THE UNCERTAINTY IN A LABORATORY FURNACE MEASUREMENT

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1. Introduction

- Scientists, engineers, industry and market, worldwide require that the quality of a measurement to be stated by an assessment of the measurement uncertainty.
- It is well established that in every physical measurement is usual to expect a difference between the value of the measured quantity and a measurement estimate of its value.
- The true value of any measurement can never be determined, since systematic or random errors (due to bias or the randomness of scatter caused probably by the limited sensitivity of the instruments) influence it and induce an uncertainty in its value.
- Measurement errors, systematic with fixed sign and magnitude over a specified period of time or unpredictable random, influence every measurement. The knowledge or estimation of error introduces a certain amount of measurement uncertainty. In other words the uncertainty of the result of a measurement reflects the lack of

- The ISO ‘‘Guide to the Expression of Uncertainty in Measurement’’ (GUM) establishes a unified method (combining random and systematic uncertainties) for evaluating and stating measurement uncertainties that has been accepted by nearly all calibration services and most test co-operations in all parts of the world.
- Although GUM is accepted everywhere as a tool for evaluation and statement of measurement uncertainties, in some cases (e.g. in complex physical measurements) its use is difficult without some necessary assumptions. In these cases a simple model function connecting complex relationships could be used.
- Measurement of uncertainty first applied in analytical measurements since it was comparatively easier to be obtained.
- Literature review showed a limited application of uncertainty measurement in materials technology measurements, e.g. in hardness measurement, in vortex flowmeter, in scratching tester, in torque machine.

2. Laboratory furnaces

- tube furnaces used in:
- materials research
- sintering of ceramics
- power metallurgy
- crystal growing
- continuous heating
- doping of semiconductors
- thermocouple calibration, etc

b. box or chamber furnaces used in:

- heat treating
- enamelling
- bonding, fusing
- melting,
- decomposition in chemical analysis
- gravimetric analysis
- curing, debonding, etc

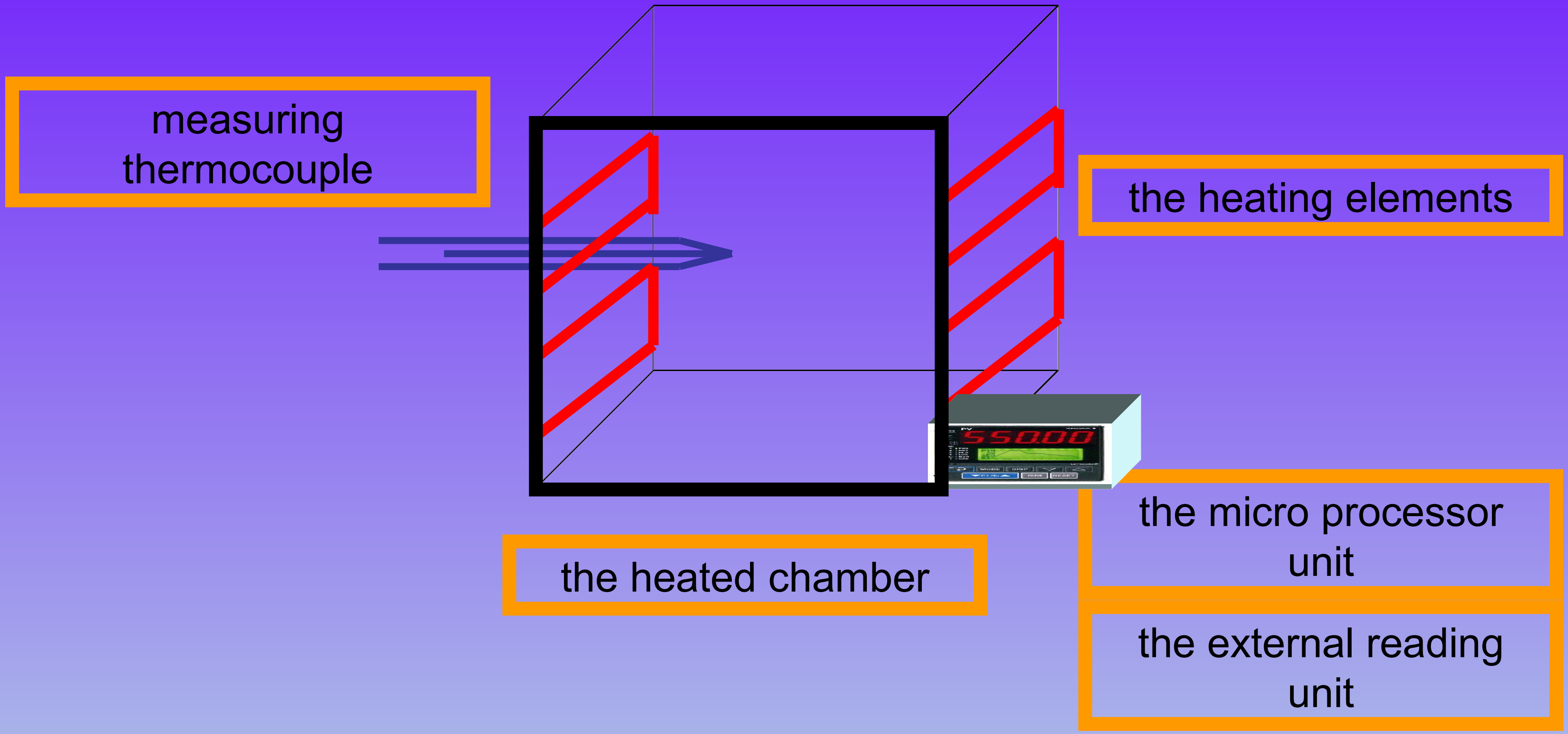
- The heated volume is usually 8 to 10 litres.

Insulation inside the furnaces include multilayer alumina and zirconia (for temperatures up to 1700^{o}C) a combination exploiting the hot strength of alumina and the low thermal conductivity of zirconia.

- **The heating elements** used in furnaces depend on its maximum operation temperatures (metallic wire up to 1300oC, silicon carbide up to 1600oC, molybdenum disilicide, $MoSi_2$, up to 1700-1800oC). Microprocessor based controllers regulate the temperature inside the furnace.

ANSI Letter/max temperature °C	Chemical composition	Special Characteristics
K -200 to 1370°C	+ Chromel (Cr-Ni) -Alumel (Al-Ni)	Good temperature precision. Sensitivity : 41 µV/°C.± 2.2 °C
N <1200°C	+ Nitrosil (Ni-Cr-Si) - Nisil (Ni,Si)	Sensitivity 39 µV/°C at 900°C. ± 2.2 °C
R up to 1600°C	+Platinum13%Rhodium (Pt-13% Rd) - Platinum (Pt)	± 1.5 °C or ± 0.25%, tolerance ± 0.5%
B up to 1680°C	+Platinum30%Rhodium - Platinum6%Rhodium (Pt-30% Rd-Pt6%,Rd)	

- A laboratory furnace consists usually of:



3. Analysis of uncertainty

Temperature measurement in a furnace is an indirect measurement in which the measured value of temperature T is computed using the known function of N independent variables X_i found by direct measurements:

$$T = f(X_1, X_2, \ldots X_N) \quad (1)$$

Estimate t of the output quantity T will be:

$$t = f(x_1, x_2 \ldots x_N) \quad (2)$$

where $x_1, x_2, \ldots x_N$ are estimates of independent variables $X_1 X_2 \ldots X_N$.

The combined standard uncertainty $u_c(y)$ can be found by combining the individual standard uncertainties and covariances using the law of propagation of uncertainty,

$$u_c(y) = \sqrt{\sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i) + 2 \sum_{i=1}^{N-1} \sum_{i=1}^{N} \frac{\partial f}{\partial x_i \partial x_j} u(x_i, x_j)} \quad (3)$$

When no covariance is present the combined standard uncertainty is reduced to:

$$u_c(y) = \sqrt{\sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i)} \qquad (4)$$

The reliability of the standard uncertainty assigned to the output estimate is determined by its effective degrees of freedom. The estimate of the effective degrees of freedom v_{eff} of the standard uncertainty u(t) associated with the output estimate t from the Welch-Satterthwaite formula:

$$v_{eff} = \frac{u^4(t)}{\sum_{i=1}^{N} \frac{u_i^4(t)}{v_i}} \qquad (5)$$

where the $u_i(t)$ (i=1,2,...,N) are the contributions to the standard uncertainty associated with the input estimate t resulting from the standard uncertainty associated with the imput estimate x_i, which are assumed to be mutually statistically independent, and v_i is the effective degrees of freedom of the standard uncertainty contribution $u_i(t)$

The expanded uncertainty U, is obtained by multiplying the combined standard uncertainty u (t) of the output estimate by a coverage factor by k, [11].

$$U=k.u(t) \qquad (6)$$

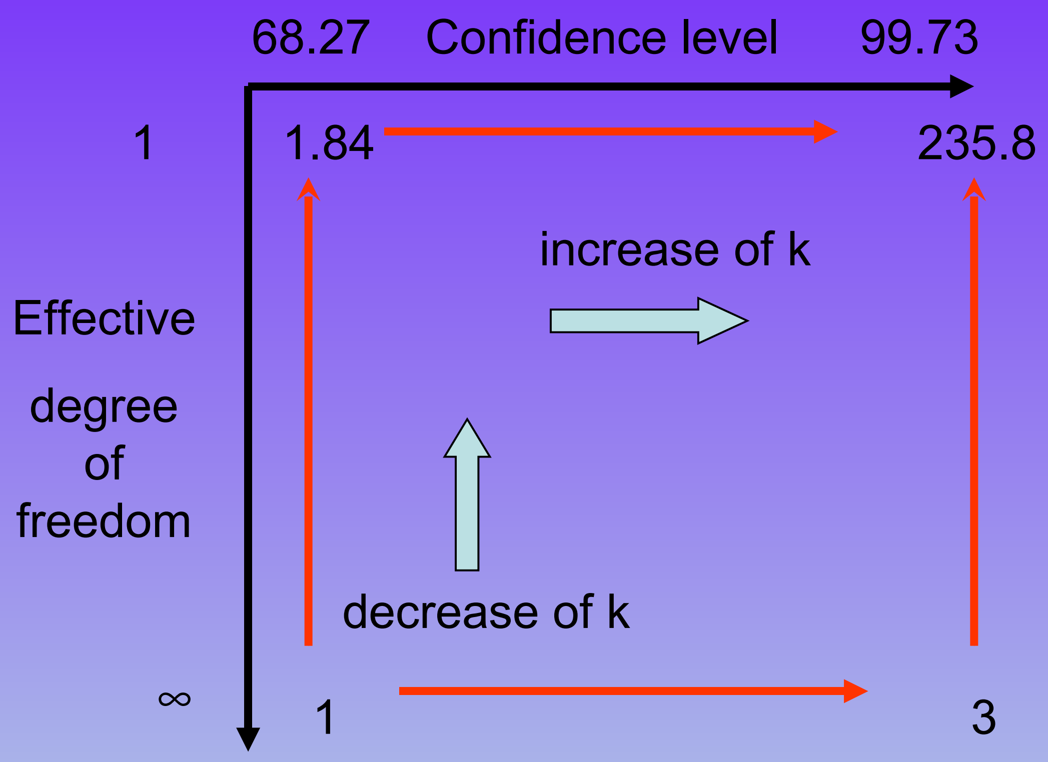
When normal distribution can be attributed to the measurand (temperature) and a significance level of 95% is acceptable, the coverage factor k is equal to 2, [12]. The coverage factor is increased as the number of effective degrees of freedom are decreased and the significance level is increased.

When the conditions of the Central Limit Theorem are met other distributions (e.g. rectangular) can be also assumed to follow normal distributions, [1]. The purpose of the expanded uncertainty U is to provide at specified significance level a confidence interval $t\pm U$ within which the value of the temperature expected to lie.

$\nu_{eff}=\infty$

Confidence level	Coverage factor
68.27	1
90	1.645
95	1.96
95.45	2
99	2.58
99.73	3

Variation of Coverage factor with confidence level and effective degrees of freedom



What we earn in confidence we lose it in uncertainty.

Uncertainty can be introduced from:

(a) improper definition of the measurand,

(b) bias in sampling or in reading of analogue instr.

(c) imperfect measurements or inadequately known effects of environmental conditions

(d) finite instrument resolution or discrimination threshold,

e) errors in values of constants and other parameters

(f) inexact values of measurement standards and reference materials,

(g) false approximations and assumptions

(h) variations in repeated observations of the measurand under apparently identical conditions

- **4. Example of calculation of temperature uncertainty in a furnace**
- Although this is a preliminary report an example of calculation of expanded temperature uncertainty at a 99% confidence level in a chamber furnace it is believed that give an indication of total uncertainty. A laboratory furnace was constructed with alumina insulation working up to 1200oC. At 1000oC the temperature **stability** inside the furnace (attained through a microprocessor) is ±0.5 oC at a confidence level of 95% and a normal distribution can be assumed. For checking **repeatability** of the temperature measurements, 9 measurements take place and a standard deviation of 0.9 oC is calculated. The temperature **uniformity** at the above temperature inside the furnace is ±2 oC and a triangular distribution can be used. Type N 1.6 mm **thermocouple**s were used measuring the temperature with **accuracy** of ± 2.2oC following rectangular distribution. The **analog to digital conversion** of the reading unit has an accuracy of 0.3%, and follows rectangular distribution.

(1) Type A uncertainties. The standard uncertainty to repeatability u_1 is :

$$u_1 = \frac{s(\bar{q}_1)}{\sqrt{9}} = \frac{0.9}{3} = 0.3\ ^{o}C \quad (7)$$

with 8 degrees of freedom.

(2) Type B uncertainties. The uncertainty due to temperature uniformity is:

$$u_2 = \frac{2}{\sqrt{6}} = 0.8165\ {}^{o}C \qquad (8)$$

with infinite degrees of freedom.

The uncertainty due to stability taking into account an accuracy of $\pm$ 0.5 ^{o}C , a confidence level of 95% and a coverage factor of k=1.96 is:

$$u_3 = \frac{0.5}{1.96} = 0.2551^{o}C \qquad (9)$$

with infinite degrees of freedom.

The type B thermocouple uncertainty following a rectangular distribution is:

$$u_4 = \frac{2.2}{\sqrt{3}} = 1.2702 \qquad (10)$$

with infinite degrees of freedom.

The combined uncertainty is:

$$u_c = \sqrt{u_1^2 + u_2^2 + u_3^2 + u_4^2} = \sqrt{0.3^2 + 0.8165^2 + 0.2551^2 + 1.2702^2} = 1.5605^{o}C \qquad (11)$$

The effective degrees of freedom are:

$$\nu_{eff} = \frac{u_c^4}{\frac{u_1^4}{n_1 - 1} + \frac{u_2^4}{\infty} + \frac{u_3^4}{\infty} + \frac{u_4^4}{\infty}} = \frac{1.5605^4}{\frac{0.3^4}{8}} = 5856.796 \approx \infty \qquad (12)$$

- The expanded uncertainty for infinite degrees of freedom at 99% confidence level (k=2.58) is:
- U=k uc=2.58*1.5605 oC=4.03 oC

5. Conclusion

Measurement accurately the temperature in a laboratory furnace is very important especially when sensitive thermal treatments and measurements are involved. In the present study an attempt is made for approaching the main sources of uncertainty in laboratory furnace temperature measurement. It seems that the main sources of uncertainty are due, to repeatability effect, to thermocouple uncertainty *per se* and also to its instability under the aggressive furnace environment, to stability and uniformity of temperature inside the furnace, to regulating microprocessor, to analogue to digital converter etc. Further research is necessary in order to investigate all the contributing to uncertainty parameters.

Thank you

your attention