Application Note - The Digestion of Lead in Children's Metal Products

Introduction:

This document provides detailed information on a test method that will be used by the U.S. Consumer Product Safety Commission's (CPSC) testing laboratory (LSC) in the analysis of Children's metal products for lead (Pb) content. The method described determines the total lead content of metal items such as, but not limited to, children's metal jewelry.

The general approach is to grind any accessible component part of a sample to a powder; digest completely in a combination of hot, concentrated nitric and hydrochloric acids; and analyze by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES).

Other analytical methods such as Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) and Flame Atomic Absorption Spectroscopy (FLAA) and Graphite Furnace Atomic Absorption Spectroscopy (GFAA) may be used under appropriate conditions as an alternative to ICP-OES using applicable, recognized analytical techniques for the alternative analytical method.

Sample Type:

Paint Chips
Sample weight: 30 - 100 mg

Supplies and Reagents:

1) *Plasma***PURE** Nitric Acid, Trace Metal Grade, (**SCP SCIENCE** cat. # 250-037-175)
2) *Plasma***PURE** Hydrochloric Acid, Trace Metal Grade (**SCP SCIENCE** cat. # 250-037-155)
3) *Digi***TUBE**s, 50ml (glass or plastic)
   (Plastic: **SCP SCIENCE** cat. # 010-500-261; Glass: **SCP SCIENCE** cat. # 010-500-079)
4) Digestion Block (**SCP SCIENCE** *Digi***PREP**®, cat. # 010-500-205 for 24 Tubes or 010-505-205 for 48 Tubes with 010-500-220 or 010-500-225 Controllers & 010-505-115 Temperature Probe)
5) Metal Cutters (Canadian Tire, Home Hardware, etc)
6) *Accu***SPEC** Distilled Water (**SCP SCIENCE** cat. # 250-310-820 for 5 Litres)
7) Rotary Grinder (such as Dremel® Tool, with carbide burr grinders) (http://www.dremel.com)
8) Orbital Shaker (http://www.coleparmer.com/catalog/product_index.asp?cls=5366)
9) CRMs for Leaded Metals (such as NIST SRM 53e, 54d, 1129, etc.)
   (http://ts.nist.gov/measurementservices/referencematerials/index.cfm)
10) *Plasma***CAL** Internal Standard
    (such as **SCP SCIENCE** 1000ug/ml Yttrium, 125 ml: 140-051-391; 250 ml: 140-051-392; 500 ml: 140-051-395)
11) *Plasma***CAL** Lead Calibration Standard
    (such as **SCP SCIENCE** 1000 ug/ml Pb, 125ml: 140-051-821; 250 ml: 140-051-822; 500 ml:140-051-825)
12) QC Standard (**SCP SCIENCE** 100 ml: 140-102-011; 250 ml: 140-102-012; 500 ml: 140-102-015)

Sample Preparation Procedure:

The method calls for two heating cycles which may be conveniently divided into two stored methods using the *Digi***PREP** Touch Screen Controller (**SCP SCIENCE** cat. # 010-500-225). Using the sample temperature DigiPROBE (**SCP SCIENCE** cat. # 010-505-115), automatically control the digestion temperature based on a constant, pre-set sample temperature. This eliminates the requirement to manually monitor and adjust for changing sample temperatures.

*Digi*PREP

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1. If the item is coated with paint or a similar surface coating, the coating should be removed and analyzed separately from the base metal for lead content, as described in the CPSC Standard Operating Procedure for Determining Lead (Pb) in Paint http://www.cpsc.gov/businfo/leadsop.pdf). Care should be taken to remove as little of the substrate metal as possible.

2. Weigh out a 30-100 mg aliquot of a component part in a labelled 50 ml *Digi***TUBE** digestion vessel.

   Component parts of children's products including metal jewelry items generally weigh several grams or more, and an aliquot (with no paint or similar surface coating, but including any electroplated coating which is considered to be part of the substrate) will have to be obtained. Samples should be cut or ground into many small pieces to increase the dissolution rate. If a grinding apparatus (such as a rotary grinding tool with disposable grinding bits) is used, then any contaminated parts must be thoroughly cleaned or disposed of between uses to prevent cross-contamination. Record the actual weight of the aliquot of the ground up component part to the nearest 0.1 mg.

3. In a chemical fume hood, add 8 ml of concentrated nitric acid to each *Digi***TUBE** and evaporate to approximately 3ml on a *Digi***PREP** digestion block at 105 ± 3°C (Sample Temperature @ 95°C with *Digi***PROBE**).

4. After cooling to below 50°C, add 2 ml of concentrated hydrochloric acid and stir.

5. Dilute with distilled water, washing the side of the *Digi***TUBE** , to 20 ml.

6. Warm up the solution to at least 60°C without boiling and gently agitate on an orbital shaker or with a stirrer or shaker bath for a minimum of 4 hours.

7. Dilute to 50 ml with distilled water directly in the *Digi***TUBE** .

8. Dilute samples so that Pb results are within the calibration range of the instrument. Generally a 1:50 dilution is sufficient. However, depending on the Pb concentration, a further dilution using a second *Digi***TUBE** may be necessary.

ICP Operating Procedures and Quality Control Measures Analysis:

1. Ignite plasma. Perform wavelength calibration or torch alignments per instrument manufacturer's recommendations.

2. Allow the instrument to become thermally stable before beginning.

3. Ensure the following element and wavelength are selected in analytical method:
   a. Pb 220.353
   b. One other Pb line, such as Pb 217.00, to ensure spectral interferences are not occurring during analysis.

4. An internal standard such as 2 µg/ml yttrium is used.

5. Perform calibration using Calibration Blank and at least 3 standards appropriate to the concentration of the analyte and instrument, such as 0.25, 0.5, 1.0 and 5.0 µg / ml. Calibration shall be performed a minimum of once a day when used for analysis, or each time the instrument is set up. Results for each standard shall be within 5% of the true value. If the values do not fall within this range, recalibration is necessary.

6. Analyze the Quality Control standard (QCS) immediately after the calibration. The analyzed value of Pb should be within ±10% of the expected value.
   If the Pb value is outside the ±10% limit, recalibration is required.

   At least one laboratory reagent blanks (LRB) must be analyzed with each sample set. If the Pb value exceeds 3 times the

method detection limit (MDL), then laboratory or reagent contamination should be expected. The source of the contamination should be identified and resolved before continuing analyses. The LRBs should be the same acid concentrations as added to the sample, and should be taken through the same digestion procedure but without added sample.

7. At least one certified reference material (CRM) should be analyzed with each batch of samples. The CRM should be similar material as the test specimen with a known amount of Pb. Analyte recoveries should be within ±15% of expected values. If recoveries are outside this limit, the source of the problem should be identified and resolved before continuing analyses.

8. Dilute any samples that have Pb values exceeding 1.5 times the high calibration standard, and reanalyze.

Calculations and Results Reported:

Results for the Pb test methods are calculated and reported as follows:

Total Percentage Pb: % Pb (wt./wt.) = [(C x D) / (W x 1000 µg/mg)] x 100% where:

C = concentration of Pb detected (in units of µg/ml)
D = dilution factor (in ml units)
W = weight of aliquot digested (in mg units)

Example:

A 50 mg aliquot of a component part was digested and diluted to 1000 ml in order to analyze by ICP. The ICP analysis found 20 µg/ml of lead in the solution, which showed that the original component part contained 40% Pb by weight.

	(C)	(D)		(W)	
Component	Concentration Detectoed on ICP (ug / ml Pb)	Dilution Factor (ml)	Total Pb (ug)	Sample wt. (mg)	%Pb
Pendant 1	20	1000	20,000	50	40

References:

[3] Standard Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques
[4] Inductively Coupled Plasma-Mass Spectrometry

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