

The brand of polarimeter used in more U.S. labs than any other.*



TECHNICAL BULLETIN 909

*2001/2002 Emmes Surveys

Polarimetry

Polarimetry is a sensitive nondestructive technique for measuring the optical activity exhibited by inorganic and organic compounds. A compound is considered to be optically active if linearly polarized light is rotated when passing through it. The amount of optical rotation is determined by the molecular structure and concentration of chiral molecules in the substance. Each optically active substance has its own specific rotation as defined in Biot's law:

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

 $[\alpha]$ = specific rotation, l = optical pathlength in dm; λ = wavelength, T = temperature, α = optical rotation, c = concentration in g/100ml.

The polarimetric method is a simple and accurate means for determination and investigation of structure in macro, semimicro and micro analysis of expensive and non-duplicable samples. Polarimetry is employed in quality control, process control and research in the pharmaceutical, chemical, essential oil, flavor and food industries. It is so well established that the United States Pharmacopoeia and the Food & Drug Administration include polarimetric specifications for numerous substances.

Research Applications for polarimetry are found in industry, research institutes and universities as a means of:

- ☐ Isolating and identifying unknowns crystallized from various solvents, or separated by high performance liquid chromatography.
- Evaluating and characterizing optically active compounds by measuring their specific rotation and comparing this value with the theoretical values found in literature.
- Investigating kinetic reactions measuring optical rotation as a function of
- ☐ Monitoring changes in concentration of an optically active component in a reaction mixture, as in enzymatic cleavage.
- ☐ Distinguishing between optical isomers.
- ☐ Analyzing molecular structure by plotting optical rotatory dispersion curves over a wide range of wavelengths.



PHARMACEUTICALS



ESSENTIAL OILS



SUGAR & CONFECTIONERY PRODUCTS



ORGANIC CHEMISTRY

In each of these applications, the AUTOPOL offers up to six discrete wavelength selections to observe the effect of wavelength upon an optically active substance.

Quality and Process Control Applications,

both in the laboratory or on-line in the factory, are found throughout the pharmaceutical, essential oil, flavor, food and chemical industries. A few examples are listed below.

Pharmaceutical Industry

Determines product purity by measuring specific rotation and optical rotation of:

- Amino acids
- Amino sugars
- Analgesics
- Antibiotics
- Cocaine
- Codeine
- Dextrose
- Diuretics
- Serums
- Steroids
- Tranquilizers
- Vitamins

Flavor, Fragrance and **Essential Oil Industry**

Utilizes polarimetry for incoming raw materials inspection of:

- Camphors
- Citric acid
- Glyceric acid
- Gums
- Lavender oil
- Lemon oil
- Orange oil
- Spearmint oil

Food Industry

Ensures product quality by measuring the concentration and purity of the following compounds in sugar based foods, cereals and syrups:

- Carbohydrates
- Fructose
- Glucose
- Lactose
- Levulose
- Maltose
- Natural monosaccharides
- Raffinose
- Sucrose
- Various starches
 Xylose

Chemical Industry

Analyzes optical rotation as a means of identifying and characterizing:

- Biopolymers
- Natural polymers
- Synthetic polymers

Features of AUTOPOL® IV

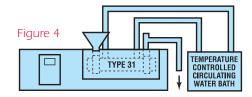
Measurement Modes

The Autopol IV can read directly in one of four measurement modes: Optical Rotation. Specific Rotation. Concentration or Specific Rotation Plus. Specific Rotation Plus allows a correction to be applied to a result for loss on drying. Each measurement mode can be activated via touchscreen selection. Sample cells can be specified at any length (e.g., 50mm, 100mm, 200mm), or entered as a precise dimension 199.98mm). (e.g., Concentration is entered as a percentage. (See Figure 3)



Figure 3

Temperature Control of Samples is allowed through rubber gasketing in the Autopol door which permits tubing to be connected from an external temperature controlled Water Bath to the jacket of a jacketed polarimeter sample tube. (See Figure 4)



Cells up to 200mm Long

Instruments of some manufacturers accept only special sample cells, with maximum lengths of 100mm. Autopols accept standard sample cells up to 200mm long. A 200mm sample cell offers twice the sensitivity as the same solution in a 100mm cell. This is especially useful for solutions having small rotations. Also some pharmacopeia monographs require a 200mm (2dm) cell like the USP monograph for Methotrexate. Rudolph Research sample cells are made to NIST standards; and the complete range of sizes and types are listed in Technical Bulletin 913. NIST certificates are available for cell length validation.



Figure 5

Multiple Wavelengths

The Autopol IV comes in single, dual and six wavelength models. Wavelength selection is completely automatic and is accomplished via menu selection. There are no lamps or filters to manually remove or insert. The following wavelengths are available: 365nm, 405nm, 436nm, 546nm, 589nm, and 633nm. Optional wavelengths include: 325nm, 880nm, 578nm and various other wavelengths are available; contact the factory for more information.

White-light source permits spectral versatility

The Tungsten-Halogen lamp employed in the Autopol is a compact, reliable, low-cost, high-intensity light source that allows any desired wavelength in the visible spectrum to be selected by means of a narrow band multilayer interference filter. (See Figure 6) The Autopol standard 10nm bandwidth is specified to permit high energy throughput and sensitivity for sample transmittances of only 0.01% (O.D. = 4), while minimizing the effects of sample ORD and color.



Figure 6

Temperature Display

The Autopol IV comes standard with a temperature probe which can be inserted into the Polarimeter cell as shown below. The temperature of either the sample compartment or the solution itself can be continuously displayed. (See Figure 7)





Just listen to our customers

"We have the AUTOPOL V from Rudolph Research Analytical (RRA)... I have used numerous polarimeters and this is by far the best. We have had it for 1-1/2 years and have had zero problems, the original lamp is still in the instrument. I recommend it over other instruments. RRA installed, trained and performed the IQOQ... It is one piece of equipment that you never worry about."

Alan Davis - Rockwell Medical Technologies

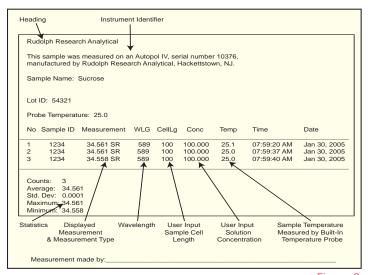


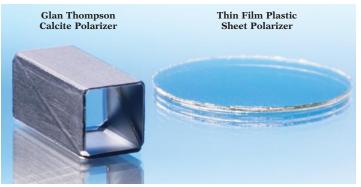
Figure 8

Quality

No matter which model Autopol you choose, Rudolph uses the same high quality optics. While other manufacturers use Polaroid Plastic Dichroic Sheet Polarizers, Rudolph does not. Instead, Rudolph uses the same high quality Glan Thompson Calcite Polarizers in all its models.

Why are high quality polarizing prisms important? Because prisms are two of the most critical optical components in the polarimeter. Polaroid polarizers are made of a polymeric plastic where the molecules are stretched and oriented in a specific direction so as to linearly polarize light. These types of plastic sheet polarizers are very inexpensive (\$50.00 USD) and are vulnerable to heat, warp over time, deteriorate from moisture, and also have greater light absorption than Calcite Polarizers. In many cases, the plastic polarizers must be replaced in 3 – 5 years. Glan Thompson Calcite Polarizers are comprised of a carbon crystalline structure similar to diamond and have excellent light transmission characteristics. The quality of these prisms is so good, Rudolph guarantees its prisms over the life of the polarimeter (See Figure 9)

Figure 9



Print Format

Numerous parameters, such as sample name, tube length, concentration, wavelength, time and date, temperature correction and statistics, can be printed after each measurement or after a group of measurements. This is accomplished by designating the required parameters, via menu entry, during the print format setup mode. A sample printout is shown to the left. (See Figure 8)

Calibration and Validation

Rudolph understands that today's cGLP/cGMP laboratories must validate measurement and temperature. Therefore Rudolph offers 6 different NIST Traceable Quartz Calibration Standards that can be matched to your application. The Autopol IV also comes with a Temperature Validation Cell so that your temperature measurement can be traced to NIST. See Technical Bulletin 913 for more information.



Rudolph PC Software

This software offers a simple, easy to use interface for collecting data from Rudolph Polarimeters. Rudolph PC is available in several different versions including PC Basic, PC Purity, PC Sample Handling, and PC 21CFR11; there is a version



of Rudolph PC software available for all of our polarimeters. All data can be exported to Excel with the click of a button.

Choosing a Polarimeter

That's right for your application and budget

Autopol I



The accuracy of our entry-level model is +/- 0.02 Degrees Optical Rotation. This accuracy is fine for food and educational applications where budget is critical but state of the art quality is still important. The Autopol I is not a pharmaceutical grade polarimeter and does not come with IQOQ (Installation Quality Operation Quality) documentation.

Please see Technical Bulletin 920 for more details on the Autopol I.

Autopol II



A general-purpose polarimeter with +/- 0.01 Degrees Optical Rotation accuracy. This accuracy is applicable for food, university education, many flavor and chemical. fragrance applications, as well as pharmaceutical inspection in some countries. We strongly recommend against using this instrument for USP, EP, JP and BP pharmaceutical applications. Although IOOO documentation is provided, Rudolph will not do an on-site IQOQ for pharmaceutical applications where a certificate of analysis will be issued. Please see the application note in yellow for further clarification.

More details on the Autopol II can be found in the Technical Bulletin 918.

Below is an example of why Polarimeters with accuracy of ± 0.02 and ± 0.01 accuracy polarimeters are not suitable for pharmaceutical applications. Please review the USP monograph for the material Ofloxacin. Ofloxacin must have a specific rotation between +1° and -1° at a concentration of 10mg per ml. Biots Law:

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

 $[\alpha]$ = specific rotation, l = optical pathlength in dm; λ = wavelength, T = temperature, α = optical rotation, c = concentration in g/100ml

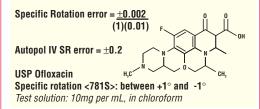
The Autopol I has an accuracy of $\pm 0.02^\circ$ optical rotation. Below is how its relative accuracy affects a hypothetical Ofloxacin sample:

$$\begin{array}{c} \left[\alpha\right]_{\lambda}^{T} \text{(Specific Rotation)} = \pm 0.02 & \text{(Autopol I)} \\ \hline (1)(0.01) \leftarrow 10 mg/ml \\ \text{(Optical pathlength in dm)} \uparrow & .01g/ml \\ \hline & 1g/100ml = 1\% = 0.01 \end{array}$$

Specific Rotation error = ± 2.0

You cannot successfully inspect a pharmaceutical (such as Ofloxacin) having a Specific Rotation between+1 and -1 and a 1% concentration, which is quite common for most pharmaceuticals, with an instrument that has an accuracy of ± 2.0 in Specific Rotation.

The Autopol IV has an accuracy of 0.002 for Optical Rotations of 1°Arc for a total unknown of +/-0.002 under the same conditions:



Autopol III



Rudolph's entry-level pharmaceutical and research grade polarimeter has +/- 0.002 Degrees Optical Rotation accuracy for rotations ≤1° Arc. This instrument comes standard with two wavelengths: 589 nm and 546 nm and will pass an FDA inspection with the appropriate Water Bath to control temperature. Please see Technical Bulletin 919 for more details on the Autopol III. IQOQ documentation is included with the instrument.

Autopol IV



This model is available in one, two and six wavelength versions and has a more expensive optical system than the Autopol III. It is the system of choice for research universities, pharmaceutical and fine chemical research departments. This instrument is also excellent for studying recimics and kinetics. Temperature control is accomplished through a circulating Water Bath as shown in Figure 4. IQOQ documentation is included with the instrument.

Autopol V



This is Rudolph's top of the line six wavelength polarimeter specifically designed with the input of the world's largest pharmaceutical companies. This unit comes standard with 21CFR11 software and Rudolph's Patented **TempTrol**TM System, which electronically heats and cools the sample to a specified temperature without the use of water circulation. This unit includes all wavelengths necessary to measure 99% of all monographs found in the USP, EP, JP and BP. IOOO documentation is included with the instrument. Please see Technical Bulletin 914 for more details on the Autopol V.

Autopol 880/Autopol 589

For Polarimeters specifically designed for the sugar industry see Technical Bulletin 910.

Markets:	Education	Food	Pharmaceutical QC	Research	Big Pharma
Specifications:	Autopol I	Autopol II	Autopol III	Autopol IV	Autopol V
Measuring Mode:	Optical Rotation, Specific Rotation, Concentration, Sugar Degrees, °Z (ISS)	Optical Rotation, Specific Rotation, Concentration, Sugar Degrees, °Z (ISS)	Optical Rotation, Specific Rotation, Concentration, Sugar Degrees, °Z (ISS)	Optical Rotation Specific Rotation, Specific Rotation Plus Concentration User Defined Scale	Optical Rotation Specific Rotation, Specific Rotation Plus Concentration User Defined Scale
Measuring Scale:	Degrees Arc Optical Rotation	Degrees Arc Optical Rotation	Degrees Arc Optical Rotation	Degrees Arc Optical Rotation	Degrees Arc Optical Rotation
Resolution:	0.01% Concentration 0.01 Specific Rotation	*	0.001% Concentration 0.001 Specific Rotation	0.001% Concentration 0.001 Specific Rotation	0.001° Arc Optical Rotation 0.001% Concentration 0.001 Specific Rotation
Accuracy:	0.05°Z (ISS) Sugar Degrees	0.01° Arc Optical Rotation 0.03°Z (ISS) Sugar Degrees	0.2% above ΰ	0.002° up to 1° 0.2% up to 5° 0.01° above 5°	0.002° up to 1° 0.2% up to 5° 0.01° above 5°
Reproducibility:		0.01° Arc Optical Rotation	_		
Measuring Range:	± 89° Arc Optical Rotation, ± 999.99° Arc Specific Rotation, 0-99.9% Concentration	± 89° Arc Optical Rotation, ± 999.99° Arc Specific Rotation, 0-99.9% Concentration	± 89° Arc Optical Rotation, ± 999.99° Arc Specific Rotation, 0-99.9% Concentration	± 89° Arc Optical Rotation, ± 999.99° Arc Specific Rotation, 0-99.9% Concentration	±89° Arc Optical Rotation, ±999.99° Arc Specific Rotation, 0-99.9% Concentration
Prism:	Glan Thompson Calcite	Glan Thompson Calcite	Glan Thompson Calcite	Glan Thompson Calcite	Glan Thompson Calcite
Optical Wavelengths:	589nm	589nm, 546nm	589nm, 546nm	365nm, 405nm, 436nm, 546nm, 589nm, 633nm (other wavelengths available: 325nm)	365nm, 405nm, 436nm, 546nm, 589nm, 633nm (other wavelengths available)
Wavelength Selection:	Fixed	Selectable/2 only	Selectable/2 only	Selectable/1, 2 and 6	Selectable/6 only
21 CFR Part 11 Compliant Software	No	No	No	No	Yes
TempTrol TM Range:	Temperature control by external Water Bath	Automatic Electronic Heating & Cooling 15°-35°C			
TempTrol™ Accuracy:	Temperature accuracy determined by Water Bath	±0.2°C			
Temp. Probe Range:	10° - 40°C	10° - 40°C	10° - 40°C	10° - 40°C	10° - 40°C
Temp. Probe Accuracy:	±0.1°C	±0.1°C	±0.1°C	±0.1°C	±0.1°C
Measurement Time:	5 measurements in less than 25 seconds (avg.)	5 measurements in less than 25 seconds (avg.)	5 measurements in less than 25 seconds (avg.)	4°/sec. slewing rate & 5 sec. nominal settling time	4°/sec. slewing rate & 5 sec. nominal settling time
Light Source:		Tungsten-Halogen 6V, 20W, avg. 2,000 hour life			Tungsten-Halogen 6V, 20W, avg. 2,000 hour life
Sample Chamber:	Accepts sample tubes up to 200 mm	Accepts sample tubes up to 200 mm			
Data Storage:	No	No	No	No	Secure Digital Card
Communication Interface:	Two RS232 serial ports, one parallel printer port	Two RS232 serial ports, one parallel printer port	Two RS232 serial ports, one parallel printer port	Two RS 232 serial ports, one parallel printer port and one auxiliary port	Two RS232 serial ports, one parallel printer port and one auxiliary port
Analog Output:	Not available	4-20mA full scale (optional)	0.1% resolution 0-10 volts or 4-20mA full scale (optional)	4-20mA full scale (optional)	0.1% resolution 0-10 volts or 4-20mA full scale (optional)
Calibration:	Automatic calibration via touch screen	Automatic calibration via touch screen	Automatic calibration via touchscreen	Automatic calibration via touchscreen	Automatic calibration via touchscreen
Display:		7.5 cm x 10 cm Graphics LCD, 320 x 240 dots cold fluorescent back lit		7.5 cm x 10 cm graphics LCD, 320 x 240 dots cold fluorescent back lit	
User Interface:	Touchscreen	Touchscreen	Touchscreen	Touchscreen	Touchscreen
Automatic Sensitivity Control:	Measures samples with transmittance as low as .1% (up to O.D. 0.03)		Measures samples with transmittance as low as 0.01% (up to O.D. 4.0)	Measures samples with transmittance as low as 0.01% (up to O.D. 4.0)	Measures samples with transmittance as low as 0.01% (up to O.D. 4.0)
Input Power:	100 - 240V, 50/60 Hz	100 - 240V, 50/60 Hz	100 - 240 V, 50/60 Hz	100 - 240V, 50/60 Hz	100 - 240V, 50/60 Hz
Operating Dimensions:		24.3" W x 12.7" H x 17.5" D 617 mm W x 323 mm H x 445 mm D	24.3" W x 12.7" H x 17.5" D 617 mm W x 323 mm H x 445 mm D	30" W x 9" H x 14" D 769 mm W x 231 mm H x 359 mm D	35" W x 10.5" H x 17" D 890 mm W x 267 mm H x 432 mm D
Shipping Dimensions:	30" W x 25" H x 23" D 762 mm W x 635 mm H x 584.2 mm D	30" W x 25" H x 23" D 762 mm W x 635 mm H x 584.2 mm D	30" W x 25" H x 23" D 762 mm W x 635 mm H x 584.2 mm D	42" W x 24" H x 21" D 1066.8 mm W x 609.6 mm H x 533.4 mm D	42" W x 24" H x 21" D 1066.8 mm W x 609.6 mm H x 533.4 mm D
Operating Weight:	42 lbs. (19.05 kg)	42 lbs. (19.05 kg)	42 lbs. (19.05 kg)	70 lbs. (32 kg)	85 lbs. (39kg)
Shipping Weight:	72 lbs. (32.6 kg)	72 lbs. (32.6 kg)	72 lbs. (32.6 kg)	115 lbs. (52.1 kg)	115 lbs. (52.1 kg)