GEOSCIENCE 777 April 1, 2014 Name ___________________

## Interference Exercise using "Virtual WDS"

The microprobe measures the intensities of characteristic X-ray lines of various elements of interest. However, there may be times where there are one or two types of interference. The worst is where the desired element's characteristic X-ray wavelength is overlapped by another's, e.g. you want to measure a small amount of vanadium in TiO2, you have to deal with V ka interfered with by Ti kb. The second type is where there is a peak in the vicinity of a potential background for the element you're trying to analyze (e.g. $^3$Ca ka near Mg ka): you need to be sure you examine all possible problems and work out clean background positions. There are two categories of interferences: first order, and higher orders, and they can fall on peak positions, or background positions. Thus, 4 combinations are possible.

Your assignment this week is to evaluate several pairs of elements for WDS peak and background interferences; for some of these, the interfering line is a higher order line of a shorter wavelength X-ray.

**Virtual WDS** is a program built around a database of peak and PHA spectra acquired on 7 different crystals on the SX50 at Cambridge, England, of pure metal and compound standards. It permits a user to model potential interferences and to work out approximate background positions.

Virtual WDS is on the desktop of the standalone probe lab computer (and SEM log-in computer).

I use Virtual WDS all the time, when someone brings in a sample with elements I'm not familiar with (or a new element in an old familiar compound). How does this go? I start with a list of elements. I first figure out what line (Ka, La, Ma) I will be using, and then which crystal might be used. Then go to Virtual WDS with a piece of paper and then systematically work thru ALL the combinations to see what might interfere with what.

In many instances there may be more than one crystal that can be used to diffract the X-ray wavelength you are trying to analyze (the FIRST one of each pair). Virtual WDS will "tell you" which crystals can reach which elements -- just click different xtals and see which elements are highlighted, or click on an element (**before** choosing " Select Elements").

This program is moderately intuitive, but needs a bit of instruction to get going, so I will give you step by step instructions for the first problem: V Ka overlapped by a line of Ti.

1. Always start with the first crystal in the list (on the left), here PET (you'll figure out later why).
2. Start up the program. (In upper right is E0: default is 15 keV.) Click on element V. A window pops up and tells you all of the lines (V Ka and La) that are possible to analyze, and with which crystals. For V Ka, it is LIF and PET, yes? Close the window.
3. Click the PET radio button at the top. (Note in passing: now all elements that can be analyzed at 15 keV on PET are shown, and impossible ones blacked out).
4. Click the "Select Elements" button on lower left. Now elements are green. In the green box below V, type in "1" (one wt %). Then click Ti green box and type in 59. Note how all other boxes have changed colors: these other elements now are showing all elements that possibly could interfere with V. Green means n=1 interference, blue means n>1 interference with V.

5. Click the “Plot” button on lower right. You now see two peaks, the black peak centered on center black line is V Ka (with its natural shape) and over it is the Ti __? Peak. You can identify peaks now by moving the cursor and hovering over the peak top…it reports the peak position in sinT units (not important) and its name…Ti Kb1. Now on your assignment page, you write in the first space “Ti Kb “.
6. This step will come in important later, but since you will use this procedure several times, I give it here. At the top menu bar is “Analytical Parameter”, click it and notice now that the option “PHA ON” is selected. Turn it off. I want you to see what the impact is when it if in the normal “OFF” position. It’s off, right? Leave it off until I tell you otherwise.
7. In the next box, I am asking you to tell me how many units away from the V peak, is the Ti Kb interfering peak. I want the difference in integer (not decimal) sin T units. To easily do that, click on the black triangle on the appropriate side of the center line (here, right side one), and drag to the top of the Ti Kb peak and let go (it will fall back down). Now read off its position in the far right box “Offset + .00107”. This we now remove decimal and say “+107 sin T units”. Meaning it is 107 sinT units to the right of the center of V Ka. Write “+107” in this box.
8. Click “Periodic Table” in top menu bar, then choose “add new matrix elements” (we’re not, but I need to move back to this window). In the lower left one of the check boxes is “Generate Sum Spectrum”. Check it. Then click the PLOT button in lower right. Toggle the log button off. Now you see the exact replica of what a wavescan of TiO2 with 1 wt% V would be. In the space, draw a rough sketch of this wavescan and be sure to put a vertical mark where the V Ka peak center is.
9. We now want to determine two background positions. Click the Log button to bottom left of the spectrum. You now see better where the peaks tails trail off. Grab the black triangles and move past the peak tails. When I did that I found -648 on the left and +816 on the right. Write these numbers in the 3rd box down.
10. The next box asks, are their any n>1 issues here, for this element and it’s paired element? Are there? Write the proper answer. (Do NOT check the box to show ALL possible interferences).
11. Next, it asks if differential PHA (“PHA ON”) would make any difference here. Write the proper answer.
12. Now repeat steps 3 to 11, this time for LIF. I expect you will see some significant differences here. Again, repeat exactly the same steps as above.
13. Then at the end, in the last box on the bottom, write a summary following the guidelines given.
14. Repeat this for 4 other pairs of elements, and then one of your choosing. Note that while in the first sample you had a first order peak overlap, you will soon encounter higher order peak and/or background complications. When there is a higher order element present, in the upper right, the element will not be in RED but will be in GREEN and have “ho” written next to it.

Problems Assigned:

1. Analytical line: V Ka. You are looking at 1 wt% V in TiO2, which has 59 wt% Ti.
2. Analytical line: W Ma. You are looking at 1 wt% W in a phase with 99 wt% Si.
3. Analytical line: Al Ka. You are looking at 1 wt% of Al in a metal with 49% wt% Ti and 50 wt% Cr .
4. Analytical line: Sr La. You are looking at 0.1 wt% Sr in feldspar which has 26 wt% Si and 7 wt% Ca.
5. Analytical line: F Ka. You are looking at 1 wt% F in a phosphate which has 18 wt% P and 2 wt% Ce. (Here there are 3 possible xtals: just do it for PC0 and TAP)

6. Your chosen compound. Consider different elements and **choose the most problematic element** (interferences)! State all knowns/givens/assumptions.

Fill in the requested information in the attached forms.

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**Some additional comments about Virtual WDS**

If you see a black vertical line that extends above the sin theta axis, that is an absorption edge. If you move the cursor to it, BELOW the sin theta axis, it will display which absorption edge it is. In theory you generally do not want to select a background position for a peak and/or pathological interference of that peak BELOW (lower) than the edge position, although in reality for major elements it is not super critical. For trace elements it could make a difference.

The default PHA setting is the differential (discrimination) mode, with a finite window set automatically under the menu item→Analytical parameters→PHA control. The first order line is black, higher order colored.

A flow chart approach would go like this:

→ Is this a peak or background interference? →If peak, first or higher order? →If higher order, will PHA (mode = differential) lessen (NOT eliminate) the interference?

→If background placement issue, is there any reasonable alternative position that can be used? →If so, what? →If not, is it higher order? →If yes, then will PHA (mode=diff) lessen it?

Latest versions 10/13/08 (new version VWDS 2008) 10/16/08 10/18/09 (New Version VDWS 2013) 3/30/14 3/31/14