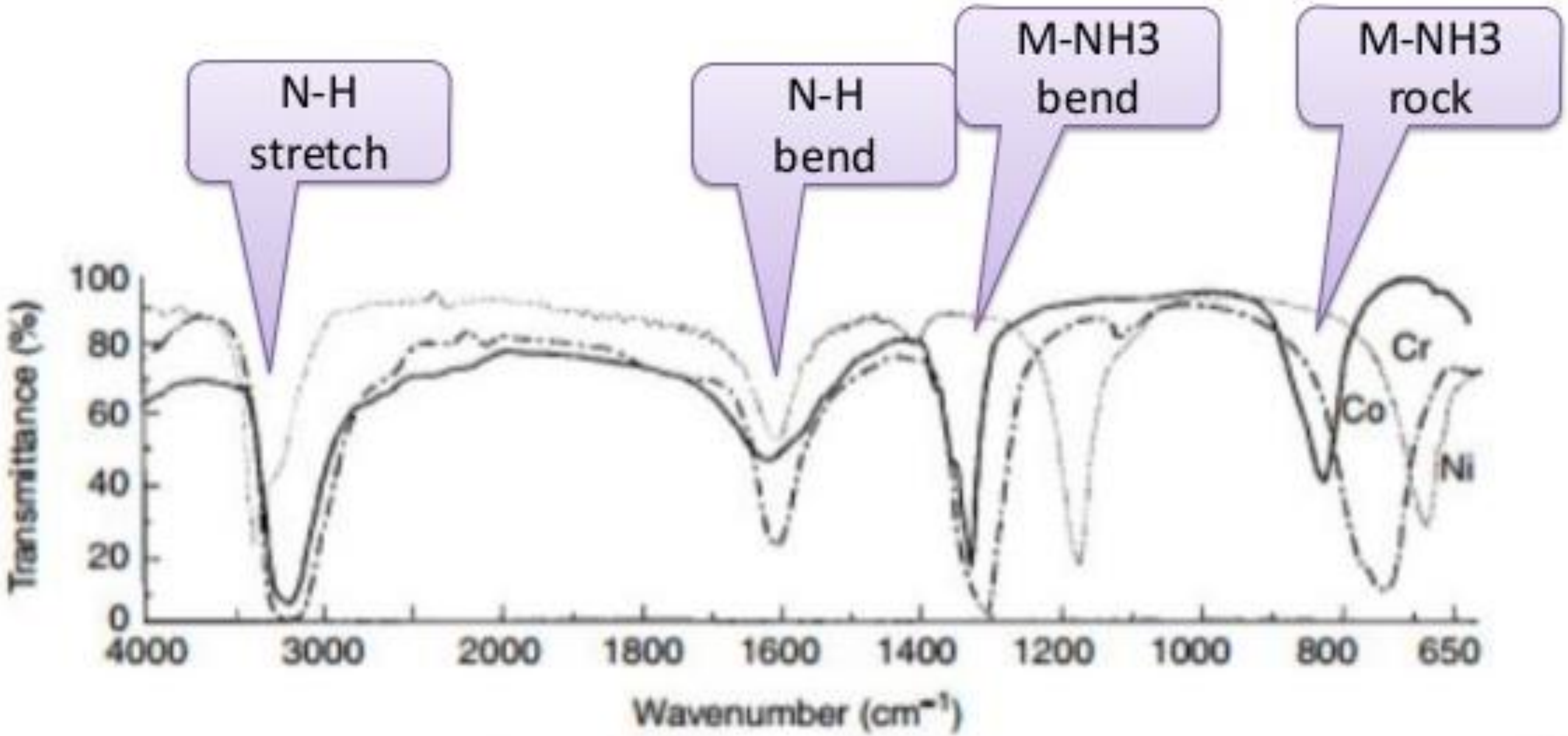
Measuring Properties of Inorganic Compounds in Lab

1. **IR spectra**
2. **UV-Vis spectra**
3. **Conductivity**

Measuring Properties of Inorganic Compounds in Lab

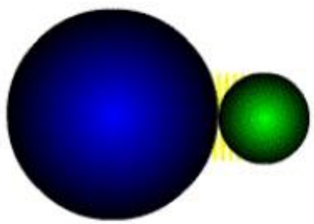
1. IR spectra

M(II)hexamine complexes



Principle of IR spectroscopy

- Molecules are made up of atoms linked by chemical bonds. The movement of atoms and the chemical bonds like like spring and balls (vibration)



Vibration of a Diatomic Molecule
Approximates an Oscillating Spring

- This characteristic vibration are called Natural frequency of vibration.

BASIC PRINCIPLE:

- A chemical substance shows marked selective absorption in IR region.
- After absorption of IR radiation the molecules of chemical substance vibrate at many rates of vibration giving rise to closely packed absorption spectrum called as **IR absorption spectrum**.
- Various bands present in the IR spectrum corresponds to characteristic functional group and bonds present in the chemical substance.
- Thus IR spectrum of a chemical substance is fingerprint for its identification.

MULL TECHNIQUE:

- In this technique a small quantity of sample is thoroughly ground in a clean mortar until the powder is very fine.
- After grinding, the mulling agent (mineral oil or Nujol) is introduced in small quantities just sufficient to take up the powder (mixture approximates the consistency of a toothpaste).
- The mixture is then transferred to the mull plates & the plates are squeezed together to adjust the thickness of the sample between IR transmitting windows.
- This is then mounted in a path of IR beam and the spectrum is run.

Demerit:

- Although Nujol is transparent throughout IR region, yet it has a disadvantage that it has absorption maxima at 2915, 1462, 1376 & 719 cm^{-1}.
- So when IR spectrum of solid sample is taken in Nujol mull, absorption bands of solid sample that happen to coincide with the absorption bands of the Nujol mull will be hidden (but others will be clearly seen in IR spectrum) and then interferes with the absorption of the sample.

- Infrared spectroscopy (IR) measures the bond vibration frequencies in a molecule and is used to determine the functional group.

- The IR region is divided into three regions:

  1. The near IR (12500-4000 cm^{-1}) (overtons region)
  2. The mid IR (4000-200 cm^{-1})
  3. The far IR (200-10 cm^{-1})

- The mid IR region is of greatest practical use to the organic compounds.


excellent
cloudy
defective areas
scratched
chipped

Cyclohexanol neat solution

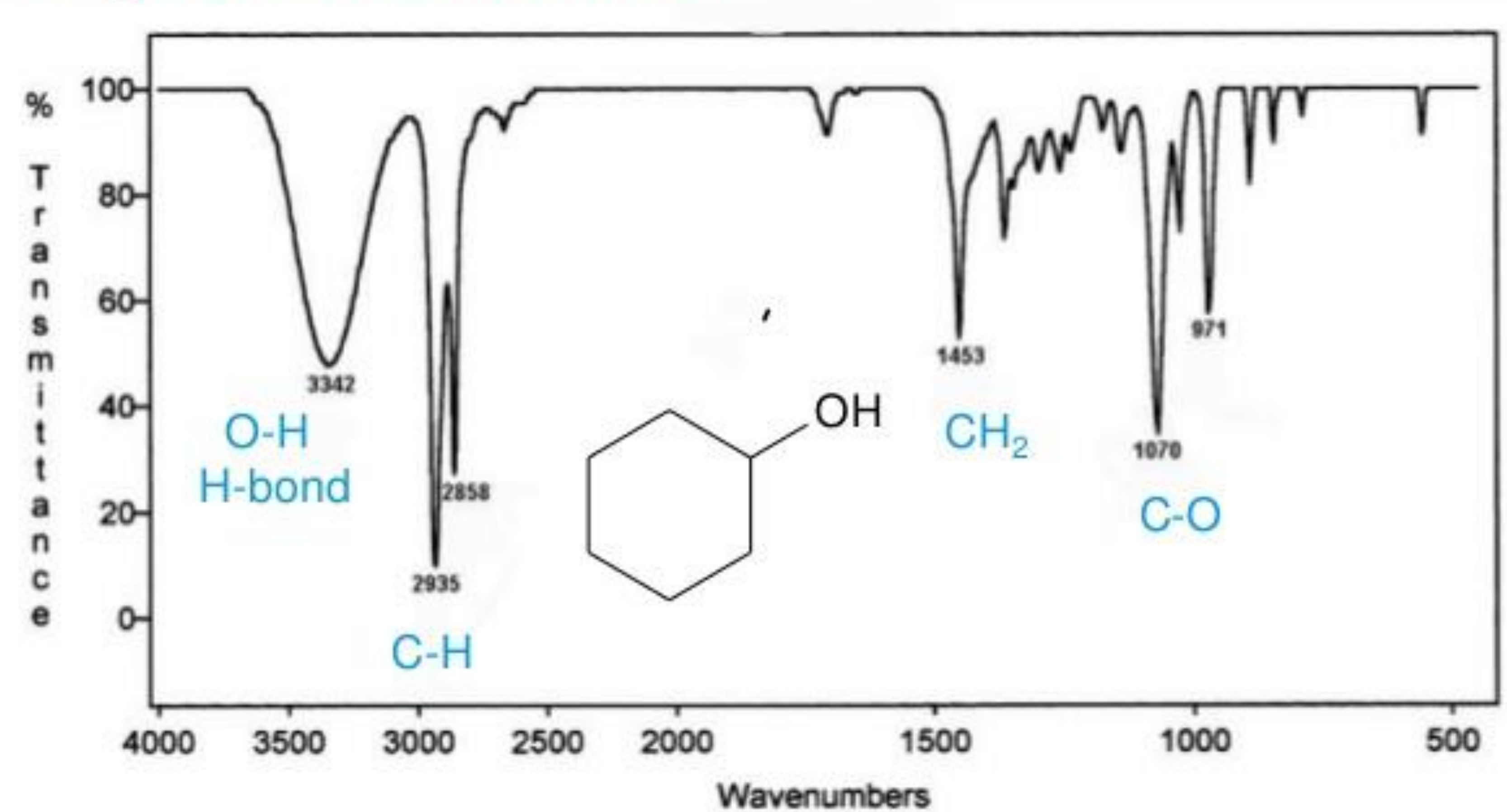


Table 13.4 Important IR Stretching Frequencies

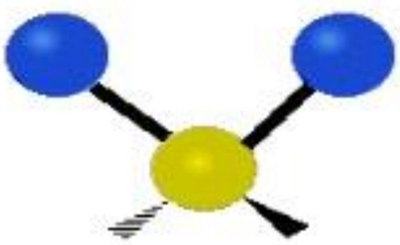
Type of bond	Wavenumber (cm^{-1})	Intensity
$C\equiv N$	2260–2220	medium
$C\equiv C$	2260–2100	medium to weak
$C=C$	1680–1600	medium
$C=N$	1650–1550	medium
	~1600 and ~1500–1430	strong to weak
$C=O$	1780–1650	strong
$C-O$	1250–1050	strong
$C-N$	1230–1020	medium
$O-H$ (alcohol)	3650–3200	strong, broad
$O-H$ (carboxylic acid)	3300–2500	strong, very broad
$N-H$	3500–3300	medium, broad
$C-H$	3300–2700	medium

MOLECULAR VIBRATIONS

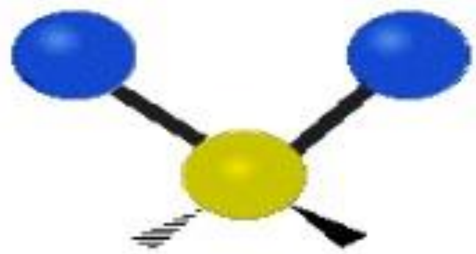
There are 2 types of vibrations.

1) Stretching vibrations
2) Bending vibrations

- 1)Stretching vibrations: in this bond length is altered.
- They are of 2 types
- a) symmetrical stretching: 2 bonds increase or decrease in length.



b) Asymmetrical stretching: in this one bond length is increased and other is decreased.



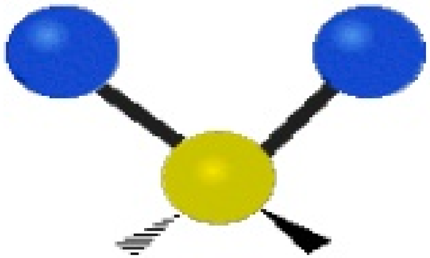
2)Bending vibrations:

- These are also called as deformations.
- In this bond angle is altered.
- These are of 2 types
- a) in plane bending→ scissoring, rocking
- b) out plane bending→ wagging, twisting

Scissoring:

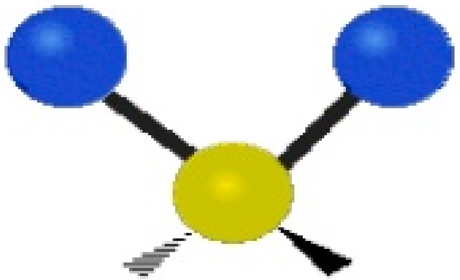
This is an in plane bending.

In this bond angles are decreased.2 atoms approach each other.



Rocking:

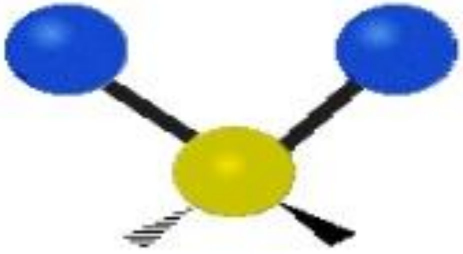
- In this movement of atoms takes place in same direction.



Wagging:

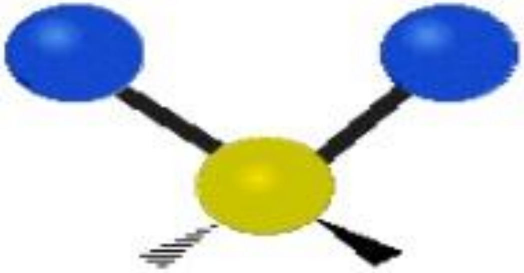
It is an out of plane bending.

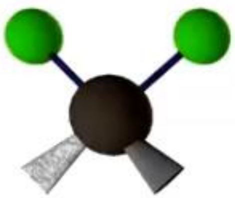
In this 2 atoms move to one side of the plane. They move up and down the plane.



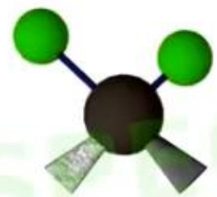
Twisting:

- In this one atom moves above the plane and the other atom moves below the plane.

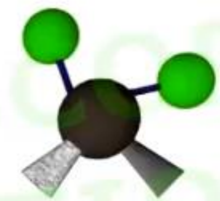




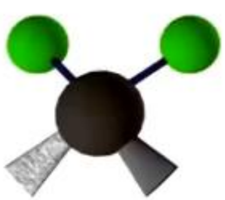
SYMMETRIC STRECHING



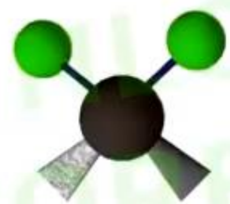
ANTISYMMETRIC STRECHING



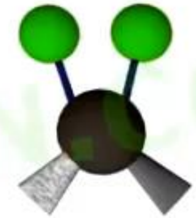
ROCKING



WAGGING

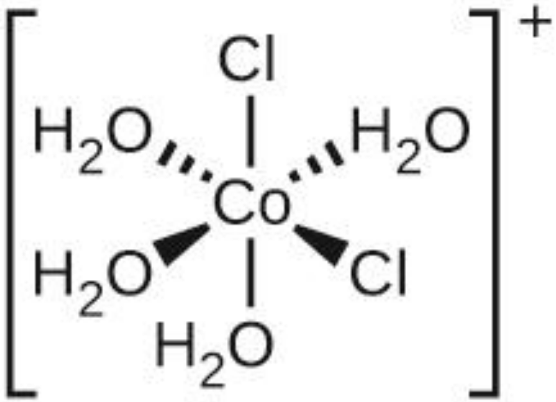


TWISTING

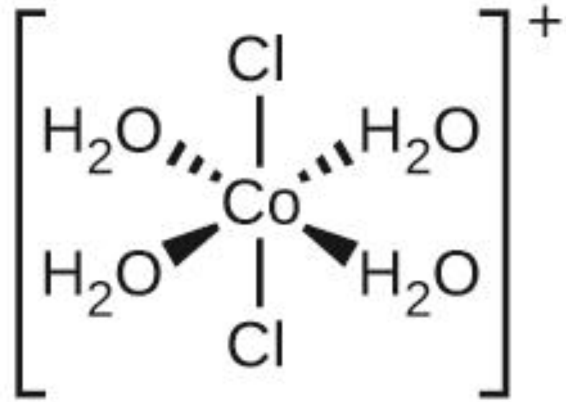


SCISSORING

Violet, *cis* form



Green, *trans* form



Here, color can distinguish the two

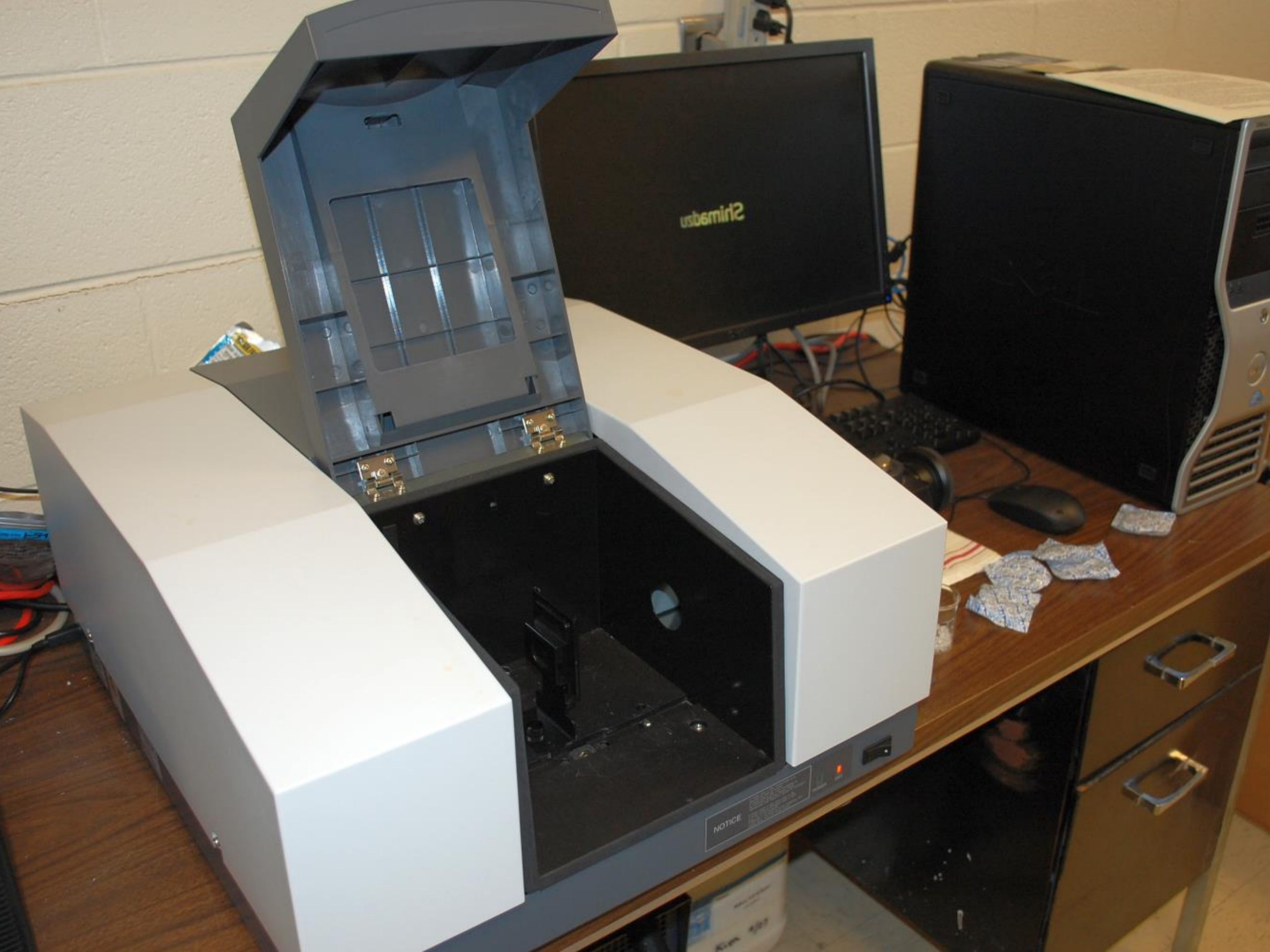
Also Far IR (500 – 300 cm^{-1})

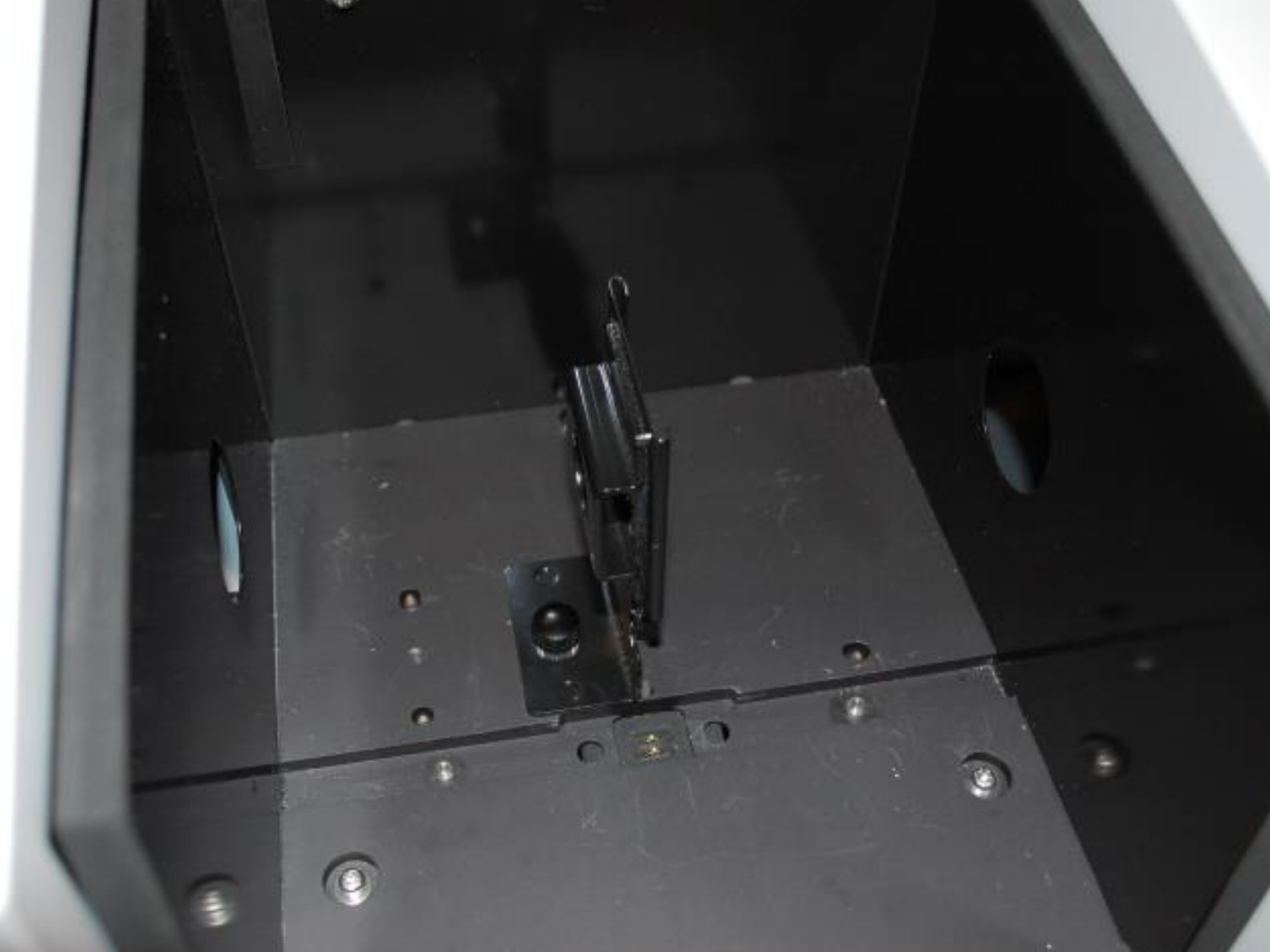
Trans: one peak

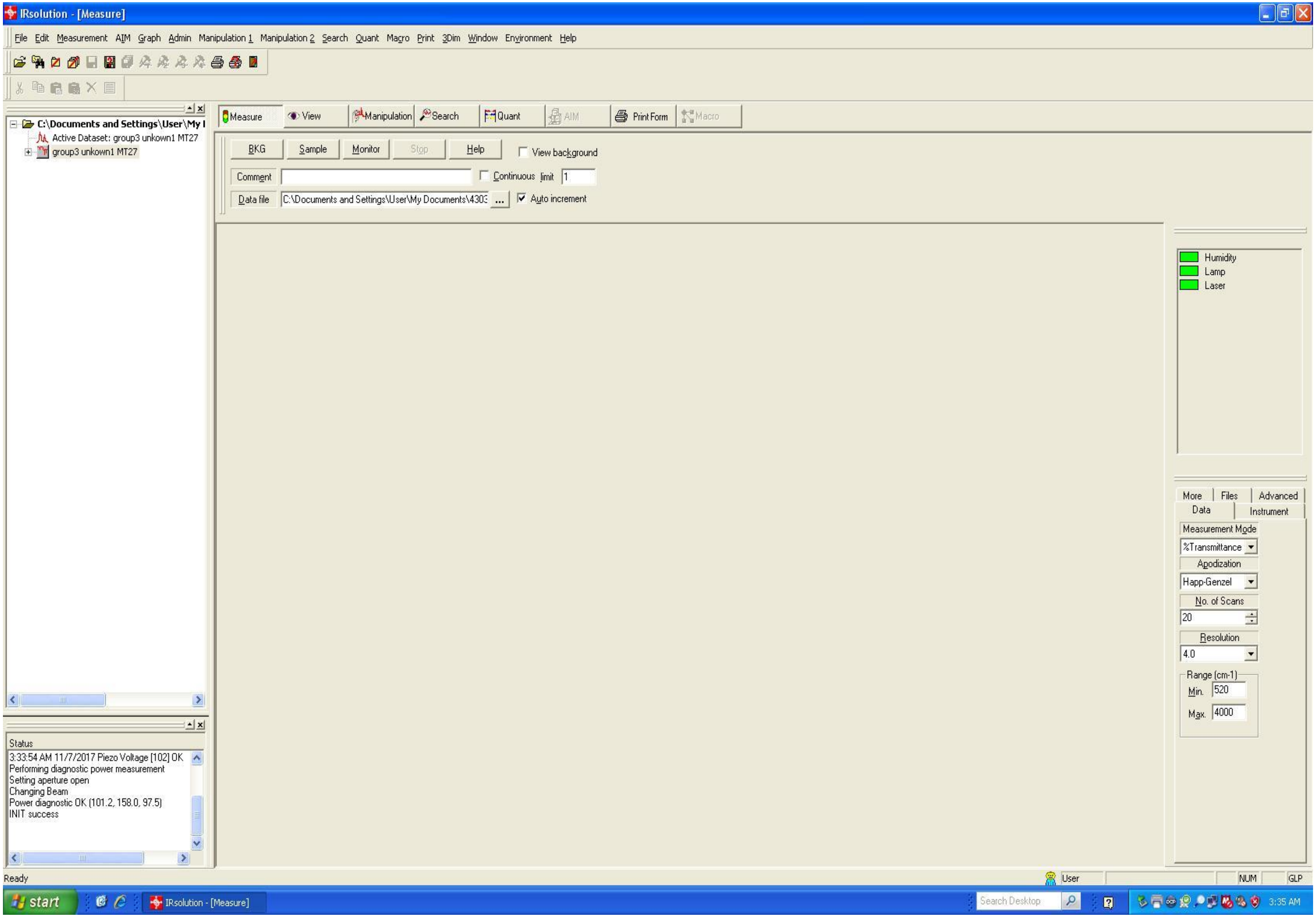
Cis: two peaks


F40-8
100g
Thermo
Nujol




Shimadzu
NOTICE




IRsolution - [Measure]
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C:\Documents and Settings\User\My I
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Measure
View
Manipulation
Search
Quant
AIM
Print Form
Macro
BKG
Sample
Monitor
Stop
Help
View background
Comment
Continuous
limit
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C:\Documents and Settings\User\My Documents\4303
Auto increment
Humidity
Lamp
Laser
More
Files
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Instrument
Measurement Mode
%Transmittance
Apodization
Happ-Genzel
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Resolution
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Range (cm-1)
Min.
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Max.
4000
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Setting aperture open
Changing Beam
Power diagnostic OK (101.2, 158.0, 97.5)
INIT success
Ready
User
NUM
GLP
start
IRsolution - [Measure]
Search Desktop
3:35 AM

Parameter	Value
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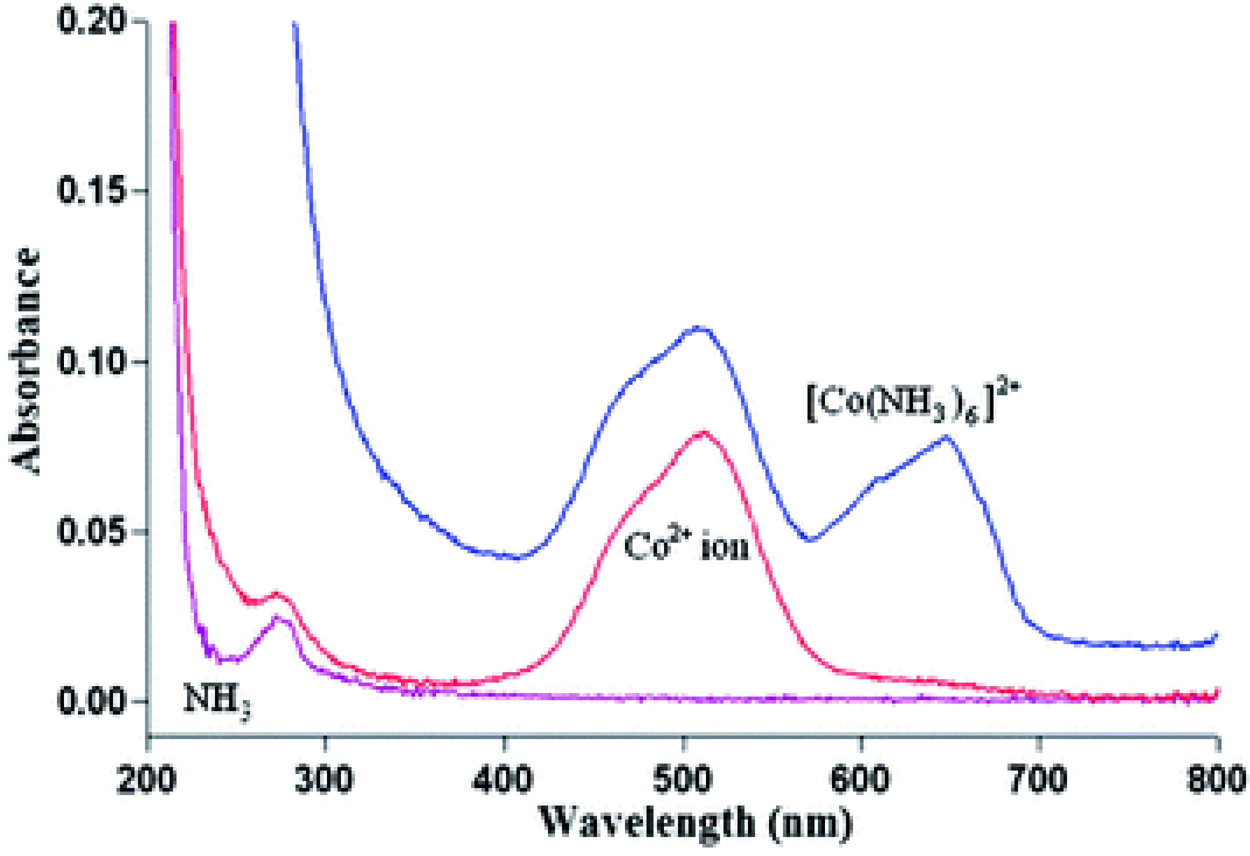
Created	10/26/2...
Dimensions	2
X-Axis	Wavenu...
Units	1/cm
Datapoints	1806
Min	518.85
Max	4000.36
Interval	1.93
Intensities	Percent ...
Units	%T
Datapoints	1806
Min	30.104
Max	103.024

No.	Peak	Intensity
1	688.59	49.176
2	702.09	55.494
3	736.81	30.104
4	972.12	52.796
5	1122.57	34.822
6	1265.30	57.307
7	1450.47	62.067
8	1606.70	75.163
9	1625.99	55.753
10	1666.50	47.974
11	2694.56	89.685
12	2740.85	86.874
13	2816.07	84.900
14	2852.72	79.526
15	2924.09	74.833
16	2951.09	80.341

Measuring Properties of Inorganic Compounds in Lab

2. UV-Vis spectra



Principles of Spectroscopy

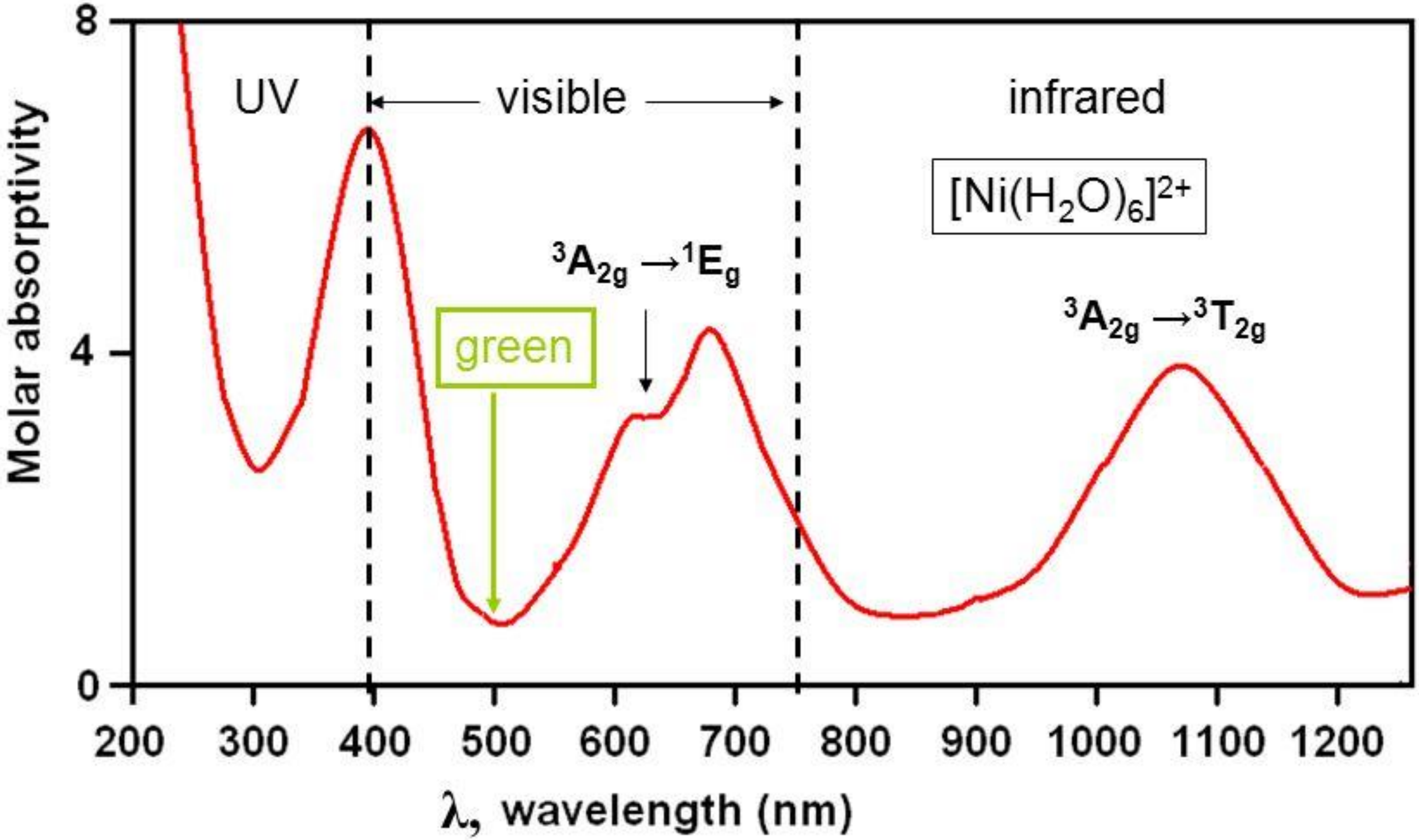
- The principle is based on the measurement of spectrum of a sample containing atoms / molecules.

- Spectrum is a graph of intensity of absorbed or emitted radiation by sample verses frequency (ν) or wavelength (λ).

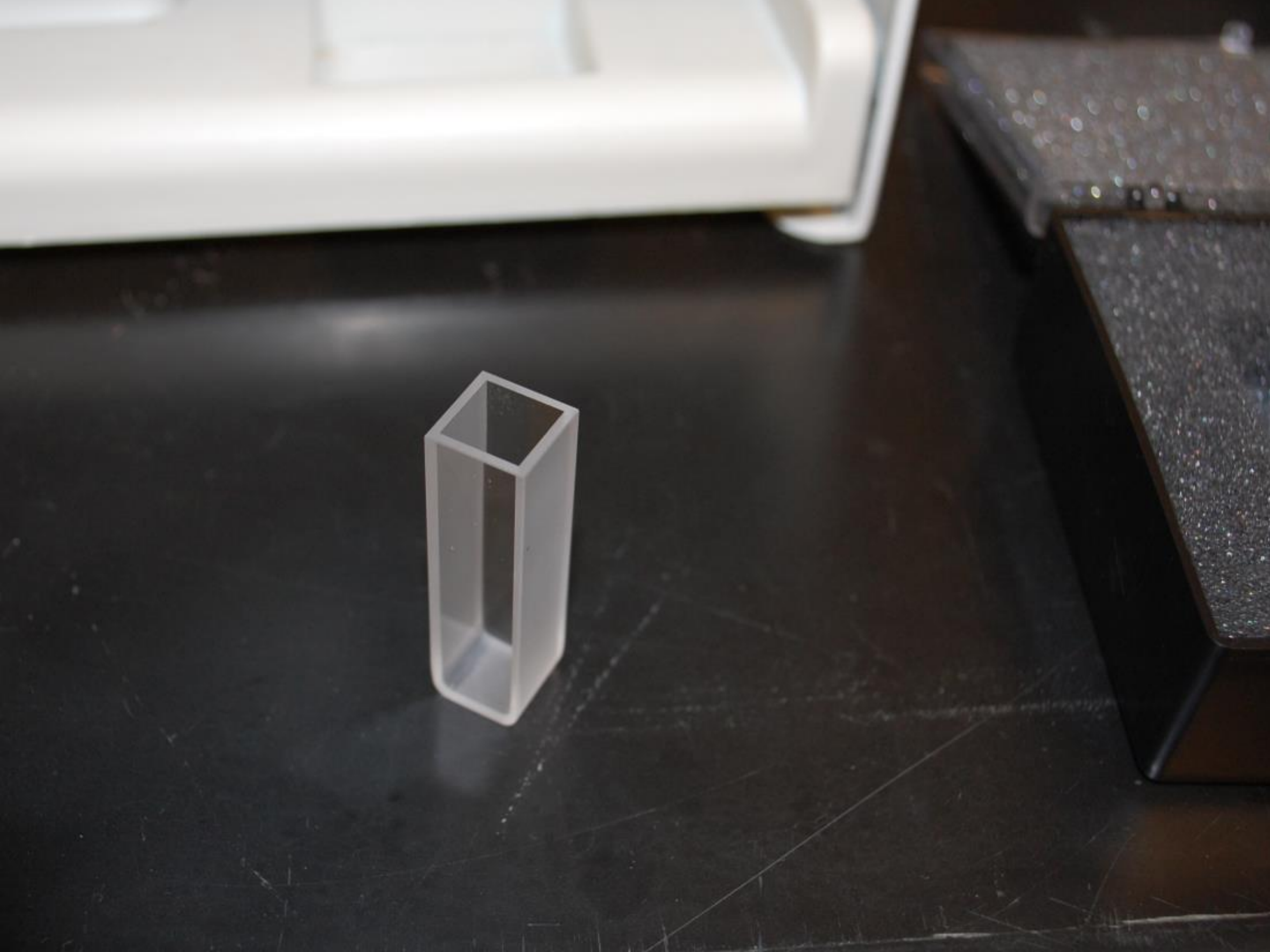
- Spectrometer is an instrument design to measure the spectrum of a compound.

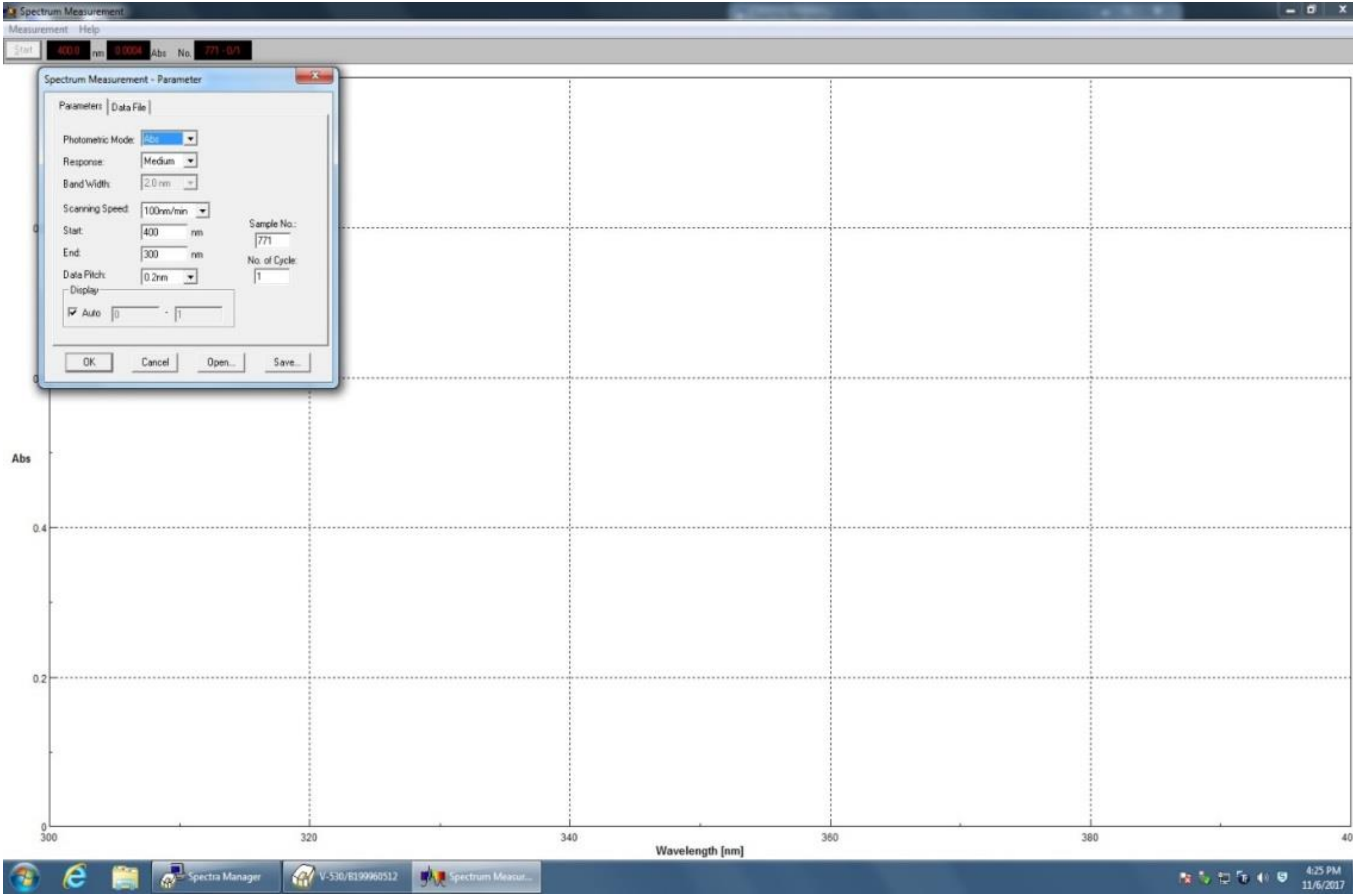
The electronic spectrum of $[Ni(H_2O)_6]^{2+}$:

The complex looks green, because it absorbs only weakly at 500 nm, the wavelength of green light.




Jasco
V-530




Spectrum Measurement
Measurement Help
Start
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Spectrum Measurement - Parameter
Parameters
Data File
Photometric Mode: Abs
Response: Medium
Band Width: 2.0 nm
Scanning Speed: 100nm/min
Start: 400 nm
End: 300 nm
Data Pitch: 0.2nm
Sample No.: 771
No. of Cycle: 1
Display
Auto 0 - 1
OK
Cancel
Open...
Save...
Abs
0.4
0.2
0
300
320
340
360
380
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Wavelength [nm]
Spectra Manager
V-530/B199960512
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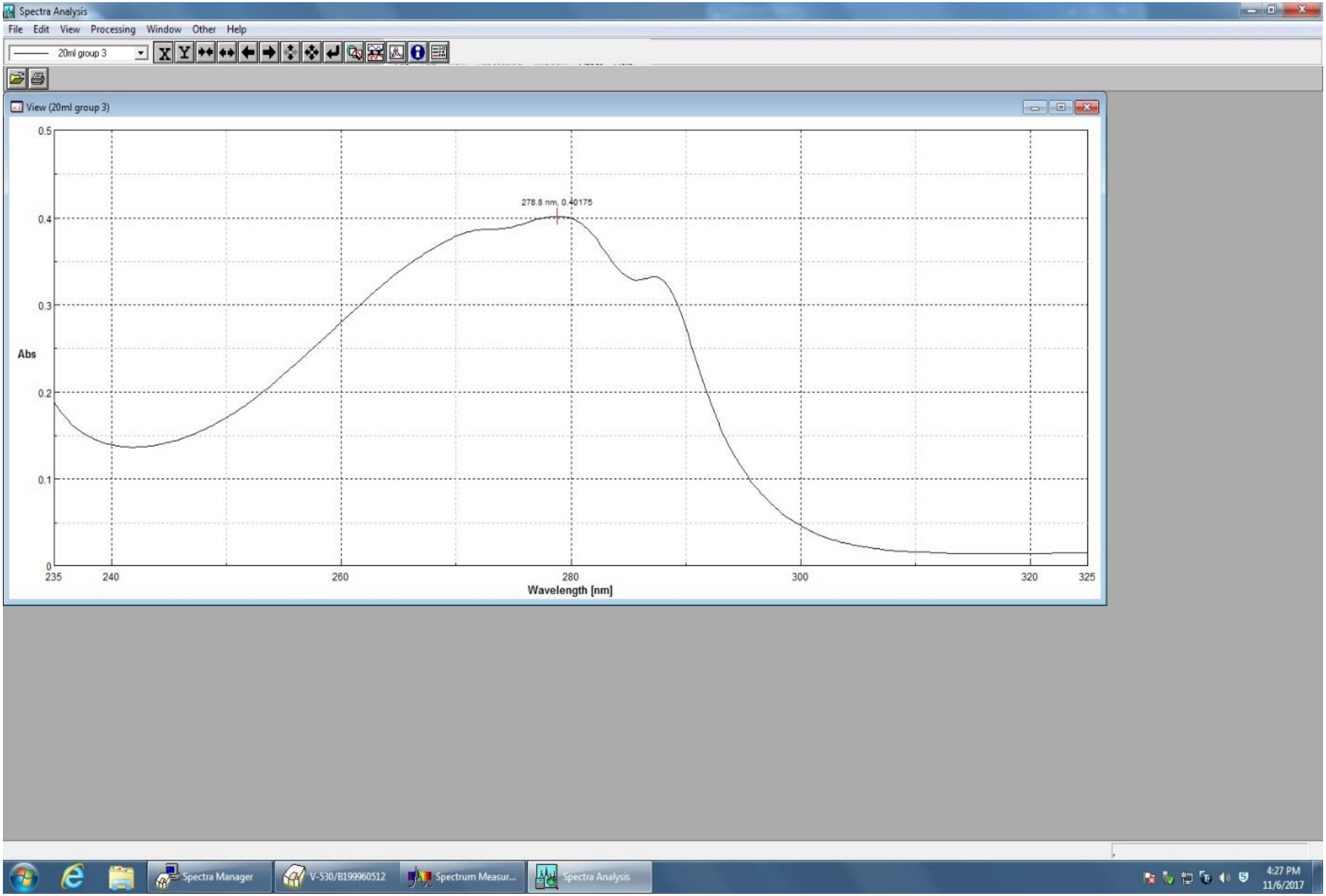

Sample vis spectrum

Measuring Properties of Inorganic Compounds in Lab

3. Conductivity

$[Co(NH_3)_3Cl_3]$ – does not ionize.

$[Co(NH_3)_4Cl_2]Cl = [Co(NH_3)_4Cl_2]^+ + Cl^-$ (2 ions)

$[Co(NH_3)_6]Cl_3 = [Co(NH_3)_6]^{3+} + 3Cl^-$ (4 ions)

$[Co(NH_3)_5Cl]Cl_2 = [Co(NH_3)_5Cl]^{2+} + 2Cl^-$ (3 ions)

As the number of ions in solution increases, their conductivity also increases.

Therefore, conductivity follows the order:

$[Co(NH_3)_3Cl_3] < [Co(NH_3)_4Cl_2]Cl < [Co(NH_3)_5Cl]Cl_2 < [Co(NH_3)_6]Cl_3$

Number of ions	molar conductivity, $\Omega^{-1}\ cm^2\ mol^{-1}$
2 (1:1)	96-150
3 (1:2)	225-273
4 (1:3)	380-435
5 (1:4)	540-560

(For aqueous solutions)


MODEL 31A
YSI
CONDUCTANCE BRIDGE
EXT CAP
LO
HI
LINE
OFF
1KHz
DRIVE
OPERATING INSTRUCTIONS
1. Connect the conductivity cell or resistance to be measured between the two blue binding posts on the top panel.
2. Connect compensating capacitor, if any, between the end blue and the black binding posts on the top panel.
3. Apply power, selecting either LINE or 1K Hertz bridge frequency.
4. Adjust SENSITIVITY control to obtain a dark "window" in the center of the null indicator.
5. Rotate the RANGE/FUNCTION switch to find the longest dark window on the null indicator. Re-adjust the SENSITIVITY control as necessary. Indication may be ambiguous between two adjacent ranges.
6. Rotate the DRIVE control to obtain the longest window on the null indicator. Re-adjust the SENSITIVITY control as necessary. It may be necessary to switch to an adjacent range if value is near one end of the dial scale.
7. Read the conductance (resistance) value off of the dial scale and multiply by scale factor from the function switch to obtain the final reading.
(For further information see instruction manual.)


CE BRIDGE
EXT.
CAP.
LO
HI
X1
µMHO
3.0
X100,000
OHMS
2.0
X10,000
OHMS


YSI
Yellow Springs Instrument Co., Inc.

OPERATING INSTRUCTIONS

1. Connect the conductivity cell or resistance to be measured between the two blue binding posts on the top panel.
2. Connect compensating capacitor, if any, between the end-blue and the black binding posts on the top panel.
3. Apply power, selecting either LINE or 1K Hertz bridge frequency.
4. Adjust SENSITIVITY control to obtain a dark ''window'' in the center of the null-indicator.
5. Rotate the RANGE/FUNCTION switch to find the longest dark-window on the null indicator. Re-adjust the SENSITIVITY control as necessary. Indication may be ambiguous between two adjacent ranges.
6. Rotate the DRIVE control to obtain the longest window on the null-indicator. Re-adjust the SENSITIVITY control as necessary. It may be necessary to switch to an adjacent range if value is near one end of the dial-scale.
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(For further information see instruction manual.)