Application Note
CD Spectroscopy

Measurement of NIR-CD spectra of nickel titrate and limonene using a J-1500 CD spectrometer

Introduction

In the near-infrared (NIR) region, observed CD spectra are a result of the combination and overtone vibrational modes of the O-H and C-H transitions (NIR-VCD), as well as the d-d transition of metallo-proteins and metal complexes (NIR-ECD). NIR-ECD spectra of metallo-protein and metal complexes are known to be sensitive to conformations of the metals ligands as well as the configuration around the central metal atom, 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 comparative analysis for many of the spectra have been accomplished.[2]

The J-1500 CD spectrometer can be used for spectral measurements into the NIR region (up to 1600 nm) using an extended wavelength range light source and detector. In this configuration, the instrument can be used for NIR-VCD measurements of vibrational transitions and NIR-ECD measurements of metal complexes. The J-1500 can also be used to measure the ECD spectrum in the UV region, primarily for the study of biological samples.

This application notes demonstrates the use of the J-1500 CD spectrometer to probe vibrational and electronic transitions of nickel tartrate solution[3] and limonene[2] in the NIR region of the spectrum.

Keywords

J-1500, electronic circular dichroism, vibrational circular dichroism, near infrared region, nickel tartrate, limonene, NIR-ECD, NIR-VCD, metal complexes, chemical

JASCO J-1500 CD spectrometer
View product information at www.jascoinc.com

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Experimental

Measurement conditions: nickel tartrate		
	UV/Vis	**NIR**
Light source	Xenon lamp	Halogen lamp (optional)
Detector	PMT	InGaAs (optional)
Measurement range	235 - 940 nm	940 - 1600 nm
Spectral band width	1 nm	16 nm
Data acquisition interval	0.1 nm	1 nm
Path length	10 mm	0.5 mm
Scan speed	200 nm/min	
Response time	1 sec	
Accumulations	1 time	
Gain	100X (InGaAs detector only)	

Measurement conditions: limonene	
Light source	Halogen lamp (optional)
Detector	InGaAs (optional)
Measurement range	1100 - 1350 nm
Measurement mode	CD/DC, UV single (Abs)
Spectral band width	16 nm
Data acquisition interval	0.1 nm
Scan speed	100 nm/min
Response time	2 sec
Accumulations	16 times
Gain	100X

A nickel tartrate solution was prepared by mixing 0.24 M nickel sulfate solution and 0.36 M sodium potassium tartrate solution with a 1:1 volume ratio. R-(+) and S-(-) limonene were used neat.

Results

A UV/Vis and NIR CD spectrum of nickel tartrate solution is shown in Figure 1. The vertical axis is converted to molar ellipticity to compensate for the difference in path length for the spectra collected in the UV/Vis region and NIR region. The spectra can be measured with high sensitivity up to 1600 nm, although some absorption from H2O is observed in the region above 1400 nm.

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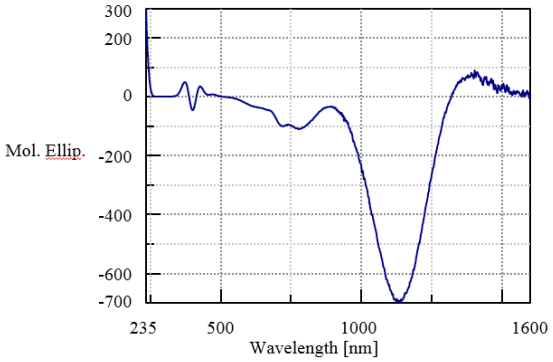

Figure 1. CD spectrum of nickel tartrate solution in the UV/Vis and NIR regions.

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

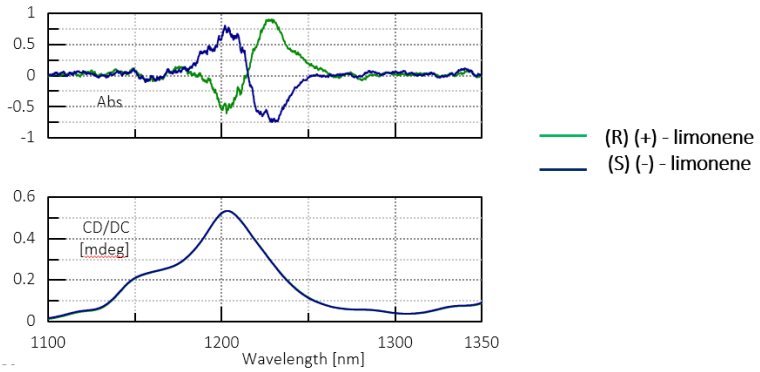

Figure 2. CD (top) and absorption (bottom) spectra of R-(+)-limonene (green) and S-(-)-limonene (blue).

Conclusion

This application note demonstrates that the J-1500 CD spectrometer can be used to probe vibrational transitions in the near infrared region of the CD spectrum with high sensitivity.

References

1. Eaton, W. A., Palmer, G., Fee, J. A., Kimura, T., and W. Lovenberg, PNAS (1971), 68, 12, 3015-3020.

2. Abbate, S., Castiglioni, E., Gangemi, F., Gangemi, R., and G. Longhi, Chirality (2009), 21, 1, 242-252.

3. Konno, T., Meguro, H., Murakami, T., and M. Hatano, Chemistry Letters (1981), 953-956.

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