissventures AG for the analyses of one e-liquid and the corresponding aerosols generated with the Sparrow electronic cigarette.

The analysis of propylene glycol and glycerine in the sample showed a proportion of approximately 50/50 for the sample Sparrow.

The concentration of nicotine in the sample Sparrow is 18 mg/ml.

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