Course 12.141: Electron Microprobe Analysis – Hints and solution for Problem Set 4

(1) Note that in the equations, if “i” is the element being considered, “j” stands for all the other elements in the sample. Our samples have only two elements, Fe and Ni. So, when calculating parameters for Fe, “i” is Fe and “j” is Ni. For example, $S_i$ for Fe will be,

$$S_{Fe} = C_{Ni} * S_{Fe-Ni}$$

where,

$$S_{Fe-Ni} = (\text{const}) \left[ \frac{(2Z_{Ni}/A_{Ni})/(E_0+E_{c(Fe\ K-shell)})}{\ln[583/(E_0+E_{c(Fe\ K-shell)})/J_{Ni}]} \right]$$

See p. 14, Eq 3.12 of Course Notes.

(2) When calculating a parameter for the specimen, you need to find a concentration-weighted average. For example,

$$\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ specimen}} = C_{Fe} * \left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Fe}} + C_{Ni} * \left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Ni}}$$

Note when the concentrations of Fe and Ni are equal, $\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ specimen}}$ becomes a simple average,

$$\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ specimen}} = \frac{\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Fe}} + \left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Ni}}}{2}$$

Also note that the standards are pure metals. So,

$$\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Fe-stand}} = \left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Fe}} = 71.4 \text{ cm}^2/\text{g}$$

$$\left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Ni-stand}} = \left( \frac{\mu}{\rho} \right)_{FeK_{\alpha} \text{ Ni}} = 90 \text{ cm}^2/\text{g}$$

(3) Note that fluorescence occurs only when the energy of the X-ray being absorbed is higher than the critical excitation energy of the atomic shell of the element it is fluorescing. Since,

$$E_{FeK_{\alpha}} \ (6.404 \text{ keV}) < E_c \ (\text{Ni K-shell}) \ (8.332 \text{ keV}),$$

FeK$\alpha$ cannot fluoresce Ni. So the fluorescence correction factor of Ni is equal 1.

(4) Your graphs should show the following:

(A) Atomic number correction deviates more and more from 1 as the composition of the sample deviates more and more from the pure element standards.

(B) There is a high positive absorption correction for NiK$\alpha$ at high Fe contents because NiK$\alpha$ is absorbed in Fe.
(C) There is a high negative fluorescence correction for FeKα at high Ni contents because NiKα fluoresces FeKα.

Following are the graphs you should obtain:

![Graph A](image1)

![Graph B](image2)

![Graph C](image3)

![Graph D](image4)