Using The Germanium Wide Angle Attenuated Total Reflectance Accessory.

The GATR is a single reflection 65°ATR accessory designed for analyzing monolayers and adsorbed species on semiconductor and metallic substrate

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1. Installing the GATR

- 1.1. Remove any accessory in the Nicolet 6700.
- 1.2. Remove the snap in baseplate.
- 1.3. Install the "ears" or sidewall adaptors from both beam path holes,
- 1.4. You need to attach the purge tube of the GATR to the connection at the back of the sample chamber.
- 1.5. The GATR should be mounted on it own base plate. Lift the GATR up and lower it down into the sample compartment of the Nicolet.
- 1.6. There is a pressure clutch drive set for 56 oz in that should be in the GATR box and mounted on top of the pressure drive.



Figure 1 Harrick GATR accessory.

- 1.7. The purge sleeves of the GATR need to align with the beam holes on the FTIR and need to be extended to fit snugly in the instrument.
- 1.8. Make sure the GATR plate and pressure foot are clean. If not you can clean them with a optical lens paper or cotton Q-tip damp with isopropanol or water. Do not rub the GATR crystal.
- 1.9. Check the flowmeter behind the FTIR, to make sure the purge is N_2 with a flow rate of 30 ft³/hr (scfh) into the FTIR.

2. Collecting the Background Reading

2.1. Open OMNIC on the computer by double-clicking the OMNIC desktop icon. You should see the tool bar shown



Figure 3 The accessory compartment showing "ears".

in Figure 3 below.

The second se		in initialites the	ore window her	p						
Experiment: De	efault - Transm	ission								
		راجا داجا		ABS 2	KA 8 2		11N	>	,	2
Open Save	Print Col Smp	Col Bkg Expt Set	Stack Sp Full Sc	Absorb % Trans	Find Pks Aut Bsin	Bsin Cor Searc	h Lib Mgr	Cut	Сору F	Paste

Figure 4 Omnic tool bar.

- 2.2. Click the menu bar icon "Expt Set" to enter the *Experiment Setup* window.
- 2.3. Under the *Bench* tab, set the accessory to *ATR*, Figure 5.
- 2.4. Under the *Collect* tab, Figure 4, set the number of scans to > 100 and the final format to what you want (we usually use single beam as described below). Enter a title for the experiment. Your window should be similar that shown in Figure 5.
- 2.5. You can run the diagnostics under the Diagnostic tab to make sure everything is ok.
- 2.6. Click OK.
- 2.7. You will need to wait about 30 minutes to make sure the background is clean enough to measure monolayers.
- 2.8. Make sure nothing is touching the Ga; click the menu bar icon "Col Bkg" to collect the background and click *OK* to start collection. (Is this how you do it or do you use a Si wafter for the background?)

Collect	3ench	Quality	Advanced Diagnostic					
Estimated time	for this collection	on: 00:01:1	17					
No. of scans:	64		File Handling					
Resolution:	4.	•	Base name:					
Data spacing:	1.928 cm-1		C:\My Documents\Omnic\autosave\0001.spa					
Final format:	Absorbance	•	Background Handling					
Correction:	None	•	Collect background before every sample					
Automatic atmospheric suppression			Collect background after every sample					
Preview data o	ollection		Collect background after 100 minutes					
Experiment des	cription:		O Use specified background file:					
This is the default experiment file.			Browse					
			Experiment title:					
			Apatite					

Figure 5 Experimental Setup in Ominc

lect Benc	h ÌG	uality	Advanced	Diag	nostic	
Max:4.48	Min:-2.21	Loc:1024	Gain:8		Sample compartm	ent:
			Autogain	-	Main	-
			Velocity:		Detector:	
			0.6329	•	DTGS KBr	-
	-		Aperture:		Beamsplitter:	
			1.00		KBr	-
	~~~~	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	- Min/May		Source:	
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10 Da	UU ata points		Spectra	al Range	Sample shuttle	
•		Single bea	m Recomm	iended i Sa	max: 4000 400 aved: 4000 400	

Figure 5: The Bench tab of the Experiment Setup

- 2.9. When the background looks stable no major changes in peaks click *Start Collection* in the top right corner of the window. An unstable background indicates that the instrument is not fully purged, so you need to wait for purging to finish. See Figure 6
- 2.10. Click Yes when prompted about adding the background to Window 1.

#### 3. Placing the Sample in the GATR Accessory

- 3.1. Make sure that the pressure applicator's tip is well above the crystal.
- 3.2. If your sample is a single solid piece, place it on the opening in the metal plate under the pressure foot.
- 3.3. If your sample is a powder:
  - 3.3.1. Hard materials must ground to a very very fine powder in a mortar and pestle to use.
  - 3.3.2. Cover the Ge surface with a thin layer of the materials. Do not allow a metal spatula to touch the Ge surface.

#### 4. Collecting the Spectrum

- 4.1. Low the pressure foot to increase the pressure applied to the sample by turning the pressure control clockwise until when??
- 4.2. Click the menu bar icon reading "Col Smp" to begin collecting the sample.
- 4.3. At the prompt, enter the sample's chemical name or formula in front of the default title

(the current date and time).

- 4.4. Click OK when prompted and press Start Collection in the top right corner of the screen.
- 4.5. Click Yes when prompted Add to Window1? Unexpected peaks in the range of O-H ( $\sim$ 3800,  $\sim$ 1600 cm⁻¹) or CO₂ ( $\sim$ 3700,  $\sim$ 2340 (doublet) and  $\sim$ 660 cm⁻¹) and sample should be re-run with ample time allowed for the system to purge.
- 4.6. Save the spectrum by clicking the Save icon. If multiple spectra are open in a window, you will need to click on the desired spectrum before clicking Save.
- 4.7. Click Set Filename to Title, and save the file to you directory.
- 4.8. You can reprocess the blank and the sample spectrum to get a %T or Absorbance spectrum.

## 5. Cleaning Up

- 5.1. Close OMNIC.
- 5.2. Clean the pressure applicator and Ge crystal. In most case where a slide sample such as Si was use no cleaning is necessary. If cleaning is necessary, be careful to use only lens tissue or a cotton tipped applicator. .

### 6. Remove the GATR

- 6.1. Remove the GATR, lift it out of the FTIR and place it in it's storage box.
- 6.2. Replace the box on the shelves.