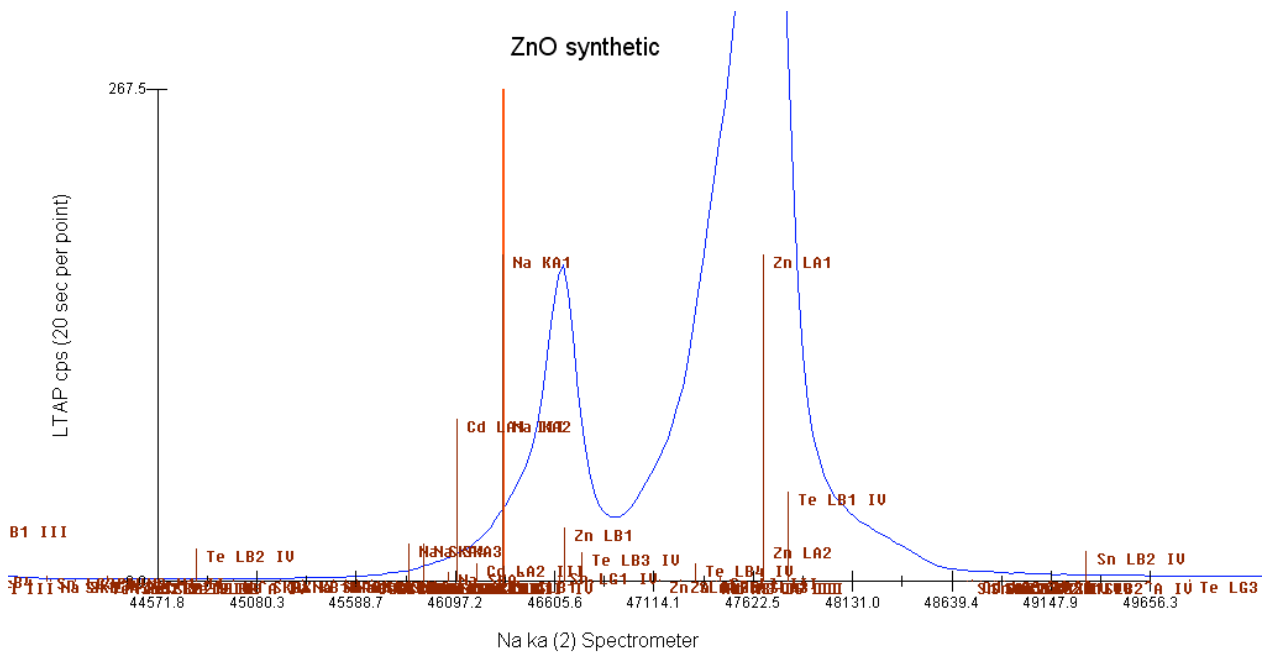
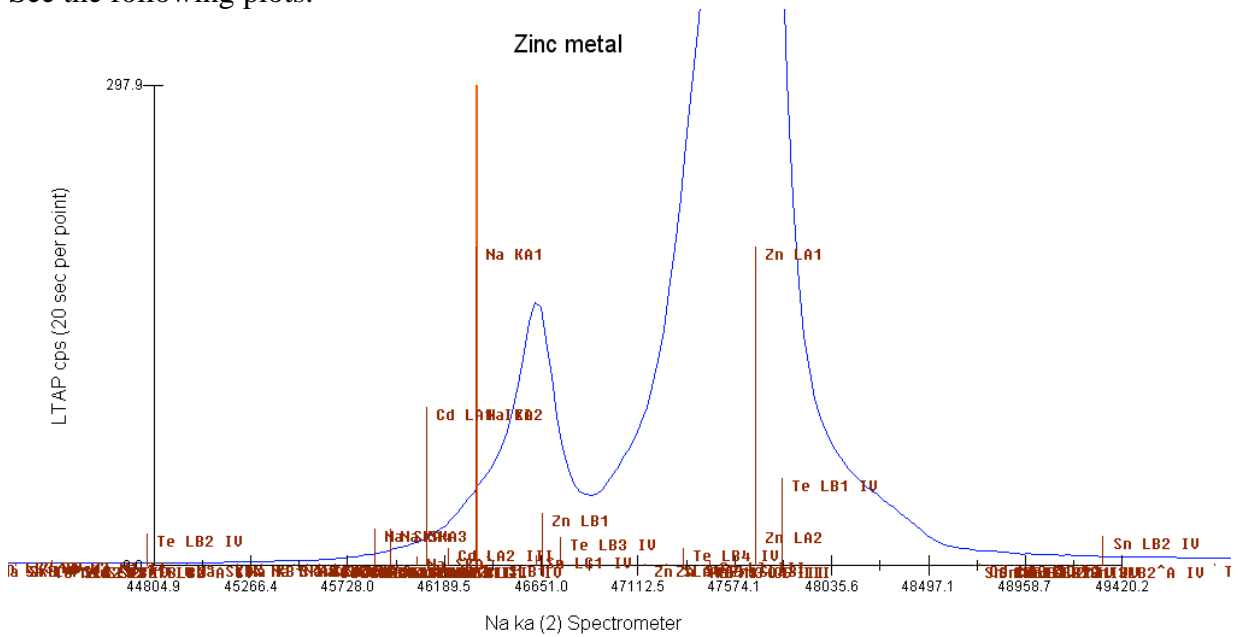


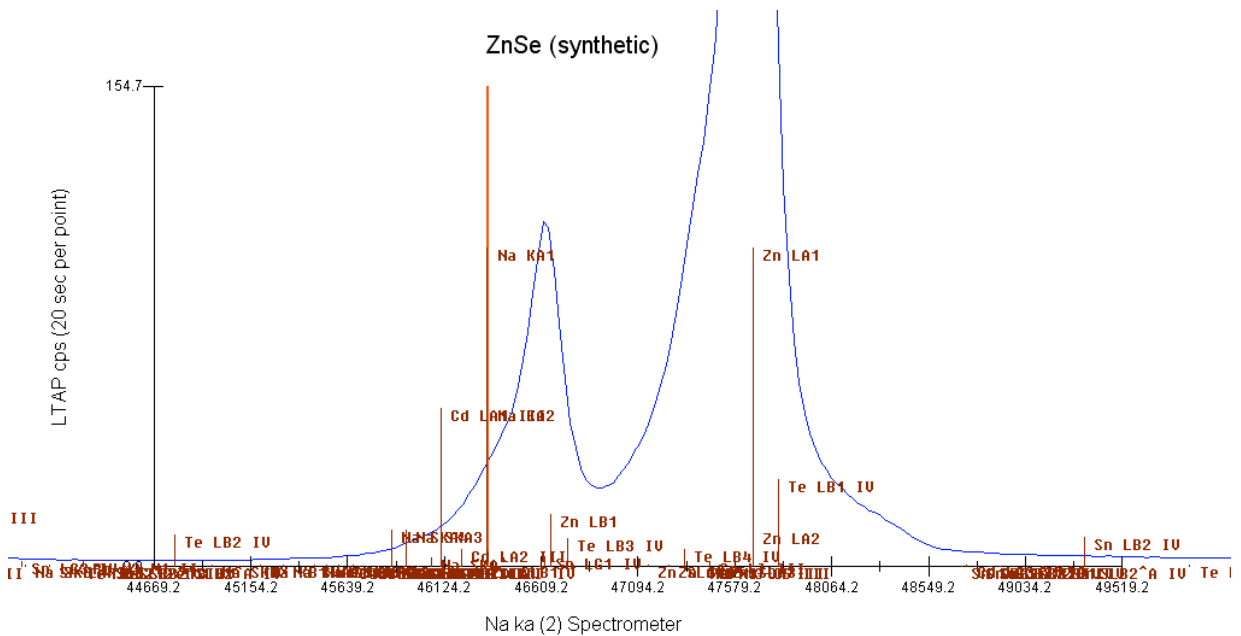
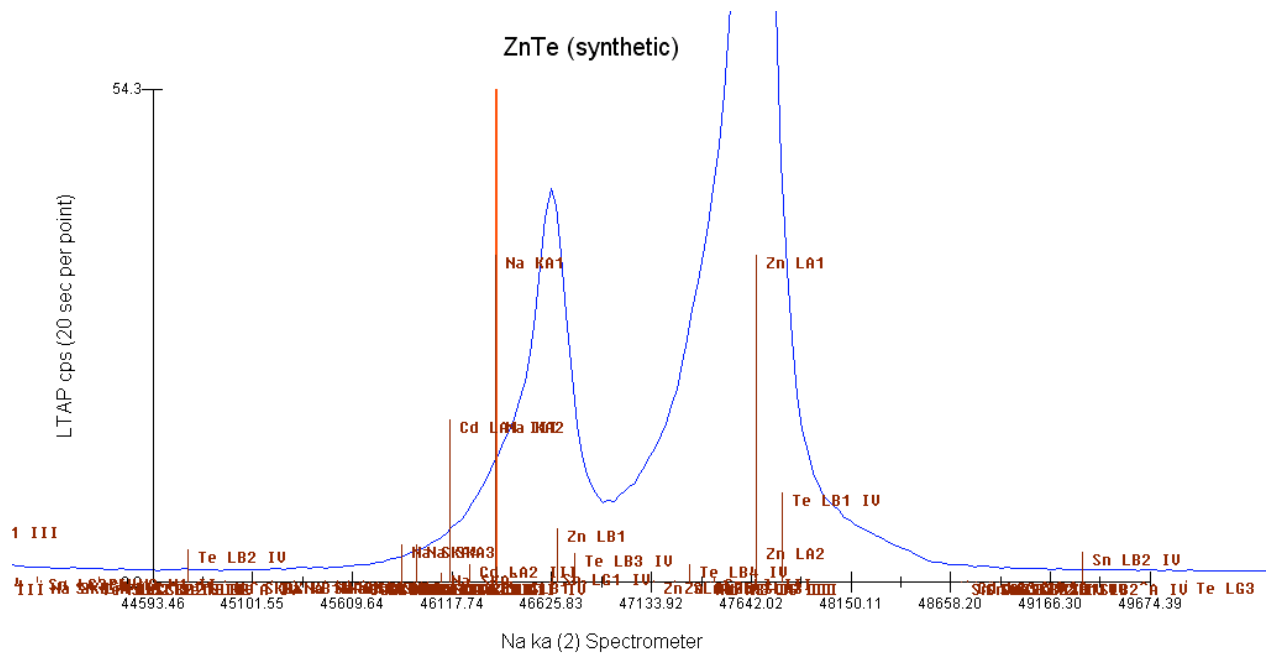
# Measuring Na in the presence of Zn (Zn, ZnO, ZnTe, ZnSe)

04/2011

The following tests attempt to characterize the Na concentration in Zn containing materials that ostensibly do NOT contain any Na. The difficulty is with the interference from the Zn Lb1 emission line, which falls very close to the Na Ka emission line.

See the following plots:

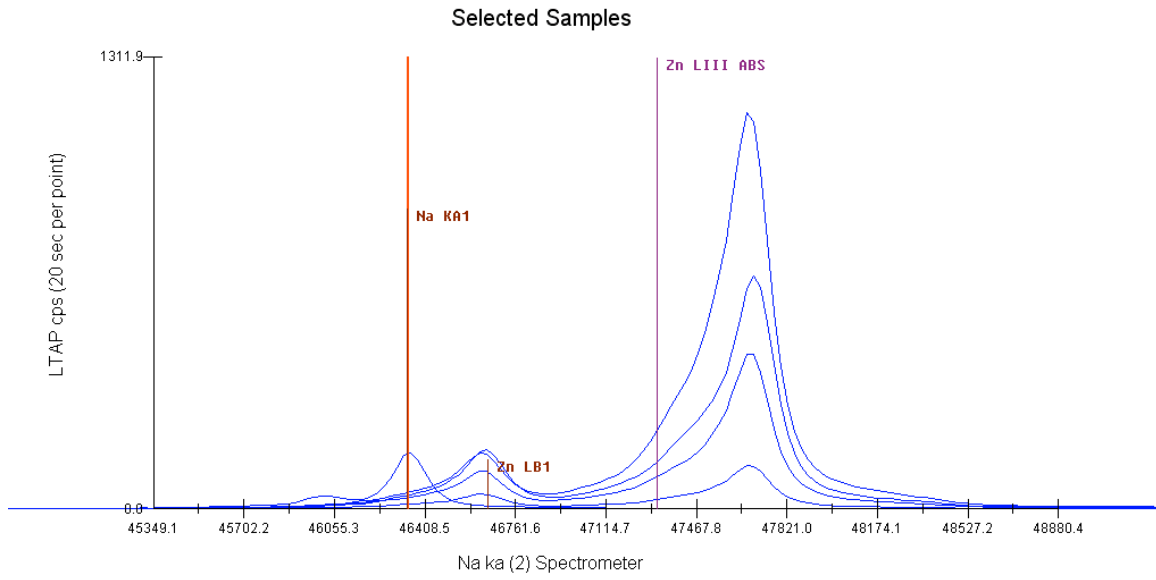




The main dilemma is that due to the fact that the Zn Lb1 transition involves the Zn N shell, which is also the valance shell for Zn, the actual emission energy of the Zn Lb1 line is somewhat dependent of the details of the molecular bonding of the compound of interest.

This can be seen in the following plot which shows the four Zn compounds (Zn, ZnO, ZnTe and ZnSe) and also an Na containing material (nepheline) which has the formula  $(\text{Na},\text{K})\text{AlSiO}_4$  which shows the degree of overlap from Zn Lb1, but more importantly one can see that the ratio of the Zn La1 to the Zn Lb1 emission lines are not consistent

between the compounds due to the presence of the Zn L3 absorption edge (and other possible edges depending on the compound) between the Zn La and Zn Lb emission lines.



The following tables are attempts to ascertain the extent of the quantification problem. All measurements were performed at 20 keV, 30 nA and 20 um beam size. First some basic parameters for Zn and Na at 20 keV.

Matrix correction parameters using FFAST MACs (Henke MAC for Na Ka in Zn of 10500) and Bastin's Proza

| Material | Zn wt% | Zn Ka ZCOR | Zn La ZCOR | Zn Lb ZCOR | Na ZCOR (Zn Ka) |
|----------|--------|------------|------------|------------|-----------------|
| Zn       | 100    | 1.00       | 1.00       | 1.00       | 4.77            |
| ZnO      | 80.34  | 1.06       | 1.28       | 0.99       | 4.58            |
| ZnSe     | 45.29  | 0.904      | 1.09       | 0.65       | 3.34            |
| ZnTe     | 33.88  | 0.913      | 2.53       | 0.94       | 3.93            |

Quant calculations for Zn Ka (no interference corrections)

| Material | Zn wt% | Zn Ka wt% | Na wt% | O wt% | O wt% (publ) |
|----------|--------|-----------|--------|-------|--------------|
| Zn       | 100    | 102.7     | 6.77   | 5.63  | n.a.         |
| ZnO      | 80.34  | 83.06     | 5.52   | 23.32 | 19.662       |
| ZnSe     | 45.29  | 46.50     | 3.89   | 3.89  | n.a.         |
| ZnTe     | 33.88  | 33.21     | 1.49   | 1.14  | n.a.         |

Quant calculations for Zn La (no interference corrections)

| Material | Zn wt% | Zn La wt% | Na wt% | O wt% | O wt% (publ) |
|----------|--------|-----------|--------|-------|--------------|
| Zn       | 100    | 105.8     | 6.78   | 5.63  | n.a.         |
| ZnO      | 80.34  | 74.86     | 5.48   | 23.13 | 19.662       |
| ZnSe     | 45.29  | 45.00     | 2.83   | 3.90  | n.a.         |
| ZnTe     | 33.88  | 27.62     | 1.88   | 1.11  | n.a.         |

Quant calculations for Zn Lb (no interference corrections)

| Material | Zn wt% (publ) | Zn Lb wt% | Na wt% | O wt% | O wt% (publ) |
|----------|---------------|-----------|--------|-------|--------------|
| Zn       | 100           | 96.11     | 6.80   | 6.45  | n.a.         |
| ZnO      | 80.34         | 102.3     | 5.69   | 24.8  | 19.662       |
| ZnSe     | 45.29         | 42.18     | 2.716  | 4.65  | n.a.         |
| ZnTe     | 33.88         | 23.72     | 1.846  | 1.25  | n.a.         |

Quant calculations for Zn Ka (with interference corrections using Zinc metal interference standard for Zn on Na and Zn on O interferences)

| Material | Zn wt% (publ) | Zn Ka wt% | Na wt% | O wt%  | O wt% (publ) |
|----------|---------------|-----------|--------|--------|--------------|
| Zn       | 100           | 100.00    | 0.00   | 0.00   | n.a.         |
| ZnO      | 80.34         | 81.86     | -0.049 | 19.29  | 19.662       |
| ZnSe     | 45.29         | 45.62     | -0.368 | 0.912  | n.a.         |
| ZnTe     | 33.88         | 32.81     | -0.375 | -0.097 | n.a.         |

Quant calculations for Zn La (with interference corrections using Zinc metal interference standard for Zn on Na and Zn on O interferences)

| Material | Zn wt% (publ) | Zn La wt% | Na wt% | O wt%  | O wt% (publ) |
|----------|---------------|-----------|--------|--------|--------------|
| Zn       | 100           | 100.02    | -0.002 | -0.001 | n.a.         |
| ZnO      | 80.34         | 73.71     | 0.478  | 19.56  | 19.662       |
| ZnSe     | 45.29         | 43.84     | -0.279 | 1.032  | n.a.         |
| ZnTe     | 33.88         | 27.73     | -0.045 | 0.141  | n.a.         |

Quant calculations for Zn Lb (with interference corrections using Zinc metal interference standard for Zn on Na and Zn on O interferences)

| Material    | Zn wt% (publ) | Zn Lb wt%     | Na wt%        | O wt%        | O wt% (publ)  |
|-------------|---------------|---------------|---------------|--------------|---------------|
| Zn          | 100           | 99.999        | 0.000         | 0.000        | n.a.          |
| <b>ZnO</b>  | <b>80.34</b>  | <b>106.48</b> | <b>-1.646</b> | <b>18.73</b> | <b>19.662</b> |
| <b>ZnSe</b> | <b>45.29</b>  | <b>42.28</b>  | <b>-0.286</b> | <b>1.478</b> | <b>n.a.</b>   |
| <b>ZnTe</b> | <b>33.88</b>  | <b>23.86</b>  | <b>0.172</b>  | <b>0.351</b> | <b>n.a.</b>   |

Ratio of Zn La to Zn Lb (cps/nA)

| Material    | Zn La        | Zn Lb        | Zn La/Zn Lb  |  |  |
|-------------|--------------|--------------|--------------|--|--|
| <b>Zn</b>   | <b>434.5</b> | <b>59.14</b> | <b>7.34</b>  |  |  |
| <b>ZnO</b>  | <b>247.9</b> | <b>62.97</b> | <b>3.94</b>  |  |  |
| <b>ZnSe</b> | <b>173.4</b> | <b>40.68</b> | <b>4.26</b>  |  |  |
| <b>ZnTe</b> | <b>47.5</b>  | <b>15.24</b> | <b>3.116</b> |  |  |

The above table demonstrates the problem in extrapolating from one Zn compound to another for the purposes of an interference correction when the peak shift/shape factors are significant due to the difference in molecular bonding energies.