Spark-Lab
Analyses for industry and science

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Report number:
2022/05/0008/011/EN

ANALYSIS REPORT

Particulars of the Client	Description	Order number
SYMETRICUS Sp. z o. o. ul. Stężycka 107 m. 1 80-174 Gdańsk	Quantitative determination of aerosol components derived from e-liquid, according to agreed specification.	ZO 2022/04/000065

The analyses have been conducted by:
Laboratorium Analiz Chemicznych Spark-Lab Sp. z o.o.
Research and Development Dept.

Date of commencement of analyses	12.05.2022
Date of completion of the analyses	24.05.2022

Sample identification:

Sample signature	Sample designation	Sample collection method	Additional information	
2022/05/0008/011	Aroma King CBD Mama Huana Bubble Gum 250mg	Sample collected and delivered by the client.	Date of delivery:	28.04.2022
			Object of analysis:	e-liquid with CBD
			Sample evaluation:	good

Results:

1. **Sample mass and puffs number for cannabidiol, tobacco-specific nitrosamines, aldehydes and ketones determination.**

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Sample mass at 40 puffs. Vaping process to acetonitrile.	SL/2020/036 Ed. 1 of 03.09.2020, NA	0,289	-	g

2. **Sample mass and puffs number for nicotine, propylene glycol, glycerin, volatile organic compounds determination.**

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Sample mass at 40 puffs. Vaping process to methanol.	SL/2020/036 Ed. 1 of 03.09.2020, NA	0,259	-	g

3. **Sample mass and puffs number for heavy metals determination.**

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Sample mass at 40 puffs. Vaping process to water.	SL/2020/036 Ed. 1 of 03.09.2020, NA	0,214	-	g

4. **Results of the heavy metals determination.**

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Content of lead **Pb**	SL/2020/042 Ed. 1 of 03.09.2020, NA	<LOQ	-	µg/g
	Content of cadmium **Cd**		<LOQ	-	
	Content of arsenic **As**		<LOQ	-	
	Content of chrome **Cr**		<LOQ	-	
	Content of nickel **Ni**		<LOQ	-	
	Content of copper **Cu**		21,3	2,1	

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5. Results of volatile organic compounds determination.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Average content of **benzene**	SL/2020/037 Ed. 1 of 03.09.2020, NA	<LOQ	-	µg/g
	Average content of **toluene**		<LOQ	-	
	Average content of **isoprene**		<LOQ	-	
	Average content of **1,3-butadiene**		<LOQ	-	
	Average content of **ethylene glycol**		<LOQ	-	
	Average content of **diethylene glycol**		<LOQ	-	

6. Results of aldehydes and ketones determination.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Average content of **formaldehyde**	SL/2020/040 Ed. 1 of 03.09.2020, NA	22,58	0,60	µg/g
	Average content of **acetaldehyde**		17,74	0,75	
	Average content of **acrolein**		36,92	0,24	
	Average content of **crotonaldehyde**		<LOQ	-	
	Average content of **diacetyl**	SL/2020/037 Ed. 1 of 03.09.2020, NA	<LOQ	-	
	Average content of **acetyl propionyl**		<LOQ	-	

7. Results of tobacco-specific nitrosamines determination.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Average content of **NNK**	SL/2020/039 Ed. 2 of 22.10.2020, NA	<LOQ	-	µg/g
	Average content of **NNN**		<LOQ	-	

8. Results of cannabidiol determination.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Uncertainty	Unit
2022/05/0008/011	Average content of **cannabidiol**	modified, SL/2019/036 Ed. 8 of 13.01.2022, NA	0,69	0,06	%
	Average number of **puffs**	NA	40	-	-
	Average cannabidiol **dose per puff**	NA	0,012	0,001	mg/puff

9. Results of nicotine, propylene glycol and glycerin determination after heating e-liquid.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2022/05/0008/011	Average content of **propylene glycol**	SL/2020/038 Ed. 1 of 03.09.2020, NA	301,0	0,6	mg/g**
	Average content of **glycerin**		444,7	2,7	
	Average content of **nicotine**		<LOQ	-	
	Average number of **puffs**	NA	40	-	-
	Average nicotine **dose per puff**		<LOQ	-	mg/puff

*Determination method: A-accredited, NA- non-accredited, AS-accredited by the subcontractor, NAS- not accredited by the subcontractor.
** amount of nicotine [mg] per 1 g of vaped liquid
LOQ – limit of quantification

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Additional information:

I. Sampling conditions:

Samples of aerosols were taken in the SMOKY-LAB apparatus. Sampling parameters:
- The air flow through the system was 1,1 L/min.
- The test consists of 3 sec. puff and 27 sec. relaxation time interval.
- Heater resistance: 1,5 Ω.
- The voltage applied to the heater: 3,7 V.
- The temperature of transfer line was set in the range of 80-100 °C.

II. Heavy metals determination method:

The aerosol was collected into the ultrapure water with nitric acid (trace analysis quality) in the absorber. The samples were analyzed directly on Agilent ICP-OES VDV 5100 System in the axial mode. The cyclon chamber and glass nebulizer was used. The RF Power was 1,20 kW and the plasma flow of argon was 12 L/min.

Table 1. The limits of quantification of heavy metals.

Subject of designation	Unit	Limit of quantification
Content of lead **Pb**	µg/g	10,00
Content of cadmium **Cd**	µg/g	10,00
Content of arsenic **As**	µg/g	10,00
Content of chrome **Cr**	µg/g	10,00
Content of nickel **Ni**	µg/g	10,00
Content of copper **Cu**	µg/g	10,00

III. Volatile Organic Compounds determination method:

The aerosol was collected to methanol in the absorber. Analysis of the standard solutions and the samples was performed with gas chromatography combined with mass spectrometry Shimadzu GCMS-QP2010 SE System. The quantitative analysis were performed in split injection mode by gradient temperature program and SIM detector mode. The Zebron WAX column was used with parameters: 30 m length; 0,25 I.D. mm and 0,25 µm of film thickness.

Table 2. The limits of quantification of volatile organic compounds.

Subject of designation	Unit	Limit of quantification
Content of **benzene**	µg/g	50,0
Content of **toluene**	µg/g	50,0
Content of **isoprene**	µg/g	50,0
Content of **1,3-butadiene**	µg/g	50,0
Content of **ethylene glycol**	µg/g	250,0
Content of **diethylene glycol**	µg/g	50,0

IV. Aldehydes and ketones determination method:

The aerosol was collected to acetonitrile in the absorber. The analytes were derivatized in acetonitrile solution by 2,4-DNPH (dinitrophenylhydrazine in phosphoric acid). Analysis of the standard solutions and the samples was performed using ultraperformance liquid chromatography with diode-array detector coupled with tandem mass spectrometry UHPLC-PDA/MS/MS Shimadzu Nexera X2 8040. The ACE C18-PFP; 100x2.10; 1.7µm; 100A column with precolumn was used for the determinations.

Table 3. The limits of quantification of aldehydes and ketones.

Subject of designation	Unit	Limit of quantification
Content of **formaldehyde**	µg/g	2,47
Content of **acetaldehyde**	µg/g	3,40
Content of **acrolein**	µg/g	4,11
Content of **crotonaldehyde**	µg/g	4,85

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Subject of designation	Unit	Limit of quantification
Content of **diacetyl	µg/g	50,0
Content of **acetyl propionyl	µg/g	25,0

**The aerosol was collected to methanol in the absorber. Analysis of the standard solutions and the samples was performed with gas chromatography combined with mass spectrometry Shimadzu GCMS-QP2010 SE System. The quantitative analysis were performed in split injection mode by gradient temperature program and SCAN and SIM detector mode. The Zebron WAX column was used with parameters: 30 m length; 0,25 I.D. mm and 0,25 µm of film thickness.

V. Tobacco-specific nitrosamines determination method:

The aerosol was collected to acetonitrile into the absorber. Analysis of the standard solutions and the samples was performed using ultraperformance liquid chromatography with diode-array detector coupled with tandem mass spectrometry UHPLC-PDA/MS/MS Shimadzu Nexera X2 8040. The ACE-PFP C18; 100x3.00; 2.6µm; 100A column with precolumn was used for the determinations.

Table 4. The limits of quantification of tobacco-specific nitrosamines.

Subject of designation	Unit	Limit of quantification
Content of **TSNA: 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK)**	µg/g	2,5
Content of **TSNA: N-nitrosonornicotine (NNN)**		2,5

VI. Nicotine, propylene glycol and glycerin determination method:

The aerosol was collected to methanol into the absorber. Analysis of the standard solutions and the samples was performed with gas chromatography combined with flame ionization detector Shimadzu GC2010 Plus System. The quantitative analysis were done in split injection mode by isothermal and gradient temperature program. The Zebron ZB-624 column was used with parameters: 30 m length; 0,32 I.D. mm and 1,8 µm of film thickness.

Table 5. The limits of quantification of nicotine.

Subject of designation	Unit	Limit of quantification
Content of **nicotine in aerosol after heating**	mg/g	2,0
Average nicotine **dose per puff**	mg/puff	0,051

Supplements, method deviations:

Method SL/2019/036 ed.8 was modified - matrix of the sample was changed.

Approved of the results and report	Dokument podpisany przez JAKUBOWSKA; LABORATORIUM ANALIZ CHEMICZNYCH SPARK-LAB SP. Z O.O. Data: 2022.05.24 14:11:40 CEST	Dokument podpisany przez Dr Łukasz Marian Guzik; Laboratorium Spark-Lab Data: 2022.05.24 14:58:58 CEST

END OF REPORT