

"Getting to know your spectrometers"

The goal of this exercise is to learn which element-lines can be diffracted by the 8 crystals and pseudo-crystals<sup>1</sup> present in our Cameca SX51 electron probe. *We now are working in wavelength units, not energy units.*

1. Using the Bragg equation (assume  $n=1$ ), and the appropriate  $2d$  spacing (in Å) for each of the 7 different crystals below, and the limits of spectrometer movement (between  $\sin \theta$  0.23000 and 0.83000), calculate and record on the attached sheet (**with at least 3 numbers** after the decimal point). **DO NOT USE RADIANS!**

1) the upper and lower wavelengths (Å) each crystal can resolve,

Abbrev	$2d$ (Å)	low Å	high Å
L I F 220	2.848		
L I F 200	4.027		
P E T	8.75		
T A P	25.75		
P C O	44.4		
P C 1	62.1		
P C 2	98.5		
P C 3	204		

2) then using the wavelengths from x-ray tables, determine the extent of (high  $Z$  to low  $Z$ ) elements whose first order  $K\alpha$  lines that correspondent to each crystal, then which element  $L\alpha$ , and then which element  $M\alpha$  lines for each crystal. Don't forget that there are 2 series (Lathanides and Actinides) that are listed "out of order" on the usual Periodic Table graphic display. **Fill in the attached table with the lowest  $Z$  element and the highest  $Z$  element for each of K, L and M lines.**

2. What 2 crystals can resolve Si Ka? \_\_\_\_\_

What 3 crystals can resolve Mn Ka? \_\_\_\_\_

What 3 crystals can resolve F Ka? \_\_\_\_\_

Can Sr Ka be resolved with LIF 200? \_\_\_\_ With LIF 220? \_\_\_\_

3. Each person should now select some multi-element phase that they could end up probing: what is it? (name and chemical composition)

<sup>1</sup> Cameca gives these PC prefixes, and JEOL uses LDE (layered diffraction elements) prefixes. Other descriptions also used: LSM (Layered Synthetic Material) and sometimes even LSD (Layered Synthetic Diffractor).

What one accelerating voltage you would realistically be using? \_\_\_\_\_ kV (think this thru, think “optimal overvoltage for one or more limiting elements”, also involved is which crystal you want to measure what particular lines. There may be multiple options. Think of the ramifications of any.)

Indicate which crystal/s you could use to acquire x-rays for the particular elements, and which line of each element (Ka, La, Ma).

Do not make a laundry list of all possible crystals for all possible lines. FOR EACH ELEMENT, SELECT ONE CRYSTAL. Make sure that each characteristic line can be properly excited. If there is another crystal that might be used, state why one is preferable over the other.

**Make a table below, list each element, which peaks that could be analyzed, and which crystal would be used for each element.** Generally we shoot for Ka, but if we can't excite it, we'll take La, and sometimes even Ma. Recall we can not run over 28 kV. Recall that for (most) geological materials, we do not measure oxygen, rather define it by stoichiometry.

4. On a periodic table which you locate on the internet, or use this one <http://0.tqn.com/d/chemistry/1/0/F/g/1/PeriodicTableEC.png> mark off the elements which each crystal or PC will diffract for WDS. Make separate charts for each family: one for Ka, one for La, one for Ma. Use different colors for different crystals: TAP (red), PET (green) and LIF 200 (blue), and your own selection for the PCs. Then print that out **in color**. MAKE SURE IT IS CLEARLY EASILY LEGIBLE AND COLORS DO NOT MIX OR OVERLAP.

Modifications: 3/12/12 3/2/13 3/7/13 3/13/14