How to: ATR spectroscopy

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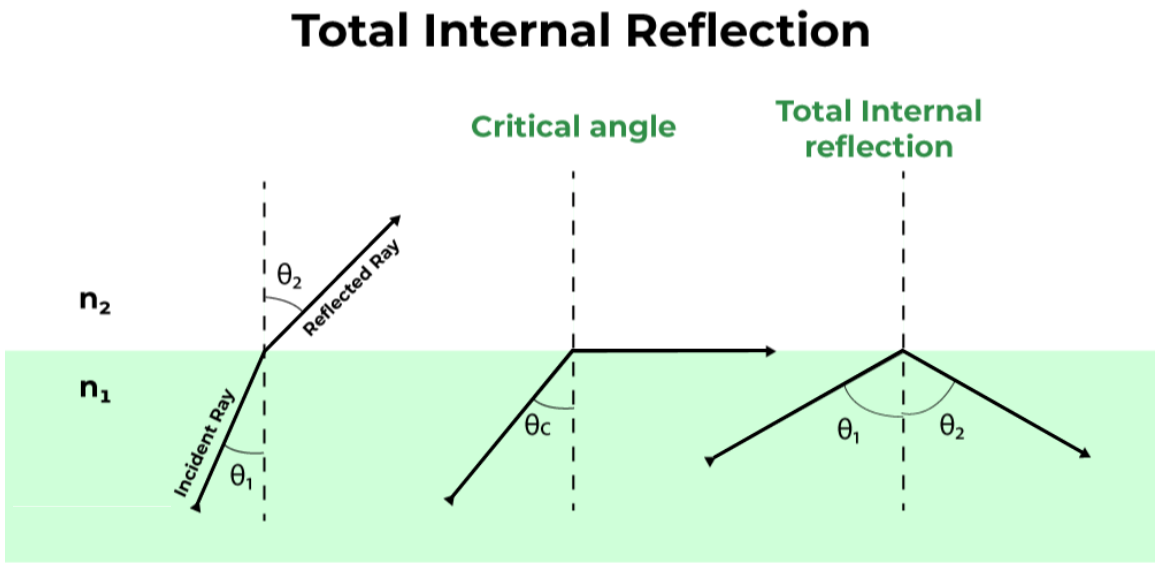
Jan 24, 2024

Thermo IS50 Advanced FTIR in our lab

Attenuated Total Reflection (ATR) Spectroscopy



Snell's law

$\theta_c = \sin^{-1}(n_2/n_1)$

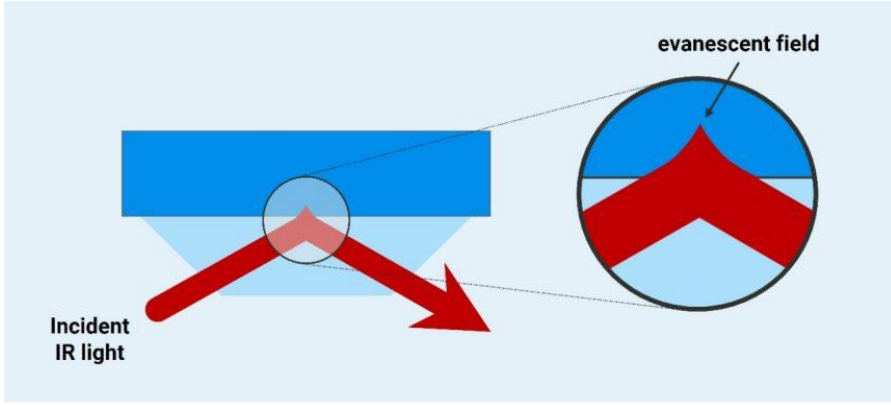


Table 1 Common materials for ATR crystals; critical angle calculated for n2=1.5; table taken from Griffiths P.R., de Haseth J. A. (2007). Fourier Transform Infrared Spectrometry. New York, USA: John Wiley &Sons.

Cell material	Refractive index	Critical angle (degrees)
Silver chloride AgCl	1.90	49°
KRS-5	2.37	40°
Zinc selenide (ZnSe)	2.40	40°
Zinc sulfide (ZnS)	2.2	43°
Germanium (Ge)	4.00	22°
Silicon (Si)	3.41	26°
Diamond	2.41	40°

Crystals have different refractive indexes as seen in the table, and most organic based samples are around 1.5

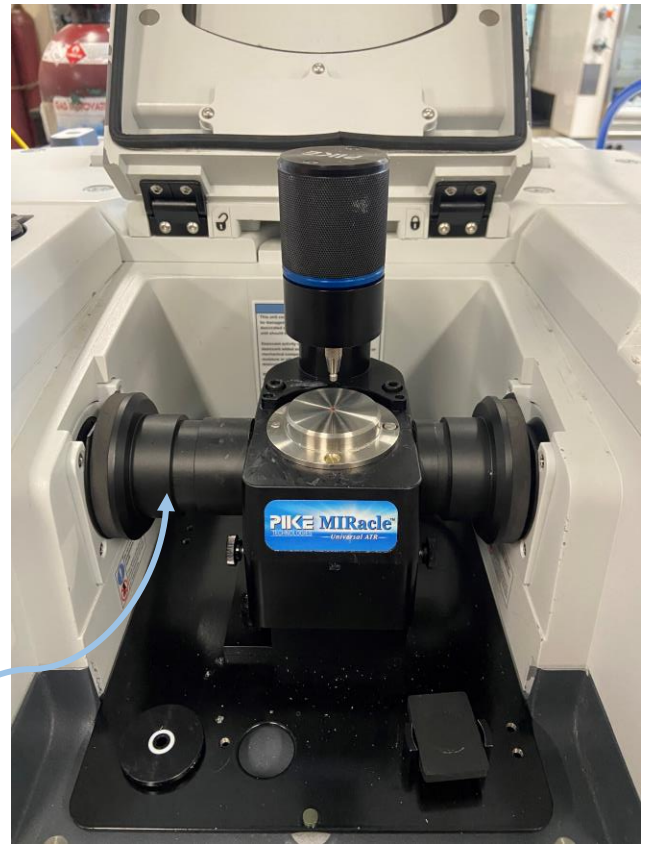
ATR has replaced a lot of transmission experiments, it requires little to no sample prep, can be done on a variety of samples, and can give same information as transmission IR spectroscopy.

https://www.geeksforgeeks.org/total-internal-reflection/

https://www.labcompare.com/10-Featured-Articles/352695-Pathlength-Considerations-With-ATR-Sampling-in-FTIR/

https://www.bruker.com/en/products-and-solutions/infrared-and-raman/ft-ir-routine-spectrometer/what-is-ft-ir-spectroscopy/atr-attenuated-total-reflectance.html

Thermo IS50 Advanced FTIR in our lab



Screws on 4 corners of baseplate to mount attachment into instrument. Adjustable to connect two sides of attachment.

There are other How To slides on the group website

Hov
Sp

Polarization Modulation Infrared Reflectance Absorption Spectroscopy(PMIRRAS)

18

Samantha Nania

Shaw Group Meeting

140908 How it Works

THE UNIVERSITY OF IOWA

Jacob Grant

Professor Scott Shaw Research Group

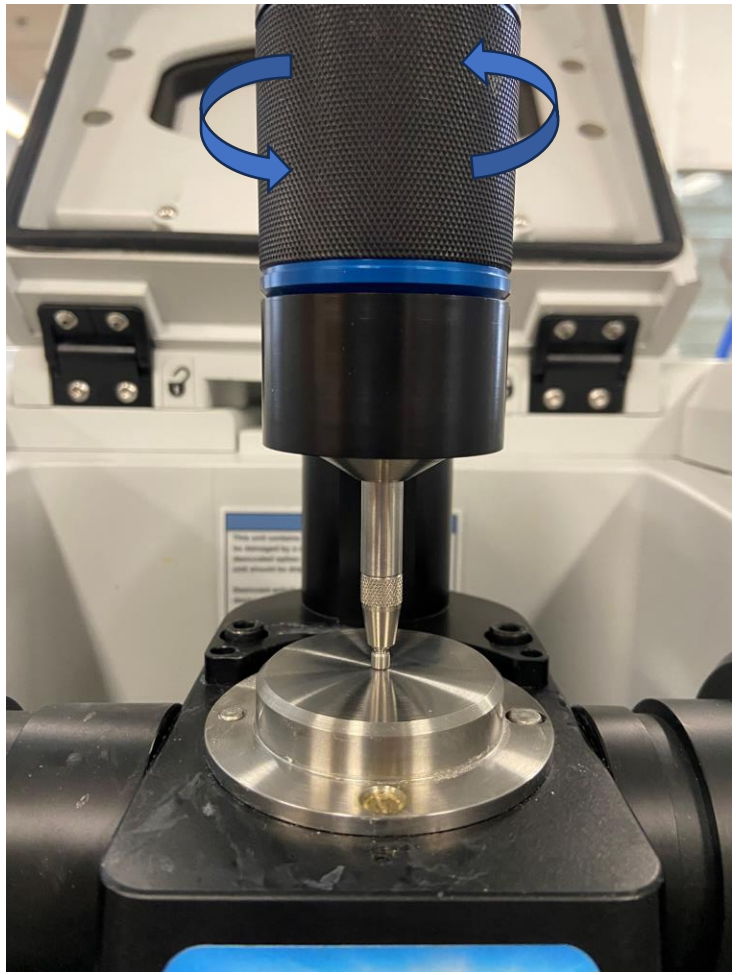
Group Meeting 141027

Turn on the instrument and gather supplies

Ethanol is used to clean the ATR crystal.

100 microliters is the typical liquid samples size.

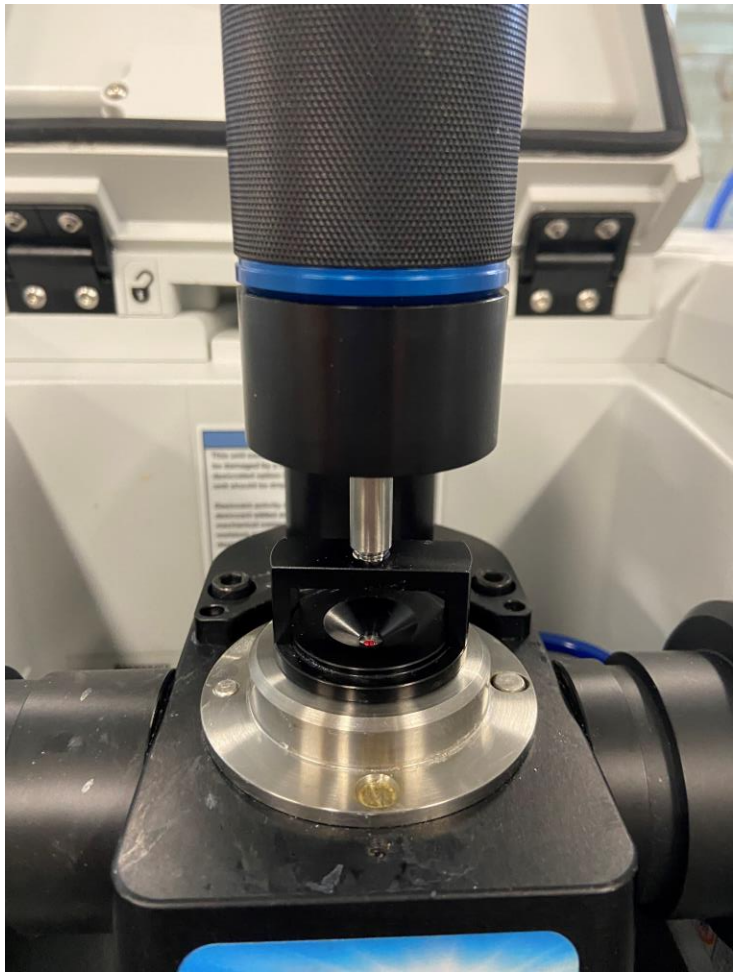
Chose your sample holder



Solid samples:

Small amount of sample needed.

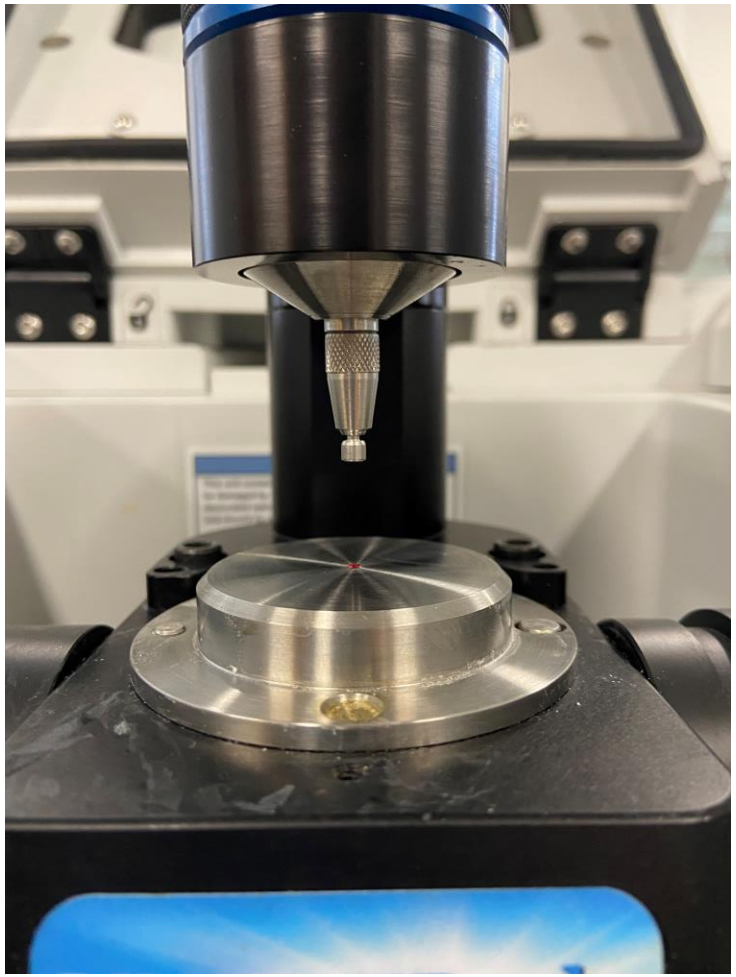
Should probably be tested if it dissolves in ethanol to prevent contamination of future samples

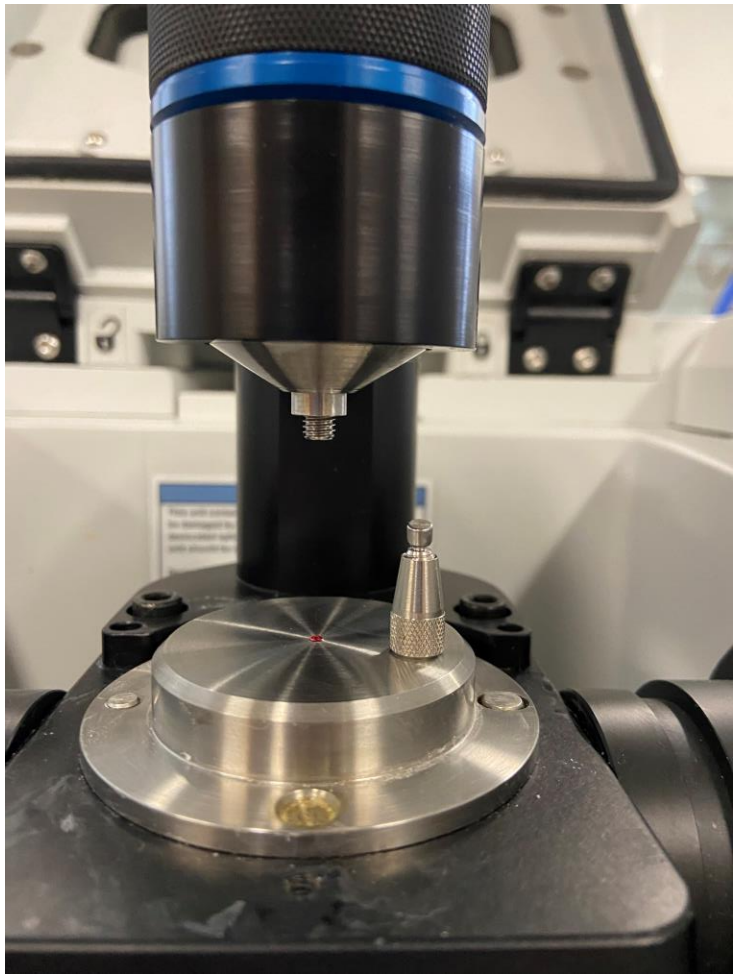


Liquid samples:

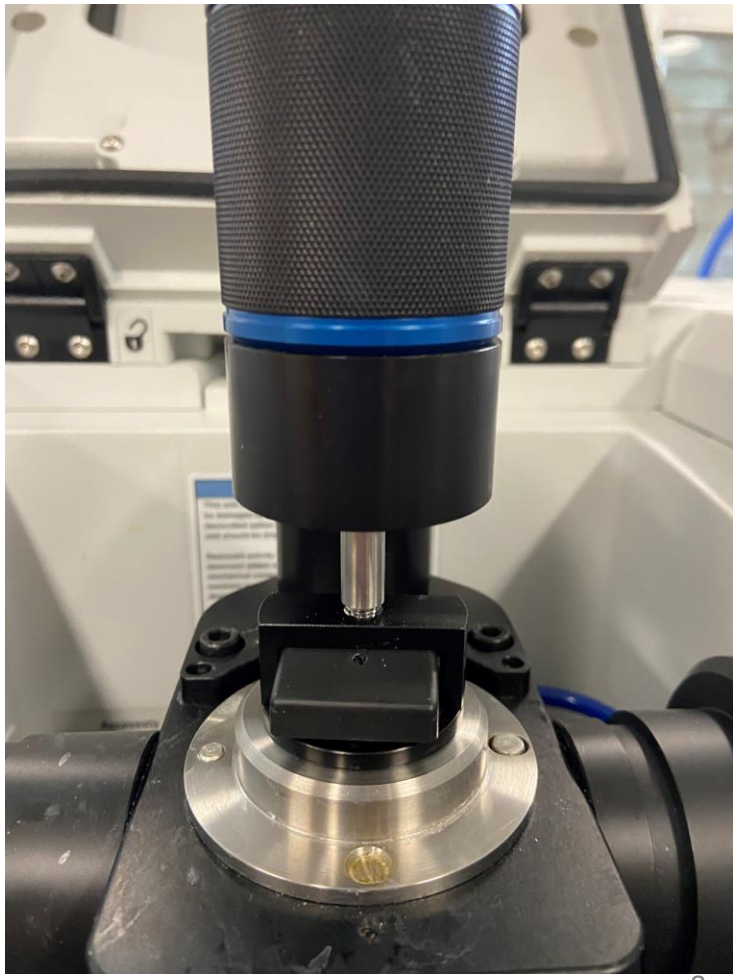
~100 microliters

How to change between solid and liquid samples

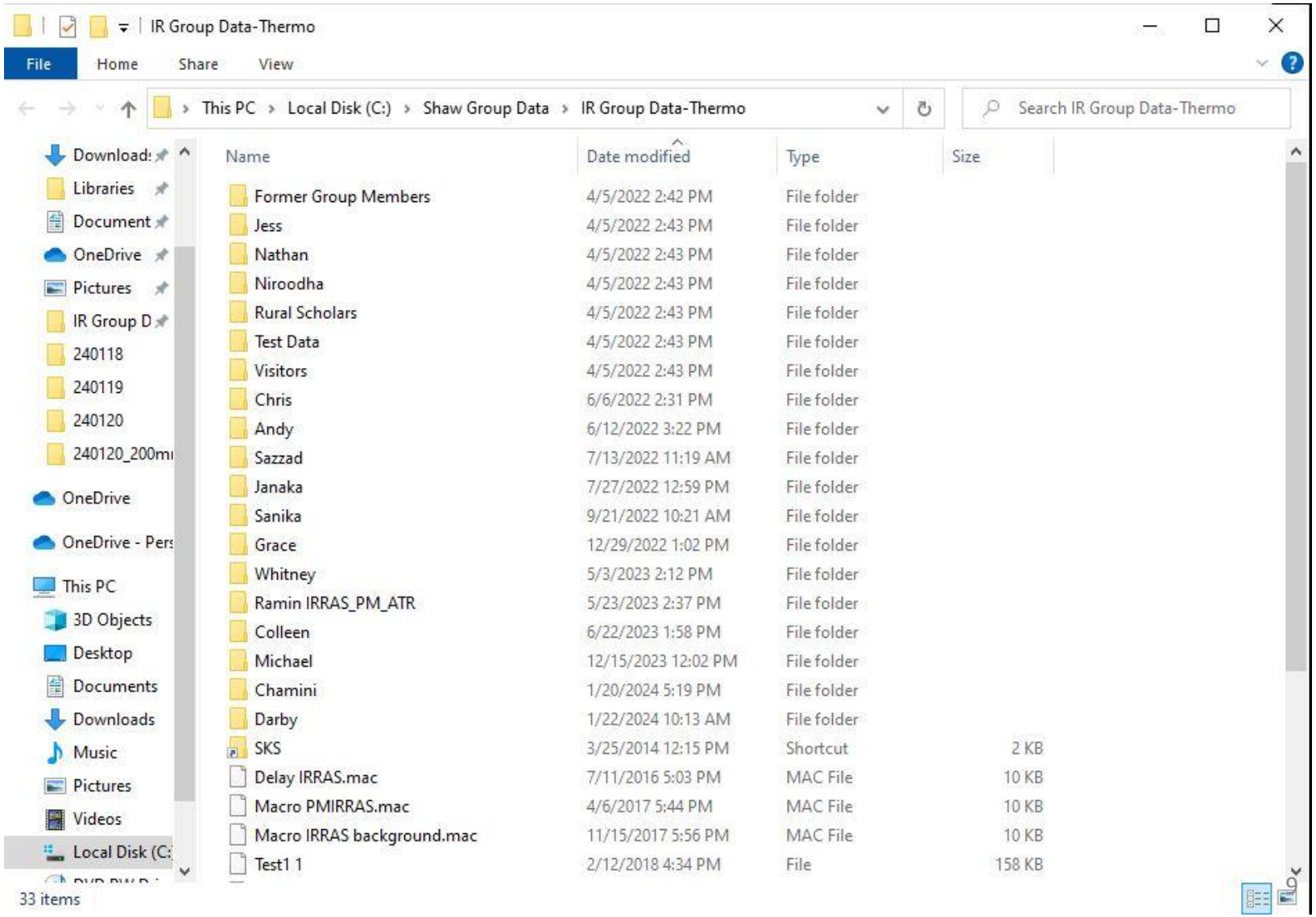




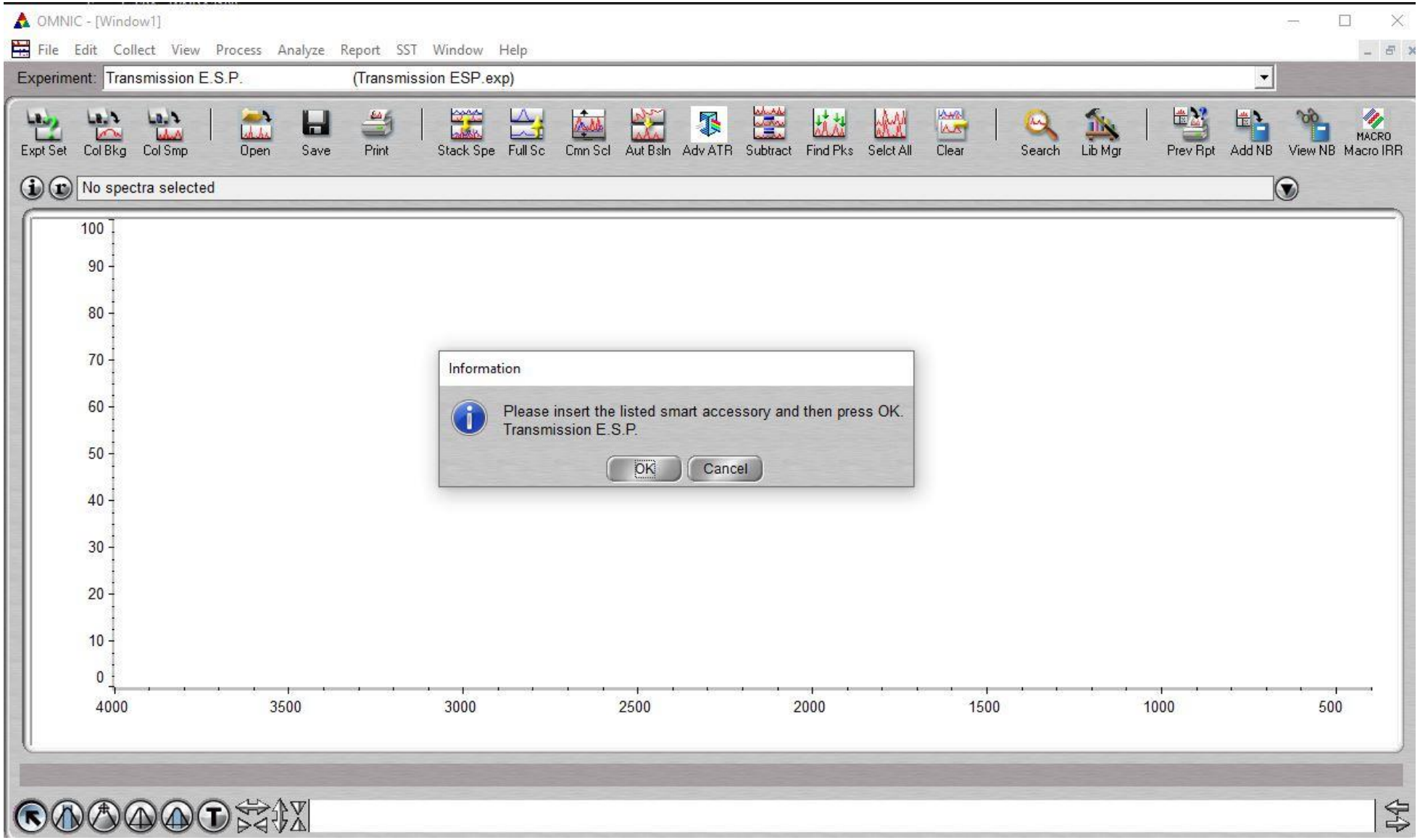
Cap helps prevent solvent evaporation



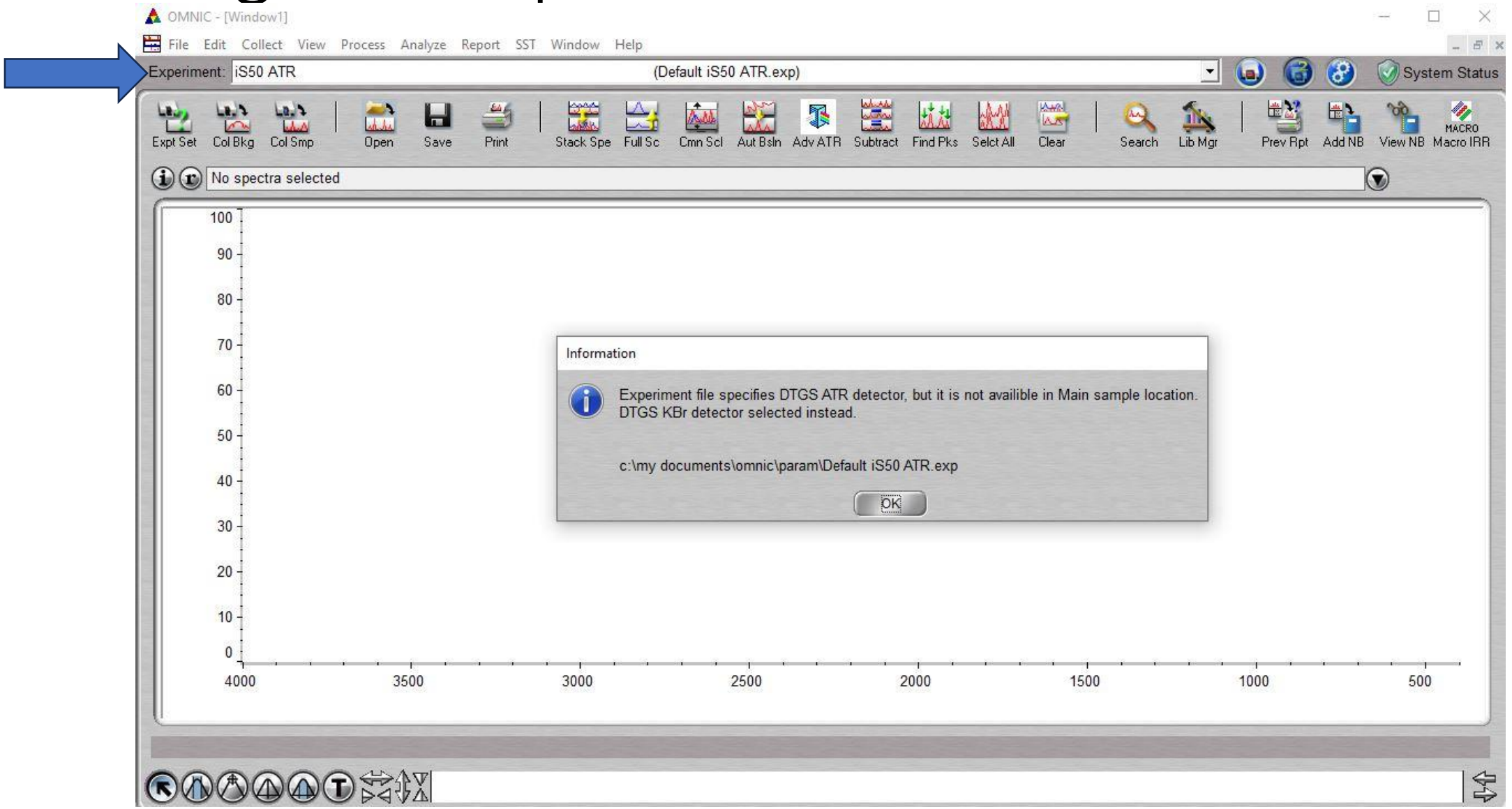
Software and where to save



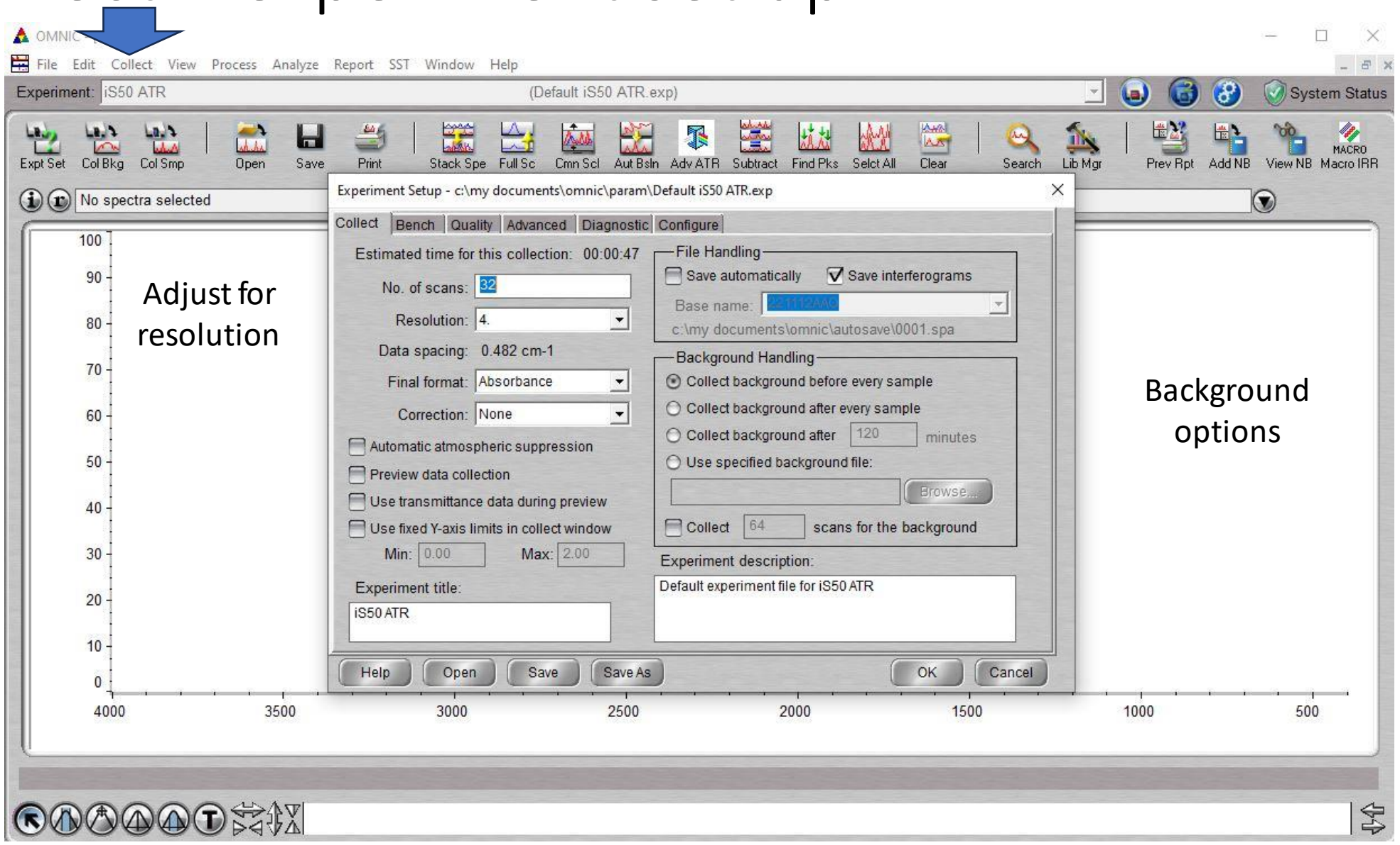
When you open OMNIC- transmission is default



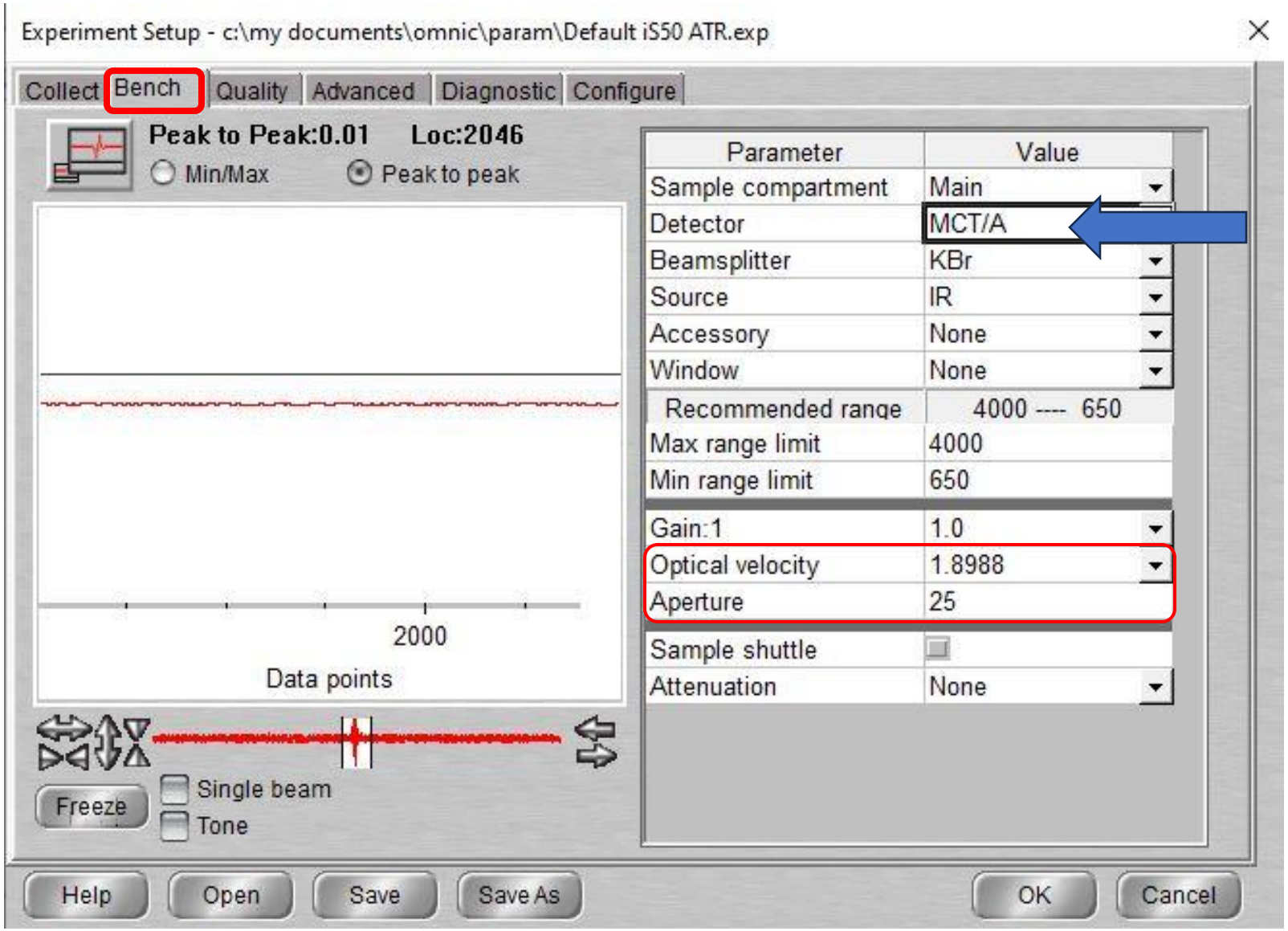
Change the experiment to iS50 ATR



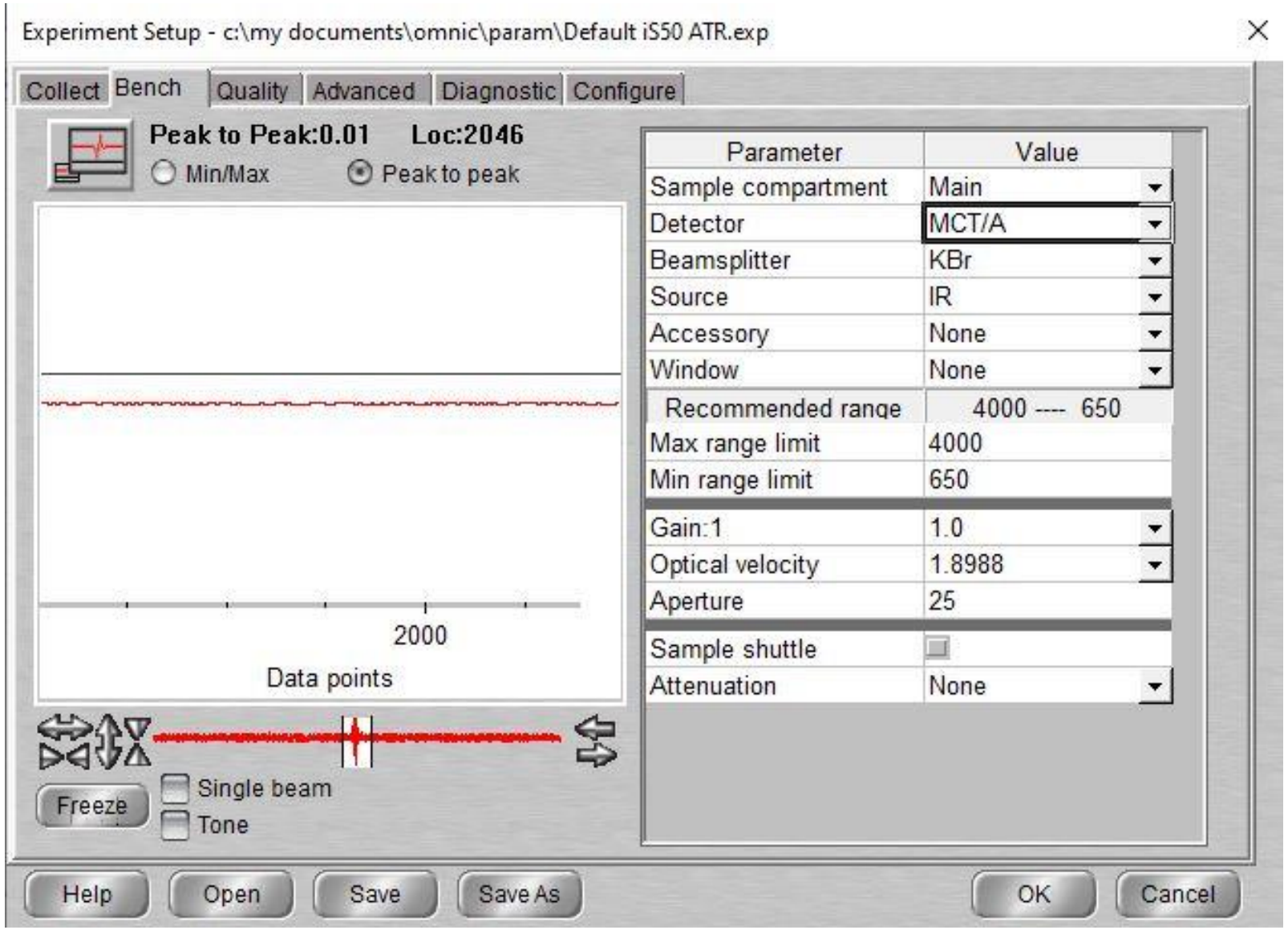
Collect > experiment set up



Bench tab- peak to peak

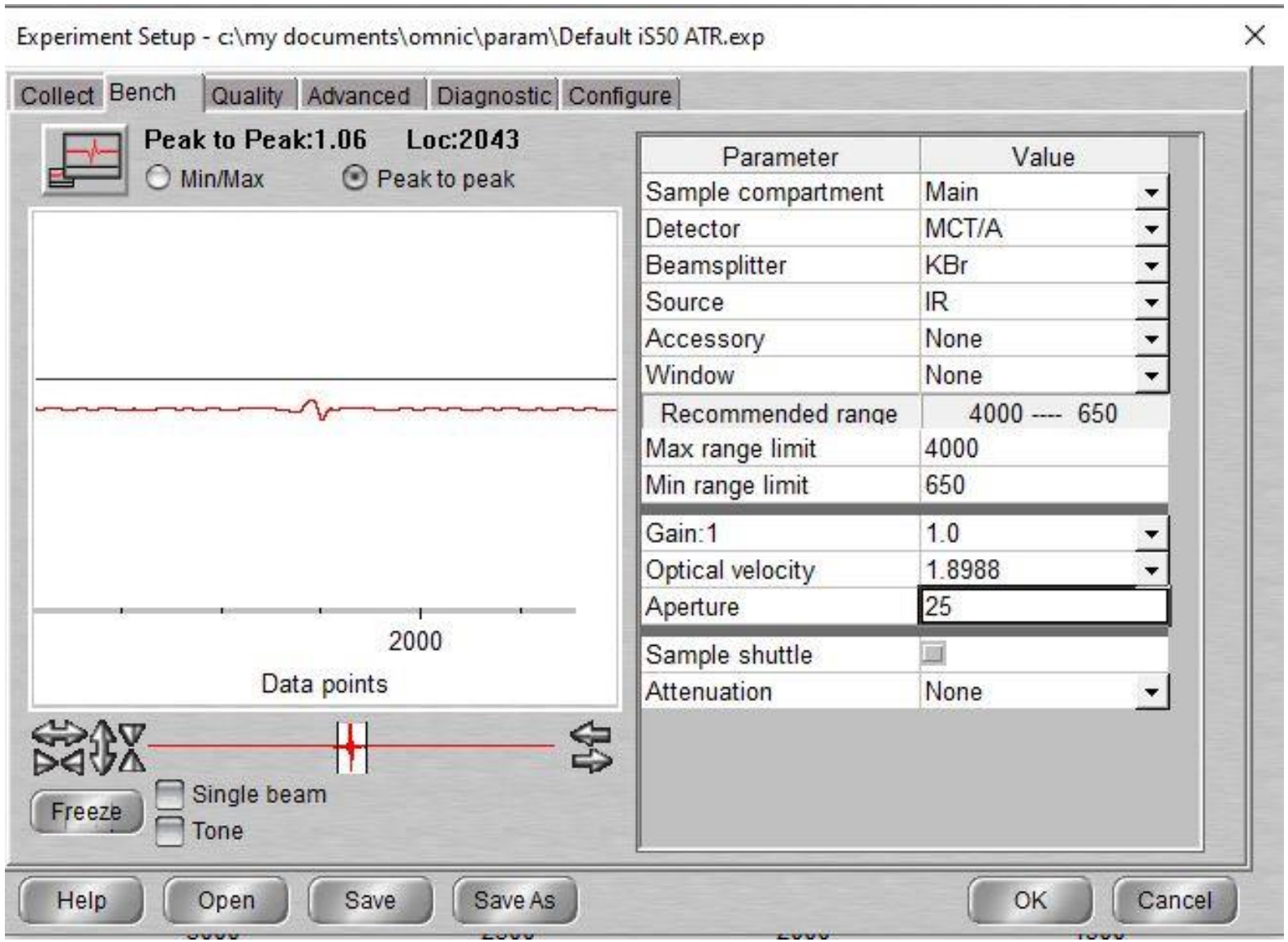


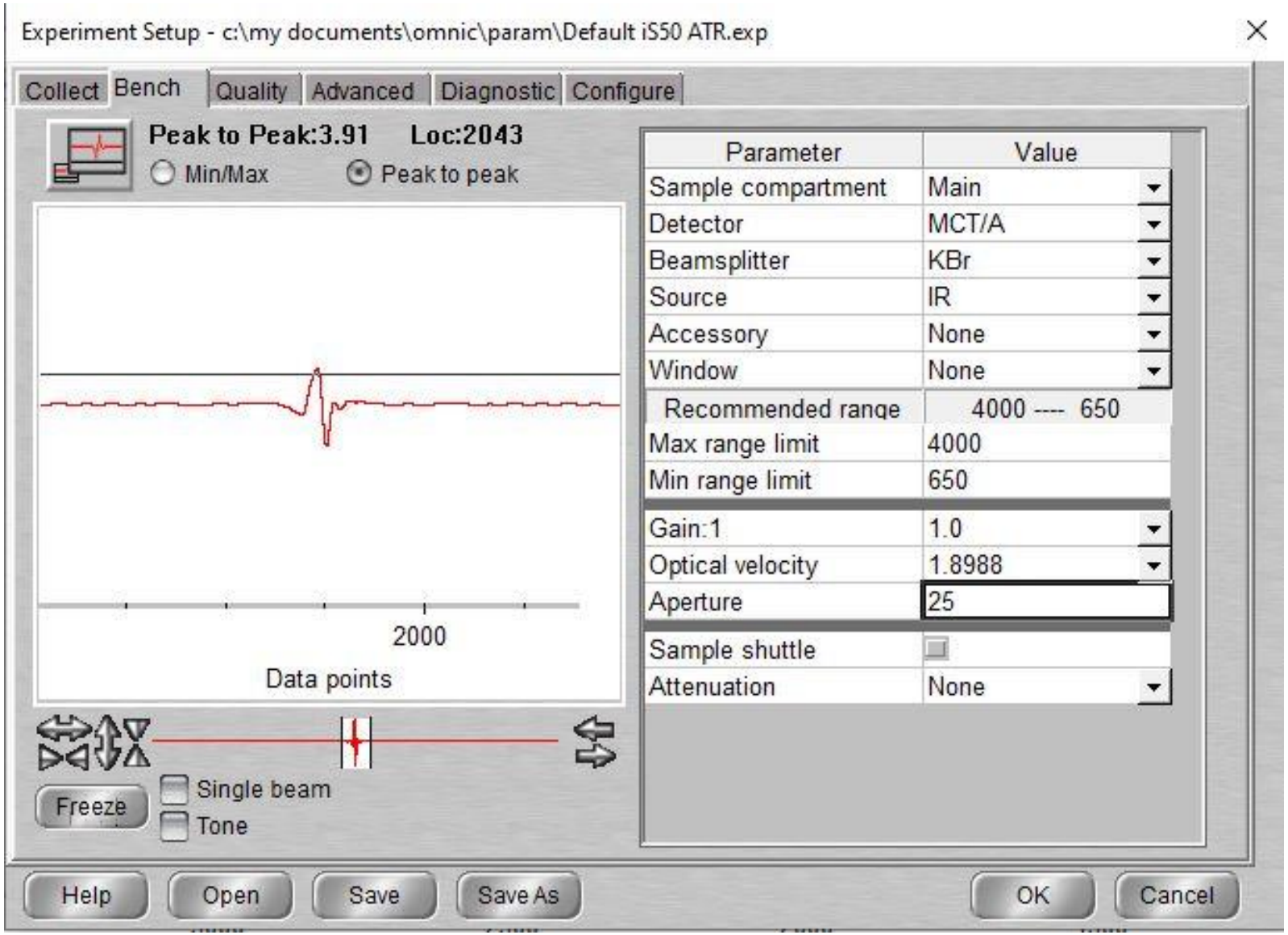
Change to MCT/A

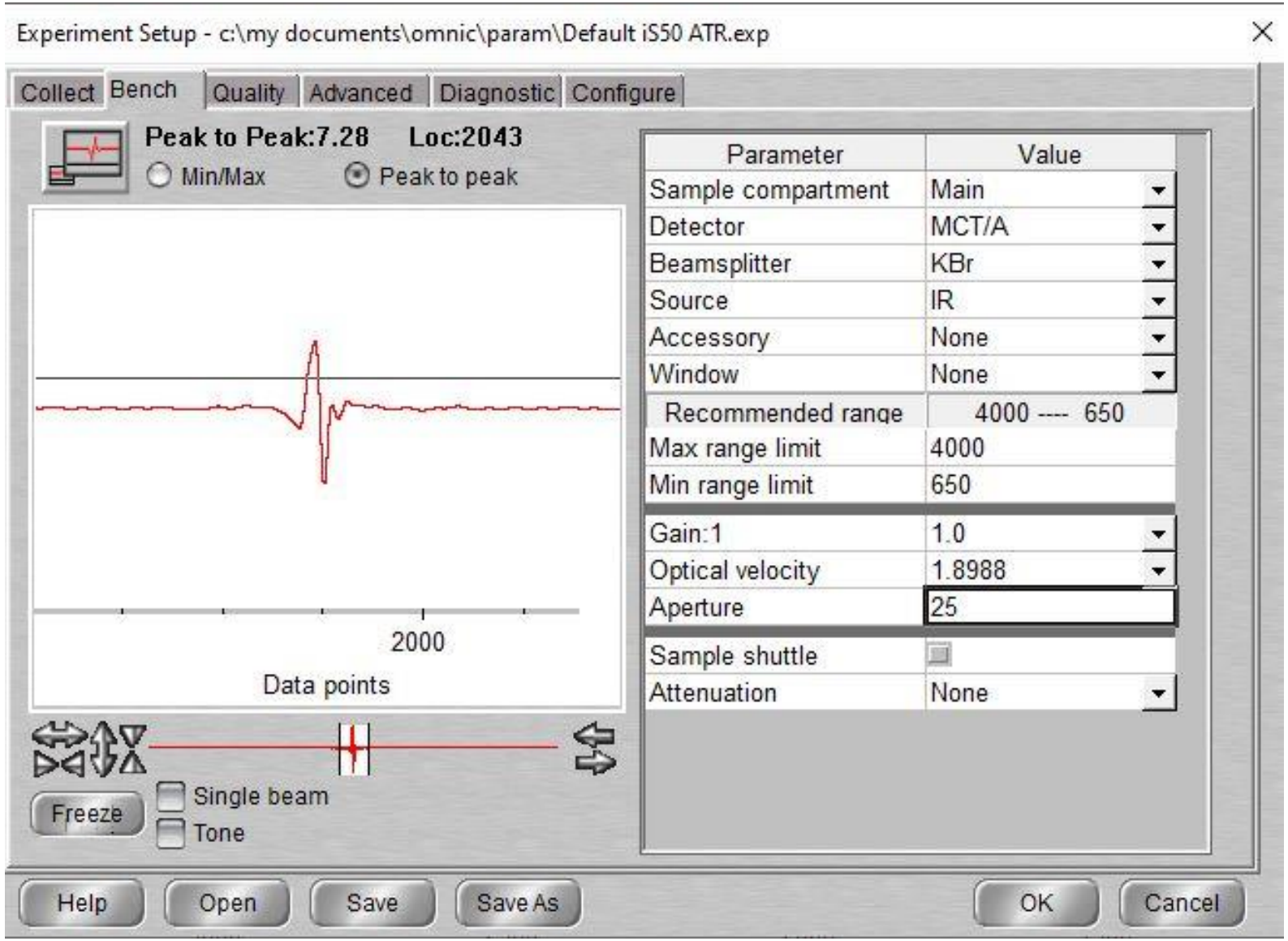

Experiment Setup - c:\my documents\omnic\param\Default iS50 ATR.exp
Collect
Bench
Quality
Advanced
Diagnostic
Configure
Peak to Peak:0.01
Loc:2046
Min/Max
Peak to peak
2000
Data points
Freeze
Single beam
Tone
Parameter
Value
Sample compartment
Main
Detector
MCT/A
Beamsplitter
KBr
Source
IR
Accessory
None
Window
None
Recommended range
4000 ---- 650
Max range limit
4000
Min range limit
650
Gain:1
1.0
Optical velocity
1.8988
Aperture
25
Sample shuttle
Attenuation
None
Help
Open
Save
Save As
OK
Cancel

Thermo
SCIENTIFIC

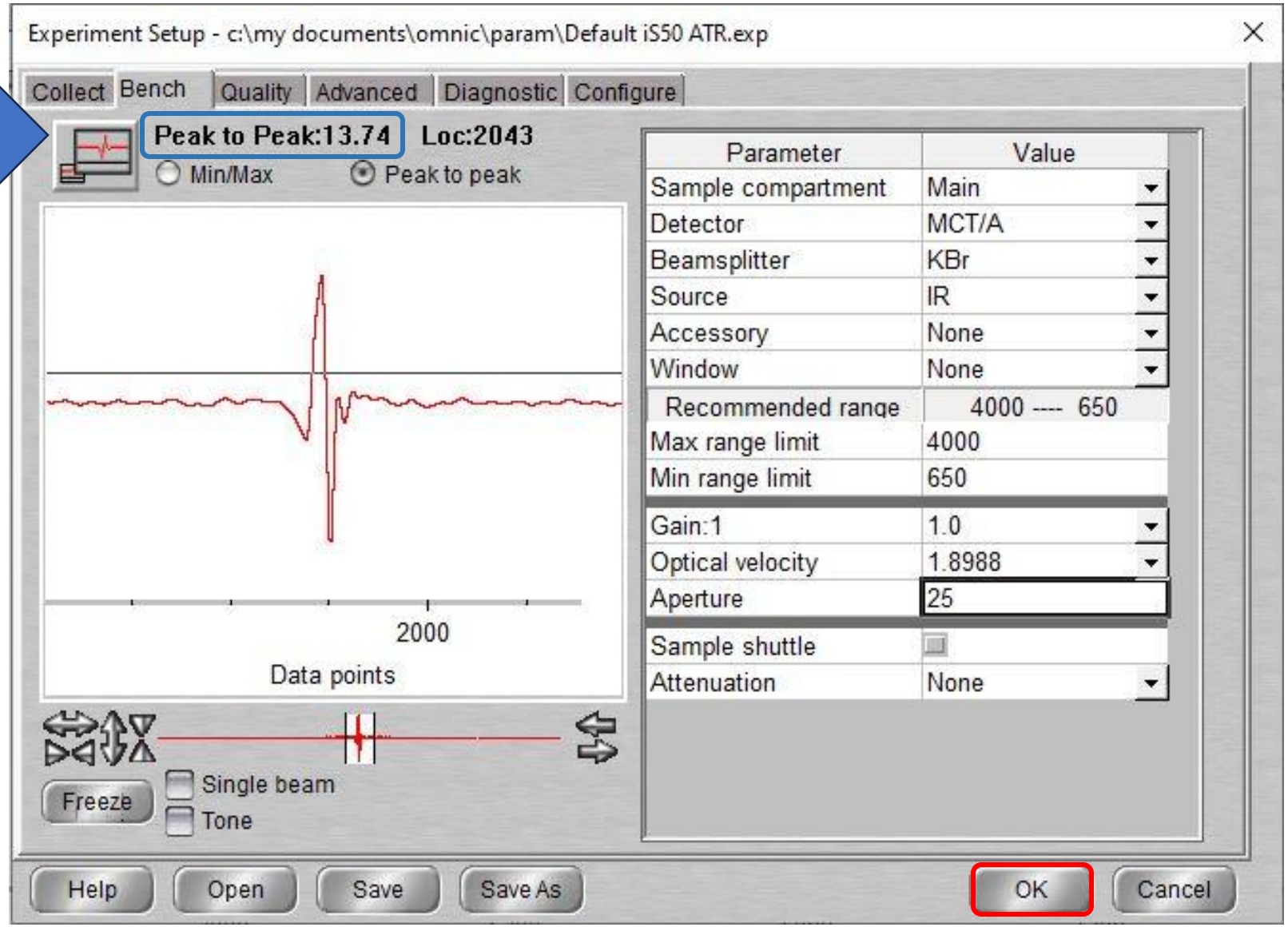
As we add liquid nitrogen, we can see the signal increase.



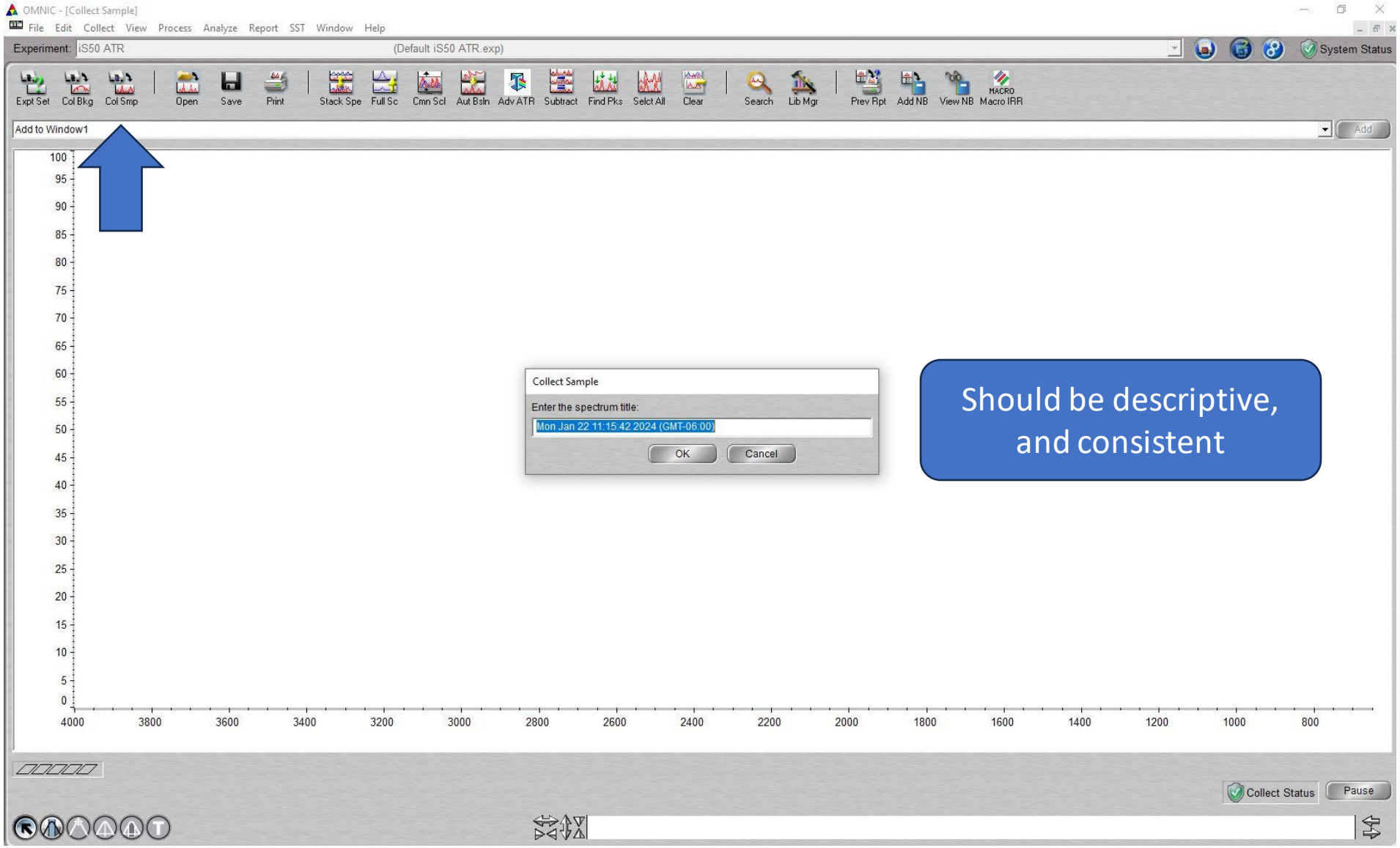

Experiment Setup - c:\my documents\omnic\param\Default iS50 ATR.exp
Collect
Bench
Quality
Advanced
Diagnostic
Configure
Peak to Peak:3.91
Loc:2043
Min/Max
Peak to peak
2000
Data points
Freeze
Single beam
Tone
Parameter
Value
Sample compartment
Main
Detector
MCT/A
Beamsplitter
KBr
Source
IR
Accessory
None
Window
None
Recommended range
4000 ---- 650
Max range limit
4000
Min range limit
650
Gain:1
1.0
Optical velocity
1.8988
Aperture
25
Sample shuttle
Attenuation
None
Help
Open
Save
Save As
OK
Cancel


Experiment Setup - c:\my documents\omnic\param\Default iS50 ATR.exp
Collect
Bench
Quality
Advanced
Diagnostic
Configure
Peak to Peak:7.28
Loc:2043
Min/Max
Peak to peak
2000
Data points
Freeze
Single beam
Tone
Parameter
Value
Sample compartment
Main
Detector
MCT/A
Beamsplitter
KBr
Source
IR
Accessory
None
Window
None
Recommended range
4000 ---- 650
Max range limit
4000
Min range limit
650
Gain:1
1.0
Optical velocity
1.8988
Aperture
25
Sample shuttle
Attenuation
None
Help
Open
Save
Save As
OK
Cancel

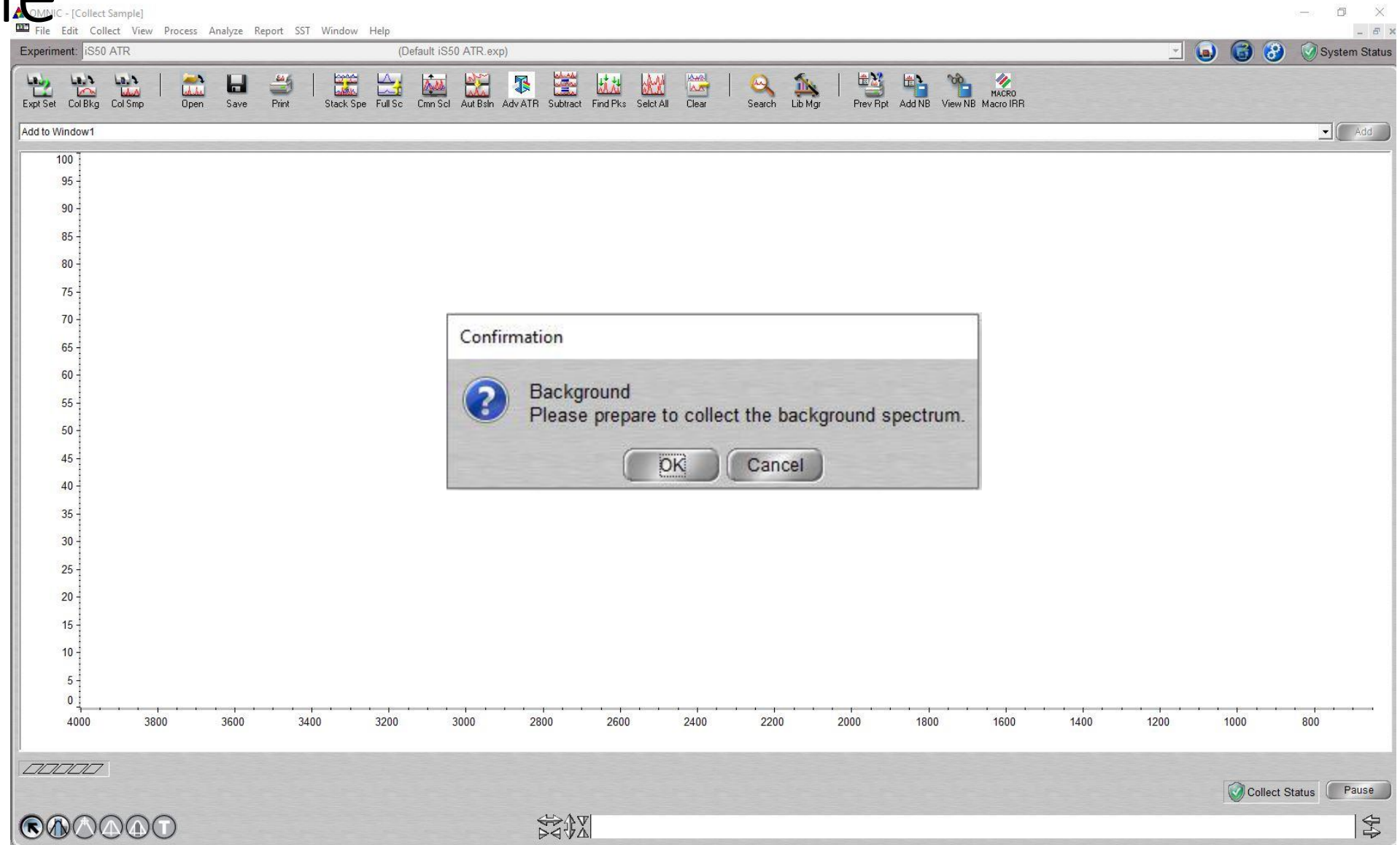
Write this down

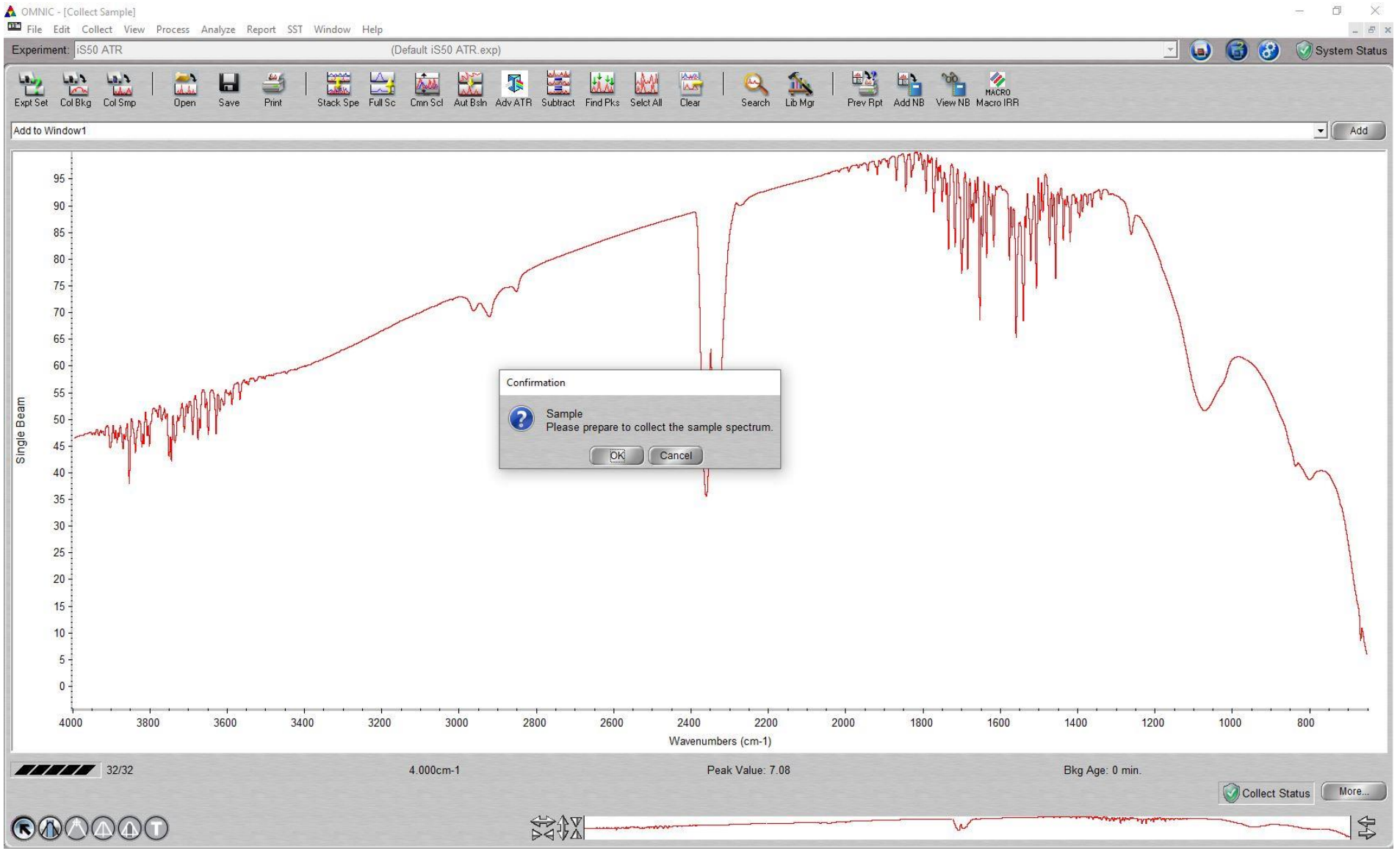


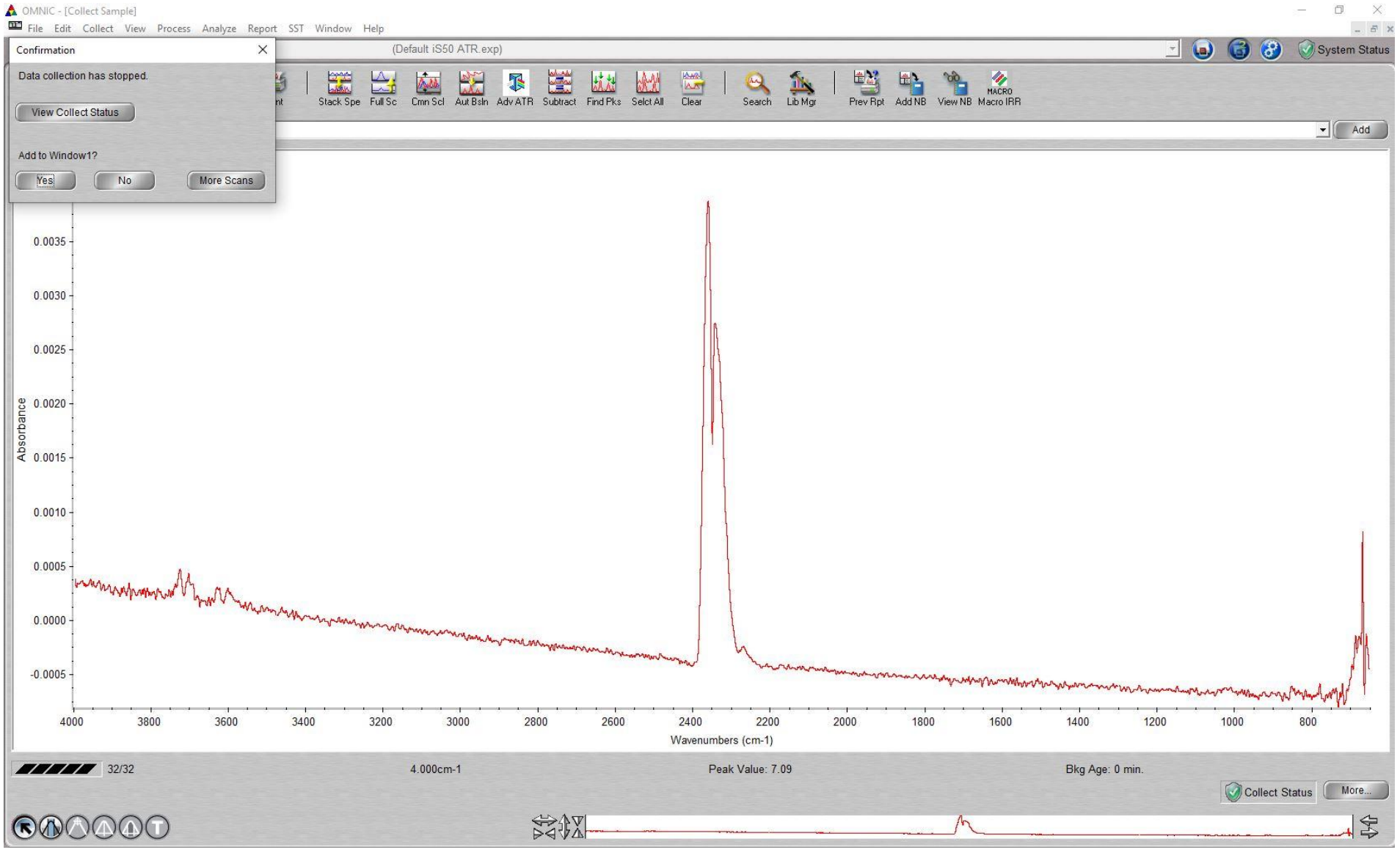
Let’s take a measurement!

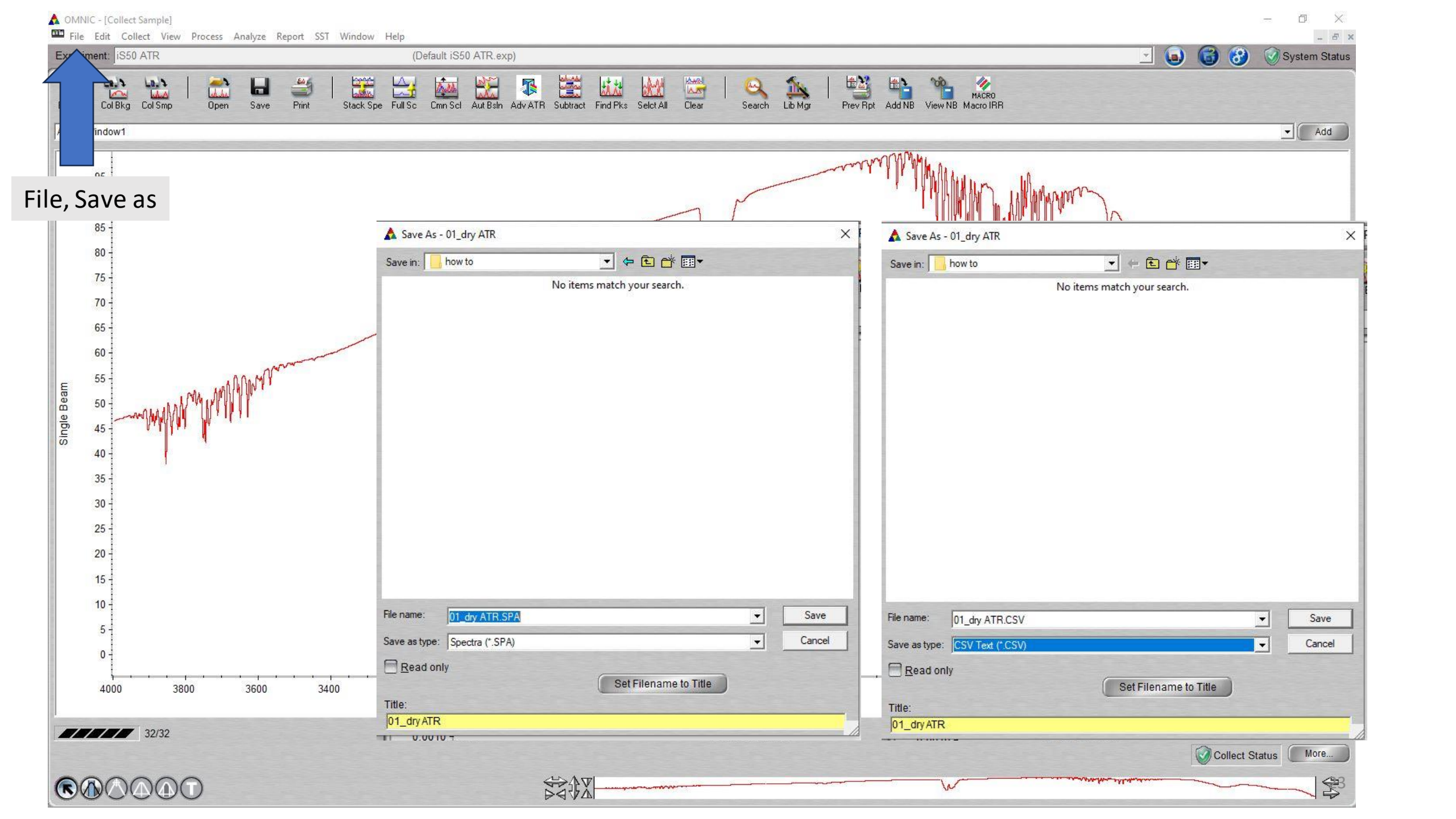


We selected “Collect background before every sample”



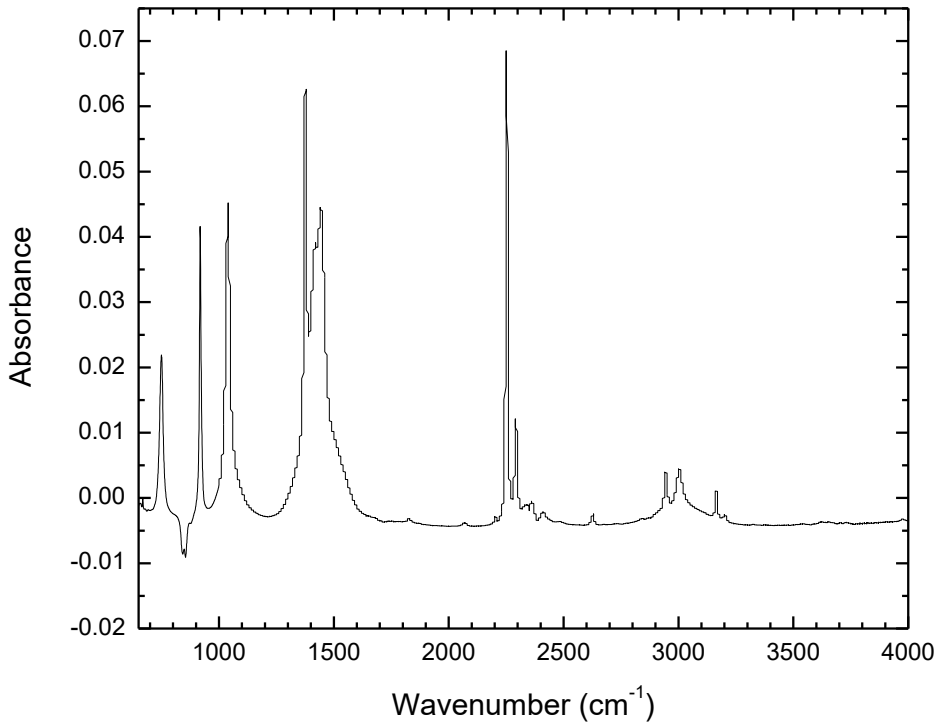

OMNIC - [Collect Sample]
File Edit Collect View Process Analyze Report SST Window Help
Experiment: iS50 ATR (Default iS50 ATR.exp)
System Status
Expt Set Col Bkg Col Smp Open Save Print Stack Spe Full Sc Cmn Scl Aut Bsln Adv ATR Subtract Find Pks Selct All Clear Search Lib Mgr Prev Rpt Add NB View NB Macro IRR
Add to Window1
Add
Confirmation
Sample
Please prepare to collect the sample spectrum.
OK
Cancel
Single Beam
Wavenumbers (cm-1)
32/32
4.000cm-1
Peak Value: 7.08
Bkg Age: 0 min.
Collect Status
More...


OMNIC - [Collect Sample]
File Edit Collect View Process Analyze Report SST Window Help
Confirmation
Data collection has stopped.
View Collect Status
Add to Window1?
Yes
No
More Scans
(Default iS50 ATR.exp)
System Status
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
MACRO
Macro IRR
Add
Absorbance
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
-0.0005
4000
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
Wavenumbers (cm-1)
32/32
4.000cm-1
Peak Value: 7.09
Bkg Age: 0 min.
Collect Status
More...

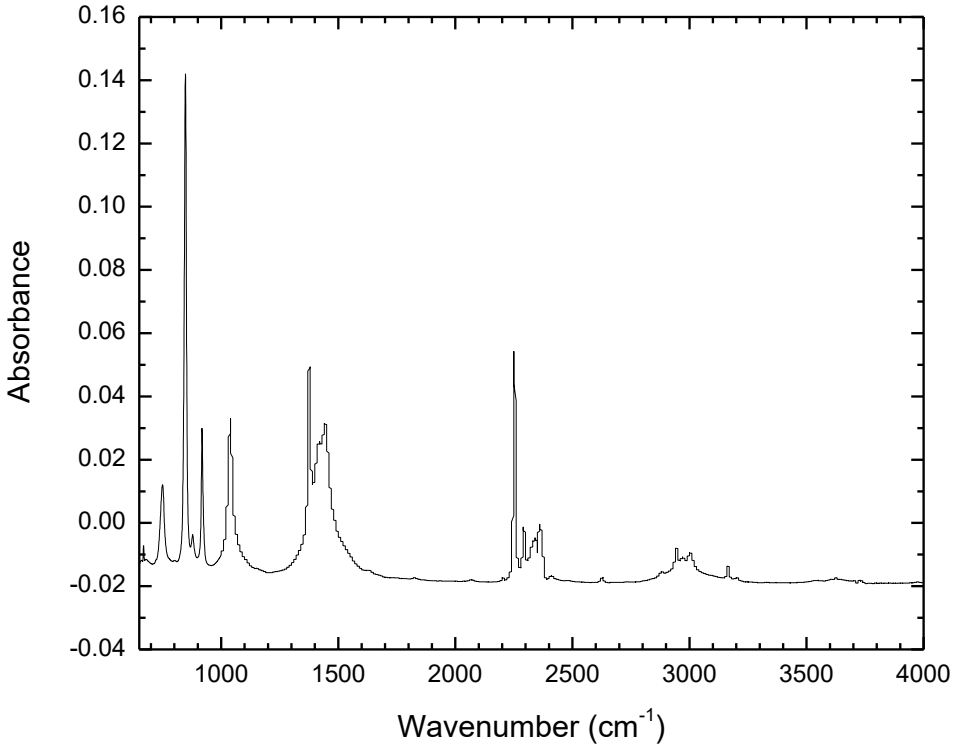

File, Save as
OMNIC - [Collect Sample]
File Edit Collect View Process Analyze Report SST Window Help
Experiment: iS50 ATR (Default iS50 ATR.exp)
System Status
Col Bkg Col Smp Open Save Print Stack Spe Full Sc Cmn Scl Aut Bsln Adv ATR Subtract Find Pks Selct All Clear Search Lib Mgr Prev Rpt Add NB View NB Macro IRR
Window1
Add
Single Beam
4000 3800 3600 3400
Save As - 01_dry ATR
Save in: how to
No items match your search.
File name: 01_dry ATR.SPA
Save as type: Spectra (*.SPA)
Save
Cancel
Read only
Set Filename to Title
Title: 01_dry ATR
Save As - 01_dry ATR
Save in: how to
No items match your search.
File name: 01_dry ATR.CSV
Save as type: CSV Text (*.CSV)
Save
Cancel
Read only
Set Filename to Title
Title: 01_dry ATR
32/32
Collect Status
More...

Examples of data

MeCN

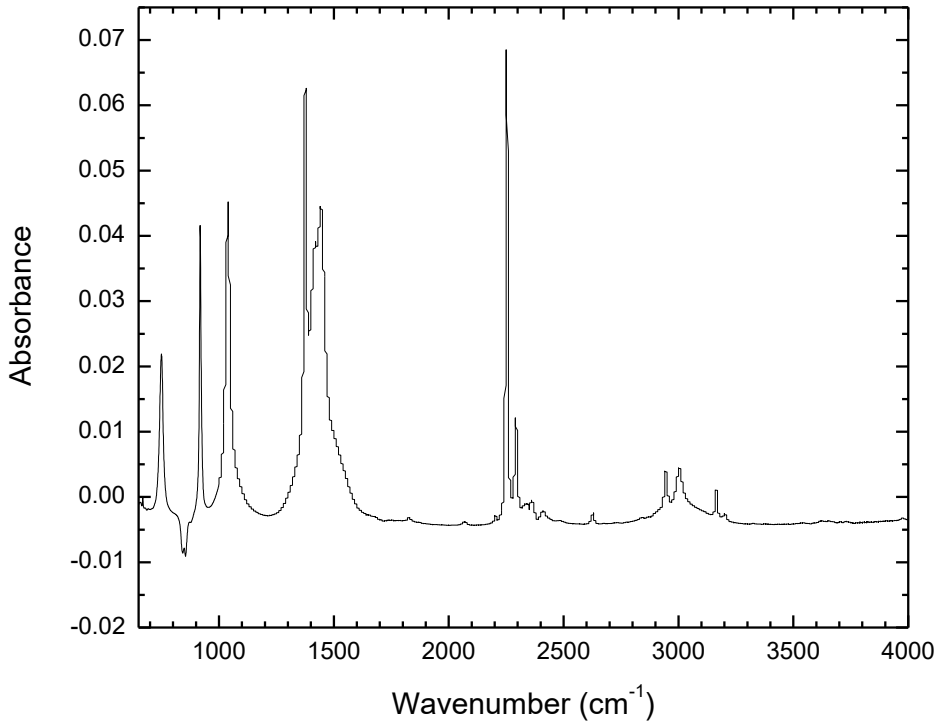


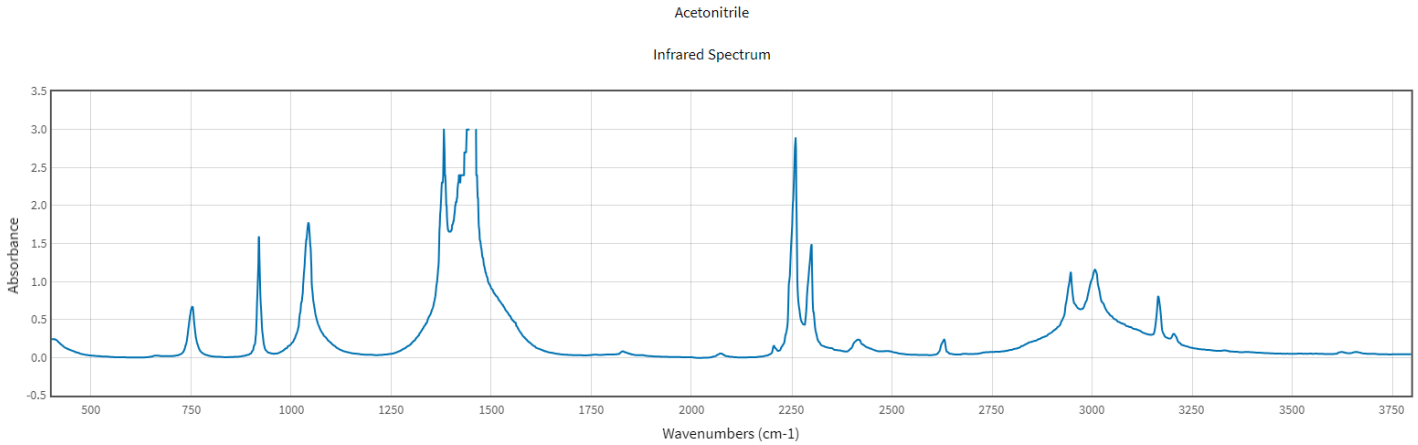
100 mM $TBAPF_6$ MeCN



Checking data with literature

MeCN





From NIST Chemistry WebBook