



# **The value of collaboration in the development of e-cigarette test methods**

**Dr Derek Mariner**

**CORESTA E-Vapour Sub-Group**

**ENDS 2017**

**St James' Court Hotel, London**

**14-15 June, 2017**



- ❖ **Introduction to CORESTA**
- ❖ **CORESTA activities**
- ❖ **Process for Development of Recommended Methods**
- ❖ **CORESTA E-Vapour Subgroup**
- ❖ **Collaborative study for CRM 84: VG, PG, water and nicotine in aerosol**
- ❖ **Standardisation and engagement**
- ❖ **Summary**



# CORESTA

Centre de  
**CO**opération pour les **RE**cherches **SC**ientifiques  
Relatives au **TA**bac

❖ Cooperation Centre for Scientific Research Relative to Tobacco



**To be recognised by our members  
and relevant external bodies  
as an authoritative source  
of publically available credible  
science and best practices  
related to tobacco and its derived products.**



## The Purpose of CORESTA

**Encourage international cooperation**  
**to actively work**  
**on tobacco-related areas of research**



## ❖ It is an Association:

- Founded in 1956 by 24 organisations from 20 countries
- Headquartered in Paris and governed under French law
- Now 150 Member organisations from 38 countries

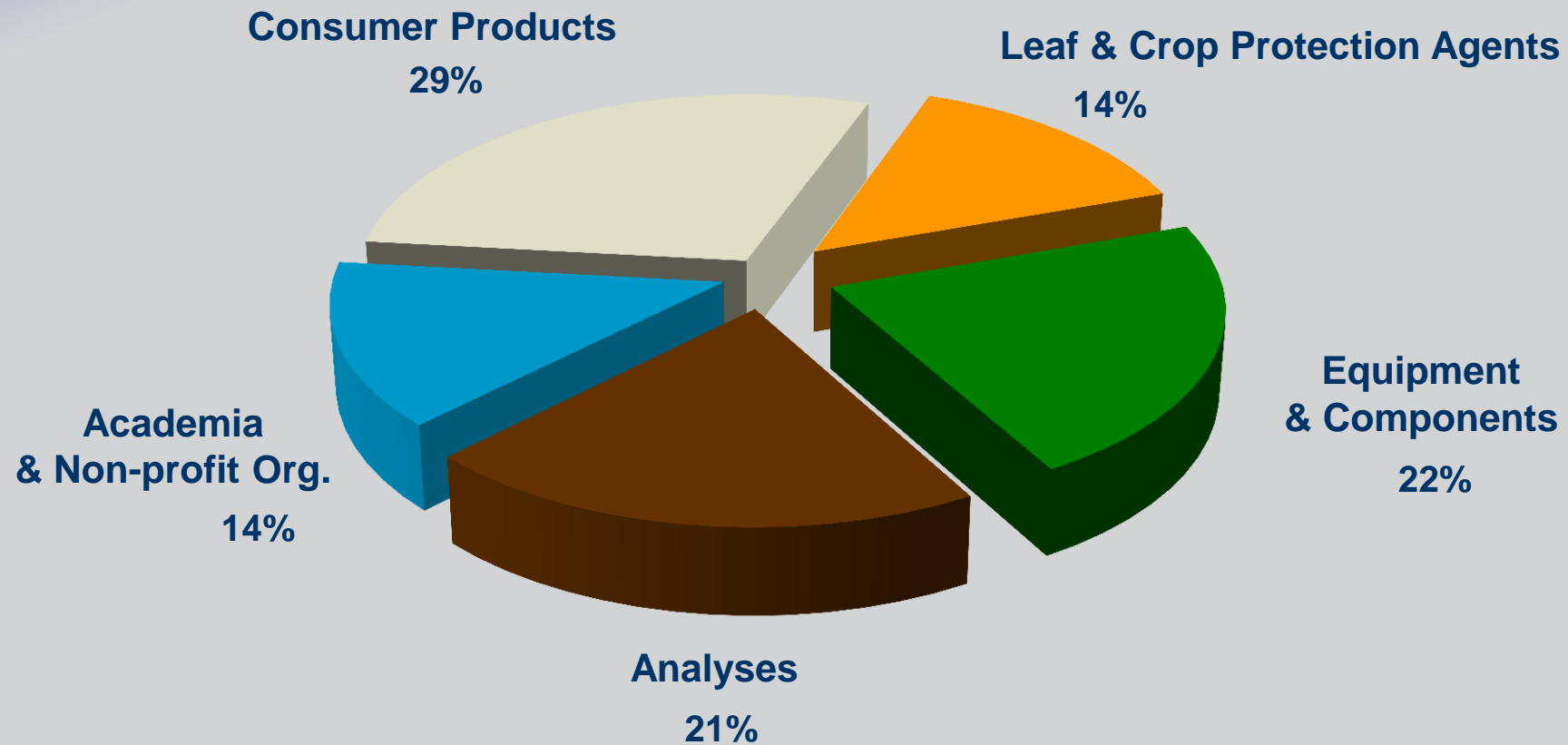
## ❖ Main bodies

- Board (12 to 14 organisations)
- Scientific Commission (20 individuals)
- General Secretariat (3 persons)
- 22 Sub-Groups and Task Forces within 4 Study Groups  
+ 3 inter-group committees

**≈ 600 persons worldwide involved in on-going work**



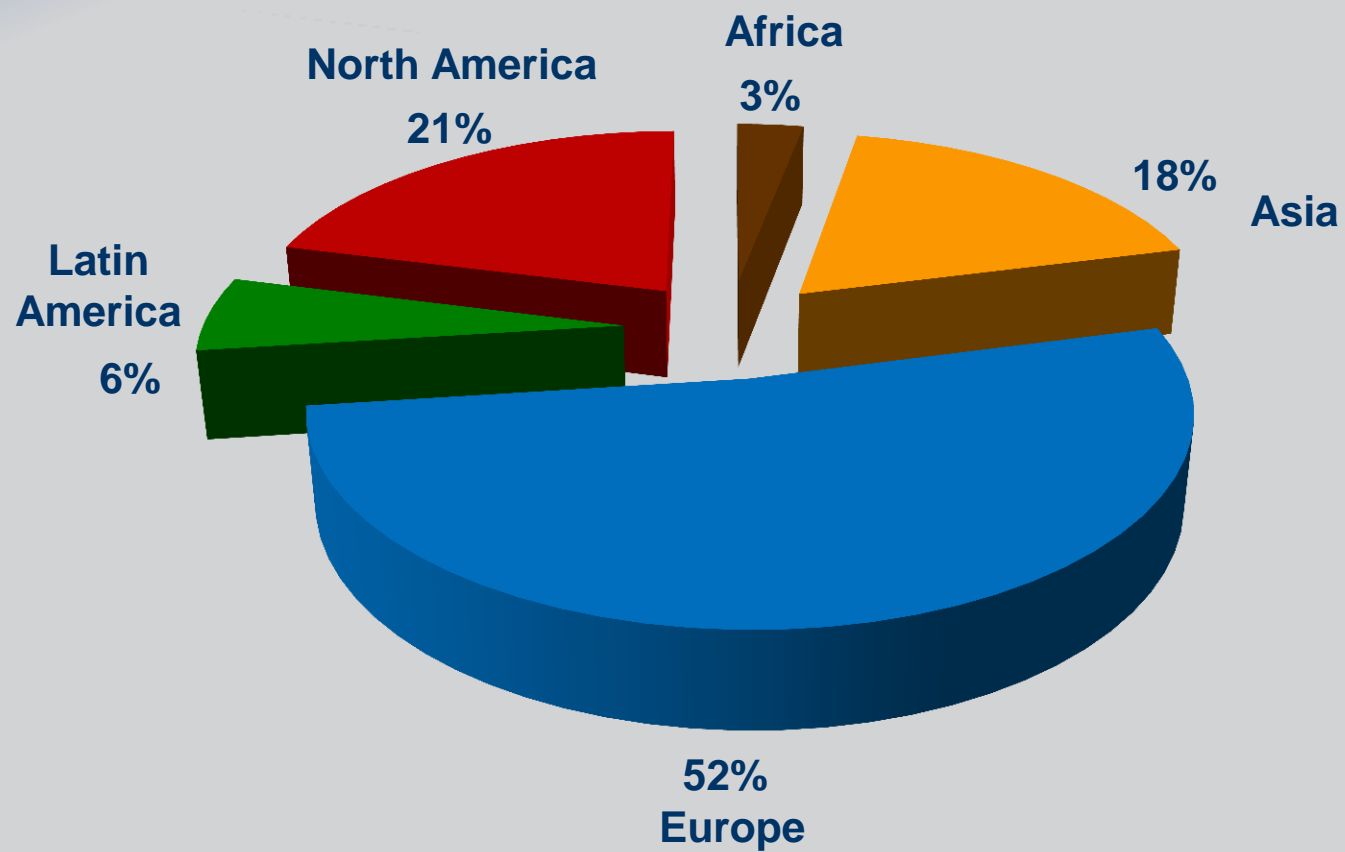
# Membership Core Activity





# Membership

## Worldwide distribution







# Activities across supply chain, product testing and product use

## ❖ Agronomy & Leaf Integrity, Phytopathology & Genetics

- Agronomy & Breeding
- Curing
- Sustainability
- Pests & plant diseases
- Agrochemical issues

Agro - Phyto

« AP »

## ❖ Smoke Science, Product Technology

- Technical specifications
- **Methods for component and emissions analysis**
- Consumer behaviour
- *In Vitro* Toxicology

Smoke - Techno

« SSPT »



## ❖ Product Technology

- SG Tobacco and Tobacco Product Analytes
- SG Physical Test Methods
- TF Cigarette Variability
- SG Routine Analytical Chemistry
- SG Cigar Smoking Methods
- **SG E-Vapour**

## ❖ Smoke Science

- SG Smoke Analytes
- SG Product Use and Behaviour
- SG Biomarkers
- SG *In Vitro* Toxicity Testing



# Smoke - Techno Achievements

## ❖ Smoke Science & Product Technology

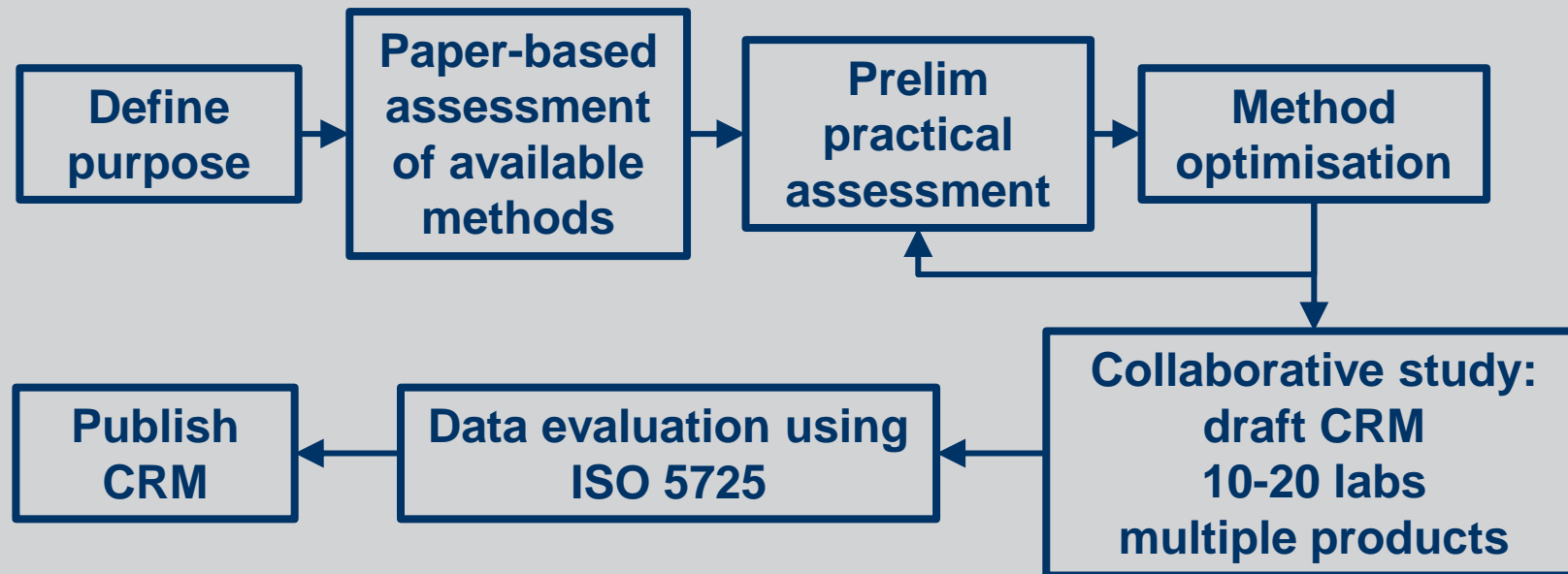
- **Developed Recommended Methods\*, Guides and Reports on tobacco, product and smoke analysis (chemical and physical)**
  - 37 ISO standards based on CORESTA Recommended Methods (CRMs)
  - + 6 CRMs currently in the process of becoming ISO standards
  - Regular collaborative studies/proficiency trials to support member labs' accreditation (agrochemicals, TNCO, physical...)
  - Protocols for *in vitro* toxicity testing of mainstream smoke
  
- **Developed Reference Materials**
  - CM 'CORESTA Monitor test piece' for smoking machine set-up + 1 for LIP testing
  - 4 smokeless tobacco products



***\*62 out of 85 CRMs are currently active, due to obsolescence/replacement of older ones***

## ❖ Approach used for the development of robust methods

### ➤ Consensus-based process



- Discussions during process provide insight into causes/reduction of intra- and inter-laboratory variability
- Methods and Reports are made available on the CORESTA website
- More recently, experience shared in peer-reviewed publications



- ❖ Formed in 2013 (originally a Task Force)
- ❖ Currently 45 member organisations: e-cig and e-liquid manufacturers, academia, regulators, equipment suppliers, and testing laboratories
- ❖ Publications on CORESTA website:
  - E-Cigarettes: A Brief Description of History, Operation and Regulation. Reference Report - February 2014
  - E-Cigarettes: Assessment of Analytical Literature from 55 Studies Published Worldwide prior to November 2013 on Commercial E-Cigarettes. Reference Report - May 2014
  - E-Liquid Preliminary Proficiency Study. Technical Report - March 2015
  - 2014 Electronic Cigarette Aerosol Parameters Study. Technical Report - March 2015
  - CRM 81: Routine analytical machine for e-cigarette aerosol generation and collection - definitions and standard conditions – June 2015
  - Guide No. 18: Sample Handling and Sample Collection of E-Cigarettes and E-Vapour Generating Products – November 2016
  - 2015 Collaborative Study for Determination of Glycerin, Propylene Glycol, Water and Nicotine in Collected Aerosol of E-Cigarettes. Technical Report - March 2017
  - CRM 84: Determination of glycerin, propylene glycol, water, and nicotine in the aerosol of e-cigarettes by gas chromatographic analysis – March 2017
- ❖ Current: CRM for Carbonyls in aerosol; Next: Metals in aerosol



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- ❖ Publications
  - **Basis for ISO/DIS 20768: “Vapour products - Routine analytical vaping machine – Definitions and standard conditions”**
  - Operation and Regulation. Reference Report - February 2014
  - Review of the Literature from 55 Studies Published Worldwide prior to November 2013 on Commercially Available E-Cigarettes. Reference Report - March 2015
  - 2014 Electronic Cigarette Aerosol Parameters Study. Technical Report - March 2015
  - **CRM 81: Routine analytical machine for e-cigarette aerosol generation and collection - definitions and standard conditions – June 2015**
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**Development has informed ISO 20714/DIS: “E-liquid — Determination of nicotine, propylene glycol and glycerol in liquids used in electronic nicotine delivery devices — Gas chromatographic method”**



# Example: CRM84 Glycerin, Propylene Glycol, Water, and Nicotine in E-Cig Aerosol by GC Analysis

## ❖ Collaborative Study (2015, report on website)

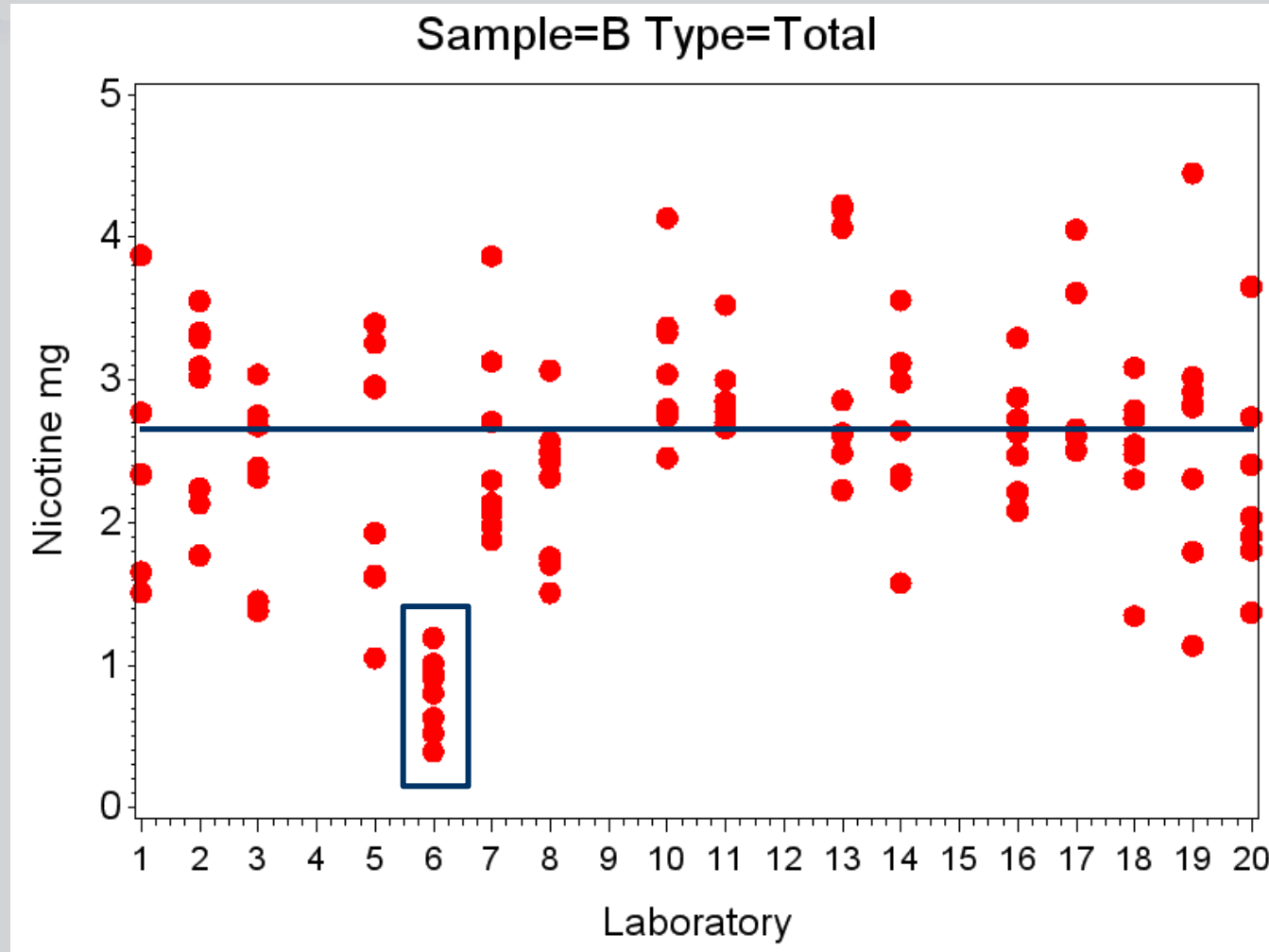
- 18 participating labs
- 4 test items (device+liquid) obtained directly from the manufacturers
- Control liquid sample
- 5-8 replicates per product per lab
- Aerosol generated in 3 puff-blocks (1-10, 11-30, 31-80) per device using CRM 81: 55ml puff volume, 3s duration, 30s interval, square profile
- Parameters: Glycerin, Propylene glycol, Nicotine, Water, Aerosol mass (ACM)
  
- Data were analyzed in basic conformance with ISO 5725(5) (and ISO 13528 for information)
- Each analyte was examined individually plus the analyte as a proportion or percentage of ACM.





# Example: CRM84 Glycerin, Propylene Glycol, Water, and Nicotine in E-Cig Aerosol by GC Analysis

Nicotine =  
Total from  
puffs 1-80





# Example: CRM84 Glycerin, Propylene Glycol, Water, and Nicotine in E-Cig Aerosol by GC Analysis

Summary Results for Nicotine			
	B	C	D
Lab	Total	Total	Total
Mean	2.59	4.86	3.20
Indiv	0.667	1.408	0.786
sr	0.334	0.704	0.393
sR	0.457	0.869	0.450
<b>r (%)</b>	<b>36.0%</b>	<b>40.5%</b>	<b>34.4%</b>
<b>R (%)</b>	<b>49.4%</b>	<b>50.0%</b>	<b>39.4%</b>

**Within lab variability**

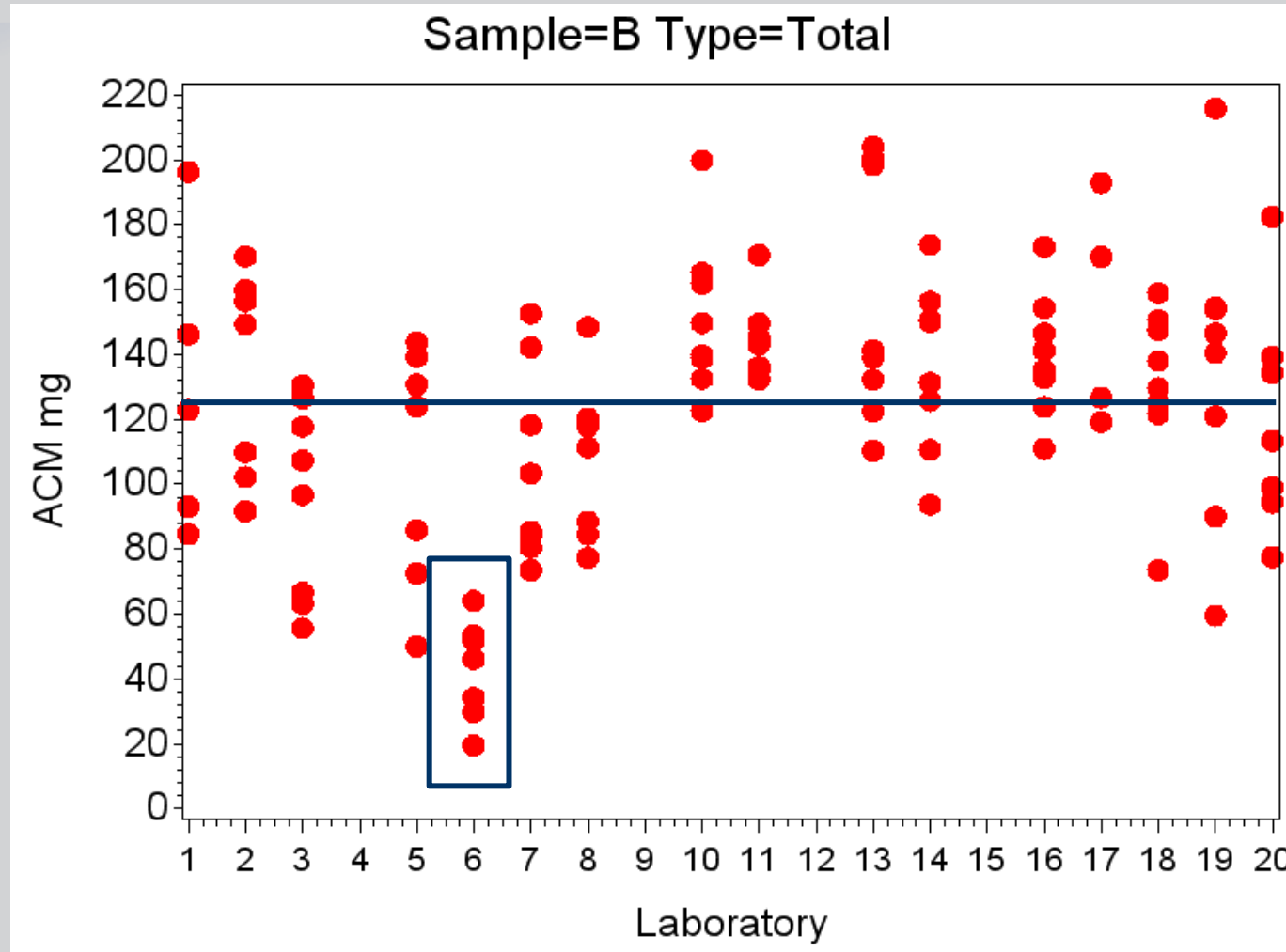
**Total variability**

- Repeatability and reproducibility calculations based on a test result being the average of four test items. Indiv is the sd among individual values (i.e., not the average of 4).
- Calculations carried out using the robust algorithms in ISO 5725-5.



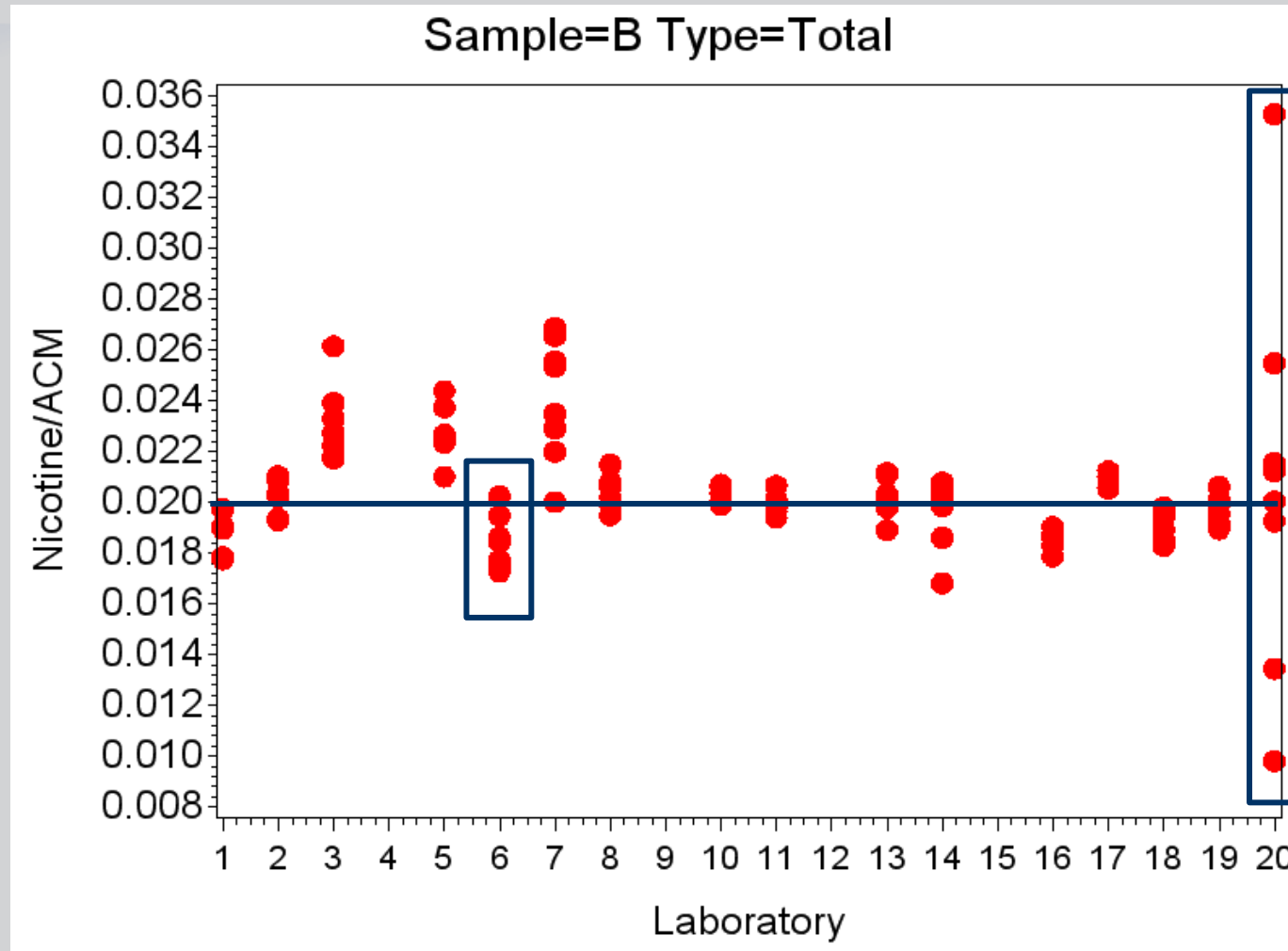
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ACM =  
Aerosol  
collected  
mass





# Example: CRM84 Glycerin, Propylene Glycol, Water, and Nicotine in E-Cig Aerosol by GC Analysis





# Example: CRM84 Glycerin, Propylene Glycol, Water, and Nicotine in E-Cig Aerosol by GC Analysis

Nicotine/ACM			
	B	C	D
	Total	Total	Total
Mean	2.03%	4.40%	2.25%
Indiv	0.08%	0.20%	0.10%
sr	0.04%	0.10%	0.05%
sR	0.16%	0.27%	0.18%
<b>r (%)</b>	<b>5.2%</b>	<b>6.5%</b>	<b>6.2%</b>
<b>R (%)</b>	<b>22.2%</b>	<b>17.1%</b>	<b>22.1%</b>

**Within lab variability**

**Total variability**

- Mean, Indiv, sr, and sR are expressed as percent of ACM. r (%) and R(%) are percent of the estimated mean.
- Repeatability and reproducibility calculations based on a test result being the average of four test items. Indiv is the sd among individual values (i.e., not the average of 4).
- Calculations carried out using the robust algorithms in ISO 5725-5.



# CORESTA and e-cig standards

## ❖ AFNOR (France)

- CORESTA participated in the ECIG Commission
- AFNOR standards
  - inclusion of the CORESTA-recommended vaping regime
  - emphasis on analytical considerations to assess and reduce variability

## ❖ CEN (Europe)

- CORESTA Liaison with TC 437 Vape and Vapour Products
- CORESTA-nominated experts in WG1, WG3 and WG4

## ❖ ISO (International)

- CORESTA Liaison with TC 126 Tobacco and Tobacco Products and SC3 Vape and Vapour Products
- SC3-WG2 on Vaping machine parameters led by CORESTA



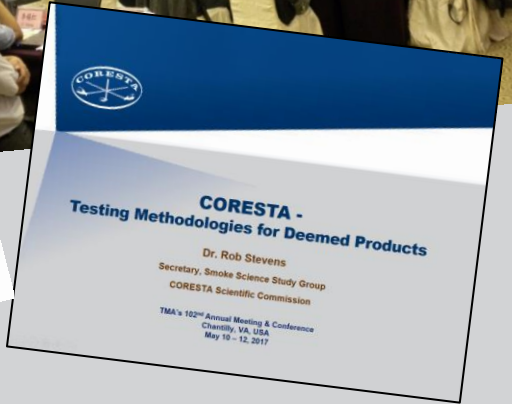
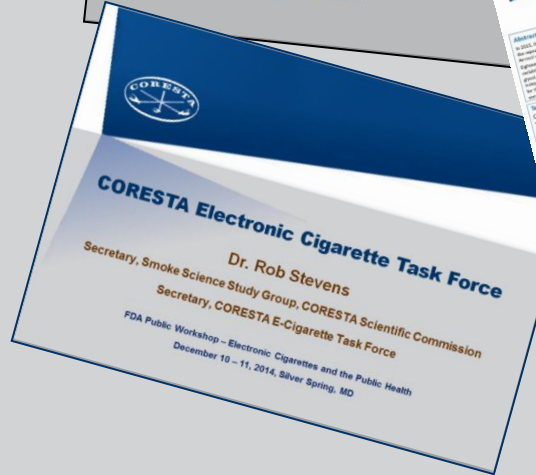
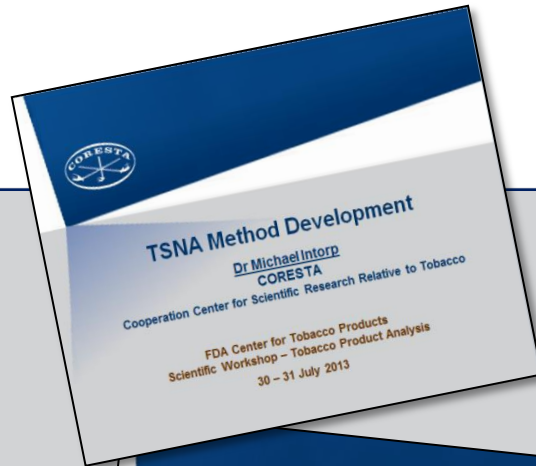
# Stakeholder Engagement

## ❖ FDA Workshops

## ❖ Agrochemical Seminars

## ❖ Conferences

- Global Tobacco & Nicotine Forum
- Tobacco Campus
- E-cig Europe
- US Tobacco Merchants Assoc.
- Global Forum on Nicotine
- ...





## **Value of collaboration: CORESTA**

- ❖ **Global interdisciplinary expertise from different sectors – non-members can get involved**
- ❖ **Focus on sharing and advancing scientific knowledge**
- ❖ **Conduct of inter-laboratory studies during development of analytical methods**
- ❖ **Track record supporting development of International Standards**
- ❖ **Emphasis on collaboration**





❖ Thank you for your attention

❖ Questions?

❖ More information available at [www.coresta.org](http://www.coresta.org)

The screenshot shows the CORESTA website homepage. At the top left is the CORESTA logo. To its right is the text "Cooperation Centre for Scientific Research Relative to Tobacco" and "Centre de Coopération pour les Recherches Scientifiques Relatives au Tabac". In the top right corner, there are links for "Join CORESTA" and "Member Access", and a search bar. Below the header is a navigation menu with items: "ABOUT US", "STUDY GROUPS", "DOCUMENTS" (which is highlighted and has a dropdown menu open), "ABSTRACTS", "MEETINGS", and "INFORMATION". The dropdown menu for "DOCUMENTS" contains: "Recommended Methods", "Reports", "Guides", and "All Documents". Below the navigation is a main banner for "Smoke-Techno 2017" with the dates "8-12 October 2017 -- Kitzbühel, Austria" and a "REGISTRATION open" button. To the right of the banner is a "Vision" section with the text: "To be recognised by our members and relevant external bodies as an authoritative source of publically available, credible science and best practices related to tobacco and its derived products." and a "More about CORESTA" button. Below the banner are three columns: "News", "Latest Documents", and "Upcoming Meetings".

**News**

- SSPT2017: online registration open  
01/06/2017
- Project 152 launched: PTM SG - 5th Round Robin Test on Air Permeability Calibration Standards  
31/05/2017
- SSPT2017 and AP2017: abstract submissions closed on 26 May 2017  
29/05/2017
- CORESTA Water Pipes Survey  
11/05/2017

**Latest Documents**

- Reports**  
**3rd Proficiency Test (2017) on Diffusion Capacity of Cigarette Papers**  
PTM Technical Report  
18/05/2017
- Reports**  
**4th Round Robin Test for Multi-Capillary Ventilation Calibration Standards (2015/2016)**  
PTM Technical Report  
30/04/2017
- Reports**

**Upcoming Meetings**

- 7 - 9 June 2017**  
Scientific Commission  
Yokohama, Japan
- 20 - 21 June 2017**  
Board  
Bristol, UK
- 17 - 20 September 2017**  
71st Tobacco Science Research Conference (TSRC)  
Bonita Springs, FL, USA