1 SCOPE

1.1 This method describes a general procedure for the determination of the concentration of lead released from kettles and is applicable to Item 28 of Part II of Schedule I to the Hazardous Products Act (HPA).

2 APPLICABLE DOCUMENTS

2.1 HPA, Schedule 1, Part II, Item 28: Hazardous Products (Kettles) Regulations, SOR91-259
2.2 M. Lanouette, “Determination of Lead Released by Kettles”, Product Safety Laboratory, Project Report 98-0439
2.3 B. Marchand, “Standard Operating Procedures (SOP) for the ICP-MS”, Product Safety Laboratory, Project Report 98-0471
2.4 M. Lanouette “Revision of Method C-09 of Reference Manual Book 5", Product Safety Laboratory, Project Report 2000-0559

3 REAGENTS AND APPARATUS

3.1 Pipetters such as Gilson Pipetman, 100 µL, 200 µL, 1000 µL
3.2 Flame Atomic Absorption Spectrophotometer (FAAS), detection limit 0.050ppm. For lower detection limit use either a graphite furnace atomic absorption (GPAAS) or an inductively coupled plasma mass spectrophotometer (ICP-MS).
3.3 Lead sheet metal (100%) as in house control sample.
3.4 Certified lead (Pb) reference solution, 1000 µg/mL.
3.5 Millipore deionized glass distilled water, pH 6.0-8.0.
3.6 Beaker, 2 liter capacity.
3.7 Nitric Acid conc. 68-70%(w/w) Reagent A.C.S. Fisher Scientific (or equivalent).

4 EXPERIMENTAL PROCEDURE

4.1 Prepare the kettle in accordance with the manufacturer’s instructions, if available. If not, rinse the kettle once with tap water, then twice with glass distilled water using approximately 200mL of water each time.
4.2 Determine the maximum capacity of the kettle by filling it with distilled water and measuring the volume with a graduated cylinder. (Maximum capacity is determined at overflowing).
4.3 Transfer 75% of the maximum capacity of water into the kettle and boil for 15 minutes using the kettle’s element and according to manufacturer’s instructions (for electric kettles). For non-electric ones, use a flat heating element like a hot plate or a domestic stove set at maximum heat in the beginning and reducing the heat by one-quarter when

the water starts boiling heavily to prevent bumping. Start counting boiling time when vapour is observed.

4.4 After boiling for 15 minutes transfer to a graduated cylinder and cool to room temperature.

4.5 Add enough concentrated HNO_3 to make 1% (v/v) of the original volume boiled (75% of the maximum capacity of water).

4.6 Make up to the original volume (75% of maximum capacity) using Millipore deionized glass distilled water pH 6.0-8.0 and transfer to a plastic (PE,PP) bottle.

4.7 Analyse by ICP-MS or Graphite Furnace AA.

5 DETERMINATION

5.1 Prepare a series of at least three aqueous lead working standard solutions of appropriate concentrations by diluting the 1000 µg/mL certified Pb reference solutions in 1% nitric acid as required.

5.2 Prepare a blank solution with Millipore deionized distilled water containing 1% (v/v) conc. HNO_3

5.3 Measure the absorbance of the sample leaching solution, as prepared in section 4, using the AAS operated in flame mode where concentrations are higher than 0.05 ppm. If concentrations are 0.05 ppm or lower, analyse by graphite furnace mode at a wavelength of 217.0 nm or 283.3 nm for lead or by ICP-MS.

5.4 If necessary, dilute the sample leaching solution with Millipore deionized glass distilled water by an appropriate factor in order to ensure that the measurements are taken within the instrument's linear dynamic range.

5.5 Prepare a calibration curve of absorbance versus concentration for lead, using the lead working standard solutions in the appropriate range, and determine the concentration of lead in the test sample solution. Determine the lead content of the blank solution and if present, subtract from the lead content of the sample.

6 REPORTING

6.1 Report the quantity of metallic lead measured directly in the sample leaching solution expressed in terms of mg/L (ppm) for each kettle tested.

6.2 Where lead is not detected, the results of analysis shall be reported as less than or equal to the detection limit of the measurement technique used, or that which is defined in Section 9.1.

6.3 Where applicable, the results of analysis shall be reported according to the following format:

Method	Sample No.	Specimen No.	Vol.(orig)* mL	Conc.Pb mg/L(PPM)
C 09	1	1A	XXXX	X.XXX

*75% of maximum capacity of kettle

7 QUALITY CONTROL PROCEDURE

7.1 In order to ensure the proper operation of the available instrumentation and that the precision and accuracy of the analytical measurements meet the specifications of the method, the following quality control procedures shall be conducted concurrently with the analysis of the test sample.

7.2 The normal and correct operation of the AAS shall be verified according to the following guidelines:

7.2.1 *For flame AAS:* Measure the absorbance of a 10 µg/mL Pb working standard solution.

7.2.2 *For graphite furnace AAS:* Measure the absorbance of 10 µL of a 0.10 µg/mL Pb working standard solution.

7.2.3 Record the absorbance of the working standard solutions in the analytical instrument's QC logbook, and verify that the measurements are within the tolerance limits of the expected values. If these control measurements fall within acceptable limits, a note shall be entered in the test sample file to the effect that the instrument calibration was found to be "within control". Should the instrument be found in a state of disrepair or out of calibration, the instrument shall immediately be repaired and/or recalibrated to meet the prescribed operating conditions prior to proceeding with the analysis.

7.3 The normal and correct operation of the test method shall be verified according to the following guidelines:

7.3.1 Measure 1000 mL using a 1L volumetric flask of Millipore deionized glass distilled water pH 6.0-8.0 into a 2 liter glass beaker. Bring the water to a boil at maximum heat on a hot plate and add a weighed 2 cm × 2 cm lead sheet (100% Pb previously washed with acetone/methanol). Reduce heat by 1/4 turn to prevent bumping and maintain boiling for 15 minutes. Ensure that the metal sheet is not floating at the surface of the water during the boiling period. After 15 minutes remove the lead sheet from beaker, transfer water to a 1000mL volumetric flask rinsing the beaker and cool to room temperature. Add 10 mL concentrated HNO_3 to have 1% HNO_3(v/v) of the original volume and bring volume up to the 1000mL using Millipore deionized glass distilled water pH 6.0-8.0 .

7.3.2 Determine the concentration of lead in the solution. Record the test result in the analytical instrument's QC logbook, (kettle section) and verify that the result is within the tolerance limits of the expected value. If this control sample result falls within acceptable limits, a note shall be entered in the test sample file to the effect that the test method was found to be "within control". Should the test result of the control sample be found to fall outside the specifications of the method, the entire analytical procedure shall be repeated, providing a fresh test sample is available [1].

8 PRECISION AND BIAS

8.1 The deviation between replicate test results, as obtained by the same analyst on two different days with the same apparatus under constant operating conditions on identical test material, should, in the normal and correct operation of the test method, meet or exceed a 30% repeatability limit at a 95% probability level for a nominal test value of 0.38 µg/mL leachable Pb (as determined in a 2 cm x 2 cm lead sheet in-house control containing 100% Pb). (Project # 98-0439)

8.2 The accuracy of the test results obtained by this method cannot be statistically determined as the result is applicable only to an individual sample article, and is dependent upon the ability to obtain truly representative test sample replicates.

8.3 The property being measured can be defined only in terms of the experimental conditions of the test method. This method therefore has no bias since the measured amount of leachable lead is defined only in terms of this method.

9 LIMIT OF DETECTION

9.1 The limit of detection of this method, as determined by ICP-MS is 0.3 ppb for Pb based on the method ASTM D4210. (Project # 98-0471)

Note 1: In view of the destructive nature of this determination on the sample's test surface, once an analysis has been conducted the same sample article cannot be retested. A repeat analysis may therefore be conducted only on a fresh test article.