



# Collaborative Study for Determination of Glycerin, Propylene Glycol, Water and Nicotine in Collected Aerosol of E-Cigarettes

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## Abstract

In 2015, the CORESTA E-Vapour Sub-Group (EVAP) conducted a collaborative study on the aerosol from electronic, prefilled “cigalike” e-cigarettes. The goal of this study was to determine the repeatability (r) and reproducibility (R) of proposed CORESTA Recommended Methods (CRMs) for the determination of nicotine, propylene glycol, glycerin, and water in aerosol. Aerosol was generated applying the standardized puffing regime given in CORESTA Recommended Method No. 81.

Eighteen laboratories participated in this international study. Each laboratory tested the same set of four e-cigarette products and one liquid control sample. The aerosol yield was highly variable with r values of 29 to 35% and R values of 33% to 57%. However, when the results were normalized for yield, the r and R values were 8% and 10 to 22 % for nicotine, propylene glycol, and glycerin, respectively. Even after normalizing for aerosol yield, however, water gave r and R values of 22 to 29% and 62 to 81%. A control sample was used to test the method independent of the e-cigarette products. The r values for this sample were approximately 5% for nicotine, propylene glycol, and glycerin, and the r value for water was 14%. The R values for the control sample were 15%, 11%, 21%, and 30% for nicotine, propylene glycol, glycerin, and water, respectively. Based on these results, the draft CORESTA Recommended Methods used in this study have been shown to be fit for the intended purpose for the determination of these constituents in aerosol generated from e-cigarettes.

## Test Method-CRM-84

### Collection

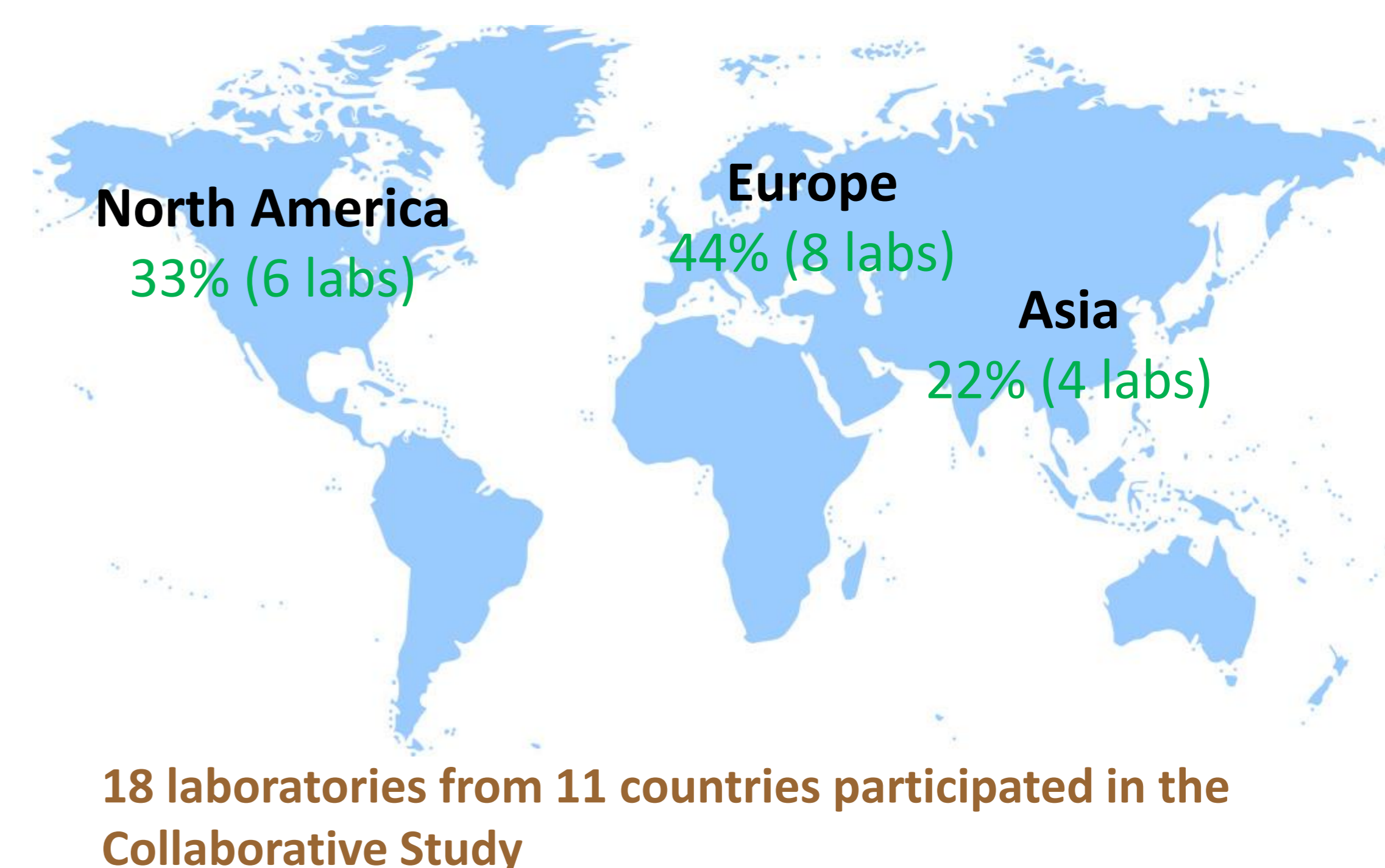
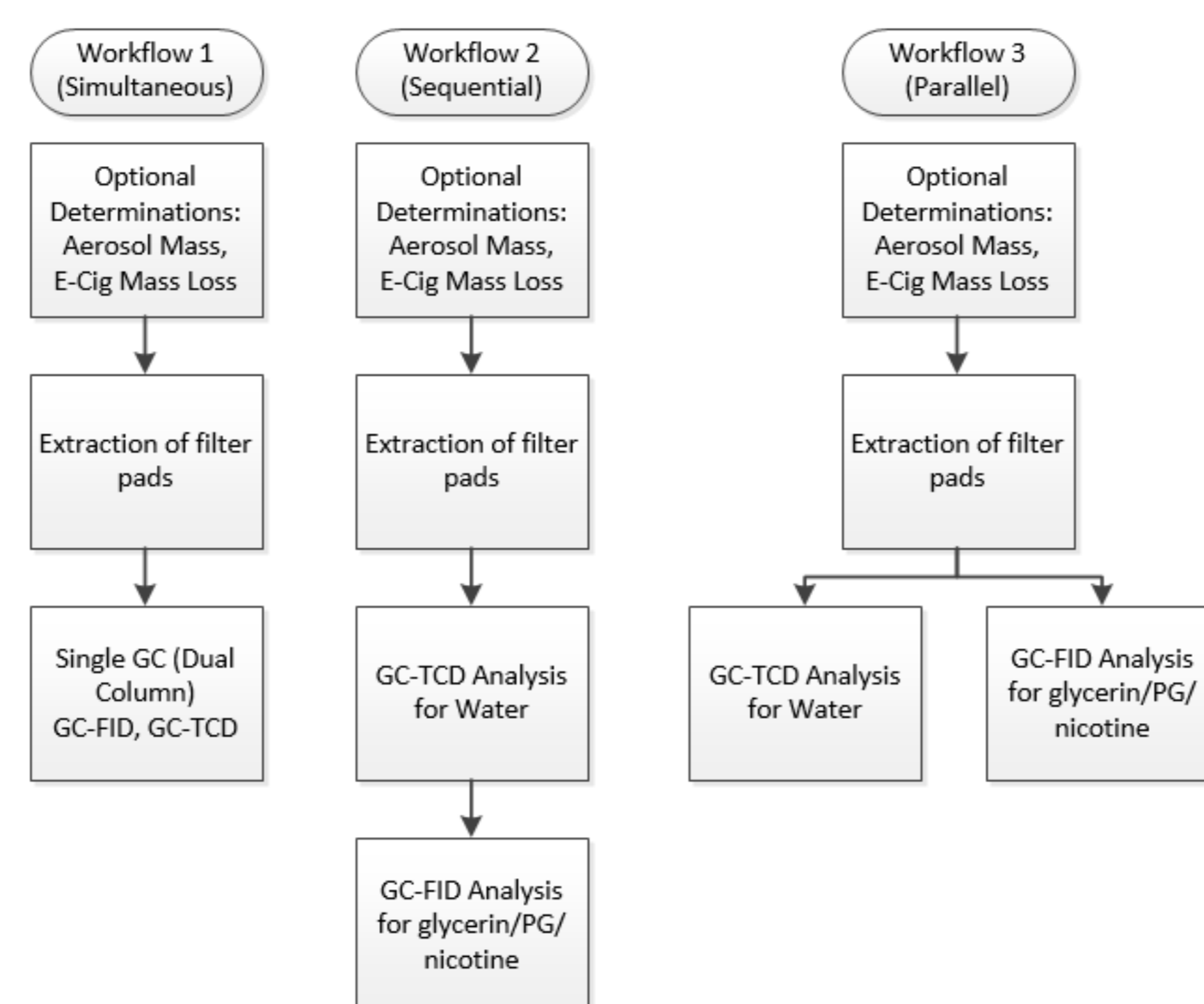
- Aerosol collection by CORESTA CRM No 81
- 55 ml puff volume, 3 second puff duration, 1 puff every 30 seconds, square wave profile
  - Humidity controlled to  $\pm 5\%$
  - Temperature controlled to  $\pm 2^\circ\text{C}$

### Sample Preparation

- Pad extraction with 20 ml, 20 ml
- Mechanical shaking

### Analysis

- GC-TCD for water
- GC-FID for other analytes
- Column: DB-ALC1 30 m x 0,32 mm x 1,8  $\mu\text{m}$

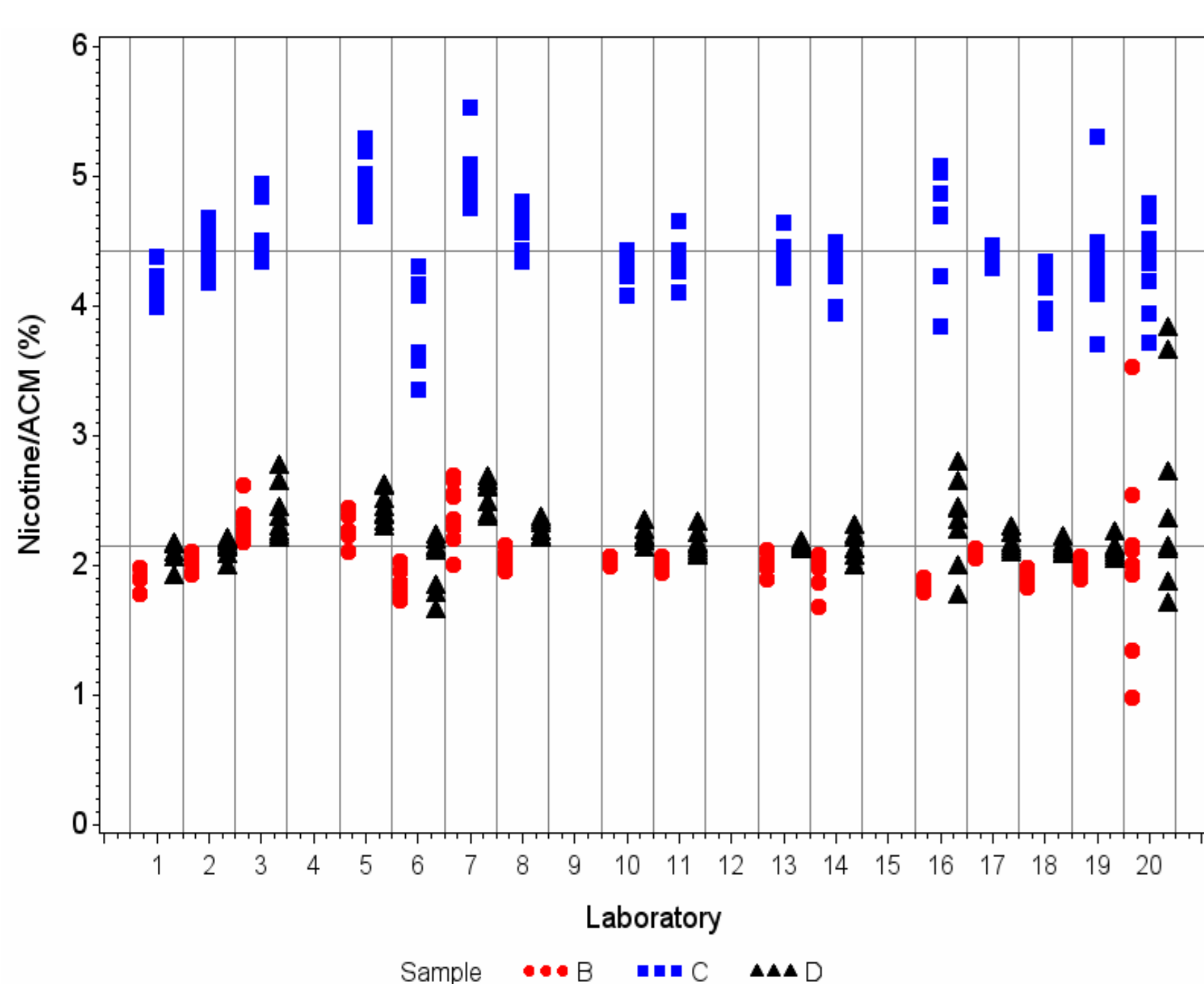


## RESULTS

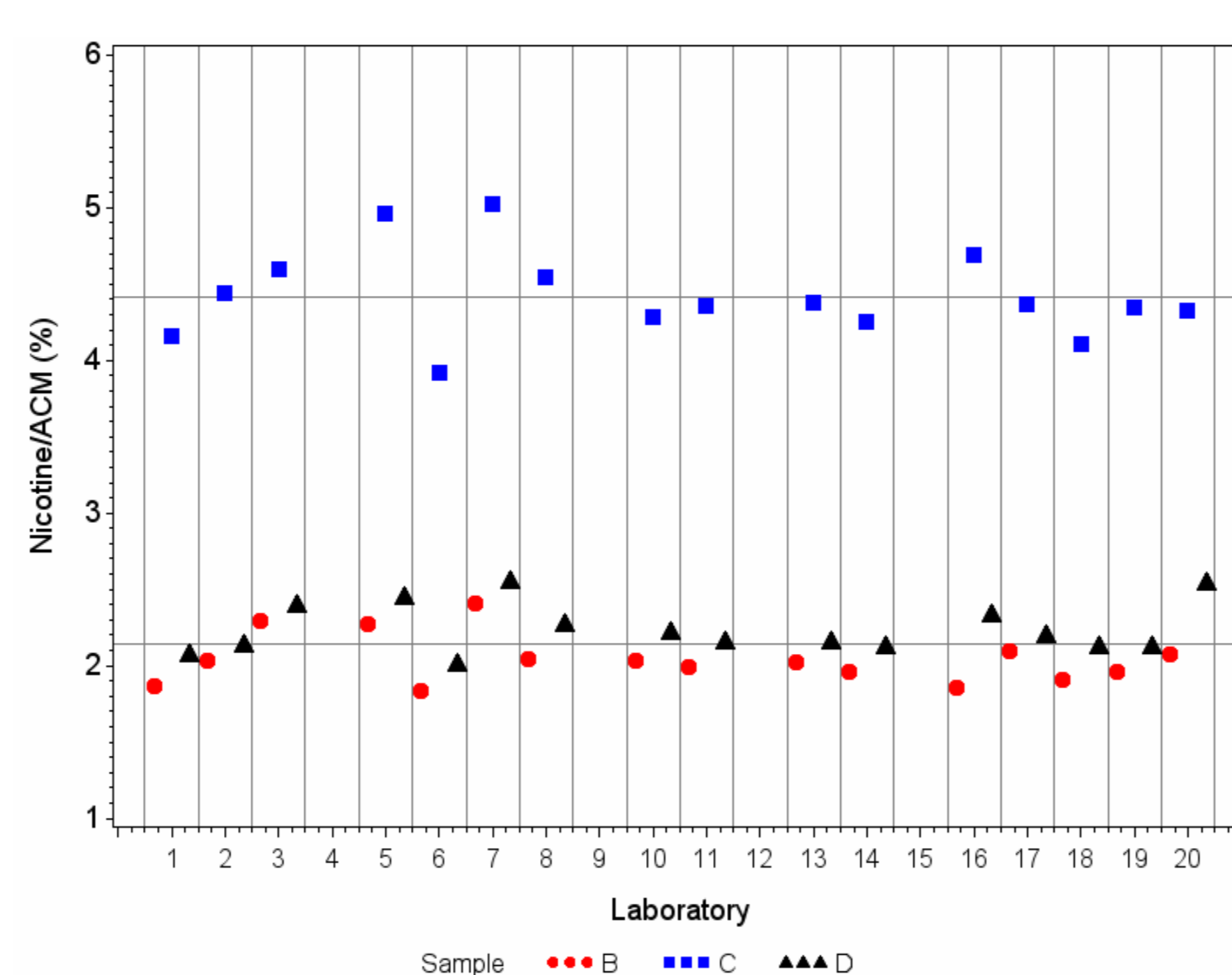
### r&R values for analytes proportioned to aerosol mass collected (ACM)

Sample Code		ACM	Nicotine/ACM	PG/ACM	Glycerin/ACM	Water/ACM
Sample A	r (%)	28.6%	--	5.30%	6.10%	22.0%
	R (%)	46.6%	--	9.80%	17.8%	64.7%
Sample B	r (%)	33.5%	5.20%	5.10%	6.10%	29.0%
	R (%)	56.7%	22.2%	9.60%	13.8%	81.5%
Sample C	r (%)	39.4%	6.50%	6.50%	8.20%	23.9%
	R (%)	47.2%	17.1%	10.2%	10.5%	62.1%
Sample D	r (%)	33.9%	6.20%	5.30%	--	21.0%
	R (%)	40.5%	22.1%	10.0%	--	53.0%

Nicotine/ACM individual results for all samples



Nicotine/ACM mean results for all samples



### r&R values for Sample F, the liquid control sample

Sample Code		Nicotine	PG	Glycerin	Water
Sample F	r (%)	4.37%	4.45%	6.07%	13.6%
	R (%)	14.8%	10.9%	20.7%	30.5%

Repeatability (r) and reproducibility (R) calculations for the aerosol results were based on a test result being the average of four test items. Calculations were carried out using the robust algorithms in ISO 5725-5; -- = Not present. For the liquid samples a test result was a single determination.

## TEST MATERIALS

Four commercial e-cigarette products (samples A-D) matched for e-liquid lot number but with varied content of major constituents were used to conduct the study. The analytes and nominal value ranges are listed below:

- Nicotine: 0 – 5.4 %
- Glycerin: Propylene Glycol (PG) 30:70 or 0:100
- Water: Content not specified

An e-liquid study control (sample F) was also included. This control was a mixture of glycerin (43 %), propylene glycol (43 %), water (12 %), and nicotine (2 %) and was prepared by one laboratory using analytical grade chemicals.

## DATA ANALYSIS

- Data were statistically analysed in conformance with ISO 5725(5) and ISO 13528. ISO 5725(5) was used instead of 5725(2) because it uses an approach less strongly affected by outliers and does not attempt to delete outliers prior to analysis.
- Each analyte was examined as is (data not shown) and as a proportion of the percentage of ACM (analyte/ACM). The r&R values are shown for the normalized yield.

## CONCLUSIONS

- The analytical method given in CRM No 84 is suitable for the determination of nicotine, PG, glycerin, and water in the aerosol generated from e-cigarettes.
- The e-cigarettes used in this study were found to vary in aerosol mass production, by device. Proportioning analyte yield to the aerosol mass collected improved method repeatability (r) and reproducibility (R).
- The e-liquid control samples distributed with the study samples demonstrated that the analytical method is fit for purpose and gives accurate and reliable analytical results.

## ACKNOWLEDGMENTS

CORESTA E-Vapour Products Subgroup (EVAP) and all laboratories participating in the study.

**References:** Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA), 2015 Collaborative Study for Determination of Glycerin, Propylene Glycol, Water and Nicotine in Collected Aerosol of E-Cigarettes, March 2017, [www.coresta.org/sites/default/files/technical\\_documents/main/EVAP-086-1-CTR\\_2015CollStudy-Glycerin-PG-H2O-Nic-CigAerosol\\_March2017.pdf](http://www.coresta.org/sites/default/files/technical_documents/main/EVAP-086-1-CTR_2015CollStudy-Glycerin-PG-H2O-Nic-CigAerosol_March2017.pdf) 6.pdf

Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA), No. 84 - Determination of Glycerin, Propylene Glycol, Water, and Nicotine in the Aerosol of E-Cigarettes by Gas Chromatographic Analysis, March 2017, [www.coresta.org/sites/default/files/technical\\_documents/main/CRM\\_84.pdf](http://www.coresta.org/sites/default/files/technical_documents/main/CRM_84.pdf)