Heavy Metals Testing by USP

USP<231> Comparison to Instrumental Methods <2232> and <233>

In January 2010, the USP proposed toxicological limits on elemental impurities, which are intended to replace the existing methods in General Chapter <231> Heavy Metals. These new impurity limits are scheduled to be official in September 2013.

Heavy metals, such as arsenic, mercury, cadmium and lead can be hazardous to human health at very low concentrations. This hazard is of greater concern with “sensitive populations,” such as pregnant women and children.

Table 1. Elemental Limits

<table>
<thead>
<tr>
<th>Element</th>
<th>Component Limit(^a) (μg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic (inorganic)(^b)</td>
<td>1.5</td>
</tr>
<tr>
<td>Cadmium</td>
<td>0.5</td>
</tr>
<tr>
<td>Lead</td>
<td>1.0</td>
</tr>
<tr>
<td>Mercury</td>
<td>1.5</td>
</tr>
<tr>
<td>Methylmercury (as Hg)(^c)</td>
<td>0.2</td>
</tr>
</tbody>
</table>

\(^a\) The limits for individual components are based on a maximum daily intake of 10g of a dietary supplement and are intended for use only with Options for Compliance with Limits of Elemental Contaminants under Individual Component Option.

\(^b\) Arsenic may be measured using a nonspeciation procedure under the assumption that all arsenic contained in the supplement is in the inorganic form. Where the limit is exceeded using a nonspeciation procedure, compliance with the limit for inorganic arsenic shall be demonstrated on the basis of a speciation procedure.

\(^c\) Methylmercury determination is not necessary when the content for total mercury is less than the limit for methylmercury. Specific monographs may provide exceptions for articles that may need to be consumed in larger quantities in order to justify the claims.

Disadvantages of the USP<231> Heavy Metals General Chapter

- ~100 year old qualitative method
- Detects only metals precipitated by sulfide ion (Pb, Hg, Bi, As, Sb, Sn, Cd, Ag, Cu, Mo)
- Nonspecific and labor intensive
- Relatively large sample size required (2 grams)
- Extra ashing step for solids and oils may result in significant loss, resulting in false negative results. This can be a particular concern for Hg.

Advantages of Proposed Limits and Procedures <2232> and <233>

- Modern sample preparation methods, such as closed vessel or microwave digestion, are used providing complete dissolution of most solid materials.
- Smaller sample sizes are required to verify absence of impurities due to improved detection limits provided by modern instrumentation.
- ICP-OES and ICP-MS multi-element analysis improves efficiency and lowers client cost.

Columbia Analytical recommends the USP<2232> and USP<233> instrumental methods for the detection of elemental impurities by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS). We have extensive experience analyzing metals in a wide variety of matrices. These instrumental methods produce reliable data at detection levels that easily meet the proposed elemental impurity limits and eliminate the difficulties associated with the bench test USP<231>. All dietary supplements with USP or NF labeling must meet the USP proposed toxicological limits listed in Table 1.

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