

# IMSERC User Manual for Rudolph Autopol IV Polarimeter

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## INTRODUCTION

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Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at <http://imserc.northwestern.edu/pcm-instruments.html> by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please **stop**, and **get help**. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

## SAFETY

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All users of IMSERC must review the general safety policies at <http://imserc.northwestern.edu/about-policies.html>. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment

You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at <https://learn.northwestern.edu>. Upon completion of the certificate, it will take overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room B170. Protective eyewear is required in this room, and gloves should be removed when using the computer.

## DATA MANAGEMENT

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Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, otherwise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their

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data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor's group folder on 'imsercdata.northwestern.edu' under others\Polarimeter every 60 minutes. Please follow instructions at <http://imserc.northwestern.edu/about-general-faq.html#data> for details about data access.

## DEFAULT INSTRUMENT STATUS

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The default working condition of Polarimeter is as follows:

1. You must start your reservation through NUcore to be able to turn on the Polarimeter. It will take 30 minutes to warm up, so be sure to start your reservation 30 minutes before you intend to collect data
2. The default 'Polarimeter' user account should be logged in. In case the computer was restarted, the password for the 'Polarimeter' account is \_\_\_\_\_ (see hardcopy next to the instrument)
3. Once the polarimeter has been turned on, the screen located on the instrument itself will initiate. Once the initiation is completed, it will state "waiting for connection"
4. Once the instrument is "waiting for connection", start the data collection software by double-pressing on the 'Rudolph' icon on the desktop of the control computer
5. Once the data collection software initializes, the screen on the instrument will state "external Connected"
6. Once the control computer is connected successfully to the instrument, the following screen will appear on the desktop:
  - a. Live reading, not corrected for temperature (figure 6a)
  - b. Parameters that can be set (T Corr. = Temperature Correction, WLG = Wavelength, Resp = Response time, measure) (figure 6b)
  - c. More parameters specific to experiments (Delay = Time delay, Cell = Length of Sample Cell, Conc./Density = concentration that is entered when measuring specific rotation, Sp. Rot. = The specific Rotation that is supplied by the user when measuring concentration) (figure 6c)
  - d. Main Menu Options (depending on the type of experiment that is being conducted, this is where you change specific parameters i.e., cell length, concentration, specific rotation etc.) (figure 6d)
  - e. The corrected optical rotation reading (figure 6e)

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**6a** Live Reading  
 $\alpha = 0.000 \text{ }^\circ\text{Arc}$

**6e** 0.000

Set Temp	Temp	T Corr.	WLG	Resp.	Measure
N/A	-64.2°C	OFF	405 nm	4 Sec	Multiple

Delay	Stable T	Cell	Conc./Density	Sp.Rot
0 Sec	N/A	100.00 mm		

**6b**

**6c**

**6d**

Status Bar: Measurement Ready      Probe Status: In holder

Optical Rotation    [α] Specific Rotation    SR - Plus    Concentration

Z °Z(589)    Temperature    Response Time    Statistics

Methods    Measurement Data

When you are done with your measurement, please remember to:

1. End your reservation in NUcore.
2. Leave lab tables clean and tools/accessories organized

If there is an error or problem with the instrument which is not addressed under the '[Troubleshooting](#)' section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. [Contact a staff member](#) for instructions

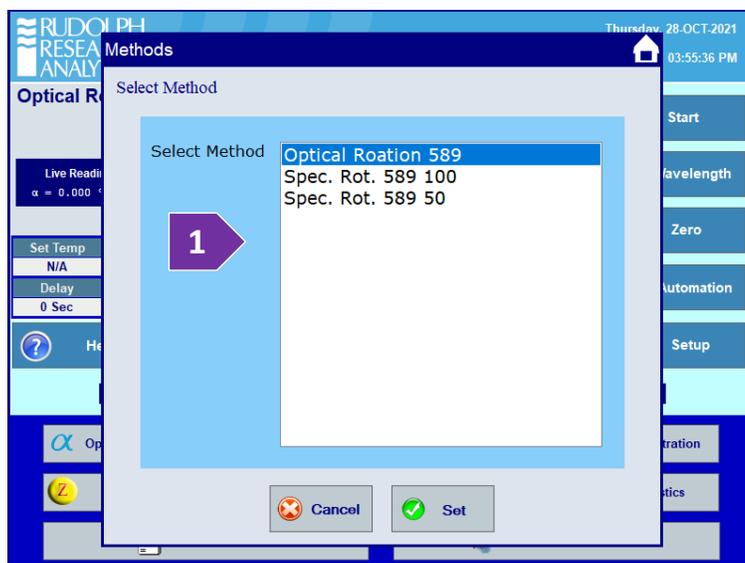
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## METHOD USAGE AND DEVELOPMENT

For collecting polarization data, a software method must be used. Depending on the existence of such method, please follow either step A or B as described below.

### A. USAGE OF A PREDEFINED METHOD

1. If a method that you intend to use has already been created, the “methods” button just needs to be selected and a window (figure 1) will appear
2. On the window, you will have the option to select the method desired, all information needed for the measurement will be prompted. The predefined method options are:



- ‘Optical Rotation 589’: This method measures total rotation independent of cell length at a wavelength of 589 nm
- ‘Specific Rotation 589-50’: This method measures a specific rotation at wavelength of 589nm for a 50mm cell
- ‘Specific Rotation 589-100’: This method measures a specific rotation at wavelength of 589nm for a 100mm cell

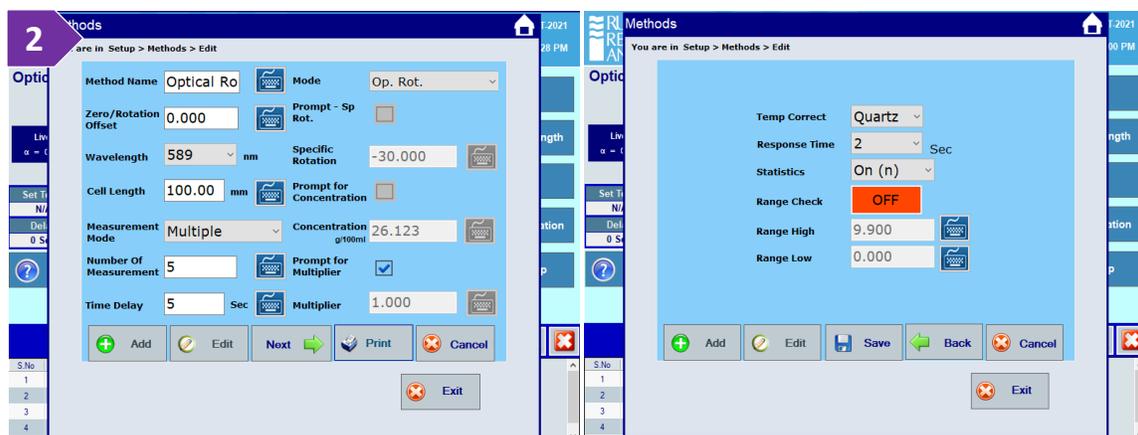
There are two cells available (50 mm and 100 mm long), and six wavelength selection, i.e., 365, 405, 436, 546, 589, and 633nm.

### B. GENERATING A METHOD

If the method you desire is not populated in the “methods” table, you can create the parameters you require by

1. Pressing on the ‘Setup’ button, then selecting the “methods” option, then finally selecting the “edit” option to edit the method of interest, or “add” to add a method to the list
2. If creating a new method, first the “add” field must be selected. Once it is, the following graphics (figure 2) will appear

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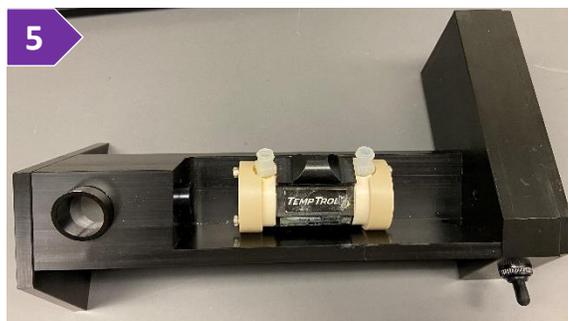


- Fill out the desired fields and save the method. Once the method is saved, it can be re-opened as saved method as explained in part A of this section
- Once a method is executed, all required parameters will need to be supplied
  - For Optical Rotation Measurements: The cell length will need to be supplied
  - For  $[\alpha]$  Specific Rotation: The cell length and concentration will need to be supplied. **The concentration is reported in a % (specifically grams per 100ml)**
  - For SR-Plus: This will allow an additional parameter to be added, in addition to the cell length and concentration, which is noted as a multiplier. The multiplier may be used to compensate for changes in a sample, such as loss from drying or filtration
  - For Concentration: The cell length and  $[\alpha]$  specific rotation(s) will need to be added

## C. MEASURING POLARIZATION

Once the proper method is selected, to measure the polarization of your sample:

- Zero the rotation by pressing on the 'Zero' button on the software
- Insert empty cell and verify that the rotation is still zero. This step ensures that the windows of the cell are not contaminated
- Fill the cell with the desired solvent and place the cell into the compartment of the instrument
- Zero the rotation
- Fill the cell with your sample, using the exact volume (1 or 2 ml), and use the bubble checker (figure 5) to ensure that there are no bubbles
- Place the cell into the compartment and record the polarization measurement by pressing on the 'Start' button. Instrument will keep taking measurements until the 'Stop' button is pressed



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7. At the end of all your measurements:
  - a. Flush the cell with solvent
  - b. Place the flushed cell into the compartment and verify that rotation is still zero ( $\pm 0.003$ )
  - c. Purge the cell with air using a syringe

## D. SAVING AND EXPORTING DATA

Once your data has been collected, the next step is to save your data. There are two options to save the data: as

a PDF or as a .txt file. They can be selected for export by pressing either the  button for the .txt file and the



button for the PDF.

## PUBLICATION

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### A. EXPERIMENTAL SECTION

*Modify the text below according to the setup and conditions you used during the measurement:*

“Optical rotation was measured at room temperature in **XXX solvent** using a Rudolph Autopol IV Polarimeter”

### B. ACKNOWLEDGEMENT

“This work made use of the IMSERC facility at Northwestern University, which has received support from the international Institute of Nanotechnology, the state of Illinois, and Northwestern University.”

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## TROUBLESHOOTING

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### A. THE SOFTWARE WILL NOT CONNECT TO THE INSTRUMENT

Begin your reservation in NUcore to turn on the instrument. We recommend starting your reservation 30 minutes before data collection.

### B. COMPUTER REQUIRES LOGIN AND A PASSWORD

The default 'Polarimeter' user account should be logged in. In case the computer was restarted, the password for the 'Polarimeter' account is \_\_\_\_\_ (see hardcopy next to the instrument). See '[Default instrument status](#)' section for more details.

### C. THERE IS AN ERROR OR PROBLEM WITH THE INSTRUMENT NOT ADDRESSED UNDER THIS TROUBLESHOOTING SECTION

If there is an error or problem with the instrument that is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
2. If you have not started your reservation using NUcore, please report problems with the instrument at <http://imserc.northwestern.edu/contact-issue.html> and place the 'Stop' sign near the instrument computer
3. [Contact a staff member](#) for instructions

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## REVISIONS

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v1.10 2022/04/21	<ul style="list-style-type: none"><li>• Added appropriate links and descriptions</li></ul>
v1.00 2021/11/15	<ul style="list-style-type: none"><li>• Release of original version of the user manual for Rudolph Autopol IV</li></ul>

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