Application Note Booklet

Polarimeter

2711P001

Optical rotation of Dextromethorphan Hydrobromide measured using 365 nm emission line of mercury lamp

Introduction

Optical rotation of Dextromethorphan Hydrobromide in accordance with the U.S. Pharmacopeia, is requested to be measured at 325 nm, however there is no emission line of 325 nm available in the polarimeter using Na lamp or Hg lamp as light source. Therefore, for the measurement of optical rotation of Dextromethorphan Hydrobromide, a polarimeter with halogen lamp as a light source and interference filter(325 nm) has been usually used, but as shown in Fig. 1, the optical rotatory dispersion (ORD) spectrum, there is a very steep slope in the short wavelength range near 325 nm, where even a very small error in the wavelength of interference filter may cause a very large error in the optical rotation measurement, which will makes the accurate measurements very difficult. On the other hand, if the measurement by using 365 nm is allowed, since no wavelength error is expected, the measurement of optical rotation can be implemented with much higher accuracy. In this experiment, Dextromethorphan Hydrobromide was measured using polarimeter with Hg lamp (365 nm), polarimeter with Halogen lamp and interference filter (325 nm) and polarimeter with ORD attachment.

Keywords: Dextromethorphan Hydrobromide, Mercury lamp, Optical rotation, ORD spectrum

Results

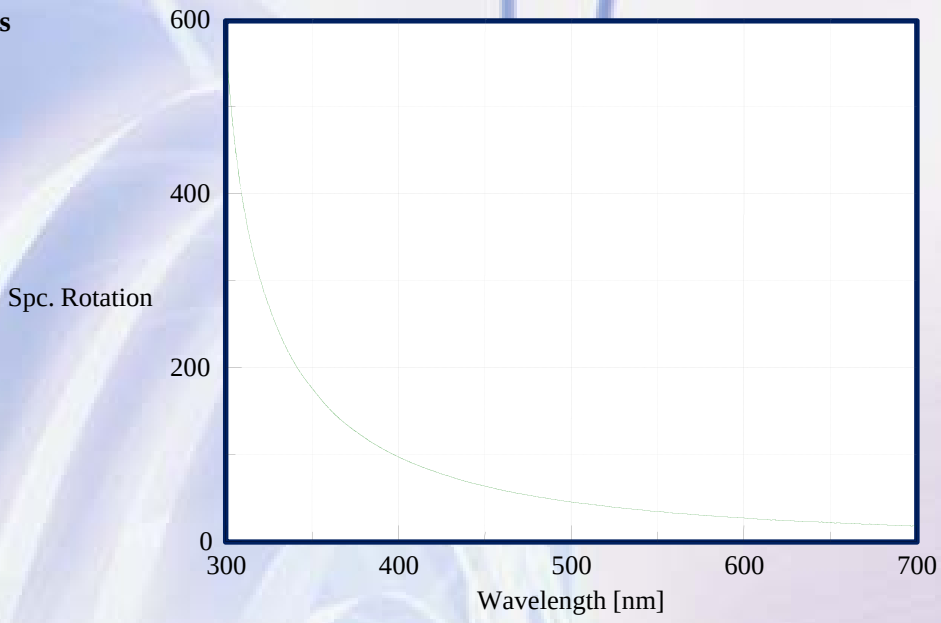

Fig. 1 ORD spectrum of Dextromethorphan Hydrobromide

<Polarimeter>

Optical rotation (365 nm, 20ºC): + 2.5655º (by JASCO P-2000 with Hg lamp)
Specific rotation $[a]_{365}^{20}$: + 142.53
Optical rotation (325 nm, 20ºC): + 4.7034º (by JASCO P-2000 with halogen lamp & interference filter)
Specific rotation $[a]_{325}^{20}$: + 261.30

<ORD attachment>

Optical rotation (365 nm, 20ºC): + 2.5706º (by JASCO P-2000 with ORD attachment)
Specific rotation $[a]_{365}^{20}$: + 142.81
Optical rotation (325 nm, 20ºC): + 4.8726º (by JASCO P-2000 with ORD attachment)
Specific rotation $[a]_{325}^{20}$: + 270.70

Optical rotation measurement of sucrose and l-menthol

Introduction

Optical rotation is the property of substances, rotating the plane of polarization when linearly-polarized light passes through such substances. This is the property which occurs specifically to optical active substances in which the refractive indices of right and left circularly-polarized light are different. Optical rotation that rotate light in a clockwise direction as viewed towards the light source is defined as *dextrorotation* (+) and the opposite, *levorotation* (-).

The angle of rotated plane of polarization is called as optical rotation and polarimeter is the instrument to measure such optical rotation. Optical rotation is proportional to cell pathlength and is related to sample concentration, measurement wavelength and temperature. The specific optical rotation $[a]_x^t$ is calculated from the following formula using temperature t (ºC), wavelength x (nm), cell pathlength l (dm), sample concentration c (g/100 mL) and measured optical rotation a. l

$$[a]^t_x = 100\ a/cl$$

JP, USP and EP suggest to measure optical rotation using D-line of Na lamp.

Polarimeter is used for several purposes such as purity certification of sugar, verification of pharmaceuticals and optical purity determination of optical active substances obtained from asymmetric synthesis in organic chemistry field.

Keywords: Optical rotation measurement, Sucrose, Menthol, ORD

Optical rotation measurement of sucrose

5 g/100mL of sucrose (Wako Pure Chemical Industries, Ltd., JIS special grade) was prepared and measured using P-2000 with 100mm cell, Na lamp D-line under 20ºC.

Fig. 1 shows the printout view of measurement result. The average of optical rotation in 5 times measurement was + 3.3264 deg. and its specific optical rotation calculated was + 66.5280 which is in good agreement with the specific optical rotation of 5 g/100mL sucrose solution described in JIS K0063 1), + 66.500.

5%Sucrose_20°C.p1d

[Data Information]
Creation Date 2012/06/11 8:56

[Measurement Information]
Instrument Name P-2000
Model Name P-2000
Serial No. A071661232

Light Source Na
Monitor wavelength 589 nm
D.I.T. 5 sec
No. of cycle 5
Cycle interval 0 sec
Temp. Monitor Holder
Temp. Corr. Factor None
Aperture(S) 8.0nm
Aperture(L) 2.0nm
Mode Optical Rotation
Factor 1

[Comment]
Sample name 5% Sucrose
Comment
User
Division
Company JASCO

		No.	Sample No.	Mode	Calc. Data	Meas. Data	PMT Voltage[V]	Temperature(C)
1	*	1	1-1	Specific O.R.	+66.5348	+3.3267	243	19.99
2	*	2	1-2	Specific O.R.	+66.5388	+3.3269	243	19.99
3	*	3	1-3	Specific O.R.	+66.5368	+3.3268	243	19.99
4	*	4	1-4	Specific O.R.	+66.5428	+3.3271	243	19.99
5	*	5	1-5	Specific O.R.	+66.5388	+3.3269	243	19.99
6	*	6	Avg.		+66.5384			
7		7	S.D		0.0030			
8		8	C.V		0.0045			

Fig. 1 Printout view of optical rotation measurement of 5 g/100mL sucrose solution

Optical rotation measurement of 1-menthol

l-menthol (Wako Pure Chemical Industries, Ltd.) was prepared under the condition suggested in JP (2.5 g ethanol (95%), 25 mL, 100 mm) and its optical rotation was measured. Its specific optical rotation was calculated to be $[a]^{20}_{D}$ = - 50.01 which is well within the JP criteria: - 45.0 ~ - 51.0.

Generally, optical rotation is increased in shorter wavelength region. For optical rotation measurement, Na lamp D-line is usually applied, while when the optical rotation of the sample is very small using D-line, by irradiating shorter wavelength light, optical rotation can be measured in easier way. In this application data, optical rotation measured using P-2000 with both Na lamp D-line and Hg lamp emission line wavelength (546, 436, 405, 365 nm) and ORD spectrum (showing wavelength dispersion of optical rotation) measured using J-1500 + ORDM-520 were compared. As shown in Fig. 3, both measured values by P-2000 and ORDM-401 are very consistent. Like this, by employing shorter wavelength light for samples with small optical rotation, the measurement can be implemented easily.

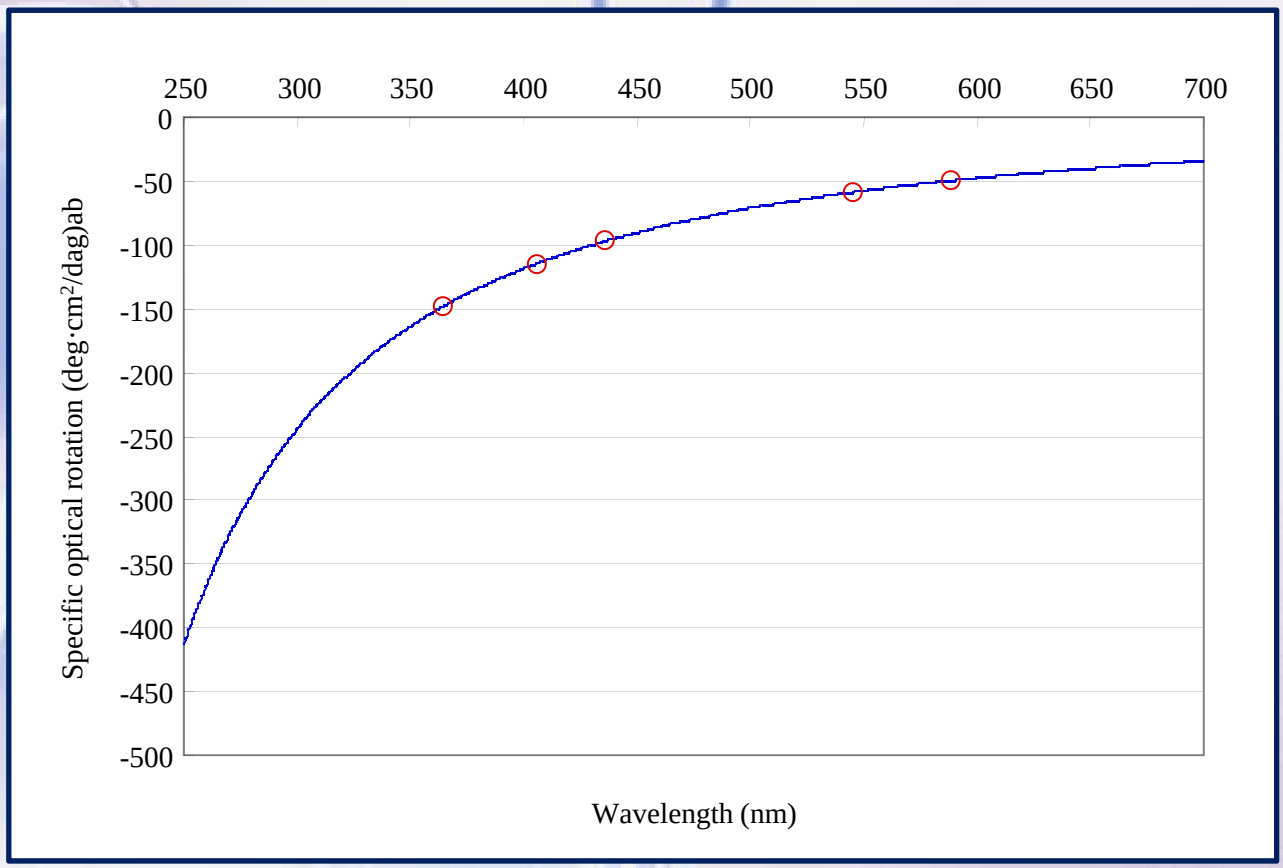

Fig. 2 Optical rotation of 1-menthol and comparison with ORD spectrum
○: P-2000 measured value —— : ORD spectrum

Reference

(1) Optical rotation of sucrose solution described in JIS K0063 is referred to Handbook of Chemistry and Physics, 42nd Edition (1960-1961), P1784, The Chemical Rubber Co., Ohio, U.S.A..

Measurement of optical rotation of pirarubicin using sodium lamp and halogen lamp

Introduction

An emission line of light source such as a sodium lamp or a mercury lamp is usually used to measure the optical rotation of pharmaceutical products. However, the Japanese Pharmacopeia and the European Pharmacopeia only mention the use of a sodium lamp as the light source.

A polarimeter using a halogen lamp and a band pass filter (BPF) can also be used, as mentioned in the United States Pharmacopeia. However, the measurement error from the transmission property, that is the difference between the center wavelength of the BPF and the wavelength of emission line, cannot be avoided.

Herein, the optical rotation of pirarubicin was measured using a sodium lamp and a halogen lamp, and the measurement error between the results was evaluated.

Keywords: Pirarubicin, Sodium lamp, Halogen lamp, Optical rotation, ORD spectrum

<ORD measurement>

Sample preparation: 10.00 mg of pirarubicin was dissolved in chloroform for final volume of 10 mL.

System

J-1500 CD Spectrometer
ORDM-520 ORD attachment

Parameters

Cell pathlength:	20 mm	Temp.:	20ºC	Bandwidth:	1 nm
Data interval:	0.2 nm	Response:	1 sec	Scan speed:	100 nm/min
Wavelength:	700-560 nm	Accumulation:	1		

Results

At first, absorption and ORD spectra of pirarubicin were measured in the region of 700-560 nm to confirm the wavelength dependence of the optical rotation (Fig. 1). The absorbance increased sharply below 600 nm and optical rotation increased gradually as a result of the Cotton effect.

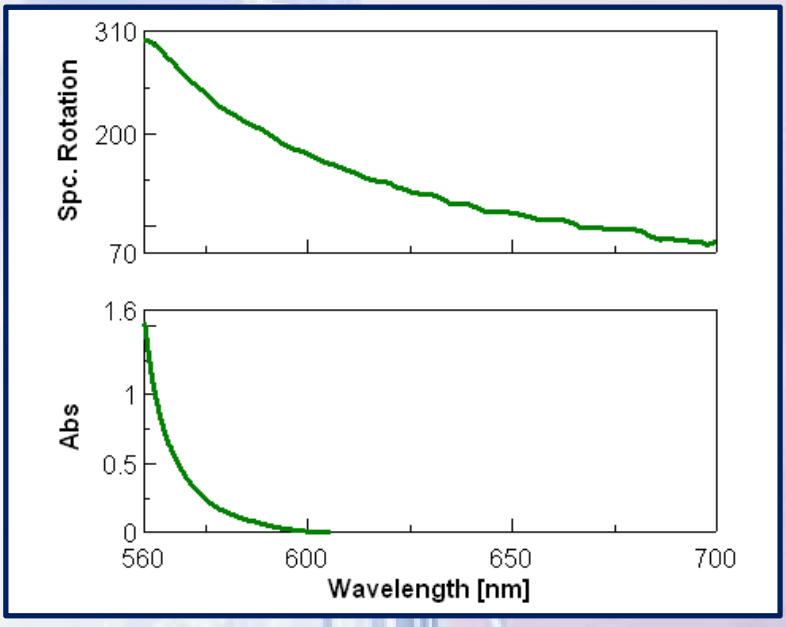

Fig. 1 Absorption and ORD spectra of pirarubicin

The transmission spectrum of a BPF and the spectrum of the D-line of the sodium lamp, overlapped with ORD spectrum are shown in Fig. 2. It is apparent to see the significant change of the specific rotation in the wavelength region within the BPF.

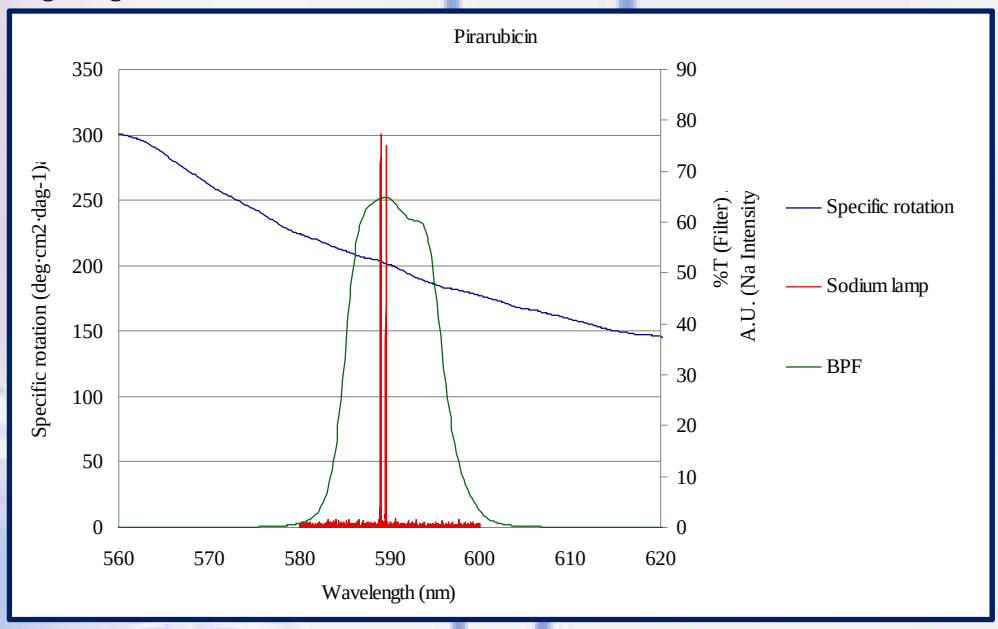

Fig. 2 ORD spectrum of pirarubicin, sodium lamp emission line and the transmission spectrum of the BPF

<Optical rotation>

Sample preparation: Identical sample for ORD measurement.

System

P-2000 Polarimeter
PTC-262 Peltier thermostated cell holder

Parameters

Measurement temp.:	20ºC
Cell pathlength:	100 mm
Wavelength:	589 nm (D-line of sodium and halogen lamp)

Results

Specific rotation of the same sample for the ORD measurement was measured using the polarimeter. When a sodium lamp is used as the light source, the result was $[a]_D^{20}$ = + 206.6 [deg·cm^2·dag^{-1}]. This value falls within the range stated in the Japanese Pharmacopeia. ($[a]_D^{20}$ = + 195 ~ + 215 [deg·cm^2·dag^{-1}]) On the other hand, when using a halogen lamp, the specific rotation was just inside this range ($[a]_{589}^{20}$ = + 196.7 [deg·cm^2·dag^{-1}]). These two results clarify the difference in the optical rotations measured at the same wavelength (589 nm) with different lamps.

Table 1 Results of the optical and specific rotation measurements

Light source	Optical rotation [deg]	Specific rotation [deg·cm^2·dag^{-1}]
Sodium lamp	+ 0.2066	$[a]_D^{20}$: + 206.6
Halogen lamp	+ 0.1967	$[a]_{589}^{20}$: + 196.7