Concept of characterization and certification of the 1R6F reference cigarette

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International ISO Standards followed

ISO Guide 34:2009 (ISO 17034:2016)
General requirements for the competence of reference material producers

ISO Guide 35:2009
Reference materials — General and statistical principles for certification

JCGM 100:2008 (revised version of GUM 1995)
Evaluation of measurement data — Guide to the expression of uncertainty in measurement

ISO-recommended approaches

- Approach A: single method in a single laboratory (better assessment of homogeneity)
- Approach B: Multiple methods in a single laboratory
- Approach C: a network of methods and/or laboratories (better estimate of the “true value”)
- Approach D: Method-defined parameters (technically different methods)

Characterization of the 1R6F follows a hybrid approach between Approach A, Approach C and Approach D.

Working principles and assumptions

- A certified value represents the best estimate of the “true” value.
- Certified values are not expected to deviate from the “true” value by more than the stated measurement uncertainty.
- The mean of method/laboratory means is assumed to be the best estimate of the certified value.

Main model for characterization (single lab/method) (ISO guide 35)

$$U_{CRM} = k\sqrt{u_{char}^2 + u_{bb}^2 + u_{lts}^2 + u_{sts}^2}$$

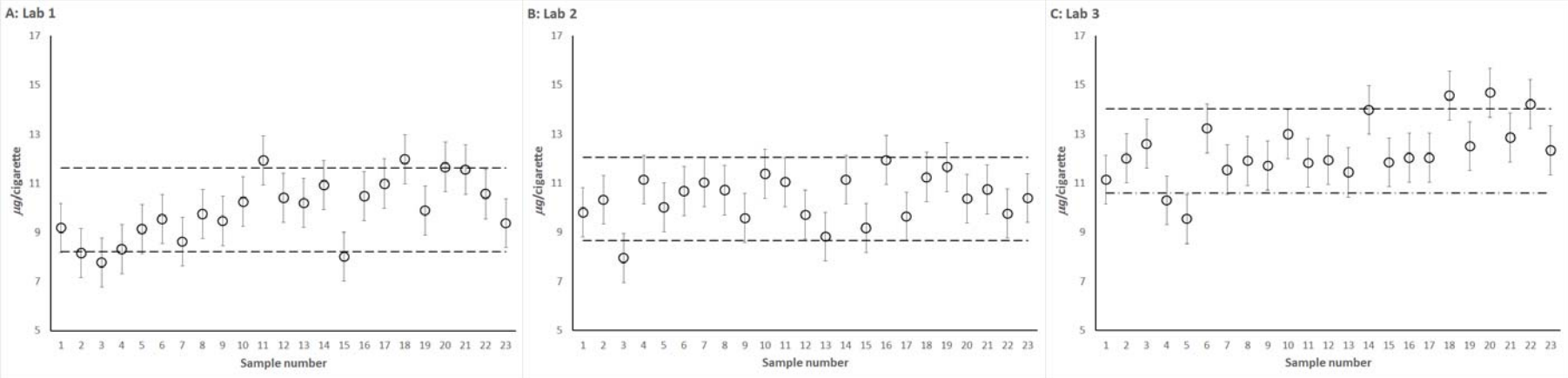
However, reference cigarettes are considered stable therefore the last 2 uncertainties relating to transport (short-term) and long-term storage stability were initially considered to be zero.

$$U_{CRM} = k\sqrt{u_{char}^2 + u_{bb}^2}$$

The $\boldsymbol{u_{bb}}$ signifies (and accounts for) the level of homogeneity as assessed by each laboratory while the $\boldsymbol{u_{char}}$ signifies (and accounts for) the level of dispersion/scatter in analytical results within that laboratory.

Single lab/method characterization of 1R6F

Crotonaldehyde ISO



	Mean	u_{bb}	u_{char}	$u_{comb,i}$
Lab 1	9.926	0.508	0.406	0.651
Lab 2	10.365	0.743	0.231	0.778
Lab 3	12.320	1.031	0.295	1.073

$$u_{comb,i} = \sqrt{u_{char}^2 + u_{bb}^2}$$

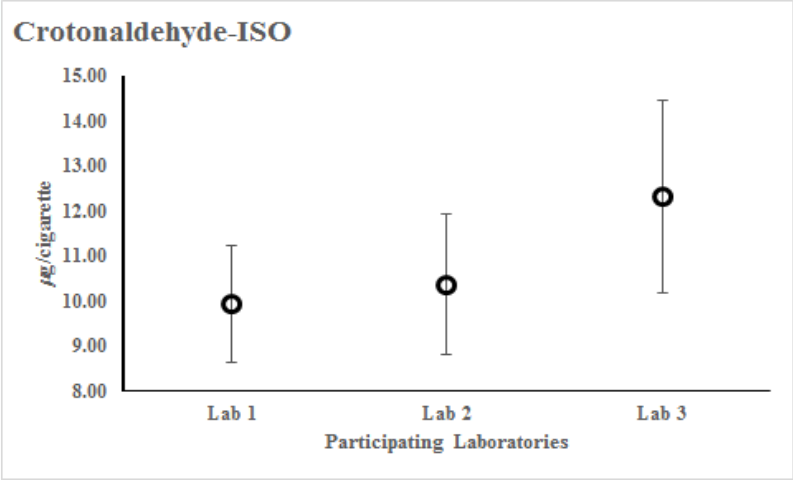
$$U_{CRM} = k\sqrt{u_{char}^2 + u_{bb}^2}$$

Single lab/method characterization of 1R6F

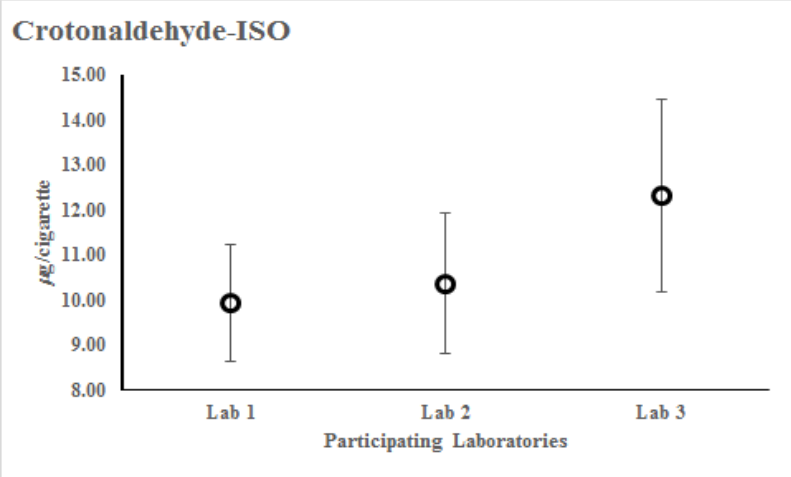
Crotonaldehyde in the ISO smoking regime							
	Mean	u_{bb}	u_{char}	$u_{comb,i}$	Within-lab combined uncertainty ($u_{comb,L}$)	Within-lab expanded uncertainty, k=2 ($U_{CRM,i}$)	Combined Within-lab expanded uncertainty, k=2 ($U_{CRM,L}$)
Lab 1	9.926	0.508	0.406	0.651	0.852	1.301	1.705
Lab 2	10.365	0.743	0.231	0.778		1.557	
Lab 3	12.320	1.031	0.295	1.073		2.145	

$$u_{comb,L} = \sqrt{\sum_{i=1}^{n} \frac{u_{comb,i}^2}{n}}$$

$$U_{CRM,L} = k\sqrt{u_{char}^2 + u_{bb}^2}$$



“Overall” expanded uncertainty



$$u_{CRM} = \sqrt{u_{fin}^2 + u_{comb,L}^2}$$

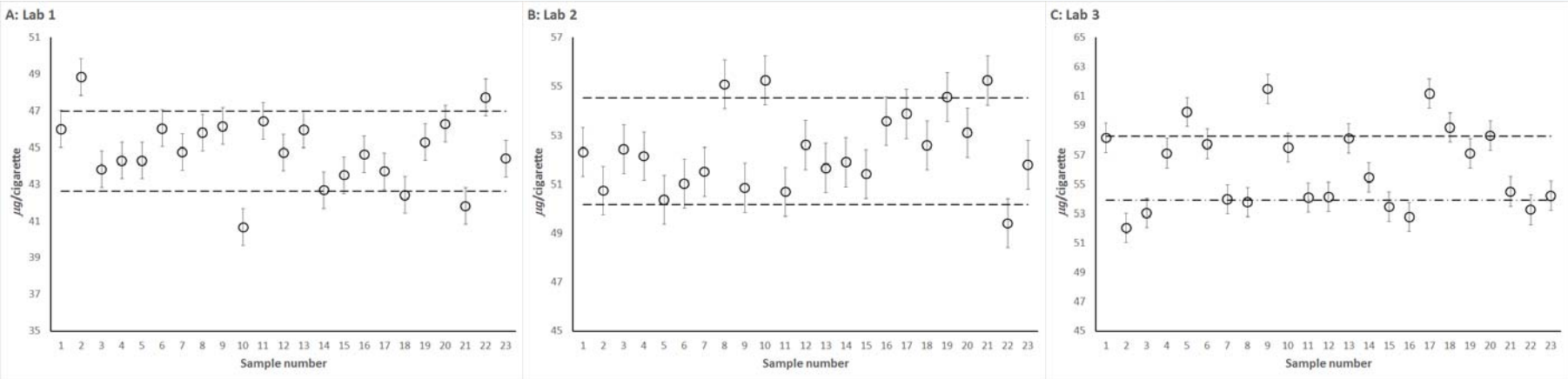
$$U_{CRM} = k\sqrt{u_{fin}^2 + u_{comb,L}^2}$$

The u_{fin}^2 signifies dispersion in “assigned values”, which demonstrate potential systematic effects

	Certified value ($\bar{x}$)	$u_{comb,L}$	u_{fin}	u_{CRM}	U_{CRM} (k=2)
Crotonaldehyde-ISO	10.870	0.852	0.742	1.130	2.260

Single lab/method characterization of 1R6F

Crotonaldehyde HCl



	Mean	u_{bb}	u_{char}	$u_{comb,i}$
Lab 1	44.798	0.834	0.486	0.966
Lab 2	52.356	0.831	0.478	0.959
Lab 3	56.105	1.096	0.713	1.308

$$u_{comb,i} = \sqrt{u_{char}^2 + u_{bb}^2}$$

$$U_{CRM} = k\sqrt{u_{char}^2 + u_{bb}^2}$$

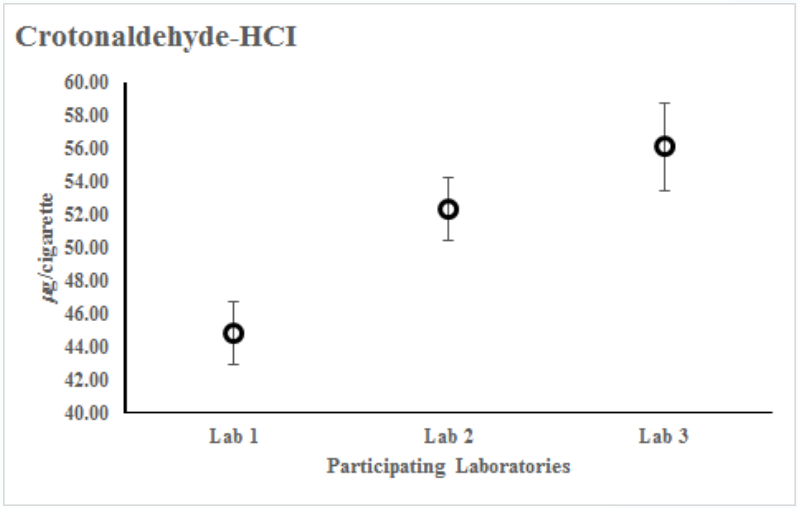
Single lab/method characterization of 1R6F

1B. Crotonaldehyde in the HCI smoking regime.

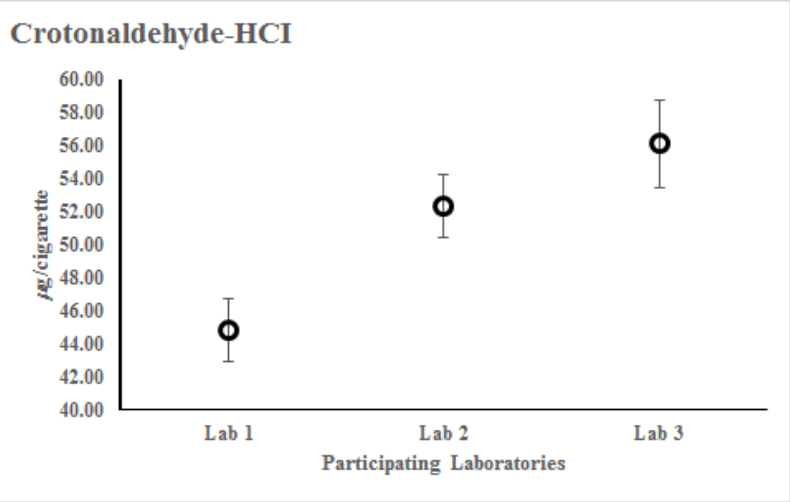
	Mean	u_{bb}	u_{char}	$u_{comb,i}$	Within-lab combined uncertainty ($u_{comb,L}$)	Within-lab expanded uncertainty, k=2 ($U_{CRM,i}$)	Combined Within-lab expanded uncertainty, k=2 ($U_{CRM,L}$)
Lab 1	44.798	0.834	0.486	0.966	1.090	1.931	2.179
Lab 2	52.356	0.831	0.478	0.959		1.918	
Lab 3	56.105	1.096	0.713	1.308		2.615	

$$u_{comb,L} = \sqrt{\sum_{i=1}^{n} \frac{u_{comb,i}^2}{n}}$$

$$U_{CRM,L} = k\sqrt{u_{char}^2 + u_{bb}^2}$$



“Overall” expanded uncertainty



$$u_{CRM} = \sqrt{u_{fin}^2 + u_{comb,L}^2}$$

$$U_{CRM} = k\sqrt{u_{fin}^2 + u_{comb,L}^2}$$

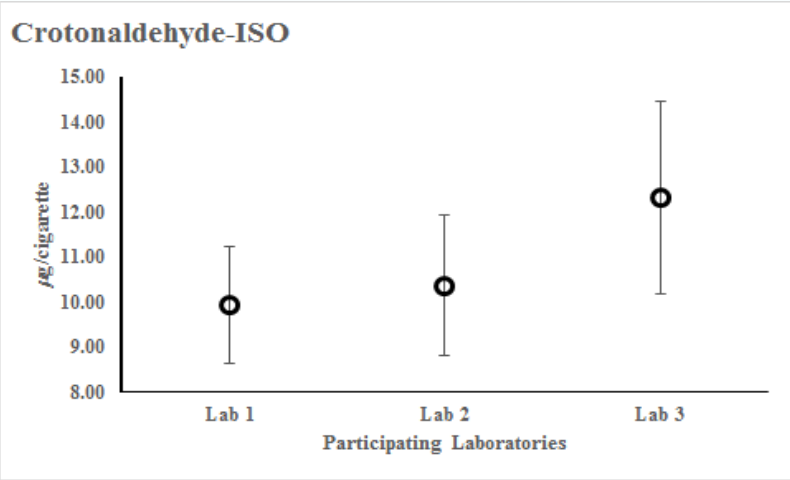
The u_{fin}^2 signifies dispersion in “assigned values”, which demonstrate potential systematic effects

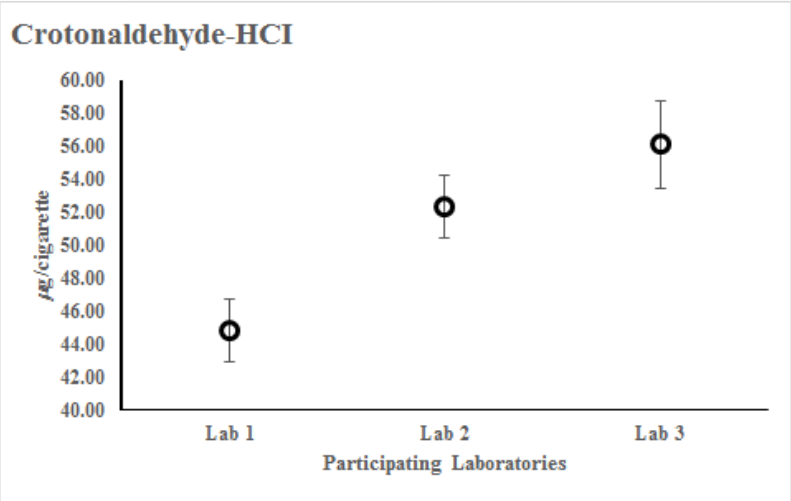
	Certified value ($\bar{x}$)	$u_{comb,L}$	u_{fin}	u_{CRM}	U_{CRM} (k=2)
Crotonaldehyde-HCl	51.0861	1.090	3.319	3.494	6.987

Coverage factor, k (ISO Guide 35)

Determine a coverage factor k to obtain an expanded uncertainty U, for which it may be assumed that the interval [$x - U$, $x + U$] contains **a large fraction of the distribution of values that could reasonably be attributed to the characteristic being certified**.

The choice of a coverage factor should be based on the required level of confidence (often 95 %), and (if applicable) the number of degrees of freedom.

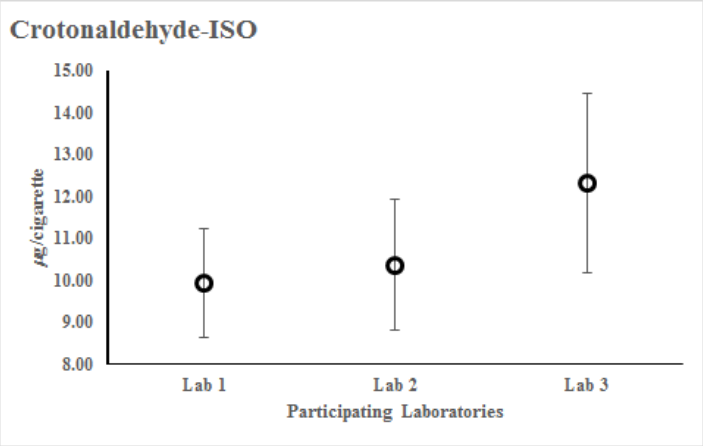


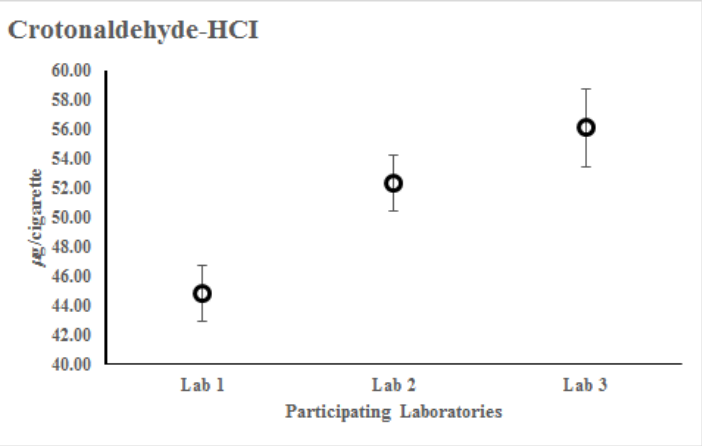


Note: Each lab analyzed 23 to 48 samples with 3 to 8 replicate analyses per sample; i.e. high degrees of freedom for each lab.

Coverage factor, k (JCGM 100:2008)

When the probability distribution characterized **approximately normal** and the effective **degrees of freedom is of significant size** one can assume that taking ***k* = 2** produces an interval having a level of confidence of approximately 95 percent, and that taking ***k* = 3** produces an interval having a level of confidence of approximately 99 percent.





Occasionally, one may find that a known **correction *b* for a systematic effect has not been applied** to the reported result of a measurement, but instead an attempt is made to take the effect into account by enlarging the "uncertainty" assigned to the result. **This should be avoided**; only in very special circumstances should corrections for known significant systematic effects not be applied to the result of a measurement.

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Thank you for your attention!