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CHARACTERIZATION OF NEW TEMPERATURE SENSOR BASED ON QUARTZ TUNING FORK OSCILLATOR

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Abstract – A new temperature system based on low cost quartz tuning forks, originally designed to be used in wristwatches, has been arranged for temperature measurements in a wide cryogenic temperature range from 77 K to 273 K.
The present paper concerns small quartz crystal tuning forks, with a fundamental frequency of 32768 Hz, as a temperature sensor by measuring the resonance frequency shift caused by an external temperature. Special attention has been given to ~~the~~ methodology, equipment and design to improve the stability in time of the output signal of the quartz tuning fork temperature sensor. After temperature calibration, a statistical study has been performed to estimate the parameters/coefficients of the fitted curves by using a Weight Total Least Squares “WTLS” method. The chi-square test has also been considered as an indicator of the agreement between the observed data and the predicted values.
Higher-order polynomial curves seem to be the ”best fits” for the experimental data provided by the temperature system. The study also shows a significant statistical improvement by introducing the fractional degree of 0.5 in the regression curves, in particular for the third degree polynomial regression. Moreover, the fractional polynomial curve results in a better smoothing of the experimental irregularities encountered at low temperatures.

Keywords: Thermometer, Quartz tuning fork, Temperature measurement

1. INTRODUCTION

Continuous monitoring and checking of reliable temperature measurements are needed in every field: energy, health, food and oil industry, concerning research, quality control and improvement of production processes.

Concerning temperature measurements, a wide number of sensors is available among which thermocouples, thermistors, and RTDs; all of them infer temperature by sensing some changes in a physical characteristic.

Cernox^{TM} resistance temperature sensors (CXRTs) and platinum resistance thermometers Pt 100 were used together at INRiM for checking the temperature in the framework of the experiment concerning the density measurements of liquefied natural gas (LNG), in the temperature range from 100 K to 111 K, at pressures up to 6 bar (0,6 MPa), within the EMRP-ENG 03 “Metrology of LNG” (2010 – 2013) [1].

During this activity, a cognitive investigation was also performed on commercially available “Quartz crystal tuning forks”, having a fundamental frequency of 32768 Hz in order to assess their changing with the ambient conditions, in a wide range of temperature below 273 K and with the purpose to be used as low cost transducer for density and temperature measurements.

Quartz thermometers were firstly introduced by the Hewlett-Packard company as high-precision, high-accuracy temperature sensors, capable to operarate over a wide temperature range [2].

In the present work the characterization of quartz fork thermometer deals with the design, methodology and equipment of a new temperature system based on quartz tuning fork, as well as with the characterization and the study of the best calibration curve.

2. THEORY

Due to its high stability, precision, and low power consumption, the quartz crystal tuning fork has become a valuable basic component for time–frequency standard in wristwatches, where the insensitivity to accelerations is an additional advantage. It is also successfully used in a large number of other additional applications as gyroscopes, micro balances, gas sensors and scanning probe microscopy to measure surface topographies with a resolution of down to several angstroms [3].

The quartz tuning fork is a bimorph cantilever based on the piezoelectric properties of quartz. The principle of operation of a crystal tuning fork is the same of the metal tuning fork. After applying an electric signal, corresponding to the stroke applied on the metal tuning fork, its tines oscillate periodically with elastic deformations that do not damage the quartz according to the fundamental resonance frequency.

The resonance frequency f_0 of a quartz crystal tuning fork can be approximately expressed by [4]:

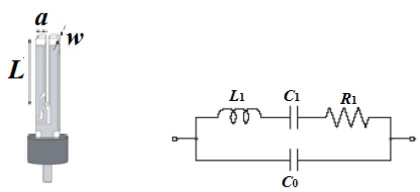

Figure 1. The equivalent circuit of the quartz crystal tuning fork.

$$f_0 \cong \frac{1.76a}{2\pi L^2}\sqrt{\frac{E}{\rho}} \qquad (1)$$

where, L is the prongs length of the tuning fork, a is their thickness, E is the Young modulus of quartz, and ρ is its density (ρ = 2650 kg/m^3). It depends mainly on the material from which it is made, the shape and size of its prongs.

By virtue of the piezoelectric properties, the tuning fork can be driven by subjecting it to a mechanical force or exciting it electrically. If a voltage is applied to the electrodes of the tuning fork its behaviour can be modeled as a series resistance-inductor-capacitor circuit based on a serial RLC circuit with a parallel capacitor (Butterworth-Van Dyke model), Figure 1. This model consists of two arms in parallel with one other. The "static arm" consists of a single capacitance C_0 (also referred to as the shunt capacitance). Herein this capacitance includes the capacitance of the bare crystal and the shunt one of its packaging. The "motional arm" consists of the series combination of a resistance R_1, an inductance L_1 and a capacitance C_1. The resulting characteristics due to these components are generally specified on a series resonance, where C_1 resonates with inductance L_1 at the crystals operating frequency. This frequency is called the crystals series frequency, f_0

$$f_0 \cong \frac{1}{2\pi\sqrt{L_1 C_1}} \qquad (2)$$

as well as this series frequency, there is a second frequency, f_1 established as a result of the parallel resonance created when L_1 and C_1 resonates with the parallel capacitor C_0

$$f_1 \cong \frac{1}{2\pi\sqrt{L_1\frac{C_0 C_1}{C_0+C_1}}} \qquad (3)$$

Generally, the capacitor $C_0 \gg C_1$ so that the fundamental oscillating frequency for a quartz tuning crystal is much more stronger or pronounced. At the resonant frequency, f_0, the reactances of the equivalent capacitor C_1 and the inductor L_1 are equal, and consequently the current is maximum.

2.1. Temperature effects

In general, the "amount" of the frequency variation due to temperature depends very much on changing of the elastic constants, the crystal cut and the crystal shape [5].

The most commonly used type of tuning fork crystal is the AT-cut crystal, where the quartz blank is in the form of a thin plate cut at a nominal angle of about 35° 15' to the optical axis of the crystal. Generally, the AT-cut crystal has a frequency-temperature characteristic described by a third-order function of temperature, which inflection point lies between 25 °C and 35 °C of the form given by

$$f(T) = f_0(T_0)(\alpha_0 + \alpha_1\Delta T + \alpha_2\Delta T^2 + \alpha_3\Delta T^3) \qquad (4)$$

where $\Delta T = (T - T_0)$, $f(T)$ any ~~one of~~ frequency at measured temperature, $f(T_0)$ is the resonance frequency at the reference temperature, usually $T_0 = 25\,°C$ (turnover temperature), and α_i, with $i = 0, 1, 2$ and 3, are the coefficients which depend on physical properties of the crystal including the angle of the cut, the ratio of dimensions, the order of overtone, the shape of plate, and the type of mounting.

In comparison to some other crystal cuts, the tuning fork crystals exhibit a typical frequency stability usually given from vendor's datasheet approximated as -0.035 ppm/°C.

3. EXPERIMENTAL

3.1. Tuning fork temperature sensor

The MS1V-T1K 32.768 kHz crystal tuning fork by Micro-Cristall (Switzerland) has been investigated as temperature sensor, by measuring the resonance frequency shift caused by an external temperature.

Figure 2 shows the picture of the MS1V-T1K crystal tuning fork and Table 1 lists its electrical characteristics.

The tuning fork component appears as a square-bodied 2.0 x 8.1 x 2.0 mm metal-can package with formed leads intended for surface mounting and reflow soldering. The quartz crystal tuning fork resonator is contained inside ~~to~~ it under low vacuum. It is a high-quality tuning fork, manufactured with a precision high-volume photolithographic process in two prongs connected at one end of crystalline quartz plate with thin-film metal electrodes deposited on the faces, operating at the fundamental mode of vibration at 32 kHz.

3.2. The Temperature measurement system

The architecture of the new temperature measurement system is shown in Fig. 3.

According to design, two independent crystal sensors XTAL1 and XTAL2 are driven by a swept sinusoidal waveform whose frequency f can be digitally programmed via serial data input in the range 25 kHz and 33 kHz (Agilent 33220A). The low-level currents of the vibrating crystals are converted to a voltage by a transimpedance amplifiers with a 5 kΩ feedback resistor. A digital analog converter - DAC (National Instruments NI-9215) provides to sample and convert simultaneously the analog waveforms of the two sensors into digital values, thus preserving the relative phase information of the signals on both analog inputs. The Personal Computer (PC) controls the temperature measurement system by a Labview program, communicating with the signal generator and making necessary acquisition and subsequent processing the

Figure 2. The MS1V-T1K 32768 kHz crystal tuning fork.

Frequency	**32768**	**kHz**
Series resistance typ./max. RS	45 / 60	kΩ
Motional capacitance typ., C1	2.1	fF
Static capacitance typ., C0	0.9	pF
Turnover temperature, T0	25 +/-5	°C
Frequency vs. temperature, Δf/f0	-0.035 ppm/°C² $(T - T0)^2$ +/-10%	ppm

Table 1. MS1V-T1K electrical characteristic at 25 °C.

measured values, and finally the results are displayed on the monitor screen.

The measurement procedure is mostly automated. The PC screen displays the Frontal panel which allows to perform the following tasks (Figure 4):

1. Set the sweep frequency range through the commands "Freq START" and "Freq STOP";

2. Display the start and end frequency ("Start SWEEP" and "Stop SWEEP" and the number of the repeated cycle ("Cycle counter");

3. Display the average of five values of resonance concerning the Amplitude and the frequency for both crystals ("FORK 1 AMP ", “FORK 1 freq” and "FORK 2 AMP” and “FORK 2 freq”), respectively;

4. Display the current value of amplitude and frequency during each cycle for both crystal ("Freq 1.1", “Amp 1.1" and "Freq 2.1”, and "Amp 2.1"), respectively;

5. Show for both crystals the typical tuning fork responses, the relationship between the output voltage V and the frequency f regarded as the vibration spectrum of tuning fork, respectively for five cycles;

and finally

6. Display the ratios between the amplitude values of XTAL 1 and XTAL 2 and the ratios between the resonance frequency values of XTAL 1 and XTAL 2.

The two XTAL1 and XTAL2 crystal sensors have been located in a small glass probe, connected through a cable to the junction box by BNC connector. The transimpedance amplifiers, the digital analog converter – DAC and all needed connections towards the Signal generator and the PC are contained in the electronic junction box.

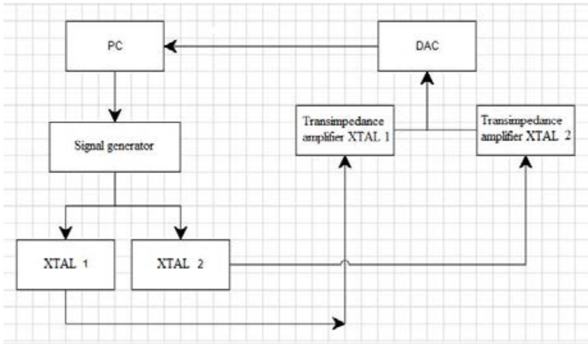

Figure 3. Simplified hardware block diagram of the temperature system.

3.3. The Temperature traceability

In order to use the designed temperature system based on quartz tuning fork for temperature measurements, one has to ensure that frequency measurements of the crystal sensors can be related to the temperature unit by means of a calibration procedure with associated measurement uncertainties. The calibration was performed after an artificial pre-aging of both sensors. Both crystals were stressed in temperature several times in order to make them mechanically stable. The resonance frequencies for both crystal were checked for short-time drift in an ice melting point bath. The resonance differences resulted about 1 ppm at 0 °C among them.

The calibration procedure aims to determine the relation, between the ratio of the two crystal sensors resonance frequency and the corresponding temperature with associated measurement uncertainties in the range of temperature 0 °C and -186 °C (273 K - 77 K approx. the LN2 boiling temperature). A Pt100 calibrated reference thermometer (Lake Shore RP13613), traceable to Italian Istituto Nazionale di Ricerca Metrologica (INRiM) was used to this purpose.

During the calibration, the crystal XTAL1 and the calibrated thermometer were placed into copper block inside a Dewar and cooled with liquid nitrogen. Temperature and beat frequency readings were taken as the block cooled down and warmed up. Instead, the crystal XTAL2 was placed into an ice melting point bath for all the calibration time. The platinum resistance was connected by four wire to the Hart Scientific 1560 Black Stack thermometer readout. The temperature data was sent to PC through the RS-232 port.

4. RESULTS AND ANALYSIS

Figure 5 shows the experimental data concerning the frequency ratio of the actual resonance frequency of XTAL1 to the resonance frequency of XTAL2 at 0 °C in the temperature range from 77 K to 273 K, fitted by a fifth-order polynomial curve.

In this work, the calibration curve, fitting the frequency ratio values versus the absolute temperature T, was investigated among the class of functions of order k , i.e.:

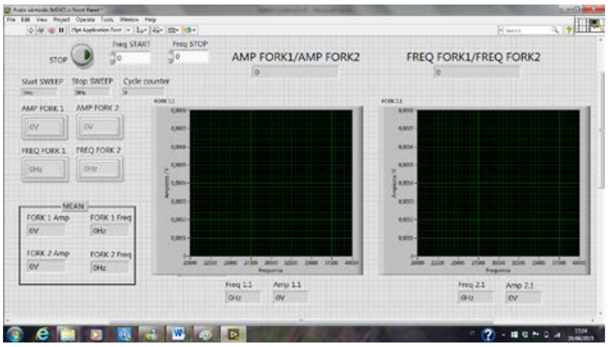

Figure 4. The PC front panel used to display and check the frequency measurements.

$$R(T) = \frac{f_{XTAL1}}{f_{XTAL2}} = \sum_{i=0}^{k} \beta_i T^i \qquad (5).$$

To estimate parameters/coefficients β_i and also to evaluate the associated uncertainty and the covariance matrix, the "Calibration Curves Computing – CCC Software" [6] has been applied. The CCC Software is an executable program for calibration problems, developed in MATLAB® environment, which was provided by the INRiM in the framework of the EMRP NEW04 project [7]. In order to fit n pairs (x, y) of measured values, where x is the independent/explanatory variable and y is the dependent/explained variable, the software can implement different regression techniques: OLS (Ordinary Least-Squares), WLS (Weighted Least-Squares) and WTLS (Weighted total Least-Squares) regressions. The model curves which can be fitted to the data are polynomial functions with exponents among $-5 \leq i \leq 5$. Among such integer exponents, fractional exponents -0.5 and 0.5 are also included in the list. Once the elaboration is performed, the software provides the following output: a) Regression curve, b) Parameter estimates and associated standard uncertainty, c) Normalized (Reduced) chi-squared value of the regression, d) Covariance matrix associated with the parameter estimates, and e) Fitted y values with the associated covariance matrix. The WTLS method has been taken into account in the present work, because both the observation vector $\boldsymbol{R}$ of the frequency ratio and the vector $\boldsymbol{T}$ of the temperature values are affected by non-negligible uncertainties.

Covariance information about the 53 available T values was given in the form of an associated 53 x 53 covariance matrix $\boldsymbol{V_T}$ having on its diagonal the square of the uncertainty (0.015 °C) of the temperature values, and as off-diagonal elements the covariances between the i^{th} and j^{th} values, constructed from an assumed correlation coefficient equal to 0.9. The 53 x 53 covariance matrix $\boldsymbol{V_R}$ associated with the frequency ratio observations was also provided to the software as an input, in which the uncertainty associated to each value was assumed equal to 3 ppm and again a correlation coefficient of 0.9 was chosen for determining the covariance terms.

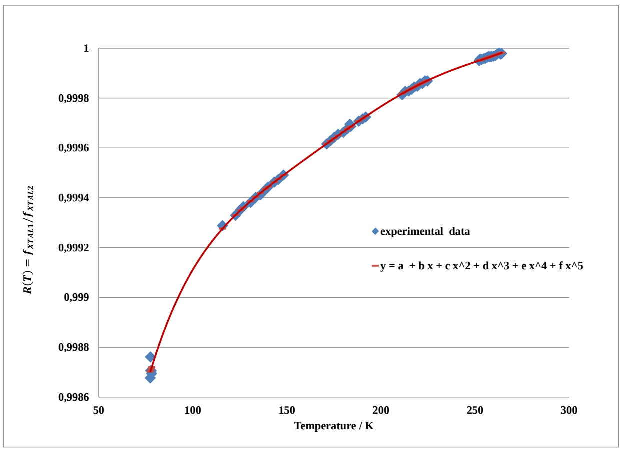

Figure 5. Calibration results in the temperature range 77 K and 273 K fitted by a WTLS regression polynomial curve of the fifth order.

4.1. The goodness of fit

The software has been launched several times by selecting different degrees/exponents of the regression curve to be fitted to the experimental data.

Table 2 shows the regression results of the polynomial curves with degree 3, 4 and 5, respectively. Moreover, fractional exponent 0.5 has been included also for the curves having degrees 3 and 4.

In order to perform a graphical residual analysis, the relative differences between the observed responses and the corresponding prediction of the regression functions are plotted in Figure 6. Moreover, the chi-square statistic was also considered as an indicator of the agreement between the observed data and the predicted values, as well as between the estimated variance of fit and the input uncertainties. The model is consistent with the observed values, if the expected value of the reduced chi-square $\tilde{\chi}^2$ (chi-square per degrees of freedom) results close to 1:

$$\tilde{\chi}^2 = \frac{\chi^2}{n-p} \cong 1 \qquad (6)$$

where n is the number of observations and p is the number of the model parameters.

Looking at Table 2, we can observe a statistical significant improvement by introducing the fractional degree of 0.5 in the regression curve, in particular for the third degree polynomial regression. This fact is evidenced by ~~the~~ reducing at 11.7 (almost 70 %) of the $\tilde{\chi}^2$ value and also it is well graphically evidenced by the residuals behaviour, red squares, in Figure 6. Moreover, the fractional polynomial curve appears to better smooth experimental irregularities at low temperatures.

The $\tilde{\chi}^2$ values around 10 do not appear unreasonably large in order to say that the considered models are sufficiently adequate. However, according to equation (6), a complete consistency test would be satisfied by appropriately enlarging the input uncertainties $\boldsymbol{V} = \begin{bmatrix} \boldsymbol{V_T} & \boldsymbol{0} \\ \boldsymbol{0} & \boldsymbol{V_R} \end{bmatrix}$, for example multiplying it by a constant factor h chosen so that expression (6) holds.

Fitted polynomial curve	Regression Result	
y = a + b x + c x^2 + d x^3	a = 0.99679 K	u(a) = 1.5e-05 K
	b = 3.44e-05 K^{-1}	u(b) = 2.4e-07 K^{-1}
	c = -1.39e-07 K^{-2}	u(c) = 1.5e-09 K^{-2}
	d = 2.06e-10 K^{-3}	u(d) = 2.8e-12 K^{-3}
	Reduced Chi square/(n-p) = 37,47	
y = a + b x^0,5 + c x + d x^2 + e x^3	a = 0.9818 K	u(a) = 4.2e-04 K
	b =3.9e-03 K$^{-0.5}$	u(b) = 1.1e-04 K$^{-0.5}$
	c = -27.06e-05 K^{-1}	u(c) = 8.5e-06 K^{-1}
	d = 5.1e-07 K^{-2}	u(d) = 1.8e-08 K^{-2}
	e = -5.7e-10 K^{-3}	u(e) = 2.2e-11 K^{-3}
	Reduced Chi square/(n-p) = 11,66	
y = a + b x + c x^2 + d x^3 + e x^4	a = 0,99517 K	u(a) = 4.9e-05 K
	b = 8,0e-05 K^{-1}	u(b) = 1,4e-06 K^{-1}
	c = -5,9e-07 K^{-2}	u(c) = 1,3e-08 K^{-2}
	d = 2,07e-09 K^{-3}	u(d) = 5,4e-11 K^{-3}
	e = -2,72e-12 K^{-4}	u(e) = 7,9e-14 K^{-4}
	Reduced Chi square/(n-p) = 13,69	
y = a + b x^0,5 + c x + d x^2 + e x^3 + f x^4	a = 0,9623 K	u(a) = 0,0027 K
	b = 0,00985 K$^{-0.5}$	u(b) = 0,0008 K$^{-0.5}$
	c = -0,00079 K^{-1}	u(c) = 7,1e-05 K^{-1}
	d = 2,2e-06 K^{-2}	u(d) = 2,2e-07 K^{-2}
	e = -4,6e-09 K^{-3}	u(e) = 5,4e-10 K^{-3}
	f = 4,2e-12 K^{-4}	u(f) = 5,7e-13 K^{-4}
	Reduced Chi square/(n-p) = 10,73	
y = a + b x + c x^2 + d x^3 + e x^4 + f x^5	a = 0,9923 K	u(a) = 0,00025 K
	b =18.0e-05 K^{-1}	u(b) = 8,4e-06 K^{-1}
	c = -1,9e-06 K^{-2}	u(c) = 1,1e-07 K^{-2}
	d = 1,0e-08 K^{-3}	u(d) = 6,7e-10 K^{-3}
	e = -2,6e-11 K^{-4}	u(e) = 1,9e-12 K^{-4}
	f = 2,7e-14 K^{-5}	u(f) = 2,3e-15 K^{-5}
	Reduced Chi square/(n-p) = 10,92	

Table 2. Regression results and reduced chi-square $\tilde{\chi}^2$ value for the fitted polynomial curves with degree 3, 4 and 5, respectively and those with fractional exponent 0.5 included.

5. CONCLUSION

Temperature can be measured in many ways, with many different types of devices.

A new temperature system based on quartz tuning forks has been arranged for temperature measurements in a wide cryogenic range from 77 K to 273 K.

The used small quartz crystal tuning fork with a fundamental frequency of 32768 Hz, originally designed for use in wristwatches, has been explored as temperature sensor by measuring the resonance frequency shift caused by an external temperature in the above range of temperature. Special attention has been given to the design for checking and improving the stability in the output signal by comparing the shift in fundamental frequency due to the temperature changing of a crystal oscillator with respect the fixed reference frequency of a similar crystal kept at the constant temperature of 0 °C. Moreover, the management of the temperature measurements is automated by a Labview program.

The new temperature system has been validate by the temperature measurements of a reference calibrated resistance thermometer. Three polynomial curves having third, fourth and fifth degrees, respectively and two curves having third and fourth degrees where the fractional exponents 0.5 was included in each one of them have been fitted to the experimental data.

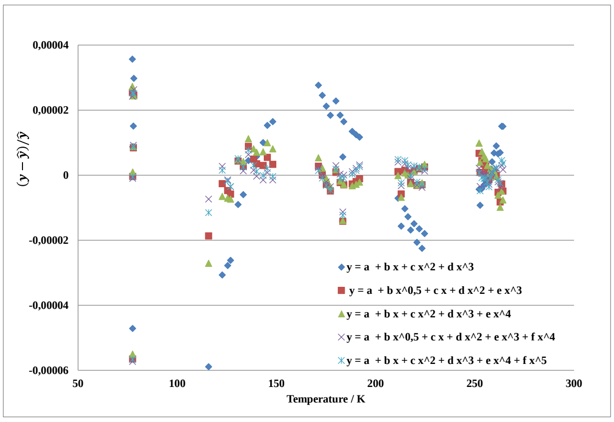

Figure 6. Relative difference between the observed responses and the corresponding prediction of five regression functions.

The parameters estimation has been performed by the application of the weighted total least square method (WTLS) and the chi-square statistic was also considered as an indicator of the agreement between the observed data and the predicted values, as well as between the estimated variance of fit and the input uncertainties. In general with the exception of the third degree polynomial curve. the higher-order polynomial curves seems to be the "best fit" to the experimental data provided by the temperature system. We also observe a statistical significant improvement by introducing the fractional degree of 0.5 in the regression curve in particular for the third degree polynomial regression. Moreover the fractional polynomial curve appears to smooth some irregularities in the fitted curves at low temperatures.

REFERENCE

[1] S. Lorefice, M. Santiano, A. Capelli, R. Romeo, A. Rivetti, G. Martini "Results of pycnometer based experiments at INRIM to measure LNG density", *Conference "Metrology for LNG"* 17 & 18 October 2013, Delft, The Netherlands

[2] B. L. Hammond, C. A. Adams and P. Smith "A linear quartz crystal temperature sensing element", *ISA Trans*, 4 (1965) 349-354.

[3] D. Zeisel, H. Menzi, L. Ullrich "A precise and robust quartz sensor based on tuning fork technology for (SF_6)-gas density control", *Zeisel Sensors and Actuators* 80 _2000. 233–236

[4] J.M. Friedt, E. Carry "Introduction to the quartz tuning fork", *Am. J. Phys*., **75,** 5, 2007, 415 – 422

[5] J. A. Kusters, "The SC Cut Crystal - An Overview", *Proc. 1981 IEEE Ultrasonics Symposium*, 402-409

[6] A. Malengo, F. Pennecchi, P. Spazzini "Calibration Curves Computing - CCC software" (Released 1.3, June 2015), http://www.inrim.it/download/CCC/index.html

[7] EMRP NEW04 "Novel mathematical and statistical approaches to uncertainty evaluation", https://www.ptb.de/emrp/new04-home.html