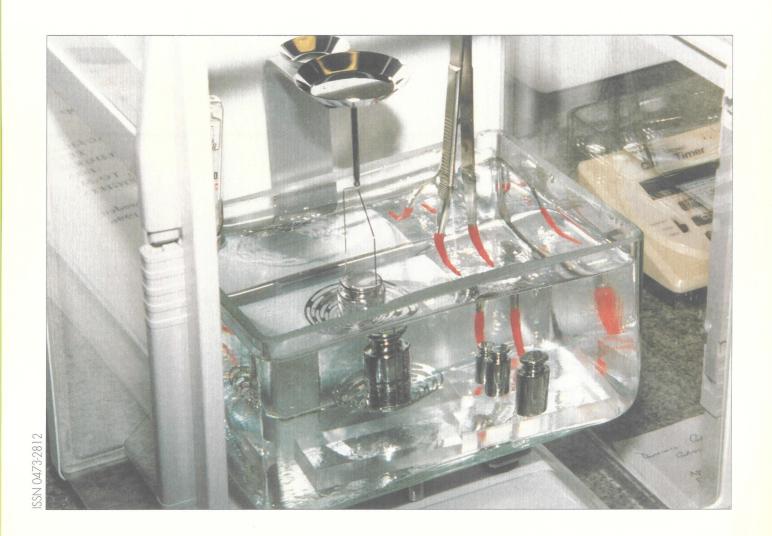


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## Organisation Internationale de Métrologie Légale

QUARTERLY JOURNAL



Test procedures being developed for OIML weights



THE OIML BULLETIN IS THE QUARTERLY JOURNAL OF THE ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE.

The Organisation Internationale de Métrologie Légale (OIML), established 12 October 1955, is an intergovernmental organization whose principal aim is to harmonize the regulations and metrological controls applied by the national metrology services of its

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Informations générales



# Editorial

### Accreditation and legal metrology

here is currently a huge development in activities connected with the accreditation of calibration and testing laboratories both at national level (laboratory accreditation systems have been established in a number of countries), at regional level (with recognition between countries in a particular region of the equivalence of their national accreditation systems, complemented by inter-regional agreements), and at international level (with ILAC being set up as a formal organization).

Another important aspect of this development is the revision of ISO/IEC Guide 25, which is currently underway; the Guide fixes a certain number of general rules which these laboratories should follow in order to satisfy the accreditation and certification (ISO 9000) requirements.

However, two types of metrological organisms exist for which the application of general accreditation principles poses a problem, namely primary laboratories and legal metrology services.

Primary laboratories maintain national measurement standards and ensure their compatibility with the corresponding standards of other countries, thereby acting as the starting point for all calibrations carried out nationally; therefore one may ask whether accreditation of primary laboratories is indeed possible. Another approach, currently under consideration within the *Convention du Mètre*, would aim at establishing agreements of recognition of the equivalence of national measurement standards and calibration certificates issued by these primary laboratories, on the basis of the results of intercomparisons of carefully selected standards.

In the field of legal metrology, other schools of thought are currently developing: firstly, national legal metrology services do not act merely as calibration and test laboratories (pattern evaluations, verifications, calibration in the context of regulations, etc.) but also as certifying (pattern approval decisions) and inspection organisms. So different accreditation and certification procedures can be applicable to them. Moreover, national legal metrology services are in fact invested by the public authority, which in the eyes of many means more than accreditation.

As a follow-up to the round table organized during the Tenth Conference and at the request of the CIML President, an OIML working group was formed to examine these questions, presided by Dr. Chappell, CIML Vice-President. It appeared that these discussions should not be confined to the single problem of accreditation, but should be broadened to encompass the more general and fundamental issue of confidence between countries as far as metrological controls are concerned.

It could transpire that accreditation is a useful tool - maybe even a necessary one - but undoubtedly insufficient to establish this confidence. Other actions, linked to the bior multi-lateral recognition of test results or to the development of the OIML Certificate System, can appear equally necessary.

It has also been noted that the Guide 25 should be complemented (in line with its Annex B) for specific application to legal metrology laboratories.

A report on this subject will be presented to the International Committee of Legal Metrology during its meeting in Rio in October this year, with a view to taking actions and decisions which could take effect in a short period of time, in order to adapt legal metrology even better to the demands of the next century.



#### CALIBRATION OF WEIGHTS

## Electromagnetic compensation comparator for heavy masses

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#### Introduction

The need for large standard masses for testing high capacity weighing instruments raises the problem of how to compare them with available certified weight standards. This paper illustrates a comparison method using a decimal weighing scale which is maintained in a state of equilibrium by means of an electromagnetic force compensation system.

# Description of the comparator and its static characteristics

The overall system is made up of a decimal weighing scale, which can be represented by a lever as illustrated in Fig. 1:

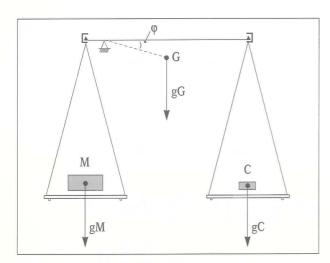


Fig. 1 Decimal weighing scale

The equilibrium of the system at no-load condition is given by:

$$M_0 \cdot a = G \cdot g \cdot \cos \phi + C_0 \cdot b \tag{1}$$

where  $M_0$  is the mass of the load receptor of the test specimen to be calibrated, G is the mass of the lever,  $C_0$  is the mass of the weight standard pan, a, b, g are respectively the length of the short arm, the long arm and the distance from the lever fulcrum to the mass center of the system, and  $\phi$  is the angle which g forms with the horizontal line.

With the test specimen M and the standard weight C loaded, the equilibrium condition gives:

$$(M + M0) \cdot a = G \cdot g \cdot \cos \varphi + (C + C0) \cdot b$$
 (2)

Subtracting (1) from (2) gives:

$$M \cdot a = C \cdot b \tag{3}$$

Equation (3) is the fundamental relation for determining the mass of the test specimen M. In order that this determination remains independent from the actual lengths of the short and long lever arms, it is necessary to compare the mass standard C with another mass standard  $C_r$ , which gives:

$$C \cdot a = C_r \cdot b \tag{4}$$

Combining (3) and (4) gives:

$$M = C^2/C_r \tag{5}$$

Equation (5) allows the M value determination accuracy to be linked to the weight standards accuracy. The propagation of the errors gives:

$$(\Delta M/M) = 2 \cdot (\Delta C/C) + (\Delta C_r/C_r)$$
(6)

In order to evaluate the overall sensitivity of the system, an infinitely small extra load dM is added onto the load pan; the system will rotate until a new equilibrium position  $\vartheta$  is reached, giving:

Since (2) still stands, the following can be written:

$$\begin{split} (M+M_0)\cdot a\cdot cos\vartheta + a\cdot dM\cdot cos\vartheta = & [G\cdot g\cdot cos\phi + (C+C_0)\cdot b]\cdot cos\vartheta \\ & + G\cdot g\cdot sin\vartheta\cdot sin\phi \end{split} \tag{7a}$$

Considering  $s = (\tan \theta/dM)$  as a measure of the system sensitivity, the following can be written for (7a):

$$s = \frac{a}{G \cdot g \cdot \sin \varphi} \tag{8}$$

Indicating Y as the vertical mass center distance from the lever fulcrum, the typical equation of the sensitivity for an infinite bending stiffness lever system can be written as:

$$s = \frac{a}{g \cdot Y} \tag{8a}$$

As already mentioned, (8a) stands on the hypothesis of the infinite bending stiffness of the lever. Thus it is necessary to properly design the cross-section of the lever, by considering the overall stress distribution on it. The maximum stressed cross-section is at the fulcrum of the lever and the bending torque at this location is:

$$M = M \cdot a = C \cdot b$$
 (9)

The expression of the strain per area unit is:

$$\sigma = \frac{M \cdot d}{I_{x}} \tag{10}$$

where M is the bending torque, d is the distance between the neutral axis and the extreme fiber of the lever, and  $I_x$  is the neutral axis based cross-sectional moment of inertia.

Indicating the ratio  $I_x/d$  by W, equation (10) can be rewritten as:

$$\sigma = \frac{M}{W} \tag{10a}$$

In order to apply equation (8a) for the sensitivity evaluation, the strain per area unit must be below a certain specified limit. A limit which can be adopted is 5 kg/mm<sup>2</sup>, as required by the Italian Legal Metrology Service [1] for the bending resistance evaluation of the weight transmission lever. The resistance modulus W can be evaluated by simply imposing the following:

#### $\sigma \leq 5 \text{ kg/mm}^2$

When  $\sigma$  is less than the above indicated limit, the lever can be assumed as showing an infinitely large bending stiffness.

# Dynamic behavior of the system without any equilibrating torque

The system's mathematical model, when the disturbing torque due to the difference of the torques referring to the two masses is acting, is nonlinear. By considering the difference dM from the nominal value of M, the motion of the lever is controlled by the following:

$$\begin{split} J \cdot d^2 \vartheta / dt^2 &= (M_0 + M + dM) \cdot a \cdot \cos\vartheta - G \cdot g \cdot \cos(\varphi - \vartheta) \\ &- (C_0 + C) \cdot b \cdot \cos\vartheta - B \cdot d\vartheta / dt \end{split} \tag{11}$$

In equation (11), B is the dynamic friction coefficient (dissipative effect of the kinetic energy), which can generally be taken as being proportional to the angular velocity of the lever, and J is the inertia moment of the lever calculated with the fulcrum F chosen as the rotation center. For (2) and by considering small oscillations of the lever, the following can be stated:

 $\sin\vartheta \rightarrow \vartheta$  and

 $\cos\vartheta \rightarrow 1$ 

The motion equation can be linearized as:

$$J \cdot d^2 \vartheta / dt^2 + B \cdot d\vartheta / dt + (G \cdot g \cdot \sin \varphi) \cdot \vartheta = a \cdot dM$$
 (12)

Equation (12) can be written as:

$$d^{2}\vartheta/dt^{2} + (B/J)\cdot d\vartheta/dt + (1/J)\cdot (G\cdot g\cdot \sin\varphi)\cdot \vartheta = a\cdot dM/J \quad (12a)$$

indicated by:

$$\omega_n^2 = G \cdot g \cdot \sin \phi / J \tag{12b}$$

which is the square of the natural pulsation, and by  $\delta\omega_n$ , with:

$$\delta = \frac{B}{\sqrt{J \cdot G \cdot g \cdot \sin \phi}}$$
 (12c)

which is the damping factor of the system. Equation (12) can be rewritten as:

$$d^{2}\vartheta/dt^{2} + \delta\omega_{n}\cdot d\vartheta/dt + \omega_{n}^{2}\cdot\vartheta = a\cdot dM/J$$
 (12d)

The evolution of the system is given for common values of d (less than unity) by the following:

$$\begin{split} \vartheta(t) &= \frac{a \cdot dM}{G \cdot g \cdot \sin \phi} \cdot \left[ 1 - \frac{e^{-\delta \omega_n t}}{\sqrt{1 - \delta^2}} \sin \left( \omega_n \cdot t \cdot \sqrt{1 - \delta^2} \right. \right. \\ &\left. + tan^{-1} \, \frac{\sqrt{1 - \delta^2}}{\delta} \, \right) \right] \end{split} \tag{13}$$

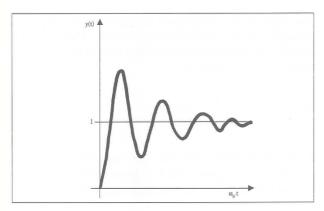


Fig. 2 Normalized evolution of the system

The normalized evolution is indicated by y(t):

$$y(t) = \vartheta(t) \cdot \frac{G \cdot g \cdot \sin \varphi}{a \cdot dM}$$
 (13a)

y(t) can be plotted versus the normalized time  $\omega_n$  t and for several values of  $\delta$  as in Fig. 2 (from reference [2] chapter 2.4).

# Dynamic behavior of the system in the presence of an equilibrating torque

In the presence of an equilibrating torque  $\tau$ , as in Fig. 3, the motion equation which controls the system in the above quoted hypothesis of "small oscillations" is:

$$J \cdot d^2 \vartheta / dt^2 + B \cdot d\vartheta / dt + (G \cdot g \cdot \sin \varphi) \cdot \vartheta = a \cdot dM + \tau$$
 (14)

Equation (14) can be rewritten as:

$$d^{2}\vartheta/dt^{2} + \delta\omega_{n}\cdot d\vartheta/dt + \omega_{n}^{2}\cdot\vartheta = (a\cdot dM + \tau)/J$$
 (14a)

The function is indicated by  $G_1(s)$ :

$$G_1(s) = [s^2 + \delta\omega_n s + \omega_n^2]^{-1}$$
(15)

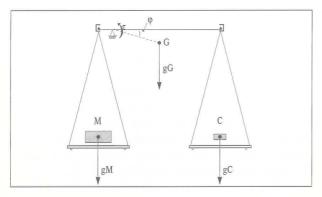


Fig. 3 The system with an equilibrating torque

and the following relation holds:

$$\Theta(s) = G_1(s) \cdot L[(a \cdot dM + \tau)/J]$$

where L is the correspondence of Laplace's transformation.

$$\vartheta(t) \to L \to \Theta(s) = \lim\nolimits_{t \to \infty} \! \int \vartheta(t) \cdot e^{-st} \cdot dt$$

Indicated by  $T_p(s)$  and T(s) respectively, the L-transformation of the disturbing and equilibrating terms  $t_p(t) = a \ dM \ /J \ and \ \tau/J$ , equation (14a) suggests the realization of the feedback control scheme as indicated in Fig. 4:

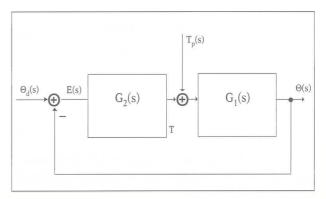


Fig. 4 Feedback control scheme

In such a control scheme the equilibrium position of the lever is:

$$\Theta_{d}(s) = 0 \tag{16}$$

The  $G_2(s)$  block has to be designed in such a way as to allow the system - in the presence of the equilibrating torque  $\tau$  - to reach, at steady state conditions, the equilibrium condition (16): in such a condition, the strength of the equilibrating torque will be proportional to the disturbance term  $t_p = a \cdot dM/J$ . From the linear feedback systems theory ([2] chapter 4), it can be seen that in order to achieve horizontal equilibrium of the lever, the  $G_2(s)$  block must be an "origin pole type" block, i.e.:

$$G_2(s) = K/s \tag{17}$$

In the time domain, the  $G_2$  block is an integrator block which is to be inserted in the open loop function describing the overall system. With the condition  $\Theta_d(s)=0$ , the L-transformation of the error function  $E(s)=[\Theta_d(s)-\Theta(s)]$  takes the form:

$$E(s) = -\Theta(s) \tag{18}$$

From the block diagram in Fig. 4, the following can be derived:

$$\Theta(s) = \frac{G_1(s)}{1 + G_1(s) \cdot G_2(s)} \cdot T_p(s)$$

$$= \frac{G_1(s)}{1 + G_1(s) \cdot G_2(s)} \cdot \frac{a \cdot dM}{J \cdot s}$$

From the theory of the L-transformation (final value theorem), the following can be deduced:

$$e_{\infty} = \lim_{s \to 0} s \cdot E(s)$$

$$= -\lim_{s \to 0} s \cdot \frac{G_1(s)}{1 + G_1(s) \cdot G_2(s)} \cdot \frac{a \cdot dM}{J \cdot s}$$
 (19)

The limit in (19) is:

$$e_{\infty} = 0 \tag{20}$$

because the block  $G_2$  tends towards  $\infty$ . (20) is the steady state equilibrium condition and the L-transformation of the equilibrating term is:

$$T(s) = G_2(s) \cdot E(s)$$

$$= -\frac{G_1(s) \cdot G_2(s)}{1 + G_1(s) \cdot G_2(s)} \cdot \frac{a \cdot dM}{J \cdot s}$$
 (21)

From (21), it can be deduced that the steady state value of the equilibrating term is:

$$t_{\infty} = \lim_{s \to 0} s \cdot T(s)$$

$$=-\lim_{s\to 0} s \cdot \frac{G_1(s) \cdot G_2(s)}{1 + G_1(s) \cdot G_2(s)} \cdot \frac{a \cdot dM}{J \cdot s}$$
 (22)

Equation (22) ensures that in steady state conditions the equilibrating torque is a measurement of the disturbing torque. The equilibrating torque is produced by an electrodynamic system: the current flowing through it is proportional to the torque strength; for these reasons (at steady state conditions) it can be written as follows:

$$t_{m} = h \cdot I = -a \cdot dM/J \tag{23}$$

where h is an appropriate constant which depends on the electrodynamic system producing the equilibrating torque and I is the steady-state current flowing in the electrodynamic system. Equation (23) is the reason for this because the steady-state current can be used as a measure of the equilibrating torque necessary to achieve the horizontal equilibrium condition. The open loop transfer function is:

$$F(s) = \frac{k}{s} \cdot \frac{1}{s^2 + \delta \omega_n s + \omega_n^2}$$
 (24)

Since in most cases the two non-zero poles are complex and conjugate, the root locus for the controlled feedback system is as the one indicated in Fig. 5.

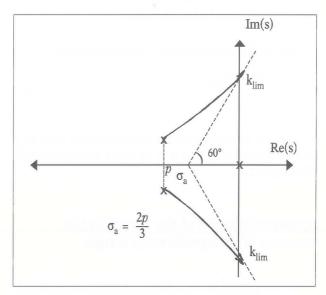


Fig. 5 Feedback system root locus

As the value of k increases, the poles move along the path plotted in Fig. 5. The value of k at which the upper and lower branches intercept the Im-axis (indicated as  $k_{\rm lim}$ ) is the one at which the system begins to show unstable behavior. The system shows greater or lower oscillations according to the longer or shorter distance of the root loci point from the Re-axis. With an appropriate choice of the k value, it is therefore possible to select the desired dynamic characteristics in terms of overshooting, settle time, etc. of the system, in order to obtain a good estimation of the system's overall behavior best suited for the mass comparison process.

In equation (23) (the basis for the measuring process) the direct component of the current appears: in order to obtain a suitable system of regulation, the current I can be seen as the DC component of a pulse width modulated (PWM) signal. Such a modulation allows the analog-digital conversion of the equilibrating current value to be put into practice with good characteristics of linearity, sensitivity and velocity [3].

In Fig. 4, the system of A/D conversion has a block structure as indicated in Fig. 6.

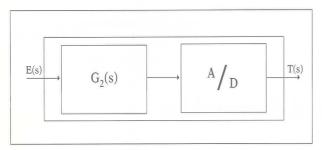


Fig. 6 Block diagram of the regulator and conversion system

The photodiode transducer on the feedback line transforms the angular displacement of the lever into an electric signal. The block diagram of the system which elaborates it is illustrated in Fig. 7.

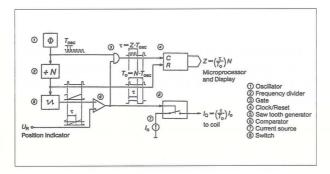


Fig. 7 Conversion block (Courtesy of Mettler-Toledo AG, Switzerland)

The slope a of the sawtooth signal is:

 $a = (I_0/T)$ 

in which  $I_0$  is the maximum current at the output and T is the period of the sawtooth signal. The above mentioned condition is such that the equilibrating signal is reproduced as the direct component of the PWM block output.

#### References

- [1] Circolare Ministeriale 5 ottobre 1937 Ministero Industria e Commercio: "Norme relative alle sollecitazioni unitarie massime nelle stadere a ponte in bilico in cassa metallica" Appendice alle istruzioni tecniche relative al Regolamento di Fabbricazione degli Strumenti per pesare e misurare
- [2] Giovanni Marro "Controlli Automatici" Edizioni Zanichelli , Bologna (3rd Edition - 1987)
- [3] K. Lang "Transducers for Weighing Instruments using Electromagnetic Force Compensation" OIML Bulletin N° 90, March 1983.

#### WEIGHTS

■ PRACTICAL TEST PROCEDURES

FOR CLASSES E<sub>1</sub> TO M<sub>3</sub> WEIGHTS

2 - 4-OCTOBER 1996 - BORÅS, SWEDEN

## Testing of weights: Part 2 – Density determination and the checklist

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#### 1 Introduction

OIML R 111 sets out the requirements for the determination of the density of weights used for different purposes [1]. By limiting the buoyancy effect to a second order contribution, measurement uncertainty in calibration can be improved, and so laboratories performing mass calibrations will have to certify that the density of test weights is within certain stipulated limits, depending on the size of the weights and the weight class in question.

In very accurate mass calibration, it is often not sufficient merely to state that a weight fulfills the requirements - in fact its actual density value must be known.

The density  $\rho_t$  of a test weight can be established in different ways, depending on whether a rough estimate, a simple mechanical measurement or an accurate determination is required. If the mass of the test weight  $m_t$  is already known, its volume and thus its density can be calculated by geometric means. Depending on weight size and accuracy requirements, different forms of hydrostatic weighing can be utilized both for verification and determination. Suitable recommended test methods based on OIML R 111 are presented in greater detail in the Draft on test methods  $[2]^*$ .

When developing the Test report format for OIML R 111, it turned out that not all the requirements in this Recommendation could be tested by instruments, e.g. markings and presentation. These and all the other

requirements in R 111 are detailed in the Nordic Group's draft as the authors also believe that some of the requirements in R 111 were diffuse and need to be clarified.

#### 2 Density determination

#### 2.1 The method of alloy specification

If the supplier specifies the alloy of the weight, then the density can be roughly estimated, assuming a reasonable average value and a certain range. Table 1 lists those alloys most commonly used for weights; the intervals given represent the expanded uncertainty of each value, calculated with a coverage factor k = 2.

Table 1 Most commonly used alloys

Alloy/material	Mean density	Interval	
platinum	21 400 kg/m <sup>3</sup>	± 130 kg/m <sup>3</sup>	
brass	$8  400  \text{kg/m}^3$	$\pm$ 300 kg/m <sup>3</sup>	
stainless steel	$7 920 \text{ kg/m}^3$	$\pm 130 \text{ kg/m}^3$	
iron	$7.850 \text{ kg/m}^3$	$\pm 400 \text{ kg/m}^3$	
carbon steel	$7~700~kg/m^3$	$\pm 400 \text{ kg/m}^3$	
cast iron (white)	$7~700~kg/m^3$	$\pm$ 600 kg/m <sup>3</sup>	
cast iron (gray)	$7\ 100\ kg/m^3$	$\pm$ 600 kg/m <sup>3</sup>	
aluminum	$2~700~kg/m^3$	$\pm 200 \text{ kg/m}^3$	
nickel-silver	$8 700 \text{ kg/m}^3$	$\pm 300 \text{ kg/m}^3$	

<sup>\*</sup>Editor's note: The test procedures and test report format for OIML R 111 is currently at Committee Draft stage (TC 9/SC 3) - Secretariat: USA.

#### 2.2 The geometric method

The volume of OIML-shaped weights can be made up of 3 or 4 parts and their determination comprises the measurement of the total height, the height of the knob, the diameter at three heights (knob, ring and cylinder) and the radii at the ring and cylinder. The formulae for the partial volumes are given in [2] and the measurement can be made with a simple vernier caliper. The applicability of the geometric method is limited to larger weight sizes, i.e. > 20 g depending on the accuracy required. Other more complicated shapes may be treated in a corresponding way [3]. The calculation is straight forward, though elaborate if performed manually; however the risk of scratching a test weight is a serious disadvantage of the method.

#### 2.3 Hydrostatic methods

Hydrostatic determination is based on the Archimedes principle and is used to measure the volume of liquid displaced when the weight is immersed. To achieve this, the amount of liquid must be weighed and its density  $\rho_1$  must be sufficiently well determined. Various techniques are used to perform hydrostatic weighings in a practical way, four of which are described below.

#### 2.3.1 Weighing the bath

A bath large enough to contain both the liquid and the weight is placed on the balance pan. The weight is attached to a suspension wire and is immersed in the liquid, but does not directly form a load on the balance.

The difference in the reading  $I_{\rm dl}$  with and without the immersed weight indicates the mass (volume) change caused by the liquid displaced. For a more accurate determination, the displacement of the weight holder and suspension wire together with evaporation of liquid off the surface should be taken into consideration. If the mass of the test weight  $m_{\rm t}$  or its conventional value  $I_{\rm t}$  is determined from a separate weighing and from the air and liquid densities (respectively  $\rho_{\rm a}$  and  $\rho_{\rm l}$ ) then the material density  $\rho_{\rm t}$  is calculated from equations (1) or (2) respectively:

$$\rho_{t} = \frac{\left(1 - \frac{\rho_{a}}{\rho_{l}}\right) \cdot m_{t} \cdot \rho_{l}}{\left(1 - \frac{\rho_{a}}{\rho_{ref}}\right) \cdot I_{dl}}$$
(1)

$$\rho_{t} = \rho_{a} + (\rho_{l} - \rho_{a}) \frac{I_{ta}}{I_{tl}}$$
(2)

where:  $\bullet$   $\rho_{ref}$  denotes the density of 8000 kg/m<sup>3</sup> for the reference weights used for the calibration of the balance in question;

- $I_{ta}$  is the indicated value when weighing the test weight in air;
- $I_{\rm dl}$  is the difference in balance indications corresponding to the liquid displaced.

#### 2.3.2 Weighing the weight

For under-pan weighing, the most suitable method is the direct weighing of the weight in the liquid suspended from a wire and a holder. In commercially available density kits the same idea is realized using a bearing arrangement as shown in Figs. 1 and 2.

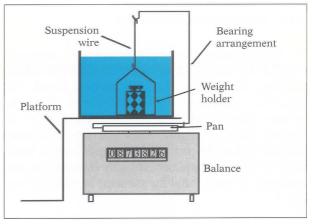


Fig. 1 Hydrostatic weighing: the weight is placed on a holder and is suspended by a wire. The water bath is placed on a suitable platform.



Fig. 2 Actual hydrostatic weighing operation in the density laboratory during the Borås Workshop, 2–4 October 1996 (see OIML Bulletin no. 1 1997, page 45).

Using the balance indication  $I_{\rm tl}$  the density  $r_{\rm t}$  of the test weight can be calculated from equation (3):

$$\rho_{t} = \frac{\rho_{l} \cdot m_{t}}{m_{t} - I_{tl} \left( 1 - \frac{\rho_{a}}{\rho_{ref}} \right)}$$
(3)

The procedure is best carried out by storing the weight in the bath and taring the balance with the holder before the weight is immersed. Depending on weight size and handling, the uncertainty of this method will be within  $5-200 \text{ kg/m}^3$  (mg weights).

Given the nominal mass of a verified weight and its class, the balance reading  $I_{\rm tl}$  alone contains sufficient information to decide whether or not the weight is within stipulated density limits. The upper and lower limits of the balance reading are tabulated for classes  $E_1$  to  $F_1$  and standard weights of 20 mg to 50 kg with a validity for a water temperature between 18 °C and 24 °C [2].

#### 2.3.3 Comparison in air and water

For best accuracy, a comparison technique is recommended that uses standards of known mass and volume (density) for weighing in both air and liquid. The equations governing the two situations allow the volume (density) of the unknown weight to be extracted. This technique requires a fine laboratory balance with under-pan weighing capabilities, a liquid bath of stable temperature and a mechanism that can exchange the weight and the standard in water [4] - see Fig. 3.



Fig. 3 Sophisticated equipment for highest accuracy density determination

It is however also possible to compare the immersed weight with suitable standards in air, which leads to a slightly different equation. This technique is capable of determining densities with an expanded uncertainty of 0.05 to  $1~{\rm kg/m^3}$  depending on the size of the weights and how carefully they are handled.

#### 2.3.4 The pycnometer method

Larger weights (> 1 kg) are difficult to handle during hydrostatic weighing. An alternative approach, which was developed within the Task Force Group [5], is to determine their volume by weighing the liquid that they displace in an indirect way. A convenient setup to accomplish this was found to be to use a vessel of constant adjustable volume which is weighed twice, firstly containing both the weight and the liquid and secondly containing only the liquid - see Figs. 4 and 5.



Fig. 4 The pycnometer, containing distilled water and the test weight, is weighed on a high accuracy nonautomatic weighing instrument.

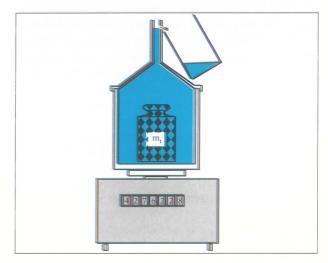


Fig. 5 Pycnometer setup with a vessel containing a liquid and the weight

If the liquid in both cases is adjusted to the same volume and has the same temperature, and if its density and the mass of the weight  $m_{\rm t}$  is known, then the difference in the balance reading  $I_{\rm l+t}$  –  $I_{\rm l}$  can be used to calculate the weight density:

$$\rho_{t} = \frac{\rho_{l} \cdot m_{t}}{m_{t} - (I_{l+t} - I_{l}) (1 - \frac{\rho_{a}}{\rho_{ref}})}$$
(4)

For density verification, the balance reading  $I_{\rm l+t}$  with the liquid and the weight itself provides enough information. If the mass and volume of the vessel and the liquid density are reasonably constant and if the weight falls into a certain class, then again low and high balance indication limits can be listed corresponding to certain stipulated density (volume) limits.

With a large filling factor, good water-tightness of the sealing and a stable and predetermined water temperature, one can reach an uncertainty of  $< 5 \text{ kg/m}^3$ . This model can easily be improved and the uncertainty reduced by taking into account the expansion of the water due to small, unavoidable temperature changes, which must be measured carefully.

#### 2.4 Immersion liquid

In this context it is recommended to use distilled and de-aerated water as an immersion liquid; this is easy to prepare and to check for purity [6]. Due to the liquid's high surface tension, an extra force comes into play, which arises due to a meniscus formation around the suspension wire. This effect can, however, be overcome or drastically reduced by using thin wires, a special preparation [7] and a comparison technique.

To reach an acceptable level of uncertainty, it is essential in all methods which use displaced liquid to avoid air bubbles on the weight or liquid compartment, if this is weighed. A suitable method to remove air is to apply an under pressure to the compartment containing the water and weight. A further critical point is the water density, which is found from the temperature by using one of the published water density tables [8].

#### 3 Checklist

#### 3.1 Introduction

As in all OIML Recommendations, the checklist in the draft R 111-2 covers the different requirements clause

by clause. Using this list in conjunction with other tests, the observer may obtain a clear overview as to whether or not the weight satisfies the requirements of OIML R 111 and may determine if it fulfills the class requirements as specified by the manufacturer.

The authors have also developed an annex for classifying old weights, which describes what factors shall be taken into consideration when classifying weights manufactured before R 111 was introduced.

The checklist includes clauses dealing with mpe, construction, material, surface conditions, adjustment, marking and presentation, based on R 111. For each part of the checklist, the authors' remarks are summarized in 3.2–3.5 below.

#### 3.2 General requirements, all classes

Whilst most of the clauses are clear, in subclause 4.3.2 which deals with the shape of weights from 1 g to 50 kg, a remark is made about classifying old weights, since many of these do not fulfill this subclause.

A remark was also made about the corrosion-resistance of weights: if the weight is not made of a material mentioned in R 111, then the manufacturer has to provide information about its resistance to corrosion.

#### 3.3 Requirements for class E weights

Some remarks have been made here too in order to avoid misunderstandings. In the second paragraph of subclause 6.2, the requirement is that the hardness shall be similar to or better than that of austentic stainless steel. It has been necessary to find practical solutions to meet this requirement, and the decision as to whether it is in fact met can be based on information provided by the manufacturer, or measured on a test specimen made from the same alloy as that of the weight. In addition, the resistance to wear shall be similar to or better than that of austentic steel. This is only applicable when the weight is made of another material. If the hardness requirement is satisfied, it is assumed that the requirement of wear is also satisfied.

For the requirements on marking (clause 10) and presentation (clause 11) a remark was made about classifying old weights; the observer is recommended to refer to the Test report format (*see previous Editor's note*).

#### 3.4 Requirements for class F weights

Remarks are made concerning subclause 6.3 (material), clause 10 (marking) and 11 (presentation). When determining that the hardness requirement in subclause 6.3

shall be at least equal to that of drawn brass (the brittleness of drawn brass is normally 28–100 J - Charpy impact test [9]), the observer must again use the information provided by the manufacturer or measure a test specimen made from the same alloy as the weights. In clauses 10 and 11 when classifying old weights, the Test report format describes how to evaluate such weights.

#### 3.5 Requirements for class M weights

The requirement in subclause 6.4.3 (material for  $M_1$  weights) states that weights of  $\leq 1$  g shall be sufficiently resistant to corrosion and oxidization, and subclause 6.5.1 (rectangular weights of 5–50 kg), requires gray cast iron to be used, or another material whose quality is similar to or better than that of gray cast iron. The material shall therefore not be brittle and shall be resistant to corrosion.

In subclause 6.5.2, the material requirements for cylindrical weights of  $\leq$  10 kg are described. The weight shall be made of a material which has a hardness and corrosion resistance at least equal to that of cast brass, and a brittleness not exceeding that of gray cast iron. This can again be determined based on information provided by the manufacturer or measured on a test specimen made from the same alloy as the weights.

In subclause 8.2.2 (surface conditions) it is stated that rectangular weights of 5–50 kg may be finished by appropriate painting. However, the authors do not recommend painting the bottom of the weight, since this will rapidly be eroded and scratched, causing dirt to collect and the mass to vary.

#### 4 Conclusion

After considering various methods used to determine the density of weights, some of the problems faced when developing the checklist have been evoked and as a result of this work, the authors feel that the present R 111 should now be revised in the light of work carried out by TC 9/SC 3 to draft the test procedures and test report format.

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The last in this series of three papers, Magnetism and Convection, will be published in the October 1997 issue of the OIML Bulletin.



#### ANALYTICAL MEASUREMENTS

# The role of certified reference materials in the Romanian traceability scheme

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#### Abstract

Traceability to the International System (SI) is increasingly demanded for measurements in chemistry. Reference materials have been widely used as measurement standards in the fields of industrial production, environmental protection and clinical chemistry, and have played an important role in ensuring the comparability of measurement results. This paper presents some aspects, practices and examples of the activity of the Reference Materials Laboratory of the National Institute of Metrology, Bucharest, in the field of spectrometric measurements regarding the role and use of reliable and suitable certified reference materials to ensure the uniformity and traceability of analytical measurements.

#### 1 Introduction

International comparability and traceability of measurements to stable references are required in measurements for environmental monitoring and protection, international trade, clinical practice, health and safety and industrial production. Measurements performed by any analytical laboratory should therefore yield reliable, traceable and comparable results. There is a growing nationwide demand to perform spectrochemical analyses in a manner that allows inter-laboratory comparisons of the results, and which creates an increasing awareness among analysts of the need to produce accurate results. In addition, an important prerequisite for international comparability and uniformity of spectrometric measurements which would provide mutual recognition of the results is traceability to specific standards, called Certified Reference Materials (CRMs) [1].

CRMs are used to evaluate the uncertainty of measurement results, to calibrate and verify measuring

instruments, to evaluate methods of measurement and to determine by comparative methods the quantities characterizing the composition or properties of substances and materials.

# 2 Some aspects of the traceability of analytical measurements and of certified reference materials

In metrology there are three fields: fundamental, industrial and legal metrology; each relies on accurate measurements, requiring the realization, maintenance and dissemination of the units (as defined on the basis of the SI) to be taken into consideration. At national and international levels, this encourages mutual recognition of the calibration certificates issued by various laboratories. The general traceability scheme for physical quantities is shown in Fig. 1, which illustrates an example of a physical measurand A correctly disseminated by the national metrological organization in one accredited laboratory (1) and incorrectly in a second laboratory (2). In order to ensure traceability, during an inter-laboratory comparison the difference  $\Delta A$  is established, its cause is identified, corrective measures are taken and the performance of the accredited calibration laboratories is tested. Thus vertical traceability (which is the ideal situation) should be complemented by horizontal traceability, which is achieved by inter-laboratory comparisons [2].

Traceability for physical quantities is relatively easy to establish, but in chemistry (as in any other field where physical quantities are measured) all measurements must be closely linked to the SI system of units using clear dissemination schemes that allow traceability to national and international standards. In chemical measurements many problems still exist due

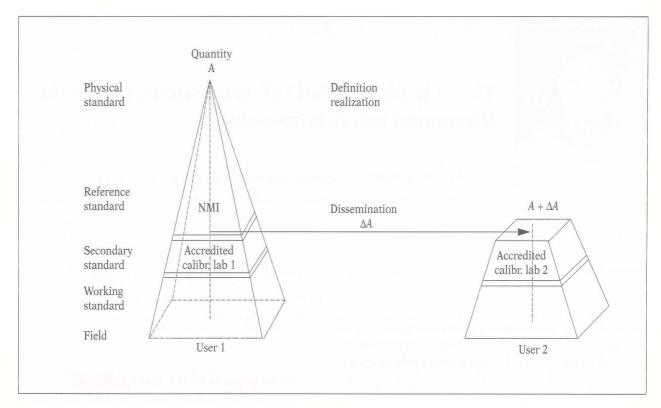


Fig. 1 The general traceability scheme

to the particular definitions and meanings of the units used, the diversity of the substances involved, the matrix effects, the standards employed (CRMs) and the assessment of measurement uncertainty.

There are several possibilities to provide traceability of chemical measurements to SI units [3], but the author favors the use of CRMs, which are defined as a special metrological category, distinct from any other standard or measuring instrument. From a metrological point of view, the CRM should be considered as a primary, secondary or working standard similar to that of a fundamental unit (e.g. the kilogram in the case of mass measurements), but with some "special" differences. For example, the chemical properties of the material used for a certain CRM are usually irreversibly altered during the measurement process and it is therefore necessary to take certain precautions whenever the CRM is used. Thus quite often the latter is divided into batches and organized into sets with specified homogeneity and stability.

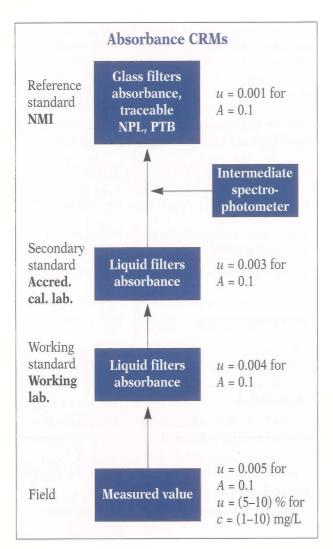
Some reference materials are themselves traceable to SI units and thus open up the possibility of creating a complete traceability chain from the field laboratory to SI units. To ensure national uniformity and accuracy of measurements, the author's Institute has developed references which may be used either as reference measurements or as CRMs. The certified reference materials used in optical measurements (molecular

absorption, wavelength, photometric linearity and stray radiant energy) and in the composition of chemicals and high purity substances - often called primary or spectrometric reference materials, shown in Table 1 - play a major part in chemical measurements performed in Romania. Establishing the traceability of such reference materials is an essential part of the certification process, achieved using the so-called metrological approach [4, 5].

Some aspects of the traceability of CRMs and the proposed categories for reference materials in terms of material composition and degree of traceability to the SI have already been described [6] and an attempt at vertical traceability of absorbance CRMs and CRMs for chemical composition is shown in Fig. 2.

Carrying out certification of reference materials in this way enables these to be used reliably in the statement of traceabilty and comparability of analytical measurements. The reference materials constitute the binding role between the traces and levels of traceability, and act as validators of the instrument and/or of the method used.

The traceability of certified values of reference materials is essentially the same as that of spectrochemical measurements - the realization of both depends on establishing a traceability system of analytical measurements. This paper highlights some uncertainty aspects of spectro(photo)metric results.



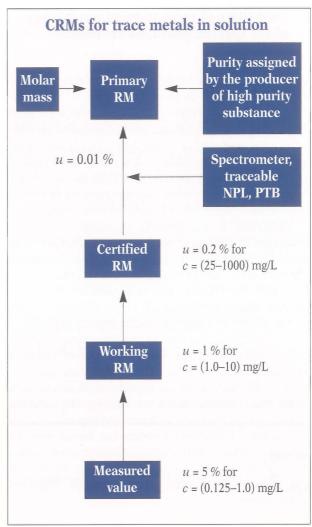


Fig. 2 Attempt at vertical traceability of absorbance and spectrometric reference materials

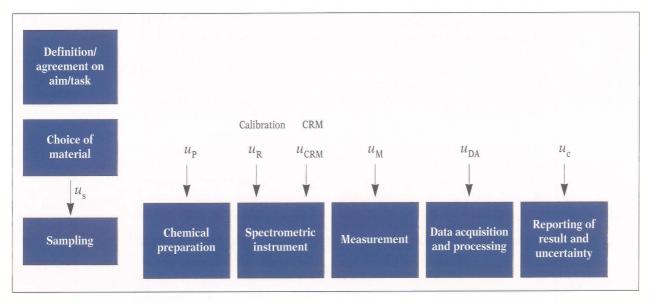


Fig. 3 The analytical spectrometric measurement system

#### **Evaluation of measurement uncertainty** of the spectro(photo)metric results

Among others, spectro(photo)metric methods are extensively utilized in our laboratories. Since ultraviolet-visible molecular spectro(photo)metry (MS), atomic absorption (AAS) and atomic emission spectrometry (ICP) are very common and versatile analytical techniques, it is important to determine the technical specification in order to choose the right instrument for the individual application and to maintain optimum spectrometer performance in daily routine analysis.

Validation is the process by which a sample, measurement method or piece of data is deemed to be useful for a specified purpose; in the case of spectro-(photo)meters, validation signifies the technical procedures and the reference materials used for evaluating the instrumental performances.

The quality of analytical results directly depends on the accuracy of absorbance measurements and on various other factors, the most important of which is the instrumentation. As far as the photometric performance of the instrument is concerned in ultravioletvisible spectro(photo)metry, it is an essential criterion that the spectral data are accurate and reproducible. In this respect, the author's Institute has issued national legal and technical metrological norms covering ultraviolet-visible molecular absorption, atomic absorption and inductively coupled plasma atomic emission spectrometers.

As the traceability of spectrometric measurements to SI standards (CRMs) is an important prerequisite for national/international comparability and uniformity of measurements, some aspects associated with the evaluation of measurement uncertainty of the spectro-(photo)metric results are also pointed out, to aid recognition of measurement results. Uncertainty and traceability are two concepts which are inherently linked in any analytical system. The Guide [5] introduces a classification between Type A and Type B standard uncertainties and indicates the two different ways of evaluating the uncertainty components:

- type A uncertainty, which may be evaluated from the statistical analysis of series of observations, and
- type B uncertainty, which may be evaluated by other means, i.e. scientific judgment based on all the available information on the possible variability of measurement results.

An analytical spectro(photo)metric process [6] usually includes eight points, shown in Fig. 3. For each point, the associated standard uncertainties below need to be estimated:

- $\bullet$   $u_s$  for sampling, which includes uncertainty due to the chemical preparation  $u_p$ ;
- $\bullet$   $u_{\rm M}$  for reproducibility of the analytic spectro-(photo)metric system, which includes the dilution factor, the weight of the sample, etc.;
- $u_{CRM}$  for the value of the calibration standards;

Table 1

Code CRMs	<b>Destination of CRMs</b>	Description of CRMs
10.01 10.02 10.03 10.04	Verification and/or calibration of the wavelength scale of the UV/VIS/NIR spectrophotometers, uncertainties within the range of (0.3–2) nm	5 ml vial (holmium oxide solution) 5 ml vial (cobalt chloride solution) filter type BG35 5 ml vial (benzen)
11.01 11.02 11.03 11.04	Verification and/or calibration of the absorbance scale of the UV/VIS/NIR spectrophotometers, uncertainties within the range of (0.003–0.020) between 235 nm and 721 nm	2 vials of 5 ml (potassium dichromate) 3 vials of 5 ml (nickel nitrate) 3 vials of 5 ml (cobalt nitrate) 5 vials of 5 ml (cobalt + nickel nitrate)
15.01 15.02 15.03 15.04	Stray light evaluation within the spectral range of (200–650) nm	10 ml bottle (KCl) 10 ml bottle (NaI) 10 ml bottle NaNO <sub>2</sub> ) 10 ml bottle (methylene blue)
16.01 16.02	Photometric linearity check of the UV/VIS spectrophotometers, uncertainty of 1 %	5 vials of 5 ml (potassium dichromate) 5 vials of 5 ml (cobalt-ammonium sulfate)
12.01–12.12	Verification and/or calibration of the AA and ICP spectrometers, uncertainty 0.2 %	100 ml bottle in nitric acid 0.2 % (Ca, Cd, Cu, Cr, Fe, Na, Mg, Mn, Ni, K, Pb, Zn)
15.01–15.06	Evaluation methods of analysis of water and waste water and verification and/or calibration of the AA and ICP spectrometers	100 ml bottle in nitric acid 0.2 % • containing 7 elements at level mg/L

- $u_{\rm R}$  for reproducibility of the calibration, and
- $u_{\mathrm{DA}}$  for suitability of the method of calibration, which includes the data treatment.

Thus it is possible to identify the sources of errors and to determine the correction in each case. The expanded uncertainty of the result *U* for spectro-(photo)metric measurement system is given by:

$$U = k \left[ u_s^2 + u_M^2 + u_{CRM}^2 + u_R^2 + u_{DA}^2 \right]^{1/2}$$

where k is the coverage factor (chosen in agreement with the user).

It is essential that the spectro(photo)metric measurement system be in a state of "statistical control", i.e. that repeated measurements of standard samples processed right through the system be consistent with the measured variance over a certain period of time.

#### 4 Practical considerations and examples

A discussion of the diagnosed instrument performance, often included in  $u_{\rm M}$ , is presented to illustrate the nature of the problems encountered and the steps that may be taken to overcome them.

Table 2 summarizes the main sources of absorbance errors on the standard ultraviolet-visible molecular spectro(photo)meter and the proposed algorithm for evaluating the uncertainty when certifying a reference measurement or a reference material.

In most cases, the determination of concentration by ultraviolet-visible spectrometry is performed by different methods:

 a linear calibration curve following the Beer-Lambert law;

Table 2

Sources of errors	Components of uncertainty Type A Type B		Estimates of magnitude of known effects	
Repeatability of measurement, each measurement including a separate analytical sample preparation	$u_{\mathrm{A,A}}$		10 <sup>-3</sup> Abs	
Combined standard uncertainty Type A	$u_{A,A} = \sqrt{\frac{\sum_{i} (A_{ij} - 1)^{i}}{n(n - 1)^{i}}}$	$\overline{A_{ij}}$ ) $^2$		
Repeatability of correction factors determined n times separately for each of the known systematic errors:				
1st correction factor due to CRM		$u_{\mathrm{B,A,CRM}}$	$10^{-3}$ Abs	
2 <sup>nd</sup> correction factor due to wavelength offset		$u_{\rm B,A,\lambda}$	10 <sup>-4</sup> Abs	
$3^{\mathrm{rd}}$ correction factor due to solution temperature		$u_{\rm B,A,t}$	$10^{-4}$ Abs	
4 <sup>th</sup> correction factor due to cuvets path-length		$u_{\rm B,A,l}$	$10^{-4}$ Abs	
5 <sup>th</sup> correction factor due to stray radiation		$u_{\rm B,A,s}$	10 <sup>-4</sup> Abs	
$6^{\text{th}}$ correction factor due to photometric linearity		$u_{\rm B,A,L}$	10 <sup>-4</sup> Abs	
Combined standard uncertainty Type B	$u_{B,A} = \sqrt{u_{B,A,CRM}^2 + }$	$u_{B,A,\lambda}^2 + u_{B,A,t}^2 + u$	$u_{B,A,l}^2 + u_{B,A,s}^2 + u_{B,A,L}^2$	
Expanded standard uncertainty	$U = u(A) = k\sqrt{u_{A,A}^2}$	$\frac{1}{4 + u_{B,A}^2} \approx 10^{-3} Abs$	S	

Table 3

Method of determination	Mathematical function	Expression for standard uncertainty	Law of propagation of uncertainty
Linear calibration curve	$c = a + b \cdot A$	$u_c^2 = \left(\frac{\partial c}{\partial a}\right)^2 \cdot u^2(a) + \left(\frac{\partial c}{\partial b}\right)^2 \cdot u^2(b) + \left(\frac{\partial c}{\partial A}\right)^2 \cdot u^2(A)$	$u_c = \frac{S_0}{b} \cdot \sqrt{\frac{1}{N} + \frac{1}{n} + \frac{\left(\overline{A}_m - \overline{A}\right)^2}{b^2 \sum \left(c_i - \overline{c}\right)^2}}$
Factor method according to Beer's law	$c = F \cdot A$	$u_c^2 = \left(\frac{\partial c}{\partial F}\right)^2 \cdot u^2(F) + \left(\frac{\partial c}{\partial A}\right)^2 \cdot u^2(A)$	$u_c = \sqrt{A^2 \cdot u^2(F) + F^2 \cdot u^2(A)}$
Known molar absorptivity	$c = \frac{M}{\varepsilon} \cdot \frac{A}{l}$	$u_c^2 = \left(\frac{\partial c}{\partial A}\right)^2 \cdot u^2(A) + \left(\frac{\partial c}{\partial l}\right)^2 \cdot u^2(l)$	$u_c = \sqrt{\left(\frac{M}{\varepsilon \cdot l}\right)^2 \cdot u^2(A) + \left(\frac{M \cdot A}{\varepsilon \cdot l^2}\right)^2 \cdot u^2(l)}$

- the factor method according to Beer's law, or
- measurement of molar absorbency.

Table 3 gives the general mathematical functions describing:

- the determination of concentration;
- the expression for uncertainty with standard uncertainties and covariances associated with the input estimate, and
- the final expression for evaluating the uncertainty.

As shown in the table, the uncertainty due the linear calibration curve may be estimated against:

- the standard deviation of the linear calibration curve  $s_0$ ;
- the slope of the curve, b:
- the number *N* of CRMs used for calibration curve;
- the number *n* of replicates, and
- the average absorbance signals  $\overline{A}_{\rm m}$  of the sample and of the CRMs  $(\overline{A})$  using the calibration curve, and their corresponding values of the concentration.

The results are traceable to the standards used for the instrument's calibration. Therefore, in accordance with the Beer-Lambert law, the uncertainty of concentration depends on the uncertainty of absorbance measurement; this uncertainty must be evaluated.

A calibration curve is set up by measuring reference solutions. The measurements yield a curve of absorbance versus concentration, and the points between the data of the reference solutions are interpolated by fitting a suitable curve, which normally follows the Beer-Lambert law and which gives rise to a straight line through the origin of the coordinate system. Some examples are shown in Fig. 4 for different types of spectro(photo)meters:

- DR 2000-wide bandwidth 8 nm (series 1);
- Hewlett Packard 8452A bandwidth 2 nm (series 2), and
- Specord M 40-wide bandwidth 1 nm (series 3), for manganese concentration measurement (which with formaldoxime forms a water-soluble orange-red color complex (CH<sub>2</sub>NO)<sub>2</sub>Mn).

Even though linearity tests may be satisfactory for characterizing the spectro(photo)meter performance, in most cases, the curves show that the increase of the spectral bandwidth causes an apparent decrease in absorbance from the true absorbance. Therefore, the accuracy of spectro(photo)metric results is related both to the performance of the instrument and to the uncertainty due to the linear calibration curve (of the instrument).

The reference concentration value based on known molar absorptivity and on optical path length is currently ensured by assuming a conventional value. Ultraviolet visible molecular absorption spectro-(photo)meters are used as measurement standards and constitute a growing network of reference points for concentration measurements.

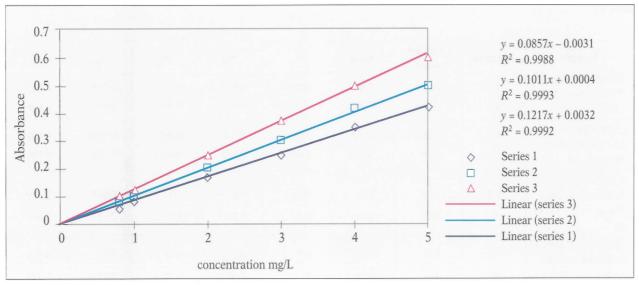


Fig. 4 The calibration curves for the concentration reference solutions of manganese

The molar absorptivity of the color complex was determined from absorbance measurements at 450 nm, by measuring the concentration of reference solutions of manganese using the method of the least squares ( $e_{450 \text{ nm}} = 10224 \text{ L/mol.cm} \pm 7 \%$ ).

Table 4 presents the evaluation of the measurement uncertainty associated with the spectro(photo)metric results using the genealogical approach. The uncertainty components are determined as follows:

- the uncertainties due to the sampling u<sub>s</sub>, the reproducibility of the system u<sub>M</sub> and the suitability of the method of calibration u<sub>DA</sub> are evaluated by the analysis of variance (ANOVA), in accordance with ISO standards [7, 8] or on the basis of professional judgment;
- the uncertainty of the certified reference materials
   u<sub>CRM</sub> used for calibration is stated according to the
   certificates of CRMs as presented above, provided
   that the traceability of the suitable CRMs is assured;
- the uncertainty due to the reproducibility of the calibration  $u_{\rm R}$  is evaluated as indicated in Table 3.

Figure 5 shows the percentage of the components corresponding to the variances as proportions of the overall variance (see Table 4), by atomic absorption using the Perkin Elmer 192 spectrometer (Block 1 in Fig. 5 for Ni-SRM NIST 14 g), by inductively coupled plasma atomic emission using the Beckman-Spectro-flame spectrometer (Block 2 for Cd- ICP Code 15474) and by ultraviolet-visible molecular absorption using the Specord M 40 spectrometer (Block 3 for Fe-CRM RNMI 12.06).

The percentage of the variance in the overall variance of CRMs is significant, but not essential: the percentage of the variance of the calibration repro-

ducibility is predominant and must be reduced in order to optimize the process. The percentages of the variances of sampling and suitability of the method of calibration (including the data processing) can be neglected. The degree to which each of these components contributes to the overall variability is obviously important when trying to reduce the process variability and improve the analytical process.

For evaluating the comparability of spectro(photo)-metric measurement results in Romania, interlaboratory comparisons were performed in 15 laboratories specializing in environmental analyses. The results of one of these comparisons are shown in Fig. 6: note that these correspond to 4 metallic components (Cd, Zn, Cu, Cr) in the concentration range of 0.125–0.5 mg/L from the reference materials Code 15.01/4 used in the program.

The results for Cd and Zn elements show a spread (as a % deviation from the certified value) within  $\pm 35$  %, and for Cu and Cr elements a spread within  $\pm 25$  %. The graphs indicate the best possible accuracy of the certified value which is obtainable and what is obtained in practice under routine conditions.

As shown above, all the elements tested are "outliers" on both the high and the low concentration sides. Neither method can be considered as being superior; the best method used can give wrong results if not correctly applied even in experienced laboratories.

It was noticed that the main reasons for the current lack of comparability at the working level also included insufficient awareness of uncertainties and sources of error, a lack of high quality reference materials and no recognized system for inter-comparison of traceable chemical measurements.

Table 4

Sample	Element	Certified value	$u_{\mathrm{CRM}}$	$u_{\rm s}$	$u_{ m M}$	$u_{\mathrm{R}}$	$u_{\mathrm{DA}}$	U
SRM-NIST 14 g (Carbon Steel AISI 1078), % wt	Ni Cu	0.030 0.043	0.003 0.003	0.008 0.005	0.010 0.012	0.007 0.008	0.005 0.003	0.015 0.016
CRM-MBH LA-80 (Law Alloy Steel), % wt	Cu Ni	0.290 3.98	0.005 0.025	0.003 0.005	0.020 0.086	0.003 0.0025	0.003 0.003	0.021 0.090
ICP Multielem. Solution Standard, Merck Code 15474, mg/L	Cd Zn	20.0	2.0 2.0	0.02 0.02	1.8 2.1	1.0	0.015 0.01	2.870 3.067
Multielem.Solution Standard, RNMI, CRM Code 15.01/4, mg/L	Cu Mn	8.0 4.0	0.25 0.20	0.02 0.02	0.35 0.30	0.16 0.10	0.02	0.459 0.370
Monoelem.Solution Standard, RNMI, CRM Code 12.05 and 12.06, mg/L	Pb Fe	3.0 5.0	0.15 0.20	0.02 0.02	0.30 0.20	0.15 0.10	0.010 0.015	0.368 0.301

For proper application of reference spectrometry methods and accurate metrological use, good guidance documents which detail the procedures to be used are essential (including when necessary details on sampling, chemical preparation, dilution techniques, etc.).

This metrological approach has recently been used in Romania to evaluate the spectrometric measurement uncertainty for the certification of several types of reference materials; the objective is to produce certified values for which the accuracy and the uncertainty are demonstrated on an experimental basis.

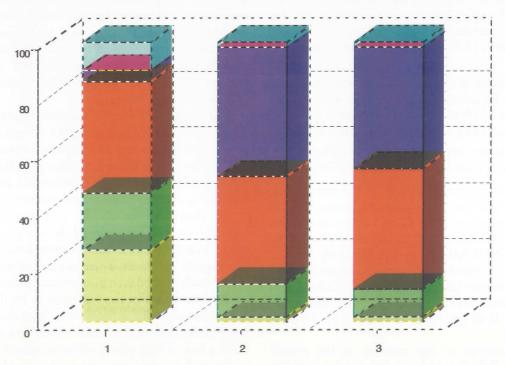
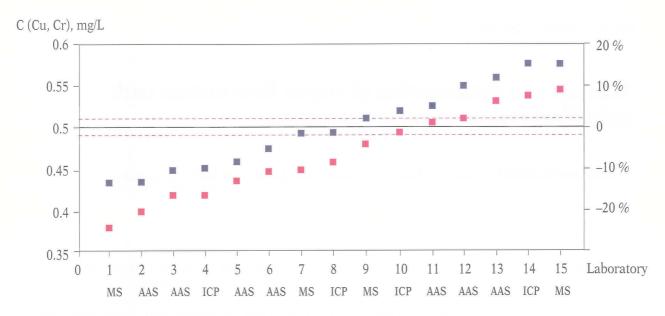


Fig. 5 Variance components of the spectro(photo)metric measurement system



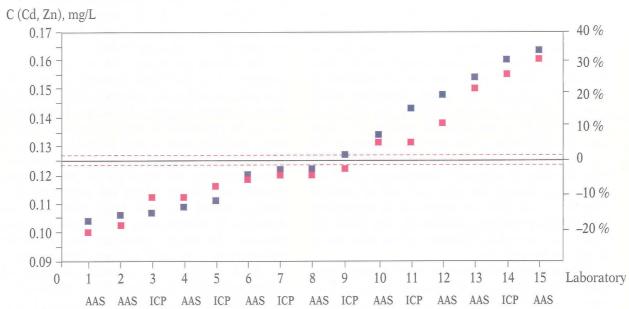


Fig. 6 Analytical data obtained during inter-laboratory comparison

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#### FLOW MEASUREMENT

# Analysis and optimization of vortex flow meters with acoustical clamp-on vortex detection

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#### Abstract

Vortex flow meters with acoustical clamp-on vortex detection can be described by a simple model of signal formation and signal analysis which is derived in this article. The relevant parameters of the system that are specific to the ultrasonic sensor principle are the phase fluctuation of the ultrasonic wave and the amplitude of the signal of interest. The phase fluctuation which increases with the growing width of the bluff body depends linearly on the Reynolds number. To a large extent it is independent of the inner pipe diameter, as well as of the axial distance between the sensors and the bluff body. The effect of ultrasonic disturbances which are propagating through the pipe wall and of multiple reflections in the fluid are suppressed by appropriate demodulation procedures. Additionally, they reduce the sensitivity to external disturbances (such as vibrations of the pipe) at the same time.

By an appropriate choice of the sensor parameters and adapted signal processing, it is possible to set up flow-meters with acoustical vortex detection with a dynamic range and accuracy comparable to conventional vortex meters. The clamp-on principle prevents direct contact between sensors and fluid and enables replacement of the sensors without direct intervention in the process.

#### 1 Introduction

Flow meters applying the vortex principle utilize a bluff body located in the fluid in order to produce vortex shedding, the frequency of which is assumed to be proportional to the volumetric flow within a certain range of the Reynolds number. Vortex meters, which in 1993 only occupied a 5.3 % share of the market, are expected to increase by about 12 % annually [1]; thus they belong to the group of flow gauges with the highest growth rate. Since they work independently of the electrical conductivity of the fluid, they supplement magnetic inductive flowmeters (which are restricted to conducting fluids).

The relevant parameters of a vortex meter are the geometry of the bluff body in combination with the flow area, as well as the principle which is applied to detect the vortex frequency. The shape of the bluff body has to be optimized in order to achieve stationary vortex shedding down to small Reynolds numbers. The distinctive measuring principles used to estimate the vortex frequency are based on the measurement of pressure or velocity, which are modulated by the vortices behind the bluff body. Acoustical vortex detection, if realized using the clamp-on technique, possesses the unique advantage that it enables a measurement to be made without direct contact with the fluid, which is an essential advantage when measuring the flow of corrosive fluids. Besides this, the simple and robust sensors can be exchanged without dismantling the meter, which avoids expensive interruptions of the process [2, 3].

#### 2 Measurement principle

The harmonically excited transmitter T emits an ultrasonic cw-signal into the fluid (Fig. 1). The vortices which are induced by the bluff body B within the streaming fluid lead to a phase modulation of the ultrasonic wave. With gaseous fluids an additional amplitude modulation occurs; this results from density gradients induced by the vortices leading to time and

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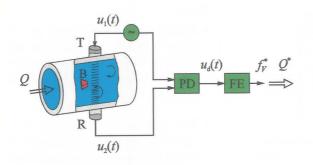


Fig. 1 Acoustical vortex detection applying ultrasound

space dependent diffraction effects as well as alternations of the absorption coefficient. Concerning liquids, which are considered in this article, this effect can be neglected due to their small compressibility.

The phase modulated signal detected by the receiver R is being coherently phase-demodulated (PD). Subsequently, the frequency of the phase modulation, which corresponds with the wanted vortex frequency  $f_v^*$ , is estimated (FE). With  $f_v^*$ , the known Strouhal number St, the width of the bluff body b and the flow area A the volumetric flow  $O^*$  can be determined:

$$Q^* = \frac{Ab}{St} f_v^* \tag{1}$$

The dimensionless Strouhal number *St* describes the influence of the shape of the bluff body and of the remaining flow area on the vortex frequency.

#### 2.1 Signal formation

Once the sensors are connected using the clamp-on technique, the transmitted wave, which in the following is described by the exciting signal  $u_1(t)$ , has to be emitted into the fluid passing through the pipe wall (see Fig. 2).

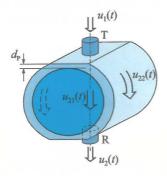


Fig. 2 Clamp-on principle and sound propagation

Due to the discontinuity of the impedances between pipe and fluid, a part of the ultrasonic signal propagates circumferentially within the pipe. Thus the received signal  $u_2(t)$  is built by the phase-modulated fluid component  $u_{21}(t)$  and the unmodulated pipe component  $u_{22}(t)$ :

$$u_1(t) = \overline{u}_1 \sin \omega_0 t \tag{2}$$

$$u_2(t) = \bar{u}_{21}\sin(\omega_0 t + \varphi_0 + \varphi_v(t)) + \bar{u}_{22}\sin(\omega_0 t + \varphi_2)$$
 (3)

 $\phi_0$  is the phase shift resulting from the acoustical path between transmitted and received waves; it depends on the distance between transmitter and receiver as well as on the sound velocities in the pipe wall  $c_{\rm pipe}$  and the fluid  $c_{\rm fluid}$ . With the assumption of a harmonical modulation the phase shift  $\phi_{\rm v}(t)$  which is caused by the vortices can be described as

$$\varphi_{v}(t) = \overline{\varphi} \sin \omega_{v} t \tag{4}$$

where  $\overline{\varphi}$  represents the amplitude of the phase modulation and the radian frequency of the vortex shedding  $\omega_v = 2\pi f_v$  is the required value.

With little expense, the ratio between the amplitudes of the signal of interest  $\bar{u}_{21}$  and the unwanted signal  $\bar{u}_{22}$  can be increased by acoustically matching the thickness  $d_{\text{pipe}}$  of the pipe wall between the transducers and the fluid to the acoustical wave length in the pipe  $\lambda_{\text{pipe}}$ . With an effective thickness of

$$d_{\rm p} \approx n \, \frac{\lambda_{\rm pipe}}{2} = n \, \frac{c_{\rm pipe}}{2f_0}, \ n \in N$$
 (5)

the pipe acts as an acoustical matching layer, resulting in an experimentally proved increase in the effective signal amplitude by a factor of 8–10 [4]. Since with common pipe materials and practically relevant ultrasonic frequencies ( $f_0 = 0.5-5$  MHz),  $d_{\rm p}$  takes minimal values (n=1) of 0.5–5 mm; this matching can easily be realized by slightly reducing the thickness of the pipe wall or by attaching additional thin matching layers.

The sound propagation, illustrated in Fig. 2, represents a rough simplification of the real situation. The limitation of the acoustical absorption in the pipe wall leads to a circulating wave propagation in both circumferencial directions, which partially results in standing waves. Multiple reflections within the fluid at the pipe wall lead to similar phenomena. In order to examine the effects of these complex superpositions on the measuring system, one can make use of their strong frequency sensitivity: after multiple reflections within the fluid and multiple propagations through the pipe wall respectively, the wave components are solely superimposed constructively with a discrete ratio between the length of the acoustical path and the

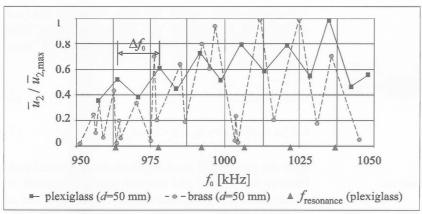


Fig. 3 Normalized received signal amplitude  $\bar{u}_2/\bar{u}_{2,\max}$  dependent on the transmitted frequency  $f_0$ 

wavelength. If the transmitted frequency  $f_0$  is varied, the magnitude of this effect changes and the same happens with the received signal amplitude (see Fig. 3). With Plexiglas pipes the difference in the acoustical impedances between fluid and pipe wall is relatively small ( $Z_0$ ,  $P_{\text{lexiglas}}/Z_0$ , water  $\approx 2.2$  with T = 20 °C). Thus a substantial part of the ultrasonic wave intensity propagates directly from the transmitter through the fluid to the receiver and the received signal amplitude contains a non-frequency-sensitive direct component on which is superimposed the frequency-dependent component resulting from the indirect propagation. The experimentally determined difference between the frequencies with maximum amplitudes corresponds with the theoretical differences  $\Delta f_0$  in consideration of the inner pipe diameter d and it is independent of the pipe material:

$$\Delta f_0 = \frac{c_{\text{fluid}}}{2d} \tag{6}$$

In contradiction to the assumptions published so far, which assume a dominant propagation of unwanted signals through the pipe wall, through this the high influence of the reflections within the fluid is verified. Due to the high difference of the impedances with metal pipes (e.g.  $Z_0$ ,  $_{\rm brass}/Z_0$ ,  $_{\rm water} \approx 24.7$  with T = 20 °C) the non-frequency-sensitive component is smaller.  $\bar{u}_2$  shows significant maxima with the differences in frequency as described by equation (6) but the periodicity is disturbed by the increasing component of the propagation through the pipe wall, which is additionally damped less in comparison to a Plexiglas pipe  $(\alpha_{\rm brass}/\alpha_{\rm Plexiglas} \approx 0.08).$ 

By applying the following realistic assumptions, the description of this complex wave propagation can be reduced to the signal model presented in Fig. 2.

Notes:

- Signals circulating once or several times within the pipe wall are not phase-modulated and therefore they can be described by the signal  $u_{22}(t)$ .
- The reflected component of a signal propagating in the fluid in a radial direction is being modulated once more while crossing the vortices. The technical relevant flow rates are very small in comparison to the speed of sound in the fluid and therefore the reflected component crosses approximately the

same vortices as the incident wave. As a result, the amplitudes of two successive phase modulations are nearly identical, their signs are inverted and by neglecting the sound attenuation, the effects of the two propagations eliminate each other. Thus a signal reflected once or several times that arrives at the receiver, possesses the same phase modulation as the signal propagating directly from the transmitter to the receiver.

Consequently, the multiple reflections and propagations, which are not exactly known in practical applications of the flow meter, do not essentially violate the assumption within the modeling of equation (3).

#### 2.2 Signal processing

In order to estimate the flow rate using equation (1) the vortex frequency  $f_{\rm v}^*$  has to be derived from the phase-modulated high frequency signal. The required phase demodulation, which transforms  $u_2(t)$  to the low-frequency range of the vortex frequency, is considered exemplary by an analog demodulation: the received signal  $u_2(t)$  is multiplied with a reference signal proportional to  $u_1(t)$ :

$$u_{\rm m}(t) = ku_1(t)u_2(t) \tag{7}$$

By applying an addition formula one obtains with equations (2) and (3)

$$\begin{split} u_{\rm m}(t) &= c_1 {\rm cos}(\phi_0 + \phi_{\rm v}(t)) + c_2 {\rm cos}\phi_2 - c_1 {\rm cos}(2\omega_0 t + \phi_0 + \\ \phi_{\rm v}(t)) - c_2 {\rm cos}(2\omega_0 t + \phi_2) \end{split} \tag{8}$$

with 
$$c_1 = \frac{\overline{u}_1 \overline{u}_{21}}{2} k$$
 and  $c_2 = \frac{\overline{u}_2 \overline{u}_{22}}{2} k$ 

By means of a bandpass filter the common and high frequency components are suppressed and the demodulated signal obtained is

$$u_{d}(t) = c_{1}\cos(\varphi_{0} + \varphi_{v}(t))$$

$$= c_{1}\cos(\varphi_{0} + \overline{\varphi}\sin(\omega_{v}t))$$
(9)

As the phase demodulation eliminates the influence of the non-modulated components, the unwanted signals resulting from the sound propagation in the pipe wall do not disturb the measurement. At the same time low-frequency disturbances of the installation, which do not result in a phase modulation of the ultrasonic wave (e.g. relatively slow mechanical vibrations of the pipe), are rejected.

In order to estimate the vortex frequency from the spectrum of the demodulated signal,  $u_{\rm d}(t)$  must possess a sufficiently high amplitude, which is achieved by strong phase fluctuations  $\overline{\varphi}$  and a high sensitivity of the cosine function of equation (9) according to  $\varphi_v(t)$ .

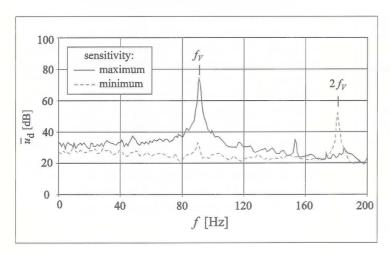


Fig. 5 Amplitude spectrum of the phase-demodulated signal

In order to achieve high sensitivity an operation near the optimum working point  $\phi_{0,\mathrm{opt}}$  is required (see Fig. 4), otherwise the sensitivity decreases severely. With a distance of  $\pi/2$  from  $\phi_{0,\mathrm{opt}}$ , the sensitivity is at a minimum and additionally a frequency doubling occurs, which would lead to an erroneous measurement with the estimation of twice the vortex frequency (see Fig. 5).

As a result of the strong dependency of the phase  $\varphi_0$  on the pipe geometry, as well as on the speed of sound within the pipe and the fluid,  $\varphi_0$  is *a priori* unknown. This makes it necessary to track the working point or to apply a demodulation principle, which works independently of the working point. Automatic tracking is realized by a phased locked loop (PLL) which has to be tuned to the high ultrasonic frequency  $f_0$ . The PLL adjusts the phase difference between the reference

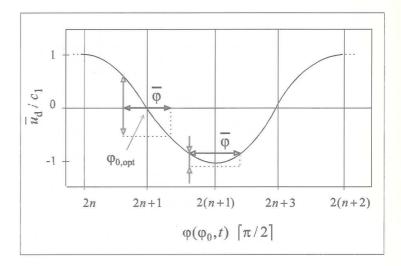


Fig. 4 Sensitivity of the phase demodulation with analog multiplication

signal  $u_1^*$  which is applied for the demodulation and the received signal  $u_2(t)$  to a given value by controlling the reference signal frequency. Independence of the working point is realized by I/Q-demodulation, in which the received signal is multiplied by the reference signal  $u_1^*(t)$  and at the same time in another channel by the reference signal which was shifted by  $\pi/2$ . Thus the sensitivities of these two channels are also shifted by  $\pi/2$  and by adequate combination a constant sensitivity is achieved [5].

The following frequency estimation determines the vortex frequency from the spectrum of  $u_{\rm d}(t)$ . This operation (which has to be realized within every vortex meter) can be implemented by a gated counter, by a Fourier transformation with an estimation of the maximum or by a PLL which is tuned to the low-frequency range of the vortices  $f_v$  [6].

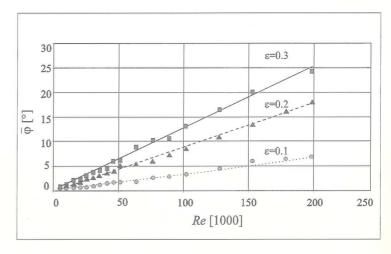


Fig. 6 Dependency of the phase shift amplitude  $\bar{\phi}$  on the relative bluff body width  $\epsilon$  (d = 50 mm)

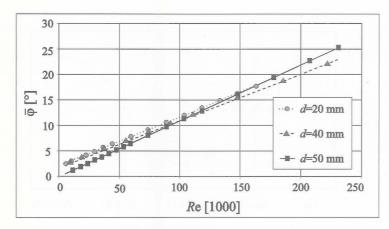


Fig. 7 Dependency of the phase shift amplitude  $\bar{\varphi}$  on the inner pipe diameter d ( $\varepsilon = 0.3$  mm)

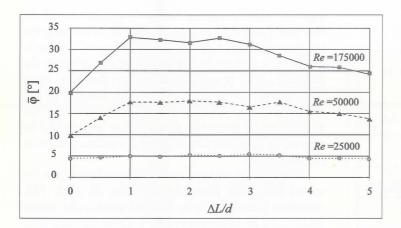


Fig. 8 Dependency of the phase shift amplitude  $\overline{\phi}$  on the relative axial distance between bluff body and transducers  $\Delta L/d$  ( $\epsilon=0.3$  mm, d=50 mm)

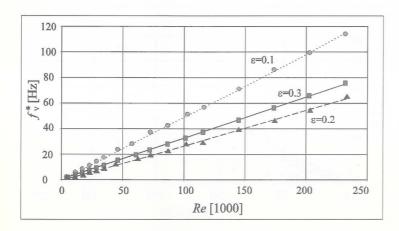


Fig. 9 Estimated vortex frequency  $f_{\nu}^*$  dependent on the relative bluff body

#### 3 Phase fluctuation

As it is evident from equation (9), the relevant factors which influence the ultrasonic system are the amplitudes of the effective signal  $(c_1)$  and of the phase fluctuation  $(\overline{\varphi})$ . A strong effective signal is attainable by means of an acoustical matching. In the following, the influence of different parameters on  $\overline{\varphi}$  is demonstrated by the results of a variety of measurements.

The dependency on the phase fluctuation on the Reynolds number Re is shown in Fig. 6. With a given ratio  $\epsilon$  of bluff body width and inner pipe diameter,  $\overline{\phi}$  increases nearly linearly with Re. The corresponding gradient rises with increasing  $\epsilon$ , resulting in substantially higher phase fluctuations. As the remaining cross-sectional area of the flow decreases with the increasing bluff body width (which results in a higher flow resistance of the flow meter)  $\epsilon$  can not be enlarged to an unlimited extent.

The modification of the inner pipe diameter d (with  $\varepsilon$  = constant) has almost no effect on the gradient of the function  $\overline{\varphi}(Re)$  (see Fig. 7), which results from two distinctive phenomena: while d decreases, the acoustical path of the effective signal in the fluid becomes smaller. At the same time, the velocity of the vortices increases and consequently the phase shift per unit length rises. The two effects compensate each other, resulting in an almost unchanged phase fluctuation.

By varying the normalized distance from the bluff body to the sensors  $\Delta L/d$  the amplitude of the phase fluctuation essentially does not change (see Fig. 8). Thus in contrast to other principles of ultrasonic flow measurement [7], this method is proved to be very insensitive to inaccuracies of the sensors' position.

#### 4 Accuracy

In the following, the dependency on the achievable accuracy of the presented flow meter on relevant parameters will be demonstrated by the influence of the relative bluff body width  $\epsilon$  and the inner pipe diameter d.

The plot in Fig. 9 of the detected vortex frequency  $f_v^*$  over the Reynolds number Re (measurement time 800 ms) shows the linear dependency which is specific to the vortex principle, and the slope of which decreases with increasing  $\epsilon$ . With large bluff body widths ( $\epsilon \geq 0.2$ ), the vortex frequency  $f_v^*$  is additionally influenced by the remaining cross-sectional area of the flow and thus the slope does not increase further. The relative deviations  $\Delta K$  of the measured values from the regression line describe the error of the flow measurements if a

constant Strouhal number is assumed and if equation (1) is applied (see Fig. 10, mean value of 5 measurements per Reynolds number). Applying the optimum bluff body width of  $\varepsilon = 0.2$  a relative error of  $\pm 0.75$  % can be achieved within a measuring range down to  $Re_{\min}$  = 8720. Assuming that the dependency on the Strouhal number on Re is known, the systematic deviations of the linearity can be eliminated easily by applying a calibration table. The remaining errors result from stochastic fluctuations of the measurement. The corresponding variation coefficients ρ (which are the standard deviations related to the corresponding mean value) dependent on the relative bluff body width  $\varepsilon$  are plotted in Fig. 11. With sufficiently high  $\varepsilon$ , the variation coefficients down to small Reynolds numbers are within an error tolerance of  $\pm 0.75$  %.

The high deviations from the linearity within very small *Re* result from the increasing variations of the Strouhal number with decreasing *Re* and thus they are specific to the vortex principle.

The high variation coefficients, which occur with small  $\varepsilon$  and very small Re, can be explained by the instationarities of the vortex shedding, and so they are also a result of the principal limit of vortex meters [8].

If pipes with constant thickness are considered, with decreasing inner pipe diameter and constant ε the ratio between the cross section of the pipe wall and the flow area enlarges and the difficulties of the clamp-on principle, which are generally considered to be the coupling of the ultrasound to the fluid, as well as the propagation through the pipe wall, should increase. In contradiction to this, Figs. 12–14 do not show a significant decrease in accuracy with a declining inner pipe diameter. The expected disturbances are suppressed by effective coupling and appropriate signal processing.

Assuming that the measuring system is optimized to the parameters derived in section 2, the measuring range and accuracy are entirely limited by quantities which are generally specific to the vortex principle. Thus it is demonstrated that by applying acoustical vortex detection, the accuracy and dynamic range of conventional vortex meters is achievable.

Since with comparable expense of manufacturing a high operational robustness and minimum maintenance problems are achieved, this is a competitive alternative to conventional detection principles.

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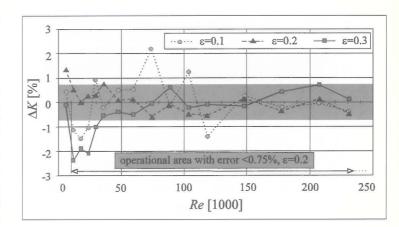


Fig. 10 Relative deviation from the linearity  $\Delta K = (f_v^* - f_{lin})/f_{lin}$  dependent on the

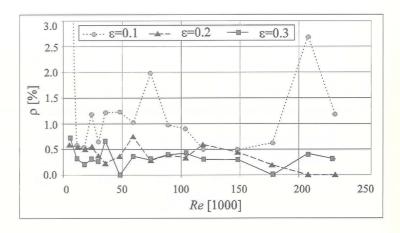


Fig. 11 Variation coefficient  $\rho$  dependent on the relative bluff body  $\epsilon$  (d = 50 mm)

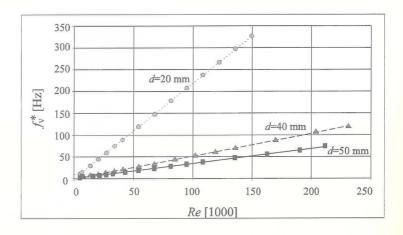


Fig. 12 Estimated vortex frequency  $f_v^*$  dependent on the inner pipe diameter d

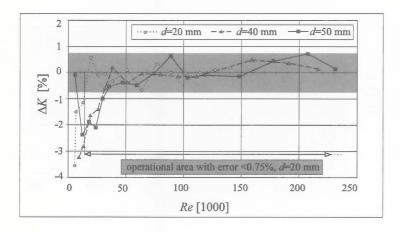


Fig. 13 Relative deviation from the linearity  $\Delta K = (f_v^* - f_{lin})/f_{lin}$  dependent on the

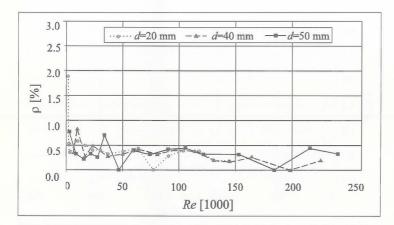


Fig. 14 Variation coefficient  $\rho$  dependent on the inner pipe diameter d ( $\varepsilon$  = 0.3 mm)

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#### CALCULATION OF UNCERTAINTY

### Pooled variance and its applications with specific reference to Type A uncertainty in the calibration of measuring instruments

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#### Abstract

This paper deals with the concept of pooled variance, the conditions under which it is valid, the categorizing of measuring instruments in order to calculate variance, and also the necessity for pooled variance for evaluating Type A uncertainty for measuring instruments received for calibration.

A method is proposed for calculating pooled variance and justification for pooling, taking a typical example of five proving rings. It has been suggested that in addition to its use for the estimation of Type A uncertainty, standard deviation from the pooled variance may be used for:

- testing the authenticity of fresh observations;
- maintenance of measuring instruments;
- fixing the maximum permissible error for regulatory purposes; and
- rejection of an instrument under calibration.

The National Physical Laboratory (New Delhi) and in particular the author have been associated with the subject of uncertainty and its dissemination for some time. Various documents and lectures [6, 7, 8] were prepared on the hithertoo accepted concepts on behalf of the Commonwealth Science Council (London) and widely distributed in training programs held under the Asia Pacific Metrology Program and at the Commonwealth India Metrology Centre, New Delhi.

To disseminate the concepts of the Guide to the participants of the National Workshop on the Role of Metrology in Total Quality Management (18–19 August 1995), in a recent paper [9] the author discussed estimation of Type A and Type B uncertainties when dealing with a single measurand or when assigning the value to an artifact. Several highly useful concepts are only briefly mentioned in the Guide.

One such important concept is the pooled variance of the instrument; its applications and methods of calculation are highlighted in this paper, as is the difficulty in finding a solution to assign Type A uncertainty to instruments received for calibration.

#### 1 Introduction

The Guide to the Expression of Uncertainty in Measurement [1] is consistent with the recommendations of the Working Group instituted by the BIPM in 1980 [2] and those of the CIPM in 1981 and 1986 [3, 4]. The GUM is the most widely accepted international document; besides some suggestions by Ernesto et al. [5] it is also a most comprehensive and concise document.

The concept of Type A and Type B uncertainties is relatively new to scientists in calibration laboratories at secondary and tertiary levels and also to users in industry, especially in developing countries. Therefore wider publicity is necessary, in terms of detailed papers on the various aspects dealt with.

#### 2 Pooled variance

The pooled variance of an instrument is the arithmetic mean of a large number of variances, after taking into consideration their respective degrees of freedom. It is valid when the value of the input supplied to the instrument remains constant for each repetition, and the variation is mainly due to the instrument under calibration. This situation exists during the calibration of many measuring instruments, e.g. when calibrating weighing instruments against standard weights or linear dial gauges against slip gauges.

A similar situation may arise during the calibration of electrical instruments, when a current or voltage of constancy by one order of magnitude or better is supplied to an ammeter/voltmeter, or during the calibration of a proving ring using a dead weight machine. In all these cases, observations mainly vary due to the instruments under calibration.

## 3 Type A uncertainty of measuring instruments received for calibration

Most instruments are calibrated by observing the reading of the instrument, the value of the standard input supplied to it being known. Ammeters, voltmeters, dial pressure gauges, linear dial gauges and weighing machines are but a few examples. Such instruments are calibrated at several points: at each point, in addition to the correction to be applied, Type A as well as Type B uncertainties are required to be given.

As per the Guide, Type A standard uncertainty is equal to the standard deviation. To calculate the latter, a large number of repetitions should be taken at each point being calibrated. However due to time and resource constraints, it is not possible to take a sufficiently large number of repetitions at each point, so the calculation of variance for the purpose of Type A uncertainty presents a problem.

Firstly the standard deviation of two or three observations has little meaning and secondly, it is more likely that there are wide variations in its values from one point to another of the same instrument. So the Type A uncertainty reported is more likely to be different not only for two calibrated instruments of the same type (for the same user) but may also be different at the various points at which the instrument has been calibrated. This will naturally confuse the user and may also cause some surprise especially when several different values of uncertainty appear on the instrument certificate.

The concept of pooled variance may be used to solve this problem. It has been suggested that the standard deviation at each point of calibration may be replaced by the square root of the pooled variance.

### 4 Calculation of pooled variance

All instruments may be divided into two categories for the purpose of calculation of pooled variances:

Category I: the variance is independent of the input quantity

All instruments having a linear relation between the input quantity and the reading on their scale fall into

this category. In this case, variances at different points of the scale may be pooled together.

Category II: the variance depends on the input quantity

All instruments having non linear scales will fall into this category. In this case, only variances for the same input quantity can be pooled together. So the pooling is to be carried out on different instruments of same kind for each input quantity, which requires more effort and a larger number of instruments before a reliable estimate of pooled variance is obtained.

## 4.1 Category I: the variance is independent of input quantity

Let an instrument be calibrated at I number of points and at each point, J number of observations have been taken. The mean of J observations at the  $i^{th}$  point is  $R_i$  and the  $j^{th}$  observation at the  $i^{th}$  point is denoted by  $r_{ij}$ . Then

$$R_{i} = [r_{i1} + r_{i2} + r_{i3} + ..... + r_{iJ}]/J = \Sigma(r_{ij})/J$$
 (1)

If the variation in the indications of the instrument is independent of the value of the indication, then to obtain the pooled variance  $S^2$  the squares from the mean value of each indication are added and the sum thus obtained is divided by I(J-1). This is illustrated in Table 1.

Table 1

Observations	Mean	Sum of squares from the mean
r <sub>11</sub> , r <sub>12</sub> , r <sub>13</sub> , r <sub>1J</sub>	$R_1$	$\Sigma (r_{1i} - R_1)^2$
$r_{21}, r_{22}, r_{23}, \dots r_{2J}$	$R_2$	$\Sigma (r_{2i} - R_2)^2$
r <sub>31</sub> , r <sub>32</sub> , r <sub>33</sub> , r <sub>3J</sub>	$R_3$	$\Sigma (r_{3i} - R_3)^2$
$r_{41}, r_{42}, r_{43}, r_{4J}$	$R_4$	$\Sigma (r_{4j} - R_4)^2$
r <sub>11</sub> , r <sub>12</sub> , r <sub>12</sub> , r <sub>11</sub>	$R_{\tau}$	$\Sigma (r_{Ii} - R_{I})^2$
	r <sub>11</sub> , r <sub>12</sub> , r <sub>13</sub> , r <sub>1J</sub> r <sub>21</sub> , r <sub>22</sub> , r <sub>23</sub> , r <sub>2J</sub> r <sub>31</sub> , r <sub>32</sub> , r <sub>33</sub> , r <sub>3J</sub> r <sub>41</sub> , r <sub>42</sub> , r <sub>43</sub> , r <sub>4J</sub>	$r_{11}, r_{12}, r_{13}, \dots r_{1J}$ $R_1$ $r_{21}, r_{22}, r_{23}, \dots r_{2J}$ $R_2$ $r_{31}, r_{32}, r_{33}, \dots r_{3J}$ $R_3$ $r_{41}, r_{42}, r_{43}, \dots r_{4J}$ $R_4$

The pooled variance is:

$$S^2 = \Sigma \Sigma (r_{ij} - R_i)^2 / I(J - 1)$$
 (2)

where i takes values from 1 to I in the first summation and j takes values from 1 to J in the second summation.

## 4.2 Category II: the variance depends on input quantity

In this case variances taken for the same input quantity magnitude are pooled together for similar kinds of instruments.

If  $s_i^2$  is the variance of  $n_i$  number of observations then the pooled variance  $S^2$  for N such sets for a particular point of similar instruments is given by:

$$S^{2} = \sum (n_{i} - 1) s_{i}^{2} / \sum (n_{i} - 1)$$
(3)

where i takes values from 1 to N.

#### 5 Uses of pooled variance

## 5.1 Estimation of Type A uncertainty of an instrument

An example follows for the justification and calculation of pooled variance from variances taken at different points of calibration of the same instrument; the pooled variances for several instruments of the same kind are themselves also pooled. The square root of the pooled variance will then be the Type A standard uncertainty for the instrument under calibration at all points.

The calibration data of five proving rings was analyzed: each of three rings A, B and C of the same type had a dial gauge as an indicating device, while each of the others (D and E) were fitted with a six-digit electronic indicating counter. The latter were much more readable.

Each ring was tested at ten points and observations for each point were repeated three times. The variances of three observations at each point are given in Table 2. The mean value of the variance, the value of the ratio between the maximum and minimum variances and the ratio of the maximum variance with the mean variance have been tabulated in the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> rows of Table 3. The greater variance has always been taken as the numerator, so that all ratios are suitable for Fisher's F test [10].

Table 3 Mean variances and ratios of maximum to minimum and maximum to mean variances

Mean var.	0.1087	0.0453	0.0360	13.000	217.767
Ratio of Max./Min.	72.6418	16.335	26.8656	49.3333	55.5807
Ratio of Max./Mean	2.2387	3.6059	2.5000	3.7948	2.6358

Fisher's F test has been applied to test the hypothesis that variances under test belong to the same population. The degree of freedom for variances at each of ten points is only 2, while the degree of freedom for the mean variance = 20.

The value of F for 2/20 degrees of freedom at 1% level is 5.85 and 3.49 at 5% level of significance. Similarly the F values for 20/20 degrees of freedom are 2.94 and 2.12 at 1% and 5% levels of significance respectively.

The application of Fisher's F test indicates that none of the variances in any of the 5 proving rings is outside the population at 1 % level of significance. The limiting value of F at 1 % level of significance for 2 degrees of freedom for each variance is 99.0.

Comparing the ratios of maximum value of the variance with the mean of variances in the set indicates that none of the maximum variance is outside the limits

Table 2 Variances of five proving rings

S. No.	A	В	С	D	E
1	0.0067	0.0267	0.0600	4.6667	20.6667
2	0.0800	0.0200	0.0867	2.0000	44.6667
3	0.0600	0.0000	0.0867	4.6667	98.0000
4	0.2600	0.1400	0.1400	4.6667	186.000
5	0.4867	0.2067	0.0267	4.6667	254.000
6	0.0600	0.0600	0.1800	56.0000	416.666
7	0.4067	0.0600	0.0867	14.0000	542.000
8	0.3467	0.0467	0.0267	98.6667	772.667
9	0.1800	0.0200	0.0067	42.0000	872.000
10	0.2867	0.3267	0.0200	28.6667	1148.000

even at 5 % level of significance for rings A, C and E and at 1 % level for rings B and D.

The ratios of the mean variances for rings A, B and C show that all mean variances belong to the same population at 1 % level of significance.

It may be noticed that the ratio of mean variances of rings E and D is 16.75, which is much larger than the F value of 2.94 for 20/20 degrees of freedom. Hence the two mean variances cannot be said to belong to the same population even at 1 % level of significance.

As the data analyzed has only two degrees of freedom and the last digit in the data is only a visual estimate, a stray case of wide variation may be neglected.

From the above, it may be reasonably concluded that all variances in the set may be pooled and the resulting square root taken as the Type A standard uncertainty. It is also reasonable to take the average of the variances for similar proving rings to form a pooled variance. When such data is accumulated over one or two years, a reliable value of the pooled variance may be established. The square root of this pooled variance may thus be taken as the Type A standard uncertainty for future use.

A similar process gives a reliable value of pooled variance when used for many similar instruments of category II.

#### 5.2 Testing the authenticity of observations

The standard deviation (S), the square root of the pooled variance, may also be used for testing the authenticity of the observations taken in the future on a similar instrument. A criterion may be formulated that the difference between any two observations should not be greater than the standard deviation or its multiple. For example assuming the normal distribution for all observations with S as its standard deviation, then no difference between any two observations should be greater than twice the value of 1.96S, at 95 % level of confidence.

The idea of pooled variance has been used for routine calibration of weights in mass standards activity of the New Delhi National Physical Laboratory for a number of years. In a precision balance for a specific range, the variation in the observations is almost independent of the denomination of weights. The standard deviation from the pooled variance is determined and used as permissible limits for the two mass values obtained for the same weight by two observers under almost the same environmental conditions. If the difference between the two values is less than 2S, their mean is taken as the mass value of the weight piece with S as one component of Type A standard uncertainty. Otherwise the mass value of the

weight is re-determined; this is a stricter criterion than that suggested in the previous paragraph.

#### 5.3 Maintenance of laboratory instruments

To ensure the correct operation of a measuring instrument, not only its calibration but also its repeatability should remain unaltered, or at least within a specified range. The standard deviation - i.e. the square root of the variance - is a good measure of the repeatability of an instrument, hence continuous monitoring of its variance should be carried out. Variances are calculated at prescribed regular intervals of time, preferably based on the same number of observations (for ease of calculation). The progressive mean of these variances, i.e. the pooled variance, is calculated. For a good working instrument the new variance should belong to the same population; to verify this Fisher's F test may be applied. The ratio of the new variance to its pooled variance should not exceed the value of F, tabulated for appropriate degrees of freedom at a prescribed level of significance (e.g. 5%). The degree of freedom of the new variance would be the number of observations -1, while for pooled variance it would be:

 $\Sigma(n_i - 1)$ , where the summation is taken over N pooled variances.

A graph of all the previous values of standard deviations should also be drawn - if it shows an upward trend and the new value exceeds a certain specified value then the instrument may either be discarded or downgraded for less precise work.

## 5.4 Fixing the maximum permissible error (mpe) of an instrument

Having determined the pooled variance, the mpe may be fixed. Indeed, mpe and standard deviation are related: an instrument having an mpe lower than its repeatability is clearly of no practical value. For example, a reasonable value of the mpe of the instrument may be twice the value of the standard deviation, i.e. the standard deviation of any instrument should not be greater than half of its maximum permissible error.

## 5.5 Rejection of an instrument received for calibration

If the pooled variance of a certain type of instruments is already known, then a particular instrument received

for calibration may be rejected if its sample variance is greater than the prescribed limits. Fisher's test may be considered again, for example the ratio of the sample variance to the pooled variance of the group of instruments may not exceed the tabulated value for the known degrees of freedom at 1 % level of significance. Attention is drawn to the ratio of the mean variances of proving rings D and E in 5.1. Had some fixed criterion existed, it would have been possible to reject the proving ring on the basis of its variance. For example, according to the criterion proposed above, proving ring E could be rejected.

#### 6 Conclusions

The concept of pooled variance is relatively new to scientists engaged in calibration work, so its potential uses have not been fully exploited. It is hoped that this work may serve as a "stepping stone" to realize the importance of this concept and the full exploitation of its applications. Use of pooled variance for fixing maximum permissible error and rejection of an instrument may also help in preparing quality manuals. National laboratories and custodians of national measurement standards should play an active role in this area. Usually, such laboratories have the capability and resources to generate sufficient data in terms of pooled variances for common measuring instruments and to discuss and finalize the criteria for (a) maintenance, (b) fixing maximum permissible errors of measuring instruments and (c) rejection of measuring instruments received for calibration.

### 7 Acknowledgments

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### METROLOGICAL INFRASTRUCTURES

## Metrological activity in Ukraine

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The State Metrology Service in Ukraine is the official governmental body responsible for industrial and legal metrology, as well as for the accreditation of verification and calibration laboratories.

### 1 History

The Inspectorate of Measures and Weights, set up in Galychyna (Western Ukraine) in 1785, was responsible for the implementation and supervision of traceability assurance, training of production personnel and maintenance of measuring instruments in district towns. In 1875 with the introduction of the metric measuring system in the Austria-Hungary Empire, metric measures began to be used in Galychyna too and unified metrological supervision by the Inspectorate was gradually implemented.

In 1875 the Russian Empire signed the *Convention du Mètre* and from 1 January 1900 the metric system was used along with the Russian measuring system. Then in 1901 the first Verification Chamber for Trade Measures and Weights in Central and Eastern Ukraine (a part of the Russian Empire) was opened in Kharkiv, which in 1922 became the Main Ukrainian Chamber of Measures and Weights - a

central scientific and technical body based on the existing verification chambers for traceability assurance.

When Ukraine gained its independence in 1992 the State Committee of Ukraine for Standardization, Metrology and Certification (Derzhstandard) was established, and traceability assurance was one its responsibilities. It now ensures State control for uniformity of measurements.

### 2 Legal Metrology

The legislative basis of the State Metrology System includes the Decree of the Cabinet of Ministers on "Traceability assurance" (no. 40-93 dated 1993-04-26). The Decree on measuring units represents the standard for measuring instruments on which present-day activities of the State Metrology Service are based.

In 1996, the Derzhstandard developed a draft law on "Metrology and Metrological Activities", which highlighted all the major aspects of the organization and management of metrological activities; its content is in line with the guidelines set out in OIML International Documents, such as D 1 "Law on metrology", D 3 "Legal qualification of measuring instruments", D 12 "Fields of use of measuring

instruments subject to verification" and other relevant documents. The draft has been submitted to Verhovna Rada of Ukraine for consideration.

Laws concerning metrology are designed to assist the development of science, technology and the national economy, to avoid the negative consequences of uncertain measurement results, to create advantageous conditions for the development of international relations and trade, as well as to establish the basic legal principles on metrology and metrological activity.

## 2.1 Normative documents on metrology

The requirements contained in normative documents which are approved by the Derzhstandard are obligatory for ministries, local authorities, enterprises, organizations, etc. The requirements contained in normative documents which are approved by ministries, departments and amalgamations of enterprises are obligatory for enterprises and organizations which fall within the management of a body which has approved these documents.

According to their field of activity, enterprises and organizations approve documents which describe in detail (without contradicting) the provisions of those normative documents approved by Derzhstandard.

## 2.2 The International System of Units (SI)

The International System of Units (SI), adopted by the General Conference on Measures and Weights and recommended by the OIML, is used in Ukraine. The Derzhstandard can introduce other units not included in the SI, and lays down their rules of representation.

The characteristics and parameters of goods (including measuring instruments) and services (including measurements, metrological certification, verification, calibration and state tests) for export may be given in units specified by the customer.

The reproduction and conservation of units in order to transmit their values to all measuring instruments used in Ukraine are achieved by national measurement standards, the basis of which is established and improved in accordance with the state programs developed by Derzhstandard in order to meet the needs of the economy.

Derzhstandard is responsible for the fulfillment of these programs, for the technical level of national measurement standards and for the optimal structure of the measurement standards base. National measurement standards are state property, are subject to Derzhstandard approval and fall under its authority. A procedure for establishing, approving, registering, storing and applying measurement standards, as well as for their comparison with international measurement standards, is laid down by Derzhstandard.

The responsibility for observing the rules and conditions of storage and the application of standards is that of the managers and scientists in organizations.

#### 2.3 Measuring instruments

Measuring instruments must comply with the operating conditions and accuracy requirements specified. It is only authorized to manufacture, use, repair, sell or hire out those measuring instruments which have been successfully verified, certified or calibrated, and it is only authorized to import those instruments whose types are included in the State Register.

Measurements which are carried out within the scope of national metrological supervision must be performed in accordance with certified measurement procedures. A procedure for the development and metrological certification of measurement procedures is laid down by Derzhstandard.

Measurement results may be used provided that the corresponding characteristics of measurement errors are known. Measurements falling within the scope of national metrological supervision may be carried out by measuring laboratories, provided that the latter are accredited to perform such measurements.

## 2.4 Financing of metrological activities

State Budgetary Funds of Ukraine finance:

- fundamental metrological research;
- establishing, improving, acquiring and maintaining national and secondary measurement standards;
- drafting normative documents;

- work on unifying time and reference frequencies, certified reference materials for the composition of substances and standard reference data on physical constants and properties of substances;
- developing, acquiring and maintaining the equipment necessary for regional Derzhstandard bodies;
- work on state metrological supervision.

Enterprises, organizations and business entities pay for work carried out such as:

- granting of licenses for the manufacturing rights and repair of measuring instruments (the amount is determined by the Cabinet of Ministers);
- performing other types of state metrological inspection (in accordance with the Rules of payment laid down by Derzhstandard in consultation with the Ministry of Economy) for work on state metrological supervision.

#### 3 Organizational structure

## 3.1 Service of metrology in Ukraine

Activities of the State Metrology Service (comprised of metrology services within ministries, departments, enterprises and organizations) are coordinated by Derzhtandard (see Fig. 1).

The State Metrology Service comprises:

- The State Service of Legal Metrology (SSLM);
- The State Service of Unified Time and Reference Frequencies (SSTF);
- The State Service of Certified Reference Materials for the

- Composition and Properties of Substances and Materials (SSRM);
- The State Service of Standard Reference Data on Physical Constants and Properties of Substances and Materials (SSSRD).

The SSLM ensures the uniformity of measurements and ensures that metrology law, other legislative acts and normative documents on metrology are observed. The SSLM comprises:

- Department of Metrology of Derzhstandard (in Kyiv);
- State Scientific Metrological Centers of Derzhstandard (hereafter referred to as "metrological bodies"):
- Metrological Centers and regional bodies of Derzhstandard in the Autonomous Republic of Krim, in regions, in Kyiv and Sevastopol and in cities of regional subordination (hereafter referred to as "regional bodies").

Derzhstandard is responsible for pursuing a unified technical policy including:

- organization of the operation of the state system ensuring the uniformity of measurements, of the performance of fundamental research in the field of metrology and of the creation and improvement of the measurement standards base;
- coordination of the activities of the Metrology Service;
- determination of general metrological requirements for measuring instruments, measurement methods and results;
- organizing and exercising state metrological inspection and supervision;
- development of or participation in the development of national, state and intersectorial programs related to the assurance of measurement uniformity;
- taking part in the metrological activity of international organizations.

Ministries, departments, local authorities, enterprises, organizations, etc. are obliged to observe Derzhstandard metrological resolutions.

Metrological bodies establish, improve and apply national measurement standards; they also develop systems for transmitting values of units of quantities, prepare normative documents on metrology, and perform state metrological inspection. Regional bodies fulfill (at regional level) the tasks and functions of Derzhstandard within their terms of reference as stipulated in their respective regulations, and those of Derzhstandard.

The SSTF provides interregional and intersectorial coordination and also carries out work to ensure the uniformity of measurements of time and frequency and also to determine the parameters of the Earth's rotation. The SSRM provides inter-regional and intersectorial coordination and develops and implements certified reference materials for the composition and properties of substances. Activity of the SSSRD is similar, also working on standard reference data of physical constants and properties of substances.

Metrology services within ministries, departments, enterprises and organizations ensure the uniformity of measurements as follows:

- verification, metrological certification, calibration and repair of measuring instruments;
- development of measurement procedures, verification and calibration procedures for measuring instruments and exercising metrological supervision.

## 3.2 Scope of activity of the State Scientific Metrology Centers

The Derzhstandard system consists of three SSMC's:

- The State Scientific-Industrial Association "Metrology" (SSIA "Metrology", Kharkiv) a National Metrology Center for traceability assurance and deals with the standard specimens of composition and properties of substances and materials, dealing with mass, force, hardness, time and frequency and radiotechnical quantities; it maintains the State Register of Standard Specimens.
- The State Scientific and Research Institute "Systema" (SSRI "Systema", Lviv) a metrology center for acoustic and hydroacoustic measurements; it specializes in the metrological assurance of measurements data acquisition systems, and assessment of analytical, measurement and testing laboratories.
- The Ukrainian Scientific-Industrial Center for Standardization, Metrology and Certification (UkrCSM, Kyiv) a metrology center for measuring pressure, vacuum, physical/chemical, electrical and magnetic quantities; it maintains the State Register of Measurement Instruments approved for use in Ukraine and the national stock of standard reference data.

There are 25 regional Centers for Standardization, Metrology and Certification and 9 city CSMCs in Ukraine.

## 4 Scope of activities of the State Metrology Service

## 4.1 State metrological inspection and supervision

State metrological inspection and supervision is exercised by the SSLM in accordance with a procedure laid down by Derzhstandard in order to ensure the observance of metrology laws, other legislative acts and normative documents. The scope of activities and principal objectives are listed below:

- measuring means and measurement data acquisition systems;
- measurement methodology and normative documents specifying the requirements for measurements;
- packed products during packaging and selling;
- other objects envisaged by metrological regulations.

State metrological supervision covers measurements used in:

- diagnosis and curing of human illnesses;
- quality inspection of drugs;
- quality and safety inspection of foods;
- environmental inspection;
- job safety;
- geodesic and hydro-meteorological work;
- trade, commercial operations and settlements, including personal and public services;
- fiscal, banking and customs operations;
- recording energy and material resources (electricity, gas, water, oil, etc.) except internal records kept by enterprises, organizations and citizens as business entities:
- work carried out on the instructions of the courts, arbitrators and other public bodies;
- mandatory product certification;
- registration of national and international records.

The following types of state metrological inspection and supervision of measuring instruments are established:

- state tests and type approval;
- metrological certification;
- verification;
- accreditation for the right to carry out state tests, verify meas-

uring instruments, perform measurements and certify measurement procedures etc.

State metrological supervision is divided into:

- observance of metrology laws, other legislative acts and normative documents, covering ministries, departments, enterprises, organizations and citizens as business entities. Ministries, departments and managerial bodies of enterprises and their amalgamations are checked for observance of metrology laws, other legislative acts and normative documents;
- control over the quantity of prepacked goods, which is applied to completed packages of any type during packaging, storage and sale in cases where the content of these packages cannot be changed without opening or damaging the package, and when a quantity of goods is expressed as a mass, volume or other quantity. The nominal quantity of goods must be indicated on a package in units of mass, volume or other quantity, as well as the limits of deviation allowed from the nominal quantity, or reference must be made to normative documents specifying these limits. Officers of Derzhstandard and its regional bodies which exercise metrological supervision must be certified in accordance with the procedure laid down by Derzhstandard and must have the status of state metrological supervisors.

Measuring instruments intended for serial production or for importation in batches are subject to state acceptance and inspection tests with type approval, and Derzhstandard enters approved types into the official "State Register of Ukraine". These tests are carried out by metrological and regional bodies (provided that they

are accredited). Tests which do not fall within the scope of state metrological supervision may also be performed by metrology services within ministries, departments, enterprises and organizations, again provided that they are suitably accredited. Measuring instruments (and/or their operating documentation) of a type approved by Derzhstandard is marked with the type approval, the form of which is established by Derzhstandard.

Measuring instruments which are not subject to state tests but which fall within the scope of state metrological supervision are subject to metrological certification, carried out by metrological and regional bodies, or by services of metrology within enterprises and organizations (if accredited).

#### 4.2 Verification Service

Measuring instruments which fall within the scope of state supervision and which are in operation, in production, under repair, on sale, for hire or imported into Ukraine are subject to verification.

Verification is also applied to working standards and reference working standards. The list of measuring instruments which are in operation and subject to verification shall be drawn up and submitted to a regional body for approval. Verification is carried out by regional bodies, so long as they are accredited.

Provided that they can justify the required technical and economic capacities (and a corresponding Derzhstandard resolution is available), accredited metrology services of enterprises and organizations may verify instruments, though managers of the latter are responsible for ensuring that this is carried out correctly.

Derzhstandard carries out accreditation of:

- metrology bodies for the right to carry out state acceptance tests of measuring instruments;
- regional bodies and services of metrology within enterprises and organizations - for the right to carry out state acceptance and inspection tests and verification of measuring instruments;
- verification laboratories of the metrology services or other organizational structures of enterprises and organizations for the right to carry out the relevant verification of measuring instruments for other enterprises, organizations, etc.;
- verification laboratories of manufacturers abroad - for the right to carry out verification of measuring instruments which are imported into Ukraine.

Derzhstandard may delegate accreditation to its metrological and regional bodies; the latter carry out accreditation of measuring laboratories of enterprises and organizations which are not subordinate to ministries and departments, for the right to perform measurements in those fields which fall within the scope of state metrological supervision.

Metrological and regional bodies carry out accreditation of metrology services within ministries, departments, enterprises and organizations for the right to carry out certification of measurement procedures which are used in those fields which fall within the scope of state metrological supervision.

Officers of regional bodies who carry out state verification of measuring instruments must be certified

and possess verificator status according to Derzhstandard procedure. State verificators may only verify those specific types of measuring instruments for which they are certified, and in compliance with the requirements in the corresponding normative documents.

#### 4.3 Calibration Service

The Calibration Service system is currently being developed. Measuring instruments which do not fall within the scope of state metrological supervision and which are manufactured, repaired, sold, hired out and imported into Ukraine are subject to calibration, as are measuring instruments which are in operation.

Calibration laboratories carrying out the corresponding calibration of measuring instruments for other enterprises, etc. must be accredited.

Metrology services within ministries and departments as well as enterprises and organizations authorized by them to accredit enterprises' calibration and measuring laboratories are subordinated to ministries and departments.

These services carry out accreditation of:

- calibration laboratories of enterprises and organizations for the right to carry out calibration of measuring instruments for the needs of these enterprises;
- measuring laboratories of enterprises and organizations for the right to perform measurements.

Accreditation of measuring laboratories which perform measurements falling within the scope of state metrological supervision is carried out with the obligatory participation of regional bodies of Derzhstandard, in compliance with the requirements of documents approved by ministries, departments and amalgamations of enterprises in consultation with Derzhstandard.

## 4.4 European and international cooperation

On 12 June 1992 Derzhstandard joined COOMET, the organization representing Central and Eastern Europe state metrological bodies. Derzhstandard is working on the programs in the framework of the five intergovernmental metrological agreements (1992) with CIS countries.

If an international agreement stipulates rules which differ from those in Ukrainian metrological legislation, then the international agreement of Ukraine is executed accordingly.

According to international agreements of Ukraine or under the Derzhstandard resolution the results of state tests, type approval, verification, calibration and metrological certification of measuring instruments carried out in foreign countries may be recognized. Great attention is paid to the development of bilateral cooperation with leading metrological bodies of the Western European countries.

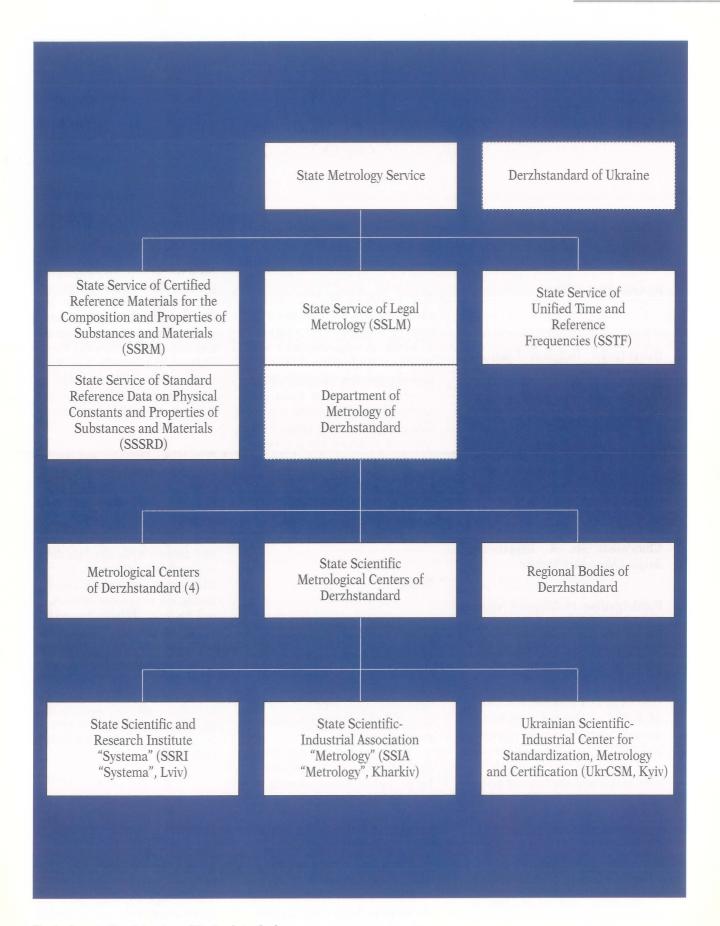


Fig. 1 Organizational structure of the Derzhstandard



## TC 8/SC 7

### Gas metering

### Secretariat: Belgium/France

The Belgian Metrology Service hosted a meeting of OIML TC 8/SC 7, held on 10–12 March 1997 in the *Maison du Gaz Naturel*, the headquarters of the Belgian Federation of gas distributors (FIGAZ), Brussels.

**Chairman:** Mr. R. Eggermont, Belgian Metrology Service

**Participation:** 18 delegates representing 9 P-member countries and FIGAZ; Ph. Degavre, BIML.

This encouragingly large attendance should be underlined: in fact nine out of the fifteen countries registered as P-members of OIML TC 8/SC 7 attended, including several Central European countries and also China. This reflects the importance of the work being carried out and the interest generated within the legal metrology community and the gas distributor federations.

The BIML would like to express its thanks to the organizers of the meeting for their hospitality and warm welcome.

#### Main points

• A 2<sup>nd</sup> working draft on measuring systems for fuel gas had been prepared by the secretariat in close cooperation with the Belgian Federation of gas distributors; this was sent out in November 1996. The main objective of this 2<sup>nd</sup> meeting was to discuss the new draft which took into consideration the decisions of the first meeting held in Brussels on 10–12 June 1996. Significant progress has been made as a consequence of the detailed review of the content.

#### Scope

The international working group confirmed the scope of the future OIML Recommendation which should apply to measuring systems for fuel gas transported or delivered by pipeline, with  $Q_{max} \geq 100 \text{ m}^3\text{/h}$  and operating pressures  $\geq 2 \text{ bar}$ .

These systems range from very large stations located at the border between two countries to industrial or community stations. In some cases, the invoice is made out in energy units; therefore, a part of the Recommendation should deal with measurements of the gas calorific value.

Lastly, it should be noted that the scope excludes systems equipped with diaphragm gas meters.

#### • Terminology

After discussing this chapter, the group approved the terms and definitions proposed by the secretariat, with some minor corrections in order to align the text with the *International Vocabulary of Basic and General Terms in Metrology (VIM)*, as well as with existing OIML Recommendations or ISO Standards.

• Principles of physics and general requirements

This clause which describes the different components of a metering station and their functions and requirements, also lays down the applicable maximum permissible errors and specific requirements for electronic devices. It will be more difficult to reach a consensus on these topics: the group has requested the secretariat to redraft some of the subclauses and to reflect on the given mpe values which apply for pattern approval and initial verification. What value should be applied for in-service verification?

#### Metrological control

The discussions on this aspect indicated that there was general agreement on the various subclauses related to marking, sealing, type approval and initial verification; only minor modifications were required.

#### • Installation requirements

Some comments on this clause were discussed, in particular the subclauses concerning piping configurations that generate swirl and which shall be avoided within a specified distance upstream from the gas meter, this value being defined at pattern approval stage.

#### · Test procedures

This important Annex to the future Recommendation should be drafted by the secretariat; it should be available at least 3 or 4 months before the next meeting in order to enable the participants of the working group to analyze the proposal in detail and to send comments to the secretariat.

The next meeting of TC 8/SC 7 is provisionally scheduled to be held on 21–23 October 1997 in Brussels.

## TC 8/SC 7

## Mesurage des gaz

### Secrétariat: Belgique/France

Sur invitation du Service belge de Métrologie, le sous-comité technique OIML TC 8/SC 7 a tenu une réunion du 10 au 12 mars 1997 à la Maison du Gaz Naturel, siège de la Fédération belge des distributeurs de gaz (FIGAZ) à Bruxelles.

**Président:** M. R. Eggermont, Service belge de Métrologie

Le BIML souhaite remercier les organisateurs de la réunion pour leur hospitalité et leur accueil chaleureux. **Participation:** 18 délégués représentant 9 pays membres-P et FIGAZ; Ph. Degavre, BIML.

Cette large participation est encourageante et doit être soulignée: en fait, neuf des quinze pays inscrits comme membres-P du TC 8/SC 7 étaient présents, y-compris des pays d'Europe centrale et la Chine; ceci reflète l'importance du travail accompli et l'intérêt que lui porte la communauté de métrologie légale ainsi que les fédérations de distributeurs de gaz.

### Points principaux

• Un 2ème projet de travail sur les ensembles de mesurage pour gaz combustible avait été préparé par le secrétariat en étroite collaboration avec la Fédération belge des distributeurs de gaz; celui-ci a été distribué en novembre 1996. L'objectif principal de cette 2ème réunion était de discuter le nouveau projet qui tenait compte des décisions prises lors de la 1ère réunion tenue à Bruxelles du 10 au 12 juin 1996. Des progrès significatifs ont été faits suite à la révision détaillée du texte.

#### • Domaine d'application

Le groupe de travail international a confirmé le domaine d'application de la future Recommandation OIML qui devrait s'appliquer aux ensembles de mesurage de gaz combustible transporté ou fourni par oléoduc, avec  $Q_{max} \geq 100 \text{ m}^3/\text{h}$  et des pressions de fonctionnement  $\geq 2$  bar.

Ces ensembles s'étendent des très grandes stations situées à la frontière entre deux pays aux stations industrielles ou communautaires.

Dans certains cas, la facture est libellée en unités d'énergie;

c'est pourquoi, il convient qu'une partie de la Recommandation soit consacrée aux mesurages du pouvoir calorifique du gaz.

Enfin, il doit être noté que le domaine d'application exclut les ensembles équipés de compteurs de gaz à parois déformables.

#### • Terminologie

Après la discussion sur ce chapitre, le groupe a approuvé les termes et définitions proposés par le secrétariat, avec quelques modifications mineures afin d'aligner le texte avec le Vocabulaire international des termes fondamentaux et généraux de métrologie (VIM), ainsi qu'avec certaines Recommandations OIML et Normes ISO existantes.

• Principes de physique et exigences générales

Cet article, qui décrit les différents composants d'une station de mesurage et leurs fonctions et exigences, fixe également les erreurs maximales tolérées applicables et les exigences spécifiques pour les dispositifs électroniques. Il sera plus difficile d'atteindre un consensus sur ces sujets: le groupe a demandé au secrétariat de revoir certains paragraphes et de réfléchir aux valeurs d'emt spécifiées qui s'appliquent lors de l'approbation de modèle et de la vérification primitive. Ouel valeurs conviennent-elles pour la vérification en service?

#### Contrôle métrologique

Les discussions sur ce sujet ont montré qu'un large consensus existait en ce qui concerne les différents paragraphes traitant du marquage, du scellement, de l'approbation de modèle et de la vérification primitive; seulement des modifications mineures ont été requises.

• Exigences pour l'installation

Plusieurs commentaires sur cet article ont été discutés, en particulier les paragraphes relatifs aux configurations de canalisations qui créent des tourbillons et qui doivent être évitées jusqu'à une distance spécifiée en amont du compteur, cette valeur étant définie lors de l'approbation de modèle.

> ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

> > INTERNATIONAL RECOMMENDATION

Procédures d'essai

Cette importante Annexe à la future Recommandation doit être rédigée par le secrétariat; il convient qu'elle soit disponible au moins 3 ou 4 mois avant la prochaine réunion afin de permettre aux participants du groupe de travail d'analyser la proposition en détail et d'envoyer leurs commentaires au secrétariat.

La date de la prochaine réunion du TC 8/SC 7 est provisoirement fixée au 21–23 octobre 1997 à Bruxelles.

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*Tél:* +33-(0)1-43 19 51 22 *Fax:* +33-(0)1-43 19 51 24

## **New publications**

R 50-2 Continuous totalizing automatic weighing instruments (belt weighers)

Part 2: Test report format

Instruments de pesage totalisateurs continus à fonctionnement automatique (peseuses sur bande)

Partie 2: Format du rapport d'essai

R 104 Pure-tone audiometers

Annex F: Test report format

Audiomètres à sons purs

Annexe F: Format du rapport d'essai

R 107-1 Discontinuous totalizing automatic weighing instruments (totalizing hopper weighers)

Part 1: Metrological and technical requirements - Tests

## Committee drafts received by BIML

March 1997 - May 1997

Stage of development	Title	TC/SC	Secretariat
I CD Revision of	OIML R 60 Metrological regulation for load cells	TC 9	USA
I CD Annex C to	R 122 Equipment for speech audiometry	TC 13	Germany



## REGISTERED OIML CERTIFICATES - CERTIFICATS OIML ENREGISTRÉS

1997.03 - 1997.05

This list is classified by issuing authority; updated information on these authorities may be obtained from BIML.

Cette liste est classée par autorité de délivrance; les informations à jour relatives à ces autorités sont disponibles auprès du BIML.

OIML Recommendation applicable within the System / Year of publication

Recommandation OIML applicable dans le cadre du Système / Année d'édition

Manufacturer / Fabricant
Certified pattern(s) / Modèle(s) certifié(s)

Issuing authority / Autorité de délivrance

Physikalisch-Technische Bundesanstalt (PTB), Germany

R 76/1992 - DE - 93.01

Sartorius AG Weender Landstraße 94-108, D-37075 Göttingen, Germany BA BA 200, BA BB 200, ...

The code (ISO) of the Member State in which the certificate was issued.

Le code (ISO) indicatif de l'Etat Membre ayant délivré le certificat.

For each Member State, certificates are numbered in the order of their issue (renumbered annually).

Pour chaque Etat Membre, les certificats sont numérotés par ordre de délivrance (cette numérotation est annuelle).

Year of issue Année de délivrance

## INSTRUMENT CATEGORY CATÉGORIE D'INSTRUMENT

#### **Automatic catchweighing instruments**

Instruments de pesage trieurs-étiqueteurs à fonctionnement automatique

R 51 (1996)

► Issuing Authority / Autorité de délivrance Physikalisch-Technische Bundesanstalt (PTB), Germany

#### R51/1996-DE-97.01

Robert BOSCH GmbH, Geschäftsbereich Verpackungsmaschinen, Stuttgarter Straße 130, 71332 Waiblingen, Germany

Types KWE 30xx and KPE 10xx (Class X(1))

#### R51/1996-DE-97.02

Optima Control Systems GmbH, Steinbeisweg 20, 74523 Schwäbich Hall, Germany

Types EC and EC ComScale,  $e \ge 0.1$  g,  $n \le 7500$ , Min = 20 g (Class X(1))

► Issuing Authority / Autorité de délivrance Sous-direction de la Métrologie, France

#### R51/1996-NL-97.01

Société ASCOREL, Z.I. de Montplaisir, Rue du Champ de Courses, BP 5, 38780 Pont-Evêque, France

MC 250 pour chargeuses à godet (matériau en vrac), Max = 5 t : e = 50 kg, n = 100; 5 t <  $Max \le 10$  t : e = 100 kg,  $n \le 100$  (Class Y(B))

#### **INSTRUMENT CATEGORY**

CATÉGORIE D'INSTRUMENT

#### Load cells

Cellules de pesée

R 60 (1991), Annex A (1993)

Issuing Authority / Autorité de délivrance
 Centro Español de Metrologia, Spain

#### R60/1991-ES-97.01

Extensotronic, S.A., C/ Colón 6, E-08912 Badalona, Barcelona, Spain

Beam load cell model TFA (Class C)

#### R60/1991-ES-97.02

Epel Industrial S.A., Ctra. Sta. Cruz de Calafell, 35 km. 9,400, 08830 Sant Boi de Llobregat, Barcelona, Spain

Beam load cell model LB2 (Class C)

Issuing Authority / Autorité de délivrance
 Sous-direction de la Métrologie, France

#### R60/1991-FR-97.01

Scaime S.A., Le bois de Juvigny, B.P. 501, 74105 Annemasse, France

Cellules de pesée à jauges de contrainte Scaime types F60X... (Classe C)

► Issuing Authority / Autorité de délivrance

Netherlands Measurement Institute (NMi) Certin B.V.,

The Netherlands

#### R60/1991-NL-97.01

Captels S.A., B.P. 34, Z.A.E. des Avants, 34270 St-Mathieu de Tréviers, France

CPA,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.02

MASTER-K, 38, avenue des Frères Montgolfier, 69680 Chassieu Cedex, France

CIP..,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.03

Tedea Huntleigh International Ltd., 60 Medinat Hayehudim, Herzliya 46120, Israël

1042,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.04

Captels S.A., B.P. 34, Z.A.E. des Avants, 34270 St-Mathieu de Tréviers, France

SAX/I,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.05

Hottinger Baldwin Messtechnic GmbH, Im Tiefen See 45, D-64293 Darmstadt, Germany

PW6.. and PW4..,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.06

Revere Transducers Europe, Ramshoorn 7, Postbus 6909, 4802 HX Breda, The Netherlands

Type 953,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.07

Hottinger Baldwin Messtechnic GmbH, Im Tiefen See 45, D-64293 Darmstadt, Germany

Z6.D./... (Class D) and Z6.C./... (Class C),  $P_i = 0.7$ 

#### R60/1991-NL-97.08

Revere Transducers Europe, Ramshoorn 7, Postbus 6909, 4802 HX Breda, The Netherlands

HCB,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.09

Epel Industrial S.A., Ctra. Sta. Cruz de Calafell, 35 km. 9,400, 08830 Sant Boi de Llobregat, Barcelona, Spain

TSF,  $P_i = 0.7$  (Class C)

#### R60/1991-NL-97.12

Mettler-Toledo Inc., 1150 Dearborn Drive, Worthington, OH 43085-6712, USA

Type 0743,  $P_i = 0.7$  (Class C)

#### INSTRUMENT CATEGORY

CATÉGORIE D'INSTRUMENT

#### Nonautomatic weighing instruments

Instruments de pesage à fonctionnement non automatique

R 76-1 (1992), R 76-2 (1993)

Issuing Authority / Autorité de délivrance

Netherlands Measurement Institute (NMi) Certin B.V., The Netherlands

#### R76/1992-NL-97.01

Mettler-Toledo A.G., Im Langacher, 8606 Greifensee, Switzerland

PG-S (Class II)

#### R76/1992-NL-97.02

Mettler-Toledo A.G., Im Langacher, 8606 Greifensee, Switzerland SR and SG (Classes II and III)

#### R76/1992-NL-97.03

Teraoka Seiko Co., Ltd., 13-12 Kugahara, 5-Chome, Ohta-ku, Tokyo 146, Japan

AW-3600 (Class III)

#### R76/1992-NL-97.04

Teraoka Seiko Co., Ltd., 13-12 Kugahara, 5-Chome, Ohta-ku, Tokyo 146, Japan

DS-516 (Class III)

#### R76/1992-NL-97.05

Teraoka Seiko Co., Ltd., 13-12 Kugahara, 5-Chome, Ohta-ku, Tokyo 146, Japan

DS-73x (Class III)

#### R76/1992-NL-97.06

Teraoka Seiko Co., Ltd., 13-12 Kugahara, 5-Chome, Ohta-ku, Tokyo 146, Japan

SM-8500 (Class III)

#### R76/1992-NL-97.07

Teraoka Seiko Co., Ltd., 13-12 Kugahara, 5-Chome, Ohta-ku, Tokyo 146, Japan

DS-515 (Class III)

#### R76/1992-NL-97.08

Mettler-Toledo Inc., 1150 Dearborn Drive, Worthington, OH 43085-6712, USA

Panther (Classes III and IIII)

#### R76/1992-NL-97.09

Ohaus Corporation, 29, Hanover Road, Florham Park, New Jersey 07932, USA

 $62 g \le Max \le 210 g$  (Class I);  $210 g \le Max \le 8100 g$  and  $12 kg \le Max \le 32.1 kg$  (Class II)

#### **INSTRUMENT CATEGORY**

CATÉGORIE D'INSTRUMENT

#### Fuel dispensers for motor vehicles

Distributeurs de carburant pour véhicules à moteur

#### R 117 (1995) [+ R 118 (1995)]

▶ **Issuing Authority** / Autorité de délivrance

Netherlands Measurement Institute (NMi) Certin B.V., The Netherlands

#### R117/1995-NL-97.01

Schlumberger Electronic Transactions, Retail Petroleum Systems Division, Industrieweg 5, 5531 AD Bladel, The Netherlands + Schlumberger Electronic Transactions RPS Div., Unit 3, Baker Road, Pitkerro Industrial Estate, Dundee DD5 3RT, UK

Model EUROTRON series, respectively HDM, UNIVERSAL, SPECTRA, H and PRIMA series (Class 0.5)

With the publication of OIML Recommendation R 50-1 & 2 Continuous totalizing automatic weighing instruments (belt weighers), and Annex F to OIML R 104 Pure-tone audiometers two important new categories of instruments are now covered by the OIML Certificate System.

Belt weighers are widely used for weighing bulk product, e.g. for loading/unloading ships and trains, for product transfers within a company, or for commercial transactions. These automatic instruments have the advantage of being rapid and relatively accurate: the maximum permissible errors in service are respectively 0.5 %, 1 % and 2 % for the different accuracy classes.

Pure-tone audiometers are designed for determining hearing threshold levels and are of clear importance in the medical field.

The possibility to deliver OIML Certificates together with test results presented in standardized format is certainly an important contribution in these fields.

Two new instrument categories covered by the OIML Certificate System

Deux nouvelles catégories d'instruments sont à présent couvertes par le Système de Certificats OIML Avec la publication de la Recommandation OIML R 50-1 & 2 *Instruments de pesage totalisateurs continus à fonctionnement automatique (peseuses sur bande)*, et de l'Annexe F à OIML R 104 *Audiomètres à sons purs*, deux nouvelles catégories importantes d'instruments ont fait leur entrée dans le Système de Certificats OIML.

Les peseuses sur bande sont très largement utilisées pour le pesage des produits en vrac; par exemple lors du chargement/déchargement des bateaux ou des trains, lors de transferts de produits au sein d'une entreprise, ou pour des transactions commerciales. Ces instruments automatiques présentent l'avantage d'être rapides et relativement précis; les erreurs maximales tolérées en service sont: 0,5 %, 1 % et 2 % respectivement pour les différentes classes d'exactitude.

Les audiomètres à sons purs sont destinés au mesurage des niveaux liminaires d'audition et sont d'une importance évidente dans le domaine médical.

La possibilité de délivrer des certificats OIML, accompagnés par des résultats d'essai présentés de façon normalisée, constitue assurément une contribution importante dans ces domaines.



## COOMET - 7th Committee Meeting / 7ème Réunion du Comité

COOMET (the Metrological Cooperation for Central and Eastern European Countries) held its Seventh Committee Meeting from 23 to 25 April 1997 at the Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Germany, under the presidency of Dr. Spurný, Slovakia.

The meeting was opened by Prof. Göbel, PTB President, who was accompanied by Prof. Kose, PTB Vice-President, and Prof. Kochsiek, Member of the PTB Presidential Board and CIML Vice-President.

Almost 40 people attended, representing Belarus, Bulgaria, Estonia, Germany, Lithuania, Moldavia, Poland, Romania, Russia, Slovakia and Ukraine, as well as EUROMET, WELMEC and OIML.

The Director of the BIML presented a short report on OIML activities since the previous meeting (in Sofia during April 1996) and emphasized the following points:

- as a result of Moldavia and Ukraine recently joining OIML, almost all COOMET countries are also members of OIML;
- OIML is strengthening cooperation with regional organizations and is seeking to avoid regional activity coming into conflict with international activity;
- the development of cooperation between international organizations such as BIPM, OIML and ILAC will have favorable consequences on regional activity;



Delegates attending the  $7^{\text{th}}$  COOMET Committee Meeting

 regional organizations (APLMF, COOMET, WELMEC, etc.) may favor the implementation of OIML Recommendations.

COOMET rapporteurs described intercomparison activities in their various fields of responsibility. Dr. Apel (PTB) was nominated as Rapporteur for legal metrology (as well as for the fields of general metrology and calibration); he will shortly be setting up a draft work program; the Director of the BIML suggested that this program should include points such as the implementation of OIML Recommendations, a mutual agreement on the recognition of pattern approvals, and the study of the European Directive on measuring instruments (currently being prepared); he also recommended that this program should be sufficiently well coordinated with WELMEC activity in order to avoid differences arising which could ultimately hinder wider European integration.

The COOMET President presented a Draft Agreement on the equivalence of national standards, currently being developed by the BIPM, and emphasized the role of regional organizations (such as APMP, COOMET, EUROMET, NORAMET, SIM, etc.) which open up the possibility for countries who are not members of the *Meter Convention* to participate in the development of this Agreement.

At the end of the meeting President Spurny was asked to accept a one-year extension of his mandate before a new President is elected during the next meeting to be held in Minsk, Belarus, 14–17 April 1998.

The Seventh COOMET Committee Meeting was a good opportunity for the Physikalisch-Technische Bundesanstalt to organize conferences and discussions on the following topics:

- uncertainty in measurement;
- world-wide globalization and metrology;
- European harmonization in legal metrology;
- European harmonization in accreditation.

COOMET (Coopération métrologique des pays de l'Europe du centre et de l'est) a tenu sa Septième Réunion de Comité du 23 au 25 avril 1997 au Physikalisch-Technische Bundesanstalt, Braunschweig, Allemagne, sous la présidence du Dr. Spurný, Slovaquie.

La réunion a été ouverte par le Prof. Göbel, Président du Physikalisch-Technische Bundesanstalt, qui était entouré des Prof. Kose, Vice-Président du PTB, et Kochsiek, Membre du Présidium du PTB et Vice-Président du CIML.

Près de 40 personnes ont représenté l'Allemagne, la Bélarus, la Bulgarie, l'Estonie, la Lituanie, la Moldavie, la Pologne, la Slovaquie, la Roumanie, la Russie et l'Ukraine, ainsi que EUROMET, WELMEC et l'OIML.

Le Directeur du BIML a présenté un bref rapport sur l'activité de l'OIML depuis la précédente réunion (Sofia, avril 1996) en insistant sur les points suivants:

- avec la récente adhésion de la Moldavie et de l'Ukraine à l'OIML, pratiquement tous les pays de COOMET sont également membres de l'OIML;
- l'OIML renforce sa coopération avec les organisations régionales et veille à éviter que l'activité régionale n'entre en contradiction avec l'activité internationale;
- le développement de la coopération entre des organisations internationales telles que le BIPM, l'OIML et ILAC aura des conséquences favorables sur l'activité régionale;
- les organisations régionales (APLMF, COOMET, WELMEC, etc.) peuvent favoriser la mise en application des Recommandations OIML.

Les rapporteurs COOMET ont décrit les activités d'intercomparaisons dans les domaines sous leur responsabilité. En ce qui concerne la métrologie légale, le Dr. Apel, PTB, a été nommé Rapporteur (en même temps que pour les domaines de métrologie générale et des étalonnages); il doit prochainement établir un projet de programme de travail; le Directeur du BIML a suggéré que ce programme comprenne des points tels que la mise en application des Recommandations OIML, un accord de reconnaissance mutuelle d'approbations de modèles, et l'étude de la Directive européenne sur les instruments de mesure (actuellement en cours de préparation); il a également recommandé que ce programme soit suffisamment coordonné avec l'activité de WELMEC pour éviter l'apparition de différences qui pourraient ultérieurement freiner une plus large intégration européenne.

Le Président de COOMET a présenté le Projet d'Accord sur l'équivalence des étalons nationaux, actuellement développé par le BIPM, et a insisté sur le rôle des organisations régionales (APMP, COOMET, EUROMET, NORAMET, SIM, etc.) qui peuvent permettre à des pays non-membres de la Convention du Mètre de participer au développement de cet Accord.

A la fin de la réunion, il a été demandé au Président Spurný d'accepter une prolongation de son mandat d'un an avant l'élection d'un nouveau Président lors de la prochaine réunion qui se tiendra à Minsk, Bélarus, du 14 au 17 avril 1998.

La Septième Réunion du Comité de COOMET a été l'occasion pour le Physikalisch-Technische Bundesanstalt d'organiser des conférences et des discussions sur les sujets suivants:

- incertitudes de mesure;
- mondialisation et métrologie;
- harmonisation européenne en matière de métrologie légale;
- harmonisation européenne en matière d'accréditation.

## **Asia-Pacific Legal Metrology Forum**

he Third Asia-Pacific Legal Metrology Forum (APLMF) meeting was held in Vancouver, Canada, on 3 November 1996 in association with the OIML Tenth International Conference of Legal Metrology. The meeting was attended by forty-seven delegates from fifteen Forum member economies, together with representatives from regional and international organizations, the International Bureau of Legal Metrology (BIML), and the Western European Legal Metrology Cooperation (WELMEC). The Forum Working Parties on Legislation and Administration, Intercomparison of Calibration & Pattern Approval Testing and Training held meetings on 1 November.

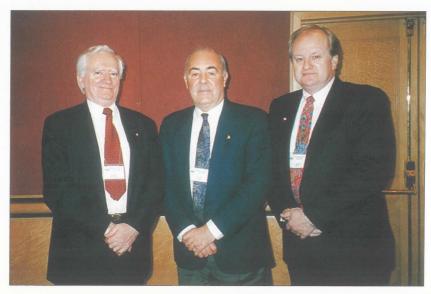
The Forum considered reports of the surveys completed during the year, particularly that on Legal Metrology Technical Infrastructure Needs of Asia-Pacific Developing Economies, conducted by Dr. Knut Birkeland in February–March 1996. The report made fifteen recommendations on legal metrology needs in the developing economies and identified modernization of the legislative and administrative basis of the legal metrology system as having the highest priority; these recommendations have been included in the work program for 1996–97.

Other reports discussed include that on Specialized Legal Metrology Testing Facilities in the region and accessibility to these facilities, National Metrological Requirements for Utility Meters and Legal Metrology Industry Consultative Structures.

The Forum considered the report of the intercomparison of nonautomatic weighing instruments, which commenced in June 1996, among the participating economies of Australia, Canada, PR of China, Germany, Japan, Republic of Korea, Malaysia, New Zealand, United Kingdom and the United States of America, and agreed to the inclusion of Russia in the project; the expected completion date of the intercomparison is June 1997. The Forum also agreed to commence the intercomparison of load cells and mass standards in 1997.

Training has been considered a very high priority need, and the joint Australia-China project on measurement skills and competency-based standards, currently developed with the China State Bureau of Technical Supervision, will be developed further with the proposal to conduct workshops/seminars on OIML R 76 in 1997. The project aims to develop a policy for harmonized measurement skills development in the Asia-Pacific region.

The Forum also discussed the APLMF input to the preparation of the Draft Mid-Term Technical Infrastructure Development Program of the APEC Standards and Conformance SubCommittee (SCSC), and the APEC Conference on Standards & Conformance held in Manila, Philippines in October 1996.



Left to right: John Birch AM, Convenor, APLMF, Richard Knapp, Chairman of the Forum meeting, Alan Johnston, President, Measurement Canada

The Forum agreed to the following work program for 1996-97:

- Publication in 1997 of a Handbook of Legal Metrology in the Asia-Pacific.
- Information on specialized testing facilities is to be included in a Directory of Specialized Testing Facilities published by the APLMF; alternatively this information could be included in the 1997 Handbook.
- A Working Group is to be established to study rice moisture meters. The Secretariat will circulate the scope and objectives of this WG and seek interest from members.
- A Working Group on Medical Measurements is to be established to study sphygmomanometers (blood pressure meters). The Secretariat will circulate the scope and objectives of this WG and seek interest from members.
- The Secretariat is to study the feasibility of establishing an Internet web site to facilitate and improve communication amongst Forum members.
- A follow-up survey on Utility Meters is to be conducted to obtain further information on a number of specific areas such as water metering, telephone metering and gas metering, covering all areas of utility metering activity.
- A workshop/seminar on legislation and administration is to be organized in 1997.
- Translation of the legislation into English is to be carried out for those economies whose legislation is currently not in English.
- Priority is to be placed on the harmonization of requirements for pre-packed goods and a Working Party meeting is to be held in association with the Fourth Forum meeting.
- A training program is to be established that is consistent with the processes and principles established in the APLMF Training Policy, and appropriate training strategies developed for the following identified priority areas:

- the mutual understanding and implementation of OIML Recommendations R 76 and R 60, and
- high capacity fluid flow and automatic weighing instruments.
- A survey is to be conducted to determine the extent of involvement of consumers with the Forum members and to examine ways in which they can participate in Forum activities.
- The scope, objectives and target outcomes for establishing the Working Group on Mutual Recognition Agreements will be formalized by the Secretariat
- A "Memorandum of Understanding" will be finalized for members of the Asia-Pacific Legal Metrology Forum.

The APLMF will hold its Fourth Forum meeting in Tsukuba (Japan) in September 1997, hosted by the National Research Laboratory of Metrology (NRLM). Following on from our discussions in Vancouver on the recommendations of Dr. Birkeland's report on technical infrastructure development and the reports on training, it is proposed to hold the following meetings and workshop/seminars in association with the Fourth Forum meeting:

- Working Party meetings Pre-packed goods, Utility metering (one day);
- Workshop/seminar on Legislation and Administration (three days);
- Workshop/seminar on High Capacity Flow Measurement (two days).

In addition, arising from the Australia-China collaboration on Measurement Skills, it is also proposed to organize a three-day workshop/seminar on the implementation of OIML R 76 *Nonautomatic weighing instruments*. It is proposed that this workshop/ seminar be held either immediately before or immediately after the Tsukuba meetings.

## NOTE ON THE THIRD MEETING OF THE JOINT OIML/CONVENTION DU MÈTRE WORKING GROUP

The third meeting of the joint OIML/Convention du Mètre working group was held at the Pavillon de Breteuil on Wednesday 19 February 1997. The meeting was in two parts, in the first part discussion concerned matters relating to the Convention du Mètre and the OIML and in the second part matters of common interest with the International Laboratory Accreditation Cooperation (ILAC). For the second part, J. Gilmour (President of ILAC) and R. Kaarls representing ILAC were present. As on the two previous occasions, the Convention du Mètre was represented by the bureau of the CIPM and the OIML by members of its Presidential Council.

The first part of the meeting was concerned with discussions on how best to move forward with the closer cooperation clearly requested both by the CIPM at its meeting in 1996 and the Conférence Internationale de Métrologie Légale in November 1996, despite the CIPM having drawn back from discussions at this time on a possible merger of the two organizations. After a preliminary review by the President of the CIPM of the events since March 1995, when the original proposals were made by the French Ministère des Affaires Étrangères, it was agreed that we now concentrate our efforts on specific activities that can be pursued together. Important among these are activities related to metrology in developing countries. It was agreed that a Seminar on this subject will be organized jointly by the BIPM, the OIML, IMEKO and the German authorities to take place at the PTB in 1998.

The second part of the meeting, at which the representatives of ILAC were present, was devoted to examining ways in which the three organizations could coordinate their activities in order to best meet the needs of the users of metrology. Among the tasks identified was the preparation of an application document for ISO Guide 25 specifically addressed to national metrology bodies.

T. J. QUINN Director, BIPM B. ATHANÉ Director, BIML

English original

## Note sur la troisième réunion du groupe de travail commun de l'OIML et de la Convention du Mètre

La troisième réunion du groupe de travail commun de l'OIML et de la Convention du Mètre a eu lieu au Pavillon de Breteuil le mercredi 19 février 1997. La réunion comportait deux parties: dans la première, les discussions concernait les matières relatives à la Convention du Mètre et à l'OIML, et dans la seconde, celles-ci ont porté sur les questions d'intérêt commun avec l'*International Laboratory Accreditation Cooperation* (ILAC). Pour la seconde partie, J. Gilmour (Président de l'ILAC) et R. Kaarls représentant l'ILAC étaient présents. Comme pour les deux réunions précédentes, la Convention du Mètre était représentée par le bureau du CIPM, et l'OIML par des membres de son Conseil de Présidence.

La première partie de la réunion a été consacrée aux discussions sur la meilleure manière de progresser vers une coopération accrue clairement demandée, à la fois par le CIPM lors de sa réunion en 1996 et par la Conférence Internationale de Métrologie Légale en novembre 1996, en dépit du fait que le CIPM avait décidé de mettre un terme aux discussions sur une éventuelle fusion des deux organisations. Après un tour d'horizon par le Président du CIPM sur les différents événements depuis mars 1995, date des premières propositions du Ministère français des Affaires Étrangères, il a été convenu de concentrer dès à présent nos efforts sur les activités spécifiques qui peuvent être entreprises conjointement. Les activités relatives à la métrologie dans les pays en développement en constituent une partie importante. Il a été décidé qu'un Séminaire sur ce sujet serait organisé conjointement par le BIPM, l'OIML, IMEKO et les Autorités allemandes, à la PTB en 1998.

La deuxième partie de la réunion, pendant laquelle les représentants de l'ILAC étaient présents, a été consacrée à l'examen des possibilités de coordination des activités des trois organisations afin de mieux répondre aux besoins des utilisateurs de la métrologie. Parmi les tâches identifiées figurait la préparation d'un document d'application du Guide ISO 25 destiné spécialement aux organismes nationaux de métrologie.

T. J. QUINN Directeur, BIPM B. ATHANÉ Directeur, BIML

Traduction en français par le BIML



## **Seventh UN/ECE Working Party Session**

The seventh session of the Working Party on Technical Harmonization and Standardization Policies of the United Nations Economic Commission for Europe (UN/ECE) was held 5–7 May 1997 at the Palais des Nations, Geneva.

The session was attended by forty-three representatives from twenty countries together with BIML, ISO, IEC, WTO, CEN, EOTC and EFTA.

In accordance with the implementation of the ECE Reforms adopted by the 52<sup>nd</sup> Meeting of the Economic Commission for Europe in April 1997, the Working Party will report to the newly created Committee for Trade, Industry and Enterprise Development (CTIED) as the Commission's Principal Subsidiary Body. Meanwhile, the number of program elements allocated to the Working Party has been reduced. However, the program element on aspects of metrology relevant to testing activities was maintained in the new work program and the growing economic importance of metrology was underlined.

It was noted that the OIML participated in a number of ECE activities such as the development of the Recommendation on "Metrology in Testing" adopted by the Working Party and by the Commission at its 49th session as decision H (49). Training seminars on metrology were organized in 1995 in Paris and in 1996 in Piestany (Slovakia) and an active role was played by the BIML.

Further training seminars on specific metrology matters will be organized. The Rapporteur for metrology informed on the preparation for a workshop on the organization and practice of interlaboratory comparisons at national and international levels. This workshop, originally scheduled for May 1997, has been postponed until the autumn.

The Working Party considered the information given by the BIML representative about a seminar on the role of metrology in economic and social development which is to be co-sponsored jointly by the three international organizations in the field of metrology - BIPM, OIML and IMEKO (TC 11) and the German national metrology institute PTB. The seminar will be organized for the benefit of countries in transition and developing countries and will be held in the first half of 1998 in Braunschweig, Germany. The UN/ECE Working Party appreciated the announcement of this seminar and was willing to co-sponsor it.

The BIML representative gave information about the Tenth International Conference of Legal Metrology and the 31st CIML Meeting held in Vancouver in November 1996. The increased OIML liaisons with international and regional organizations and new developments in communications were reported on, and other activities such as OIML technical work, certification, developing countries and publications were also presented.

Representatives of other organizations (ISO, IEC, WTO, etc.) and national Institutes reported as well on developments in the field of standardization and related activities since the sixth session of the Working Party in May 1996. The session discussed, among other main points of the agenda: coordination of standardization and metrology activities (document Standardization List"); harmonization (ECE Recommendations on standardization policies): conformity assessment: quality policy; intergovernmental agreement on technical harmonization, and a draft study on evaluating the role and impact of norms, standards and regulations in international trade. The consideration of these subjects will be continued during the eighth session of the Working Party which is planned to be held in Geneva in May 1998.

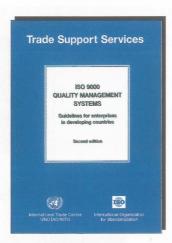
Information about OIML cooperation with the UN/ECE was also published in the July 1995 and July 1996 OIML Bulletins.

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## **General Information**



This guide presents the methodology for implementing the elements of the ISO 9000 quality management system, with particular reference to small and medium-sized enterprises in developing countries. It also provides guidance on preparing such enterprises for third-party (purchasers or authorities) certification processes.



This issue of the *Bulletin du BNM* presents a new version of the French National Standards, presented as a collection of synopses of all fundamental and derived units, realized in BNM laboratories and in some external laboratories. They contain a wealth of information on uncertainties in the realization of French standards as well as on the comparisons which ensure their national, and when applicable, international traceability.

# NWMLT 2 SEPT. 97

To mark the 10<sup>th</sup> anniversary of their move to Teddington, NWML are holding an Open Day which will include a series of presentations on the following topics:

- The National Measurement System
- CE marking
- Uncertainty of measurement
- The measuring instruments Directive update
- Petrol pump certification
- Mass metrology
- EMC testing
- The role of WELMEC
- PADS/EMeTAS
- OIML R 60 certification
- Laboratory accreditation
- NWML's customer service standards

This Open Day will give visitors the opportunity of meet NWML staff and discuss how services can best be provided. A complimentary buffet lunch will be provided and those wishing to attend should contact Richard Sanders at NWML as soon as possible.

Fax: +44 181 943 7270

## Bibliography

▶ Intelligent and mass vortex flowmeters

E. Ribolini, InTech (USA), Vol. 43 n° 2, pp. 30–32, 37 - Feb. 1996.

A stochastic approach to the determination of calibration intervals

A. Bobbio - Dipartimento di Inf., Torino University, Italy; A. Montefusco; P. Tavella.

Proceedings of the 1996 Joint conference: IEEE Instrumentation and Measurement Technology Conference and IMEKO TC 7. (Cat. N° 96CH35936), Brussels 4–6 June 1996 (New York: IEEE 1996) pp. 54–57 vol. 1.



July 1997

7-9 TC 9 TEDDINGTON, UK Instruments for measuring mass and density

Revision R 60: Metrological regulation for load cells

9-11 TC 9/SC 2 TEDDINGTON, UK

Automatic weighing instruments

Automatic instruments for weighing road vehicles in motion

#### October 1997

TC 13
Measuring instruments for acoustics and vibration

Z7
Seminar on legal metrology
RIO, BRAZIL

29
OIML Development Council meeting (morning)

29-31 32<sup>nd</sup> CIML meeting (starting in the afternoon of 29<sup>th</sup> October)

Note: A meeting of the Sistema Interamericano de Metrologia (SIM) will be held on 28th October.

The OIML is pleased to welcome the following new:

### **Committee Members:**

Israel: Mr. G. Deitch

Tunisia: Mrs. Ghaïet El-Mouna Annabi

## **Corresponding Member:**

Bosnia and Herzegovina



## 7th International Congress for Historical Metrology

25-28 September 1997

### Siegen University (Germany)

The 7<sup>th</sup> International Congress for Historical Metrology will take place at Siegen University from Thursday 25 (2pm) to Saturday 27 September (7pm); in addition an excursion to local historical sites has been organized for the Sunday afternoon.

The Congress will have as its main themes:

- Methodology and material structures of historical metrology;
- Historical metrology in Asia (China and Japan) and in ancient times;
- Historical metrology, mathematics, natural science and technique;
- Metrology and notions of value and money

Those wishing to attend or present a paper should contact the organizers,

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### **PUBLICATIONS**

classified by subject and number

International Recommendations International Documents Other publications

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Généralités		Fields of use of measuring instruments subject to verification	
R 34 (1979–1974)	60 FRF	Domaines d'utilisation des instruments de mesure assujettis à la vérification	
Accuracy classes of measuring instruments Classes de précision des instruments de mesurage		D 13 (1986)	50 FRF
R 42 (1981–1977)	50 FRF	Guidelines for bi- or multilateral arrangements on the recognition of: test results - pattern approvals -	
Metal stamps for verification officers Poinçons de métal pour Agents de vérification		verifications Conseils pour les arrangements bi- ou multilatéraux de	
<b>D 1</b> (1975)	50 FRF	reconnaissance des: résultats d'essais - approbations de modèles - vérifications	
Law on metrology Loi de métrologie		<b>D 14</b> (1989)	60 FRF
<b>D 2</b> (being printed - en cours de publication) Legal units of measurement Unités de mesure légales		Training of legal metrology personnel - Qualification - Training programmes  Formation du personnel en métrologie légale -	
• • • • • • • • • • • • • • • • • • •		Qualification - Programmes d'étude	
<b>D3</b> (1979)	60 FRF	<b>D 15</b> (1986)	
Legal qualitication of measuring instruments Qualification légale des instruments de mesurage		Principles of selection of characteristics for the examination of measuring instruments  Principes du choix des caractéristiques pour l'examen	
<b>D</b> 5 (1982)	OFRE	des instruments de mesure usuels	
Principles for the establishment of hierarchy schemes for measuring instruments		<b>D 16</b> (1986)	80 F.K.F
Principes pour l'établissement des schémas de hiérarchie des instruments de mesure		Principles of assurance of metrological control Principes d'assurance du contrôle métrologique	
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Principles of metrological supervision Principes de la surveillance métrologique		Pattern evaluation and pattern approval Essai de modèle et approbation de modèle	

<b>D 20</b> (1988) Initial and subsequent verification of measuring instruments and processes Vérifications primitive et ultérieure des instruments et processus de mesure	80 FRF	D 23 (1993) Principles of metrological control of equipment used for verification Principes du contrôle métrologique des équipements utilisés pour la vérification	80 FRF
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Road and rail tankers Evaluation des étalons de débitmétrie et des dispositifs	*	100 FRF	testing water meters	
Camions et wagons-cuernes utilises pour l'essat des compleurs d'eau			Evaluation des étalons de débitmétrie et des dispositifs	
	Camions et wagons-cuernes		utuises pour i essat des compleurs d'equ	

D 25 (1996)	60774	R 110 (1994)	8011
Vortex meters used in measuring systems for fluids Compteurs à vortex utilisés dans les ensembles de mesurage de fluides		Pressure balances Manomètres à piston	
D 26 (being printed - en cours de publication) Glass delivery measures - Automatic pipeties Mesures en verre à délivrer - Pipeties automatiques		Temperature Températures <sup>(2)</sup>	
Gas measurement Mesurage des gaz, <sup>(1)</sup>		R 18 (1989) Visual disappearing filament pyrometers Pyromètres optiques à filament disparaissant	60 FR
<b>R 6</b> (1989)	20 FK)	R 48 (1980–1978)	50 FR
General provisions for gas volume meters Dispositions générales pour les compteurs de volume de gaz		Tungsien ribbon lamps for calibration of optical pyrometers  Lampes à ruban de tungstène pour l'étalonnage	
<b>R 31</b> (1995)	NO FRE	des pyromètres optiques	
Diaphragm gas meters Compteurs de gaz à parois déformables		<b>R 75</b> (1988)  Heat meters	60 FR
R 32 (1989)	<b>60 FR</b> 1	rieat meters Compteurs d'énergie thermique	
Rotary piston gas meters and turbine gas meters Compteurs de volume de gaz à pistons rotatifs et compteurs de volume de gaz à turbine		R 84 (1989) Resistance-thermometer sensors made of platinum, copper or nickel (for industrial and commercial use) Capteurs à résistance thermométrique de platine, de cuivre ou de nickel (à usages techniques et commerciaux)	60 FR
Pressions (2)		D 24 (1996) Total radiation pyrometers Pyromètres à radiation totale	60 FR
<b>R 23</b> (1975–1973)	60 FRF	<b>P 16</b> (1991)	100 FF
Tyre pressure gauges for motor vehicles Manomètres pour pneumatiques de véhicules automobiles		Guide to practical temperature measurements	
<b>R 53</b> (1982)	60 FRF	AND AND THE PROPERTY OF THE STREET	
Metrological characteristics of elastic sensing elements used for measurement of pressure.  Determination methods		Electricité Électricité	
Caractéristiques métrologiques des éléments récepteurs élastiques utilisés pour le mesurage de la pression.		<b>R 46</b> (1980–1978)	80 14
Méthodes de leur détermination		Active electrical energy meters for direct connection of class 2	
<b>R 97</b> (1990)	60 FRF	oi ciass 2 Compteurs d'énergie électrique active à branchement direct de la classe 2	
Barometers Baromètres			
<b>R 101</b> (1991)	S.O.F.R.F	<b>D 11</b> (1994) General requirements for electronic measuring	
Indicating and recording pressure gauges, vacuum gauges and pressure vacuum gauges with elastic sensing elements (ordinary instruments)		instruments Exigences générales pour les instruments de mesure électroniques	
Manomètres, vacuomètres et manovacuomètres indicateurs et enregistreurs à élément récepteur élastique (instruments usuels)		Acoustics and vibration	
<b>R 109</b> (1993)	60 FRF	Accoustique et vibrations <sup>(1)</sup>	
Pressure gauges and vacuum gauges with elastic sensing elements (standard instruments) Manomètres et vacuomètres à élément récepteur élastique (instruments étalons)		R. 58 (being printed - en cours de publication) Sound level meters Sonomètres	
See also "Liquid measurement" D 25 – Voir aussi "Mesarage des liq     See also "Medical instruments" – Voir aussi "Instruments médical		R 88 (being printed - en cours de publication) Integrating-averaging sound level meters Sonomètres intégrateurs-moyenneurs	

	<b>R 102</b> (1992)	50 PRF	R 123 (being printed - en cours de publication)	
	Sound calibrators		Portable and transportable X-ray fluorescence spectro- meters for field measurement of hazardous	
	Calibreurs acoustiques Annex (1995)	80 FRF	elemental pollutants	
	Test methods for pattern evaluation and test report format		Spectromètres à fluorescence de rayons X portatifs et	
	Méthodes d'essai de modèle et format du rapport d'essai		déplaçables pour la mesure sur le terrain d'éléments : polluants dangereux	
	<b>R 103</b> (1992)	60 FRF	<b>D 22</b> (1991)	80 FRF
	Measuring instrumentation for human response		Guide to portable instruments for assessing airborne	
	to vibration Appareillage de mesure pour la réponse des individus aux		pollutants arising from hazardous wastes	
	vibrations		Guide sur les instruments portatifs pour l'évaluation des polluants contenus dans l'air en provenance des	
٠	R 104 (1993)	60 FRF	sites de décharge de déchets dangereux	
	Pure-tone audiometers Audiomètres à sons purs			
	Annex F (1997)	100 FRF	Physico-chemical measurements	
	Test report format Format du rapport d'essai		Mesures physico-chimiques	
	итали протиськи			
	Environment			
	Environnement		<b>R 14</b> (1995)	60 FRF
	R 82 (1989)	so FRF	Polarimetric saccharimeters Saccharimètres polarimétriques	
	Gas chromatographs for measuring pollution from			
	pesticides and other toxic substances		<b>R 54</b> (in revision - en cours de révision)  pH scale for aqueous solutions	
	Chromatographes en phase gazeuse pour la mesure des pollutions par pesticides et autres substances toxiques		Echelle de pH des solutions aqueuses	
	<b>R 83</b> (1990)	80 FRF	<b>R 56</b> (1981)	50 FRF
	Gas chromatograph/mass spectrometer/data system for analysis of organic pollutants in water		Standard solutions reproducing the conductivity of	
	Chromatographe en phase gazeuse équipé d'un spectro-		electrolytes Solutions-étalons reproduisant la conductivité	
	mètre de masse et d'un système de traitement de données		des électrolytes	
	pour l'analyse des polluants organiques dans l'eau		<b>R 59</b> (1984)	S0 FRF
	<b>R 99</b> (1991)	100181	Moisture meters for cereal grains and oilseeds	
	Instruments for measuring vehicle exhaust emissions Instruments de mesure des gaz d'échappement des véhicules		Humidimètres pour grains de céréales et graines oléagineuses	
	<b>R 100</b> (1991)	80 FRF	<b>R 68</b> (1985)	SOFRE
	Atomic absorption spectrometers for measuring metal		Calibration method for conductivity cells	SVIRE
	pollutants in water  Spectromètres d'absorption atomique pour la mesure		Méthode d'étalonnage des cellules de conductivité	
	des polluants métalliques dans l'eau		<b>R 69</b> (1985)	50 FRF
	R 112 (1994)	80 FRF	Glass capillary viscometers for the measurement of	
	High performance liquid chromatographs for measure-		kinematic viscosity. Verification method	
	ment of pesticides and other toxic substances Chromatographes en phase liquide de haute performance		Viscosimètres à capillaire, en verre, pour la mesure de la viscosité cinématique. Méthode de vérification	
	pour la mesure des pesticides et autres substances toxiques			
	<b>R 113</b> . (1994)	80 F.K.F.	R 70 (1985)	50 FRF
	Portable gas chromatographs for field measurements		Determination of intrinsic and hysteresis errors of gas analysers	
	of hazardous chemical pollutants		Détermination des erreurs de base et d'hystérésis des	
	Chromatographes en phase gazeuse portatifs pour la mesure sur site des polluants chimiques dangereux		analyseurs de gaz	
	· · · · · · · · · · · · · · · · · · ·		<b>R 73</b> (1985)	50 FRF
	R 116 (1995) Inductively coupled plasma atomic emission spectro-		Requirements concerning pure gases CO, CO <sub>2</sub> , CH <sub>4</sub> , H <sub>2</sub> ,	
	meters for measurement of metal pollutants in water		O <sub>2</sub> , N <sub>2</sub> and Ar intended for the preparation of reference gas mixtures	
	Spectromètres à émission atonique de plasma couplé		Prescriptions pour les gaz purs CO, CO <sub>2</sub> , CH <sub>4</sub> , H <sub>2</sub> , O <sub>2</sub>	
	inductivement pour le mesurage des polluants métalliques dans l'eau		N, et Ar destinés à la préparation des mélanges de gaz de référence	

<b>R 92</b> (1989)	60 FRF	<b>R 93</b> (1990)	60 FRE
Wood-moisture meters - Verification methods and		Focimeters	
equipment: general provisions Humidimètres pour le bois - Méthodes et moyens de		Frontofocomètres	
vérification: exigences générales		R 114 (1995)	80144
R 108 (1993)	60 FRF	Clinical electrical thermometers for continuous measurement	
Refractometers for the measurement of the sugar		Thermomètres électriques médicaux pour mesurage	
content of fruit juices  Réfractomètres pour la mesure de la teneur en sucre		en continu	
des jus de fruits ,		R 115 (1995)	80 FRF
R 121 (1996)	eo PAT	Clinical electrical thermometers with maximum device Thermomètres électriques médicaux avec dispositif	
The scale of relative humidity of air certified against saturated salt solutions		à maximum	
Échelle d'humidité relative de l'air certifiée par rapport		R 122 (1996)	o0 FRE
à des solutions saturées de sels		Equipment for speech audiometry	
R 124 (being printed - en cours de publication)		Appareils pour l'audiométrie vocale	
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Réfractomètres pour la mesure de la teneur en sucre des moûts de raisin		Secondary standard dosimetry laboratories for the calibration of dosimeters used in radiotherapy	
,		Laboratoires secondaires d'étalonnage en dosimétrie pour	
<b>D 17</b> (1987)  Hierarchy scheme for instruments measuring the		l'étalonnage des dosimètres utilisés en radiothérapie	
viscosity of liquids		Testing of materials	
Schéma de hiérarchie des instruments de mesure de la viscosité des liquides		Essais des matériaux	
·		Approximation and security for spirit K 18 1 to morning in a	
Medical instruments		<b>R 9</b> (1972–1970)  Verification and calibration of Brinell hardness	OFRE
Instruments médicaux		standardized blocks	
<b>R 7</b> (1979–1978)	60 FRF	Vérification et étalonnage des blocs de référence de	
Clinical thermometers, mercury-in-glass with maximum device		dureté Brinell	•
Thermomètres médicaux à mêrcure, en verre,		R 10 (1974–1970)  Verification and calibration of Vickers hardness	ou FKI
avec dispositif à maximum		vermeation and calibration of vickers nardness standardized blocks	
<b>R 16</b> (1973–1970)		Vérification et étalonnage des blocs de référence de	
Manometers for instruments for measuring blood pressure (sphygmomanometers)		dureté Vickers	
Manomètres des instruments de mesure de la tension		R 11 (1974–1970)	60 FRF
artérielle (sphygmomanomètres)		Verification and calibration of Rockwell B hardness standardized blocks	
<b>R 26</b> (1978–1973)		Vérification et étalonnage des blocs de référence de dureté	
Medical syringes Seringues médicales		Rockwell B	
<b>R 78</b> (1989)	50 FRF	R 12 (1974-1970)	W.K.
Westergren tubes for measurement of erythrocyte		Verification and calibration of Rockwell C hardness standardized blocks	
sedimentation rate Pipettes Westergren pour la mesure de la vitesse		Vérification et étalonnage des blocs de référence de dureté	
de sédimentation des hématies		Rockwell C	
R 89 (1990)	80 FRF	R 36 (1980–1977)	60 FRF
Electroencephalographs - Metrological characteristics -		Verification of indenters for hardness testing machines Vérification des pénétrateurs des machines d'essai de dureté	
Methods and equipment for verification Electroencéphalographes - Caractéristiques métrologiques -		<b>R 37</b> (1981– <i>19</i> 77)	60 FRT
Méthodes et moyens de vérification		Verification of hardness testing machines (Brinell system)	
<b>R 90</b> (1990)	80 FRF	Vérification des machines d'essaí de dureté (système Brinell)	
Electrocardiographs - Metrological characteristics - Methods and equipment for verification		<b>R 38</b> (1981–1977)	60 FRF
Electrocardiographes - Caractéristiques métrologiques -		Verification of hardness testing machines (Vickers system)	
Méthodes et moyens de vérification		Vérification des muchines d'essai de dureté (système Vickers)	

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INTERNATIONAL RECOMMENDATIONS RECOMMANDATIONS INTERNATIONALES		R 22 (1975–1973) International alcoholometric tables (trilingual French-English-	150 FRF
R 4 (1970– <i>1972</i> ) Volumetric flasks (one mark) in glass	SOFIF	Spanish) Tables alcoométriques internationales (trilingue français- anglais-espagnol)	
Fioles jaugées à un trait en verre		R 23 (1975–1973)	60 FRF
R 6 (1989) General provisions for gos volume meters	80 FRF	Tyre pressure gauges for motor vehicles Manomètres pour pneumatiques de véhicules automobiles	
Dispositions générales pour les compteurs de volume de gaz		R 24 (1975–1973) Standard one metre bar for verification officers	50 FRF
R 7 (1979–1978) Clinical thermometers, mercury-in-glass with maximum device Thermomètres médicaux à mercure, en verre, avec	60 FRF	Mètre étalon rigide pour agents de vérification <b>R 26</b> (1978–1973)	50 FRF
dispositif à maximum		Medical syringes Seringues médicales	
<b>R 9</b> (1972– <i>1970</i> )  Verification and calibration of Brinell hardness	€0 FRF	R 29 (1979–1973)	50 FRF
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duraté Brinell  R 10 (1974–1970)	60 FRF	<b>R 30</b> (1981) End standards of length (gauge blocks)	60 FRF
Verification and calibration of Vickers hardness standardized blocks		Mesures de longueur à bouts plans (cales étalons)  R 31 (1995)	80 FRF
Vérification et étalonnage des blocs de référence de dureté Vickers		Diaphragm gas meters Compteurs de gaz à parois déformables	
R 11 (1974–1970) Verification and calibration of Rockwell B hardness	60 FRF	<b>R 32</b> (1989) . Rotary piston gas meters and turbine gas meters	60 FRF
standardized blocks Vérification et étalonnage des blocs de référence de dureté Rockwell B		Compteurs de volume de gaz à pistons rotatifs et compteurs de volume de gaz à turbine	
<b>R 12</b> (1974–1970)	60 FRF	<b>R 33</b> (1979–1973)  Conventional value of the result of weighing in air	50 FRF
Verification and calibration of Rockwell C hardness standardized blocks		Valeur conventionnelle du résultat des pesées dans l'air R 34 (19791974)	60 FRF
Vérification et étalonnage des blocs de référence de dureté Rockwell C		Accuracy classes of measuring instruments  Classes de précision des instruments de mesurage	
R 14 (1995) Polarimetric saccharimeters	oo FRE	<b>R 35</b> (1985)	80 FRF
Saccharimètres polarimétriques		Material measures of length for general use Mesures matérialisées de longueur pour usages généraux	
R 15 (1974–1970) Instruments for measuring the hectolitre mass of cereals Instruments de mesure de la masse à l'hectolitre des céréales	80 64	R 36 (1980–1977) Verification of indenters for hardness testing machines	60 FRE
R 16 (1973–1970)	50 FR	Vérification des pénétrateurs des machines d'essai de dureté <b>R 37</b> (1981-1 <i>977</i> )	(O fRF
Manameters for instruments for measuring blood pressure (sphygmomanometers)		Verification of hardness testing machines (Brinell system) Verification des machines d'essai de dureté (système Brinell)	
Manomètres des instruments de mesure de la tension artérielle (sphygmomanomètres)		R 38 (1981–1977)	O N
R 18 (1989) Visual disappearing filament pyrometers	o n	Verification of hardness testing machines (Vickers system) Vérification des machines d'essai de dureté (système Vickers)	
Pyromètres optiques à filament disparaissant		<b>R 39</b> (1981– <i>1977</i> ) Verification of hardness testing machines (Rockwell	OO PRE
<b>R 21</b> (1975–1973) Taximeters = Taximetres	60 FRE	systems B,F,T-C,A,N) Vérification des machines d'essai de dureté (systèmes Rockwell B,F,T-C,A,N)	
, 400		· · · · · · · · · · · · · · · · · · ·	

	<b>R 40</b> (1981–1 <i>977</i> ) Skindard graduated pipettes for verification officers Pipettes graduées étalons pour agents de vérification	60 FRF	R 51-2 (1996) Automatic catchweighing instruments. Part 2: Test report format Instruments de pesage trieurs-étiqueteurs à fonctionnement	300 FRF
,	R 41 (1981–1977) Standard burettes for verification officers Burettes étalons pour agents de vérification	60 FRF	automatique. Partie 2: Format du rapport d'essai  R 52 (1980)  Hexagonal weights, ordinary accuracy class	SO FRE
	R 42 (1981-1977) Metal stamps for verification officers Poinçons de métal pour agents de vérification	SO FRE	from 100 g to 50 kg Poids hexagonaux de classe de précision ordinaire, de 100 g à 50 kg	(A FRE
	R 43 (1981-1977) Standard graduated glass flasks for verification officers Fioles étalons graduées en verne pour agents de vérification	60 FRF	R 53 (1982) Metrological characteristics of elastic sensing elements used for measurement of pressure. Determination methods Caractéristiques métrologiques des éléments récepteurs élastiques utilisés pour le mesurage de lo pression. Méthodes	60 FRF
	R 44 (1985) Alcoholometers and alcohol hydrometers and thermometers for use in alcoholometry	50 FRF	de leur détermination  R 54 (in revision - en cours de révision)	
	Alcoomètres et aréomètres pour alcoal et thermomètres utilisés en alcoométrie		pH scale for aqueous solutions Echelle de pH des solutions aqueuses	
	R 45 (1980-1977) Casks and barrels Tonneaux et lutailles	50 FRF	R 55 (1981) Speedometers, mechanical odometers and chronotachographs for motor vehicles. Metrological regulations Compteurs de vitesse, compteurs mécaniques de distance	50 FRF
	R 46 (1980–1978) Active electrical energy meters for direct connection	80 FRF	et chronotachygraphes des véhicules automobiles. Réglementation métrologique	
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	R 47 (1979-1978) Standard weights for testing of high capacity weighing machines	60 FRF	R 58 (being printed - en cours de publication) Sound level meters Sonomètres	
	Poids étalons pour le contrôle des instruments de pesage de portée élevée		R 59 (1984)	80 FRF
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	Lampes à ruban de lungstène pour l'étalonnage des pyromètres optiques		R 60 (1991) Metrological regulation for load cells Réglementation métrologique des cellules de pesée	80 FRF
	R 49 (in revision - en cours de révision) Water meters intended for the metering of cold water Compteurs d'eau destinés au mesurage de l'eau froide		Annex (1993)	80 FRF
	R 50-1 (1997)	1.50 FRF	Test report format for the evaluation of load cells Format du rapport d'essai des cellules de pesée	
	Continuous totalizing automatic weighing instruments (Belt weighers). Part 1: Metrological and technical requirements - Tests		R61-1 (1996) Automatic gravimetric filling instruments.	150 FRF
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	Automatic catchweighing instruments, Part 1: Metrological and technical requirements - Tests	WIN .	Caractéristiques de performance des extensomètres métalliques à résistance	
	Instruments de pésage trieurs-étiqueteurs à fonctionnement automatique. Partie 1: Exigences métrologiques et		R 63 (1994) . Petroleum measurement tables	50 FRF
	techniques - Essais		Tables de mesure du pétrole	

R 64 (1985) General requirements for materials testing machines	2 <b>50 FRF</b>	R 79 (being printed - en cours de publication) Information on package labels Etiquetage des préemballages	
Exigences générales pour les machines d'essai des matériaux			
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R 66 (1985) Length measuring instruments Instruments mesureurs de longueurs	AO FRF	Measuring devices and measuring systems for cryogenic liquids (including tables of density for liquid argon, helium, hydrogen, nitrogen and oxygen) Dispositifs et systèmes de mesure de liquides cryogéniques	
R 68 (1985) Calibration method for conductivity cells Méthode d'étalonnage des cellules de conductivité	<b>50 m</b> F	(comprend tables de masse volumique pour argon, hélium, hydrogène, azote et oxygène liquides)	
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R 70 (1985)  Determination of intrinsic and hysteresis errors of gas analysers  Détermination des erreurs de base et d'hystérésis des  analyseurs de gaz	<b>30 FNF</b>	Gas chromatograph/mass spectrometer/data system for analysis of organic pollutants in water Chromatographe en phase gazeuse équipé d'un	
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Fixed storage tanks, General requirements Réservoirs de stockage fixes. Prescriptions générales		R 84 (1989) Resistance thermometer sensors made of platinum, copper	60 FRF
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