Application Note

Simultaneous Multi-Probe (SMP) Measurement of CD and LD using Diffuse Reflectance (DRCD) for Alanine powder samples

Introduction

Generally, a sample for CD measurement needs to be in a liquid state to allow transmission measurement, more recently there has been increasing requirement for CD measurement of samples that are insoluble or may change structure in solution. These types of sample are best measured in solid state. Although CD measurement can be made in transmission for solid samples, there is a requirement for sample preparation; such as making a pellet and if sample dilution is required, sample recovery can be difficult. As an alternative, measurement using Diffuse Reflection (DR) CD is recommended. 1), 2) This application note illustrates the use of DRCD measurement with the DRCD-575 diffuse reflectance accessory (See Fig. 1.) optimized for the J-1500 CD spectrometer with the multi-probe simultaneous CD/LD measurement.

DRCD uses incident light to illuminate a solid sample and an integrating sphere to collect the diffusely reflected light which is focused onto a detector (See Fig. 2.) However, CD measurement of solid samples can be influenced by LD (linear dichroism) derived from optical anisotropy, this needs to be evaluated and removed in order to minimize artifacts in the CD measurement.

The J-1500 CD spectrometer allows the estimation of artifacts in the CD signal by using a quad digital lock-in amplifier which offers the Simultaneous Multi-Probe function (SMP) measurement of both CD and LD.

In this example of DRCD measurement, samples of L- and D-alanine powder were used.

Keyword : powder sample, amino acid, Diffuse reflectance circular dichroism, DRCD method, L-Alanine, D-Alanine

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Fig. 1 Model DRCD-575 Powder CD Unit

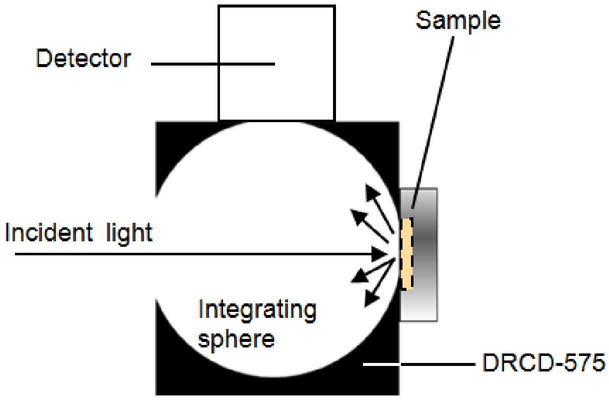

Fig. 2 Optical layout, DRCD-575

DRCD-575 Diffuse Reflectance Accessory

As shown in Figure 2, Diffuse Reflectance CD (DRCD) can be measured by placing a powder sample in the measurement position for diffuse reflection (opposite to the inlet port of incident light) and by locating the detector in close contact with the integrating sphere at 90 degrees to the incident light axis.

Also, Diffuse Transmission measurement can be made by setting the sample at the inlet port of incident light and a diffuse plate (white plate) is located at the sample position for diffuse reflection measurement. In diffuse transmission measurement mode, dilution is often required and it is sensitive enough for measurement of very small amounts of sample.

Measurement

In order to minimize the influence from optical anisotropy, the alanine powder sample was well ground using a pestle and mortar and then the simultaneous CD and LD measurement was performed using the multi-probe function of the J-1500 CD spectrometer.

System configuration:

P/N: 7000-J006A	J-1500-150ST CD Spectrometer
P/N: 7069-J034A	PML-534 FDCD PMT Detector
P/N: 7069-J025A	FLM-525 N2 gas flow meter
P/N: 7069-J075A	DRCD-575 Solid state(powder) CD measurement unit (powder cell incl.)

Conditions:

Wavelength range:	300-200 nm
CD/LD sensitivity:	10000 mdeg/1.0 delta OD
Scan speed:	100 nm/min
Response:	1 sec
Photometric mode:	DRCD, DRLD
Data interval:	0.1 nm
Spectral bandwidth (SBW):	2 nm
Number of accumulation:	5

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Results

The DRCD and DRLD spectra of L- and D-alanine powder samples are shown in Fig. 3. In the CD mode, L- and D-alanine spectra obtained are mirror images showing the same intensity of plus and minus sign respectively. In the LD mode, the signal intensity of each L and D spectrum obtained is less than +/- 0.005 ΔOD. These results indicate that the influence from optical anisotropy can be considered to be at a negligible level.

This application confirms that the J-1500 CD spectrometer fitted with the DRCD-575 diffuse reflectance accessory with multi-probe function is an excellent tool for the analysis of solid-state powder materials.

This powerful technique can be expanded to CD measurement of other solid-state materials such as metal complexes and super molecules.

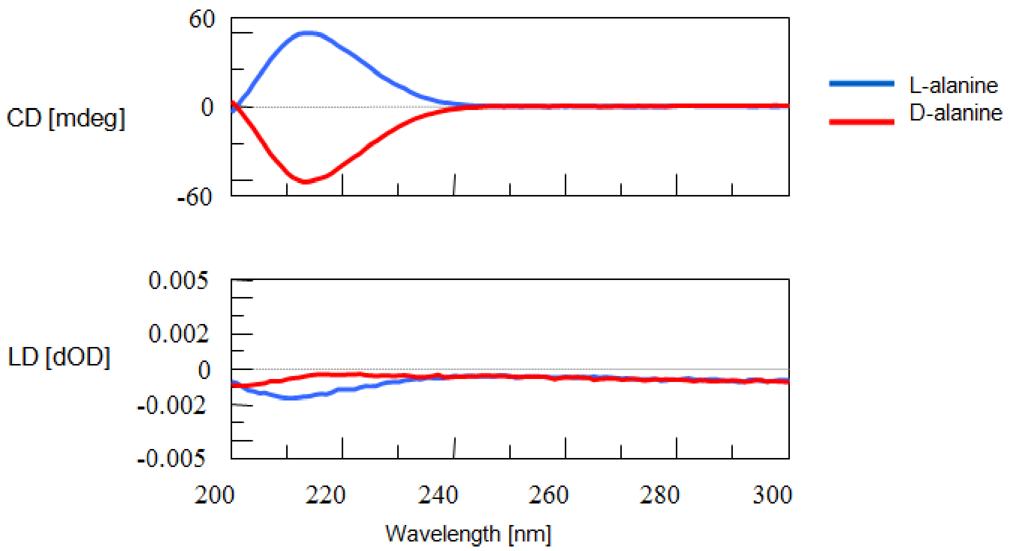

Fig. 3 CD and LD spectra of L- and D-alanine (accumulated)

References Ettore Castiglioni and Paolo Albertini, CHIRARITY, 2000, 12, 291-294.
Huibin Qiu, Yoshihira Inoue and Shunai Che, Angew. Chem. Int. Ed. 2009, 48, 3069-3072

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