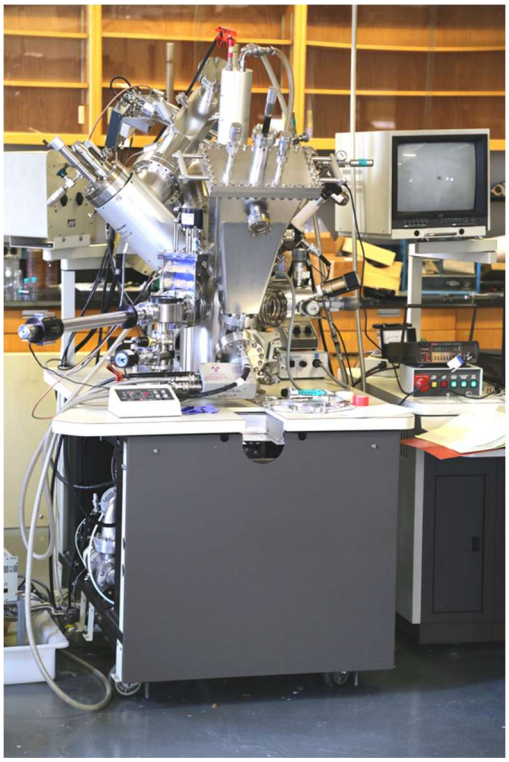
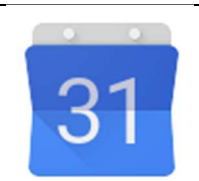
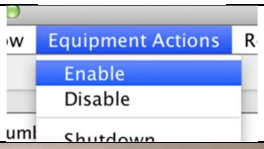
COLUMBIA UNIVERSITY
IN THE CITY OF NEW YORK
COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

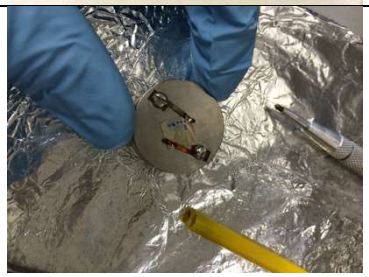
Phi 5500 XPS
Standard Operating Procedure

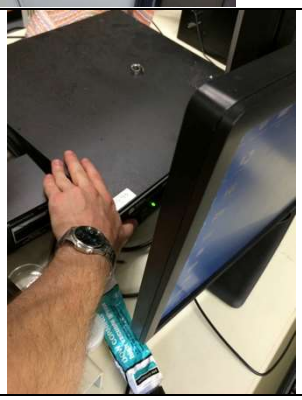


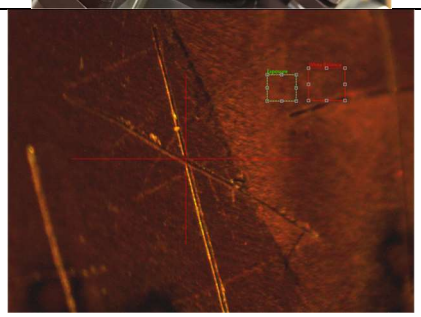
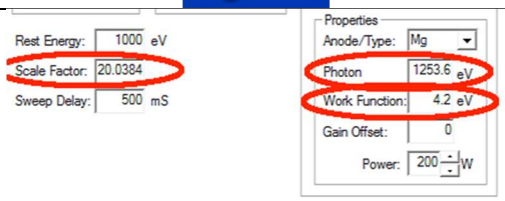
These instructions are intended for reference only, and will *not* replace the thorough training required for proper system operation. Contact Daniel Paley (dwp2111) with questions or to report a system problem.

SOP prepared by Dr. Daniel Paley and Amrita Masurkar.

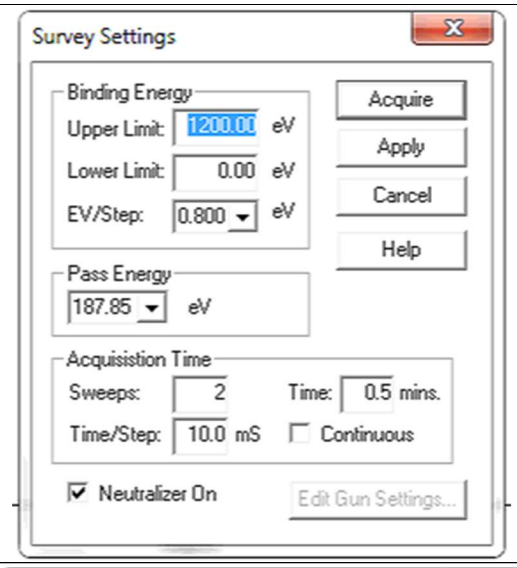
1.	The XPS is very sensitive, the operation is mostly manual, and you can easily cause major damage. If anything seems unusual, **stop immediately** and **call Dan** at 434-953-9497.	
2.	Users typically do not load their own samples. Contact staff in advance to schedule sample loading.	
3.	Enable the tool in **BADGER**	
4.	Check the chamber base pressure is in the 10^{-10} range. If it is above the 10^{-10} range, contact staff.	
5.	Check the N_2 cylinder for the load lock purge: cylinder open, regulator set to 3 psi, ball valve open.	

6.	Check the loading fork is rotated correctly (THIS SIDE UP). Check the AVC remote: V3 open (green), all others red; 5 bars on vacuum indicator. If the chamber pressure is high or the AVC remote is not as shown, contact staff or the superuser.	 
7.	Prepare your sample. Wear clean gloves and do not contaminate the puck with vacuum grease. **If your sample is a powder, you must discuss it with staff.**	
8.	Press Backfill Intro on the AVC remote. Wait for the cover to pop gently off the load lock. Attach your sample to the fork, using the upper ring on the puck. Replace the cover; do not use grease! Press Pump Intro and set a timer for 10 minutes.	

9.	Power up the X-ray source: Turn on the Power switch; select STANDARD Source; press BOTH, Mg, or Al buttons. Press red High Voltage button (loud relay noise will start).	
10.	Use voltage knob to slowly set 10 kV **on the left dial**.	
11.	On the digital display, turn the power to 100 W by pressing the arrows. Slowly increase voltage to 15 kV by turning the knob more. Then adjust power to 200 W with arrows.	
12.	Turn on the card rack power and RBD 147 interface box (behind the monitor).	 

13.	Start the ToupView software. Select the camera under Camera List. Set the camera to moderate zoom (halfway out) without touching anything besides the zoom wheel. **The black zoom wheel turns with no resistance. If there is any resistance, you're adjusting the wrong part.**	 
14.	Use the stage x and y axes to put your sample in the crosshairs. Zoom the camera all the way in. Adjust the stage z axis until the center of the image is in focus. Now your sample is aligned with the analyzer focal point.	
15.	Start AugerScan and say yes to allow changes to the computer.	
16.	In System-Hardware Properties-XPS, set the scale factor, photon energy, and work function for the X-ray anode you are using. Current values are Mg 1253.6 eV, 4.55 eV, 20.0528; Al 1486.6 eV, 3.24 eV, 20.0081; but updated values are posted on the instrument.	

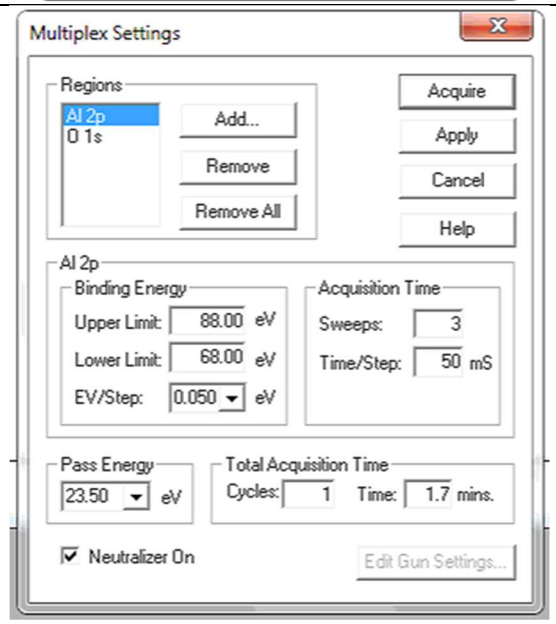
17. Record a survey scan: Acquisition→New Survey. Binding energy is 1200 to 0 for Mg or 1400 to 0 for Al. EV/Step can be between 0.4-1.6. Pass Energy is 187.85. Time/Step is 10 ms. Sweeps should be at least 2 but can be as many as you need.

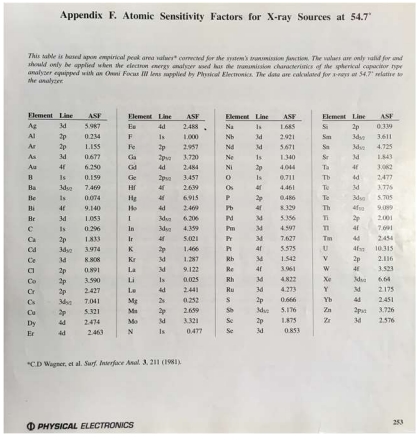


18. Record high quality scans of individual peaks: Acquisition→New Multiplex. The binding energy ranges can be adjusted using the data collected in your survey scan. EV/step can be 0.025 to 0.1, but 0.05 is usually suitable. Pass energy is 23.5 eV. Time/Step is 50 ms. Sweeps can be any number, but commonly between 1 and 10. Note that each region has to be configured individually.

Semi-quantitative XPS is possible. You should be sure to collect the peaks listed on page 253 of the XPS Handbook.

You should always measure the C 1s peak because it is a useful binding energy reference to correct for sample charging.





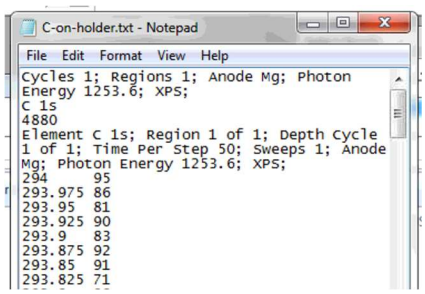
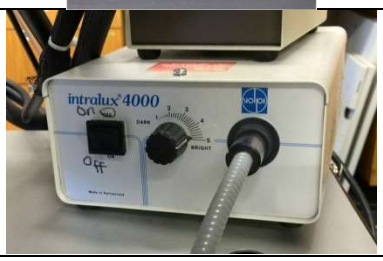
Appendix F. Atomic Sensitivity Factors for X-ray Sources at 54.7°

This table is based upon empirical peak area values corrected for the system's transmission function. The values are only valid for and should only be applied when the electron energy analyzer used has the transmission characteristics of the spherical capacitor type analyzer equipped with an Omni Focus III lens supplied by Physical Electronics. The data are calculated for x-rays at 54.7° relative to the analyzer.*

Element	Line	ASF	Element	Line	ASF	Element	Line	ASF	Element	Line	ASF
Ag	3d	5.987	Eu	4d	2.488	Na	1s	1.685	Si	2p	0.339
Al	2p	0.234	F	1s	1.000	Nb	3d	2.921	Sm	$3d_{5/2}$	3.611
Ar	2p	1.155	Fe	2p	2.957	Nd	3d	5.671	Sn	$3d_{5/2}$	4.725
As	3d	0.677	Ga	$2p_{3/2}$	3.720	Ne	1s	1.340	Sr	3d	1.843
Au	4f	6.250	Gd	4d	2.484	Ni	2p	4.044	Ta	4f	3.082
B	1s	0.159	Ge	$2p_{3/2}$	3.457	O	1s	0.711	Tb	4d	2.477
Ba	$3d_{5/2}$	7.469	Hf	4f	2.639	Os	4f	4.461	Te	3d	3.776
Be	1s	0.074	Hg	4f	6.915	P	2p	0.486	Th	$3d_{5/2}$	5.705
Bi	4f	9.140	Ho	4d	2.469	Pb	4f	8.329	Ti	$4f_{7/2}$	9.089
Br	3d	1.053	I	$3d_{5/2}$	6.206	Pd	3d	5.356	Tl	2p	2.001
C	1s	0.296	In	$3d_{5/2}$	4.359	Pm	3d	4.597	Tl	4f	7.691
Ca	2p	1.833	Ir	4f	5.021	Pr	3d	7.627	Tm	4d	2.454
Cd	$3d_{5/2}$	3.974	K	2p	1.466	Pt	4f	5.575	U	$4f_{7/2}$	10.315
Ce	3d	8.808	Kr	3d	1.287	Rb	3d	1.542	V	2p	2.116
Cl	2p	0.891	La	3d	5.122	Re	4f	3.961	W	4f	3.523
Co	2p	3.590	Li	1s	0.025	Rh	3d	4.822	Xe	$3d_{5/2}$	6.64
Cr	2p	2.427	Lu	4d	2.441	Ru	3d	4.273	Y	3d	2.175
Cs	$3d_{5/2}$	7.041	Mg	2s	0.252	S	2p	0.666	Yb	4d	2.451
Cu	2p	5.321	Mn	2p	2.659	Sb	$3d_{5/2}$	5.176	Zn	$2p_{3/2}$	3.726
Dy	4d	2.474	Mo	3d	3.321	Sc	2p	1.875	Zr	3d	2.576
Er	4d	2.463	N	1s	0.477	Se	3d	0.853			

*C.D Wagner, et al. *Surf. Interface Anal.* 3, 211 (1981).

PHYSICAL ELECTRONICS 253

19.	Spectra are saved in .txt format as well as binary .asn format. You can take away your spectra in .txt format using a USB drive and process/plot/curve-fit them in your preferred data processing software. Basic peak fitting is possible in AugerScan; contact staff for a demo. You can download Augerscan using the link provided here.	 https://www.dropbox.com/s/h7ypitjaqhk63u0/augerscan-3-3.zip
20.	When all measurements are completed, turn off the card rack power and black interface box.	
21.	On X-ray source, decrease power to 0 W (upper panel) and then decrease voltage to 0. Wait 2 minutes, then switch off the X-ray source control.	
22.	Turn off the chamber light.	

23.	Contact staff to remove sample.	
24.	Turn off the X-ray source control.	
25.	**BADGER LOGOUT:** Don't forget to disable the tool in badger after you're done.