JoVE: Science Education

Lead Analysis of Soil Using Atomic Absorption Spectrometry (AAS)

--Manuscript Draft--

Manuscript Number:	10021
Full Title:	Lead Analysis of Soil Using Atomic Absorption Spectrometry (AAS)
Article Type:	Manuscript
Section/Category:	Manuscript Submission
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Science Education Title Lead Analysis of Soil Using Atomic Absorption Spectrometry (AAS)

Overview

Atomic absorption spectrometry is based on the absorption of discrete wavelengths of light by ground-state, gas-phase free atoms. Atoms of different elements absorb characteristic wavelengths of light. A hollow cathode lamp (fig 1) is used to emit light with the specific frequency that can be absorbed. The energy absorbed excites the electrons in the target element from their ground-state to a higher-energy state. The amount of light absorbed is proportional to the concentration of the element in the sample. Using a standard curve, the concentration of the element in the sample can be determined.

AAS is an elemental analysis technique that can provide quantitative information on over 70 different elements. Concentrations as low as ppt (parts per trillion) can be determined for some elements, with ppb (parts per billion) and ppm (parts per million) being more common for various metals. This method has several benefits over others. For example, this technique measures the total concentration of an element, regardless of its form. In addition, the wavelength used is specific to the element being tested, so there will be no interference from other elements in the sample, making it a fast and easy technique.

Principles

Lead occurs naturally in soil in levels ranging from 50 – 400 ppm. However, with the widespread use of lead in paint and gasoline, in addition to contamination by industry, urban soils often have concentrations of lead significantly greater than background levels – in some places up to 10,000 ppm. Lead does not biodegrade, but remains in the soil.

Serious health risks are associated with lead poisoning. Children are particularly at risk, and exposure can lead to developmental and behavioral problems for them. The Environmental Protection Agency has set a standard for lead in soil at 400 ppm for play areas and 1200 ppm for non-play areas.

Lead in soil is also of concern when it is soil that is to be used for gardening. Plants take up lead from the soil. Therefore, if one eats vegetables or herbs grown in contaminated soil, lead can be ingested. Contaminated soil particles can also be breathed in while gardening or brought into the house on clothing or footwear. It is recommended that soils with lead levels greater than 400 ppm not be used for gardening. It is further recommended that soil with lead levels between 100 and 400 ppm not be used for leafy vegetables or herbs due to the fact that lead can be stored in the leaves. Root vegetables should also not be grown in this soil due to the fact that lead can accumulate in plant roots as well.

Procedure

1. Soil Preparation

1.1. Mix sample thoroughly and sieve using a USS #10 sieve.

1.2. Dry soil in an oven at 40°C for 24 hours.

2. Sample Digestion

2.1. Using an analytical balance, weigh out 1g of soil sample and place in a digestion tube. Record the weight of the sample to four decimal places.

2.2. In a hood, add 5 mL of water to the digestion tube.

2.3. Add 5 mL of concentrated HNO_3 to the digestion tube.

2.4. Mix the slurry with a stirring rod. Cover the digestion tube with a tear drop glass stopper.

2.5. Put the digestion tube in the block digester and heat the sample to 95°C and reflux for 10 minutes without boiling. REMEMBER- THIS CONTAINS CONCENTRATED ACID.

2.6. Allow the tubes to cool to room temperature. Add 5 mL of concentrated HNO_3 to the digestion tube, replace the drop glass and reflux for an additional 30 minutes. If brown fumes are generated, repeat this step over and over until no brown fumes are given off by the sample.

2.7. Evaporate the solution to a 5 mL volume without boiling.

2.8. Allow the tubes to cool. Add 2 mL of distilled water and 3 mL of 30% H_2O_2. Cover with the glass stopper and heat to begin the peroxide reaction. Be careful that the solution does not boil over. Heat until the bubbling stops and allow to cool.

2.9. Continue to add 30% H_2O_2 in 1 mL increments with warming until the bubbling is minimal. Do not add more than a total of 10 mL of the 30% H_2O_2.

2.10. Cover the sample with the glass teardrop stoppers and heat until the volume is reduced to 5 mL without boiling.

2.11. Add 10 mL concentrated HCl to the sample and cover with the glass teardrop stopper. Heat to 95°C and reflux for 15 minutes.

2.12. Allow the tubes to cool. Filter the sample if there are particulates using filter paper (or similar) and collect filtrate in a 100 mL volumetric flask. Dilute the sample volume to 100mL with distilled water.

3. Analyzing Samples on Atomic Absorption Spectrometer

Comment [DM1]: Have told authors we'll need screen capture (non-still) for all of this.

3.1. Turn on the computer and the spectrometer.

3.2. Set parameters on instrument:

    3.2.1 Acetylene pressure > 700 kPa (~100 psi)

  - 3.2.2 Acetylene valve set to 11 psi
  - 3.2.3 Air valve set to 45 psi

3.3 Open the SpectraAA software.

3.4. Open a new worksheet in the software.

3.5. Choose “Add Method” and click on Pb to do a Lead Analysis.

3.6. Set Type/Mode parameters:

  - 3.6.1 Type = Flame
  - 3.6.2 Element = Pb
  - 3.6.3 Sampling Mode = Manual
  - 3.6.4 Instrument Mode = Absorbance
  - 3.6.5 Flame Type = Air/Acetylene
  - 3.6.6 Air Flow = 13.5
  - 3.6.7 Acetylene Flow = 2.0
  - 3.6.8 Online Diluter Type = SIPS

3.7. Set the Measurements parameters:

  - 3.7.1 Measurement Mode = PROMT
  - 3.7.2 Calibration Mode = Concentration
  - 3.7.3 Times: Measurement = 10
  - 3.7.4 Times: Read Delay = 10
  - 3.7.5 Replicates: Standard = 3
  - 3.7.6 Replicates: Sample = 3
  - 3.7.8 Precision (%): Standard = 1.0
  - 3.7.9 Precision (%): Sample = 1.0

3.8. Set the Optical parameters:

  - 3.8.1 Lamp Position = Ca #4
  - 3.8.2Lamp Current (mA) = 10.0 mA
  - 3.8.3 Wavelength = 422.7 nm

3.8.4 Slit = 0.5 nm

3.8.5 Background = BC Off

3.9. Set the SIPS parameters:

3.9.1 Nebulizer Uptake Rate = 5.0 mL/min

3.9.2 Right Pump = none

3.9.3 Standard Additions = Unselect

3.9.4 Calibration Mode = Auto Set Std Concentrations

3.9.5 Dual Pump Calibration = Unselect

3.10. Under the Standards tab, a list of standards will automatically populate for the particular test.

3.11. Exit the Edit Method menu and click on the “Labels” tab. Input information regarding sample names and number of samples.

3.12. On the “Analysis” tab, use the “Select” button to highlight the samples to be analyzed.

3.13. Turn on the flame by pressing the ignite button on the instrument.

3.14. Zero the instrument by aspirating a blank and pressing the “Alt” and “Read” keys simultaneously.

3.15. Place the pump tubing in the blank solution and press “Start.” Once the calibration has been performed, place the pump tubing in the sample and press the “Read” key. Continue for all samples.

3.16. Turn off the instrument by pressing the red power off button on the instrument. Turn off all gas tanks and remove all samples.

Representative Results

The software will create the calibration curve (figure 2) and automatically determine the concentration of the Pb in the samples.

The values given on the worksheet are mg/L of Pb in the sample *solution*. Additional calculations must be done to convert this number to the ppm of Pb in the *soil* sample.

Example:

For a soil sample that weighed 1.2523 g before digestion and was measured by the AAS to have 6.0 mg/L of Pb in the 100 mL solution sample.

$$\underline{100\ \text{mL solution}} \times \frac{6.0\ \text{mg Pb}}{\text{L solution}} \times \frac{1\ \text{L}}{1000\ \text{mL}} = 0.6\ \text{mg Pb}$$

$$\frac{0.6\ \text{mg Pb}}{1.2523\ \text{g soil}} \times \frac{1000\ \text{g}}{1\ \text{kg}} = \frac{479\ \text{mg Pb}}{\text{kg soil}} = 479\ \text{ppm}$$

Applications

Atomic Absorption Spectrometry is a useful technique to analyze a wide-range of environmental samples (e.g. water, soil, sludge, sediment) for a large number of elements (e.g. heavy metals). This experiment highlights the use of flame AAS to determine the Pb content in soil (table 1). However, it could also be used to measure concentrations of Cu, Fe, Mn, K, Na, Mg, and Zn in soils.

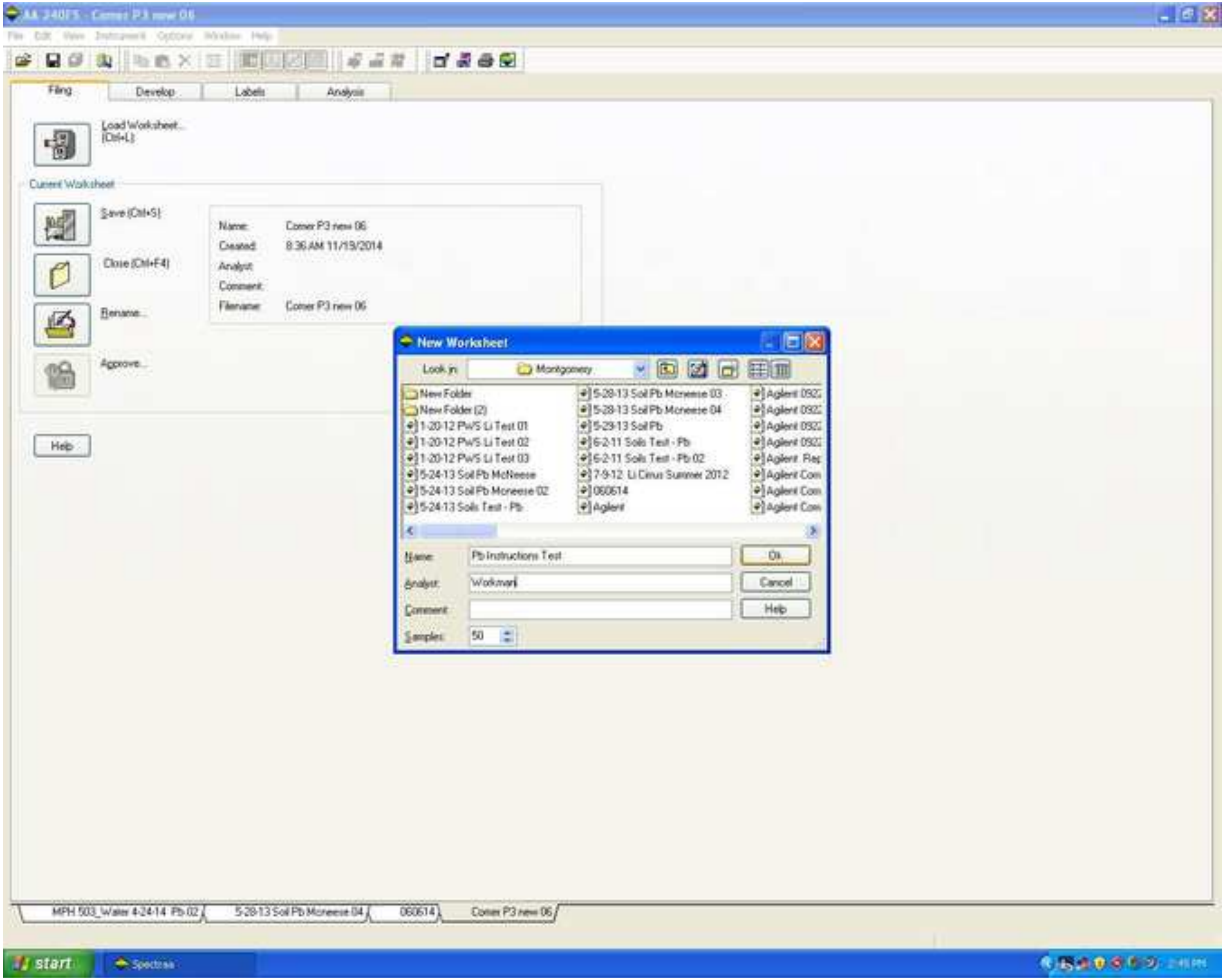
Atomic Absorption Spectrometry can also be used on non-environmental samples, including:

- Water analysis (Ca, Mg, Fe, Al, Ba, Cr)
- Food analysis (Cd, Pb, Al, Cu, Fe)
- Additives in oils (Ba, Ca, Na, Li, Zn, Mg, V, Pb, Sb)
- Fertilizers (K, B, Mo)
- Clinical samples (blood, serum, plasma, urine, Ca, Mg, Li, Na, K, Fe, Cu, Zn, Au, Pb)
- Cosmetics (Pb)
- Mining (Au)

Legend:
Figure 1: Hollow cathode lamps from an atomic absorption spectrometer

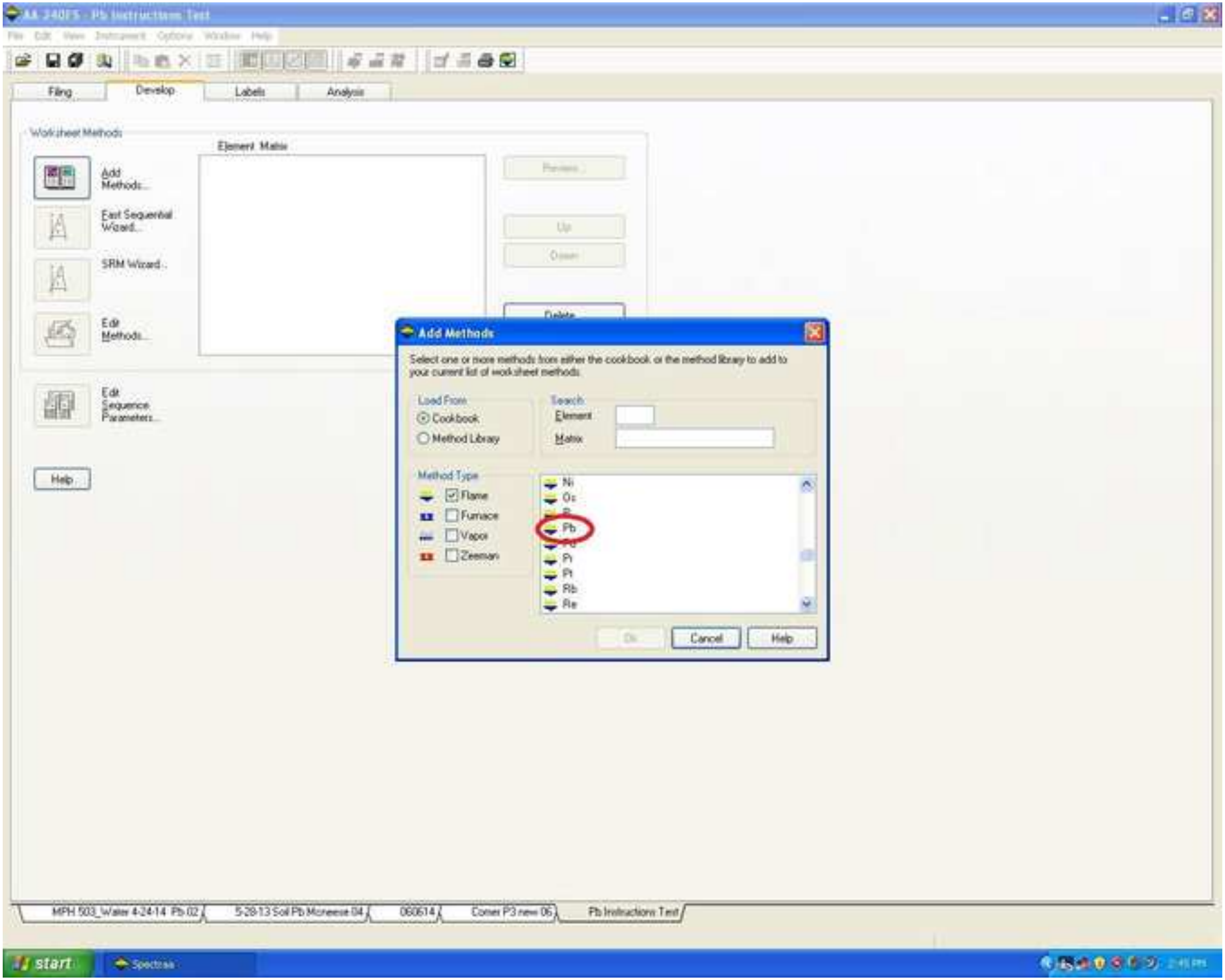
Figure 2: Calibration curve


Internet Explorer
Windows Media Player
Google Desktop
HP Photosmart Essential 2.01
HP Solution Center
Roxio Creator Home
Shop for HP Supplies
CHE265_Pb1
Help and Support
start



Name: Corner P3 new 06
Created: 8:36 AM 11/19/2014
Analyst:
Comment:
Filename: Corner P3 new 06

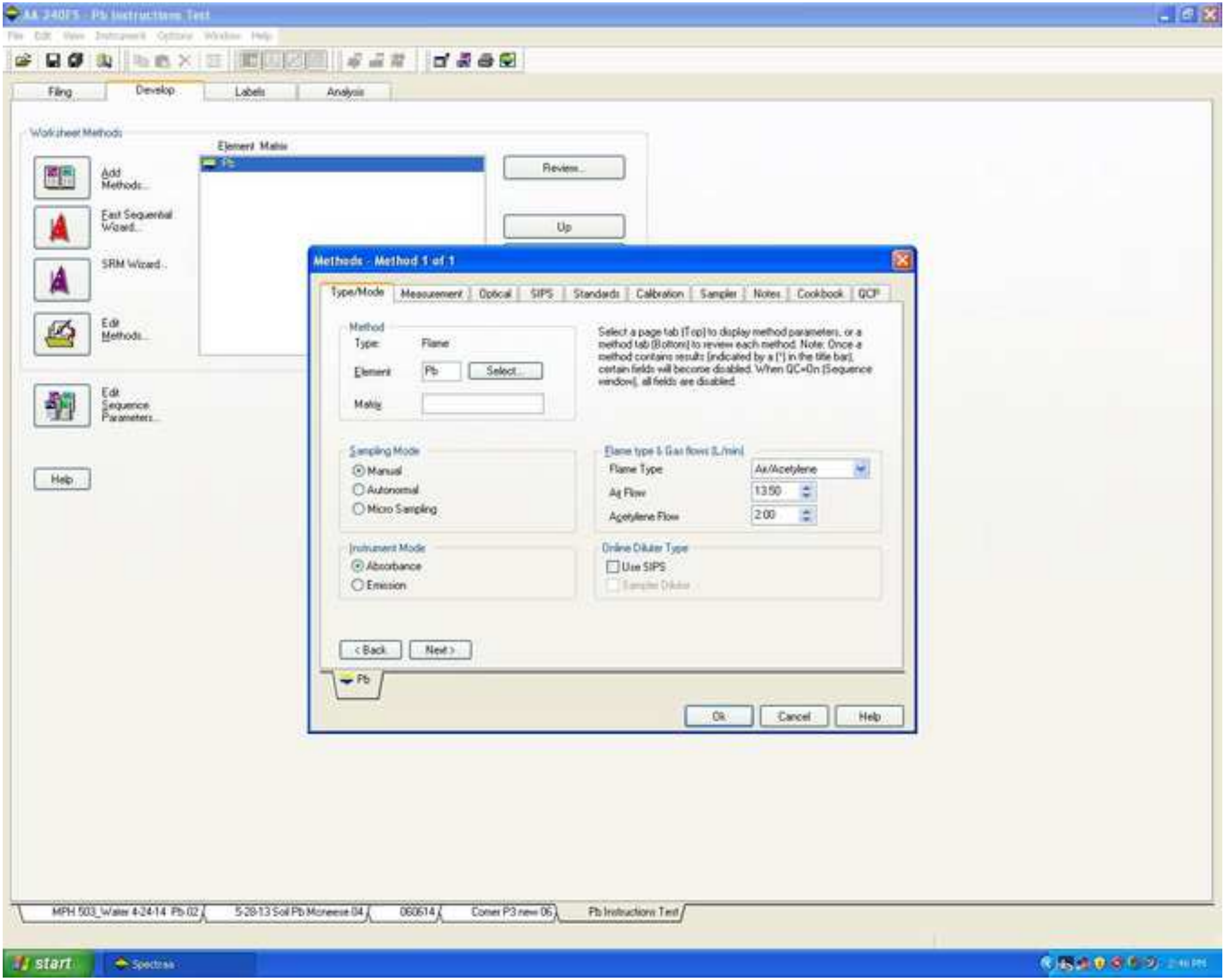
Name: Pb Instructions Test
Analyst: Workman
Comment:
Samples: 50



Select one or more methods from either the cookbook or the method library to add to your current list of worksheet methods.

Load From: ● Cookbook ○ Method Library

Method Type: ☑ Flame ☐ Furnace ☐ Vapor ☐ Zeeman



Method

Type: Flame

Element: Pb

Matrix:

Select a page tab (Top) to display method parameters, or a method tab (Bottom) to review each method. Note: Once a method contains results (indicated by a (*) in the title bar), certain fields will become disabled. When QC=On (Sequence window), all fields are disabled.

Sampling Mode

● Manual

○ Autonormal

○ Micro Sampling

Flame type & Gas flows (L/min)

Flame Type: Air/Acetylene

Air Flow: 13.50

Acetylene Flow: 2.00

Instrument Mode

● Absorbance

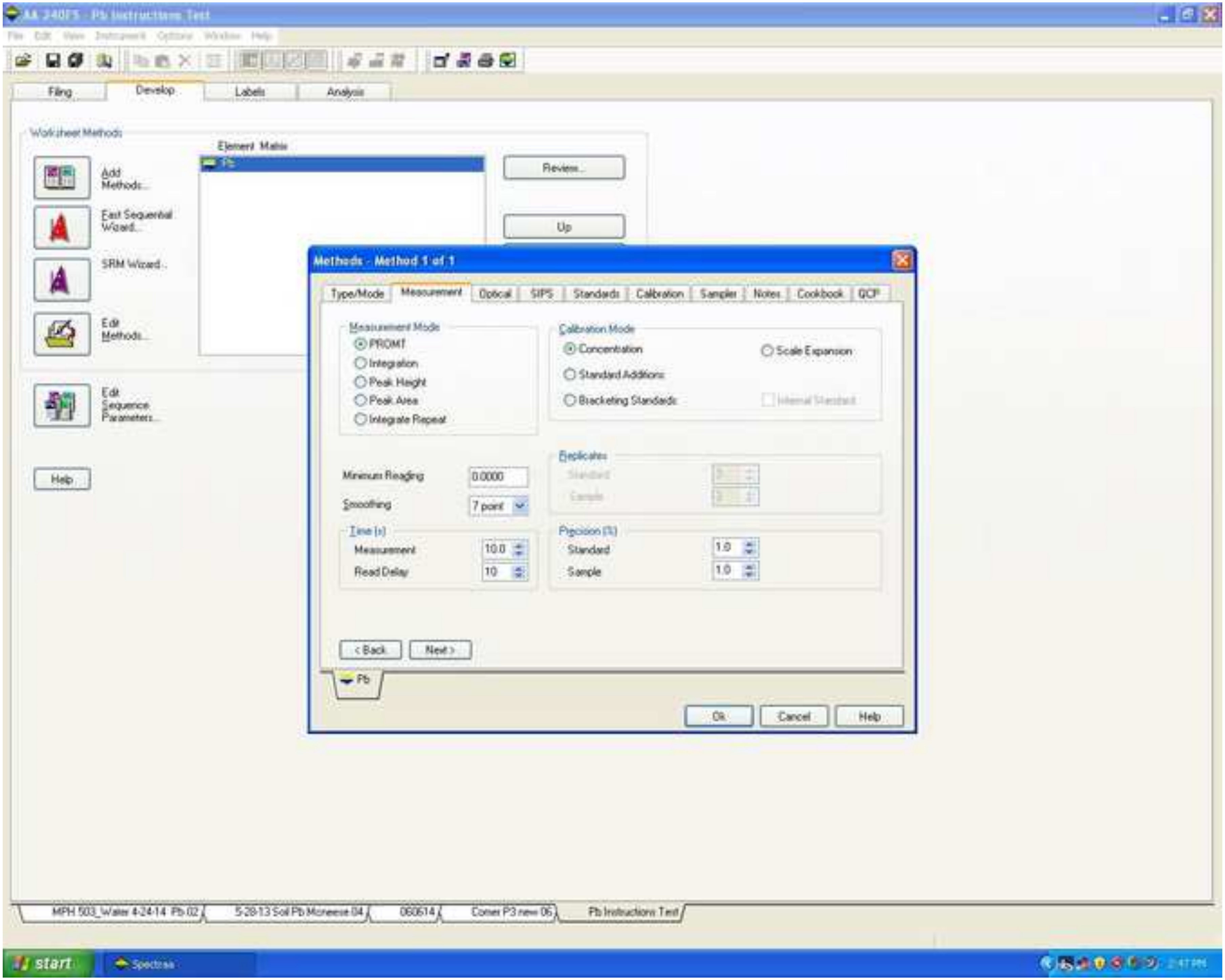
○ Emission

Online Diluter Type

☐ Use SIPS

☐ Sampler Diluter

Click here to download Photo or Graphic File: Frye and Workman_Lead Analysis of Soil Using AAS_Figure 6.jpg



Methods - Method 1 of 1

Measurement Mode
- ● PROMT
- ○ Integration
- ○ Peak Height
- ○ Peak Area
- ○ Integrate Repeat

Calibration Mode
- ● Concentration
- ○ Scale Expansion
- ○ Standard Additions
- ○ Bracketing Standards
- ☐ Internal Standard

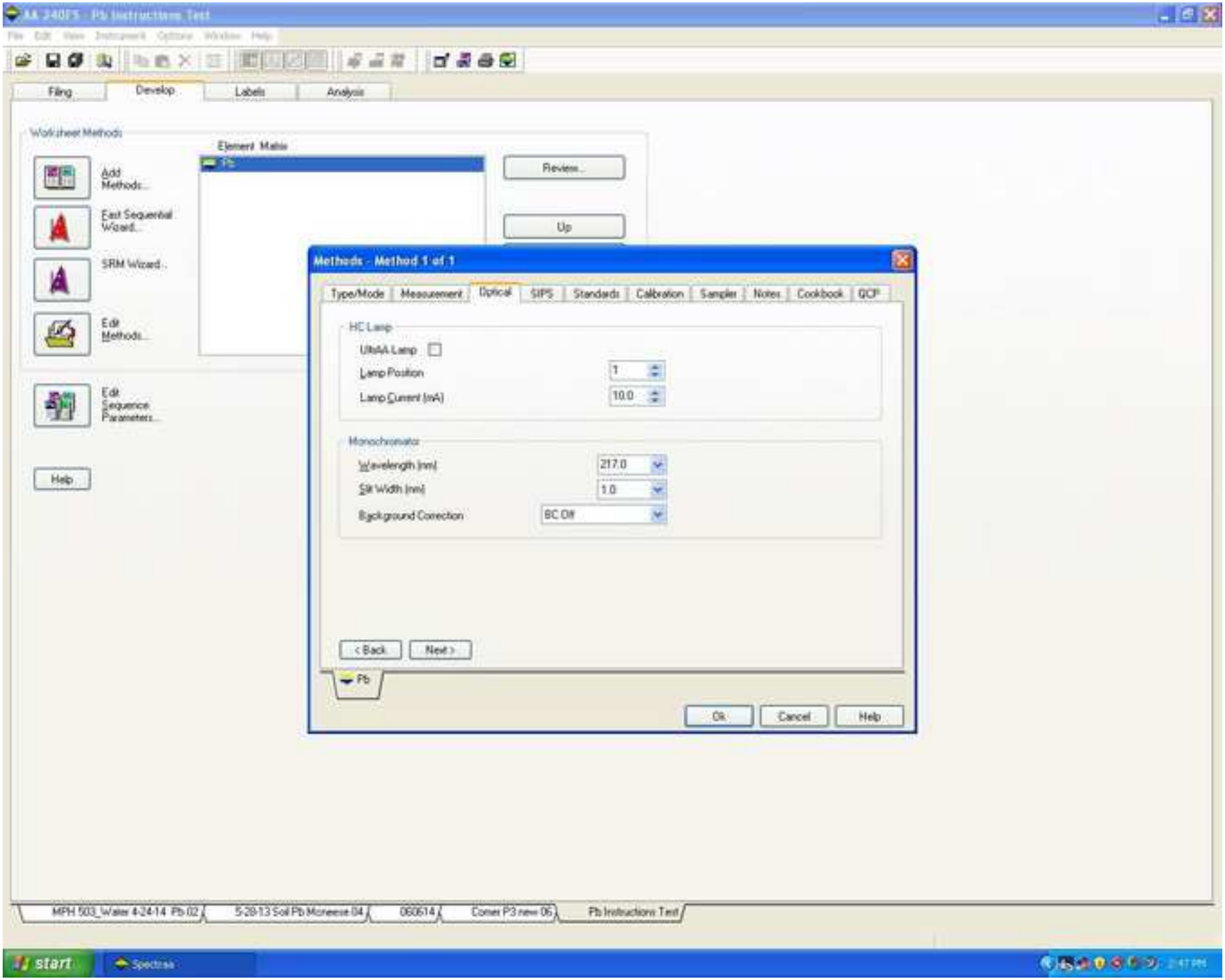
Minimum Reading: 0.0000

Smoothing: 7 point

Time (s)
- Measurement: 10.0
- Read Delay: 10

Replicates
- Standard
- Sample

Precision (%)
- Standard: 1.0
- Sample: 1.0



HC Lamp

UltrAA Lamp ☐

Lamp Position: 1

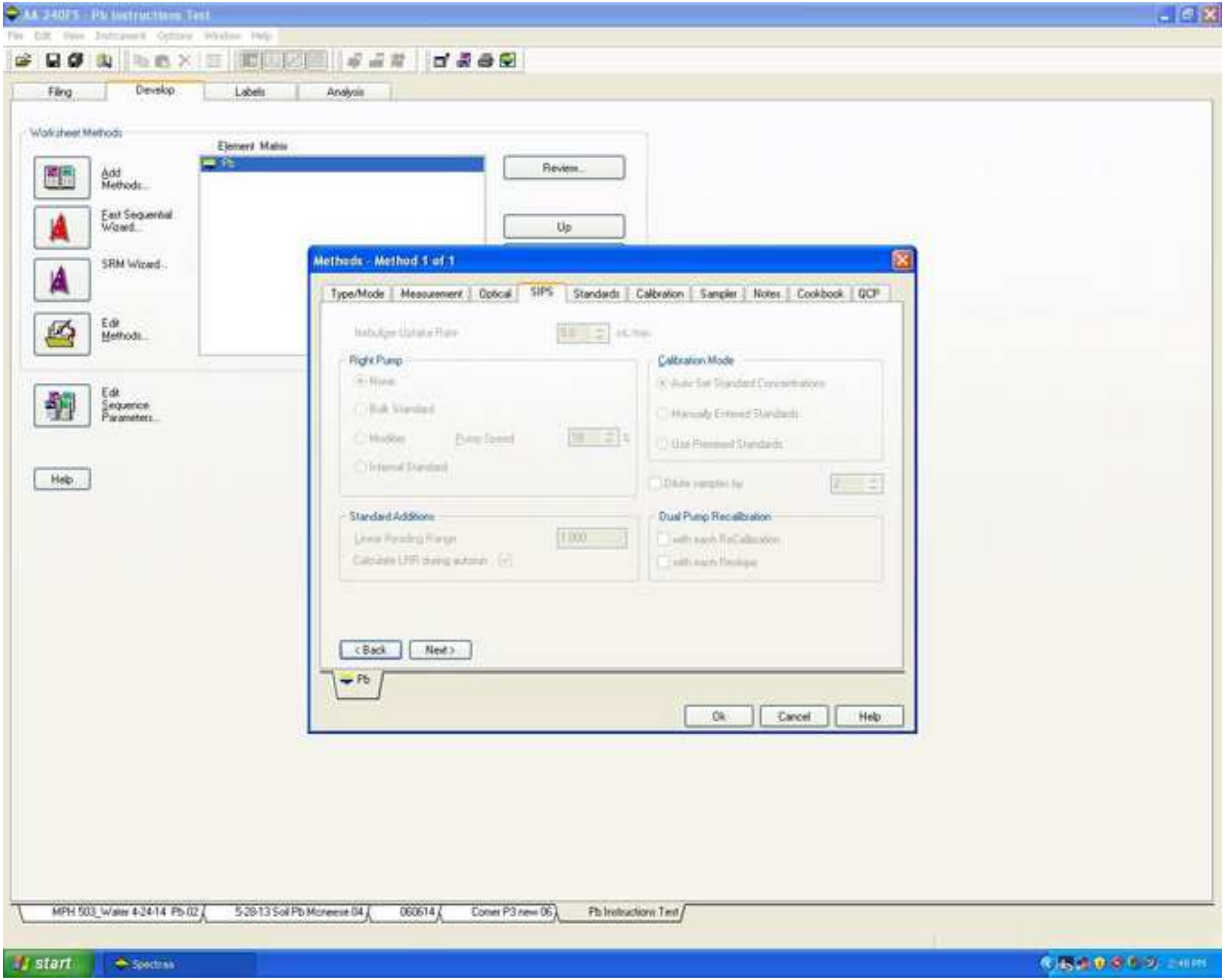
Lamp Current (mA): 10.0

Monochromator

Wavelength (nm): 217.0

Slit Width (nm): 1.0

Background Correction: BC Off



Nebulizer Uptake Rate mL/min

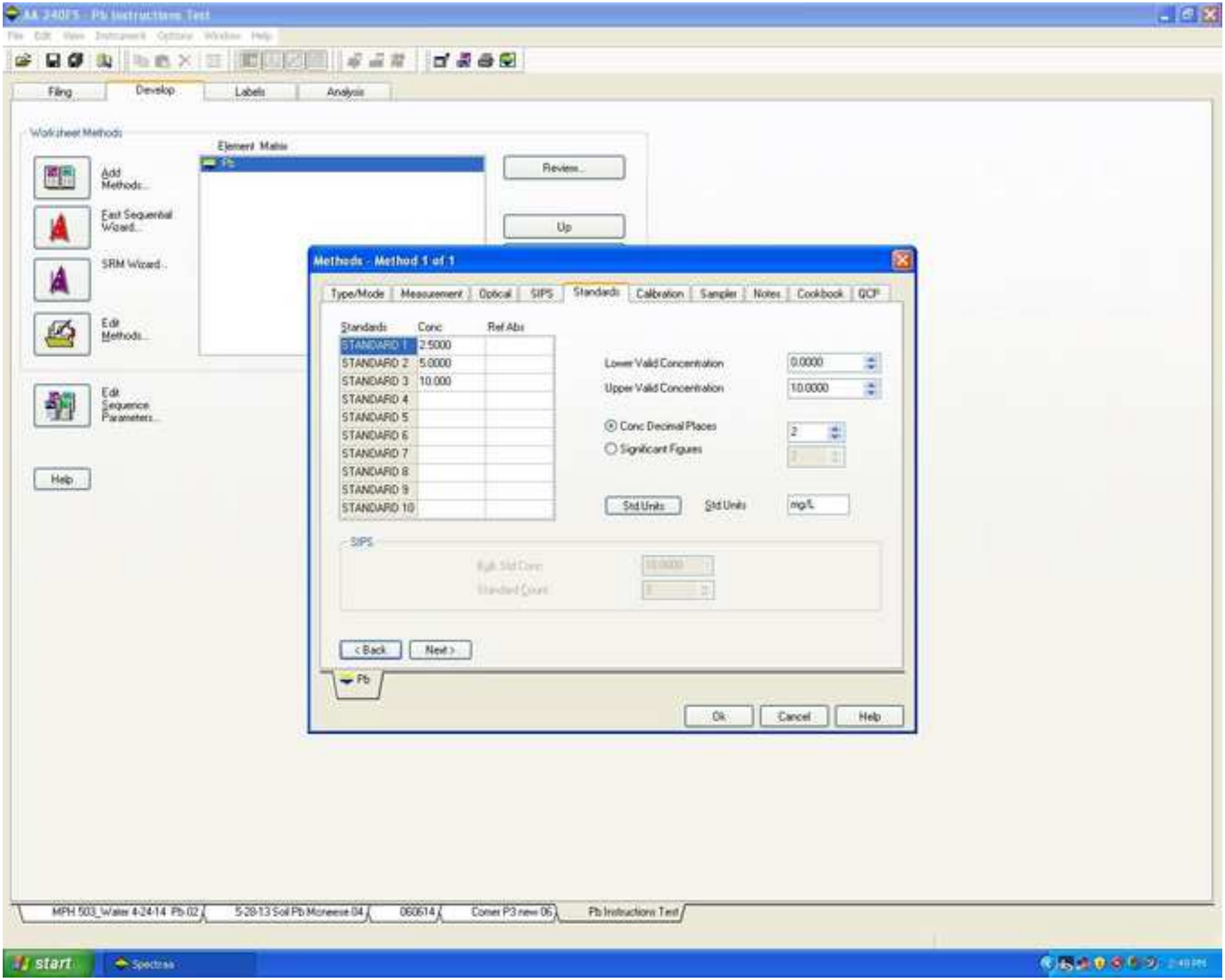
Right Pump
● None
○ Bulk Standard
○ Modifier Pump Speed %
○ Internal Standard

Calibration Mode
● Auto Set Standard Concentrations
○ Manually Entered Standards
○ Use Premixed Standards

☐ Dilute samples by

Standard Additions
Linear Reading Range 1.000
Calculate LRR during autorun ☑

Dual Pump Recalibration
☐ with each ReCalibration
☐ with each Reslope



Standards	Conc	Ref Abs
STANDARD 1	2.5000	
STANDARD 2	5.0000	
STANDARD 3	10.000	
STANDARD 4		
STANDARD 5		
STANDARD 6		
STANDARD 7		
STANDARD 8		
STANDARD 9		
STANDARD 10		

Lower Valid Concentration 0.0000

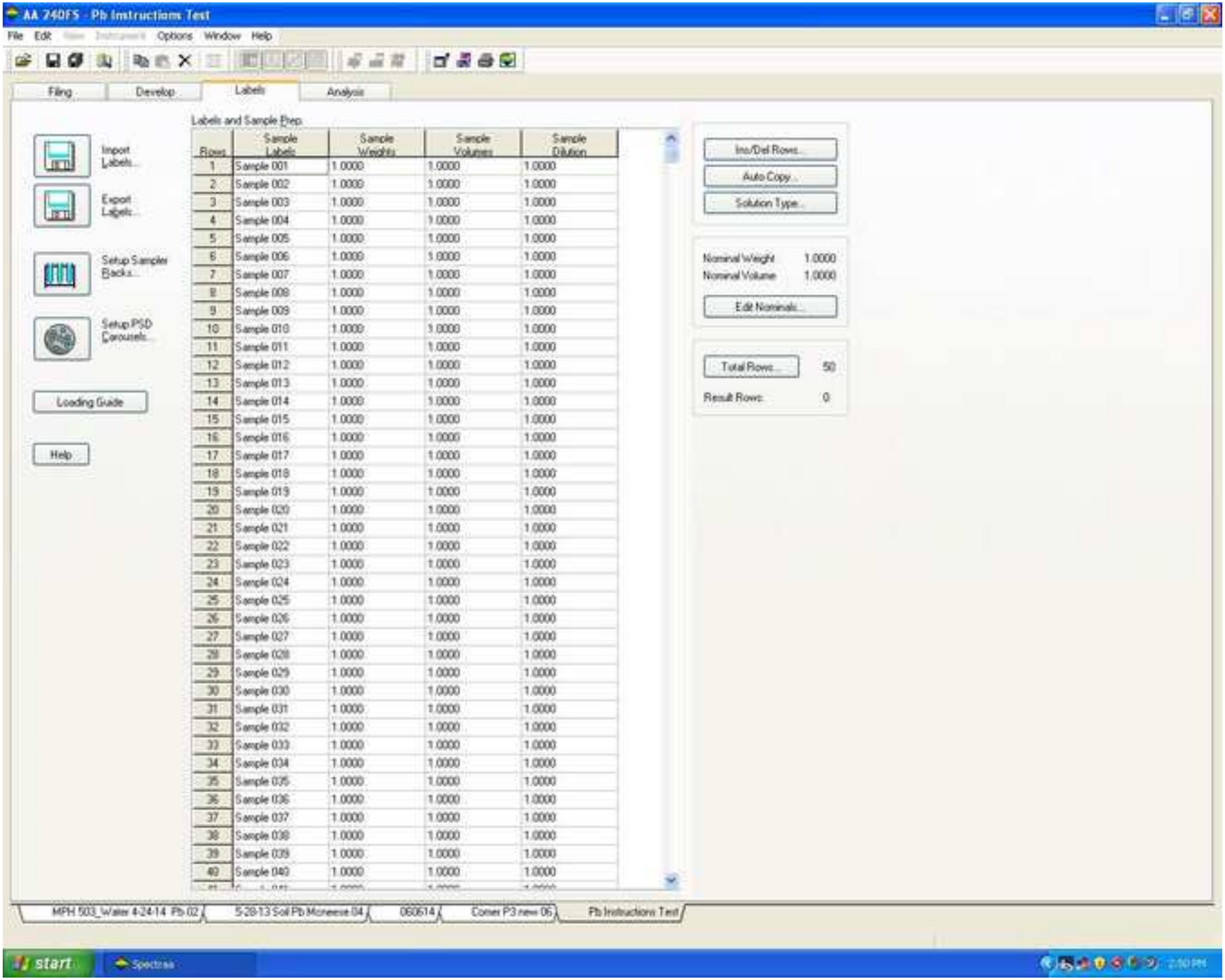
Upper Valid Concentration 10.0000

● Conc Decimal Places 2

○ Significant Figures

Std Units mg/L

Click here to download Photo or Graphic File: Frye and Workman_Lead Analysis of Soil Using AAS_Figure 10.jpg



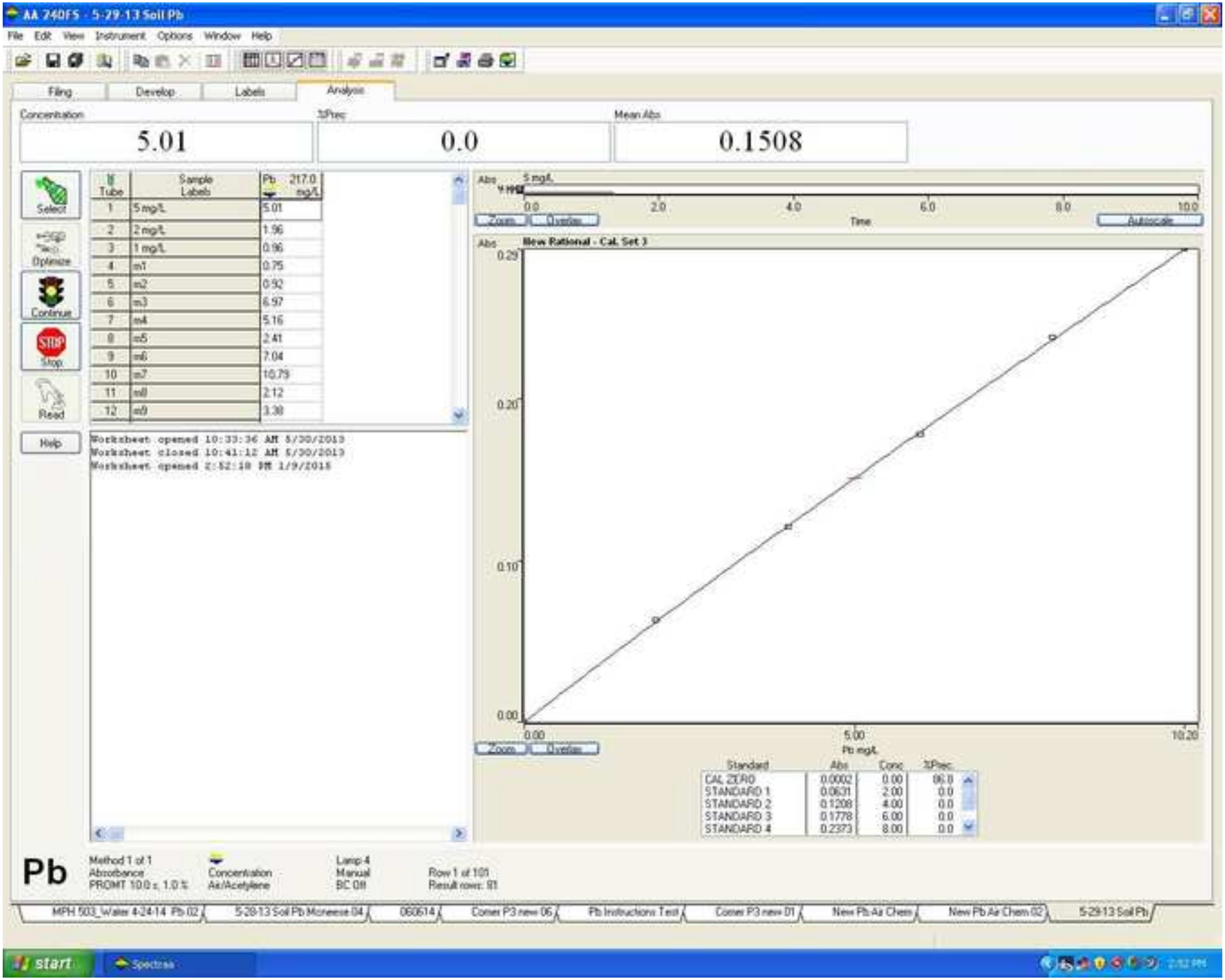
Labels and Sample Prep

Rows	Sample Labels	Sample Weights	Sample Volumes	Sample Dilution
1	Sample 001	1.0000	1.0000	1.0000
2	Sample 002	1.0000	1.0000	1.0000
3	Sample 003	1.0000	1.0000	1.0000
4	Sample 004	1.0000	1.0000	1.0000
5	Sample 005	1.0000	1.0000	1.0000
6	Sample 006	1.0000	1.0000	1.0000
7	Sample 007	1.0000	1.0000	1.0000
8	Sample 008	1.0000	1.0000	1.0000
9	Sample 009	1.0000	1.0000	1.0000
10	Sample 010	1.0000	1.0000	1.0000
11	Sample 011	1.0000	1.0000	1.0000
12	Sample 012	1.0000	1.0000	1.0000
13	Sample 013	1.0000	1.0000	1.0000
14	Sample 014	1.0000	1.0000	1.0000
15	Sample 015	1.0000	1.0000	1.0000
16	Sample 016	1.0000	1.0000	1.0000
17	Sample 017	1.0000	1.0000	1.0000
18	Sample 018	1.0000	1.0000	1.0000
19	Sample 019	1.0000	1.0000	1.0000
20	Sample 020	1.0000	1.0000	1.0000
21	Sample 021	1.0000	1.0000	1.0000
22	Sample 022	1.0000	1.0000	1.0000
23	Sample 023	1.0000	1.0000	1.0000
24	Sample 024	1.0000	1.0000	1.0000
25	Sample 025	1.0000	1.0000	1.0000
26	Sample 026	1.0000	1.0000	1.0000
27	Sample 027	1.0000	1.0000	1.0000
28	Sample 028	1.0000	1.0000	1.0000
29	Sample 029	1.0000	1.0000	1.0000
30	Sample 030	1.0000	1.0000	1.0000
31	Sample 031	1.0000	1.0000	1.0000
32	Sample 032	1.0000	1.0000	1.0000
33	Sample 033	1.0000	1.0000	1.0000
34	Sample 034	1.0000	1.0000	1.0000
35	Sample 035	1.0000	1.0000	1.0000
36	Sample 036	1.0000	1.0000	1.0000
37	Sample 037	1.0000	1.0000	1.0000
38	Sample 038	1.0000	1.0000	1.0000
39	Sample 039	1.0000	1.0000	1.0000
40	Sample 040	1.0000	1.0000	1.0000

Nominal Weight 1.0000
Nominal Volume 1.0000

Total Rows 50

Result Rows 0



Concentration: 5.01

%Prec: 0.0

Mean Abs: 0.1508

Tube	Sample Labels	Pb 217.0 mg/L
1	5 mg/L	5.01
2	2 mg/L	1.96
3	1 mg/L	0.96
4	m1	0.75
5	m2	0.92
6	m3	6.97
7	m4	5.16
8	m5	2.41
9	m6	7.04
10	m7	10.79
11	m8	2.12
12	m9	3.30

```
Worksheet opened 10:33:36 AM 5/30/2013
Worksheet closed 10:41:12 AM 5/30/2013
Worksheet opened 2:52:18 PM 1/9/2015
```

Standard	Abs	Conc	%Prec
CAL ZERO	0.0002	0.00	86.0
STANDARD 1	0.0631	2.00	0.0
STANDARD 2	0.1208	4.00	0.0
STANDARD 3	0.1778	6.00	0.0
STANDARD 4	0.2373	8.00	0.0

Pb
Method 1 of 1
Absorbance
PROMT 10.0 s, 1.0 %

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 101
Result rows: 91

Soil Lead Level (ppm)	**Level of Contamination**
Less than 150	None to very low
150 – 400	Low
400 – 1,000	Medium
1,000 – 2,000	High
Greater than 2,000	Very High

PI Name: Margaret Workman & Kimberly Frye, DePaul University

Science Education Title: Lead Analysis of Soil using Atomic Absorption Spectrometry (AAS)

Overview

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Atomic Absorption Spectrometry is an elemental analysis technique that provides quantitative information on over 70 different elements. Concentrations as low as ppt can be determined for some elements, with ppb and ppm being more common for various metals. This method has several benefits over others. For example, this technique measures the total concentration of an element, regardless of its form. In addition, the wavelength used is specific to the element being tested, so there is no interference from other elements in the sample, making it a fast and easy technique.

Principles

Lead occurs naturally in soil in levels, ranging from 50 – 400 ppm. However, with the widespread use of lead in paint and gasoline in addition to contamination by industry, urban soils often have concentrations of lead significantly greater than background levels – up to 10,000 ppm in some places. Lead does not biodegrade, but remains in the soil.

Serious health risks are associated with lead poisoning. Children are particularly at risk. Millions of children in the U.S. are exposed to soil containing lead. This exposure can cause developmental and behavioral problems in children. These problems include learning disabilities, inattention, delayed growth, and brain damage. The Environmental Protection Agency has set a standard for lead in soil at 400 ppm for play areas and 1200 ppm for non-play areas.

Lead is also of concern in soil, when it's used for gardening. Plants take up lead from the soil. Therefore, if one eats vegetables or herbs grown in contaminated soil, lead can be ingested. In addition, contaminated soil particles can be breathed in while gardening or brought into the house on clothing and footwear. It is recommended that soils with lead levels greater than 400 ppm should not be used for gardening. It is further recommended that soil with lead levels between 100 and 400 ppm not be used for leafy vegetables or herbs, because lead can be stored in the leaves. On a similar note, root vegetables should not be grown in this soil, because lead can also accumulate in plant roots.

Procedure

1. Soil Collection

   1.1. In undisturbed areas, collect soil from the upper 1-2 inches of the soil. If sampling vegetable gardens, collect 6-inch deep samples.

      1.1.1. Use a shovel to dig a 6-inch deep hole to expose a smooth vertical area of soil.

      1.1.2. Cut a 1-inch thick slice from the vertical face. Collect a 1-inch wide sample from the center of the slice that extends from the surface to 6 inches below the surface.

   1.2. Take 10 samples from an area and put them in a container.

~~1.~~2. Soil Preparation

   ~~1.1.~~2.1. Mix sample thoroughly by shaking for 2 min and sieve using a USS #10 sieve.

   ~~1.2.~~2.2. Dry soil in a 40 °C oven for 24 hr.

~~2.~~3. Sample Digestion

   ~~2.1.~~3.1. Using an analytical balance, weigh out 1 g of the soil sample and place in a digestion tube. Record the weight of the sample to four decimal places.

   ~~2.2.~~3.2. In a hood, add 5 mL of water to the digestion tube.

   ~~2.3.~~3.3. Add 5 mL of concentrated HNO_3 to the digestion tube.

   ~~2.4.~~3.4. Mix the slurry with a stirring rod. Cover the digestion tube with a teardrop glass stopper.

   ~~2.5.~~3.5. Put the digestion tube in the block digester and heat the sample to 95 °C and reflux for 10 min without boiling (**Figure 1**). Remember that this contains concentrated acid.

   ~~2.6.~~3.6. Allow the tubes to cool. Add 5 mL of concentrated HNO_3 to the digestion tube, replace the drop glass, and reflux for an additional 30 min. If brown fumes are generated, repeat this step over and over until no brown fumes are given off by the sample.

   ~~2.7.~~3.7. Evaporate the solution to a 5 mL volume without boiling.

   ~~2.8.~~3.8. Allow the tubes to cool. Add 2 mL of distilled water and 3 mL of 30% H_2O_2. Cover with the glass stopper and heat to begin the peroxide reaction. Be careful that the solution does not boil over. Heat until the bubbling stops and allow to cool.

~~2.9.~~3.9. Continue to add 30% H_2O_2 in 1 mL increments, warming until the bubbling is minimal. Do not add more than a total of 10 mL of the 30% H_2O_2.

~~2.10.~~3.10. Cover the sample with the glass teardrop stoppers and heat until the volume is reduced to 5 mL without boiling.

~~2.11.~~3.11. Add 10 mL concentrated HCl to the sample and cover with the glass teardrop stopper. Heat to 95 °C and reflux for 15 min.

~~2.12.~~3.12. Allow the tubes to cool. If there are particulates, filter the sample using Whatman 41 filter paper (or similar) and collect filtrate in a 100 mL volumetric flask. Dilute the sample volume to 100 mL with distilled water.

~~3.~~4. Analyzing Samples on Atomic Absorption Spectrometer

~~3.1.~~4.1. Turn on the computer and the spectrometer.

~~3.2.~~4.2. Set parameters on the instrument:

  ~~3.2.1.~~4.2.1. Set the acetylene pressure to > 700 kPa (~100 psi).

  ~~3.2.2.~~4.2.2. Set the acetylene valve set to 11 psi.

  ~~3.2.3.~~4.2.3. Set the air valve 45 psi.

~~3.3.~~4.3. Open the SpectrAA software (**Figure 2**).

~~3.4.~~4.4. Open a new worksheet (**Figure 3**).

~~3.5.~~4.5. Choose “Add Method” and click on Pb to do a Lead Analysis (**Figure 4**).

~~3.6.~~4.6. Set Type/Mode parameters to the following (**Figure 5**):

  ~~3.6.1.~~4.6.1. Type = Flame

  ~~3.6.2.~~4.6.2. Element = Pb

  ~~3.6.3.~~4.6.3. Sampling Mode = Manual

  ~~3.6.4.~~4.6.4. Instrument Mode = Absorbance

  ~~3.6.5.~~4.6.5. Flame Type = Air/Acetylene

  ~~3.6.6.~~4.6.6. Air Flow = 13.5

  ~~3.6.7.~~4.6.7. Acetylene Flow = 2.0

  ~~3.6.8.~~4.6.8. Online Diluter Type = SIPS

~~3.7.~~4.7. Set the Measurements parameters to the following (**Figure 6**):

~~3.7.1.~~4.7.1. Measurement Mode = PROMT

~~3.7.2.~~4.7.2. Calibration Mode = Concentration

~~3.7.3.~~4.7.3. Times: Measurement = 10

~~3.7.4.~~4.7.4. Times: Read Delay = 10

~~3.7.5.~~4.7.5. Replicates: Standard = 3

~~3.7.6.~~4.7.6. Replicates: Sample = 3

~~3.7.7.~~4.7.7. Precision (%): Standard = 1.0

~~3.7.8.~~4.7.8. Precision (%): Sample = 1.0

~~3.8.~~4.8. Set the Optical parameters to the following (**Figure 7**):

~~3.8.1.~~4.8.1. Lamp Position = Ca #4

~~3.8.2.~~4.8.2. Lamp Current (mA) = 10.0 mA

~~3.8.3.~~4.8.3. Wavelength = 217.0 nm

~~3.8.4.~~4.8.4. Slit = 1.0 nm

~~3.8.5.~~4.8.5. Background = BC Off

~~3.9.~~4.9. Set the SIPS parameters to the following (**Figure 8**):

~~3.9.1.~~4.9.1. Nebulizer Uptake Rate = 5.0 mL/min

~~3.9.2.~~4.9.2. Right Pump = none

~~3.9.3.~~4.9.3. Standard Additions = Unselect

~~3.9.4.~~4.9.4. Calibration Mode = Auto Set Std Concentrations

~~3.9.5.~~4.9.5. Dual Pump Calibration = Unselect

~~3.10.~~4.10. Under the Standards tab, a list of standards automatically populates for the particular test (**Figure 9**). A 1000 ppm Pb standard for atomic absorption spectrometry purchased from a chemical supply company is used and automatically diluted by the instrument. A new calibration curve is generated each time a new set of samples is run.

~~3.11.~~4.11. Exit the Edit Method menu and click on the “Labels” tab. Input information regarding sample names and number of samples (**Figure 10**).

~~3.12.~~4.12. Using the “Analysis” tab, use the “Select” button to highlight the samples to be analyzed.

~~3.13.~~4.13. Turn on the flame by pressing the ignite button on the instrument.

~~3.14.~~4.14. Zero the instrument by aspirating a blank and pressing the “Alt” and “Read” keys simultaneously.

~~3.15.~~4.15. Place the pump tubing in the blank solution and press “Start.” Once the calibration has been performed, place the pump tubing in the sample and press the “Read” key. Continue for all samples.

~~3.16.~~4.16. Turn off the instrument by pressing the red power off button on the instrument. Turn off all gas tanks and remove all samples.

Representative Results

The software creates the calibration curve and automatically determines the concentration of the Pb in the samples (**Figure 11**).

The values given on the worksheet are mg/L of Pb in the sample solution. Additional calculations must be done to convert this number to the ppm of Pb in the soil sample.

Example: For a soil sample that weighed 1.2523 g before digestion was measured by the AAS to have 6.0 mg/L of Pb in the 100 mL solution sample (**Table 1**).

$$\underline{100\text{ mL solution}} \times \frac{6.0\text{ mg Pb}}{\text{L solution}} \times \frac{1\text{ L}}{1000\text{ mL}} = 0.6\text{ mg Pb}$$

$$\frac{0.6\text{ mg Pb}}{1.2523\text{ g soil}} \times \frac{1000\text{ g}}{1\text{ kg}} = \frac{479\text{ mg Pb}}{\text{kg soil}} = 479\text{ ppm}$$

Applications

Atomic Absorption Spectrometry is a useful technique to analyze a wide range of environmental samples (e.g., water, soil, sludge, and sediment) for a large number of elements (e.g., heavy metals). This experiment highlights the use of flame AAS to determine the Pb content in soil. However, it could also be used to measure concentrations of Cu, Fe, Mn, K, Na, Mg, and Zn in soils.

Zinc is an important micronutrient and is needed for protein synthesis. Zn helps regulate the expression of genes needed to protect cells when under environmental stress conditions. Zinc deficiency is a large problem in crop and pasture plants around the world, resulting in decreased yields. It is estimated that half of all soils used for cereal production have a zinc deficiency. This leads to a zinc deficiency in the grain. As a result, zinc deficiency in humans is a serious nutritional problem worldwide, affecting 1/3 of the world’s population. A typical range of zinc in soils is 10 – 300 mg/kg with a mean of 55 mg/kg.

Iron is the fourth most abundant element on Earth. However, it is mostly found in forms ~~that are~~ not available for plants, such as in silicate minerals or iron oxides. Iron is involved in photosynthesis, chlorophyll formation, nitrogen fixation, and many enzymatic reactions in plants. Iron deficiency in soil is rare, but it can become unavailable in excessively alkaline soils. Symptoms of iron deficiency in soil include leaves turning yellow and a decrease in yield. A typical range of iron in soils is 100 – 100,000 ppm with a mean of 26,000 ppm.

Copper is an essential micronutrient for plants. Copper promotes seed production, plays a role in chlorophyll formation, and is essential for enzyme activity. Copper deficiency can be seen by light green to yellow leaves. The leaf tips die back and become twisted. If the deficiency is severe enough, growth of the grain can stop and the plants die. Available copper in soils can vary from 1 to 200 ppm. Availability of copper is related to the soil pH – as pH increases, the availability of copper decreases.

Atomic Absorption Spectrometry can also be used on non-environmental samples, including:

- Water analysis (Ca, Mg, Fe, Al, Ba, Cr)
- Food analysis (Cd, Pb, Al, Cu, Fe)
- Additives in oils (Ba, Ca, Na, Li, Zn, Mg, V, Pb, Sb)
- Fertilizers (K, B, Mo)
- Clinical samples (blood, serum, plasma, urine, Ca, Mg, Li, Na, K, Fe, Cu, Zn, Au, Pb)
- Cosmetics (Pb)
- Mining (Au)

Legend

Comment [JR1]: Figures 1-11 and Table 1 are from the author.

Figure 1: Digestion tubes in a block digester.

Figure 2: A desktop icon to click, which opens the SpectrAA software.

Figure 3: A screenshot of a user creating a new worksheet.

Figure 4: After choosing “Add Method,” click on Pb to do a Lead Analysis.

Figure 5: A screenshot of the Type/Mode parameters that need to be set.

Figure 6: A screenshot of the Measurements parameters that need to be set.

Figure 7: A screenshot of the Optical parameters that need to be set.

Figure 8: A screenshot of the SIPS parameters that need to be set.

Figure 9: A list of standards under the Standards tab that was automatically populated for the test.

Figure 10: The Labels tab, where information can be input regarding sample names and number of samples.

Figure 11: The calibration curve and the concentration of the Pb in the samples automatically determined by the software.

Table 1: Soil lead levels measured in ppm and the corresponding levels of contamination.