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Metrology at Philip Morris Europe*

by

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SUMMARY

The importance of the metrology function at Philip Morris Europe (PME), a multinational organisation producing at over 40 sites in the European, Middle Eastern and African Regions is presented. Standardisation of test methods and equipment as well as the traceability of calibration gauges to the same reference gauge are essential in order to obtain comparable results among the various production centers. The metrology function as well as the qualification of instruments and the drafting of test and calibration operating procedures for this region are conducted or co-ordinated by the Research and Development Department in Neuchâtel, Switzerland.
In this paper the metrology function within PME is presented based on the measurement of the resistance to draw for which the PME R&D laboratory is accredited (ISO/CEI 17025), as both a calibration and a testing laboratory. The following topics are addressed in this paper: traceability of calibration standards to national standards; comparison of results among manufacturing centres; the choice, the budget as well as the computation of uncertainties. Furthermore, some practical aspects related to the calibration and use of the glass multi-capillary gauges are discussed. [Beitr. Tabakforsch. Int. 19 (2000) 171–175]

ZUSAMMENFASSUNG

Die Bedeutung der Funktion der Metrologie bei Philip Morris Europe (PME), einem multinationalen Unternehmen mit mehr als 40 Produktionsstätten in Europa, im Mittleren Osten und in Afrika, wird dargestellt. Die Standardisierung der Testmethoden und der Geräte und die Anpassung der Kalibrierungen an dasselbe Eichmaß sind von grundlegender Bedeutung, um vergleichbare Resultate in den verschiedenen Produktionsstätten zu erzielen. Die Funktion der Metrologie sowie die Angleichung der Instrumente und der Entwurf von Test- und Eichverfahren für diese Region wird von der Abteilung für Forschung und Entwicklung in Neuchâtel in der Schweiz durchgeführt oder koordiniert.
Diese Arbeit beschreibt die Bedeutung der Metrologie innerhalb des Unternehmens anhand der Messung des Zugwiderstands, wofür das Forschungs- und Entwicklungslabor PME R&D gemäß ISO/CEI 17025 als Eich- und Versuchslabor akkreditiert ist. Die folgenden Themen werden in der Arbeit untersucht: Das Anpassen der Eichungen an nationale Standards, der Vergleich der Ergebnisse in den unterschiedlichen Produktionsstätten des Unternehmens, die Auswahl, die Kosten, das Abwägen der Unsicherheitsfaktoren sowie einige praktische Aspekte im Bereich der Eichungen und des Gebrauchs multikapillarer Manometer aus Glas. [Beitr. Tabakforsch. Int. 19 (2000) 171–175]

RESUME

L'importance de la fonction métrologie chez Philip Morris Europe (PME) une entreprise multinationale avec une production répartie sur plus de 40 sites en Europe, Moyen Orient et Afrique, est présentée. La standardisation des méthodes d'essai et des instruments, ainsi que la traçabilité et le raccordement des étalons à une même référence, sont indispensables pour assurer des résultats comparables entre les différents centres de production. La fonction métrologie ainsi que l'homologation des instruments et l'élaboration des procédures d'essai et d'étalon-

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nage pour cette zone sont assurés par le Département de Recherche et Développement à Neuchâtel, Suisse.
Cette communication décrit la fonction métrologie au sein de l'entreprise en utilisant dans ce cas de figure la mesure de la résistance au tirage pour laquelle le laboratoire de PME R&D est accrédité selon la norme ISO/CEI 17025 en tant que laboratoire d'étalonnage et laboratoire d'essai. Cette communication aborde le raccordement des étalons à des étalons nationaux, la comparaison des résultats avec ceux obtenus par des usines affiliées, le choix, le bilan et le calcul des incertitudes ainsi que quelques aspects pratiques liés à l'étalonnage et à l'utilisation de jauges multicapillaires en verre. [Beitr. Tabakforsch. Int. 19 (2000) 171–175]

INTRODUCTION

Standardisation of test methods and instruments as well as traceability of calibration standards to the same reference standard are required in order to obtain comparable results among the various production centres of Philip Morris Europe (PME). The metrological function, i.e. the qualification of instruments, the drafting of standard test and calibration operating procedures, the organisation of interlaboratory collaborative tests, the audits as well as the co-ordination of expert working groups is carried out by the Research and Development Department in Neuchâtel, Switzerland.
The resistance to draw (RTD) of cigarettes and the pressure drop of filter rods, two important parameters for the tobacco industry, are chosen as an example for reviewing the metrology function within PME.
Philip Morris Europe R&D have obtained accreditation according to ISO/CEI 17025 both as a calibration laboratory for RTD-glass-multicapillary-gauge calibration and as a testing laboratory for RTD measurements.

CALIBRATION PROCEDURE

The equipment described below is required for the calibration of glass multicapillary gauges (RTD gauges) (1, 2):

- Laboratory air conditioned to 60 ± 5% RH and 22 ± 2 °C (3)
- Reference glass multicapillary gauges certified by the Swiss Federal Office of Metrology
- Source of volumetric flow of 17.5 ± 0.3 cm^3/s generated by a critical flow orifice system
- Certified manometer for the measurement of differential pressure
- Flow measuring instrument having an expanded uncertainty of ± 0.09 cm^3/s at the 17.5 cm^3/s level
- Certified barometer for measuring atmospheric pressure

The first step of the RTD-gauge calibration process is to check the constant air flow of 17.5 cm^3/s using a flow-measuring instrument to which an RTD gauge is connected, firstly one of 500 mm water gauge (WG) and secondly one of 100 mm WG. The difference in flow between the two measurements should not be larger than 0.3 cm^3/s.
Before proceeding with the measurements it is necessary to ensure that the RTD gauges to be calibrated are clean and are fitted with plastic tubing that covers one end of the gauge during the entire measurement process. This plastic tubing allows manipulation of the RTD gauges without direct contact with them, thus preventing warming up of these gauges that may give rise to erroneous results.
In order to ascertain reliable measurements the equipment as well as the RTD gauges must be placed in the laboratory at least one hour before measurements start in order to allow for thermal equilibrium to be reached. The RTD gauges must be placed away from any heat sources that could perturb their thermal equilibrium.
It is necessary that the laboratory conditions be within the tolerances stated above during the equilibration period, as well as during the measurements.
The manometer is zeroed before any RTD gauge is connected to it. The certified RTD gauges are connected and measured one after the other starting with the gauge having the higher RTD. The same procedure is repeated for each RTD gauge to be calibrated. Each gauge is measured three times.

Calculation of RTD values and checking of results

The certified reference RTD gauges can be measured at an atmospheric pressure that is different from the one prevailing during the determination of their nominal values by the Swiss Federal Office of Metrology. The RTD measurements done at PME R&D are correlated with the available nominal values by means of a weighted linear regression. A weighted linear regression is necessary due to the fact that the uncertainty of the gauges is not constant but it is a function of the RTD level.
$$\mathrm{RTD}_{\mathrm{measured}} = a_w + b_w \cdot \mathrm{RTD}_{\mathrm{certified}}$$
The weighted linear regression uses a weight at each level of RTD that is calculated as follows:

$$w_i = \frac{1}{s_{y_i}^2/n_i + b^2 \cdot s_{x_i}^2}$$

where:

- n_i : number of measurements (equal to 3)
- $s_{y_i}^2$: variance of the three measurements of the certified gauges
- b : slope of the linear regression (non weighted)

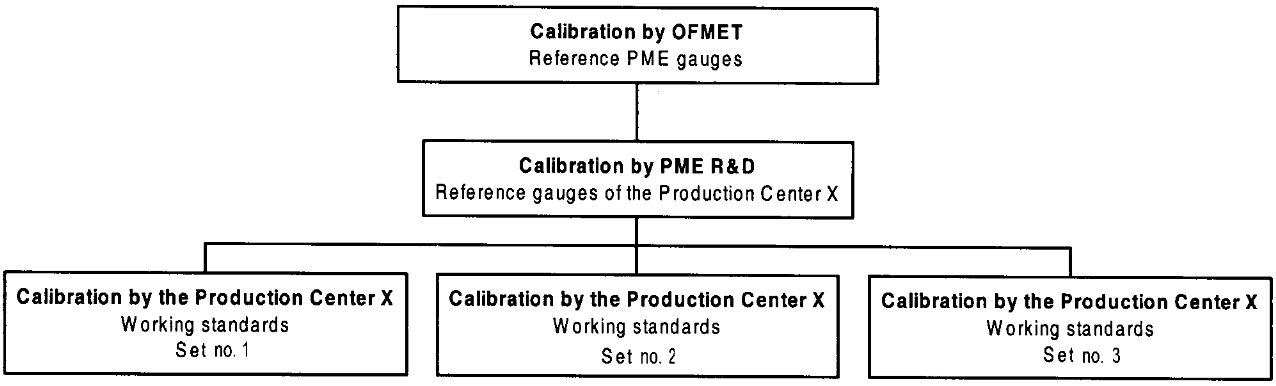

Figure 1.
Schematic representation of RTD-calibration-standard traceability (OFMET is the abbreviation in French for the Swiss Federal Office of Metrology)

Note: The Excel sheet that does the calculations performs as well a non-weighted linear regression in order to obtain the slope that is subsequently used in the calculation of the weights

$s_{x_i}^2$: variance of certified gauges obtained from the uncertainty indicated in the calibration certificate

The RTD value of the gauge to be calibrated is obtained by fitting the values of the three measurements into the weighted regression line obtained previously via measurements of the certified reference gauges. The final result for each gauge is then the average of the three calculated values.

A check of the measurements is conducted based on the obtained r^2 (coefficient of determination) as well as the value of the standard deviation of the three measurements of each gauge. The r^2 must not be lower than 0.99998. The standard deviation of the three measurements must be lower than a maximum value. The maximum values at each level are defined based on historical measurements having an $r^2 > 0.99998$.

Calculation of measurement uncertainty during the calibration of the RTD gauges

The calculation of uncertainties is performed based on the guidelines described in the ISO and EAL (European Cooperation for Accreditation of Laboratories) guides (4–6). The total variance is given by:

$$s_{\text{total}}^2 = \frac{s_{w-x'}^2}{3} + s_{\text{cert-gauge}}^2 + s_{\text{man}}^2$$

$$+ \frac{s_{w-y/x}^2}{b_w^2} \cdot \left(\frac{1}{W} + \frac{(\bar{X}' - \bar{X}_w)^2}{\mathrm{SS}_w} \right)$$

where:

$s_{w-x'}^2$: variance of the three measurements of the gauge to be certified

$s_{\text{cert-gauge}}^2$: variance of the certified gauge of an equivalent level

Calculation of the standard deviation associated with the manometer uncertainty

The calibration certificate of the manometer indicates the uncertainty at a 95% confidence level as a percentage of the measured value, with a stated minimum uncertainty. The standard deviation associated with this uncertainty is:

$$s_{\text{man}} = \frac{\mathrm{Max}\left(X \cdot \frac{\text{Measured value}}{100} ; Y \right)}{2}$$

where:

X : uncertainty at 95% confidence level given in the calibration certificate as a percentage of the measured value

Y : minimum uncertainty stated in the calibration certificate

Calculation of variance obtained from the weighted linear regression

$$\frac{s_{w-y/x}^2}{b_w^2} \cdot \left(\frac{1}{W} + \frac{(\bar{X}' - \bar{X}_w)^2}{\mathrm{SS}_w} \right)$$

where:

$s_{w-y/x}^2$: residual variance of weighted regression

b_w : slope of the weighted linear regression

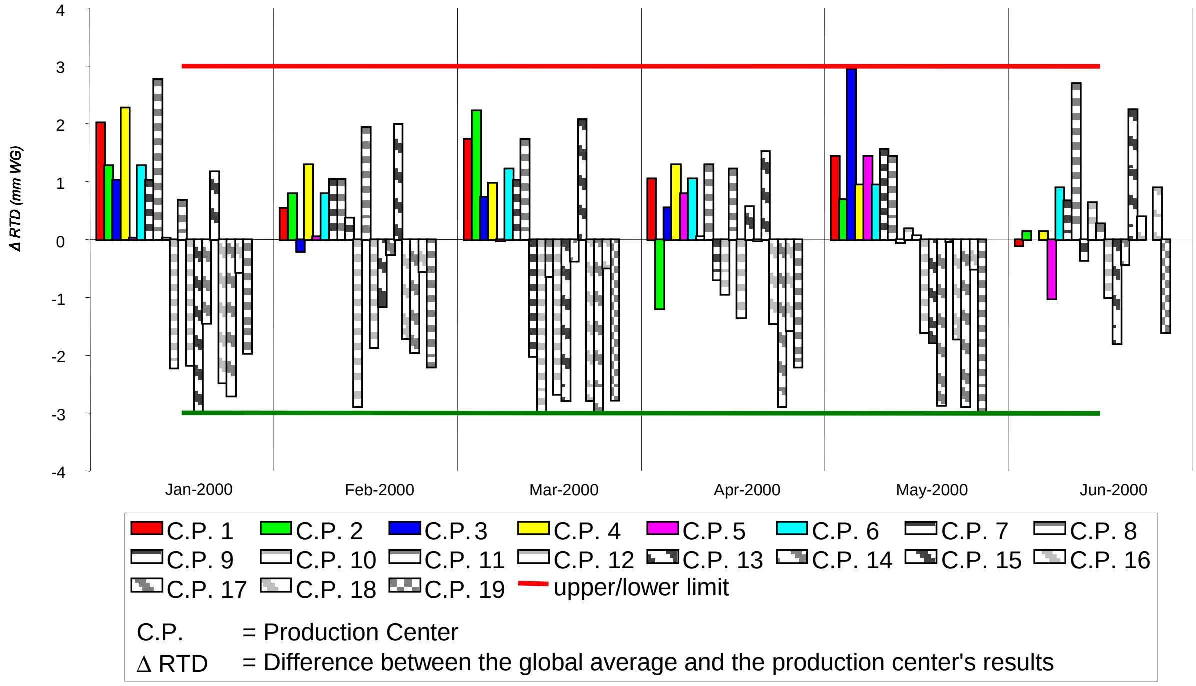

Figure 2.
Interlaboratory collaborative test for RTD measurements

W	:	sum of weights; $W = \Sigma w_i$
$\bar{X}'$	:	average of the calculated RTD values; $X' = (Y' - a_w)/b_w$
$\bar{X}_w$	:	weighted average; $\bar{X}_w = \Sigma(w_i \cdot X_i)/W$
SS_w	:	sum of squares of errors for $\bar{X}$; $SS_w = \Sigma w_i \cdot (X_i - \bar{X}_w)^2$

The expanded uncertainty at a 95% confidence level, calculated on the basis of s_{total} obtained from the equation described above, is between 2 to 3 mm WG in the usual range of measurement (100 to 800 mm WG).

DISCUSSION

The PME reference RTD calibration standards, i.e. the reference glass multicapillary gauges, are certified by the Swiss Federal Office of Metrology every two years thus ensuring traceability to the national standards. These calibration standards are of the highest metrological quality available in Switzerland owing to their direct calibration using the Swiss Federal Office of Metrology's equipment.

The RTD gauges calibrated and certified by the accredited laboratory of PME R&D are distributed to the various production centres. The frequency of checking of the gauge-calibration by PME R&D is specified in the appropriate regional directives. These RTD gauges are the reference RTD calibration standards, available in each production centre. They are used by the production centres solely for the calibration of the working standards that are used regularly for calibrating and checking the RTD measuring instruments (*see* Figure 1). These calibrations of working standards are conducted within each production centre using the procedure described in the corresponding internal PME method.

Checks and follow up of results are done by means of regular interlaboratory collaborative tests. Weekly measurements are conducted on a monitor sample prepared in advance specially for these interlaboratory tests. These tests are conducted using an instrument that has been designated as a reference instrument and that is located, most of the times, in a laboratory and not on the production floor. The results are then reported to PME R&D who calculate monthly averages, graph and comment the results and post these reports on the company intranet (see Figure 2). This allows the various production centres to check their results as well as to compare them with the global average. It is up to the production centre to ensure that the RTD results obtained with the other instruments available on site are comparable to those of the reference instrument.

A laboratory having a metrological function within PM USA covers the region of the Americas as well as part of Asia and Australia. A comparison of the reference calibration standards utilised by that laboratory with those of PME R&D showed a maximum difference of 1 mm WG, a difference that is within the uncertainty obtained at PME R&D or at the laboratory of PM USA. Based on these results and taking into account the stand-

ardisation of test methods within Philip Morris, worldwide standardisation of RTD measurements has been achieved.
Traceability of the RTD calibration standards utilised by PME to Swiss national standards as well as qualification of all RTD instruments are necessary in order to maintain a high quality of results. This is a prerequisite for obtaining consistent results within Philip Morris but also comparable results with PME's suppliers. However, as described above, some basic precautions should be taken in order to ensure reliable RTD results (cleaning of the gauges, thermal stability, etc).

CONCLUSION

The metrological function co-ordinated by the R&D laboratory enables PME to maintain measurement-result reliability at the various production centres by means of a programme of calibration-standard certification as well as regular interlaboratory tests. This allows for local testing of the product with respect to its conformity to the specifications, the ultimate goal being the satisfaction of our consumers by offering them consistently high-quality products.

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