

How-To: Fourier Transform Infrared Spectroscopy (FTIR)

Jacob Grant

Professor Scott Shaw Research Group

Group Meeting 141027



Beer-Lambert Law

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

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

$$A = \epsilon bc$$

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

I, I₀ = intensity (Wm⁻²) of transmitted and incident radiation, respectively

ε = molar absorptivity (m²mol⁻¹ or M⁻¹cm⁻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)

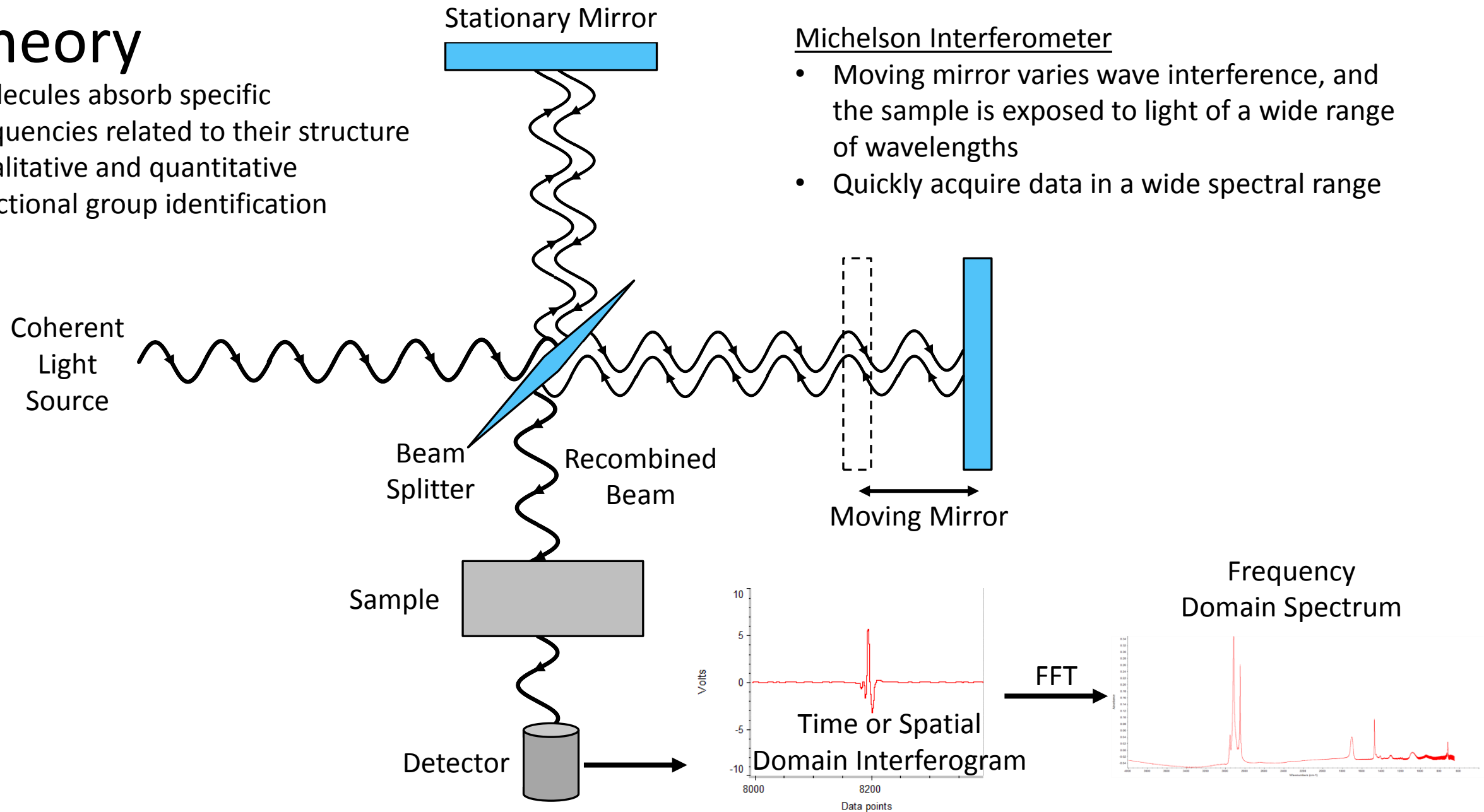
- Absorbance is determined in the IR spectrum, and concentration should be known for a liquid sample
- Spacers of accurately determined thickness can be used for path length, and molar absorptivity can then be calculated
- If molar absorptivity and concentration can be determined, the thickness of a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

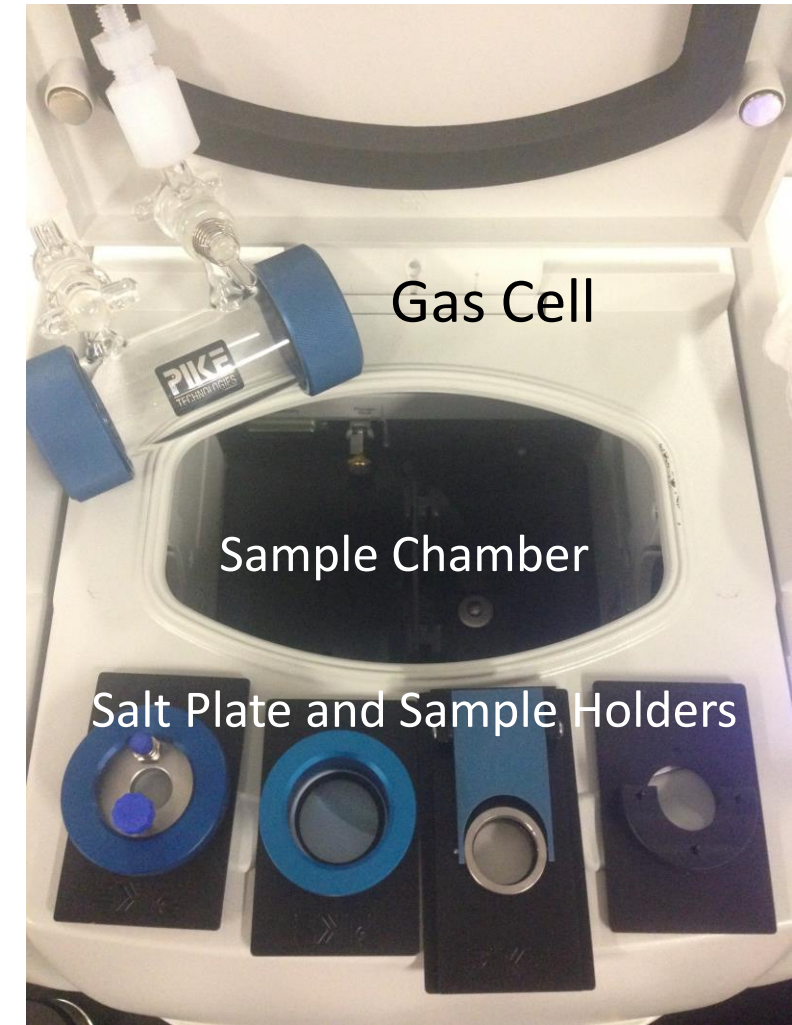
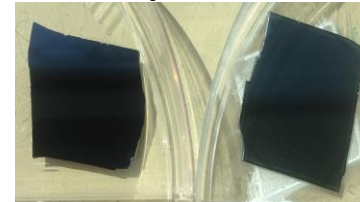
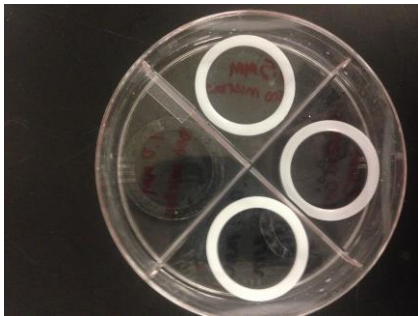
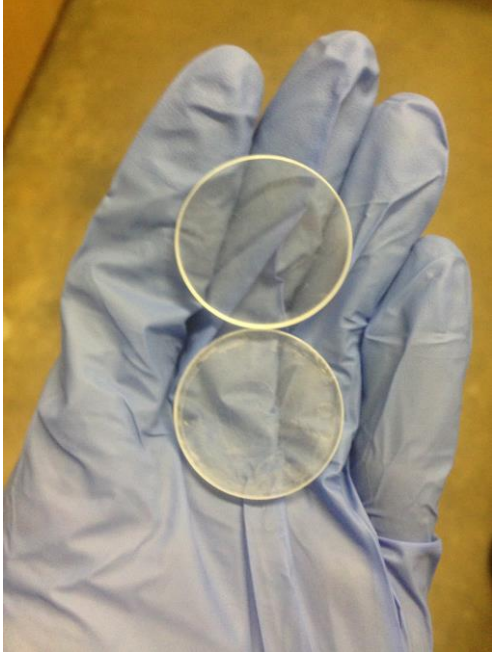
- Moving mirror varies wave interference, and the sample is exposed to light of a wide range of wavelengths
- Quickly acquire data in a wide spectral range



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



OMNIC - [Window1]

File Edit Collect View Process Analyze Report SST Window Help

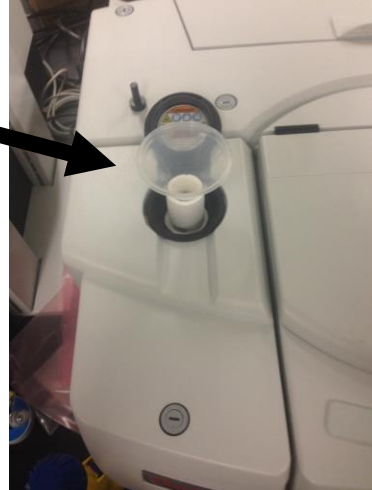
Experiment: Transmission E.S.P. (Transmission ESP.exp)

Expt Set Col Bkg Col Smp Open Save Print Stack Spe Full Sc Cmn Scl Aut Bsln Adv ATR Subtract Find Pks Sclt All Clear Search Lib Mgr Prev Rpt Add NB View NB

No spectra selected

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”



OMNIC - [Window1]
 File Edit Collect View Process Analyze Report SST Window Help
 Experiment: Transmission E.S.P. (Transmission ESP.exp)

Experiment Setup - c:\my documents\omnic\param\Transmission ESP.exp

Collect Bench Quality Advanced Diagnostic Configure

Estimated time for this collection: 00:05:47

No. of scans: 128
 Resolution: 2
 Data spacing: 0.241 cm⁻¹
 Final format: Absorbance
 Correction: None

Automatic atmospheric suppression
 Preview data collection
 Use transmittance data during preview
 Use fixed Y-axis limits in collect window
 Min: 0.00 Max: 2.00

Experiment title:
 Transmission E.S.P.

File Handling
 Save automatically Save interferograms
 Base name:
 c:\my documents\omnic\autosave\0001.spa

Background Handling
 Collect background before every sample
 Collect background after every sample
 Collect background after 100 minutes
 Use specified background file:
 Browse...
 Collect 64 scans for the background

Experiment description:
 This is the Transmission experiment file.

Help Open Save Save As OK Cancel

RECORD ALL PARAMETERS

Experiment Setup - c:\my documents\omnic\param\Transmission ESP.exp

Collect Bench Quality Advanced Diagnostic Configure

Peak to Peak: 0.30 Loc: 2044
 Min/Max Peak to peak

Outside acceptable range: 2.0 - 9.8 (Gain: 1)

2000
 Data points

Freeze Single beam Tone

Help Open Save Save As OK Cancel

Parameter	Value
Sample compartment	Main
Detector	MCT/A
Beamsplitter	KBr
Source	IR
Accessory	Transmission E.S.1
Window	None
Recommended range	4000 ---- 650
Max range limit	4000
Min range limit	650
Gain:1	1.0
Optical velocity	1.8988
Aperture	80
Sample shuttle	<input type="checkbox"/>
Attenuation	None

4000 3800 3600 3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600

35
30
25
20
15
10
5
0

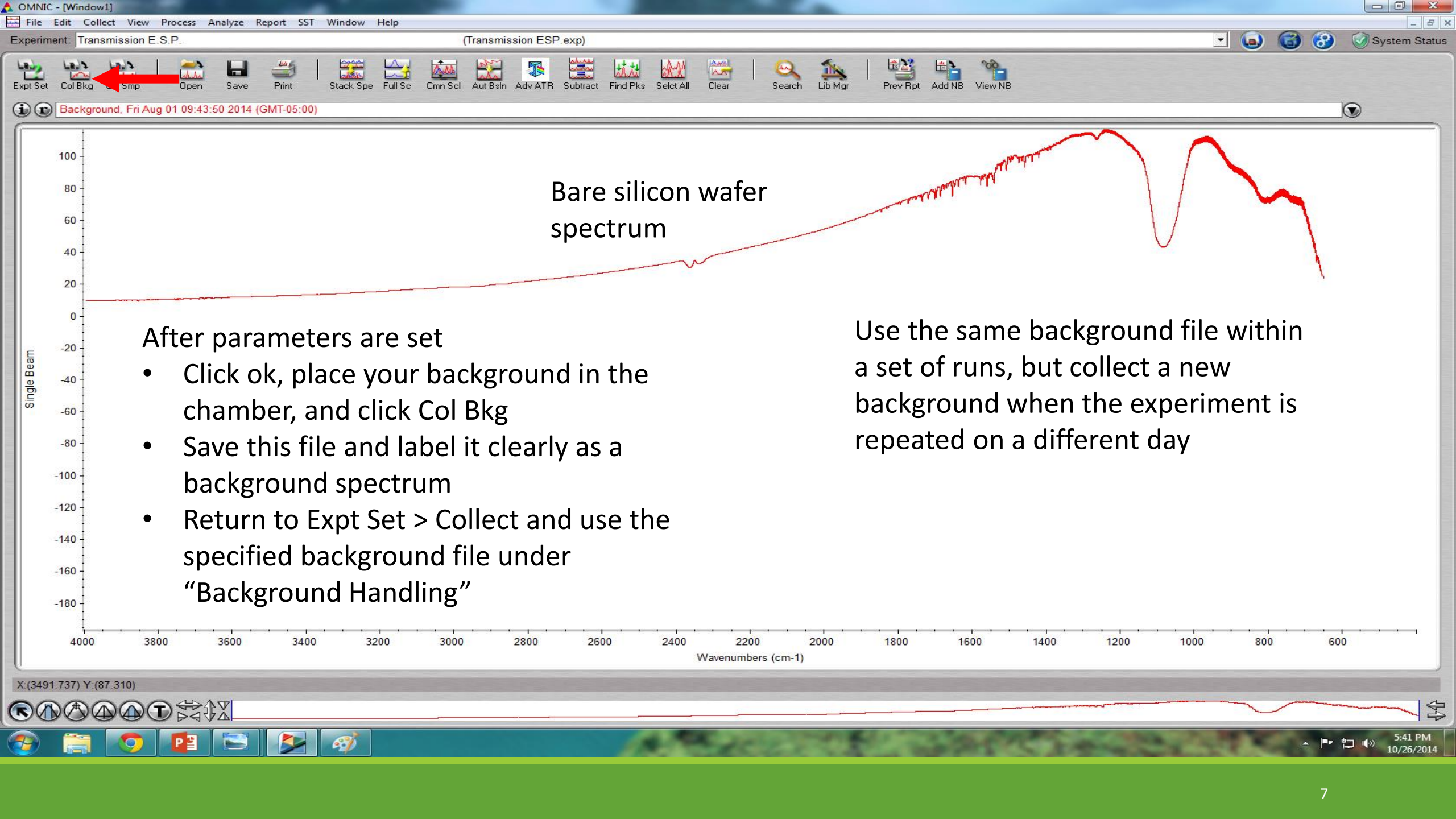
Expt Set – Collect
 No. Scans – 128
 Resolution – 2
 Final Format – Absorbance
 Typically use specified background file

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

System Status

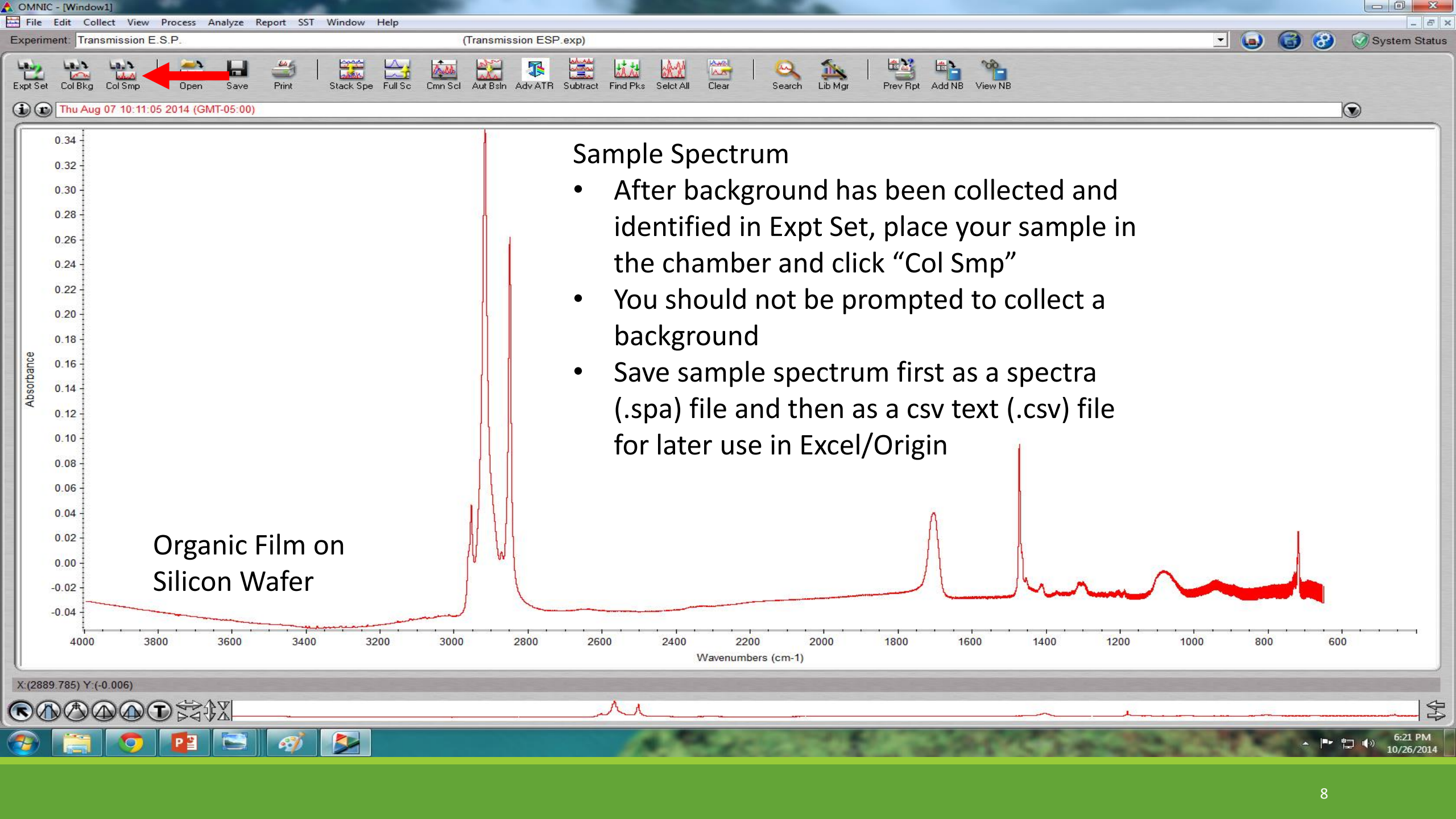
5:20 PM
 10/26/2014



After parameters are set

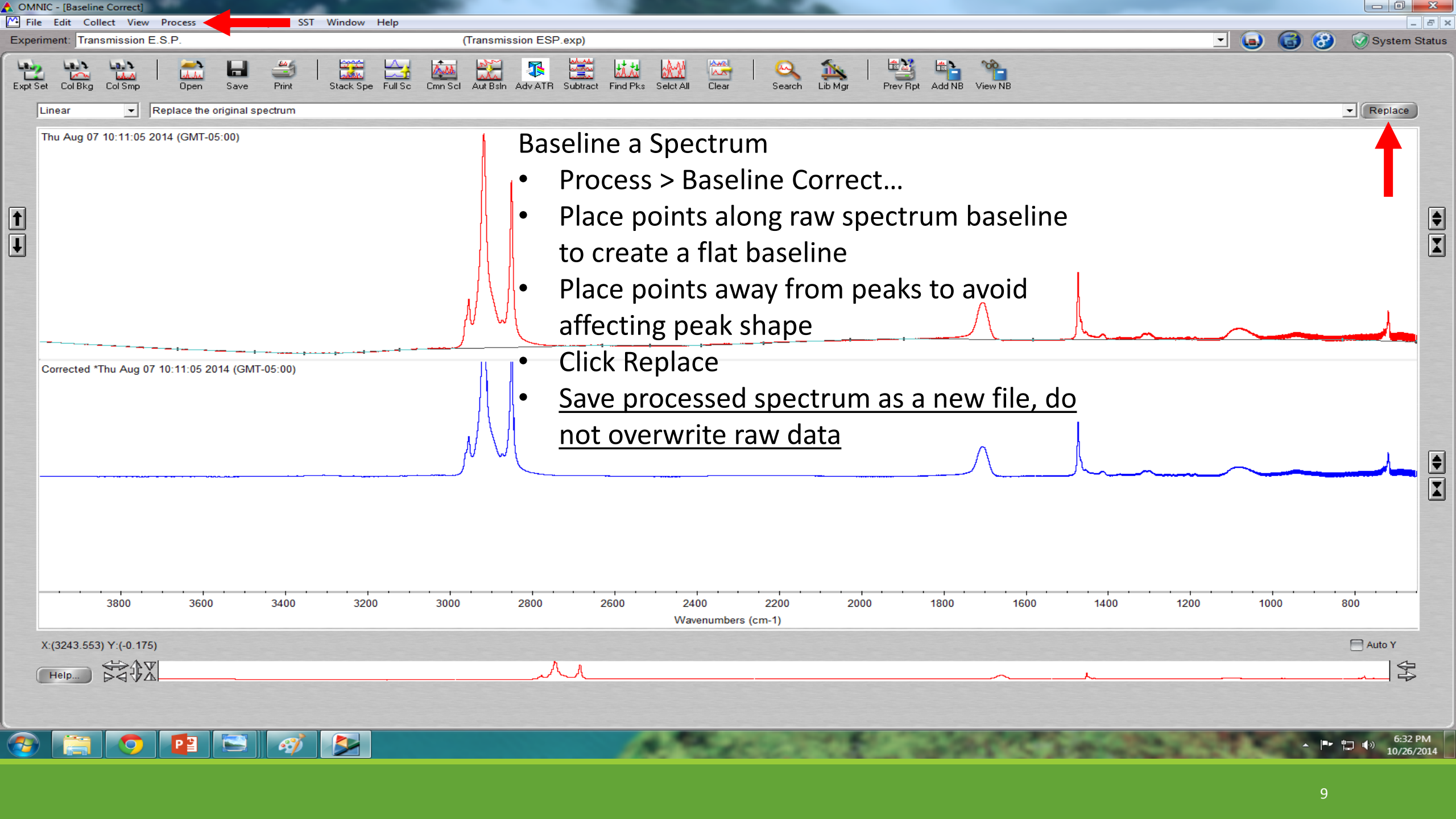
- Click ok, place your background in the chamber, and click Col Bkg
- Save this file and label it clearly as a background spectrum
- Return to Expt Set > Collect and use the specified background file under "Background Handling"

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



Process

Experiment: Transmission E.S.P.

(Transmission ESP.exp)

System Status



Linear Replace the original spectrum Replace

Thu Aug 07 10:11:05 2014 (GMT-05:00)

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

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)

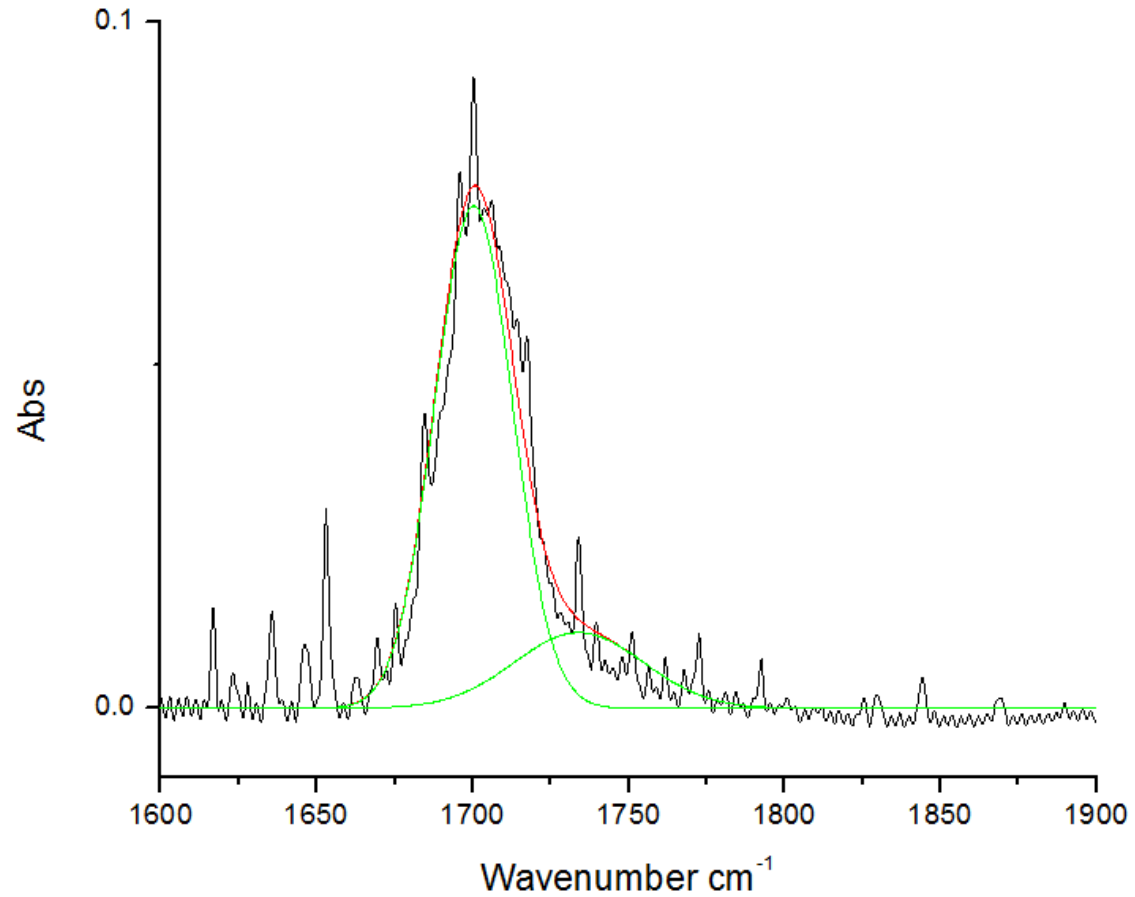
X:(3243.553) Y:(-0.175)

Auto Y



Further Processing

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



The dialog box shows the following parameters:

Parameter	Value	Vary?	Error	Dependency
y0	0	<input type="checkbox"/>	± 0	0
xc1	1700.32841	<input type="checkbox"/>	± 0	0
w1	24.93709	<input checked="" type="checkbox"/>	± 1.52618	0.44823
A1	2.28822	<input checked="" type="checkbox"/>	± 0.14731	0.626

Iteration history:

```
Reduced Chi-sqr = 0.00076
Total 119 rounds in this session
(123)-----Levenberg-Marquardt-----
Successfully progressed 1 rounds.
Reduced Chi-sqr = 0.00076
Total 120 rounds in this session
(124)-----Levenberg-Marquardt-----
Successfully progressed 1 rounds.
Reduced Chi-sqr = 0.00076
Total 121 rounds in this session
(125)-----Levenberg-Marquardt-----
Successfully progressed 1 rounds.
Reduced Chi-sqr = 0.00076
Total 122 rounds in this session
```

Buttons: Chi-Sqr, 1 Iter., 10 Iter., 10 Simplex Iter., Done, Basic Mode