How-To: Fourier Transform Infrared Spectroscopy (FTIR)

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Group Meeting 141027

Beer-Lambert Law

$$T = \frac{I}{I_0} = e^{-\varepsilon bc}$$

$$A = -ln(T) = -ln\left(\frac{I}{I_0}\right)$$

$$A = \varepsilon bc$$

T = transmittance (relative amount of light passing through a sample)

I, I_0 = intensity (Wm^{-2}) of transmitted and incident radiation, respectively

ε = molar absorptivity (m^2mol^{-1} or $M^{-1}cm^{-1}$) or how strongly a chemical species absorbs light at a given wavelength

b = path length (distance light travels through the chemical species)

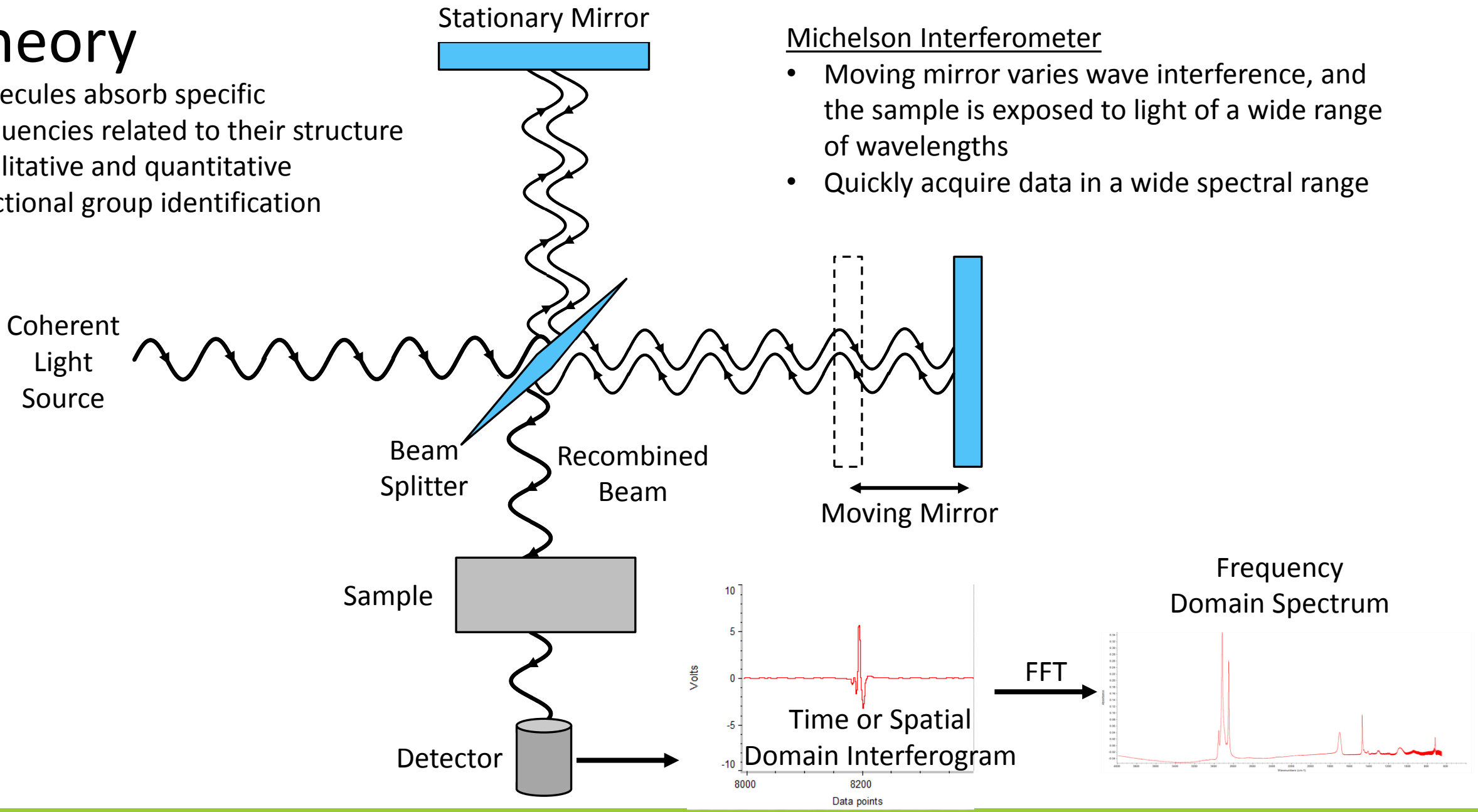
c = molar concentration of chemical species

A = absorbance (relative amount of light absorbed by sample)

- Absorbance is determined in the IR spectrum, and concentration should be known for a liquid sample
- Spacers of accurately determined thickness can be used for path length, and molar absorptivity can then be calculated
- If molar absorptivity and concentration can be determined, the thickness of a film can be calculated by solving for the path length

Theory

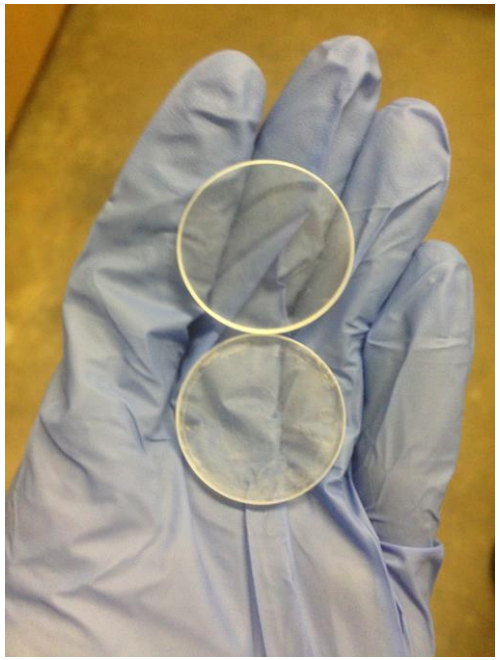
- Molecules absorb specific frequencies related to their structure
- Qualitative and quantitative functional group identification

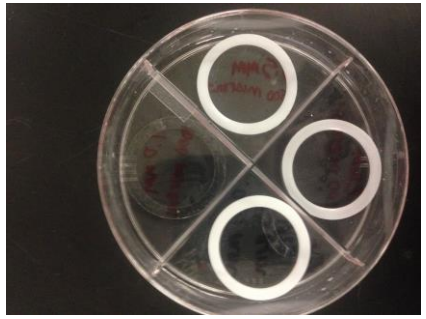


Michelson Interferometer

- Moving mirror varies wave interference, and the sample is exposed to light of a wide range of wavelengths
- Quickly acquire data in a wide spectral range

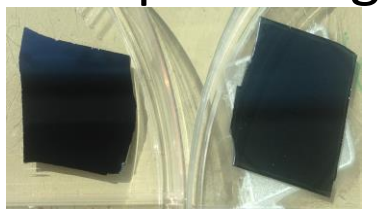
Sample Preparation

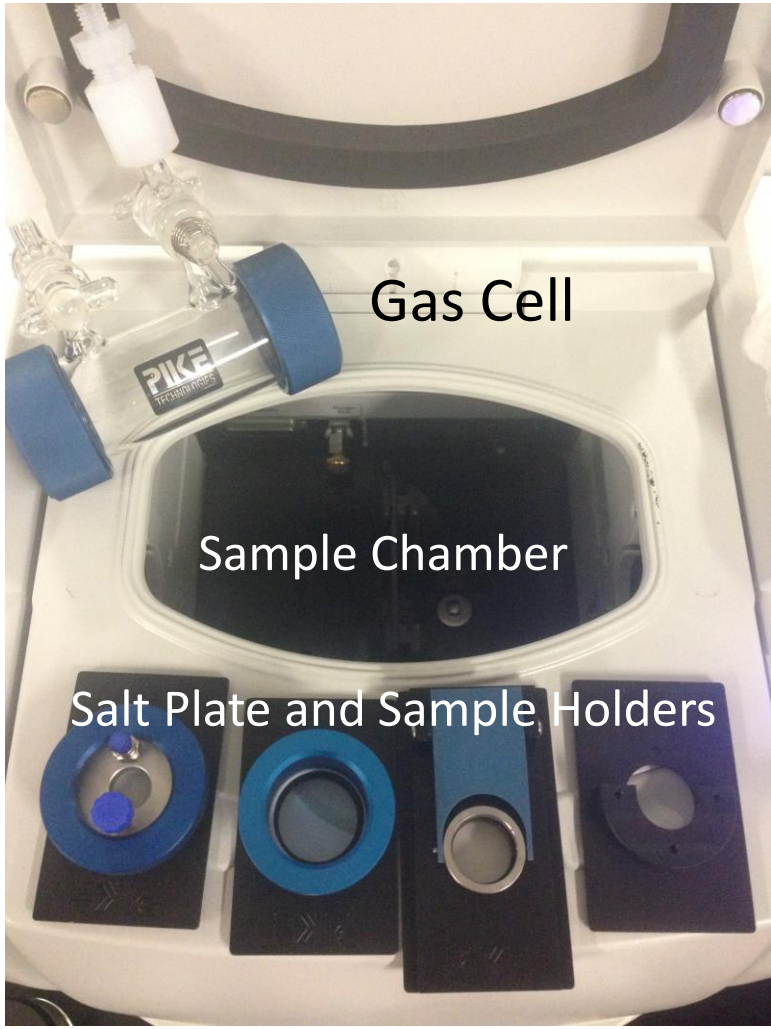




KBr Salt Plates

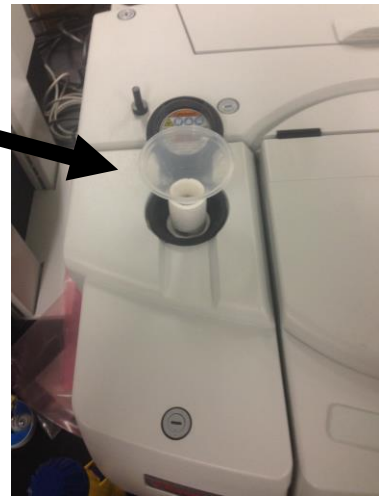
- Handled via edges (fragile) with gloves
- No fingerprints or water exposure, clean copiously with ethanol or acetone
- Collect a background spectrum with clean KBr plates secured in sample holder
- Place a few drops of sample solution on one salt plate, press the plates together, and collect a sample spectrum
- Use a (clean) spacer between the salt plates with sample when the path length must be known

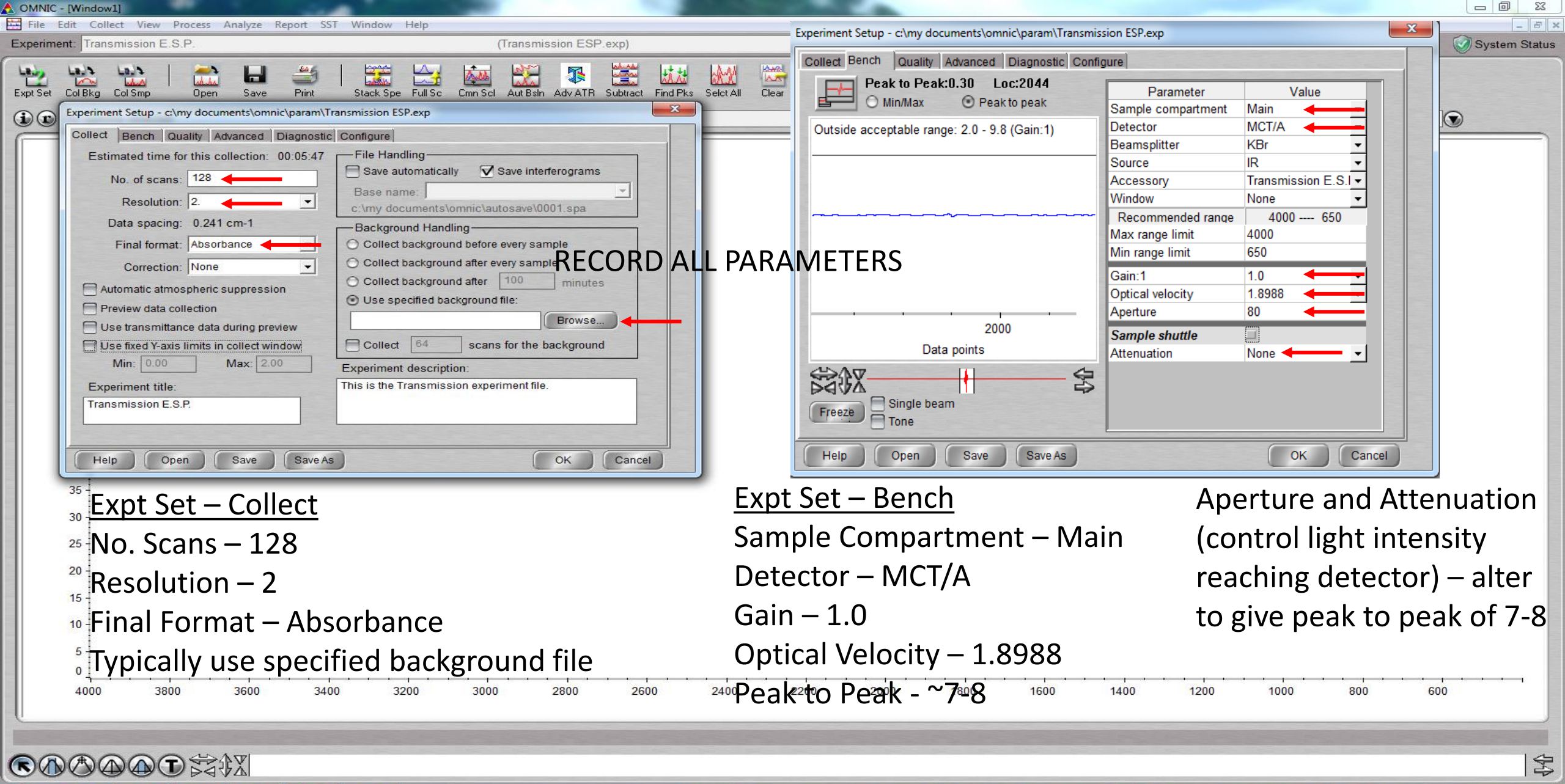




Software Operation

- Must cool detector with liquid nitrogen
- Software – “Omnic”
  - Instrument initializes and attains stable interferogram
  - Instrument will indicate all tests passed
- Save Data – “IR Group Data”
- Experiment – “Transmission E.S.P.”
- Set Parameters – “Expt Set”





RECORD ALL PARAMETERS

Expt Set – Collect

No. Scans – 128

Resolution – 2

Final Format – Absorbance

Typically use specified background file

Expt Set – Bench

Sample Compartment – Main

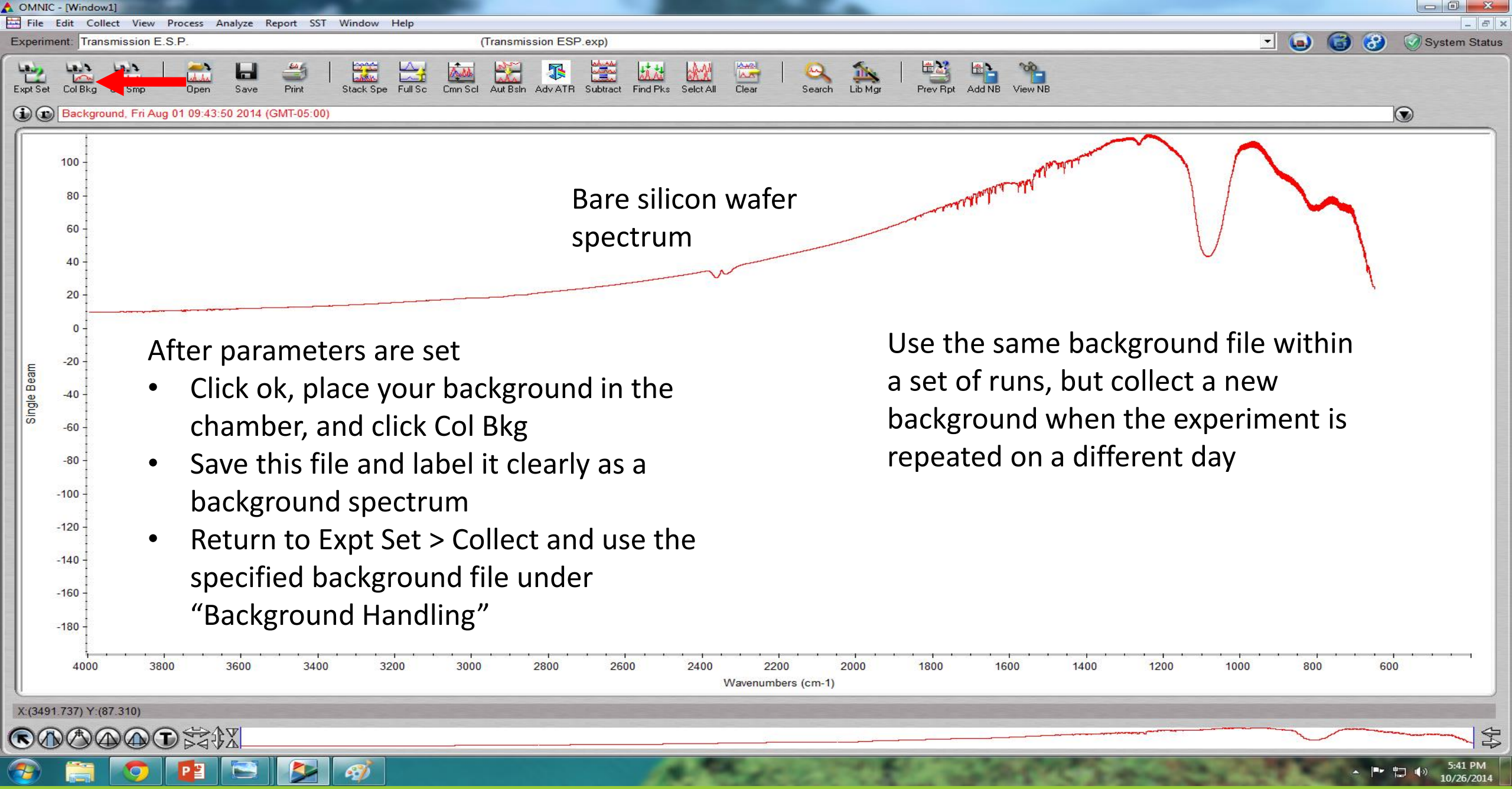
Detector – MCT/A

Gain – 1.0

Optical Velocity – 1.8988

Peak to Peak - ~7-8

Aperture and Attenuation (control light intensity reaching detector) – alter to give peak to peak of 7-8

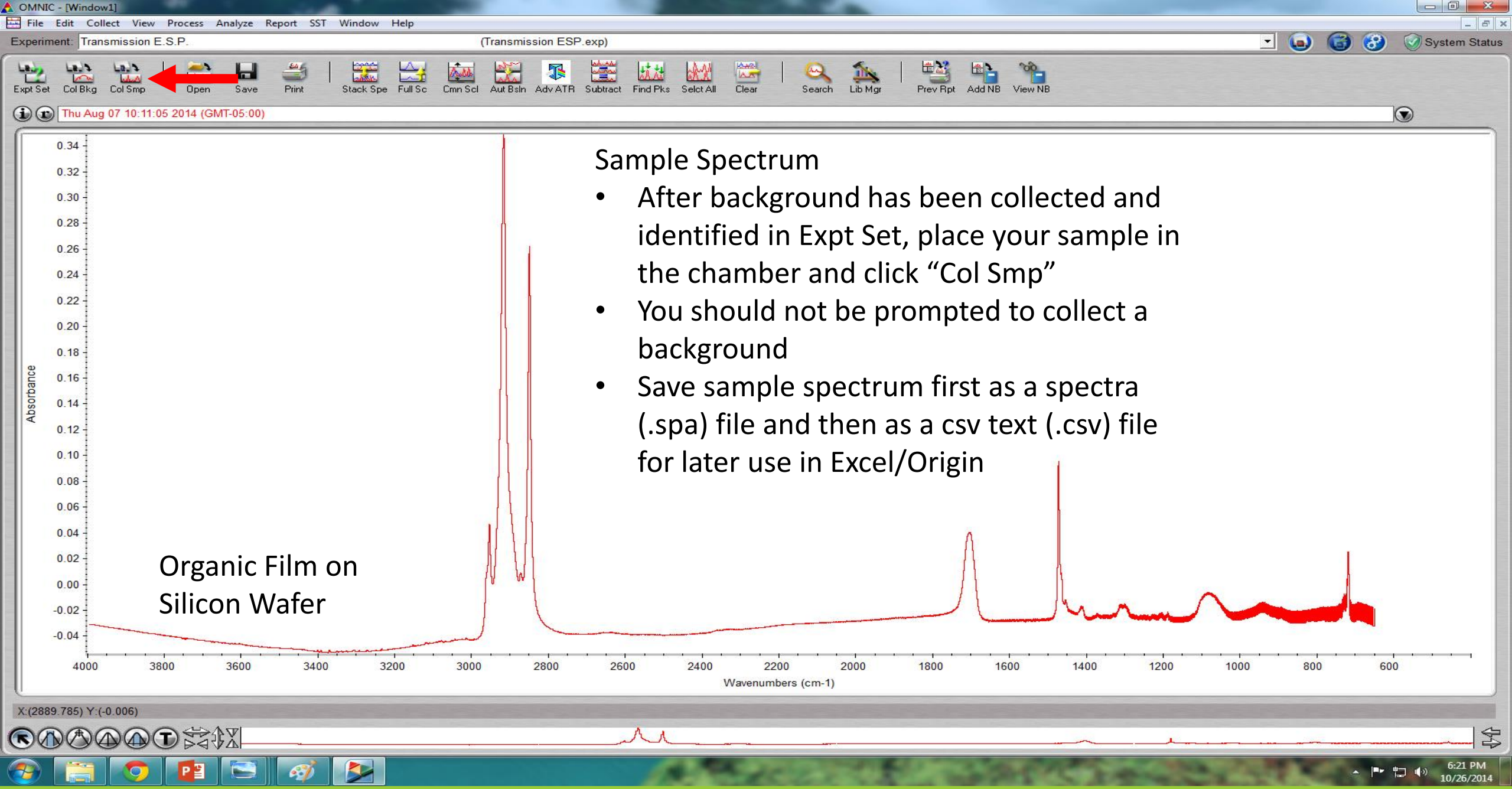


Bare silicon wafer spectrum

After parameters are set

- Click ok, place your background in the chamber, and click Col Bkg
- Save this file and label it clearly as a background spectrum
- Return to Expt Set > Collect and use the specified background file under “Background Handling”

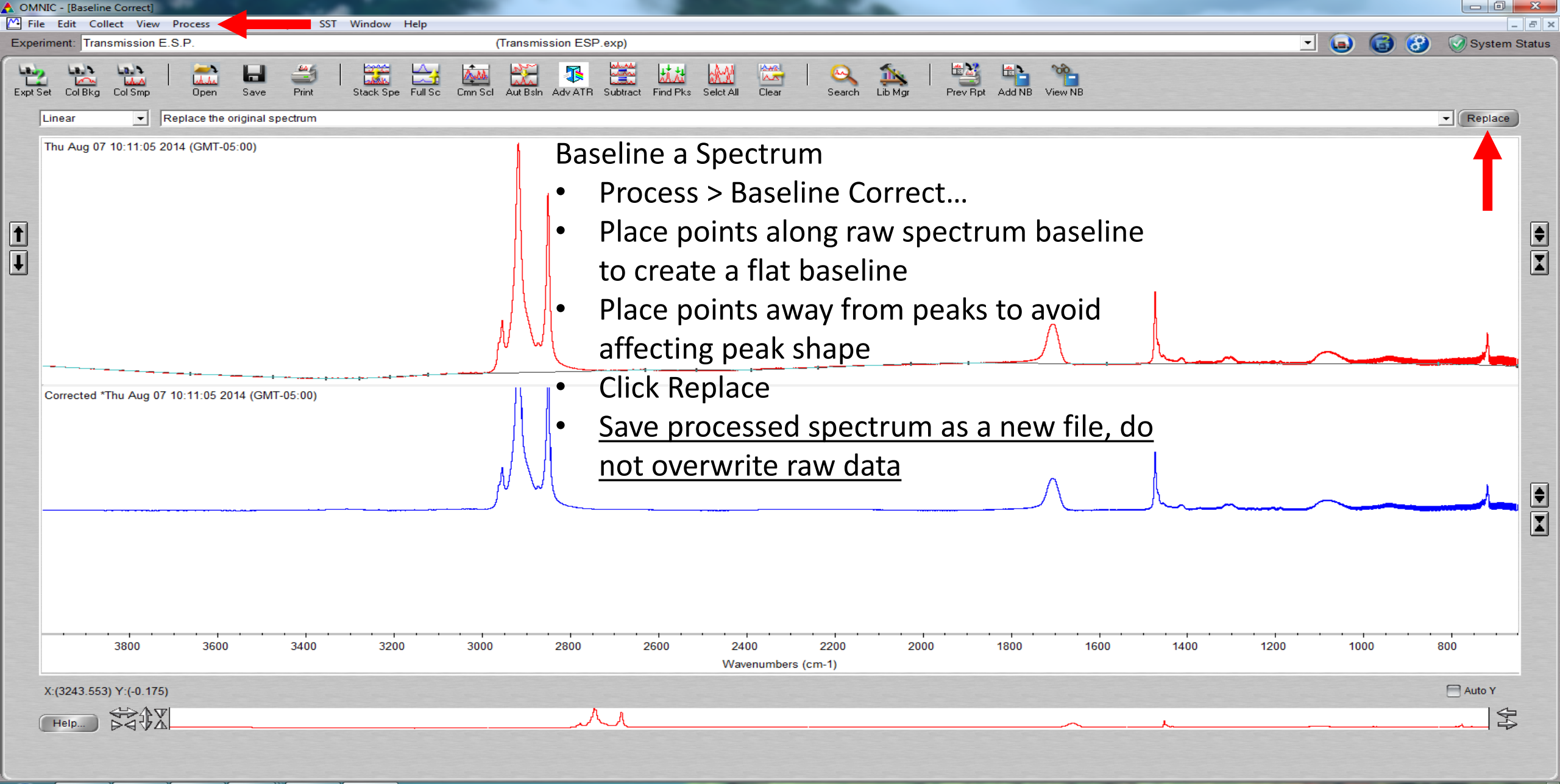
Use the same background file within a set of runs, but collect a new background when the experiment is repeated on a different day



Sample Spectrum

- After background has been collected and identified in Expt Set, place your sample in the chamber and click “Col Smp”
- You should not be prompted to collect a background
- Save sample spectrum first as a spectra (.spa) file and then as a csv text (.csv) file for later use in Excel/Origin

Organic Film on Silicon Wafer


OMNIC - [Baseline Correct]
File Edit Collect View Process SST Window Help
Experiment: Transmission E.S.P. (Transmission ESP.exp)
System Status
Expt Set Col Bkg Col Smp Open Save Print Stack Spe Full Sc Cmn Scl Aut Bsln Adv ATR Subtract Find Pks Selct All Clear Search Lib Mgr Prev Rpt Add NB View NB
Linear
Replace the original spectrum
Replace
Thu Aug 07 10:11:05 2014 (GMT-05:00)
Corrected *Thu Aug 07 10:11:05 2014 (GMT-05:00)
3800 3600 3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800
Wavenumbers (cm-1)
X:(3243.553) Y:(-0.175)
Auto Y
Help...
Baseline a Spectrum
• Process > Baseline Correct…
• Place points along raw spectrum baseline to create a flat baseline
• Place points away from peaks to avoid affecting peak shape
• Click Replace
• Save processed spectrum as a new file, do not overwrite raw data
6:32 PM
10/26/2014

Further Processing

With the CSV files, spectra can be opened in Origin for thorough peak fitting and analysis