A4 – DETERMINATION of HEAVY METAL CONTENT of FALL OUT by ELECTROTHERMAL ATOMIC ABSORPTION SPECTROPHOTOMETRY

Preworke
1. Outline the procedures for cleaning and working with glassware when using EAAS
2. Why are solutions made up in acid rather than just ultrapure water?

SAFETY
Extreme caution must be exercised when working with hydrofluoric and perchloric acids.

4.1 Introduction
Heavy metals are a major health problem, particularly when they are associated with airborne particulates, as this means they can be readily transported into the lungs and stomach and directly absorbed into the body. These substances accumulate in the body and eventually may lead to severe health problems such as dementia and growth retardation, even at relatively low doses. For this reason, health authorities are continually revising the acceptable level of lead particulates in the atmosphere and reducing the value.

In this practical you will use the electrothermal atomic absorption spectrophotometer to determine the level of lead in our fall out sample. Other methods such as conventional atomic absorption spectroscopy do not normally provide the sensitivity to conduct successful determinations of lead as the levels are too low.

4.2 Procedure
Part 1 – Sample preparation
1. Dry your fall out sample in an oven for at least an hour at 105°C.
2. Carefully weigh 0.1-1.2g of your sample in duplicate into a platinum crucible.
3. Wet the solution with a few drops of water then add 1mL perchloric acid (72%) and 5mL hydrofluoric acid (48%)
4. Place a lid on the crucible and heat at 200-225°C until the evolution of white fumes ceases. Do not take to dryness. If all solids are not dissolved consult the teacher.
5. Add 10mL of modulab water then 5mL of HNO₃ (5M Aristar) and gently boil the solution until dissolution is complete.
6. Quantitatively transfer the contents of the crucible to a 100mL volumetric flask with washing of the crucible and lid, then make the volume up to the mark.

Part 2 – Standard preparation
1. Using the stock solutions in the laboratory prepare a 1000µg/L Pb stock solution in 1M HNO₃. (Note: EAAS works in the ppb (µg/L) range! Do not prepare the normal ppm solutions which are used in other analysis.
2. Using the stock solution prepared in the previous step, prepare a 20µg/L Pb standard solution.
3. Prepare a 0.5% ammonium dihydrogen phosphate solution. This is the chemical modifier.
Part 3 – Analysis

1. Place the solutions prepared in the steps above in the autosampler as instructed by the teacher.
2. Under supervision run the solutions on the EAAS and obtain a print out of the results.
3. Report the concentration of Pb in your sample on a dry weight basis in appropriate units.

4.3 Questions

1. What is the purpose of adding HF and HClO₄ to the samples?
2. Suggest some potential sources of Pb in particulate pollutants in this area.

4.4 References

1. Methods for Graphite Tube Atomic Absorption Analysis – Varian
2. Advanced Instrumental Techniques Notes