

Measure Pb content in cocoa powder and liquor by Z-max



Quantification of Unprecedented Low-level Pb By Monochromatic Excitation XRF

Z-max is a portable monochromatic energy-dispersive x-ray fluorescence analyzer using a monochromatic beam optimized for Pb detection. It delivers unprecedented Pb LOD taking advantage of the superior signal/background (S/B) ratio. The system is also employed a fast silicon drift detector (SDD) tuned to a minimum detector background. It can rapidly survey and quantify low-level Pb in food with no or minimal sample preparation. Figure 1 shows the schematics of the system.

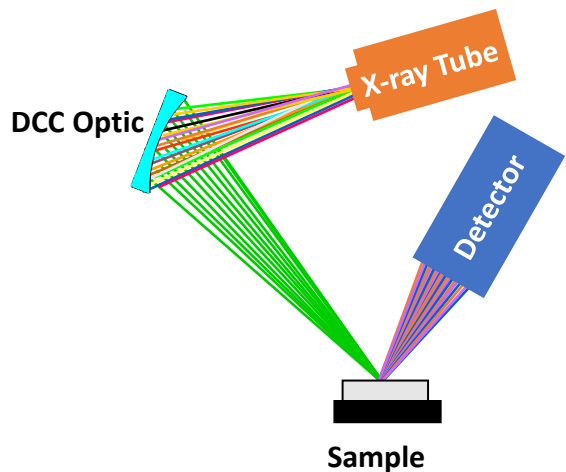


Figure 1. Schematic of monochromatic energy dispersive x-ray fluorescence analysis

Fundamental parameters (FP) approach

Z-max applies the fundamental parameters (FP) approach to calibrate the system, using reference material to refine FP parameters. The FP approach allows JP500 to measure various types of food with one calibration curve. Custom calibration curves can also be established to improve accuracy for specific sample types. Figure 2 shows a Pb calibration in Z-max using standard water base solution. The water-based solution can also be used for food type matrixes.

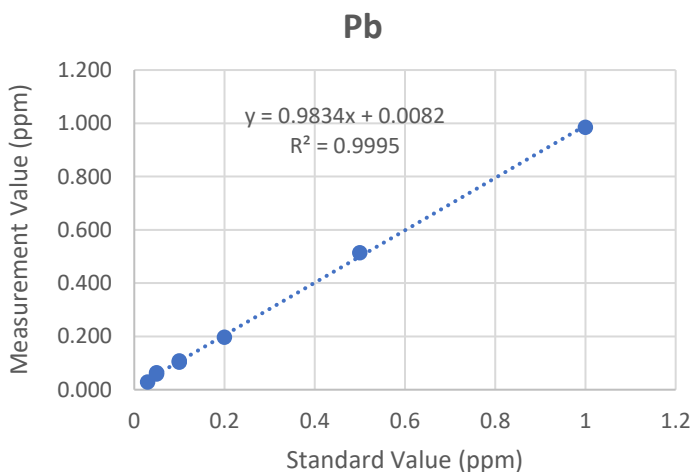


Figure 2. Pb calibration curve in Z-max

Precision and Limit of Detection (LOD)

The critical control level of Pb in chocolate production is between 0.05-0.1 ppm. A cocoa powder sample with Pb level around 0.09 ppm is used to evaluate the precision near 0.1 ppm. Table 1. shows the results of 11 repeats and Table 2. shows the results of two-point average. The limit of detection is about 15 ppb by using the two-point average of 20 minutes measurement.

Table 1. Repeats of single point 1200 s (ppm)

Sample	NIST 2384	Liquor 1	Liquor 2
1	0.040	0.062	0.109
2	0.032	0.079	0.095
3	0.035	0.075	0.095
4	0.026	0.065	0.105
5	0.038	0.070	0.114
6	0.046	0.064	0.116
7	0.038	0.074	0.093
8	0.023	0.073	0.109
9	0.034	0.084	0.107
10	0.027	0.064	0.108
11	0.040	0.057	0.091
SD	0.0071	0.0080	0.0088
Average	0.034	0.070	0.104
Reference Value/ICP	0.0357	0.063	0.11

Table 2. Two-point average 1200s (ppm)

Sample	NIST2384	Liquor 1	Liquor 2
1	0.036	0.071	0.102
2	0.033	0.077	0.095
3	0.030	0.070	0.100
4	0.032	0.067	0.110
5	0.042	0.067	0.115
6	0.042	0.069	0.104
7	0.030	0.073	0.101
8	0.028	0.078	0.108
9	0.031	0.074	0.107
10	0.034	0.060	0.099
SD	0.0048	0.0052	0.0059
Average	0.034	0.071	0.104
Reference Value/ICP	0.0357	0.063	0.11

Correlation with ICP testing

Cocoa powder is obtained and tested by the Z-max. The cocoa powder was well mixed, and three samples are taking from each batch. The average of three sample tests from JP500 is reported. Cocoa liquor samples were heated and stirred before a test. Two samples were taken for each cocoa liquor. The average of the two sample tests is reported. All tests used a 20mins measurement time in Z-max. The same batches of cocoa powder and liquor were also tested using the ICP-MS method. Figure 3 shows the high consistency of results obtained from Z-max and ICP-MS.

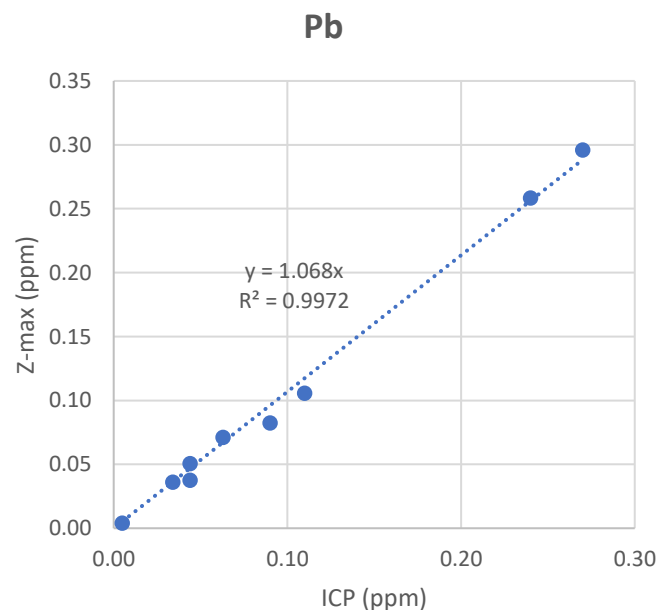


Figure 3. Z-max vs. ICP-MS results