Name _____

Lab 11 WDS5 Time Dependent Intensity ("Volatile Elements") in EPMA 4/15/14

The purpose of this exercise is to show that for some samples, there are non-steady state responses of some of the elements in the sample to the electron beam. For many years, people used the term "volatile element" which has lost favor and now is probably more accurately called "time dependent intensity" variation (of the count rate). Mainly it is seen as rapidly dropping Na and K counts in hydrous silicic glasses, and a related "grow in" (slight increase) in Al and Si counts. In today's lab we will investigate three glasses and two minerals whose stage positions should already be entered into the Position database (under Automate):

Std 6018 Corning Haplogranite "Minimum" Melt – Hydrous
Std 6017 Corning Haplogranite "Minimum" Melt – Anhydrous
Std 6010 USGS BIR (Icelandic Basalt Glass)
Std 820 NMNH Anorthoclase (Na-K feldspar mineral)
Std 840 NMNH Kakanui Hornblende mineral

1. Open up Probe for EPMA (PfE), create a 777 file for today, Set up a PfE run, as "Unknown" sample type. We will be using (Analytical Conditions) 15 kV, 20 nA, 0 micron beam diameter.

2. Setup elements on following spectrometers
SP1-TAP – Al Ka (+1600, +1080, average)
SP2-TAP – Na Ka (bkg +1050, -1540, linear)
SP3-PET – K Ka (bkg +452, -443, linear)
SP4- TAP – Si Ka (bkg +1800, +1050, average)
Using the cookbook background positions above. Check your typing! One element per spectrometer.

3. While we are not doing quant, the software is fussy and requires that we pretend will be using standards. Go to the "standard" menu bar at top of PfE window, right click, and select Add/Remove Stds to Run, and double click on 840.

4. We will peak and check PHA on Na and K feldspars.
Use microcline std 217 to peak the spectros and check the PHAs for Ka lines of K, Al and Si; and use albite std 201 to peak and do PHA for Na Ka.

5. Now we will examine the test subjects.
First we will delve into two buttons you haven't seen before:
Acquisition Options: upper right, Spectrometer Motion, change from Asynchronous to Synchronous (this minimizes 'irradiation without counting' time)
And then **Special Options**: change from Normal Acquisition, to Self Calibration TDI Acq., and change the number of TDI count intervals from 5 to 15, and lastly
Under Count Times, change the On-Peak Time from 10 to 30 seconds. Note: we do this to **accentuate the trends (if there are any).**

We will thus acquire for each of the 4 elements of each sample, 15 data points (count rate data), of 2 seconds each, and then look at the count vs time plot. Note that we are NOT doing any quantitative analysis, only using PfE to acquire experimental data. Keep the sample type as "unknown" throughout.

6. Start with 6018, the hydrous glass. Make a new unknown sample with the proper name, and click "start standard/unknown acquisition". When this first spot is finished, move the stage to a spot at least 50 microns away. Acquire another set of data.

   When finished:

   a. Click the Analyze! main button, then click Standard Assignments (this is where Donovan put the TDI), then select the first element. In the bottom right there is a yellow Display TDI Fit button. First click adjacent button for error bars. Note that to the left are two radio buttons to change the fit from Log-Linear (default) to Log-Quadritic ("hyper-exponential") which may be more appropriate for *some* samples (hint).
   b. Click "Display TDI Fit" to view the TDI plot. Play with the radio buttons to get better fit if appropriate. These plots can be printed, or copied to clipboard (for export). You will have 2 overlapping plots for each sample for each element. The point of these TDI fits is to extrapolate back to time=0 to estimate the correct intensity (counts) for elements which change with time.
   c. You will want to create a Word document of plot captures of the TDI plots (for documenting all this, for everyone in the class—rather than drawing sketches as we did before). So…. When you display a TDI plot, and have a nice fit of the data points (log-linear or log-quadratic fit), click yellow "Copy to ClipBoard" button and paste it (with appropriate titling where necessary) into a Word document. Do this for all 4 elements.
   d. Then, still on the sample, in the Analyze! window, click the "Data" button. Move the Analyze! window to the side and observe the "log" window
      --Find the "On-Peak (off-peak corrected) or MAN On-Peak X-ray Counts and the lines below it which have the P-B counts, Average and other statistics, AND then below that the backgrounds = Off-Peak (calculated) X-ray Counts.
      →Do a screen grab of this bunch of data, as you need both these sets of numbers, to reconstruct the **uncorrected** P+B counts, which you will compare with the **TDI-corrected** P+B counts, below.
   e. Now do the arithmetic to determine the time=0 extrapolated counts. The software apparently doesn't output this data readily, so we need to do it by hand[1]. When the TDI fit window is open, determine the ln number at t=0 and use a calculator to do the e^x calculation (on TI 30XIIS calculator, it is 2nd LN button), e.g. for ln of 7.5, e^x is 1080). This is P+B. You will make a table for each sample, with the non-TDI counts, the TDI-corrected counts, and the relative difference when TDI is done. Such as below

	Al	Na	K	Si
6018				
NoTDI	1620	131	375	6486
TDI	1604	245	544	6438
%dif	-1.0%	+187%	+145%	-0.7%

7. Count similarly for 6017, then then 6010, then anorthoclase and finally hornblende, doing 2 spots on each. Thus you will have 20 TDI plots (4 elements x 5 different materials).

[1] Actually, there is a way. Under RUN, then in 4th section down, 'Display TDI …Intensities' where the y-intercept is output. I had forgotten all about this option…

8. Fill out the attached sheet, calculating the % change in the "after TDI correction" relative to the counts outside recorded by the software, without the TDI correction (it just sums up all the counts gathered during all of the counting intervals).

.

Write up a report describing the TDI behavior of the 5 materials, and comment, where appropriate, which of the two TDI curve fits seems to yield the proper extrapolation to time=0. Discuss whether or not the composition and nature (crystalline vs glass) may have something to do with the degree to which some elements migrate under the electron beam.

Version: 4/15/13 4/16/13 4/15/14