

CD Measurement of alanine powder by using of DRCD method with Multi-probe function

Introduction

Generally, a sample for CD measurement needs to be a liquid (solution) sample, while recently there has been increasing requirements of CD measurement for hardly soluble sample or the sample whose structure may change in a solution. Such sample has to be measured in the solid state as it is. Although the transmission method can be applied to the CD measurement for such solid samples, there needs sample preparation such as making a pellet and also, if the sample dilution is required, it is not easy to recover the sample. For solving those difficulties in such transmission method, diffuse reflection (DR) CD method has been recommended.^{1), 2)} This application note illustrates the DRCD measurement by using of model DRCD-575 (See Fig. 1.) optimized for the J-1500 CD spectrometer with the multi-probe function.

DRCD is a method, in which the sample powder is located at the measurement point for diffuse reflection in integrating sphere and the CD is measured by utilizing diffuse reflection light (See Fig. 2.) and therefore, the DRCD can be effectively applied to the powder sample. However, in CD measurement of such solid sample, the influence of LD (linear dichroism) derived from the optical anisotropy of solid sample may have to be taken into consideration in order to minimize the CD artifact. The model J-1500 CD spectrometer allows the users to estimate the CD artifact easily by an incorporated quad digital lock-in amplifier allowing multi-probe function which enables the simultaneous measurement of CD and LD. As an example of the DRCD measurement, L- and D-alanine powder were used as sample.

Keywords: powder sample, amino acid, DRCD method



Fig. 1 Model DRCD-575 Powder CD Unit

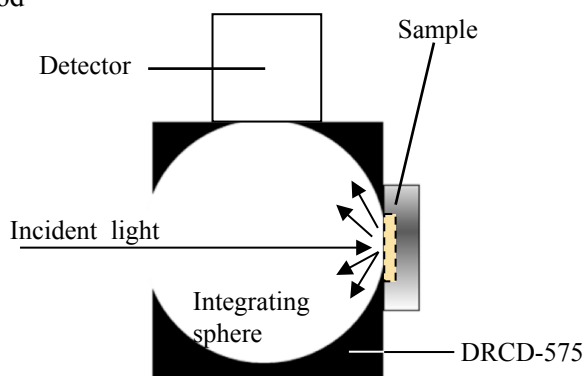


Fig. 2 Optical layout, DRCD-575

DRCD-575

The DRCD can be measured by placing the powder sample at the position for diffuse reflection measurement (opposite end of inlet port of incident light) and by locating the detector in close contact with the integrating sphere at 90 degrees side from the incident light axis. Also, the diffuse transmission measurement can be carried out when the sample is set at an inlet port of incident light and the diffuse reflection plate (white plate) is located at the sample position for diffuse reflection measurement. In the diffuse transmission measurement, the dilution of sample may be often required, while the very small amount of sample can be measured.

Measurement

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

System configuration

P/N: 7000-J006A	J-1500-450ST CD Spectrometer
P/N: 7069-J034A	PML-534 FDCD PMT Detector
P/N: 7069-J025A	FLM-525 N ₂ gas flow meter
P/N: 7069-J075A	DRCD-575 Solid state(powder) CD measurement unit

- *1) This application can be applied to the J-1500-150ST (P/N: 7000-J005A)
- *2) N₂ gas supply and regulator are required separately.
- *3) Water-circulator for cooling 450W Xe source needs to be prepared locally.
- *4) Powder CD unit can be applied to the transmission measurement using KBr pellet method.
- *5) Cell holder for powder sample is a standard item in DRCD-575.

Measurement parameters

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

Results

The DRCD and DRLD spectra of L- and D-alanine powder are shown in Fig. 3. In the CD mode, L- and D-alanine spectra obtained are in the mirror image 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 delta OD that is low enough. Such results indicate that the influence from optical anisotropy is considered to be in negligible level.

It is confirmed that the DRCD-575 Powder CD unit and J-1500 with multi-probe function can be used for the measurement of DRCD of alanine sample sufficiently powdered. It is expected that such powerful measurement technique will be able to expand the application of solid sample CD to the metal complex or super molecule.

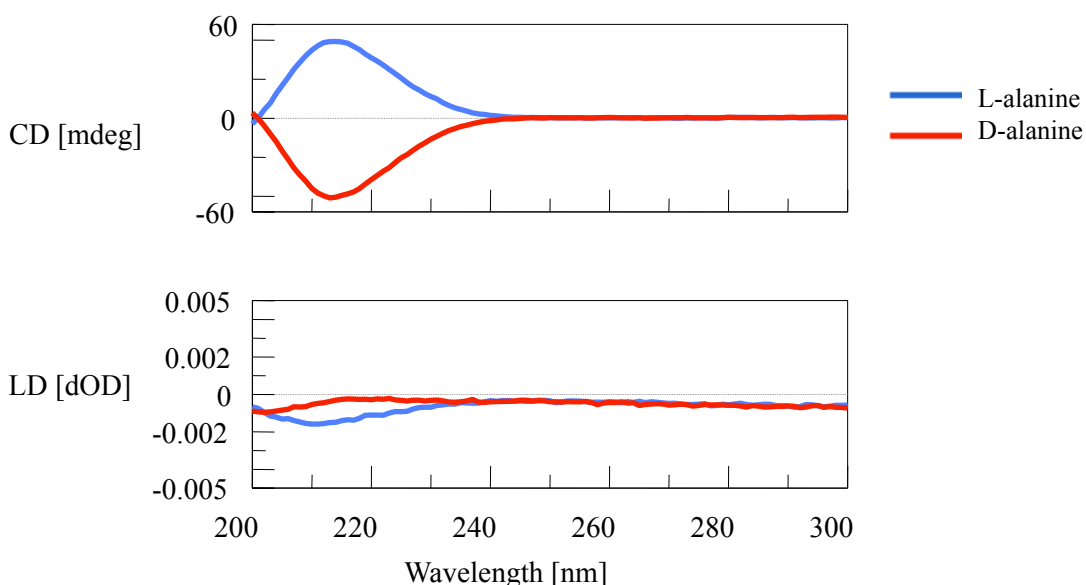


Fig. 3 CD and LD spectra of L- and D-alanine (accumulated)

Reference

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Huibin Qiu, Yoshihira Inoue and Shunai Che, *Angew. Chem. Int. Ed.* **2009**, *48*, 3069-3072.

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