

**STANDARDS FOR CD SCALE CALIBRATION**

Calibration of the vertical scale of CD spectrometers has been a widely discussed topics, a few suitable standards are reported here, but discussion on absolute calibration will be reported in another report.

ACS and CSA, well defined band at around 290.5 nm and opposite intensity band at 192 nm.

ACS is easier to handle since less deliquescent and it's the standard supplied with Jasco instruments:

a 0.06% aqueous solution (60mg in 100ml) of d-10-camphorsulfonate in a 10mm cell should give a signal of +190.4 mdeg ($[\Theta]_{290.5} = +7910$)

(1S)-(+)-10-camphorsulfonic acid (CSA)

FW 232.30 $[\alpha]_D^{20} +19.9^\circ$ c2, H₂O $[\Theta]_{291} = +7820$ available from Aldrich C210-7 or from Sigma C-1395

(1R)-(-)-10-camphorsulfonic acid (CSA)

FW 232.30 $[\alpha]_D^{20} -21^\circ$ c2, H₂O $[\Theta]_{291} = -7820$ Aldrich 28,214-6

(1R)-(-)-10-camphorsulfonic acid, ammonium salt (ACS)

FW 249.33 $[\alpha]_D^{22} -18.4^\circ$ c=5.3, H₂O $[\Theta]_{290.5} = -7910$ Aldrich 18,836-0

(1S)-(+)-10-camphorsulfonic acid, ammonium salt (ACS)

FW 249.33 $[\alpha]_D^{25} +20.9^\circ$ c=6, H₂O $[\Theta]_{290.5} = +7910$

available from Katayama Chemical, it can be ordered directly from Jasco

Pantolactone is a good standard for UV region with a strong band at 219 nm.

A 0.03% aqueous solution (15mg in 100 ml) of D-(-)-pantolactone in a 10mm cell should give a signal of -186 mdeg ($[\Theta]_{219} = -16160$)

(R)-(-)-Pantolactone

FW 130.15 $[\alpha]_D^{25} -49.8^\circ$ c=2, H₂O $[\Theta]_{219} = -16160$ Aldrich 23,781-7

(S)-(+)-Pantolactone

FW 130.15 $[\alpha]_D^{20} +53^\circ$ c=2.4, H₂O $[\Theta]_{219} = +16160$ Aldrich 43732-8

Epiandrosterone shows a positive band at 304 nm

A 0.05% solution (w/v) in dioxane in a 10mm cell should give a signal of 187.3 mdeg

Epiandrosterone

FW 290.45 $[\alpha]_D^{20} +92^\circ$ c=1, CH₃OH $[\Theta]_{304} +10880$ Aldrich 23,440-0, Sigma E-3375

Tris (ethylenediamine) Co complex has a very strong band at 490 nm and other weaker ones toward the UV, with a very strong band of opposite polarity at about 209 nm.

This sample is unfortunately not commercially available.

2(+)-D-[Coen₃]Cl₃•NaCl•6H₂O

FW 857.78 $[\alpha]_D +128$ $[\Theta]_{490} +6431$

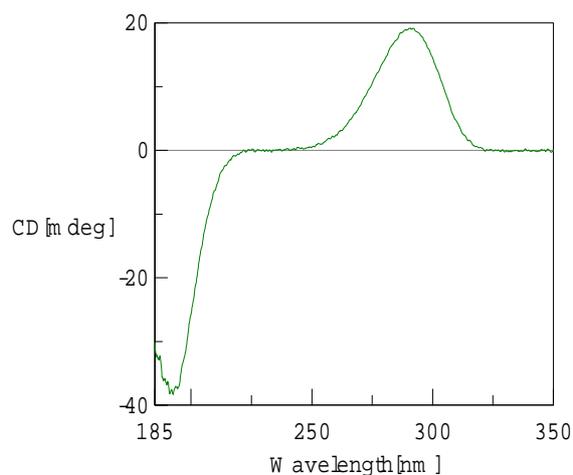
2(-)-D-[Coen₃]Cl₃•NaCl•6H₂O

FW 857.78 $[\alpha]_D -128$ $[\Theta]_{490} -6431$

Examples:

ACS spectra in a 1 mm cell is very effective to test both absolute calibration (at 290.5 nm) and band ratio intensity 192/290.

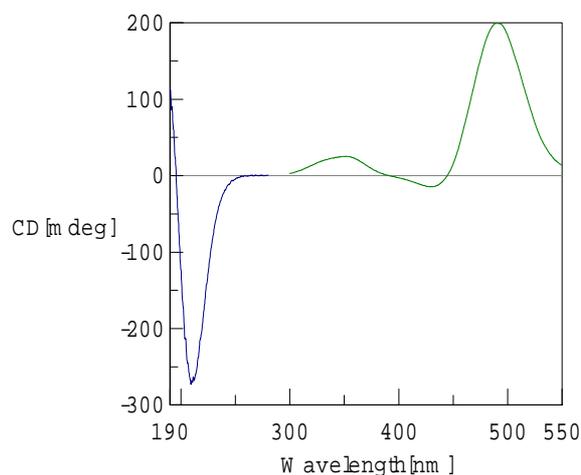
It's therefore the proper sample for applications dealing with UV and far UV.



The spectra of Co complex is shown on the right.

Two different concentrations are necessary to collect all bands.

This sample is very suitable to check overall performances when visible range is also a concern.



References:

Takakuwa T., Konno T., Meguro H. *Anal.Sci.* 1 (1985) 215
Schippers P.H., Dekkers H.P.J.M. *Anal.Chem.* 53 (1981) 778
European Pharmacopoeia (Addendum 2001) 2.2.41