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Implementing ANSI/NCSL Z540 in an EPA Calibration Laboratory

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May 9, 2008

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Outline

- The APPCD Metrology Laboratory (MetLab)
- *The Guide to the Expression of Uncertainty in Measurement* (GUM), adopted as ANSI/NCSL Z540
- Examples of uncertainty analysis in our MetLab

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APPCD Metrology Laboratory (MetLab)

- The MetLab was Created in 1995 with a $24K budget and some donated/scavenged equipment.
- Now the MetLab operates with about $180K per year with equipment that was purchased new and under the MetLab control at all times.

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APPCD MetLab

- One set of calibration equipment for the entire division (no duplicate costs).
- Calibration equipment's calibrations are up to date and documented in a secure database.
- On-site calibrations reduce downtime and help identify measurement errors.
- Reduced technician training
- Principal investigator time

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APPCD MetLab

Calibrations are comparative using NIST traceable in-house standards.

Standards are compared to the device under test (DUT). Usually the DUT is not adjusted, but a linear adjustment equation is provided to compensate for most systematic error. These are useful with computer DAQ.

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APPCD MetLab Temperature Calibration

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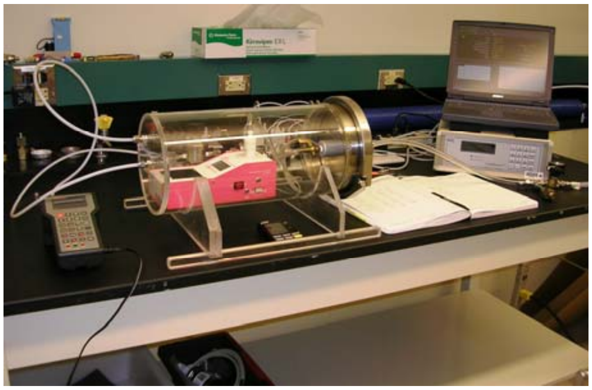
APPCD MetLab Flow Calibration



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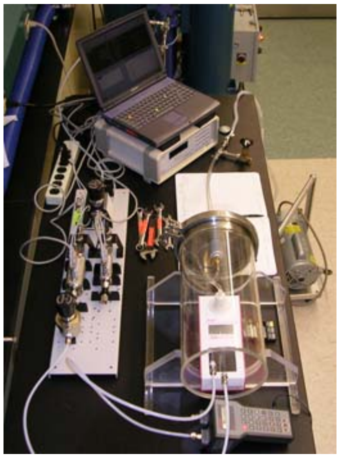
APPCD MetLab Flow Calibration



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APPCD MetLab Temperature Calibration

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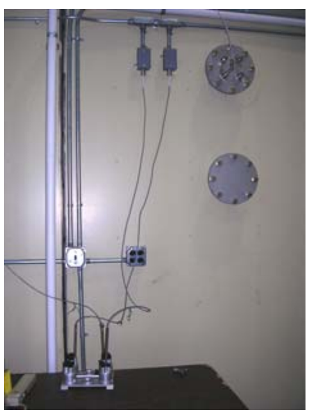
APPCD MetLab Relative Humidity (RH) Calibration



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APPCD MetLab RH Calibration

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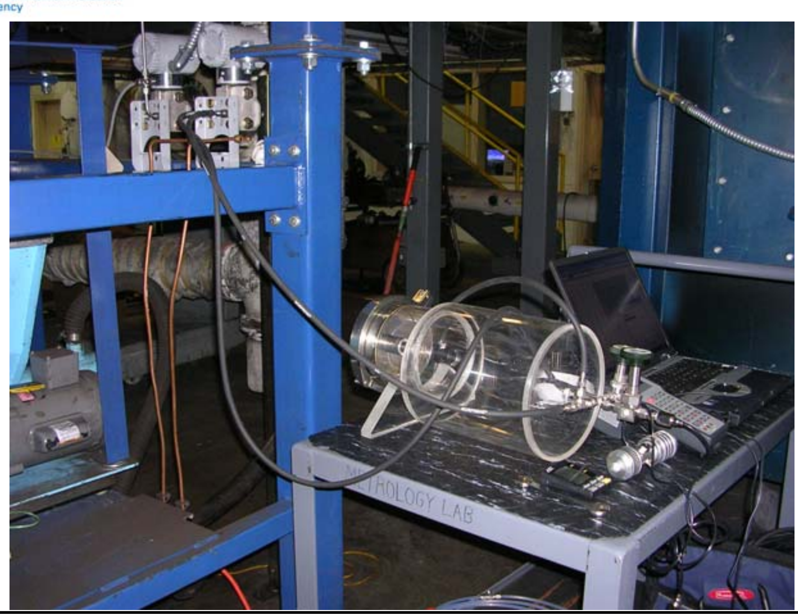
Consistent Improvement



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Consistent Improvement

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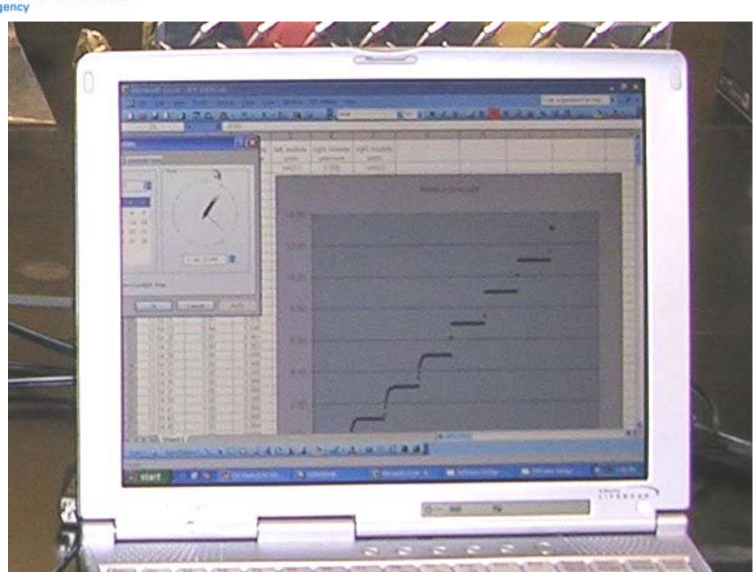
Consistent Improvement



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Scavengers

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Pack Rats?

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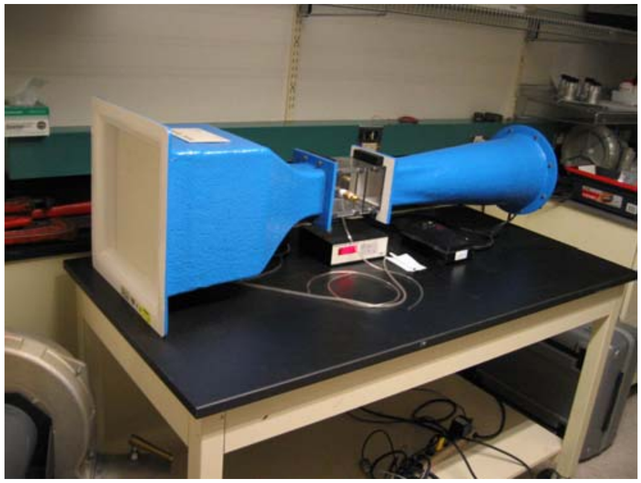
Small Wind Tunnel



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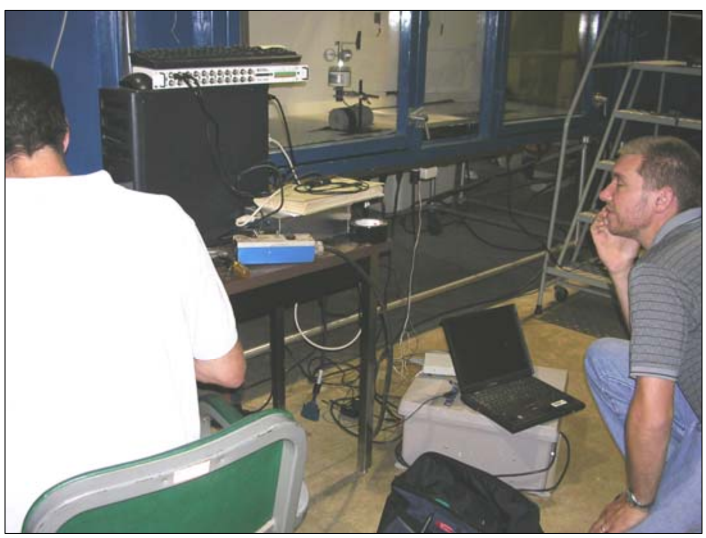
Large Wind Tunnel



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Electronic Calibration

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Calibration Report

- Once the DUT has been tested a calibration report is generated that includes the raw data and a statement of the uncertainty of the DUT.
- This uncertainty is determined using the GUM

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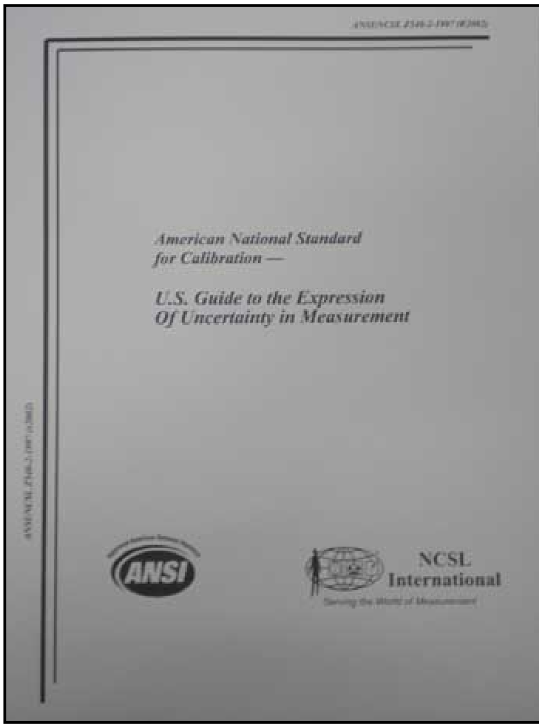

This guide is identical to the ISO/IEC 98 (1995) *Guide to the Expression of Uncertainty in Measurement* (GUM) with the exception of minor editorial changes to facilitate its use in the United States.

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The Intent of the Guide

- To give guidance on “a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement…”

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A Simple Example

A Straight Forward Uncertainty Calculation

Gas flow measurements require temperature and pressure to be expressed in absolute mass per time. So for a gas flow device to be calibrated the temperature and pressure uncertainties factor into the total uncertainty estimation.

$$Q = Flow = \frac{V}{t}$$

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A Straight Forward Uncertainty Calculation

$$u_c^2(Q) = \sum_{i=1}^{N} \left(\frac{\partial Q}{\partial x_i} \right)^2 u^2(x_i)$$

$$Q = \frac{V}{t}$$

$$u_c^2(Q) = \left(\frac{\partial \left(\frac{V}{t} \right)}{\partial (V)} \right)^2 u^2(V) + \left(\frac{\partial \left(\frac{V}{t} \right)}{\partial (t)} \right)^2 u^2(t)$$

$$u_c^2(Q) = \left(\frac{1}{t} \right)^2 u^2(V) + \left(\frac{-V}{t^2} \right)^2 u^2(t)$$

Q =	flow
V =	volume
t =	time
u_c =	standard uncertainty

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GUM 5.1.6

"If Y is of the form $Y = cX_1^{P_1} X_1^{P_2} \ldots X_1^{n_1}$ and the exponents p_i are known positive or negative numbers having negligible uncertainties, the combined variance, equation (10), can be expressed as

$$\left[u_c(y) / y \right]^2 = \sum_{i=1}^{N} \left[p_i u(x_i) / x_i \right]^2 \text{ “}$$

$$\left(\frac{u_c(Q)}{Q} \right)^2 = \left(\frac{u\ (V)}{V} \right)^2 + \left(\frac{u\ (t)}{t} \right)^2$$

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A Straight Forward Uncertainty Calculation

When calculating mass emissions volume must be converted into mass using the ideal gas law.

$$PV = nRT = NkT$$

$$V_1 = Nk(T_1/P_1)$$

$$N = (P_1V_1)/(kT_1)$$

P =	pressure
R =	universal gas constant
n =	number of moles
k =	boltzmann constant
N =	number of molecules

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Don't Waste Time

$$u_c^2(N) = \left(\frac{\partial\left(\frac{P_1V_1}{kT_1}\right)}{\partial(P)} \right)^2 u^2(P) + \left(\frac{\partial\left(\frac{P_1V_1}{kT_1}\right)}{\partial(V)} \right)^2 u^2(V) + \left(\frac{\partial\left(\frac{P_1V_1}{kT_1}\right)}{\partial(T)} \right)^2 u^2(T)$$

$$u_c^2(N) = \left(\frac{V_1}{kT_1}\right)^2 u^2(P) + \left(\frac{P_1}{kT_1}\right)^2 u^2(V) + \left(\frac{-P_1V_1}{kT_1^2}\right)^2 u^2(T)$$

$$N = \frac{P_1V_1}{kT_1}$$

$$u_c^2(N) = \left(\frac{N}{P_1}\right)^2 u^2(P) + \left(\frac{N}{V_1}\right)^2 u^2(V) + \left(\frac{-kN^2}{P_1V_1}\right)^2 u^2(T)$$

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Don't Waste Time

$$N = \frac{P_1 V_1}{kT_1}$$

$$\left(\frac{u_c(N)}{N}\right)^2 = \left(\frac{u(P)}{P}\right)^2 + \left(\frac{u(V)}{V}\right)^2 + \left(\frac{u(T)}{T}\right)^2$$

$= (0.06\%)^2 + (1\%)^2 + (0.3\%)^2 = 1.1\%^2$

$U_c(N) = 1.04\%$

These are just the uncertainties do to the standards

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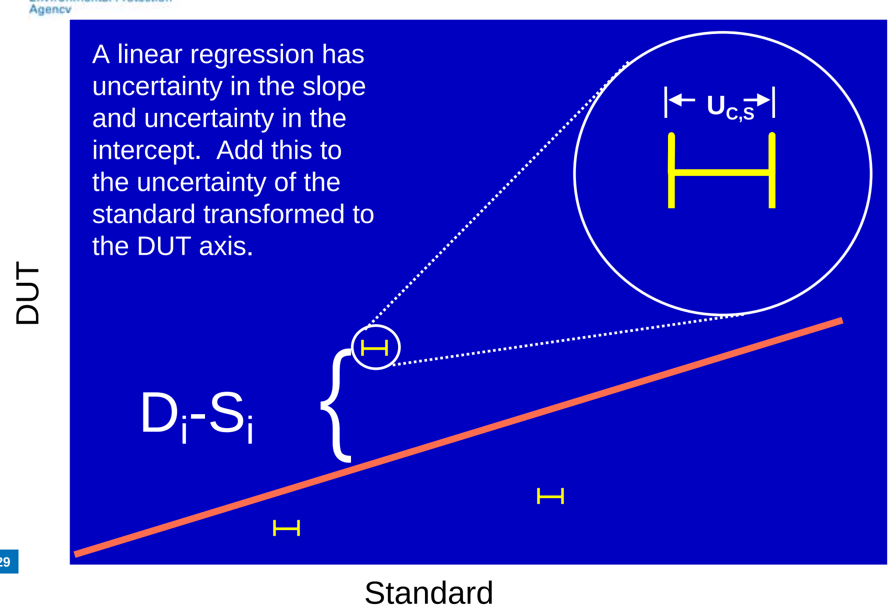
Add in the Variability of the DUT



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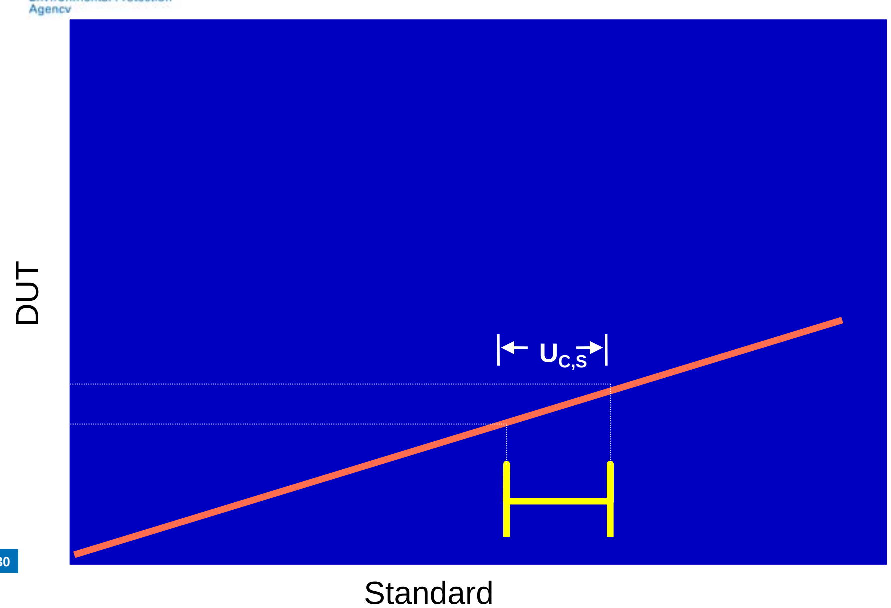
Translate Uc of Standard to DUT



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A Not-So-Straight Forward Uncertainty Calculation

The determination of the ozone concentration, C, in air

$$C = \frac{-1}{2\alpha L}\frac{T_{mes}}{T_{std}}\frac{P_{std}}{P_{mes}} ln(D)$$

α is the absorption cross-section of ozone at 253.7 nm in standard conditions of temperature and pressure. The value used is: 1.1476×10^{-17} cm^2/molecule [1, 4]. In (1):

- L is the optical path length of one of the cells,
- T_{mes} is the temperature measured in the cells,
- T_{std} is the standard temperature (273.15 K),
- P_{mes} is the pressure measured in the cells,
- P_{std} is the standard pressure (101.325 kPa),
- D is the product of transmittances of two cells :

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SRP1 in APPCD MetLab

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International Comparison CCQM-P28, Ozone at ambient level (Pilot study)

These instruments are manufactured by NIST and are basically identical. Each standard organization calculated their own uncertainty and the results vary to a greater extent than one might think.

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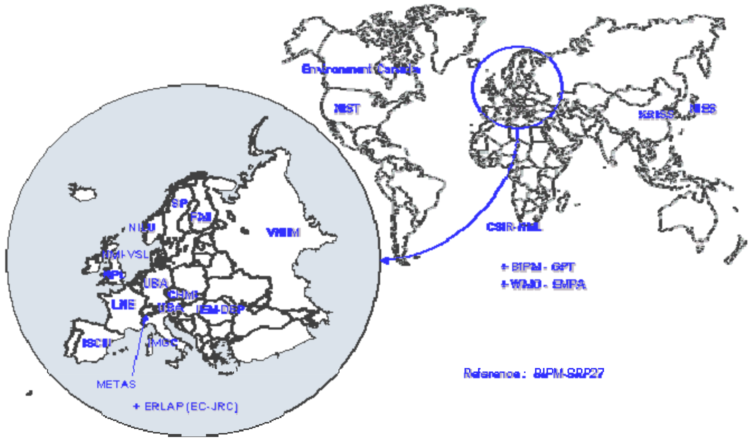
International Bureau of Weights and Measures (BIPM) CCQM-P28



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BIPM CCQM-P28

- BIPM sent out SRP 27 for comparison to other national standard laboratories comparing it to SRP 28 in between each iteration.
- BIPM provided the uncertainty calculated for their SRP along with the method used to obtain it.

$$u(x) = \sqrt{\left(\frac{u(D)x}{D\ln(D)}\right)^2 + \left(\left(\frac{u(2L)}{2L}\right)^2 + \left(\frac{u(P)}{P}\right)^2 + \left(\frac{u(T)}{T}\right)^2\right)x^2}$$

$$u(x) = \sqrt{(0.28)^2 + 2.09 \cdot 10^{-7} x^2}$$

Where x is the best estimate of C and U(x) is the uncertainty of x at 95% confidence

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Component (y)	Uncertainty $u(y)$				Sensitivity coefficient $c_i = \frac{\partial x}{\partial y}$	contribution to $u(x)$ $\lvert c_i \rvert \cdot u(y)$
	Source	Distribution	Standard Uncertainty	Combined standard uncertainty $u(y)$		
Optical Path $2L$	Measurement Scale	Rect.	0.0011 cm	0.014 cm	$-\frac{x}{2L}$	$\frac{u(2L)\cdot x}{2L}$
	Repeatability	Normal	0.01 cm			
Pressure P	Pressure gauge	Rect.	0.029 kPa	0.034 kPa	$-\frac{x}{P}$	$\frac{u(P)\cdot x}{P}$
	Difference between cells	Rect.	0.017 kPa			
Temperature T	Temperature probe	Rect.	0.087 K	0.087 K	$\frac{x}{T}$	$\frac{u(T)\cdot x}{T}$
Ratio of intensities D	Scalers resolution	Rect.	8×10^{-6}	1.4×10^{-5}	$\frac{x}{D\ln(D)}$	$\frac{u(D)\cdot x}{D\ln(D)}$
	Repeatability	Triang.	1.1×10^{-5}			
Absorption Cross section α	Conventional value		8.6×10^{-20} cm²/molecule	8.6×10^{-20} cm²/molecule	$-\frac{x}{\alpha}$	$\frac{u(\alpha)\cdot x}{\alpha}$

Table 1: Uncertainty budget for the SRPs maintained by the BIPM

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BIPM SRP 28 Control

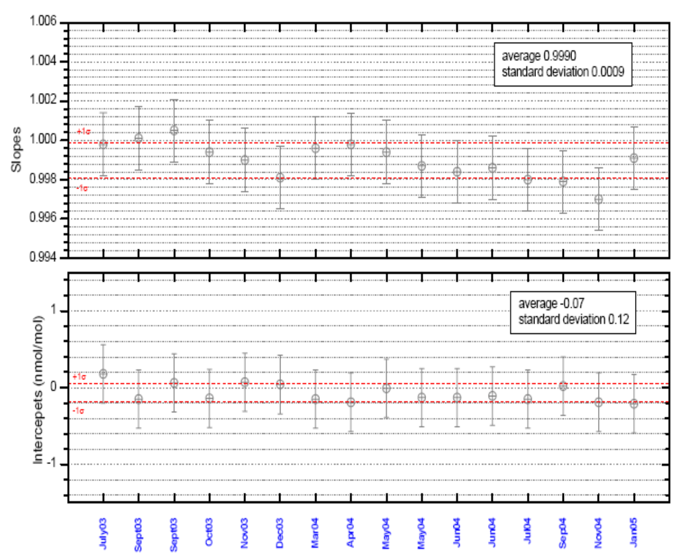

Figure 3 : Results of BIPM-SRP28 versus BIPM-SRP27 comparisons performed during the pilot study

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Results

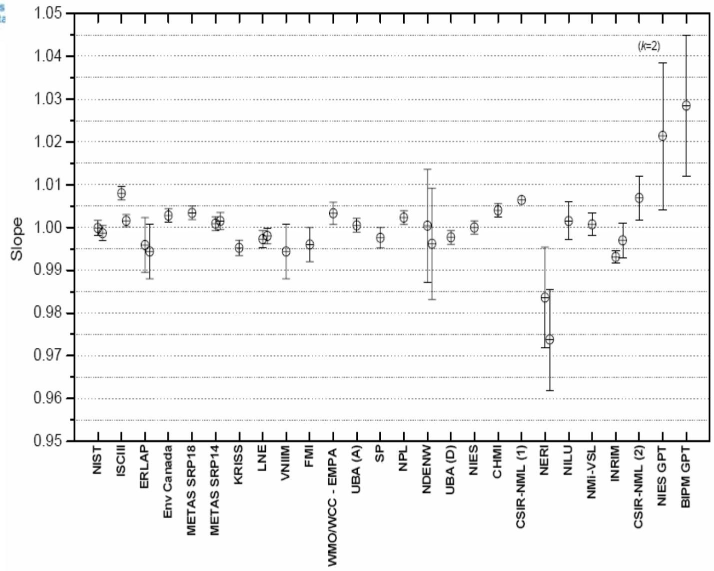

Figure 1 : Slopes of the regression

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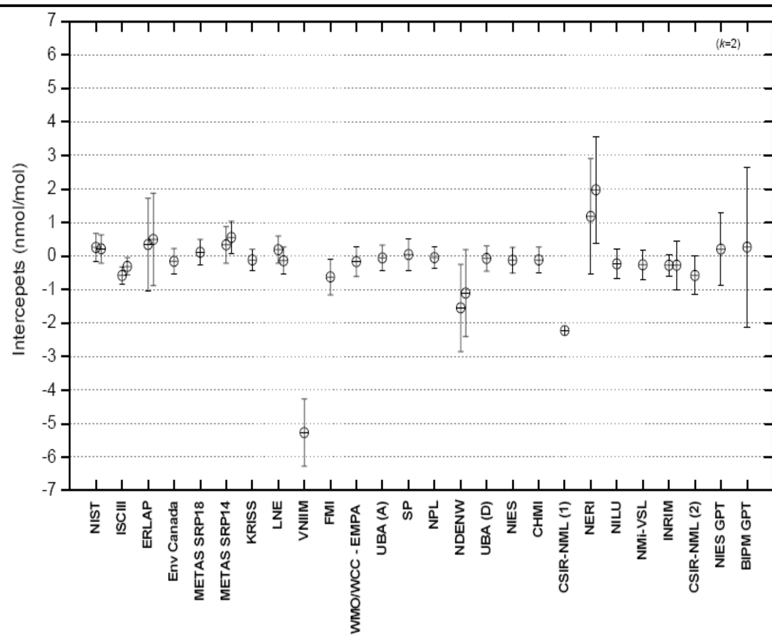

Figure 2 : Intercepts of the regression

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International Comparison CCQM-P28, Ozone at ambient level (Pilot study)

"As a final result of the pilot study, the difference from the reference value (BIPM-SRP27 measurement result) and its related uncertainty were calculated for each ozone standard at the two nominal ozone mole fractions of 80 nmol/mol and 420 nmol/mol."

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BIPM CCQM-P28

	INRIM	ISCIII	FMI	METAS	METAS	NPL	SP	EMPA	EMPA
SRP Number	2	22	24	14	18	20	11	15	15
Ozone Concentration (nmol/mol)	200	200	199.27	200	201	200	117.2		200
Uncertainty (nmol/mol)	0.937303	0.31749	0.384543	0.372022	0.372022	0.221775	0.28	0.25	0.235321
% Uncertainty	0.47%	0.16%	0.19%	0.19%	0.19%	0.11%	0.24%	N/A	0.12%

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NPL (United Kingdom)

Term	Factors affecting uncertainty	Means of evaluating	component
L	Measurement uncertainty	Estimate	u(L)
	Environmental factors (thermal expansion)	Estimate	negligible
T	Measurement uncertainty, including temperature gradients	Estimate	u(T)
	Drift	From repeat measurements	included in u(rep)
P	Measurement uncertainty, including pressure gradients	Estimate	u(P)
	Drift	From repeat measurements	included in u(rep)
I/I_o	Measurement uncertainty, including source and detectors repeatability and drift	From repeat measurements	u(rep)
	Instrument resolution	Estimate	negligible
	Environmental factors (effect of temperature on source and detectors)	Estimate	u(E)
	Detector non-proportionality	Estimate	u(NP)

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Component (y)	Source	Distribution	Standard Uncertainty
Optical Path $2L$	Measurement Scale	Rect.	0.0011 cm
	Repeatability	Normal	0.058 cm
Pressure P	Pressure gauge certificate	Normal	0.020 kPa
	Repeatability	Normal.	0.010 kPa
	Difference between cells	Rect	0.017 kPa
Temperature T	Temperature probe certificate	Normal	0.01 K
	repeatability	Normal	0.005 K
Ratio of intensities D	Scalers resolution	Rect.	8×10^{-6}
	Repeatability	Normal	5.2×10^{-6}

Term	size	distribution	Standard u/c
u(L)	2×10^{-4} m	Rect	1.2×10^{-4} m
u(T)	0.3 K	rect	0.17 K
u(P)	100 Pa	rect	58 Pa
u(E)	0.1 nmol/mol	gaussian	0.1 nmol/mol
u(rep)	0.1 nmol/mol	gaussian	0.1 nmol/mol
u(NP)	4×10^{-4} x/B $[I/I_o]$	rect	2.3×10^{-4} x/B $[I/I_o]$

$$u(x) = \sqrt{(0.14)^2 + 7.4.10^{-7} x^2} \quad \text{nmol/mol}$$

$$u(x) = \sqrt{0.19^2 + (4.28 \cdot 10^{-4} x)^2}$$

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Assumptions

Some of the shortcuts in the Guide assume that the uncertainty is small compared to the measurement, that the data is normally distributed and that the individual uncertainty components are not correlated.

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Expanded Uncertainty

$$U_e = ku_c$$

For normally distributed data with infinite degrees of freedom the confidence intervals correspond to:

k=1 68%

k=2 95.5%

K=3 99%

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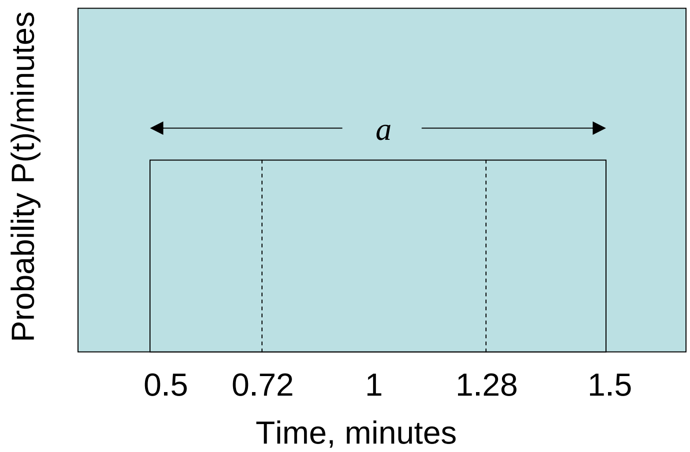
Uncertainty of a Rectangular Distribution



$$u_c(t) = \frac{a}{2\sqrt{3}} = 0.28$$

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Uncorrected Bias

Thermocouples (TC) work off the principle that two dissimilar metal wires induce a current when they are joined at one end. Off the production line each TC is not tested, but instead the uncertainty is based on the variance of the TCs off the production line

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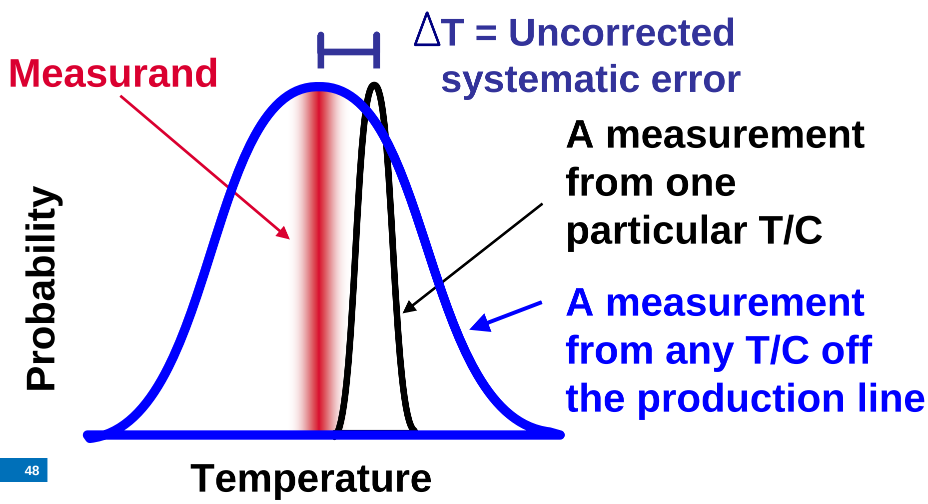
Probability Distribution Function



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APPCD Metrology Laboratory Personnel

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Questions?

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