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Calibration of Quartz Control Plates by High Resolution Polarimetry

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ABSTRACT

A high resolution polarimeter is being assembled at Inmetro for calibrating quartz control plates, standards for saccharimeters and industrial polarimeters. Here is shown the first measurements by this new instrument of plates previously calibrated at the PTB.

Keywords: polarimetry, quartz control plates, laboratory automation.

1 INTRODUCTION

The metrological traceability of standards is of a great importance for the National Industry competitiveness. Quartz control plates are transfer standards used to calibrate polarimeters and saccharimeters, a polarimeter specialized in measurement of saccharosis concentration. These instruments are used in the sugar cane industry, food, chemistry and pharmaceutical industries as well. They measure the angle of rotation of the polarization plane of a radiation after traversing a chiral medium. This angle is related to the concentration of the substance or to the thickness of the crystal under test. The angle rotation from a Quartz control plate corresponds to the rotation introduced by a standard solution of saccharosis measured under the established conditions prescribed by norms the from the ICUMSA (International Commission for Uniform Methods of Sugar Analysis) SPS-1(1998) and the OIML (Organization Internationale de Métrologie Légale) R14

(1995). The recommendations of these organizations concerning the construction of saccharimeters, reference quartz plates and procedures for calibrations are internationally recognized and followed for most of the countries that Brazil has trade and business, what motivated the Brazilian Industry to ask for this service here in Brazil.

2 OBJECTIVES

The Optical Division of Inmetro (Diopt) is building a high resolution polarimeter, Alvarenga et all (2005), for calibration of quartz control plates in a collaboration with the the Polarimetry Laboratory of PTB, Schulz et all (2006), the only institution that performs primary calibrations of quartz control plates. Inmetro´s instrument is similar to the new recently built at PTB. A first prototype was assembled here with parts already available and a semi-automatic method was used to perform the measurements in standards already calibrated in PTB, Alvarenga et all (2008). Recently the remaining imported parts of the equipment has arrived and the new instrument is being assembled, automated and programmed in the Interferometry Laboratory (Laint). It has a high resolution automatic rotation stage with an optical encoder to rotate a polarizer, and a linear stage to position the quartz plate, what permits fast repetitiveness of the measurements, achieving greater reproducibility, and will give a much smaller uncertainty to the results.

3 METHODS AND EXPERIMENTAL SETUP

The polarimetric method for measuring polarization rotation angles of optically active substances is well known, and is condensed in reference Alvarenga et all (2005). In a simple polarimeter, a beam of light crosses two polarizers where its intensity is thus monitored by a detector. Beginning with the first polarizer fixed – selects the incoming polarization plane – the other, mounted on a graduated circle, is rotated 90º and consequently no light reaches the detector. On the graduated circle this is the reference point. The second step is to introduce the sample in the light pathway between both polarizers. The detector will indicate a raise in the signal, indicating that the polarization plane of the light was rotated. The third step is to restore the condition of minimum intensity as it was before (the null condition), and this is done by rotating the second polarizer away from the reference point until the signal in the detector drops again to a minimum. The actual restoration angle read in the graduated circle corresponds to the rotation angle of the radiation polarization plane caused by the sampled substance.

This polarization rotation angle is temperature and wavelength dependent as seen in Bunnagel et all (1966), Zander et all (1974), and Emmerich et all (1998), and for crystalline substances the alignment of their optical axis and the radiation beam direction plays an important role as well. A high resolution polarimeter thus must obey the ICUMSA and OIML specifications SPS-1 and R-14 for their construction and operation, and for a primary instrument, the requirements are much more stringent, in order to keep the uncertainty as low as possible.

The new polarimeter under construction, shown in Figure 1, utilizes the 633 nm radiation of a stabilized He-Ne laser, to comply with the requirement of well

known incident wavelength. High quality optical elements as a Faraday isolator, to cut backreflections into the laser tube, and calcite Glan-Taylor polarizers with high extinction ratio. An automatic programmable robust rotation stage is used for mounting the analyzer with an optical encoder with 36000 lines, two reading heads and an interpolation card for a very precise angle reading. An automatic programmable linear stage is used for the positioning of the quartz plate in and out of the laser beam. Two thermalization chambers with circulating water bath, for mounting the quartz plates are under construction; meanwhile the plates are in a sample holder inside a small foam cup with a high resolution temperature sensor attached, and all the linear stage is inside a metallic insulated box, where the sample holder slides in and out of the laser beam. A second temperature sensor (air model) is located over the metallic box and it registers the room temperature.

A very important step is the exact alignment of the whole system and of the quartz plates every time they are mounted and for this it is used an autocollimator. The quartz plate standards are made under directions established by the ICUMSA norms SPS-1 (1998) an they are tested for these conditions before the polarimetric measurements. Standards that failed the pretests are not calibrated. Quartz plates are discs of a clear, transparent, free of inclusions or any defects crystalline quartz, cut perpendicular to the quartz optical axe and making an angle deviation smaller than 10 minutes of arc. Flatness and parallelism of the faces are tested by interferometric measurements, their actual dimensions are measured: should be 16 mm in diameter, and maximum thickness of 1,6 mm. Inmetro has seven quartz plate standards of a very high quality bought from Bernhard Halle GmBh, Germany. These standards were characterized and calibrated at the high resolution polarimeter of PTB in 2005, and are being measured in our developing setups. In our setup all instruments are being programmed under LabView and a main program reads and registers the temperature, the angular position from the encoder, and the radiation intensity from the detection system.

The procedure starts with mounting the quartz plate in a sample holder attached to a positioning platform (covered by a foam “cup”) mounted over the motorized sliding table, that is covered by the insulated box. An autocollimator is used to exactly align the plate. The temperature sensor is positioned inside the “cup” as close as possible to the plate. After covering with the box, the whole system is set to thermalize overnight. Figure 1 shows the actual setup. From right to left: the laser beam crosses the optical isolator, a quarter waveplate, the rotating polarizer, the quartz plate under measurement, the Faraday modulator, the fixed polarizer, and reaches the photodetector.

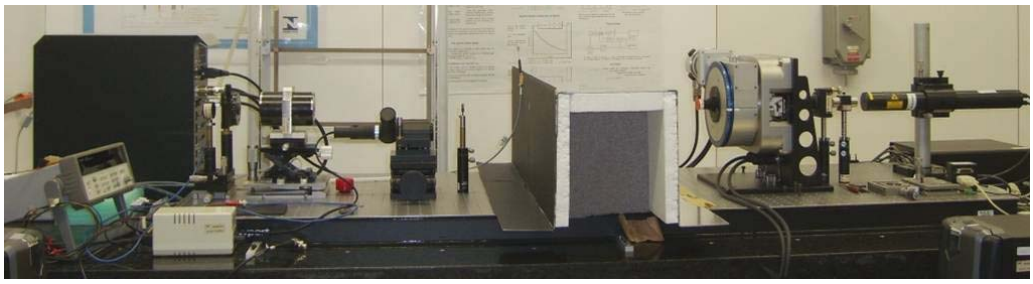

Figure 1: Photo of the new polarimeter under construction. Main components from right: stabilized He-Ne laser, analyzer in a rotator, insulated box covering the linear stage, autocollimator, Faraday modulator, polarizer, photodetector.

Two different methods for determining the null condition are under implementation in the radiation detection system. One employs a Faraday modulator built at the laboratory and a f/f modulation technique with a lock-in. The resultant acquired points near the null region are fitted by a strait line, and the crossing Y=0 point determines the angular position. The other method measures directly the output of the photodetector with a multimeter. The resultant points acquired near the region of minimum intensity are fitted to a second order polynomial, and the minimum point is calculated from de fitting parameters. This last method has already being used here by an earlier Prototype built with a manual polarizer rotator and detailed in reference Alvarenga et all (2008), which results are shown in Table 1. This Prototype polarimeter has a device to rotate the polarizer with a digital display that shows the angular position within 15″ of uncertainty in the reading. A semi-automatic acquisition mode was used, where the angular position of each step was digitized in a window in a LabView program that acquires the temperature and photodetector output from the instruments, and records all data in a table. The drawbacks are that the instrument is manually activated, what translates in long runs for acquisition of refined data point by point, and it suffers from backlash problems, what introduces errors after repetitive series of measurements. The new automatic equipment permits several repetitions of the measurements in the same day, and even will fully calibrate one quartz plate a day, contrary to the Prototype that took two weeks for measurements of only one quartz plate. At the Prototype the null detection point was obtained by direct reading in dark conditions, and use of fitting routines to the data points. By that time, our modulator was not ready yet. Our new instrument will run measurements in both configurations in order to establish comparison conditions.

4 RESULTS AND DISCUSSION

The results of the measurement of four quartz plates obtained with the Prototype are shown in Table 1. The first column shows the identification, the second column shows the PTB calibration values' obtained for the three levorotatory plates (negative sign) and one dextrorotatory plate; the third column shows the Prototype results, each one is the average of several repetitions. The presented expanded uncertainty has a coverage factor k=2. These results agree with PTB's in one tenth of a degree: they are close but not good enough for a primary calibration service, where uncertainties at the order of $\pm 0{,}001°$ are desirable.

Table 1- Polarization angular rotation and uncertainties of the quartz plates measured in the Prototype and calibrated at the PTB.

Plate Number	PTB (U k=2)	Prototype (U k=2)
IP880	-(29,749□0.001)□	-(29,750□0.003)□
IP882	-(29,749□0.001)□	-(29,752□0.004)□
IP883	-(29,747□0.001)□	-(29,749□0.002)□
IP886	(29,817□0.001)□	(29,814□0.008)□

The setup with the new equipment is being tested with both acquisition modes: the modulation and the direct reading. In both of them a Series starts with the plate in front of the laser beam, the analyzer is rotated with a larger speed until reach a previous assigned angular position, them it slowly advances though a region around 1° at the minimum in order to have points read at an average step of (0,0026±0,0003)°. Next, it scans the angular region 180° apart, and continuously turning, repeat two more scans and pauses while the linear stage moves the quartz plate away from the laser beam. The analyzer now advances to the next minimum intensity region corresponding to the two crossed polarizers (which minimum is the angular reference position); continuously turns until four scans are acquired. The rotation stage pauses again and the quartz plate is positioned in front of the laser beam. This is repeated four times, in order to have 16 scans for the quartz plate and 16 scans for the reference. The minimum (or null) angular position is determined from the fittings of parabolas (straight lines) to the region slowly scanned. The quartz plate angular rotation is obtained by the difference from the averaged four scans of the reference and each plate angular position. The angular rotation of the plate should be determined at 20,00°C and thus a correction formula (1) is used where the measured temperature during each slow scan is averaged. The resulting 16 repetitions are then averaged to give one Series result. The acquisition of one Series takes about one hour, whereas each slow scan region takes about 1 minute. During one day this Series can be repeated to study the effect of the overall Laboratory ambient conditions in the average rotation value. The seven plates were mounted and measured, and some of them have been mounted again at different days, to study repeatability conditions, using the direct method. The modulation method, which is the one in use at PTB, is still under implementation, our results are not yet satisfactory.

Figure 2 shows a plot of one slowly scanned region using the Faraday modulator, for sample IP886. The inset shows the linear fitting to the null adjacencies, where the Y=0 crossing corresponds to the angular position. For quartz plate IP886 the PTB´s calibration value is (29,817±0.001)º and the resulting average and standard deviation times 2 for this series of 16 repetitions of our measured polarization angular rotation is: (29,803±0.008)º, where the declared uncertainty U corresponds to a coverage factor k=2. This result is worse than that obtained by the earlier Prototype, as seen in Table 1. The main drawback is the correct choice of the modulation parameters, a compromise between excitation frequency and amplification power, what demanded an acquisition of a new, more potent amplifier to feed the Faraday modulator, still under tests. At this point we are still improving the modulation parameters.

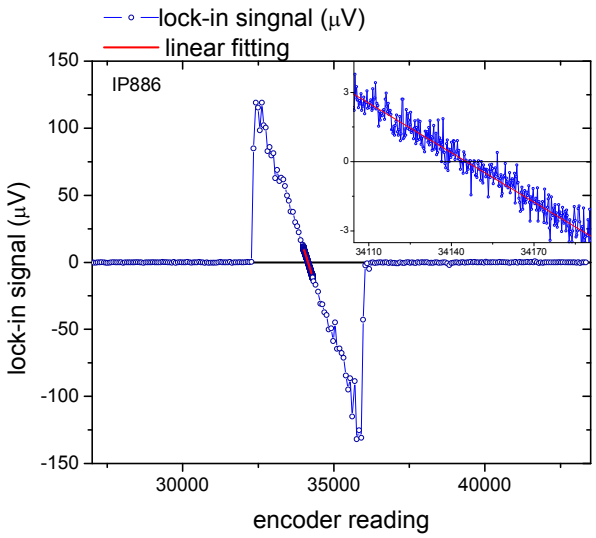

Figure 2: Photodetector f/f modulated signal output as a function of rotary encoder reading. The inset shows detail of a linear fitting through the zero signal crossing region.

Figure 3 shows a slow scanned region using the direct method. The inset presents the parabolic fitting to the minimum region. The parabola is a good approximation for the real phenomena just close to the minimum intensity region. The choice of the fitted region was made taking into account the residuals and the quality parameters for the fitting. Another test consisted in purposely choosing a larger and a smaller set of points and compare the determination of the minimum position from the parameters, in order to account for subtle variation in the choice of fitting points. This last test showed that a mistake in the choice of the group of points for the fittings resulted in an uncertainty around 8 × 10-5 angular degrees, what is one order of magnitude smaller than the standard deviation of the averaged 16 measurements. The evaluation the uncertainty due to the encoder reading was done in two steps: measuring the spread of the readings with the rotation stage standing still, and reading this position as a function of time, during a few hours, in order to account for Laboratory temperature variations over the hardware. In the most pessimistic case, this uncertainty can be estimated at the order of 4 × 10-5 angular degrees. The actual acquisition time is about 1 minute per slow scan, and about 1 hour for the whole 16 repetitions of one Series. Considering the acquisition time and the actual angular step of 0,0026°, this uncertainty in the position due to the encoder is very small.

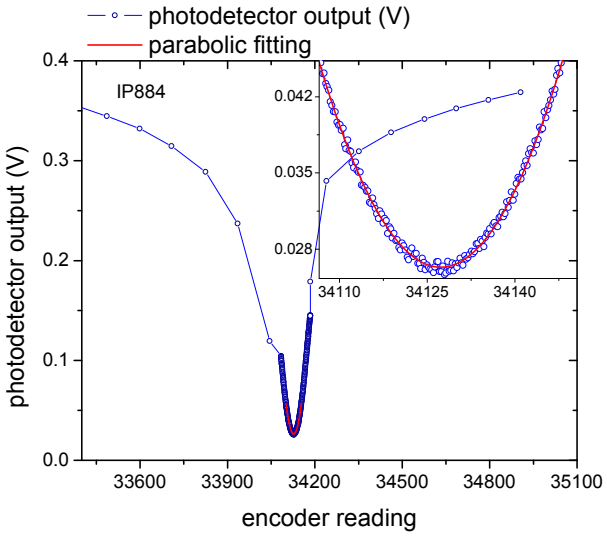

Figure 3: Photodetector output as a function of rotary encoder reading. The inset shows detail of a parabolic fitting through the minimum intensity region.

The data analysis was performed using OriginPro 8 software. The value of the polarization rotation angle of each measurement at T=20 °C, R(θ,T), was calculated by the expression stated at the norm OIML R14 (1995):

$$R(\theta,T)=\frac{\theta}{1+aT-20a} \qquad (1)$$

where α = 0,000144 is the quartz thermal expansion coefficient, θ is the measured polarization rotation angle, and T is the average temperature near the quartz plate measured during the slow scan.

From each Series of 16 measurements, an average value and correspondent standard deviation was taken. This takes into account the repeatability. Plates that have been measured more than one Series, or being mounted again at different days, had their values averaged by a weighted average. This takes into account the reproducibility.

Table 2 shows the values of the averaged measurements of the 7 standard quartz plates that have already being calibrated at the PTB. The uncertainty is U, k=2. The negative signal corresponds to the levorotatory quartz plates, by convention. Comparing the values measured at the earlier Prototype, in Table 1, with our values measured at the new setup, one can see that there is a great improvement in the precision and exactitude. Comparing our measurements with the calibrated values at the PTB, one can see a very good agreement for IP880, IP884 and IP 885, even though the uncertainty in IP884 is greater than expected. Plates IP881 and IP886 presents a mean value and deviation that is still considered in a good agreement. IP882 and 883, although their mean values differ from the PTB´s by 0,002°, their uncertainties are not good: IP882 presents a too large uncertainty, due to the reproducibility factor: from six repeated Series, one presented a value too low. We examined the ambient factors, the data analysis, and we couldn't find any reason to exclude it from the weighed average, se we keep it.

Table 2- Polarization angular rotation and uncertainties of the quartz plates measured in the laboratory and calibrated at the PTB. Identification of plate and average method is indicated in the first column; second column shows the values calibrated at PTB and the third column shows the results of our measurements at the Laboratory.

Plate Number	PTB (U k=2)	Laboratory (U k=2)
IP880 [a]	-(29,749±0,001)°	-(29,7491±0,0004)°
IP881 [a]	-(29,750±0,001)°	-(29,7511±0,0008)°
IP882 [c]	-(29,749±0,001)°	-(29,751±0,008)°
IP883 [b]	-(29,747±0,001)°	-(29,7489±0,0002)°
IP884 [c]	(29,859±0,001)°	(29,859±0,003)°
IP885 [a]	(29,796±0,001)°	(29,797±0,002)°
IP886 [a]	(29,817±0,001)°	(29,819±0,001)°

[a] repeatability: Series of 16 measurements; simple average

[b] reproducibility: different Series, same mounting; weighed average

[c] reproducibility: different Series and mountings; weighed average

IP883 presents a very small uncertainty, derived from the weighed average of two Series, one measured at one day, and the other at the following day. Their mean individual values and standard deviation (k=1) are –(29,749±0,0005)° and –(29,7488±0,0004)°, what shows a very good repeatability of the measurements, in these two Series comprising 32 repetitions. The uncertainty calculated just as the standard deviation of the average of these values, without weighing, leads to a larger value of ± 0,0002°, what becomes closer to the PTB´s when making k=2, thus resulting in ± 0,0008°. The results are presented with the small number in order to be consistent in the comparisons. Reproducibility measurements are under way, and those figures may change.

The results of the measurements presented in Table2 are depicted in Figure 4; the same scale is used in the four graphs to compare the uncertainties. Red triangles are the calibration values measured at the PTB in their high resolution polarimeter, declared uncertainty is U, k=2. Blue dots and blue diamonds are the levorotatory and dextrorotatory plates measurements performed at the first assembling of the new polarimeter set up at Diop/Laint. The uncertainty is k=2.

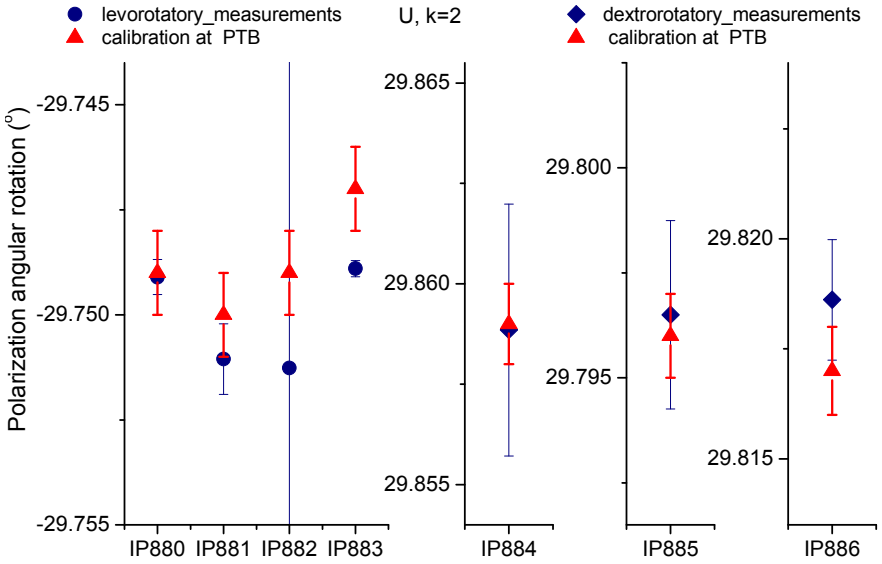

Figure 4: First results of the measurements of the polarization angular rotation of seven quartz control plates by the new equipment at Diopt/Laint: 4 levorotatory and 3 dextrorotatory. Triangles are previous calibration values measured at the high resolution polarimeter at PTB.

5 CONCLUSIONS

Here we presented the first results of the measurements of seven quartz control plates realized in the new set up of the high resolution polarimeter under construction at Diopt/Laint. Comparison with a previous Prototype showed a large improvement in the repeatability and time spent for data acquisition: whereas in the Prototype one slow scan took 30 min, now it takes 1 minute. One Series of 16 repetitions (32 slow scans plus translating movements) now takes 1 hour. Time is important not only to fasten calibrations, but to assure ambient factors are in the best stable conditions during the Series measurement. The results show good agreement with the calibrated values at PTB, and the uncertainties are close but not yet the desirable for a calibration service. The assembling of the Polarimeter is still under improvement. Considering that the thermalization chambers with the special sample holder are not yet available, and the measurements were not performed under the final assemblies, these results are still very promising for offering soon a calibration service for the Industry.

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