Rudolph Autopol III S2 Polarimeter User's Guide

Version 1.0

Edited by Dr. Hartmut G. Hedderich

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The following guide describes the use of the Rudolph Autopol III Polarimeter. The guide is intended to assist instrument users after the initial training session.

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The Rudolph Autopol III Polarimeter measures:

- Optical rotation of a compound (required input: pathlength in mm)
- Specific rotation of a compound (required input: pathlength in mm and concentration in g/100ml)

1. Hardware

The instrument is depicted above. It has a chamber for the polarimeter cell, which can take cells up to 200mm length. It can handle center fill and flow-through tubes. Steel rails position the cell accurately in the optical path. The instrument has a build-in temperature probe (range 10° to 100°C).

The Autopol III has two light sources providing 589nm (sodium line) and 546nm (mercury line) wavelengths. The measurement range for optical rotation is ±89° with a resolution of 0.001° arc and ±999.99° arc for specific optical rotation (0-99.9% concentration). The accuracy is 0.002° arc up to 1° as well as 0.2% above 1°.

The instrument is controlled via a touch screen (imbedded Windows 7 OS).

The user provides a polarimeter cell for the measurements. RIC does not have cells for general use.

2. Principles

The polarimeter measures the optical activity of organic and inorganic compounds in solution. Plane-polarized light passes through a solution containing the compound of interest and the light rotation is measured. The degree of rotation depends on the structure and concentration of chiral molecules in solution.

The specific rotation is defined as:

$$[\alpha]_{\lambda}^{T} = \frac{\alpha_{\lambda}^{T}}{c \cdot l}$$

with [α] = specific rotation

α = observed rotation in degree arc

l = pathlength in dm (decimeter)

c = concentration in g/100ml (or density for neat liquids)

T = temperature in °C

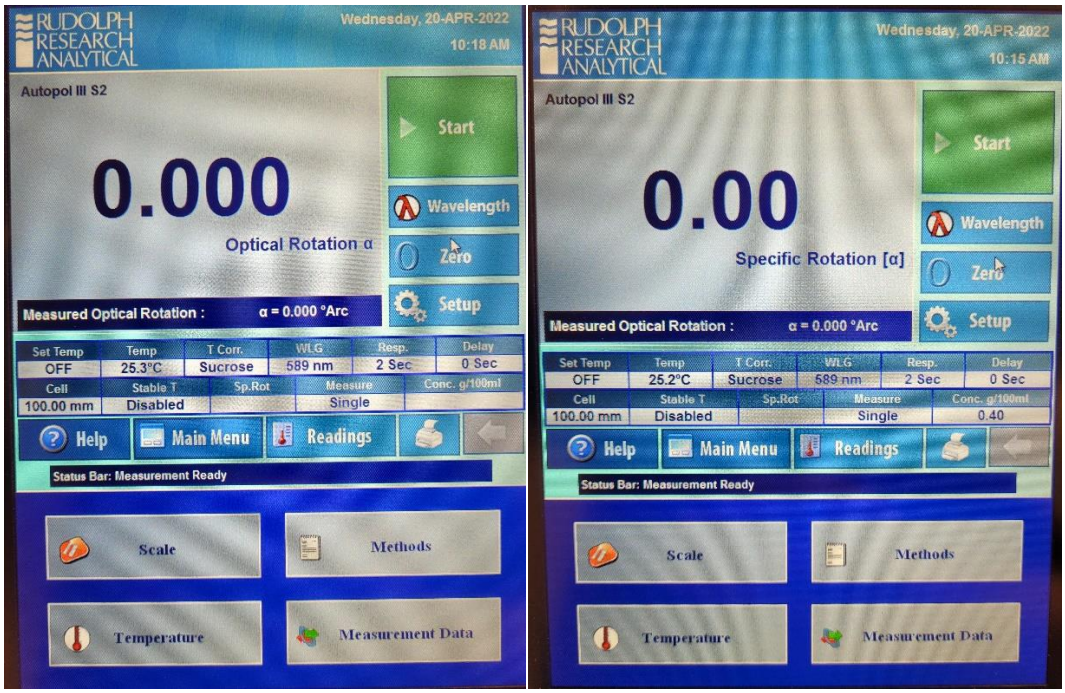
and λ = wavelength of measurement

3. Startup

Remove dust cloth. Turn the instrument on and wait for 10 minutes to allow the instrument to warm up.

The screen looks as follows:

Optical Rotation: Specific Rotation:



- Press **Zero** to zero the instrument
- Select the desired wavelength by pressing the **Wavelength** key. The default is the 589nm sodium D line, second option is the 546nm mercury line.
- Press **Scale** to select the measurement mode:
  - Optical Rotation (input pathlength in mm)
  - Specific Rotation (input pathlength and concentration in g/100ml)
- Fill a clean(!) cell with your solvent. Place cell in instrument and press **Zero** to remove any influence of your solvent to your measurement.
- Remove and clean cell carefully. Dry cell.
- Fill the cell with your sample solution and place in instrument. Make sure that you have no bubbles in your solution!
- Press **Start** and record the measurement.
- Remove cell. Clean and dry cell for further measurements.

- When finished with your measurements please turn the instrument off and put dust cloth over the instrument.

4. Problems with measurement

There are two different kind of problems that you can observe:

- Polarimetric Imbalance:
  - Physical or chemical changes within the sample (i.e.: reactions, T changes)
  - Voltage fluctuations
  - Changing wavelengths with cell in sample chamber
  - Noise contribution from light source, PMT, sample absorption, sample scattering
- Sample Errors:
  - Particulate matter
  - Air bubbles (!)
  - Incomplete mixing of sample in solvent
  - Thermal gradients

5. Notes