

# Thermal expansion measurement of gauge blocks

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## ABSTRACT

An instrument for the measurement of the thermal expansion coefficient near room temperature of gauge blocks and other samples of similar shape and size has been developed. The length dilatation is measured by a differential plane mirror interferometer. A special interference phase detection technique compensates for non-linearity errors caused by polarization mixing. In combination with an electronic phase meter this allows to achieve nanometer accuracy. Since the measurements are done in vacuum, no compensation for the refractive index of air has to be made. For samples with good thermal conductivity the slow heat exchange by thermal radiation allows for a small temperature gradient of the sample and a good stability in the thermal equilibrium. From the thermal expansion curve, measured in a temperature range typically between 10 °C and 30 °C, the linear and quadratic expansion coefficients are evaluated at 20°C, the reference temperature for length. It is shown, that for the investigated gauge block materials the room temperature expansion can be very accurately described with two coefficients within a few parts in 10<sup>9</sup> per degree. A detailed analysis of the measurement uncertainty demonstrates the capability of the measurement instrument, which is confirmed by the results of an international comparison.

**Keywords:** Thermal expansion coefficient, gauge block metrology, high-resolution interferometry

## 1. INTRODUCTION

According to the international standard ISO 1, all length measurements have to be referred to the reference temperature 20 °C. This means that for all precise dimensional measurements, corrections have to be applied, if the measurements are not carried out at 20 °C exactly, taking into account the thermal expansion coefficient of the standards or workpieces to be measured. In gauge block metrology, the knowledge of the expansion coefficient is often an important component in the uncertainty budget. If, for example, a 100 mm gauge block is calibrated at a temperature deviating by 0.2 °C from 20 °C and the expansion coefficient is known from a manufacturer specification to be within an interval of  $\pm 1 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ , this would contribute to the standard uncertainty by 0.012  $\mu\text{m}$ , which is almost comparable to the combined uncertainties stated by leading national metrology institutes for interferometric gauge block calibration.

In order to reduce this uncertainty contribution, but also to investigate the thermal expansion of gauge blocks more generally and to provide thermal expansion measurements as a service for customers, a dilatometer for the accurate absolute measurement of thermal expansion coefficient of gauge blocks and similarly shaped samples has been developed. The instrument works near room temperature, the length measurement is done by optical interferometry.

## 2. DEFINITION OF THE THERMAL EXPANSION COEFFICIENT

The linear coefficient  $\alpha_T$  of thermal expansion at the temperature  $T$  is defined as

$$\alpha_T = \frac{1}{L} \left. \frac{dL}{dT} \right|_T, \quad (1)$$

where  $L$  is the length of the specimen. Since the expansion is not linear,  $\alpha_T$  depends on the temperature of the material. It is convenient to introduce a temperature scale offset by the reference temperature, i.e.  $t = T - 20 \text{ }^\circ\text{C}$ . The dependence on temperature of  $\alpha_t$  can be expressed by a polynomial<sup>1</sup>

$$\alpha_t = a + b \cdot t + c \cdot t^2 + d \cdot t^3 + \dots \quad (2)$$

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An alternative and more common representation is obtained from the Taylor series development of the length  $L$

$$L(t) = L_{20} (1 + \alpha \cdot t + \beta \cdot t^2 + \gamma \cdot t^3 \dots) \quad (3)$$

where  $\alpha = a$ ,  $\beta = b/2$  and  $\gamma = c/3$ . The dilatometer described here measures the length  $L(t)$  within a temperature range of typically  $\pm 10$  °C (i.e.  $T \in [10$  °C, 30 °C]). The coefficients are then obtained by numerically fitting the function of Eq.(3) through the measured points (see section 4).

### 3. DESIGN OF THE INTERFERENCE DILATOMETER

The complete measurement system is schematically shown in Fig.1. It is composed of a vacuum chamber and a temperature chamber of copper, thermostatically controlled by a water cooling and heating system. The laser and the interferometer optics for the length measurement are placed outside the vacuum. The measurement beams reach the sample through an optical window in the vacuum chamber. The sample and the interferometer are arranged vertically. The entire system is fully computer controlled, i.e. the thermostat, the temperature and the length measurement system. In the following sections, particular aspects will be discussed in detail.

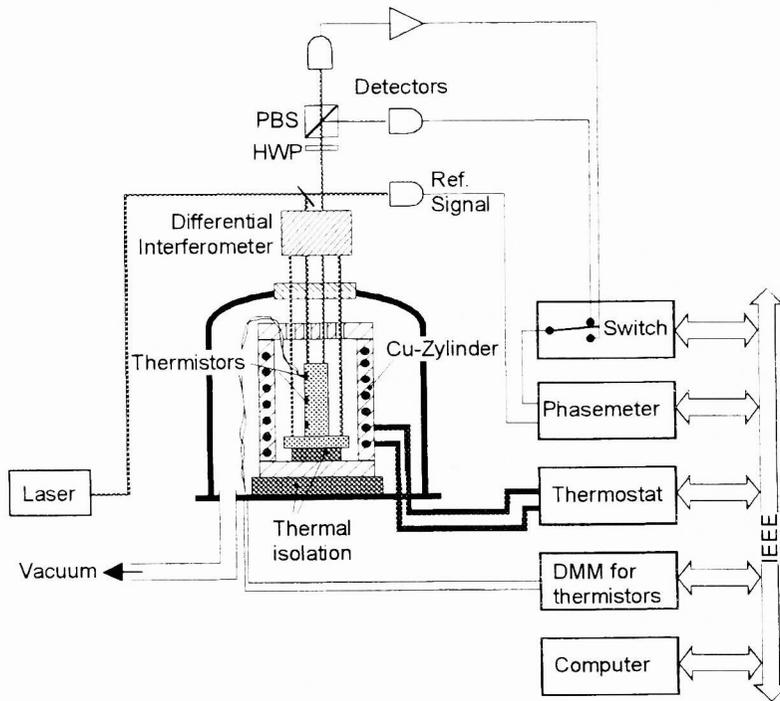


Figure 1. Schematic set-up of the interference dilatometer system.

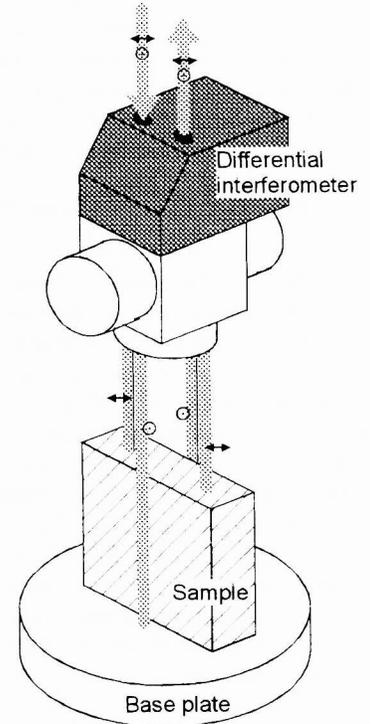


Figure 2. Arrangement of the optical beams of the differential plane mirror interferometer on the gauge block.

#### 3.1 DIFFERENTIAL LENGTH MEASUREMENT

The length change of the sample is measured by a differential plane mirror interferometer (HP 10715A). The use of a differential interferometer provides the optimum geometry for absolute dilatometry<sup>2,3</sup>. The optical interferometer acts in fact directly as the reference frame, i.e. no other parts can influence the measurement by spurious expansion. The optical configuration of the interferometer and the sample to be measured is schematically shown in Fig.2. The interferometer provides four beams, arranged in a rectangular cross section. Two beams across the diagonal belong together, the pairs having orthogonal polarization states between each other. The interferometer measures the difference between the sum of

the optical path lengths of each pair. The change of optical path length within the measurement volume due to any change of the refractive index is completely eliminated by the fact, that the interferometer measures in vacuum. The only remaining influence which acts on the stability of the expansion measurement is the stability of the interferometer itself. This was measured to be below  $2.5 \text{ nm}/^\circ\text{C}$ <sup>4</sup>. Since the interferometer optics is situated outside the vacuum chamber and the laboratory is temperature controlled, the long term stability of the interferometric length measurement is expected to be within an interval of  $\pm 1 \text{ nm}$ .

### 3.2 HETERODYNE INTERFERENCE DETECTION

The differential plane mirror interferometer used is a double-path polarizing interferometer and provides a sensitivity of  $\lambda/4 \cong 157 \text{ nm}$  for one interference fringe. The use of a Zeeman-stabilized HeNe-Laser with two orthogonally polarized optical frequencies allows for heterodyne detection at the beat frequency. The measurement of the interference phase with an electronic phase meter provides almost unlimited resolution. Dependent on the bandwidth of the detectors and the phase meter, rms phase noise below  $1^\circ$ , i.e. in the sub-nanometer region can easily be obtained<sup>5</sup>.

Limiting factor for the accuracy in heterodyne interferometry is the non-linearity produced by the polarization mixing or cross talk. This gives rise to periodic errors of about  $\pm 5 \text{ nm}$ , dependent on the quality of the polarization optics, the laser source and the alignment. By the application of an appropriate detection technique,<sup>6</sup> polarization cross talk errors can be substantially reduced. The principle of detection is shown in Fig. 1. A polarizing beam splitter inserted in the output beam of the interferometer produces two interference signals with opposite phase (or opposite sign). It can be shown, that the difference between these two signals is not affected by most of the polarization cross talk errors. Therefore, the interference phase measurement is accomplished by measuring the phase of both signals in turn with respect to the reference signal and calculating the average of the two. By this method, the interpolation linearity of the heterodyne interferometer can be improved to deviations below  $1 \text{ nm}$ .

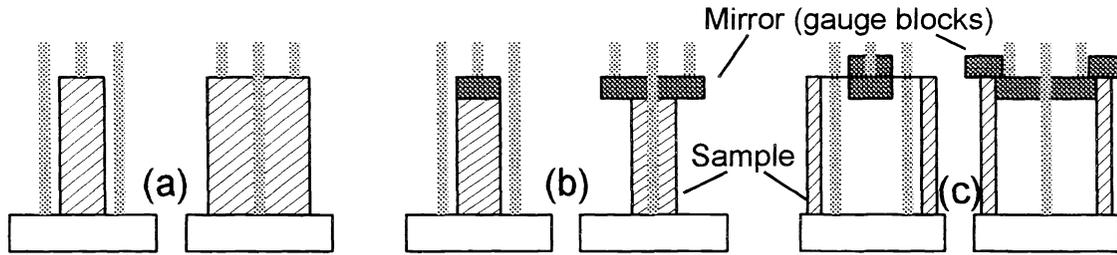
### 3.3 TEMPERATURE CHAMBER

The chamber which accommodates the sample consists of a copper cylinder. A water helix in the cylinder wall connected to a thermostatic bath allows to change the temperature of the cylinder. The sample is placed in the center of the cylinder on a thermal isolation. The thermostatic cylinder is placed in a vacuum chamber in order to avoid the influence of air refraction on the length measurement (change of the refractive index with pressure and strong turbulence during heating or cooling). This means that the heat exchange between the sample and the cylinder is done only by radiation. The temperature uniformity of the sample depends on the temperature uniformity of the cylinder and the ratio between heat exchange by thermal radiation and the thermal conductivity of the sample material. Although the walls of the cylinder are uniformly tempered, the top and bottom cover of the cylinder, which follow passively, are also influenced by the wall temperature of the vacuum chamber, which is at room temperature. Temperature uniformity of the cylinder wall has been found to be within  $20 \text{ mK}$ , differences between top and bottom cover with respect to the wall temperature were always smaller than  $80 \text{ mK}$  for a deviation from room temperature of about  $10^\circ\text{C}$ . On  $100 \text{ mm}$  gauge blocks maximum temperature gradients of  $35 \text{ mK}$ ,  $45 \text{ mK}$ ,  $120 \text{ mK}$  and  $150 \text{ mK}$  have been measured for the different materials steel, tungsten carbide, aluminum oxide ceramic and zerodur, respectively. As long as these gradients are linear, the use of the average temperature for the determination of the thermal expansion coefficient will not lead to significant errors. The contributions due to residual temperature non-uniformity, which have to be taken into account for the measurement uncertainty evaluation, amount to  $5 \text{ mK}$ ,  $4 \text{ mK}$ ,  $12 \text{ mK}$  and  $13 \text{ mK}$  for the four different materials mentioned above. The temperature uniformity, but also the time constant for temperature changes (see section 4) could certainly be improved by introducing a helium atmosphere – helium has excellent thermal conductivity – in the vacuum chamber at sufficiently low pressure ( $\sim 1 \text{ hPa}$ ), in order to keep the refractive index sufficiently low.

### 3.4 SAMPLE GEOMETRY

As mentioned earlier, the instrument has been designed essentially for gauge blocks. These are wrung to a flat platen with the upper measurement surface of the gauge block and the platen surface acting as mirrors for the differential interferometer (Fig.3a). Other samples than gauge blocks are possible, provided that they are sufficiently parallel (better than  $0.3 \text{ mrad}$ ). If the end surfaces are polished, one of these can directly be used as a mirror, otherwise a small gauge block with known expansion coefficient can be fixed on top of the sample (Fig.3b). Also tube shaped samples can be measured. An assembly of three gauge blocks appropriately wrung together can be placed as top mirror without introducing an additional thermal expansion (Fig.3c). The limit sample dimensions are:  $130 \text{ mm}$  for the length,  $9 \text{ mm}$  for

the smaller width of a prismatic or the diameter of a cylindrical sample and 21 mm for the inner diameter of a tube shaped sample.



**Figure 3.** Different possible sample geometries to be used with the four measurement beams of the differential interferometer: (a) prismatic (gauge block), (b) cylindrical, (c) tube.

#### 4. MEASUREMENT PROCEDURE

For the expansion measurements, the temperature is changed typically in the range of 10 °C to 30 °C in steps of 3 °C to 5 °C. The measurements are performed at each point in a quasi-equilibrium, the difference between sample and the chamber temperature being smaller than 0.05 °C, which corresponds after a temperature change of a few degrees to a residual rate of about 0.3 mK/min. Waiting for an equilibrium allows for a better temperature measurement accuracy and a better uniformity of the sample temperature.

The change of the chamber wall temperature by the circulating water is relatively fast (a few minutes). On a step like change of the thermostat, the sample temperature follows an exponential function. The time constant depends on the absorption and the reflectivity of the sample surface for heat radiation and the heat capacity of the material. It has been measured to be between 1½ and 3 hours (Table 1). For achieving the quasi-equilibrium in each point, 4.6 times the time constant has to be waited, which can be more than half a day. A full cycle of expansion measurement lasts up to four days. Although this might seem to be long, the system is fully automatic and the long measurement time does not present any problem, if not a large series of samples has to be measured. A controlled Helium atmosphere would probably speed up the measurement process by a factor of two to three.

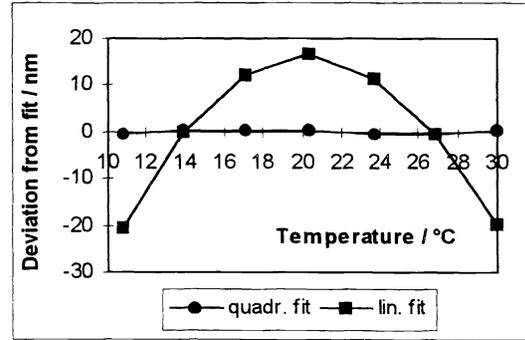
Material	Time constant	Stabilisation time
Steel	185 min	13 h
Tungsten carbide	140 min	11 h
Ceramic	90 min	7.5 h
Zerodur	90 min	7.5 h

**Table 1.** Time constant and stabilisation time for reaching a thermal equilibrium within the vacuum temperature chamber for different gauge block materials.

A complete measurement cycle results in the expansion function  $L(t)$ , sampled in usually 5 to 7 measurement points. A quadratic function according to Eq.(3) is then fitted through these points in order to obtain the parameters  $\alpha$  and  $\beta$ . Figure 4 shows the measurement of a 100 mm tungsten carbide gauge block and a graphical representation of the residuals from a linear and a quadratic fit through the expansion curve. With a quadratic fit function, the residuals in the measurement points are usually smaller than 1 nm, i.e. no significant result could be obtained for the cubic parameter  $\gamma$ , or – in other words – the thermal expansion in the temperature range of 10 °C to 30 °C is sufficiently well described within a few parts in  $10^9$  with two parameters only, at least for the investigated gauge block materials. The determination of the quadratic coefficient  $\beta$  is very significant; for the temperature range above, a linear fit would give rise to residuals of about 100 nm or 35 nm for steel and tungsten carbide, respectively.

The fit residuals (for the example in Fig.4 smaller than 0.6 nm) are not only caused by random variations in length measurement, but also by uncertainties in the temperature measurement. In fact, residuals of 1 nm correspond to temperature deviations of approximately 1 mK or 2 mK for a 100 mm steel or tungsten carbide gauge block, respectively. Because length and temperature measurements are uncorrelated, the random deviations in each individual measurement quantity (length and temperature) are likely to be smaller than those found from the residuals.

$t / ^\circ\text{C}$	$\Delta L(t) / \mu\text{m}$	$\delta L_{\text{lin}} / \text{nm}$	$\delta L_{\text{quad}} / \text{nm}$
10.759	0	-20.4	-0.4
13.901	1.324	0.1	0.3
17.083	2.674	12.3	0.4
20.375	4.078	16.5	0.3
23.659	5.488	11.5	-0.6
26.848	6.865	-0.4	-0.4
30.042	8.251	-19.6	0.4



**Figure 4.** Measured thermal expansion  $\Delta L(t) = L(t) - L(t=10.759^\circ\text{C})$  for a tungsten carbide gauge block and residuals  $\delta L_{\text{lin}}$  and  $\delta L_{\text{quad}}$  from linear and quadratic fit with coefficients  $\alpha = 4.279 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$  and  $\alpha = 4.276 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$ ,  $\beta = 0.0039 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$ , respectively.

## 5. UNCERTAINTY OF MEASUREMENT

The uncertainty of measurement of the interference dilatometer shall be evaluated according to the ISO Guide<sup>7</sup>. The estimation is made for 100 mm gauge blocks of different material, assuming a measurement cycle of 7 points regularly spaced by 3.33 °C ranging from 10 °C to 30 °C. The combined uncertainty is essentially determined by four contributions:

- uncertainty of the fit due to random uncertainties in temperature and length measurement;
- uniformity of the temperature within the sample;
- calibration of the temperature sensors;
- drift in length measurement.

Other contributions such as the uncertainty of the optical wavelength and of the absolute length of the sample or cosine error are negligible. The square of the standard uncertainty of the linear thermal expansion coefficient is then given by

$$\delta^2 \alpha = c_{\text{Lfit}}^2 \delta^2 L + c_{\text{tfit}}^2 \delta^2 t + c_{\text{tu}}^2 \delta^2 t_u + c_{\text{tc}}^2 \delta^2 t_c + c_{\text{Ld}}^2 \delta^2 L_d \quad (4)$$

where  $\delta L_i$  and  $\delta t_i$  are the standard uncertainties of the input quantities and  $c_i$  the sensitivity coefficients.

### 5.1 UNCERTAINTY OF THE FIT ( $\delta L$ , $\delta t$ )

The uncertainty of the fitted parameter caused by random variations of the input quantities has been numerically simulated by introducing random errors in length and temperature to the least squares fit calculation. The following sensitivity coefficients have been found:  $c_{\text{Lfit}} = (1/L) \cdot 3.2 \cdot 10^{-2} \text{ }^\circ\text{C}^{-1}$  and  $c_{\text{tfit}} = \alpha \cdot 3 \cdot 10^{-2} \text{ }^\circ\text{C}^{-1}$

### 5.2 TEMPERATURE UNIFORMITY ( $\delta t_u$ )

The sample temperature uniformity is best near room temperature, whereas the largest temperature gradients are found at the extremes of the temperature range. From three temperature sensors fixed at the bottom, mid height and top of the sample, the maximum deviations of the average temperature obtained from the three readings from the real average temperature of the sample has been estimated to 7 mK, 5 mK, 17 mK and 20 mK for steel, tungsten carbide, ceramic and zerodur, respectively. The sensitivity coefficient is  $c_{\text{tu}} = 2\alpha/\Delta T$ , where  $\Delta T = 20 \text{ }^\circ\text{C}$  is the range of the temperature change.

### 5.3 CALIBRATION OF THE TEMPERATURE SENSORS ( $\delta t_c$ )

The temperature sensors (thermistors with 10 k $\Omega$  nominal resistance at 25 °C) are calibrated with respect to a Pt25 standard platinum resistance thermometer in the temperature range between 10 °C and 30 °C. The absolute value of the calibration is only of second order importance, but the slope and the stability will have to be considered. This is estimated to <10 mK for the full range including the drift since the last calibration, i.e.  $\delta t_c \cong 6 \text{ mK}$  standard uncertainty with the sensitivity coefficient  $c_{\text{tc}} = \alpha/\Delta T$ .

## 5.4 STABILITY OF THE INTERFEROMETER ( $\delta L_d$ )

The drift of the interferometer during the measurement cycle is estimated from the closing error (after a the full temperature range the temperature is set to the initial value and this last measurement point is compared to the fitted expansion curve), which is usually a few nanometers and has never been larger than 6 nm after four days of measurement. A long term stability test obtained from an „expansion“ measurement without sample resulted in a maximum drift value of 1.6 nm. The sensitivity coefficient is  $c_{L_d} = 1/(L \cdot \Delta T)$ .

## 5.5 COMBINED STANDARD UNCERTAINTY

The standard uncertainty for the linear expansion coefficient depends on the material (expansion coefficient, thermal conductivity) and the sample length. Based on the uncertainty contributions discussed above, the combined uncertainty is calculated for four 100 mm gauge blocks of different material (table 2). For the examples given, the standard uncertainty is always smaller than  $10^{-8} \text{ }^\circ\text{C}^{-1}$ .

Quantity	100 mm steel gauge block			100 mm tungsten carbide gauge block		
	$u(x_i)$	$c_i$	$u_i(y)$	$u(x_i)$	$c_i$	$u_i(y)$
Length, random	1 nm	$3.2 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$0.32 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	1 nm	$3.2 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$0.32 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
Temperature, random	1 mK	$0.34 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$0.34 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	1 mK	$0.28 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$0.28 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
Temperature uniformity	7 mK	$1.2 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$8.4 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	5mK	$0.94 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$4 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
Temperature calibration	6 mK	$2.4 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$3.6 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	6 mK	$1.88 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$2.8 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
Length stability	3.6 nm	$5 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$1.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	3.6 nm	$5 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$1.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
	<b>Combined std. uncertainty</b>		<b><math>9.3 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}</math></b>	<b>Combined std. uncertainty</b>		<b><math>5.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}</math></b>

Quantity	100 mm ceramic gauge block			100 mm zerodur gauge block		
	$u(x_i)$	$c_i$	$u_i(y)$	$u(x_i)$	$c_i$	$u_i(y)$
Length, random	1 nm	$3.2 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$0.32 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	1 nm	$3.2 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$0.32 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
Temperature, random	1 mK	$0.13 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$0.13 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	1 mK	0	0
Temperature uniformity	17 mK	$0.43 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$7.3 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	20 mK	0	0
Temperature calibration	6 mK	$0.86 \cdot 10^{-6} \text{ }^\circ\text{C}^{-2}$	$1.3 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	6 mK	0	0
Length stability	3.6 nm	$5 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$1.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$	3.6 nm	$5 \cdot 10^{-4}/(\text{mm}^\circ\text{C})$	$1.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}$
	<b>Combined std. uncertainty</b>		<b><math>7.6 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}</math></b>	<b>Combined std. uncertainty</b>		<b><math>1.7 \cdot 10^{-9} \text{ }^\circ\text{C}^{-1}</math></b>

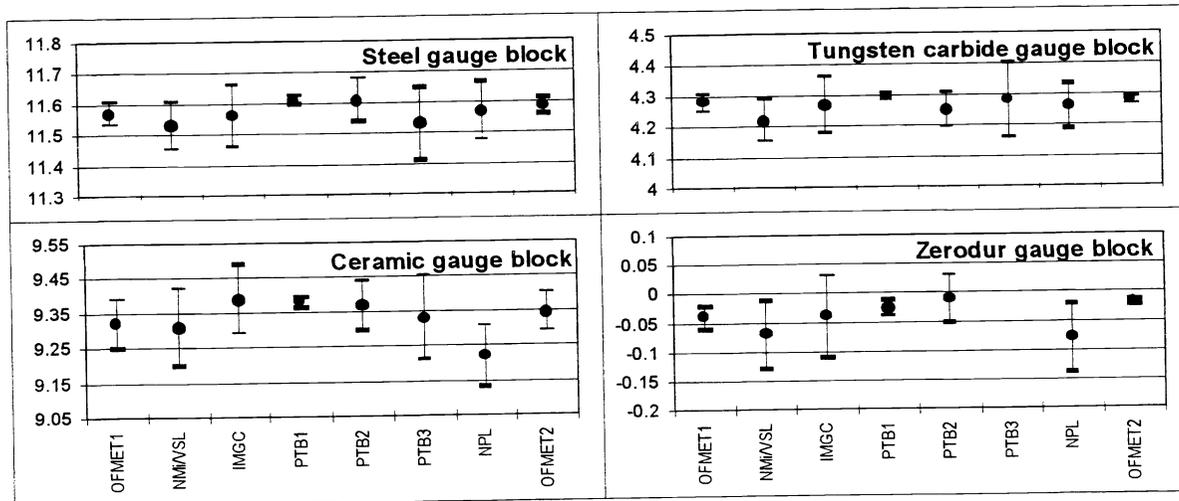
**Table 2.** Measurement uncertainty budget for four 100 mm gauge blocks of different material, i.e. steel, tungsten carbide, ceramic and zerodur.  $u(x_i)$  are the standard uncertainties of the input quantities,  $c_i$  the sensitivity coefficients and  $u_i(y) = u(x_i) \cdot c_i$  the corresponding contributions to the combined uncertainty.

## 6. INTERNATIONAL COMPARISON

In 1994, an interlaboratory comparison of measurements of the thermal expansion coefficient has been carried out<sup>8</sup>. Seven laboratories of five European national metrology institutes took part with the Swiss Federal Office of Metrology (OFMET) as the pilot laboratory. The linear thermal expansion coefficient at room temperature (20 °C) of four 100 mm gauge blocks of different material had to be measured.

Various types of instruments were used for the measurements. Common to all but one instrument was the interferometric length measurement. Some laboratories carried out their measurements on their usual absolute gauge block interferometer (NMI/VSL, PTB1, IMGC), changing the temperature of the gauge blocks by a thermostatic cooler / heater or by the laboratory temperature. Others have developed dedicated instruments for thermal expansion measurement, exclusively for gauge blocks (NPL, OFMET) or for more general, larger specimens (PTB3). PTB2 used a mechanical comparator and thermal expansion standards calibrated by PTB1.

The measurement results in units of  $10^{-6} \text{ }^\circ\text{C}^{-1}$  together with the associated uncertainties are shown in Fig.5. The agreement expressed as the difference between the largest and the smallest value was  $0.08 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$  for steel,  $0.075 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$  for tungsten carbide,  $0.17 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$  for ceramic, and  $0.07 \cdot 10^{-6} \text{ }^\circ\text{C}^{-1}$  for zerodur.



**Figure 5.** Results of the European interlaboratory comparison of thermal expansion measurements of four 100 mm gauge blocks. The results are expressed in  $10^{-6} \text{ }^{\circ}\text{C}^{-1}$ .

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