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		LAP: Calibration
SUBJECT: Requirements for Laboratories Calibrating Flow by Primary Methods		

The purpose of this bulletin is to advise laboratories performing primary-method-based calibrations of flow and related quantities about specific NVLAP policies and requirements for ISO/IEC 17025 assessments. This bulletin covers information required for a laboratory's calibration and measurement capabilities listing on its scope of accreditation, provides detail about determination of measurement uncertainty in flow measurements, and also provides detail about proficiency testing for flow and related quantities. Review of these three areas is an integral part of the NVLAP assessment of laboratories performing calibration of flow and related quantities.

Note there are new requirements in this document which laboratories must address by September 1, 2017.

Scope of Accreditation

The scope of accreditation, calibration and measurement capability (CMC), and associated notes are to inform customers and assessors of the accredited capabilities of the laboratory, including the range of conditions (i.e. fluid type, pressure, temperature) the laboratory provides during the calibration of a customer's device. Laboratories shall provide a complete list of the ranges of pertinent parameters that are accredited for the facility. Scopes for flow laboratories shall include:

- 1. **Measurand**: The NVLAP list of measurands for flow laboratories are: liquid flow, gas flow, air speed, liquid volume, and hydrometer correction.
- 2. **Range of Measurand**: The minimum and maximum values of the measurand over which the laboratory is accredited shall be listed in the CMC table.
- 3. **CMC Uncertainty:** The 95% confidence level expanded uncertainty for the output of the calibration, including uncertainties contributed by the device under test (i.e. not the uncertainty of the reference standard alone) shall be listed in the CMC table. The CMC lists the smallest uncertainty of measurement that is normally available to the laboratory's customers for a nearly ideal flow meter (best existing device). The uncertainty related to the device under test shall be repeatability based on the normal number of measurements performed at each flow set point, corrected for the finite sample size using the Welch-

Satterthwaite method as described in the GUM [1] or appropriately applied *t*-values. The uncertainty in customer calibration reports will nearly always be larger than the uncertainty stated in the CMC table because of extra uncertainty components that arise when the meter under test is not the best existing device (see section below entitled Uncertainty in Customer Calibration Reports). More information on these topics and references to example uncertainty analyses for flow can be found in *Working Group for Fluid Flow Guidelines* [2]. The *WGFF Guidelines* are applied to national metrology institutes when they generate CMCs for the Bureau International des Poids et Mesures.

- 4. **Instrument Type**: In some cases, a laboratory is only qualified to calibrate particular meter types, for instance, its own product. In those cases, the types of devices that the laboratory is accredited to calibrate shall be stated as a note to the scope of accreditation. For example, "Stated CMC best uncertainty is for calibration of electromagnetic flow meters. Calibration of other flow meter types is available at higher uncertainty."
- 5. **Fluid Type**: Example entries are: water, dry air, nitrogen, natural gas, carbon dioxide, mineral oil, gasoline, etc.
- 6. **Pressure of Fluid**: Minimum and maximum values of pressure of the fluid applied to the device under test during calibration.
- 7. **Temperature of Fluid**: Minimum and maximum values of temperature of the fluid applied to the device under test during calibration.
- 8. **Kinematic Viscosity of Fluid**: Minimum and maximum values of kinematic viscosity of the fluid applied to the device under test during calibration.

Fluid type, pressure, temperature, and kinematic viscosity of the fluid shall be entered in the Remarks section of the CMC table or in the Notes section.

Uncertainty for a Customer Calibration Report

The uncertainty listed in the NVLAP scope of accreditation will almost always be smaller than the uncertainty in a calibration report for a customer's instrument. A customer calibration report shall give the uncertainty of the reported quantity which is not flow alone, but is usually a "performance indicator" like discharge coefficient, meter factor, or some dimensionless quantity appropriate for the meter type. Therefore, the uncertainty in a calibration report includes not just the uncertainty of the reference standard and the reproducibility of the best existing device. It will include additional uncertainty components not included in the CMC related to: 1) instrumentation associated with the meter under test (e.g., frequency, pressure, temperature

¹ Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008.

² WGFF Guidelines for CMC Uncertainty and Calibration Report Uncertainty, Working Group for Fluid Flow, October 21, 2013, <u>http://www.bipm.org/utils/en/pdf/ccm-wgff-guidelines.pdf</u>.

sensors...), 2) fluid properties, and 3) repeatability or short-term reproducibility for the customer's meter under test (generally larger than for the best existing device).

Note that the reported uncertainty of the performance indicator must not be less than the uncertainty stated in the CMC at that point in the operating range.

In some cases, the uncertainty of the fluid properties indirectly impacts the uncertainty of the performance indicator. For example, calibration data for a turbine meter is often presented as Strouhal number St versus Roshko number Ro. The Strouhal number is defined as:

$$St = \frac{f \pi D^3}{4\dot{V}} \tag{1}$$

where f is the rotor frequency of the meter under test, D is the diameter of the meter under test, and \dot{V} is the actual volumetric flow at the meter under test. The Roshko number is:

$$Ro = \frac{f D^2}{v} \tag{2}$$

where ν is the liquid kinematic viscosity. If the change in *St* with respect to *Ro* is large (the calibration curve is not flat), uncertainty of the Roshko number shall be accounted for in the uncertainty of the Strouhal number. The following example of text from a turbine meter calibration report explains this in more detail. The example provides quantitative values for the uncertainty of the frequency, the volumetric flow, and the kinematic viscosity to illustrate the magnitude of these components. The uncertainties of these quantities are based on the measurement techniques used for their determination. A sample of text from a calibration report is given as an example in the following section. The uncertainty values used in the example do not necessarily apply to all turbine meter calibrations, and calibration laboratories should apply values based on their own independent uncertainty analysis.

Example Uncertainty Analysis for a Calibration Report

An analysis was performed to assess the uncertainty of the calibration factors obtained for the meter under test. This process involves identifying all of the significant uncertainty components and quantifying each of them at the 68 % confidence level. Additionally, we determine the sensitivity coefficients (*S*) for each component by partial differentiation of equation 1 and equation 2. The uncertainty terms are then combined by the root-sum-of-squares method (RSS) to obtain the relative combined uncertainty, u_c , which in turn, is multiplied by a coverage factor (k = 2) to give the relative expanded uncertainty, $U_e = k u_c$ at a confidence level of approximately 95 %.

$$\frac{U_{\rm e}(St)}{St} = k \cdot \frac{u_{\rm c}(St)}{St} = k \cdot \sqrt{\left(s_f \frac{u(f)}{f}\right)^2 + \left(s_{\dot{V}} \frac{u(\dot{V})}{\dot{V}}\right)^2 + \left(s_{RO} \frac{u(RO)}{RO}\right)^2 + u(R)^2} \tag{3}$$

The uncertainty components for this calibration include the relative standard uncertainty of the frequency measurements of the meter under test, u(f)/f, the relative standard uncertainty of the actual volumetric flow, $u(\dot{V})/\dot{V}$, the relative standard uncertainty of *Ro*, and the

reproducibility of the meter under test, u(R) [3]. Partial differentiation of equation 1 shows the sensitivity coefficient for frequency, $s_f = \partial St / \partial f \cdot f / St = 1.0$ and the sensitivity coefficient for volumetric flow, $s_{\dot{v}} = \partial St / \partial \dot{V} \cdot \dot{V} / St = -1.0$. The sensitivity coefficient s_{RO} is determined by the St, Ro relationship of the meter under test by: $s_{RO} = \Delta St / \Delta Ro \cdot Ro / St$. The value of s_{RO} is not constant, but varies over the span of the St versus Ro calibration curve. In general, s_{RO} is nearly zero at high flows where a turbine meter calibration curve is typically flat; however, s_{RO} can be large at low flows where a turbine meter's calibration curve has a non-zero slope. The third term under the radical in equation 3 shows how s_{RO} affects the uncertainty of $U_e(St)/St$. When the magnitude of s_{RO} is near zero, it reduces the contribution of u(RO)/RO to $U_{e}(St)/St$. In contrast, when s_{RO} is large it amplifies the contribution of u(RO)/RO to $U_e(St)/St$, and in some cases can be the dominate source of uncertainty in equation 3. The relative standard uncertainty of the frequency measurements for the meter under test is determined by assuming 1 pulse count can be missed out of the total pulses counted. This component is assumed to have a rectangular distribution and hence is calculated by $u_t = 1/(f \cdot t)/\sqrt{3}$, where t is the duration of the calibration point. The relative standard uncertainty for volumetric flow from the prover is 0.028 %.

Because *St* is a function of *Ro*, the uncertainty in *Ro* must be considered. The uncertainty in *Ro* is calculated by:

$$\frac{U_{\rm e}(Ro)}{Ro} = k \cdot \frac{u_{\rm c}(Ro)}{Ro} = k \cdot \sqrt{\left(s_{\nu} \frac{u(\nu)}{\nu}\right)^2 + \left(s_f \frac{u(f)}{f}\right)^2} \tag{4}$$

The uncertainty components include the relative standard uncertainty of the frequency measurements of the meter under test, u(f)/f, and the relative standard uncertainty of the kinematic viscosity, u(v)/v. Partial differentiation of equation 2 shows that the sensitivity coefficient for frequency is 1 and the sensitivity coefficient for kinematic viscosity, $s_v = \partial Ro/\partial v \cdot v/Ro$, is -1. The standard relative uncertainty of the frequency is calculated as discussed in the prior paragraph. The standard relative uncertainty in kinematic viscosity in this calibration service is 0.10 %. The uncertainty of the kinematic viscosity is determined by how well we can characterize the fluid using a densimeter and a falling ball viscometer and how well we can measure the temperature at the meter under test during calibration.

To measure the flowmeter reproducibility, the standard deviation of the mean of 10 measurements was used to calculate the relative standard uncertainty (*i.e.*, the sample standard deviation of the mean divided by the average and expressed as a percentage) at each of the nominal flows. The flowmeter reproducibility is a type A uncertainty, while all of the other uncertainty components are type B. Using the uncertainty values given above and equation 3 yield the relative expanded uncertainties for the Strouhal number between 0.06 % and 0.08 %.

³ Note that the uncertainty in flow meter diameter can be neglected during usage of the flowmeter as long as the same reference diameter is used.

Requirements for Proficiency Testing

ISO/IEC 17025 accredited laboratories are required to provide the results of proficiency testing to assessors that demonstrate that they achieve their uncertainty specifications. Accredited laboratories are responsible for obtaining proficiency test results and shall use transfer standards and proficiency test participants with uncertainty comparable to their own (i.e. within a factor of 2).

An accredited laboratory is responsible for performing internal comparisons between systems with overlapping measurement ranges and maintaining records (including graphical representations) of the results of these internal comparisons. An accredited laboratory is also responsible for maintaining control charts that show the stability of calibration results from periodic calibrations of check standards using the same reference standard. Internal comparison and check standard data should be used to reduce the proficiency testing (external comparison) workload. For example, an accredited laboratory that maintains five reference standards is not required to perform proficiency testing (external comparisons) with each of the five reference standards, rather, one proficiency test will suffice if there are successful internal comparison results that connect that one reference standard to the other four.

NVLAP-accredited laboratories performing flow calibrations are not required to use NIST for proficiency testing. However, they are *encouraged* to use NIST for proficiency testing except 1) when the laboratory has CMC uncertainty *greater than twice* the NIST uncertainty specified for the measurand in the BIPM Key Comparison Data Base (<u>www.bipm.org</u>) or 2) when the measurand or needed range is not supported by NIST. If NIST or another NMI is not used, accredited laboratories with comparable uncertainty (i.e. within a factor of 2) should be used in the proficiency testing.

Note that this laboratory bulletin does not nullify the following two clauses from NIST Handbook 150-2-2016:

3.4.3.1 Calibration proficiency tests may be organized by NVLAP in consultation with NIST experts for parameters where laboratories are operating at or near NIST uncertainties.

3.4.4 a) Where NVLAP provides coordination for PT, a laboratory accredited for the parameter and range within the specified uncertainty for the test shall participate.

The proficiency test requirements for flow laboratories are:

- 1. Comparisons will be performed at least every five years, generally following the *Guidelines for CIPM Key Comparison* [4].
- 2. An unstable transfer standard can lead to inconclusive proficiency test results. Preliminary tests shall be conducted to assess the stability of the transfer standard to be used and its sensitivity to transport, environmental conditions, fluid properties, etc. The transfer standard shall be demonstrated to have reproducibility less than the accredited labs' uncertainty

⁴ *Guidelines for CIPM Key Comparisons*, March 1, 1999, http://www.bipm.org/utils/en/pdf/guidelines.pdf.

through preliminary testing or (if such a device does not exist) the transfer standard must have the best commercially available calibration stability. At minimum, calibration results for the transfer standard from the beginning and the end of the proficiency test done by one of the participants shall be used to quantify transfer standard stability.

- 3. The comparison shall be blind, i.e. the pilot laboratory or some third party will not share measurement results with the laboratory seeking accreditation until all measurements are complete. Results cannot be altered to improve agreement.
- 4. For successful accreditation, the standardized degrees of equivalence at all test flows, defined as the difference between the accredited laboratory and the proficiency test provider divided by the root-sum-square of the two laboratory uncertainties and the uncertainty introduced by the transfer standard, shall be less than or equal to 1 for 95 % or more of the proficiency test set points.
- 5. The comparison shall be performed at 2 or more values of the measurand that differ by at least a factor of 5, but wider ranges are encouraged if the transfer standard is capable.
- 6. Normally, laboratories have ranges of measurand in their scope that cannot be covered by a single transfer standard. Therefore, the accredited laboratory should choose different ranges of the measurand for different comparisons so that over time, proficiency is demonstrated over the entire range of the measurand.

Implementation of Requirements

Uncertainty budgets—laboratories shall verify that their flow uncertainty budgets and associated procedure documents meet the requirements set out above. Any uncertainty budgets requiring revision to comply with these requirements shall be revised and uploaded to the NVLAP portal by September 1, 2017.

Proficiency testing—laboratories shall verify that their proficiency testing plans covering flow capabilities meet the requirements set out above. Any plans requiring revision to comply with these requirements shall be revised and uploaded to the NVLAP portal by September 1, 2017.

Assessment During Implementation Period

During the implementation period (now through September 1, 2017), assessors will assess against these requirements. However, no nonconformities on *new* requirements contained herein will be cited. Instead, assessors will document as *comments* those areas where laboratories do not yet comply.