Application Note

CD-0027

CD measurement at NIR region by J-1500
-Measurement of nickel tartrate and limonene-

Introduction

In the NIR region, observed CD spectra (NIR-VCD) are a result of the combination and overtone vibrational modes of the O-H and C-H transitions as well as the CD spectra (NIR-ECD) derived from the d-d transitions of metallic proteins and metal complexes. NIR-ECD spectra of the metal protein and metal complexes are known to be sensitive to quite detailed structures including the conformation of metal ligands as well as the configuration around the central metal, which is used for structural analysis of these molecules.[1)] For NIR-VCD, theories and calculation methods to interpret the NIR-VCD spectra have been introduced, and comparison analysis with many of the spectra has been accomplished. [2)]

The J-1500 CD Spectrometer can be used for data collection in the NIR region (up to 1600cm^{-1}) by replacing the detector and light source. In this configuration the instrument can be used for NIR-VCD measurement of the vibration transitions and NIR-ECD measurements of molecules including metal complexes, as well as ECD spectra in the UV region, primarily used for measurement of biological samples. Examples of the NIR-CD spectra of a nickel tartrate solution[3)] and limonene[2)] by the J-1500 CD Spectrometer are shown below.

Keywords: NIR-ECD, NIR-VCD, metal complex

Sample preparation

1. Nickel tartrate solution

Nickel sulfate solution of 0.24 M and sodium-potassium tartrate solution of 0.36 M mixed in a 1:1 volume ratio to prepare the nickel tartrate solution.

2. Limonene

Liquid (R)-(+) and (S)-(-) limonene in a 10 mm pathlength cylindrical cell.

Measurement condition

1. Nickel tartrate solution

Light source:	UV/Vis: Xe lamp	NIR: Halogen lamp (option)
Detector:	UV/Vis: PMT	NIR: InGaAs (option)
Measurement range:	UV/Vis: 235 - 940 nm	NIR: 940 - 1600 nm
Band width:	UV/Vis: 1 nm	NIR: 16 nm
Data interval:	UV/Vis: 0.1 nm	NIR: 1 nm
Path length:	UV/Vis: 10 mm	NIR: 0.5 mm

Scan speed: 200 nm/min
Response: 1 second
Accumulation: 1
Gain: 100x (InGaAs detector only)

2. Limonene

Light source: Halogen lamp (option)
Detector: InGaAs (option)
Measurement range: 1100 – 1350 nm
Measurement mode: CD/DC, UV single (Abs)
Band width: 16 nm
Data interval: 0.1 nm
Scan speed: 100 nm/min
Response: 2 seconds
Accumulation: 16
Gain: 100x

JASCO INTERNATIONAL CO., LTD
11-10, Myojin-cho 1-chome, Hachioji, Tokyo 192-0046, Japan
Tel: +81-42-649-3247, Fax: +81-42-649-3518

Result

1. Nickel tartrate solution

CD spectrum from the UV region to the NIR region of the nickel tartrate solution is shown in Figure 1. The vertical axis is converted to Molar ellipticity (Mol. Ellip) because the path length in the NIR region is different from the spectrum collected in the UV/Vis range. The spectrum can be measured with high sensitivity up to 1600 cm^{-1}, although any light absorptions from H_2O is observed in the region greater than 1400 nm.

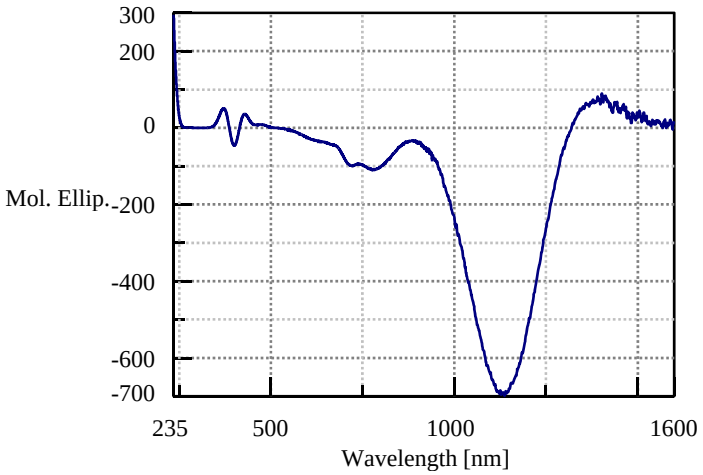

Fig. 1 CD spectrum of the nickel tartrate solution

Concentration of the nickel tartrate [3] : 0.1188 M in H_2O

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2. Limonene

Absorption and CD spectra derived from the double overtone of the C-H vibrational transition of the (R)-(+) and (S)-(-)-limonene are shown in Figure 2. The limonene of a racemic form was used for the blank of the CD spectrum. The limonene was measured with pathlengths of 10 mm and 2 mm because there is no appropriate solvent to serve as the blank for the liquid limonene at room temperature in the absorption spectrum. The difference spectrum (absorbance equivalent to the 8 mm path length) is multiplied by 1.25 and converted to the absorbance equal to a 10 mm path length. The very weak CD signal below 1 mdeg can thus be measured with high-sensitivity.

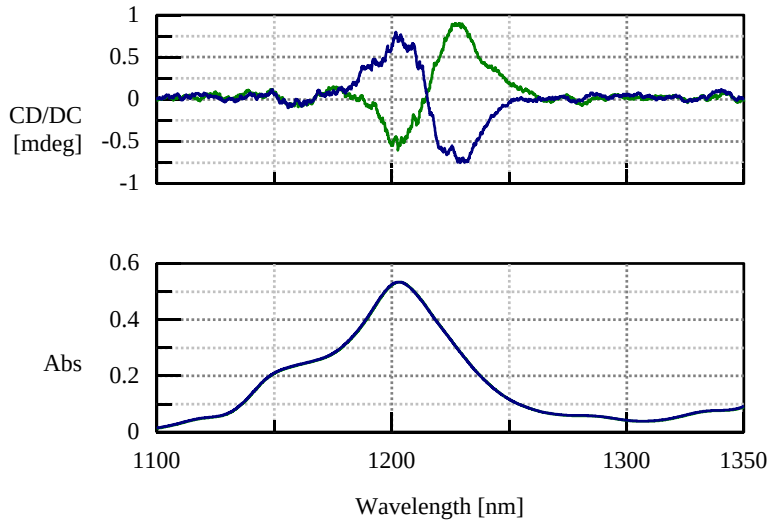

Fig. 2 CD spectrum of the limonene
(R) (+) - limonene
(S) (-) - limonene

Reference

(1) William A. Eaton, Graham Palmer, James A. Fee, Tokuji Kimura, and Walter Lovenberg, *Proc. Nat. Acad. Sci. USA*, 68, 12, 3015-3020, (1971)

(2) Sergio Abbate, Ettore Castiglioni, Fabrizio Gangemi, Roberto Gangemi, and Giovanna Longhi, *CHIRALITY*, 21, Issue 1E E242-E252, (2009)

(3) T. Konno, H. Meguro, T. Murakami, and M. Hatano, *CHEMISTRY LETTERS*, 953-956, (1981)

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