## Analytical Method Changes for Elements in TDS Foods Starting in 2014

In 2014, we updated the methods of measuring elements in foods analyzed for the TDS. Compared with previous methods, the newer methods can detect and differentiate the elements at lower levels. If you're comparing TDS data on elements in foods over time, be aware that an apparent increase in positive findings starting in 2014 might be due to the improved ability to detect them.

The newer methods and instruments have enabled better precision, with lower limits of detection (LOD) and limits of quantification (LOQ) and have reduced interferences by food matrixes. For comparative purposes, the methods used to analyze elements for the TDS, and their LODs and LOQs, are listed in the table below, according to date of use (i.e., before and starting in 2014) and are expressed as ranges. The LODs and LOQs are expressed as ranges, to reflect differences in sample sizes used in digestion procedures, as necessitated by differences in the composition of the various foods included in the TDS.

**Brief Background and Comparison of Previous vs. Newer Methods**

Until 2014, the laboratory used graphite furnace-atomic absorption (GFAA), using a dry-digestion procedure; hydride generation-AA (HGAA), using a wet-digestion procedure; and cold vapor-AA (CVAA), using microwave assisted digestion. Some elements are still measured by inductive coupled plasma atomic emission spectroscopy (ICP-AES), but now use the microwave-assisted process of sample digestion. In 2014, the laboratory changed to methods that use inductive coupled plasma mass spectroscopy (ICP-MS) instrumentation, for analysis, and a microwave-assisted process, for sample digestion.

The microwave-assisted digestion method currently being used is more efficient and requires less time, a smaller sample size, and less acid than did the former digestion procedures. The microwave-assisted digestion process presents fewer safety hazards and reduced potential for sample contamination, compared with the previous methods. In general, these new digestion methods reduce unwanted effects from the food matrix in which the element being analyzed is found. This is due to the food-sample digests being diluted more than they were by the previous methods. The greater dilution mitigates the potential effects of the matrix without a resultant loss in LOD and LOQ, because ICP-MS instrumentation is inherently more sensitive than were the previous methods.

ICP-MS, which measures atomic mass, instead of emissions or light absorption, has several advantages over the previous techniques. In addition to being more sensitive and selective, and less prone to food-matrix interferences, it can be used to measure multiple analytes simultaneously, making it more efficient than previous methods, which measured only a single analyte at a time.

**Summary of LOD and LOQ***

| Element | Pre-2014 LOD (mg/kg) | Pre-2014 LOQ (mg/kg) | Pre-2014 Method | 2014 to Present LOD (mg/kg) | 2014 to Present LOQ (mg/kg) | 2014 to Present Method |
|---|---|---|---|---|---|---|
| As | 0.002 - 0.02 | 0.002 - 0.02 | HGAA | 0.001 - 0.004 | 0.008 - 0.3 | ICP-MS |
| Ca | 0.6 - 4 | 2 - 20 | ICP-AES | 1 - 4 | 10 - 40 | ICP-AES |
| Cd | 0.001 - 0.004 | 0.003 - 0.012 | GFAA | 0.0004 - 0.001 | 0.008 - 0.03 | ICP-MS |
| Cu | 0.05 - 0.4 | 0.2 - 2 | ICP-AES | 0.04 - 0.15 | 0.4 - 1.5 | ICP-AES |
| Fe | 0.2 - 0.9 | 0.5 - 3 | ICP-AES | 0.3 - 1.3 | 3 - 11 | ICP-AES |
| Hg | 0.01 - 0.02 | 0.04 - 0.07 | CVAA | 0.0004 - 0.001 | 0.004 - 0.015 | ICP-MS |
| I* | 0.03 - 0.06 | 0.3 - 0.6 | Colorimetric | 0.03 - 0.06 | 0.3 - 0.6 | Colorimetric |
| K | 2 - 10 | 5 - 40 | ICP-AES | 0.9 - 3.5 | 9 - 32 | ICP-AES |
| Mg | 0.6 - 4 | 2 - 20 | ICP-AES | 0.4 - 1.5 | 4 - 15 | ICP-AES |
| Mn | 0.05 - 0.4 | 0.2 - 2 | GFAA | 0.002 - 0.009 | 0.02 - 0.07 | ICP-MS |
| Mo | 0.2 - 0.9 | 0.5 - 3 | ICP-AES | 0.003 - 0.010 | 0.02 - 0.09 | ICP-MS |
| Na | 2 - 9 | 5 - 30 | ICP-AES | 0.8 - 3.5 | 8 - 11 | ICP-AES |
| Ni | 0.009 - 0.04 | 0.03 - 0.2 | GFAA | 0.005 - 0.02 | 0.05 - 0.2 | ICP-MS |
| P | 2 - 20 | 7 - 50 | ICP-AES | 1 - 4 | 10 - 40 | ICP-AES |

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|---|---|---|---|---|---|---|
| Pb | 0.004 - 0.02 | 0.02 - 0.06 | GFAA | 0.001 - 0.004 | 0.01 - 0.04 | ICP-MS |
| Se | 0.002 - 0.02 | 0.007 - 0.05 | HGAA | 0.002 - 0.008 | 0.02 - 0.07 | ICP-MS |
| Sr | 0.03 - 0.2 | 0.09 - 0.6 | ICP-AES | 0.02 - 0.09 | 0.2 -0.8 | ICP-AES |
| U | Not Analyzed | Not Analyzed | Not Analyzed | 0.007 - 0.003 | 0.006 - 0.02 | ICP-MS |
| V | 0.05 - 0.4 | 0.2 - 2 | ICP-AES | 0.003 - 0.01 | 0.03 - 0.1 | ICP-MS |
| Zn | 0.1 - 0.4 | 0.2 - 2 | GFAA | 0.07 - 0.25 | 0.6 – 2 | ICP-MS |

* The new methods do not apply to iodine until 2016.

The TDS will continue to monitor the development of new technologies that enable efficient detection and differentiation of low levels of analytes in foods.