Experiment 19: ICP-MS Determination of Lead

SYNOPSIS

The isotopic composition of lead in a sample, as well as the quantitative amount of lead in a sample is determined by Inductively Couple Plasma Mass Spectrometry.

READINGS p. 330-331 in Critical Reviews

INSTRUMENTS

ICP-MS
Peristaltic pump

Solutions

0.1% NITRIC ACID
Internal standard:
(monitors instrumental drift in counts per second (cps)): 50 ppb Th, V,Sb
Lead standards
10, 50, 100, 200 ppb in 0.1% nitric acid, with 50 ppb internal standard (thorium, vanadium, antimony)
Sample:
Your final volume should be no more than 0.1% nitric acid. Higher acidities destroy the sampling cone. Your final volume of your digestate should be at least 10 mL because of the inefficient nebulaization of the sample.

Procedure

Specific instructions come with the instrument. Generally you will do the following:

1. Tune the intensity of the plasma
2. Tune the quadrapoles by setting the voltages of the extraction and collection plates which pull the cation ions from the plasma into the high vacuum system.
3. Calibrate the mass sensitivity of the instrument so that a certain voltage fluctuation in the quadrapole will select for a certain mass. The internal standard solution is used for this step.
4. Set up an acquisition method for your sample. For isotope ratioing you will want to peak hop to acquire data as fast as possible to avoid the flicker of the plasma changing from one lead isotope peak to another. For quantitative analysis, peak scanning mode is useful.
5. Acquire spectrum with internal standard and rinses between each acquisition.

REPORT: In addition to materials, methods, and results your report should include the following information.

1. Report the relative standard deviation of the multiple digestates.

2. What is the LOD, LOQ, linear range and r value of your standard curve for this method? How does your LOD compare with the expected value?
3. Convert the ppm of your LOD to ppm in your sample (soil, paint, etc.)
4. Did your internal standards for the instrument (Th, V, and Sb) show your technique to be reliabel? What is the purpose of these standards?
5. Did your sample internal standards come out to be reliable?
6. Do you attribute all the variation in your measurements to either the ICP-MS and the digestion process, or must you consider the soil and the types of lead present in the soil?
7. What is the ultimate possible detection limit of this instrument (compare the cps for one of your standards and its %rds).
8. What is the relative standard deviation for your measurement of 3 ppm Pb?
9. What are the main background peaks in this measurement?
10. Why do you get Gaussian shaped peaks?
11. What is the rsd of the determination of the isotope ratios? How might this be important in litigation over paint samples?
12. Why can you get such great LOD with this method. Hint, how long does it take to acquire data ona single peak?
13. What is the main instrumental error?
14. How does the sample matrix affect the atomization process?
15. What was the estimated time for turn around in samples?
16. Are there any problems with disposal of hazardous materials?
17. How easy would it be to instruct a technician on this method?
18. How easy would it be to construct a paper trail for this method?