CHAPTER-4

CALIBRATION OF PC BASED POLARIMETER BASED ON MALUS' LAW
4.1 CALIBRATION OF PC BASED POLARIMETER

In the previous chapters, the design and development of hardware and software for PC based polarimeter using Malus law are described. This chapter is confined to the calibration of PC based polarimeter using standard values and to present experimental results of various samples. Based on these results the performance of the system is evaluated.

In order to calibrate the PC based polarimeter the quartz standards are selected, since quartz is optically stable over time. Changes in its optical rotation are the result of optical stress or surface scratches caused by physical handling.

A quartz plate consists of one or two thin discs of optically worked crystalline quartz mounted in a tube, forming a ‘standard’ sample for a polarimeter.

Quartz exists in two forms; one form, called Dextro-Rotary quartz, rotates polarized light in a clockwise direction, the other form, Laevo-Rotary quartz rotates in an anti-clockwise direction.

The specific rotation of quartz (i.e. the rotation of a 1mm thick plate) at 20°C for light of wavelength 589.4400nm (sodium yellow) is 21.7°. Variations with both temperature and wavelength are well known for quartz.

The standards employed for the calibration in the present work are given in the Table 4.1. Fig 4.1 shows the photograph of these standards.
<table>
<thead>
<tr>
<th>S.NO</th>
<th>OPTICAL ROTATION (at 650 nm) in deg</th>
<th>DEXTRO/LAEVO</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21.5</td>
<td>Dextro</td>
</tr>
<tr>
<td>2</td>
<td>29</td>
<td>Dextro</td>
</tr>
<tr>
<td>3</td>
<td>50.5</td>
<td>Dextro</td>
</tr>
<tr>
<td>4</td>
<td>8.5</td>
<td>Dextro</td>
</tr>
<tr>
<td>5</td>
<td>21.5</td>
<td>Laevo</td>
</tr>
<tr>
<td>6</td>
<td>29</td>
<td>Laevo</td>
</tr>
<tr>
<td>7</td>
<td>50.5</td>
<td>Laevo</td>
</tr>
<tr>
<td>8</td>
<td>8.5</td>
<td>Laevo</td>
</tr>
</tbody>
</table>

Table 4.1: list of Quartz Standards
Fig 4.1 Quartz Standards
Before testing with the above standard samples, first, the sample tube is removed and $S_1$ value measured. This value is $I = I_1 = 8.2 \text{mV}$. This value is entered in the ‘angle of rotation’ sheet (see Fig 3.1a) which is the front panel of the LabVIEW (there, it is written as VO). The quartz standard is placed now. This rotates the polarized light passing through it. Hence the output of $S_1$ according to Malus will be less than $I_1$. This is seen in $S_1$ o/p (mV) column, and the result (optical rotation) will be displayed in the ‘Result’ column using the formula $\theta = \cos^{-1}\sqrt{(I/I_1)}$ by the LabVIEW. The same process is repeated with all standards and the results are tabulated as shown in Table 4.2.
<table>
<thead>
<tr>
<th>S.NO</th>
<th>Θ STANDARD VALUE (TV)</th>
<th>Θ MEASURED VALUE (MV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21.5, Dextro</td>
<td>21.3, Dextro</td>
</tr>
<tr>
<td>2</td>
<td>29.0, Dextro</td>
<td>28.8, Dextro</td>
</tr>
<tr>
<td>3</td>
<td>50.5, Dextro</td>
<td>50.3, Dextro</td>
</tr>
<tr>
<td>4</td>
<td>8.5, Dextro</td>
<td>8.4, Dextro</td>
</tr>
<tr>
<td>5</td>
<td>21.5, Laevo</td>
<td>21.5, Laevo</td>
</tr>
<tr>
<td>6</td>
<td>29.0, Laevo</td>
<td>28.8, Laevo</td>
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<td>7</td>
<td>50.5, Laevo</td>
<td>50.3, Laevo</td>
</tr>
<tr>
<td>8</td>
<td>8.5, Laevo</td>
<td>8.4, Laevo</td>
</tr>
</tbody>
</table>

Table 4.2: Results with the Quartz Standards
From the above results, the instrument performance characteristics like accuracy, precision and resolution were evaluated.

4.2 Performance characteristics of PC based polarimeter

4.2a. Accuracy

Accuracy of a measuring system is defined as the closeness of the instrument output to the true value of the measured quantity (as per standards). However in usual practice, it is specified as the %deviation of inaccuracy of the measurement from its true value (TV).

Accuracy can be specified in the following form.

\[
\text{% of the true value (TV) = \frac{\left(\text{Measured value (MV)} - \text{True value (TV)}\right) \times 100}{\text{True value}}}
\]

Accuracy of the instrument mainly depends on the inherent limitations of the instrument as well as on the shortcomings in the measurement process.

Inherent shortcomings of the instrument:

Errors are inherent in instruments because of their mechanical structure. They may be due to construction, calibration or operation of the instruments or measuring devices. These errors may cause the instrument to read low or high.

The instrumental errors in our PC based polarimeter arises due to the following reasons as discussed below.
1) The most important design aspect is to keep the distances as $BS_1A_2=BS_1BS_2=BS_2A_3=BS_2A_1$, so that any changes in the light intensity will equally appear for all $S_1$, $S_2$, $S_3$. Any disturbance to the above condition will seriously affect the accuracy of the instrument. Hence, the disturbance to the above condition is the prime source of instrumental error (systematic error).

2) After the analyzers 1 and 2, the sensors $S_1$ and $S_2$ should be placed without any gap. If there exists any gap, as the reduction in the intensity is directly proportional to the square of the optical path, $S_1$ and $S_2$ record less intensity which gives the error in the readings. Hence, accuracy of the instrument will be reduced. Therefore any disturbance to the above condition is another source of instrumental error (systematic error).

3) Limitations in the resolution of the photo sensor (OPT-301) and DAQ card (USB-6259).

Accuracy of the instrument w.r.t standard 1; $\theta = 21.5$, D

$\% TV = -0.2/21.5*100 = -0.93 \%$

Accuracy w.r.t standard 2; $\theta = 29$, D

$\% TV = -0.2/29 = -0.68 \%$

Accuracy w.r.t standard 3; $\theta = 50.5$, D

$\% TV = -0.2/50.5*100 = -0.39 \%$

Accuracy w.r.t standard 4; $\theta = 8.5$, D
% TV = \(-\frac{0.1}{8.5} \times 100\) = -1.1 %

Accuracy w.r.t standard 5; \(\theta = 21.5\), L

% TV = \(\frac{0}{21.5} \times 100\) = 0%

Accuracy w.r.t standard 6; \(\theta = 29\), L

% TV = \(-\frac{0.2}{29}\) = -0.68 %

Accuracy w.r.t standard 7; \(\theta = 50.5\), L

% TV = \(-\frac{0.2}{50.5} \times 100\) = -0.39 %

Accuracy w.r.t standard 8; \(\theta = 8.5\), L

% TV = \(-\frac{0.1}{8.5} \times 100\) = -1.1 %

Average accuracy = -0.65 % of TV.

4.2b. Precision (Repeatability)

Precision is defined as the ability of the instrument to reproduce a group of measurements of the same measured quantity made by the same observer, using the same instrument, under same conditions. The inconsistency in the measured values is due to random or accidental errors.

To calculate the precision of the instrument a standard of 29\(^o\) is selected and the optical rotation is measured. The same experiment is repeated for 8 times for the same input (standard) and obtained the output readings as follows.

28.6, 28.6, 28.5, 28.5, 28.6, 28.6, 28.5, 28.6

Mean reading = 28.96 (TV)
1). % of deviation for 28.6(MV) from 28.96(TV) = \( \frac{28.6 - 28.96}{28.96} \times 100 \)

\[ = \frac{-0.36}{28.96} \times 100 \]

\[ = 1.24\% \]

2). % of deviation for 28.5(MV) from 28.96(TV) = \( \frac{28.5 - 28.96}{28.96} \times 100 \)

\[ = \frac{-0.46}{28.96} \times 100 \]

\[ = 1.58\% \]

**Average precision = ± 1.376 % of TV**

4.2c. Resolution

Resolution is the smallest input change that can be detected (measured) by the instrument. It is calculated for PC based polarimeter as follows.

The PC based polarimeter uses a 16-bit data acquisition (DAQ) card. The reference voltage of its A/D converter is 10v.

Then the LSB value = \( \frac{\frac{1}{2^{16}}}{1} \times 10V = 10 V / 65,535 \)

\[ = 152uv \]

This is the **resolution** of DAQ card. This implies the smallest change in the input of A/D converter that can be detected by the PC = 152uv.

Now, the smallest change in optical rotation (i/p) that causes a change of 152uv at sensor S1 is to be calculated.
From the screen shots 1 (Fig 3.1a), it is evident that before placing the quartz standard (21.5°), sensor $S_1$ output ($I$) = 8.2mv.

After placing the quartz standard, sensor $S_1$ output ($I$) = 7.1mv (due to an optical rotation of 21.3°).

I.e. a rotation of 21.3° is causing a change in the output of $S_1$ which is equal to $(8.2 - 7.1) \text{mv} = 1.1 \text{mv}$.

Or, for a change of 1.1mv, the rotation required = 21.3°. Then for a change of 152uv, the rotation required is

$$= \frac{21.3}{1.1 \text{mv}} \times 152 \text{uv} = 2.9°$$

which is the resolution of PC based polarimeter.

This resolution can be further increased in future by using a 24-bit DAQ card. The LSB value of 24-bit DAQ is $(\frac{1}{2^{24}-1}) \times 10 \text{V} = 0.06 \text{uV}$. Then the resolution of PC based polarimeter from above procedure becomes $21.3/1.1 \text{mv} \times 0.06 \text{uv} = 0.01°$.

Finally, the PC based polarimeter has the following specifications.
4.3 SPECIFICATIONS OF PC BASED POLARIMETER

Measurement mode: optical rotation, specific rotation
Measuring Scales: Angular degrees
Measuring Range: (+/- 90°)
Wavelengths: 650nm, 589nm
Wavelength selection: Manual
Accuracy: - 0.65 % of TV
Precision: ± 1.376 % of TV
Resolution: 2.9° (with 16-bit DAQ)

Light source: 100 watts tungsten halogen lamp
Sample compartment: Accepts sample tubes of lengths up to 2.5cm. Collar dia = 30mm
Power requirements: 220v AC line voltage, 50HZ
Frame material: GI tube of 1 inch dia