ANALYSIS REPORT

Particulars of the Client	Description	Order number
EXPRAN Sp. z o.o. ul. Fabryczna 3 26-200 Końskie	Quantitative determination of aerosol components derived from e-liquids, according to agreed specification. Quantitative determination of nicotine in e-liquid.	ZO 2020/11/000088

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

Date of commencement of analyses	07.12.2020
Date of completion of the analyses	23.12.2020

Sample identification:

Sample signature	Sample designation	Sample collection method	Additional information	
2020/12/0005/001	Buttermilch	Sample collected and delivered by the client.	Date of delivery:	30.11.2020
			Object of analysis:	Viscous e-liquid
			Sample evaluation:	unresevedly

Results:

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

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	Sample mass at 40 puffs. Vaping process to methanol.	SL/2020/036 Ed. 1 of 03.09.2020, NA	0,247	-	g

2. **Sample mass and puffs number for tobacco-specific nitrosamines and aldehydes and ketones determination.**

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	Sample mass at 40 puffs. Vaping process to acetonitrile.	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
2020/12/0005/001	Sample mass at 40 puffs. Vaping process to water.	SL/2020/036 Ed. 1 of 03.09.2020, NA	0,425	-	g

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

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	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**		<LOQ	-	

ANALYSIS REPORT

5. Results of the content of volatile organic compounds.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	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 content.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	Average content of **formaldehyde**	SL/2020/040 Ed. 1 of 03.09.2020, NA	4,34	0,14	µg/g
	Average content of **acetaldehyde**		4,41	0,56	
	Average content of **acrolein**		<LOQ	-	
	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
2020/12/0005/001	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 the nicotine, propylene glycol, glycerin and aroma compounds determination after heating e-liquid.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Standard Deviation	Unit
2020/12/0005/001	Average content of **nicotine**	SL/2020/038 Ed. 1 of 03.09.2020, NA	< 2,0	-	mg/g**
	Average content of **propylene glycol**		416,0	18,8	mg/g
	Average content of **glycerin**		364,8	12,3	
	Avarage of **vanilin**	SL/2020/054 Ed. 1 of 16.12.2020, NA	1,888	0,043	mg/g
	Average number of **puffs**	NA	40	-	-
	Average nicotine **dose per puff**	NA	< 0,051	-	mg/puff

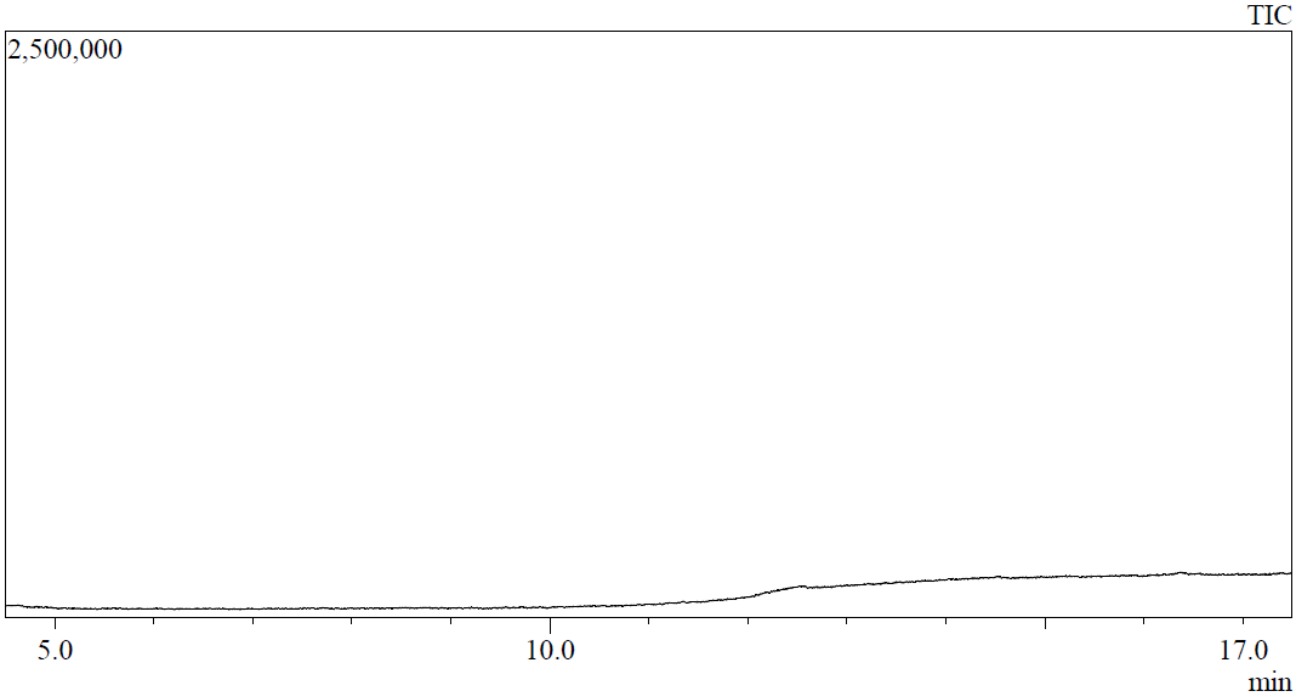
9. Results of nicotine content in liquid.

Sample signature	Subject of determination	Method identification*	The result of the analysis	Uncertainty	Unit
2020/12/0005/001	Average content of **nicotine**	SL/2019/044 Ed. 2 of 14.08.2020, A	< 2,0	0,2	mg/ml

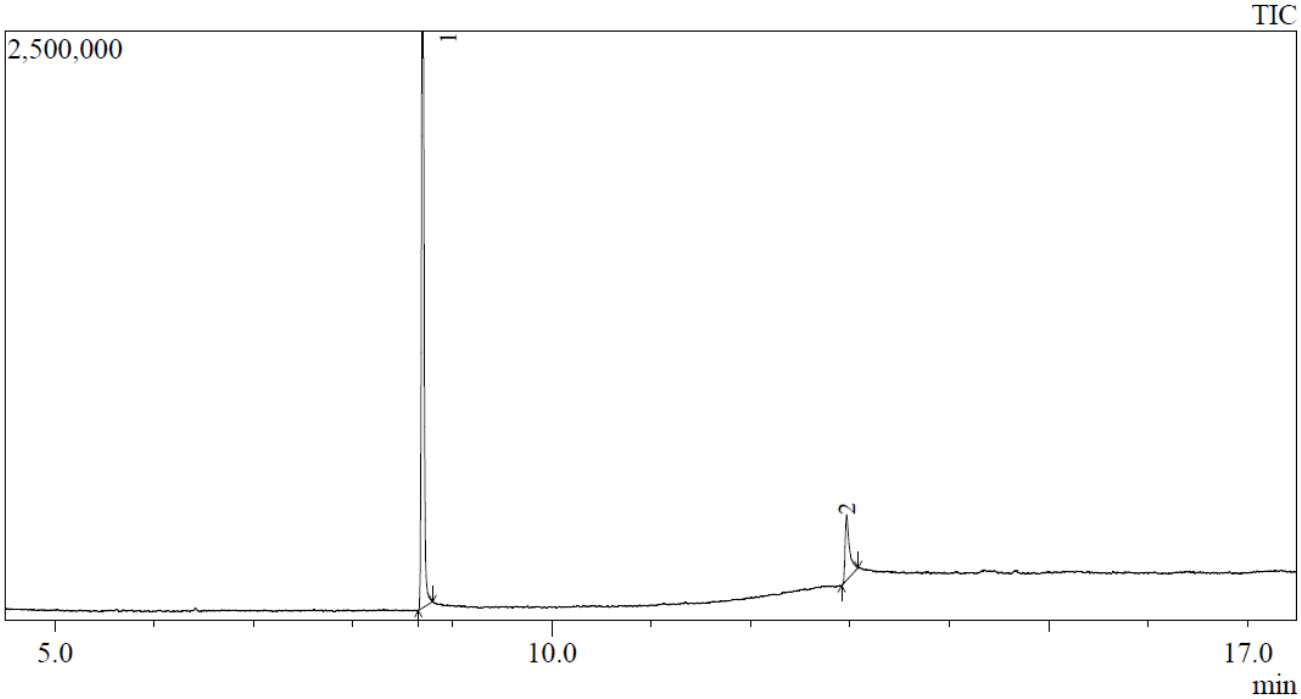
Determination method: A-accredited, NA- non-accredited, AS-accredited by the subcontractor, NAS- not accredited by the subcontractor.
** amount of nicotine [mg] per 1 g of vaped liquid
LOQ – limit of quantification

ANALYSIS REPORT

10. Chromatograms.



Pic. 1. Chromatogram of sample blank after full procedure.



2020_12_005_001

ID#	Name	R.Time
1	Propylene Glycol	8.701
2	Glycerin	12.966

Pic. 2. Chromatogram of sample 2020/12/0005/001 and compound table from NIST11 and NIST11s library.

ANALYSIS REPORT

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. Limits of quantification of heavy metals

Subject of designation	Unit	Limit of quantification
Content of lead **Pb**	µg/g	5,00
Content of cadmium **Cd**	µg/g	5,00
Content of arsenic **As**	µg/g	5,00
Content of chrome **Cr**	µg/g	5,00
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
Content of **toluene**	µg/g	50
Content of **isoprene**	µg/g	50
Content of **1,3-butadiene**	µg/g	50
Content of **ethylene glycol**	µg/g	250
Content of **diethylene glycol**	µg/g	50

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.

ANALYSIS REPORT

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

Subject of designation	Unit	Limit of quantification
Content of **formaldehyde**	µg/g	2,76
Content of **acetaldehyde**	µg/g	3,79
Content of **acrolein**	µg/g	4,58
Content of **crotonaldehyde**	µg/g	5,41
Content of **diacetyl	µg/g	50
Content of **acetyl propionyl	µg/g	25

**The aerosol was collected to acetonitrile 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.

VII. Method of qualitative analysis of aerosol generated from the e-liquid (SCAN mode):

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

Approved of the results and report	Mariej Barc	Dokument podpisany przez Barc; Laboratorium Analiz Chemicznych Spark-Lab Sp. z o.o. Data: 2020.12.23 12:07:29 CET	Julian Guzik	Dokument podpisany przez Guzik; LABORATORIUM ANALIZ CHEMICZNYCH SPARK-LAB SP. Z O.O. Data: 2020.12.23 12:04:13 CET

END OF REPORT

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