

Optical Rotation Measurement for small amount of sample

Introduction

The optical rotation measurement is a common analytical method used for the evaluation of synthesized compounds and new compounds isolated and purified from natural products, while in most of the cases, the amount of compounds obtained from the synthesis for which many steps are required or target compounds obtained from valuable natural products is usually very small. Therefore, it is essential to enable the measurement of such sample with the amount as small as possible and a series of cells have been lined up. This report proves the optical rotation of the minimum 1 mL sample can be measured.

Key words: Small amount sample measurement, polarimeter, cell, pirarubicin

Table 1 Cell sizes and recommendable (minimum) sample volume

	Light pathlength: 100 mm	Light pathlength: 50 mm
ϕ 10 mm	9 mL	5 mL
ϕ 3.5 mm	1.6 mL	1.4mL (1mL)
ϕ 2.5 mm	1.4 mL (1mL)	- -

The values in () are the minimum sample volume required for the measurement



Fig. 1 ϕ 2.5mm x 100mm cell

Features

- Model PTC-262 Peltier cell holder enables temperature control for the measurement
- Minimum 1 mL sample measurement (when using cells of ϕ 3.5 mm x 50 mm and ϕ 2.5 mm x 100 mm)

(Note)

- A temperature sensor probe inside a cell cannot be used when the sample volume is minimum.
- It is necessary to make sure that there is no bubble in the light path in the cell, especially near the window. Any bubble should be removed from the light path because bubbles may cause an inaccurate measurement.
- If the amount of the sample available is sufficient, it is recommended to use a cell of ϕ 10 mm x 100 mm in order to obtain the most simple and stable measurement results.

Optical rotation measurement of pirarubicin

Among the pharmaceuticals described in pharmacopoeias such as EP, USP and BP, there are some for which the sample volume specified for the optical rotation measurement is very small because they are very precious. As one of the examples, the optical rotation of pirarubicin ($[\alpha]_D^{20}$: +195 ~ +215° (10 mg, chloroform, 10 mL, 100 mm)) indicated in the JP 16th Edition was measured with cells of ϕ 2.5 mm x 100 mm cell and ϕ 3.5 mm x 100 mm. The result shows that even a tiny amount of sample can be measured without any problem.

Sample preparation

10 mg of pirarubicin was accurately weighted and added into a 10 mL measuring flask, dissolved with chloroform and then diluted to 10 mL in total at 20°C.

Measurement conditions

Instruments: P-2000 Polarimeter
 PTC-262 Peltier thermostatted cell holder
 Cells: Glass cells (size: ϕ 2.5 mm x 100 mm, ϕ 3.5 mm x 100 mm)
 Light source/Wavelength: Deuterium lamp / D line
 Measurement temperature: 20°C

Measurement results

	ϕ 2.5mm x 100mm glass cell	ϕ 3.5mm x 100mm glass cell	Description in JP ($[\alpha]_D^{20}$ (10 mg, 10 mL, 100 mm))
Optical rotation	+0.2066°	+0.2021°	-
Specific optical rotation	+206.6°	+202.1°	+195 ~ +215°