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HIGH-ACCURACY POLARIMETRIC CALIBRATION OF QUARTZ CONTROL PLATES

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Abstract: Quartz Control Plates used as standards for the quality control of polarimetric and saccharimetric measurements have to be calibrated with low uncertainty. A set-up for this is operated at PTB, and INMETRO is building a similar one. In both set-ups, the null detection of the polarimetric signal is of high importance. A novel procedure for this is proposed and tested which avoids the use of an electronic feedback loop.

Keywords: Polarimetry, Saccharimeter, Calibration.

1. INTRODUCTION

Polarimetric and saccharimetric measurements based on polarimetry are important in many branches, for example in the food or sugar industry, in the chemical industry, in the pharmaceutical industry, etc. Those methods are based on the measurement of the polarization rotation angle of light after passing through an active medium. A saccharimeter is a special kind of polarimeter designed to measure the amount of saccharosis present in a sugar solution, and the result is expressed as Sugar degrees (°Z), in a scale defined by the ICUMSA, the International Commission for Uniform Methods of Sugar Analysis. To calibrate instruments such as polarimetric saccharimeters according to the OIML [1], Quartz Control Plates (QCPs) are used as standards, and the ICUMSA gives specifications and recommendations for such standards [2].

The QCPs used for calibrating the industrial measuring systems have to be traceably calibrated themselves before they are used. The Physikalisch-Technische Bundesanstalt, PTB, as the National Metrology Institute of Germany, performs first-level calibrations of QCPs of several institutes and companies, using a low-uncertainty polarimeter. Historically, the polarimetric methods applied to sugar and quartz have been developed at PTB since the 19th century.

On the other hand, such calibration service is of high importance for Brazil - as it is a big producer of sugar cane and sugar byproducts, for both the internal market and foreign trade. The payment centers in the sugar factories rely upon saccharimetric measurements for the determination of the product prices. Furthermore, traceability is one of the primary requests of the ISO 17025 standard, and all calibration laboratories must be in compliance with it. The increasing number of sugar factory laboratories seeking accreditation has led INMETRO, the Brazilian National Metrology and Normalization Institute, to build a reference polarimeter for calibration of quartz transfer standards used to control the saccharimeters in the sugar factories. For this purpose, a collaboration with PTB has been established since 2004.

In this presentation, we report on recent developments in the high-accuracy polarimetric calibration of quartz control plates that are used as transfer standards.

2. THE QUARTZ CONTROL PLATES (QCPs)

Substances that are able to rotate the polarization plane of the light are called “optically active”, and sugar and crystalline quartz belong to this class. In a sugar solution, the polarization rotation is proportional to the number of sucrose molecules present in the pathway of the light. The rotation is temperature-, concentration- and wavelength-dependent. The ICUMSA unified the analytical methods for the sugar industry, and set the standards for the International Sugar Scale (ISS) and the specifications for saccharimeters [2]. The ISS is defined upon a prescribed sugar solution called “standard” or “normal” solution, and essentially relates the sucrose concentration to the light polarization rotation. Using those solutions as a standard for the verification of instruments, has, however, many disadvantages, e.g. a degradation due to fungi and bacterial attack and an alteration in the concentration due to evaporation. Also, the polarization rotation is very sensitive to the temperature [3]. The search for a stable substance to act as a substitution standard led to the choice of crystalline quartz which is oriented and cut in such a way that it matches the polarization rotation of a normal sugar solution, but without bringing about the drawbacks mentioned above. Its wavelength dependence of the polarization rotation is similar to sucrose, however, it is much less sensitive to temperature variation [4]. Quartz plates are thus made in accordance with the rules prescribed by the ICUMSA [2].

3. COLLABORATION PTB-INMETRO

In PTB's set-up, a special electronics is used which performs the measurement automatically. The angle of rotation is adjusted with very low uncertainty using a complex feedback control loop. This device was built by a small company in the 80s already and is no longer commercially available. So INMETRO has to find another way of realizing the polarimetric set-up. As large numbers of calibrations are expected, an automatic set-up is necessary.

The aim of the collaboration between PTB and INMETRO was to evaluate an alternative method of high-accuracy polarimetric measurements using a set-up built from commonly available standard components and to compare its estimated performance with the set-up currently in operation at PTB. This involves, for example, the use of a very high precision automatic rotation stage with an optical encoder, for rotating the polarizer and reading the rotation angle. Accurate temperature reading of the QCP can be achieved by the use of high-precision temperature sensors and a high-quality temperature stabilization of the QCP. Also, a new detection technique of the null point is tested at PTB which allows the actuators and data acquisition to be completely computer-controlled, for example by a LabView program (INMETRO). The actual status of the INMETRO set-up is testing the components and parts already available. To estimate the accuracy that can be achieved with the new method, PTB's set-up was modified in such a way that it operates similarly to the new detection principle which is proposed in the following.

4. RECENT DEVELOPMENTS

4.1. Calibration set-up at PTB

The principle of PTB's calibration set-up goes back to fundamental polarimetric principles as described by Flügge [5] or Bünnagel [6]. As light source, a Helium-Neon laser (633 nm) is typically used, but other light sources are possible, too. The rotation of the vibration direction of linearly polarized light by the specimen is measured as follows:

A polarizer P (see fig.1) generates the linearly polarized light and a second polarizer acts as analyzer A for the state of extinction with its vibration direction perpendicular to the first one in the initial state, i.e. without the QCP inserted in the optical path.

When the QCP is inserted between the polarizers, the vibration direction of light is rotated by the specimen-characteristic angle α. When the polarizer P is rotated by α, too, the state of extinction can be adjusted again. By this, α is determined.

For a high resolution detection of this null condition, measuring the transmitted light intensity behind the analyzer is not sufficient. Therefore, a modulation technique is applied according to [6]. It uses a Faraday modulator (fig.1) and applies a lock-in technique. This increases the sensitivity of the determination of the state of extinction by several orders of magnitude.

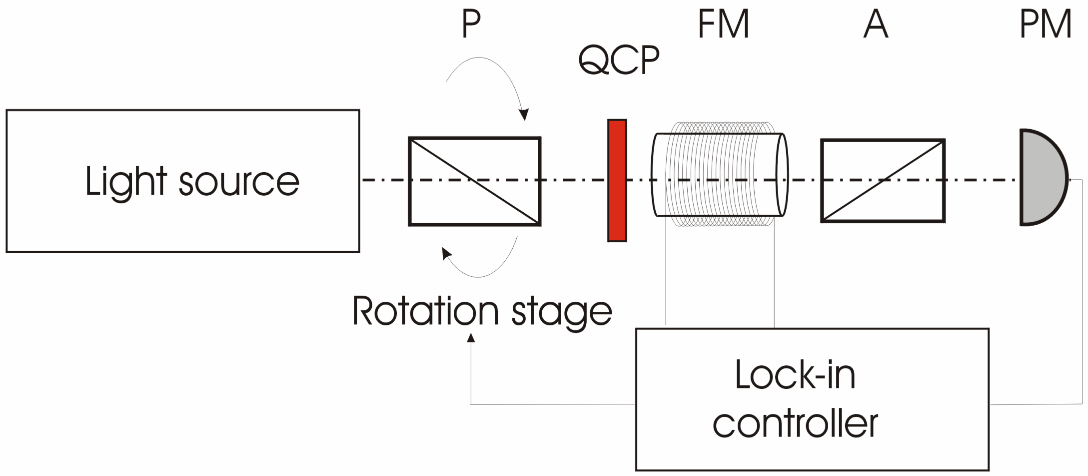

Fig. 1. Measurement principle of the polarimetric calibration set-up of PTB.
FM: Faraday modulator, PM: Photomultiplier, P: Polarizer, A: Analyser, QCP: Quartz Control Plate.

Let Ψ be the deviation of the analyzer angle from the orientation perpendicular to the vibration direction of light after passing through the QCP. For Ψ = 0, with a current of zero through the Faraday modulator, no light will reach the detector (for ideal components). The effect of an alternating modulator current with frequency f in this case is that for a positive and a negative current, the vibration direction of light is antisymmetrically rotated, thus leading to a symmetric intensity on the detector. For Ψ ≠ 0, this symmetry is lost (see fig. 2) and the lock-in amplifier used detects and measures the deviation from the balanced state with high sensitivity. The balanced state is characterized by a maximum of the signal components with frequency 2f.

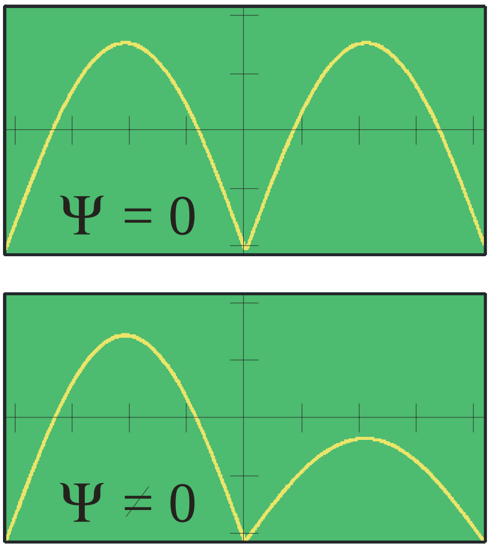

Fig. 2. Principle of the signal forms of the detector signal in the balanced and unbalanced state

The special electronics that was built in the 80s, as mentioned before, generates an electronic feedback signal for the motorized high-accuracy rotation stage of the polarizer and adjusts the state of extinction automatically. After this adjustment, the angle information is read from the rotation stage with a high-accuracy angle encoder by the operator.

Fig.3 shows the flowchart of this unit. Initially, the electronics receive a start command and start to turn the rotary stage with the polarizer P at a rather high velocity (6 revolutions per minute). When the angle comes near to the state of extinction, the electronics detect the modulated signal with frequency f and switch to a slow velocity (1 revolution per minute). When the 2f-signal is detected, the continuous motion is stopped and a PID loop controller (applying a feedback signal that is the sum of a component **p**roportional to the detected signal, a second one corresponding to the **i**ntegral and a third one regarding the **d**ifferential of the detected signal) adjusts the rotary stage to the maximum of the 2f-signal.

Parallel to this, the high voltage (HV) applied to the PM is increased because the output signal decreases rapidly. After the system has reached a stable position, the angle is read out.

The typical output voltage (from the PM signal) to drive the polarizer motor as a function of the rotation angle of the polarizer rotation stage is shown in fig. 4.

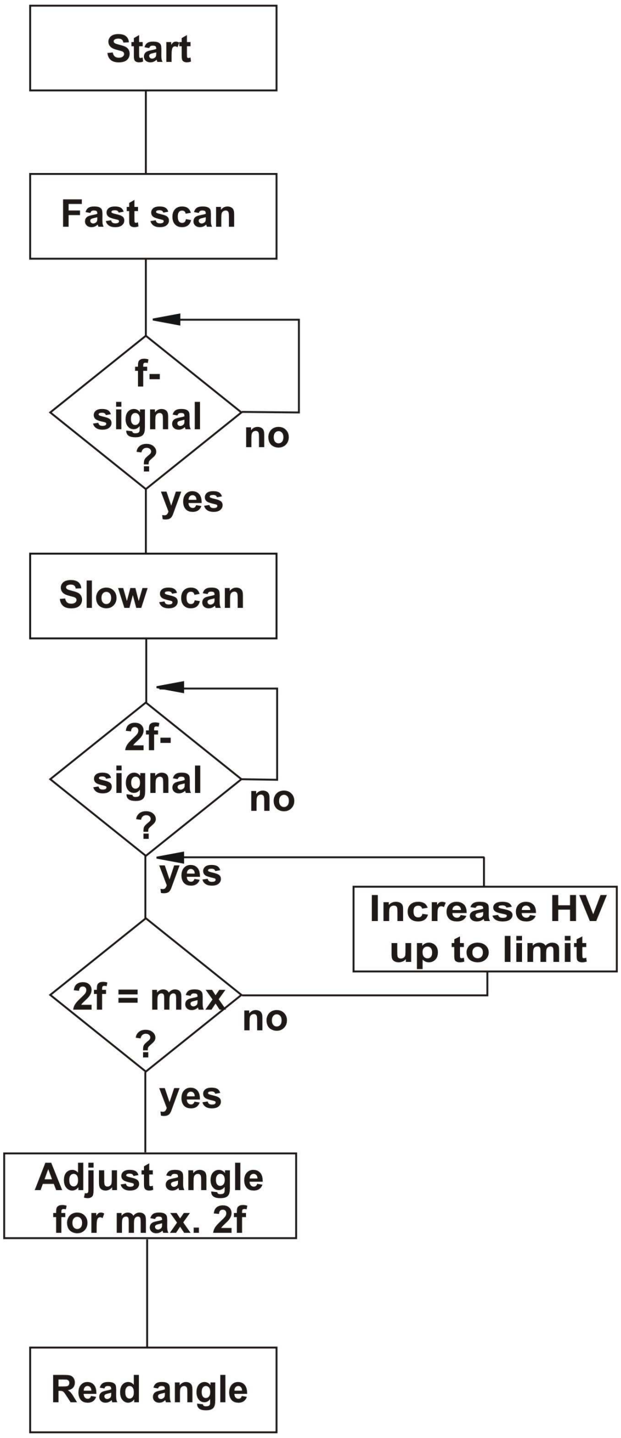

Fig. 3. Flowchart of the automatic electronic feedback unit for angle adjustment.

4.2. Uncertainty of the calibration set-up at PTB

For a QCP to be qualified as highly accurate calibration device according to [2], several prerequisites have to be fulfilled. Therefore a quality test is essential which guarantees that the flatness and parallelism of the QCP faces, the purity and the play of the plate in the holder are within stringent limits. As the polarimeter used for determining the optical rotation of the QCPs is not suitable for detecting unqualified specimens, additional measuring set-ups using interferometric or classical polarimetric techniques (see references in [6]) are necessary for testing these properties of the QCP. Specimens not satisfying the specifications are rejected.

Once the QCP has passed the quality test, it is adjusted within the polarimeter. The angular orientation with respect to the system's optical axis is checked by means of an autocollimator. This is of high importance especially in systems which use a laser as light source. The effects of lasers as light sources in polarimeters are discussed in detail in [7].

For the set-up with high quality components and a proper alignment, the main uncertainty influences are the specimen temperature, the null detection and the rotary encoder reading. In the following, the uncertainty which is achievable with the set-up is analyzed.

With a typical sensitivity coefficient in the order of 0.01°/K (depending on the wavelength used and the rotation value of the QCP), the temperature of the specimen has to be known with an accuracy of about 0.01 K for an uncertainty contribution of 0.0001°. A highly accurate stabilization and measurement of the temperature of the QCP is therefore necessary. This is achieved by air temperature stabilization of the laboratory and by mounting the specimen in an additional housing with through-flow of water from a thermostat. A threefold stabilization with an additional housing is possible, too. After inserting the specimen, temperature equilibrium has to be reached. The specimen's temperature is measured by an absolutely calibrated temperature sensor with an uncertainty of 0.005 K. Additionally a well-characterized, long (49 mm) reference quartz [3], positioned close to the QCP and with the same temperature stabilization and measurement, is moved into the measurement position after the QCP has been measured, and its optical rotation is measured, too. This additional measurement is used as a sensitive cross-check.

Rotary encoders can have very low uncertainties, in particular when they have been calibrated by appropriate means [8]. For the encoder used in this set-up, an uncertainty of 0.0001° is a realistic value.

The uncertainty contribution from the null detection was determined by repeated detection. The feedback loop of the electronic controller can adjust the rotation angle with a reproducibility and uncertainty of 0.0002°.

According to the ISO guidelines [9], the uncertainty of PTB's set-up is 0.0005° for an expansion factor of k=2, corresponding to a 95% probability for the value of the measurand to be within the stated range.

5. ALTERNATIVE METHOD TO DETERMINE THE STATE OF EXTINCTION

As the electronics described above is no longer available on the market, a different method for determining the state of extinction with commonly available standard components had to be evaluated and is presented in the following.

With progress in computer capabilities, measurement strategies have changed. Feedback loops for adjusting the state of extinction could be realized by software, instead of electronics. To eliminate also any possible offsets with a proportional loop controller, a different null detection is proposed.

To find the desired angle where the lock-in signal is zero, the polarizer stage is rotated in one direction and the angle information is recorded from the angle encoder with high accuracy. This can be done stepwise or in continuous motion. Parallel to the positional measurement points, the lock-in signal is captured. To the data points in the vicinity of $\Psi = 0$, a straight line is fitted and from this, the angle corresponding to $\Psi = 0$ is determined (see fig.5).

5.1. Results

The alternative method proposed in the previous section was tested and experimental results are presented in the following. For the test measurements, the automatic electronic feedback system of PTB's set-up was inhibited as far as possible. Unfortunately, several components of the electronics could not be deactivated, namely several components introducing time constants into the output signal and the automatic high-voltage control (HV) of the photomultiplier (PM). As a consequence, the test conditions are not optimized for indicating the limits of the proposed method.

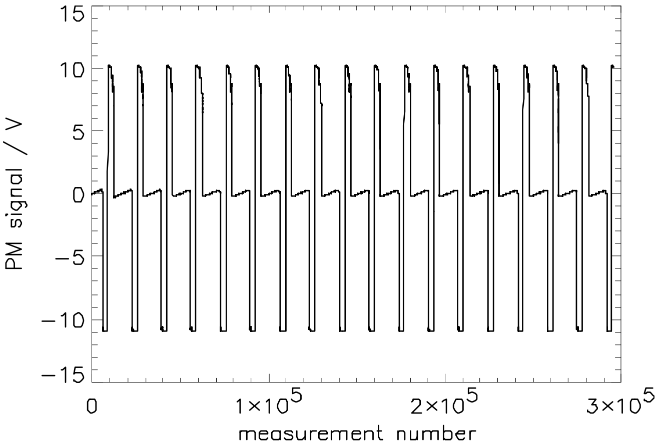

Fig. 4. Output voltage of photomultiplier as a function of the measurement number during continuos rotation of the polarizer.

During a continuous rotation of the polarizer at the lowest possible speed (about 0.01 revolution per minute) the photomultiplier signal and the angle encoder output were recorded (see fig. 4). Due to the automatic photomultiplier supply voltage control loop, the large parts of the output signal are close to zero, as shown in fig. 4. This corresponds to relative orientations of polarizer and analyzer away from extinction, where a large amount of light can pass and consequently the PM supply voltage is driven down by the electronics.

The relevant data are in the ascending flanks of the signal, where the PM supply voltage is at its maximum. Fig. 5 shows this part of the measurement data enlarged as a function of the rotation angle. A line is fitted to the edge data. From this linear fit, the angle corresponding to an ordinate value of zero can be extracted, giving the result for the state of extinction.

For the measurement sequence captured with the modified automatic set-up and shown in fig. 4, a reproducibility of less than 0.001 degree was achieved. This is one prerequisite for high-accuracy polarimetric measurements.

For a new measurement set-up designed for this method, only the small angular range around the state of extinction would be used and the data can be captured more precisely. Additionally, a photomultiplier electronics can be used that is free of the restrictions of PTB’s automatic set-up. By this means, an even better accuracy seems to be possible.

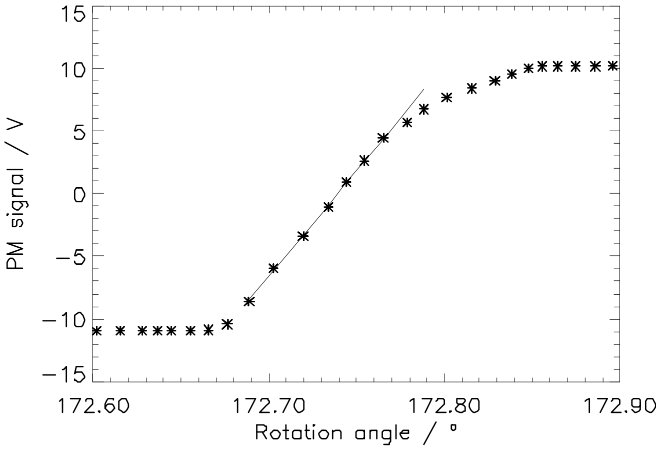

Fig. 5. Output voltage of photomultiplier (PM) as a function of the rotation angle (arbitrary units) corresponding to an ascending flank of the signal of fig. 4.

6. CONCLUSION

A method for determining the rotation angle of Quartz Control Plates with commonly available standard components is presented and first measurements verifying the potential of this method have been performed. It is based on continuously scanning the angle coordinate and fitting a straight line to the slopes of the modulated detector signal. Starting from PTB’s proved set-up, uncertainty contributions are listed. The new method can be expected to reach an uncertainty of at least the same order of magnitude when operated with suitable equipment.

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