AA Determination of Lead: Standard Addition Method

Apparatus Atomic Absorption Spectrometer, 25 ml volumetric flasks (8), Micropipette

Chemicals 500 ppm solution lead (standard-stock), de-ionized water

Procedure:

Prepare the laboratory sample as follows; paint about a 4"×4" area of a glass or plastic sheet provided. Let it dry in air for a few minutes in the fume hood. Use a blow drier to hasten the process. Make sure to dry the paint well, if not it tends to form lumps at the point of scraping.

Scrape the dried paint sample with a blade and weigh *accurately* about 0.7 - 0.9g of the dried paint sample. Transfer the weighed sample into a 150mL beaker carefully. Extract the lead present in it by *gently* boiling in ~50mL of 0.05M nitric acid solution for about 20 min.; keep the beaker covered with a watch glass. Cool the acid extract to room temperature. Filter into a 100 mL volumetric flask *quantitatively*. Dilute it to the mark. This is your unknown solution.

Prepare a 50ppm solution as the *working standard* solution from the standard stock solution in a 100mL volumetric flask.

Prepare a series of solutions (25.00mL - in 25mL volumetric flasks) containing the same volume (5.00mL) of Pb^{+2} unknown solution, but different volumes *of the working standard* of Pb^{+2}, varied in a systematic fashion as shown in the table below. Mix all the solutions well.

Volume of unknown (mL)	5.00	5.00	5.00	5.00	5.00	5.00	5.00	5.00
Volume of standard addition (mL)	0	1.00	2.00	3.00	4.00	5.00	6.00	7.00

Verify the atomic absorption spectral line to be used for the monitoring of Pb.

Run the Perkin Elmer Atomic Absorption Spectrometer; see the instructions to set up the instrument. Use option B (see below).

Measure the absorbance of the set of solutions made above, (set the program to read the average of three replicates measurements per sample) starting with the solution containing the lowest amount of the analyte, see operating instructions of the AA spectrometer.

Clean up and return all glassware (you may use a few milliliters of acetone for drying if necessary).

Plot the observed absorbance vs. the volume of standard *additions*. From the best-fit line equation of the plot (focusing in the linear region of the plot), calculate the concentration of Pb in the 'first undiluted unknown' solution prepared.

Express the concentration in units of ppm and mol/L of Pb in the *first undiluted unknown* solution. Calculate the weight percent of lead in the dried solid sample (% w/w).

Perkin-Elmer 1100 Operating Procedure (Option A)

Turn ON instrument (Green On/Off)

Element Select Enter Z Element (Pb) -automatic
Enter Current Lamp Current (10mA) and Mode - automatic

Setup automatically sets to wavelength, Energy 60-80, slit and lamp current.
BG-Corr

Gain optimizes electronics (any time)

Instrument Calibration
Program Using arrow Keys to move around the different fields if necessary
Set # replicates to 3, Enter
Set standard concentration units Mg/L, and sample units Mg/L
Enter standard value (a single standard) Enter
Continuous
Open Air (58 psi)
Open acetylene tank (12-15 psi)
Flame ON
Setup Examine optimization indicators, Gain if necessary
Run
Introduce Blank (Millipore water) , wait ~20 s, press *Auto-Zero*
Std 1-
Introduce (aspirate) standard 1, wait ~20 s, STD 1
.
.
Main Keys
Display Data

Measuring Samples
Introduce sample, wait ~20s
Read
Repeat Read to make sure the preceding reading is correct, record absorbance (mean of triplicate measurements)
Introduce the blank, wait ~10s, *Auto-Zero* and repeat the preceding three steps

Shut Down Procedure:

Run Millipore water (2 min)
Flame OFF
Atom Ctrl

Acetylene - close
Air close

Check Gases
Check F & O (press as many times as need to reduce the pressure to zero)
Turn OFF instrument

Perkin-Elmer 1100 Operating Procedure (Option B)

Turn ON instrument (Green On/Off)

Element Select Enter Z Element (Pb) -automatic
Enter Current Lamp Current (10mA) and Mode - Flame

Setup automatically sets to wavelength, Energy 60-80, slit and lamp current.
BG-Corr

Gain optimizes electronics (any time)

Instrument Calibration
Program Using arrow Keys to move around the different fields if necessary
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