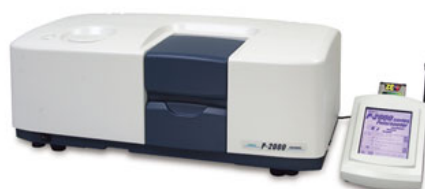


Optical Rotation Measurement for Small Sample Amounts

By Leah Pandiscia, PhD

Introduction

Optical rotation measurement is a common analytical method used for the evaluation of synthesized compounds and compounds isolated and purified from natural products. In most of cases, the amount of material obtained during synthesis and those obtained from natural product purification is usually very small. Therefore, it is essential to be able to measure these samples using the smallest amounts which requires the use of small volume cylindrical cells. This application note demonstrates the measurement of optical rotation using a sample volume of only 1 mL.



P-2000 for Optical Rotation Measurement

Table 1. Cell sizes and recommended (minimum) sample volumes

Cell Diameter	Cell path length: 100 mm	Cell path length: 50 mm
10 mm	9 mL	5 mL
3.5 mm	1.6 mL	1.4 mL (1mL)
2.5 mm	>1.4 mL (1mL)	-
The values in () are the minimum sample volume required for the measurement.		



Fig.1 Cylindrical Cell 2.5 mm D x 100 mm L

Features

- P-2000 Polarimeter with sodium lamp and 589nm filter
- PTC-262 Peltier cell holder for temperature controlled measurement
- Minimum 1 mL sample volume (when using cells of 3.5 mm D x 50 mm L and 2.5 mm D x 100 mm L)

Experimental

Sample preparation

10 mg of pirarubicin was accurately weighed and added to a 10 mL measuring flask, dissolved with chloroform and then diluted to 10 mL total volume (at 20°C).

Measurement conditions

Instrument:	P-2000 Polarimeter
	PTC-262 Peltier thermostatted cell holder
Cells:	Glass cells (size: 2.5 mm D x 100 mm L, 3.5 mm D x 100 mm L)
Light source/Wavelength:	Sodium lamp / D589 line
Measurement Temperature:	20°C

Note:

- Temperature should be recorded prior to measurement as the probe may interfere with the light path.
- Make sure that there are no bubbles in the cell, especially near the windows. Bubbles may cause inaccurate measurement.
- If there is sufficient available sample available, it is recommended that a cell of 10 mm D x 100 L mm is used to obtain more simple and stable measurement results.

Keywords

pirarubicin, polarimeter, P-2000, optical rotation

Result

Measurement Results

	2.5 mm D x 100 mm L glass cell	3.5mm L x 100 mm L glass cell	Description in JP ([α] _D ²⁰ : +195 ~ +215° (10 mg, 10 mL, 100 mm))
Optical rotation	+0.2066°	+0.2021°	-
	+206.6°	+202.1°	+195 ~ +215°

	2.5 mm D x 100 mm L glass cell	3.5mm L x 100 mm L glass cell	Description in JP ([α] _D ²⁰ : +195 ~ +215° (10 mg, 10 mL, 100 mm))
Specific optical rotation (α)			

Conclusion

Optical rotation measurement of pirarubicin

Among the pharmaceutical compounds described in the various pharmacopoeias such as EP, USP and BP, there are requirements for optical rotation measurement of small sample volumes of valuable material. One such example is the measurement of optical rotation of pirarubicin ([α]_D²⁰: +195 ~ +215° (10 mg, chloroform, 10 mL, 100 mm)) indicated in the JP 16th Edition was measured with cells of 2.5 mm D x 100 mm L cell and 3.5 mm D x 100 mm L. The result shows that even the smallest amounts of sample can be easily measured.

About the Author

Leah Pandiscia received her PhD from Drexel University where she studied Biophysical Chemistry. She is a Spectroscopy Applications Scientist at JASCO.