



FutMon

Associated Beneficiary 14: Forest Service
DAFF (Ireland)



Hot issues with Aqua Regia!

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Presentation will discuss briefly:

- Use of Blank Subtraction following Aqua Regia digestion
- Effects of digestion temperature on recovery of specific elements



Use of Blank Subtraction following Aqua Regia digestion

Problem:

- It was observed that there was significant variability in the results of the FSCC reference sample for a number of elements.

Solution:

- Upon digestion at 114°C, all samples were diluted 1 in 50 before analysis on the ICP-MS.
- The results from 27 samples determined with and without blank subtraction were compared by plotting on a control chart.
- In addition, the standard deviation for each set of results was calculated.



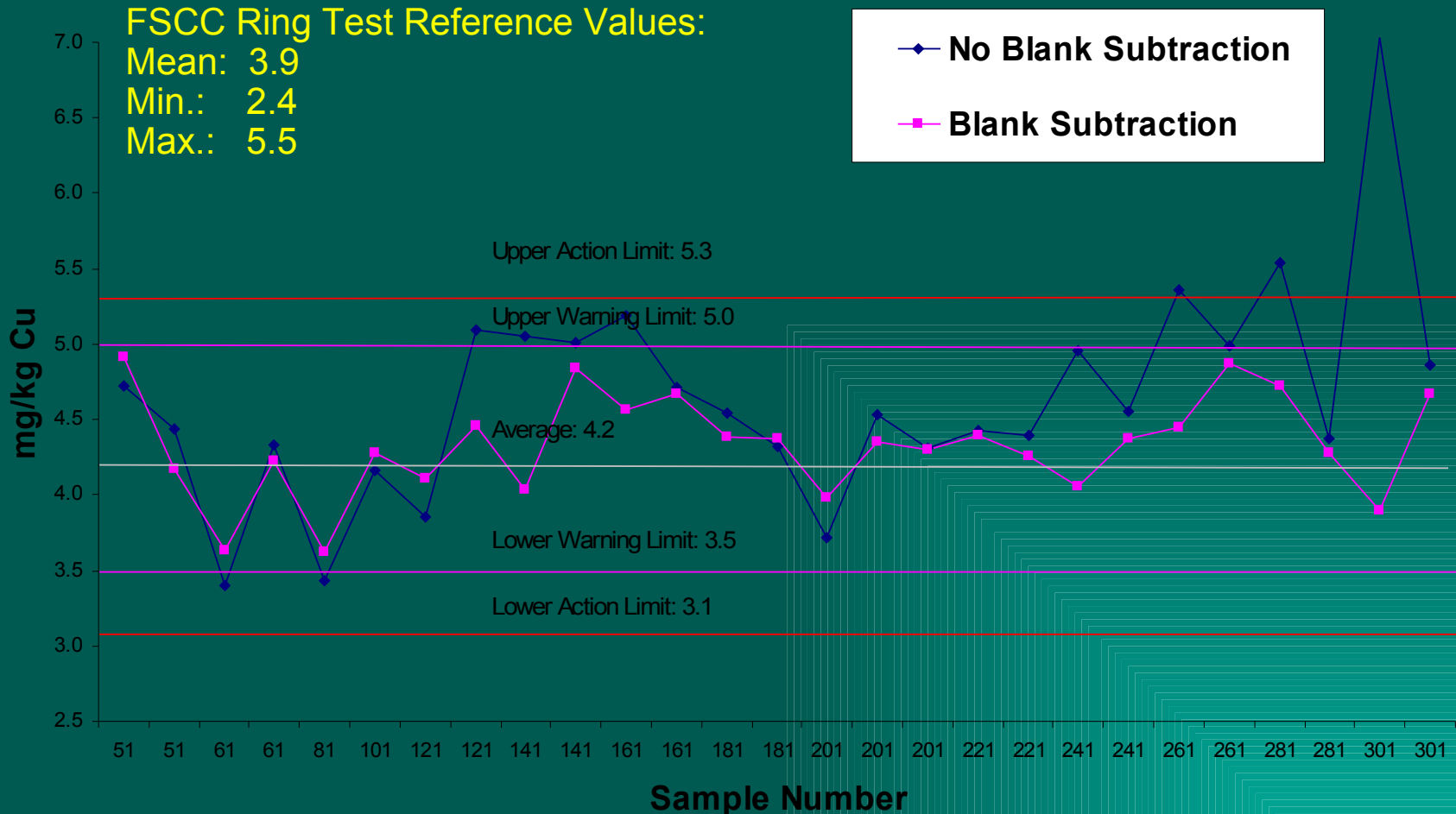
Cu Results for the FSCC Reference Sample

FSCC Ring Test Reference Values:

Mean: 3.9

Min.: 2.4

Max.: 5.5





The effect on Standard Deviation with Blank Subtraction

Element	% Decrease in STD with Blank Subtraction
Ca	2%
Cd	12%
Cu	67%
Fe	0%
K	44%
Mg	50%
Mn	1%
Ni	60%
P	10%
Pb	4%
Zn	3%

Conclusion:

- There was a significant decrease in standard deviation with Cu, Ni, Mg and K.
- Less significant decrease observed with remaining elements.
- Finally, blank subtraction corrected for any contamination present and improved reproducibility of the sample results and was used in all subsequent analysis.



Effects of digestion temperature on recovery of specific elements

Problem

- For Ca, Mg, Zn and K, the initial results from open digestion were below tolerable limits for the FSCC reference sample.

Solution

- Sought advice from other laboratories, namely the Laboratory of Forest Environmental Chemistry (Poland) and the Forestry Commission Laboratory (**UK**).
- Determined that extraction temperature was a possible cause of this problem.
- Raised temperature from 114°C to 160°C

Results Obtained with Increased Extraction Temperature

Temperature	Ca (mg/kg)	K (mg/kg)	Mg (mg/kg)	Zn (mg/kg)
114°C	146.5 $\sigma = 25.4$ $n = 14$	1286.0 $\sigma = 126.5$ $n = 14$	1091.4 $\sigma = 125.5$ $n = 14$	16.6 $\sigma = 0.9$ $n = 14$
160°C	265.1 $\sigma = 29.8$ $n = 13$	1682.3 $\sigma = 61.6$ $n = 14$	1369.0 $\sigma = 48.8$ $n = 14$	18.5 $\sigma = 0.6$ $n = 14$
% difference	101.5%	30.8%	25.4%	11.7%
FSCC Inter-Laboratory Mean	353.6	1640.5	1348.2	20.2

Conclusion:

- A significant increase in the results for Ca and to a lesser extent Mg, Zn and K in the FSCC sample was observed when the extraction temperature was increased from 114°C to 160°C .
- Therefore, an extraction temperature of 160°C was used in all subsequent analysis.



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Thank you for your attention!