EVALUATION OF MEASUREMENT UNCERTAINTIES FOR POLARIMETRIC CALIBRATION OF QUARTZ CONTROL PLATES

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Abstract:
Quartz control plates are commonly used in the sugar and ethanol industry as transfer standards for the calibration of saccharimeters and therefore require calibration certificates with low uncertainties. We discuss the different physical factors influencing the assessment of the uncertainty budget for the determination of the polarization rotation angle measurements of these quartz control plates.
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Keywords: uncertainty evaluation, polarimetry, quartz control plates.

1. INTRODUCTION

The Optical Metrology Division of Inmetro has built a reference polarimeter [1] for polarimetric calibration of quartz control plates, transfer standards being used by the sugar and ethanol industry for the calibration of saccharimeters. Quality control of the saccharimeters will soon became regulated in Brazil, and the national regulating norms are being developed by the Legal Metrology Division of Inmetro, Dimel.

The polarimeter project started with a collaboration of the Polarimetry Laboratory of the Physikalisch-Technische Bundesanstalt, PTB, Germany [2, 3]. A detailed description of the setup can be found in [1] and references therein. The main improvement since that last communication is the finalization of the thermalization chambers at Inmetro's machine shop, a key part of the instrument. This improved setup is now under characterization, in order to determine the uncertainty budget. The influence factors and their contribution to the uncertainties are being disclosed.

2. THE POLARIMETER

The quartz control plate (QCP) has the shape of a disc, cut from a single crystal quartz gem, with plane parallel round and polished surfaces of 16 mm in diameter and thickness up to 1,6 mm. The crystal´s optical axis is oriented perpendicular to the surface of the plate. The polarimeter measures the angle of rotation of the polarization plane of polarized monochromatic radiation transmitted through the QCP under test. Figure 1 shows the actual polarimeter set up: (1) frequency stabilized He-Ne laser, $\lambda = 633$ nm; (2) optical isolator; (3) fixed Glan-Thompson polarizer (P1);

(4, 13) iris; (5) autocollimator; (6) step motor driver connected to chamber A; (7) motorized linear stage; (8) thermalized chamber A with a rotating sample holder; (9) chamber B with fixed sample holder; (10, 11) temperature sensors inside A and B wells; (12) air temperature sensor; (14) Glan-Thompson analyzer (P2) mounted in a rotary stage with an optical encoder; (15) photodetector; (16) linear and rotary stage drives; (18) closed loop thermalizing bath; (20) computer running LabView for automation, data acquisition and analysis. The main set up is enclosed under a protective cover against air currents and stray light during the measurements (dotted line), made of thick F5 EPS with a layer of black EVA at the inside. Figure 2 shows the new thermalization chambers A and B, mounted over a platform pushed by a linear stage underneath. Water from a very stable bath circulates though both chambers, maintaining the temperature of the samples at 20°C.
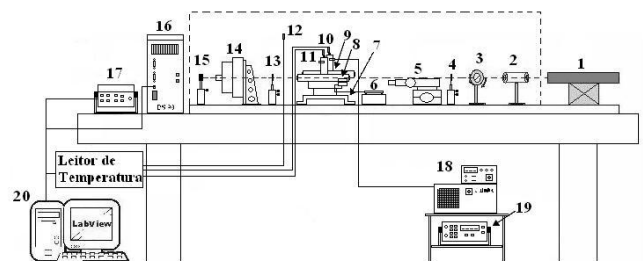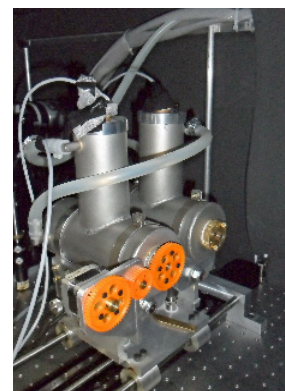

Fig. 1. The polarimeter setup.

Fig.2. The thermalization chambers: chamber A, at the left, connected to a step motor; chamber B at the right.

2.1. The Measurement Method

As shown in Figure 1, from right to left: the laser beam crosses the optical isolator, the fixed polarizer, the QCP under test, the rotating polarizer, and reaches the photodetector. The QCP under test is mounted inside a rotating cylinder inserted into the thermalized chamber A, and chamber B contains a permanently installed QCP, used as reference standard for crosschecking system performance and validation. The measurement procedure is automated in LabView environment.

The measurement procedure is automated by a LabView routine that acquires the photodetector signal, the temperature sensors readings and the optical encoder readings of the rotation stage, besides controlling the movements of the rotation stage, the linear translation stage and the stepping motor of the sample chamber. The polarization rotation angle results from the difference in angle position of a point of minimum intensity reading when the laser crosses P1 and P2 is rotated, and this minimum position after a QCP is placed between P1 and P2 in front of the laser beam, and P2 is rotated. This minimum position is obtained by fitting a second degree polynomial to the acquired intensity versus encoder reading, by a LabView routine. The plate mounted in chamber A is measured after four successively rotations of 90° in the plane perpendicular to beam; at each position the rotation stage moves the. The linear stage moves chamber B to the front of the laser and the QCP mounted is also measured, but this sample tube stays fixed, so this sample is always measured at the same position. The series are repeated 3 more times.

2.2. The influence factors

Supposing the QCP are free of imperfections, compliant with the constructions standards [4, 5], the main influence factors are the wavelength of the radiation, the plates' temperature, alignment of the plate and optics, and the determination of the minimum intensity point.

2.2.1. The wavelength factor

The polarization rotation wavelength dependence has been established after precise measurements [6, 7] realized in the PTB, and a mathematical expression was derived from fittings to the experimental data [8] and [9], and is valid in the range 546-900 nm:

$$[\alpha] = a_0 + \frac{a_1}{\lambda^2} + \frac{a_2}{\lambda^4} + \frac{a_3}{\lambda^6} \tag{1}$$

Where: $[\alpha]$ is the specific rotation of quartz in °/mm; λ is the wavelength in μm; $a_0 = -0.1963657$; $a_1 = 7.262667$; $a_2 = 0.1171867$; $a_3 = 0.0019554$

The ICUMSA SPS-1 [4] specifies that the radiation incident wavelength must be "well known up to 0.0001 nm". In order to comply with this requirement the polarimeter utilizes the radiation of a calibrated stabilized He-Ne laser with wavelength $(632.990964 \pm 0.000027)$ nm. To exemplify the influence of the wavelength, a variation in λ of this magnitude implies in a polarization rotation angle α, when calculated for a QCP of 1 mm thickness:

for $\lambda = 0.632990991$ μm $\Rightarrow \alpha = 18.6899367974°$
for $\lambda = 0.632990937$ μm $\Rightarrow \alpha = 18.689940154668°$
The difference is $\delta = 0.000003°$

A variation in the laser wavelength of the size of its expanded uncertainty leads to a variation in the polarization rotation of the order of 10^{-6} degrees. The ICUMSA standards [4] recommend that the uncertainty in the polarization rotation should be less than $\pm 0.001°$, which is 10^3 larger than the variation introduced by the stabilized laser. Thus, the stabilized He-Ne laser wavelength uncertainty has a very small contribution to the total uncertainty budget.

2.2.2. The temperature contribution

There is a dependence of the rotation angle with the temperature for quartz, which generates a correction factor when the measurement is realized at temperatures different from the recommended temperature of 20 °C. This dependence has been determined from fittings to experimental data [9], resulting in the equation:

$$\frac{\alpha_{t°C}}{\alpha_{20°C}} = 1.0 + \left(a_1 + \frac{a_2}{\lambda} + \frac{a_3}{\lambda^2} \right) \cdot (t - 20) \tag{2}$$

Where λ is the wavelength (in μm), $\alpha_{20°C}$ is the rotation at 20 °C and t is the measured temperature (°C); with:
$a_1 = 1.280924 \times 10^{-4}$; $a_2 = 1.0852636 \times 10^{-5}$;
$a_3 = -9.0311692 \times 10^{-7}$

The ICUMSA standard [4] recommends that the QCP temperature must be known with a tolerance of 0.1 °C. To evaluate this influence in the value of the polarization rotation, consider a 1.6 mm thick QCP measured by a He-Ne laser of $\lambda = 0.6329914$ μm, and a variation in temperature of 0.1°C:

for t $= 20.0°C \Rightarrow \alpha = 29.7510°$
for t $= 20.1°C \Rightarrow \alpha = 29.7514°$

This introduces a variation of $\delta = 0.0004°$ in the polarization rotation.

This variation contributes significantly to the total uncertainty in rotation, which is recommended by the standard [4] to stay below a limit: "the total uncertainty in polarization rotation should be less than $\pm 0.001°$".

The uncertainty in the temperature determination arises mainly from two factors: (a) the actual value of the temperature at the QCP during the measurement, and (b) the calibration of the temperature sensors.

Factor (a) has been studied for several measurement configurations where the temperature sensors were positioned inside the chambers, in the air close to chambers, in contact with the QCP, and the data were acquired during several days. A complete characterization of the temperature inside the chambers, and in the air around the chambers was realized, in order to know the temperature gradient inside the chamber at the sample position, and in the air just outside the chambers. From those measurements results, the setup has been improved, and actually the temperature in the thermalization chambers where the QCP is mounted is now

stabilized better than 0.001°C during each minimum intensity measurement. For that same above mentioned example, the variation in rotation introduced by 0.001°C is $\delta = 0.000\,004°$. This means that the temperature influence now is kept very low, and the stabilized temperature introduces a very small uncertainty component to the uncertainty budget. This is achieved by the thermalization chambers with a very stable circulating bath, and also by enclosing the whole polarimeter under an insulating box. Keeping the temperature factor under control allowed us to study the alignment factor influence in the polarization rotation.

Factor (b), the calibration of the temperature sensors, is very important, because the actual temperature reading enters the final calculation of the rotation, and a poor calibrated temperature sensor will introduce a systematic error in the measurements, as well as enlarge the uncertainty.

2.2.3. The Alignment factor

There are two main alignment components for the alignment factor: (a) the alignment of the polarizers, and (b) the alignment of the QCP relative to the optical axis of the polarimeter.

The QCP polarization rotation is measured as a difference of two determinations: the reference, measured when the laser crosses just the entrance polarizer and the rotating analyzer, and measurement with the QCP. Thus, the first uncertainty contribution, when just the analyzer rotates, is evaluated by the standard deviation of the repetitivity and the reproducibility of the reference measurements. This contribution reflects a misalignment of the optical axis of the polarimeter. Evaluating this factor for over a hundred measurements, this contribution is of the order of 0.00005°.

The dominant factor for the uncertainty budget arises from a slight misalignment of the QCP inside the rotating sample tube. Misalignment can introduce errors in the rotation angle: the assumption of perpendicularity of sample faces and laser beam is related to the necessary parallelism of the quartz optical axis and laser beam. That is why the quartz plate optical axis direction should not deviate by more than 0.1′ from the vertical to the plate face and the plate tilt angle has a tolerance of no more than 10′ [4]. An autocollimator is used to assist in the QCP alignment, besides the laser beam retroreflection. Measurements of the polarization rotation varying this tilt angle were realized to estimate the value of the error. An expression for evaluation of the alignment of the laser versus QCP, considering the increase in thickness introduced by tilt angles was derived. QCP polarization rotation was measured for tilt angles varying from 0° to 1°. The variation introduced by a tilt angle of 1° can be as large as ~ -0.003° in polarization rotation. These measurements showed that the negligible increase in the optical path (thickness) introduced by these tilt angle is not the preponderant factor in the polarization rotation angle variation: the contribution is negative, whereas an increase in thickness contributes with a positive value. This contribution is due to the deviation of the quartz optical axis from the normal, that is, the contribution from

the quartz crystallographic a-axis and b-axis play a role in decreasing the amount of polarization rotation.

This factor is very important, because the QCP must rotate about its center during the measurements series, as the ICUMSA standard recommends [4]. This is accomplished by rotating the sample tube inside the thermalization chamber by a stepping motor, as seen in Figure 2. The whole thermalization chamber can be adjusted in order to stay parallel to the laser beam, but the QCP inside must be well mounted, in order to keep the tilt angle relating to the laser beam at a minimum. This misalignment factor is reflected in the spread of the values as the QCP turns 90° four times sequentially, and this series is repeated three more times. Re-mounting the QCP several times gives the reproducibility. This influence factor was evaluated and is of the order of ± 0.0005°, and thus this is the dominant factor.

2.2.4. The determination of the point of minimum intensity

There are two factors to consider: the rotary encoder reading, and the data analysis method. The angular position is determined by an optical 36000 lines rotary encoder with two reading heads and an interpolation card; the resolution is 0.0003°. The data analysis method consist in determining the read angular position of minimal intensity by fitting a second order polynomial to an angular region of 0.6° around this point, where it is a very good approximations for the measured signal. This small region contains close to 120 points, providing good fittings, as shown by the plotted residuals. The minimum is thus obtained from the resulting parameters. The optical rotation results from the difference of the averaged repeated measurements of the reference position (without QCP) and each temperature corrected measurement of the QCP. This correction to 20 °C is effectuated using equation (2), which can be rewritten as:

$$\alpha(r,t) = \frac{r}{1 + b(t - 20)} \tag{3}$$

Where r is the measured QCP rotation at $t \neq 20°C$, t is the measured temperature, and b is the calculated factor in eq. (2) for for $\lambda = 0.632990964\ \mu m$. The sensibility coefficients are:

$$\frac{\partial \alpha}{\partial r} = [1 + bt - 20b]^{-1} \tag{4}$$

$$\frac{\partial \alpha}{\partial t} = \frac{-br}{[1 + bt - 20b]^2} \tag{5}$$

The combined uncertainty in each measurement i due to temperature is $(\alpha_i \pm u_{ci})°$:

$$u_{ci}(\alpha_i(r,t)) = \sqrt{\frac{(\Delta r)^2}{[1 + b(t - 20)]^2} + \frac{b^2 r^2 (\Delta t)^2}{[1 + b(t - 20)]^4}} \tag{6}$$

where: Δr is the uncertainty of the reference measurement, and Δt is the uncertainty of the averaged temperature during each run. Here one can clearly see where the temperature

factor contribution enters the calculation of each polarization rotation measurement. During each run 24 measurements are performed, and the values are averaged. This average standard deviation reflects the spread in the values due to the 90° rotation of the tube containing the QCP inside the thermalization chamber. This alignment factor offers still the possibility to further improve the mechanical alignment of the QCP, although our results so far achieved already comply with the ICUMSA recommendation.

3. CONCLUSIONS

The metrological characterization of a polarimeter assembled at Diopt/Inmetro is under way, in order to assess the uncertainty budget for polarimetric calibration of quartz control plates.

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