WDS Identification Assignment

With this assignment you will have an opportunity to identify major and minor elements in an unknown material. You are given **four wide WDS scans** (15 kev, 20 nA) of the material, each using a different crystal, for the same material.

Students used to do this exercise "the old fashioned way" (manually) using paper copies of the scans and books with the wavelength tables. But it makes more sense to get you started using the powerful Probe for Windows software to figure out what the elements are (see attached sheet for instructions). But you can't assume the computer will do all the work… **you must use common sense and a sense of strategy**.

If you wish, you may work in groups of 2 or 3 on the computer. But each person must turn in their own individual writeup.

You will be looking at two different computer displays. The first question below uses the Probe "EDS computer" which is over next to the main probe keyboard and monitor. When you are finished with this quick look-see, you work on the OTHER computer ACROSS the room.

I supply you with an EDS spectrum of the unknown. (On the Probe EDS computer, second keyboard, monitor up high, with WINEDS running, go to file->Open->2003->Oct21-unknown.ems) This simulates the real world situation, where most modern microprobes have EDS to give you a first estimate of the major elements present. **The exercise here simulates an assignment of scanning the full WDS spectra for all major and minor elements, where you need to explain to your boss what every peak means. Remember, this is an old Be-window detector…cannot see elements below Na).**

===
-> Important guidelines for X-ray ID for both EDS and WDS:
• If there's a beta line, there must be an alpha.
• An alpha is *usually* ~10X as intense as a beta.
• If there's a K line, there's going to be an L line (if there are enough shells)
• You can't create an x-ray line if your E_o is below the critical excitation voltage
Guidelines for WDS
• If there's a higher order reflection (n >1) line, then there's going to be a first order line. On the other hand if you have a strong first order line, there is a certain probability that you might see a 2nd or higher order, depending upon how well that crystal reflects higher orders (and sometimes a crystal can do odd, not even or vice-versa). TAP has strong higher order reflections.

• You should start with the scan that covers the lowest wavelength; and on each scan, go from low to high wavelength . WHY? (answer this is your writeup)   (Note: we will not be using the LIF 220 crystal, only the LIF 200 one.)

• You always FIRST try to get some 'hits' by looking for potential Ka lines excited by your E0, for the shortest wavelengths—which will be the _____ crystal, right? If no clear hits, go for La lines. If not there then the Ma lines. And you would, for the very first attempt, go for a <u>strong</u>, short wavelength, line. Warning: **do not first** look at the "all wavelength in the universe listings"-- you will get lost in the forest's underbrush and not recognize the big tree 6 feet away from you!

Accessing probe files on "Probe3" Dell in Room 306

        This stand alone windows computer in the probe lab (table opposite the probe, by the air conditioner) is for your use. It is a 2.8 mhz Dell known as "Probe3". The main computer for the probe is Kontron "Probe4".

        <u>Read completely through all the directions before you start.</u>

1. On Probe3 , first look at the bottom menu stripe—is there a label saying "Probe for EPMA"? If so, the program is already running. (If not, go to 2 below). Click the box, and the application window will appear. If the correct file is open (092401.mdb), go to 4 below. If not, you will need to close the current file (*.mdb), by File->Quit.   Go to 3 below.

2. If Probe for Windows is not running, then click the icon on the desktop that says "shortcut to v9-Probe for Windows-E". It will ask if you want to interface with hardware. Yes will give you full simulation options (for offline training); No gives you only ability to evaluate data. For this exercise, No is sufficient.

   NOTE: We have another version (10) also on this computer, but it is only for accessing files generated on a different microprobe with totally different settings (a Jeol 8530). Do NOT attempt to run that version.

3. You need to Open an existing file (in this case, 092401.mdb) which has the data to be evaluated. File->Open-> (browse the main folder "Probe User Data Copies", under that 777, then Fall 2001 to get to 092401.mdb. If you ever get a window (e.g. DataMotor) asking if you want to overwrite some parameters, click NO.

4. To evaluate the wavescans, you need to go to the PLOT! Menu, and immediately select (radio button) Wavescans at top left. The wavescan, that covers the whole spectrometer range, was collected in 500 channels, that span 10 'pages' in the database. **In this case you will select ALL from Wa21 thru Wa30** (just drag down and highlight all with the mouse). If you do not do this, you will glitch. Also, if you include Wa31, you will glitch. The title is "Mystery Wavescan Take II" which is tagged as Wa21, and the following 9 files simply say "continued".

5. You need to select which variable will be X and what will go with it in Y. X can be either spectrometer units (=sin theta units), or angstrom units, or energy (KV) units, and Y will be the associated <element n> wavescan counts. The elements (which may or may not be in the sample, are ones I chose to align each spectrometer with), and stay as the default (element) name for the spectrometer. The list does not explicitly tell you which crystal is used for each wavescan, just the spectrometer number. But here is what you need to known: Si(1) = TAP, Ca(3)=PET, Fe(5)=LIF and O(2)=PC1.

   → **SELECT SPECTROMETER (SIN THETA) UNITS FOR X AXIS**

6. Remember, you want to start with the SHORTEST wavelengths and ID those elements on the spectrometer that looks at the shortest wavelengths FIRST. And the order of the crystals, from shortest wavelengths to longest (say for Ka) is: LIF then PET then TAP and finally PC1.

2

7. How to read off the spectrometer position (sin theta, or Å)→ Below the KLM markers is a box that shows [position counts] for wherever the cursor is POINTING.

8. I suggest you first qualitatively get an overview of the scan when you first open it up – just eyeball it, how many peaks are obvious at the default vertical scale? OK, then magnify the bottom 1/3 – ¼ of the vertical scale, looking for shorter peaks. See any? Those 2 initial views will provide you with your initial numbers of peaks you will want to identify.

The default plot is y in linear scale, but you "may" be curious to look at the y in log. If you decide to try this: in the Plot window, click on the 4ᵗʰ icon from the top right (looks like a blue printer with paper coming out, "system"). Click Style, and then click Log Data "y".
To undo it, just go back and uncheck that box.

9. Here are some specific instructions on using the KLM markers on the top right side of the PLOT window. These can make the job much easier than paging thru a reference book -- but there is a major limitation, which is computer memory -- the computer has all the data that is the reference tables but cannot keep it all in active memory - by default, it includes only the major lines, which are usually good enough to ID the major peaks, but smaller peaks (which generally includes some of the higher order peaks) may not be displayed automatically. I will explain in a moment how to do that.

10. You have SOME initial information because you know from the EDS spectrum what the major peaks are, right? So you will be able to see those pretty simply - click the Specific element button and type in the element you wish to see, and if any of the major lines are present, they will appear. That will help reduce some of the "unknowns".

11. You can also "click thru the periodic table" by entering a low Z element (say B or C) in the Specific element box and then clicking the arrow on the top left of the box to scroll thru the elements and look for "hits".

12. What do you do if you have a clear peak present but you don't get a hit? Then you have to zoom in on the spectral region (x axis) and then click "Load Xray Database" button, then Set Minimum Intensity to 0.01 (default is usually 0.25), and set the Start and Stop Angstroms to just below and just above the peak, and then click Load New Range, and click Close, and then you must re-enter the elements in the Specific Element window. Important! Just because the Minimum shows "0.01" does not mean the relevant low intensities will be shown – you MUST make sure that the "Start Angstroms" and "Stop Angstroms" be set for a small range centered on the peak you are trying to identify.

13. The most difficult is the PC1 as some the markers may not be perfectly aligned for the n>1 peaks. (This is a somewhat complicated issue…)Use logic.

14. There are more than one way to do each crystal's wavescan – remember, you want to write down the sin theta position, then the element and specific x-ray type, then order if n>1

--For LIF there only 4 peaks to identify and you can easily identify 2 of them by selecting the major elements, but it may take step 10 above to identify the smaller peaks (program can't keep everything in memory all the time).

--For PET and the rest, there are many more peaks and here it makes sense to go more methodically and start at the left end (low end), and go peak by peak to the right.

Final note:

I expect that you will find and identify minimally the following number of peaks (lines). LIF 4; PET 11; TAP 16 or 17; PC1 6-8. I supply a spreadsheet with many possible peak positions, listed within 5-10 units—if you disagree with one or more, that is fine…just be able to back up what you put down.

Don't wait until next Monday to use the computer for this, as I can guarantee that it will be busy.

If you wish to have access over the weekend or evenings. you can check out a key (room 306) from Jansi in the office ($1).

You will EACH turn in a writeup that includes:
  ➢ A spreadsheet table listing all elements, which lines, which orders if >1, and the crystal and sin theta position of each – organized by crystal, and then in increasing sin theta order for each crystal -- USE THE FORMAT I SUPPLY.
  ➢ A discussion of the procedure you used (EDS, WDS, order of evaluation)
  ➢ Why does it makes most sense to start with the shortest wavelengths first?
  ➢ If there are any ambiguities for any of the elements/lines, discuss.

Be sure to answer the questions above, particularly explaining why you start with LIF, and with the low sin theta end of the spectrum.

Also answer this question: True/False: It would be possible, **on one specific crystal**, to observe an n=2 peak, but not the n=1 peak of a particular element (as a general statement, not specifically for these spectra). Explain your answer. Extra credit if you can give a real example.

Versions
10/17/05 10/16/06 11/2/06 9/29/08 11/1/09 3/19/12 3/10/13 3/10/14 3/20/14 3/26/14

sin theta			
LIF			
	Element	Line	Order
43621			
48108			

sin theta			
TAP cont			
	Element	Line	Order
39230			
52235			

76728					52703			
83438					54991			
					55389			
PET					64824			
22128					67098			
35268					68214			
38393					78217			
40080					82371			
44192					82949			
66368								
70629					**PC1**			
76756					23389			
77265					24129			
80865					29272			
81452					33304			
					39122			
TAP					56166			
23970					57082			
26122					72621			
27674					76415			
32493								
36040								