



Development of Machine Smoking History and Recent Learning

Tobacco Product Analysis - A Scientific Workshop - July 2013

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- ❖ **CORESTA is an international organization with 138 members that promotes collaborative pre-competitive scientific research into all aspects of tobacco**
- ❖ **It is the only international organization that represents both the product and the leaf growing communities with members from:**
 - **the main tobacco product manufacturers;**
 - **the scientific equipment manufactures,**
 - **independent laboratories (including some regulatory laboratories);**
 - **third party suppliers to the industry including leaf growing and leaf suppliers**
- ❖ **Scientific activities within CORESTA are conducted through Sub-Groups and Task Forces, each devoted to a specialist topic**
- ❖ **The research on TNCO and smoking machine methodology is conducted by the Routine Analytical Chemistry Sub Group (RAC)**



Agenda

- ❖ **Smoke Formation Processes**
- ❖ **Factors Relevant to the Development of Smoking-Machine Methodology**
- ❖ **Development of Standards**
 - **Federal Trade Commission in the USA**
 - **CORESTA and ISO**
 - **Harmonization of Smoking-Machine Methods**
- ❖ **Recent Learning on Factors Relevant to Smoking-Machine Methodology**



Mainstream Cigarette Smoke

- ❖ **Smoke is composed of semi-liquid droplets suspended in a gas**
- ❖ **Particles may contain organic and inorganic material**
- ❖ **Particles will generally contain material which has condensed from the hot gas stream, and may contain high molecular weight material which is the result of explosive cell rupture and mechanical suspension**
- ❖ **High concentration and small size lead to rapid coagulation**
- ❖ **Particle composition may initially be non-homogeneous, but coagulation acts to homogenize the sample.**
- ❖ **Aerosol particles are lightly charged, with overall electrical neutrality**
- ❖ **At least 4700 constituents of cigarette mainstream smoke have been identified (Green and Rodgman, 1996) with the vast majority observed at very small concentrations**



Factors Relevant to the Development of Smoking-Machine Methodology



Smoking-Machine Methodology

- ❖ **There are three essential elements and requirements for a smoking-machine method for such work:**
 - **It should enable the cigarette to be smoked mechanically under specified condition in a reproducible and controlled manner**
 - **The smoke should be collected quantitatively and consistently**
 - **The smoke should be analyzed in a reproducible manner**

- ❖ **Two types of constant volume machines have dominated smoking methodology for many years: linear smoking machines and rotary smoking machines**

- ❖ **Because of the complex nature of tobacco smoke, the design of a perfect collection system is challenging**
 - **Different methods (Electrostatic trapping, Physical trapping, sorption trapping) can give slightly different component values**



Development of Standards



Development of Standards

- ❖ **‘Standards’ are documented agreements containing technical specifications or concise criteria to be used consistently as rules or guidelines**
- ❖ **A standard method was required to produce comparable results although it was recognized that this did not replicate human smoking**
- ❖ **In 1969 CORESTA published a recommended international method for machine-smoking of cigarettes**
- ❖ **In 1970 ISO/TC126 set up a Working Group of experts to consider an ISO standard for machine-smoking cigarettes,**
- ❖ **The resultant standards were published in 1977 and were essentially identical to the CORESTA Recommended Methods**



Smoking-Machine Standards Harmonisation

- ❖ **By the late 1980s various standard methods were in use around the world for determining yields but details in butt length, capture method and air flows within smoking machine varied**
- ❖ **Based on the types of differences outlined above, ISO decided that there was a need to further “harmonize” the smoking methods used into a single standard and CORESTA was asked to undertake the necessary experimental work to produce a revised standard**
- ❖ **A large amount of experimental work and collaborative experiments were undertaken by CORESTA between 1989 and 1992 and a revised standard was published in 1991**
- ❖ **With the introduction of the 10-1-10 ‘ceilings’ for “tar”, nicotine, and CO in the EU small differences in ISO CO yields under the standard between machines came under scrutiny**
- ❖ **A further harmonization exercise was undertaken and air straightening measures taken which improved correlation in yields**



CORESTA Efforts to improve proficiency

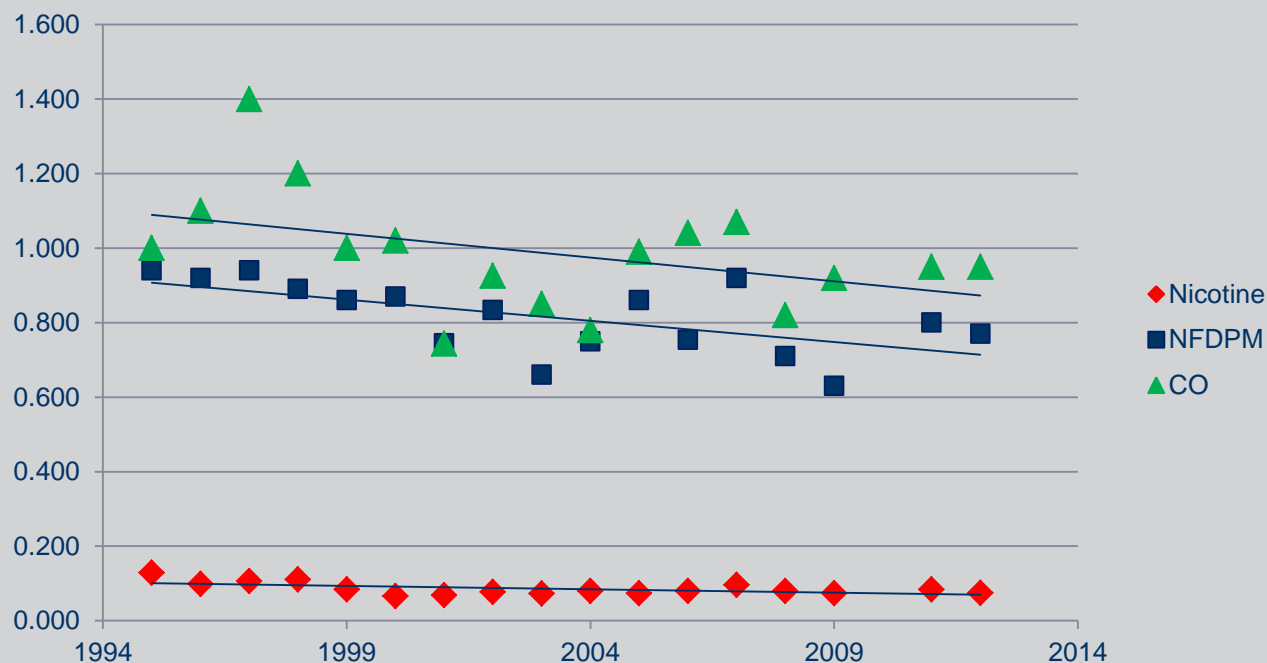
- ❖ **The CORESTA RAC group carries out regular correlation studies to both establish the suitability of standard monitors supplied to laboratories and to reinforce good practice and so improve r and R capabilities of TNCO analysis within and between labs**
- ❖ **There has been steady improvement in capability of participants since recent harmonization exercises**



CORESTA Efforts to improve proficiency

- ❖ **Steady improvements in r and R have been made by CORESTA study participants through development of standards and sharing of key operational parameters.**

Repeatability CORESTA cross checks ISO

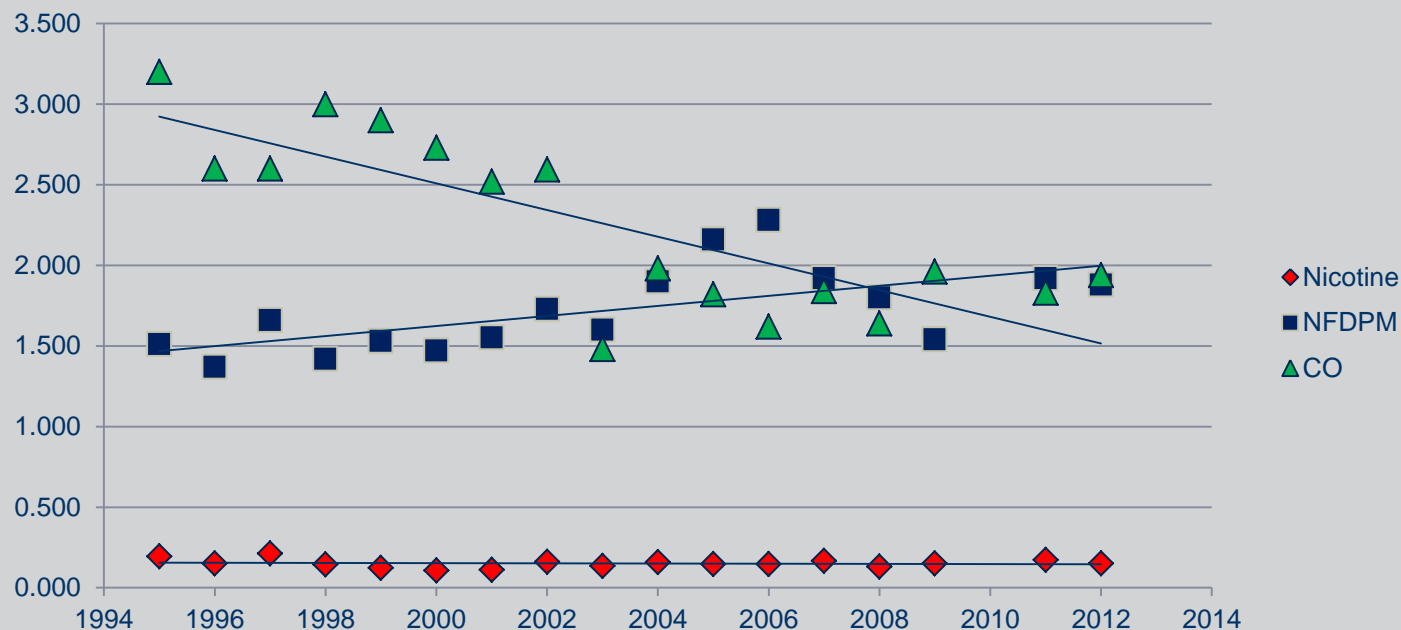




CORESTA Efforts to improve proficiency

- ❖ These “knowledge based” improvements are shared through sharing with machine providers, member labs, codifying in CORESTA recommended methods and participation of members in alternate studies

Reproducibility CORESTA cross checks ISO





Recent Activities/Developments that Impact Machine-Smoking Methodologies



Machine-Smoking Methodologies

Recent Activities/Developments

- ❖ **Intense Smoking Regimes such as the Canadian Intense Regime amplified the remaining small differences between machine types and encouraged a deeper investigation into causes.**
- ❖ **The Health Canada method (HCI) [1] references ISO3308 [4] but:**
 - **this method was developed specifically for the 35ml puff every 60 seconds method described there in**
 - **the more intense puffing regime in HCI unsurprisingly creates a different smoke aerosol than ISO, and this behaves in a different manner.**



Applying ISO methodology to Health Canada

- ❖ It became clear in 2012 that there was a problem with comparisons between machine types when smoking HCI [1]regimes.
- ❖ A number of collaborative studies showed a lower yield for rotary smoking machines when smoking HCI. There was no immediately apparent reason for this discrepancy [2].

	ISO Linear - Rotary		CI Linear - Rotary	
	Absolute mg/tp	% of mean	Absolute mg/tp	% of mean
CO	-0.56	-3.9%	-0.73	-2.7%
TPM	0.60	3.4%	5.47	12.8%
NFPDM	1.00	7.0%	2.62	9.0%
Water	-0.49	-26.0%	2.76	25.6
Nicotine	0.06	4.6%	0.12	4.4%
Puff count	0.31	3.4%	0.22	1.8%



Variability and Accuracy of TNCO Yield

- ❖ **ISO3308 [4] defines the smoking machine so this should not be a source of yield differences**
 - **ISO3308 less prescriptive than most believe**
 - **Onus is on smoking machine manufacturers to interpret ISO3308 and get consistent yields**
- ❖ **Air Flow Turbulence**
- ❖ **Lighting Temperature**
- ❖ **Systematic capture errors**



- ❖ **ISO30038 specifies the magnitude of air flow but not the direction.**
- ❖ **Measurement is made through an omnidirectional probe and no information is provided on acceptable levels of turbulence of the air.**
- ❖ **It has been shown that for some products such as those with printed wrappers that yields can differ depending on the direction of air flow.**
- ❖ **Furthermore highly turbulent air flow can increase yields of CO, air straightening kits are commonplace on rotary smoking machines to overcome the worst effects of this problem.**
- ❖ **Even modest changes in turbulence can change yields.**



Turbulence and TNCO Yield Variability

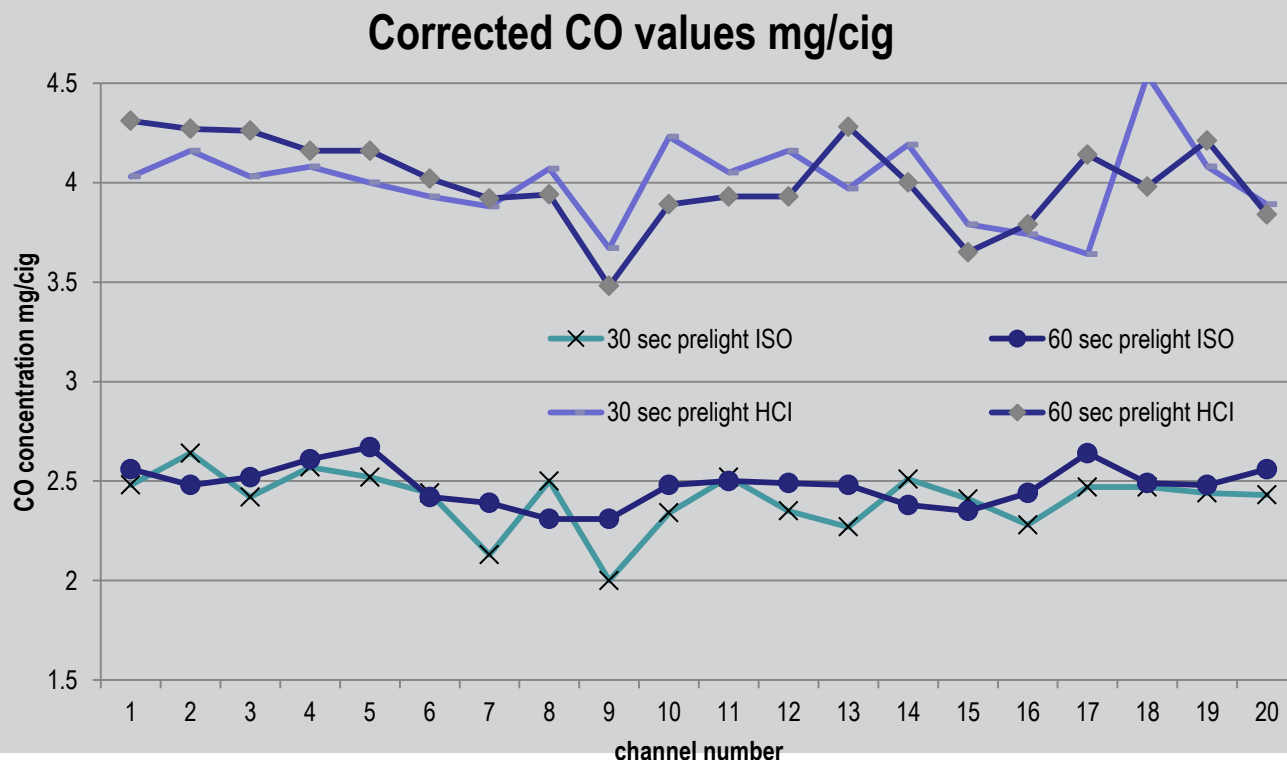
- ❖ Comparing yields for standard and more turbulent flow increased variability is observed as is a slight increase in puff count, CO yield and TPM. The differences are statistically significant for the more intense regime
- ❖ There is no restriction or guide for turbulent flow in ISO3308

		ISO			HCI		
		Standard	Increased turbulence	Difference	Standard	Increased turbulence	Difference
TPM	Mean/mg/tp	51.5	50.8	-0.7	118.3	123.1	4.9
	SD	1.5	2.9	1.4	5.6	4.4	-1.2
	T.Test	0.354			0.004		
Puff Count	Mean/tp	8.45	8.53	0.09	11.79	12.17	0.38
	SD	0.29	0.31	0.02	0.30	0.39	0.09
	T.Test	0.379			0.001		
CO	Mean/mg/tp	13.46	13.55	0.10	25.68	26.27	0.59
	SD	0.70	1.03	0.33	0.88	1.07	0.19
	T.Test	0.730			0.066		



Lighting and CO Yield Variability

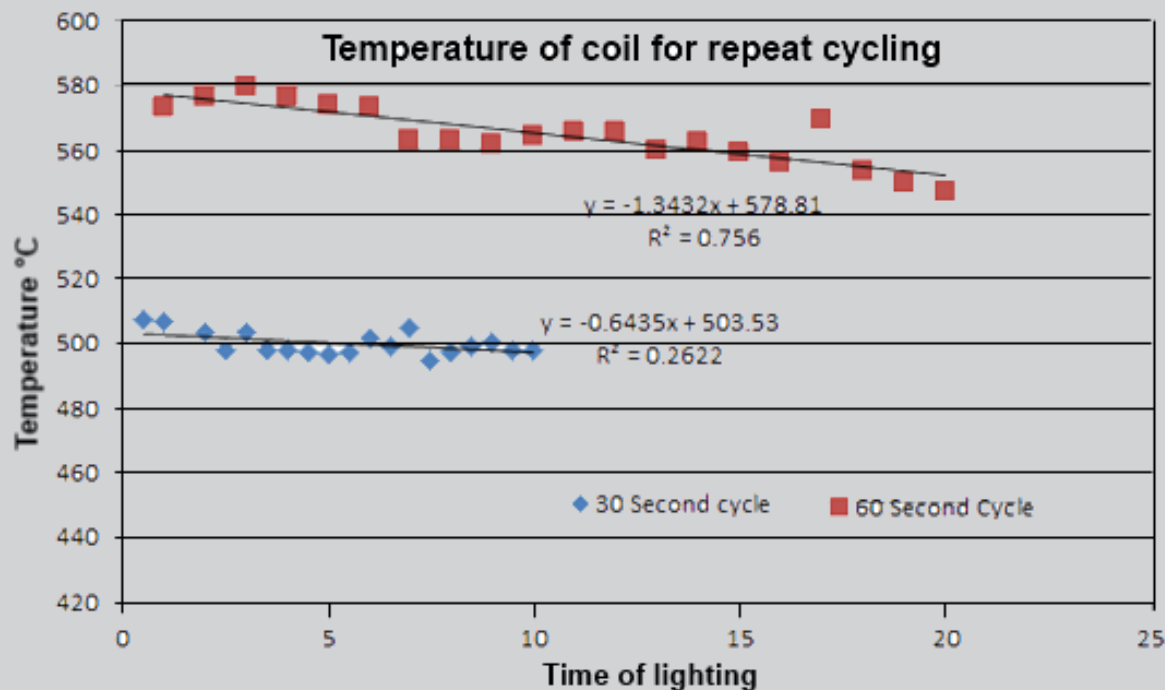
- ❖ Using two different lighting temperatures and monitoring the CO yield of a monitor test piece for the first two puffs shows different yields
- ❖ 30second pre-light corresponds to 500° and 60 seconds to 580°C
- ❖ Under HCl conditions the yields were statistically equivalent but not under the less intense puffing of ISO 35ml puff [3]





Lighting and CO Yield Variability

- ❖ Lighting is performed normally using a resistively heated coil.
- ❖ Determination of the lighting temperature and how this impacts yields is further complicated by ageing effects of the lighters
- ❖ To minimise variability in CO caused by lighting time and temperature requires control





Systematic Capture Errors

- ❖ **Insertion depth can vary by 1.7mm**
- ❖ **Overwrap and rode length vary due to manufacturing tolerances – typically ± 1 mm**
- ❖ **Overall tolerance build up for lighting point is up to 6 mm and for termination up to 4 mm**
- ❖ **If each rod is not treated individually for lighting and termination, variability can be introduced.**
- ❖ **The impact of cigarette holders becomes significant for HCl smoking and requires additional care for all smoking experiments to ensure consistency of TNCO yield**



Overview of recent findings

- ❖ **There are methodological features of ISO3308 that can introduce variability in TNCO yields**
- ❖ **The interpretation of ISO3308 has reached a consensus however the standard does not highlight all design features that contribute to variability**
- ❖ **Continuing proficiency testing, understanding of sources of variability, procedures applied that eliminate variation in yield have succeeded in achieving increased r and R performance over time**
- ❖ **The ISO standard was developed for ISO smoking and was not designed to be scalable for other regimes that generate higher water vapour levels**
- ❖ **Application of ISO3308 conditions to other puffing regimes is unlikely to achieve comparable r and R values.**



References

1. Health Canada Test Method T-115 – Determination of Tar, Water, Nicotine and Carbon Monoxide in Mainstream Tobacco Smoke, 1999-12-31
2. “2011 Collaborative study of CORESTA Monitors #6 (CM6) and #7 (CM7) for the determination of test piece weight, TPM, water, Nicotine, NFDPM, Carbon monoxide and puff count obtained under mainstream ‘ISO’ and ‘Intense’ smoking regimes”. Thomas Verron, 2011
3. Instrumental factors affecting reproducible smoke generation for routine TNC analysis. ACS 2013 I.Tindall
4. ISO 3308:2012 Routine analytical cigarette-smoking machine -- Definitions and standard conditions

See also

- Smoking machine design and yield errors under intense smoke regimes . Part 1: The influence of dead volume on yield. I.F.Tindall, L.Crumpler, T.Mason TSRC 2012
- Smoking machine design and yield errors under intense smoke regimes . Part 2: The influence of puff volume on desorption of semi-volatile smoke components I.F.Tindall, L.Crumpler, T.Mason TSRC 2012