

6.5 Appendix - Answer Key

6.2.1 Qualitative Identification of Lead in Ceramicware and Solder Alloys Using Test Kits

1. **Which ions interfere with the test for leachable Pb in ceramicware and Pb in solders?** In principle, other than Pb, Sn and Ba form colored complexes with the rhodizonate salt. The Sn complex is purple. The Ba complex is yellow-orange. Both are persistent and can obscure a positive response to Pb. Sn interference is not a practical issue for ceramicware because Sn is not utilized in ceramic pigments or glazes. However, BaO is used as a base in a number of glazes. Most Ba-based glazes yield a matte finish. If the Ba is leachable, one will get a reaction to Ba. For solders, Sn is a serious but a manageable problem, since most solder alloys have a high percentage of Sn. However, the limit of Pb detection in a Sn-Pb solder ranges from 10-20% Pb. The precautions in the method (e.g. cleaning the surface in order to remove oxides with silicon carbide paper and dabbing the swab rather than rubbing with the swab) minimizes the Sn interference.
2. **What are the principal chemical reactions (no chemical formula are required) embodied in these test methods and kits?** There are three reaction steps that need to be identified: (1) oxidation of the Pb, if the lead is in the metallic state, (2) chelation of the Pb by the food acid (citrate or tartrate) employed by the kit, and (3) the formation of the color complex, Pb-rhodizonate.
3. **How would one utilize each test in selecting ceramicware items for quantitative analysis?** For ceramicware, any positive result on a decoration or glaze on the food-bearing surface would trigger a sample collection. The collection of cups, mugs, or pitchers would be based on a positive test of either the QCT or RAT.

6.2.2 Leachable Lead and Cadmium in Ceramics by Flame Atomic Absorption Spectrometry

1. **For the brand and model of instrument utilized, demonstrate the steps employed to obtain maximum sensitivity for the two elements, and to eliminate or minimize interferences.** This "question" calls for the analyst to physically demonstrate the set-up of the instrument. The steps that need to be demonstrated are as follows: proper positioning of the burner and proper flame characteristics, in order to achieve optimal sensitivity; optimizing the light source, lamp, slit, wavelength; and optimizing the nebulizer to achieve the specified sensitivity.
2. **What are the possible sources of interference?** Interferences arise from the sample matrix. They may be physical or chemical in nature. They can be due to high levels of certain other elements that cause non-specific absorption or can be caused by changes in the atomic species of the element of interest. Interferences can be due to spectral overlap, but these are rare for FAAS, at least for these elements. They may be caused by physical differences (e.g. sample viscosity) in the analytical test solution that alter the "delivery" of the analytes into the aerosol and flame.
3. **How does an instrument minimize or reduce these interferences?** Explain how the particular instrument in the laboratory accomplishes background correction (deuterium lamp, optics, and electronics).
4. **What steps could one use to correct background interference?** Aside from the instrumental techniques, some or all of the following may prove useful: sample dilution, standard additions,

suppressors, different wavelength, etc.

5. **What measures of instrumental performance verify instrumental “calibration”?**
Characteristic concentration, detection limit, quantitation limit, linearity, and range of linearity.
6. **Identify and give the acceptable range for other method quality control measures that are utilized to assure accuracy and precision of the analysis?** Characteristic concentration: within 20% of specification. Precision of 10 replicate determinations of a midlevel standard yields a RSD of $\leq 5\%$. Sample concentration limit: absorbance reading of 0.05 equal to or less than 1.0 mg Pb/liter and 0.1 mg Cd/liter. Result of ICS 95-105% of expected. Recovery of leachate spike 80-110% of expected. No carry-over of analyte between samples. No quantifiable Pb or Cd in method blank.
7. **Why are samples stored in the dark during leaching with 4% acetic acid?** Leaching ceramicware in the presence of light causes accelerated cadmium leaching.