به نام خدایی که به شدت کافیست

# Workshop in Analytical Instrumentation for Atomic Absorption Spectroscopy

**By: Ali Sorkheh & DSA Co.**

Daghighshimi@yahoo.com

**www.daghighshimi.ir**

شماره ثبت : ۱۱۷۳۵
دقیق شیمی آزما

# Atomic Spectroscopy for Metal Analysis

## Introduction to the Principles of Atomic Spectroscopy

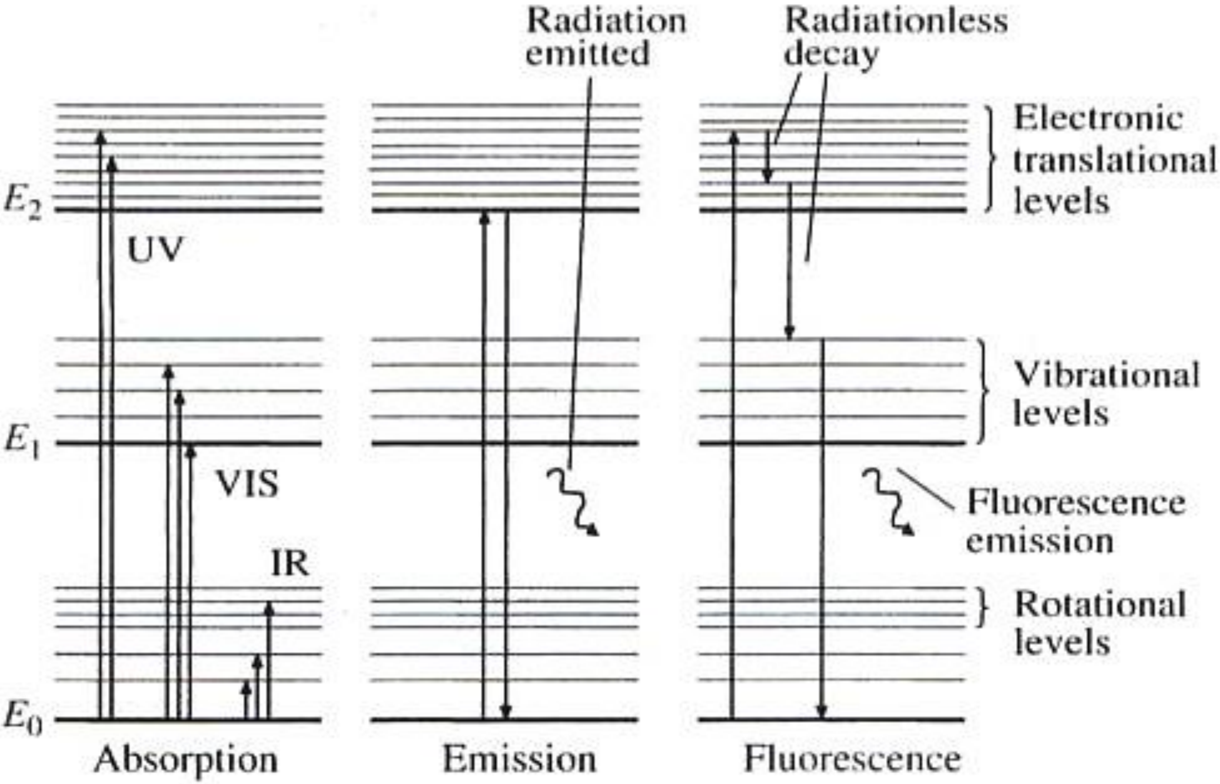
- **3 major types of atomic spectroscopy:**

- **Absorption** – light of a wavelength characteristic of the element of interest radiates through the atom vapor. The atoms absorb some of the light. The amount absorbed is measured.

- **Emission** – sample is heated to excitation/ionization of the sample atoms. Excited and ionized atoms decay to a lower energy state through emission. Intensity of the light emitted is measured.

- **Fluorescence** – a short wavelength is absorbed by the sample atoms, a longer wavelength (lower energy) radiation characteristic of the element is emitted and measured

By:A.Sorkheh@yahoo.com



$E_0$ = Ground level; $E_1$, $E_2$ = Excited states

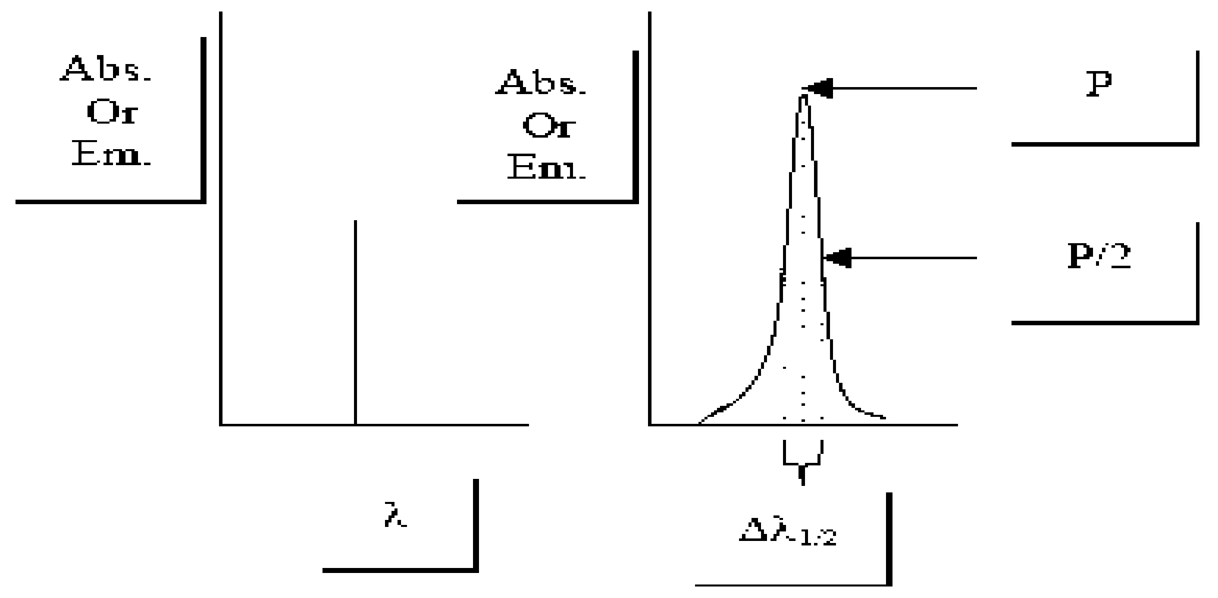
Energy spacing: vibration > rotation >> translation

**Figure 8.4** Three types of spectroscopy—absorption, emission, and fluorescence

By:A.Sorkheh@yahoo.com

# Atomic Line Width

- It is taken for granted that an atomic line should have infinitesimally small **(or zero) line width** since transition between two quantum states requires an exact amount of energy. However, careful examination of atomic lines reveals that they have finite width. For example, try to look at the situation where we expand the x-axis (wavelength axis) of the following line:



- The **effective line width** in terms of wavelength units is equal to $Dl_{1/2}$ and is defined as the width of the line, in wavelength units, measured at one half maximum signal (P).

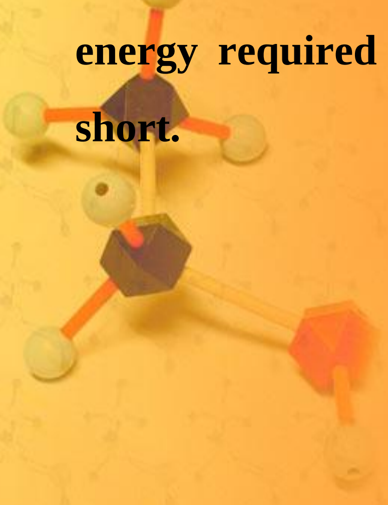
- **The question** which needs a definite answer is **what causes** the atomic line to become **broad ?**

By:A.Sorkheh@yahoo.com

# Reasons for Atomic Line Broadening

## 1. The Uncertainty Principle

We have seen earlier that Heisenberg uncertainty principle suggests that nature places limits on the precision by which two interrelated physical quantities can be measured. It is not easy, will have some uncertainty, to calculate the energy required for a transition when the lifetime of the excited state is short.



By:A.Sorkheh@yahoo.com

- The **ground state lifetime is long** but the lifetime of the **excited state** is **very short** which suggests that there is an **uncertainty** in the calculation of the transition time. We have seen earlier that when we are to estimate the energy of a transition and thus the wavelength (line width), it is required that the two states where a transition takes place should have infinite lifetimes for the uncertainty in energy (or wavelength) to be zero:

$$DE>h/Dt$$

- Therefore, atomic lines should have some broadening due to uncertainty in the lifetime of the excited state.

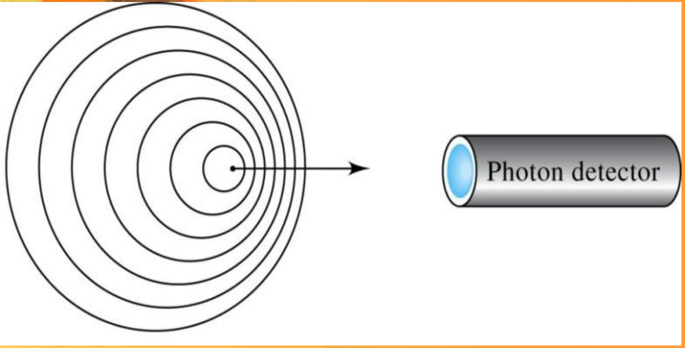
- The broadening resulting from the uncertainty principle is referred to as **natural line width** and is unavoidable.

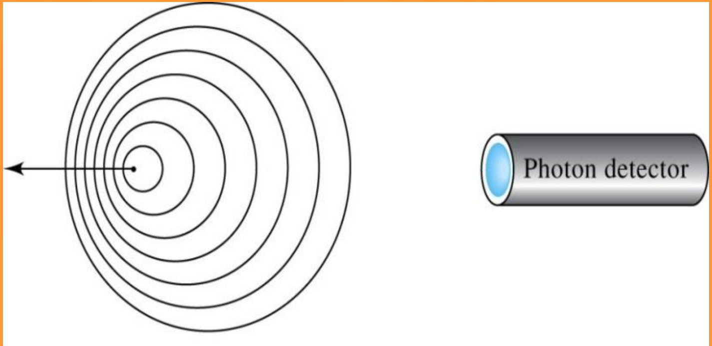
# 2. Doppler effect

- **Emitted or absorbed wavelength changes as a result of atom movement relative to detector**

- ✓ **Wavelength decrease if motion toward receiver**

- ✓ **Wavelength increases if motion away from receiver**

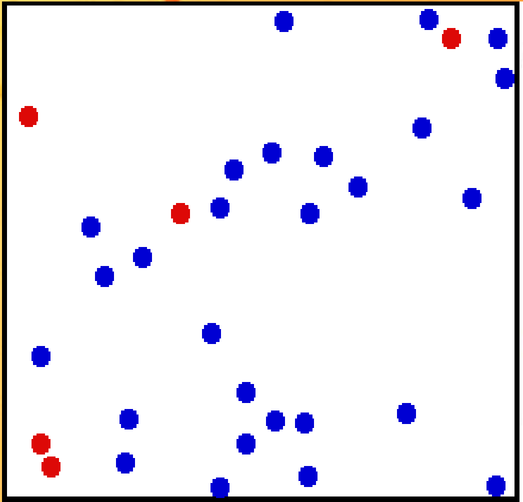


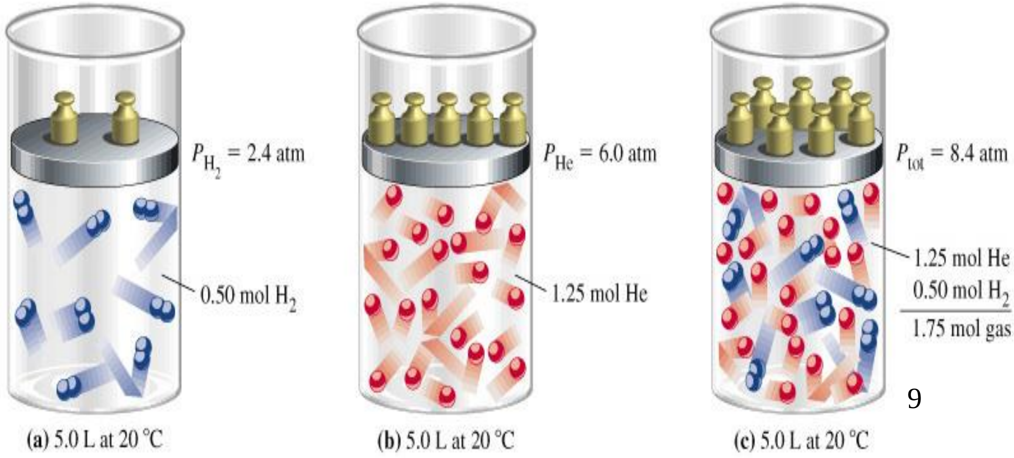


**Usage in measurement of velocity of galaxies, age of universe and big bang theory**

# 3-Pressure(Lorentz or Random or Stark or Holtzmark) broadening

* Collisions with atoms/molecules transfers small quantities of vibrational energy (heat) - ill-defined ground state energy effect worse at high pressures:





- ✓ **Lorentz broadening**: **Collisions of different molecules in hight concentration.**

- ✓ **Stark broadening**: **Collisions of charged particles.**

- ✓ **Vandevals effect** : **Collisions of no charged(neutrals) particles.**

- ✓ **Holtzmark broadening**: **Collisions of same atomes.**

By:A.Sorkheh@yahoo.com

# Atomic Absorption Spectroscopy(AAS)

## Theory & Principle of OPERATION

- AAS is an analytical technique that measures the concentrations of elements.

- It makes use of the absorption of light by these elements in order to measure their concentration .

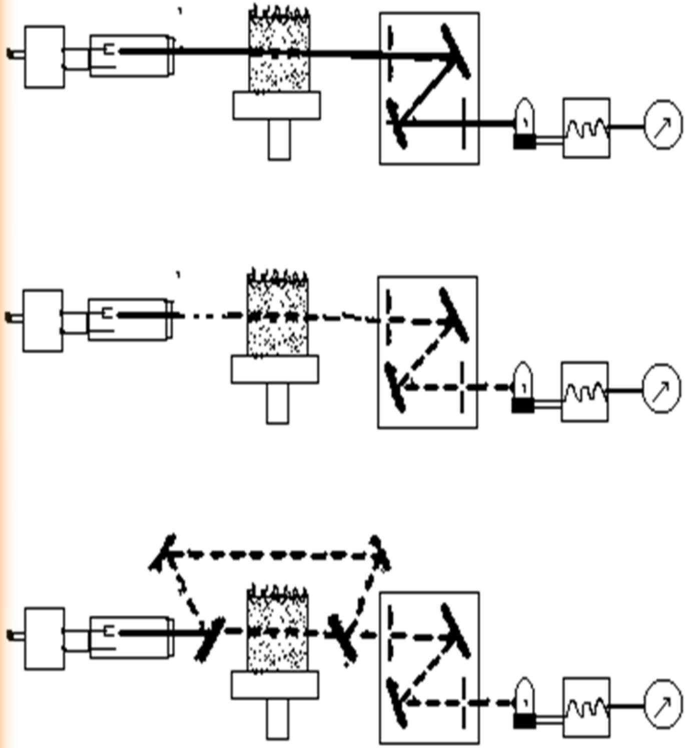
- Atomic-absorption spectroscopy quantifies the absorption of ground state atoms in the gaseous state .

- The **atoms absorb UV** or **Vis** light and make transitions to higher electronic energy levels .

- The analyte **concentration** is determined from the amount of absorption.

By:A.Sorkheh@yahoo.com

# Basic AA Design

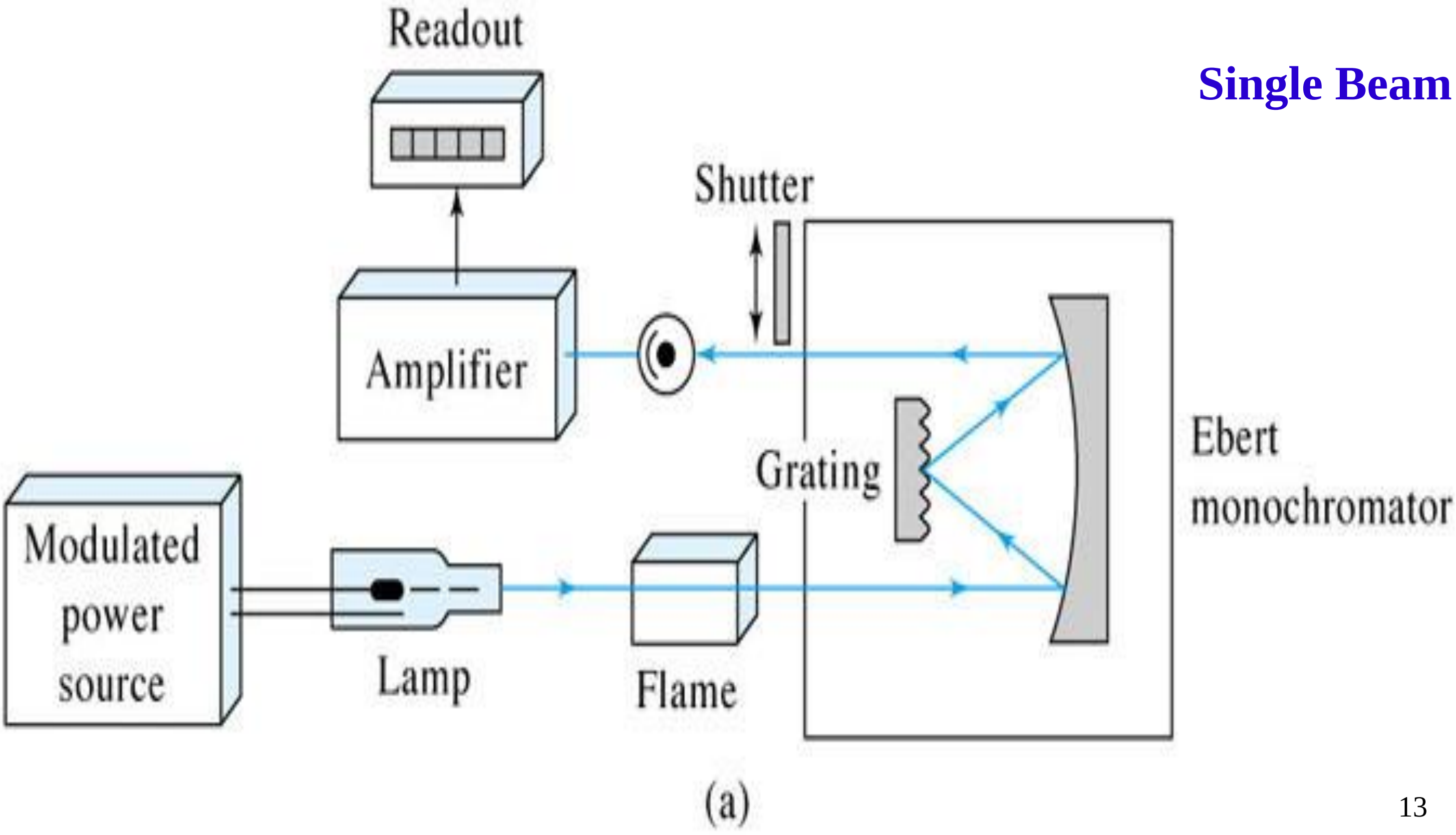


- First instruments were ***DIRECT Current(DC), SINGLE Beam*** **designs**
- **Next generation were *ALTERNATINGCurrent(AC)* to try to compensate for high Drift & Instability**
- **Final designs had to "split" the signal into a *"DOUBLE Beam"* path to fully correct for Noise, Drift & Errors**

By:A.Sorkheh@yahoo.com

# Single Beam or Double Beam AAS Systems ?

**Single Beam**



By:A.Sorkheh@yahoo.com

# Single Beam Systems

- The light source comprising of a hollow cathode lamp emits sharp atomic line of the element whose determination is required.

- The light is modulated (switched on and off) rapidly by means of a rotating chopper located between the light source and the flame.

- Modulation can also be achieved by pulsing the power (switched on and off rapidly) to the light source.

By:A.Sorkheh@yahoo.com

- **Modulation serves to differentiate the light coming from the source lamp, from the emission and from the flame.**

- **The modulated light is led to the flame where ground state atoms of the element of interest are present and after absorption is led to the monochromator which isolates the wavelength of interest which is then led to the detector.**

By:A.Sorkheh@yahoo.com

## Advantages of Single Beam Systems

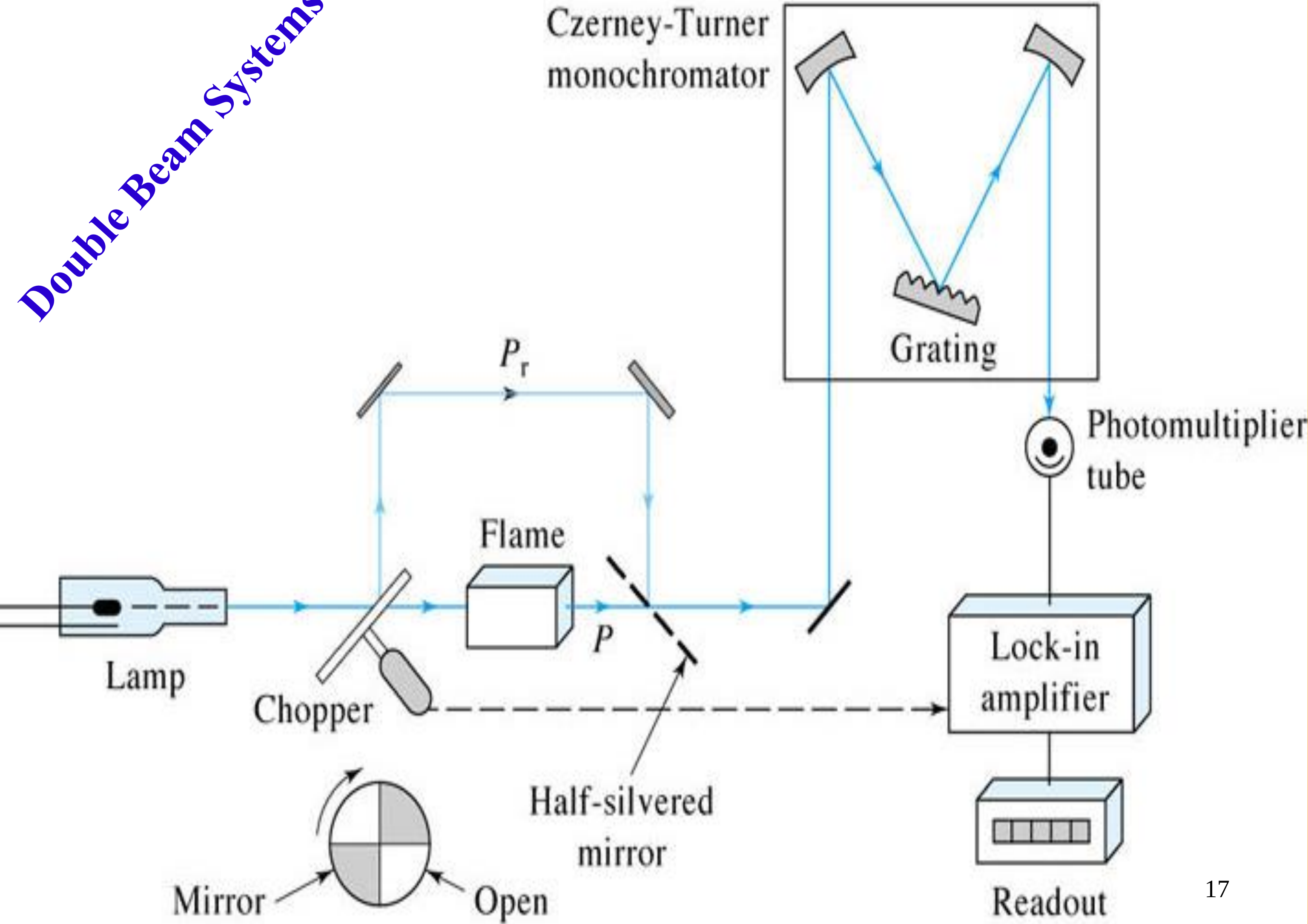
- ✓ Single beam instruments are less expensive
- ✓ High energy throughput due to non-splitting of source beam results in high sensitivity of detection

## Disadvantages

- ✓ Instability due to lack of compensation for disturbances like electronic circuit fluctuations, voltage fluctuations, mechanical component's instability or drift in energy of light sources. Such drifts result in abnormal fluctuations in the results.

By:A.Sorkheh@yahoo.com

# Double Beam Systems



By:A.Sorkheh@yahoo.com

- **The light beam from the source is split into sample beam and reference beam by the mechanical chopper.**

- ✓ **The reference beam monitors the lamp energy.**

- ✓ **The sample beam reflects sample absorption.**

- **The observed absorbance measurement is the ratio of the sample and reference beams which are recombined before moving to the monochromator.**

- **This arrangement compensates the effects due to drift in lamp intensity, electronic and mechanical fluctuations which affect both the sample and reference beams equally.**

# ❖ Advantages of Double Beam Systems

- Modern improvements in optics permit high level of automation and offer the same or even better level of detection as compared to earlier single beam systems. Instability factors due to lamp drift, stray light, voltage fluctuations do not affect the measurement in real-time.

- Little or no lamp warm up time is required. This not only improves throughput of results but also conserves lamp life.

- The cost factor is more than offset by the advantages offered by modern double beam systems and therefore these have become the preferred choice.

By:A.Sorkheh@yahoo.com

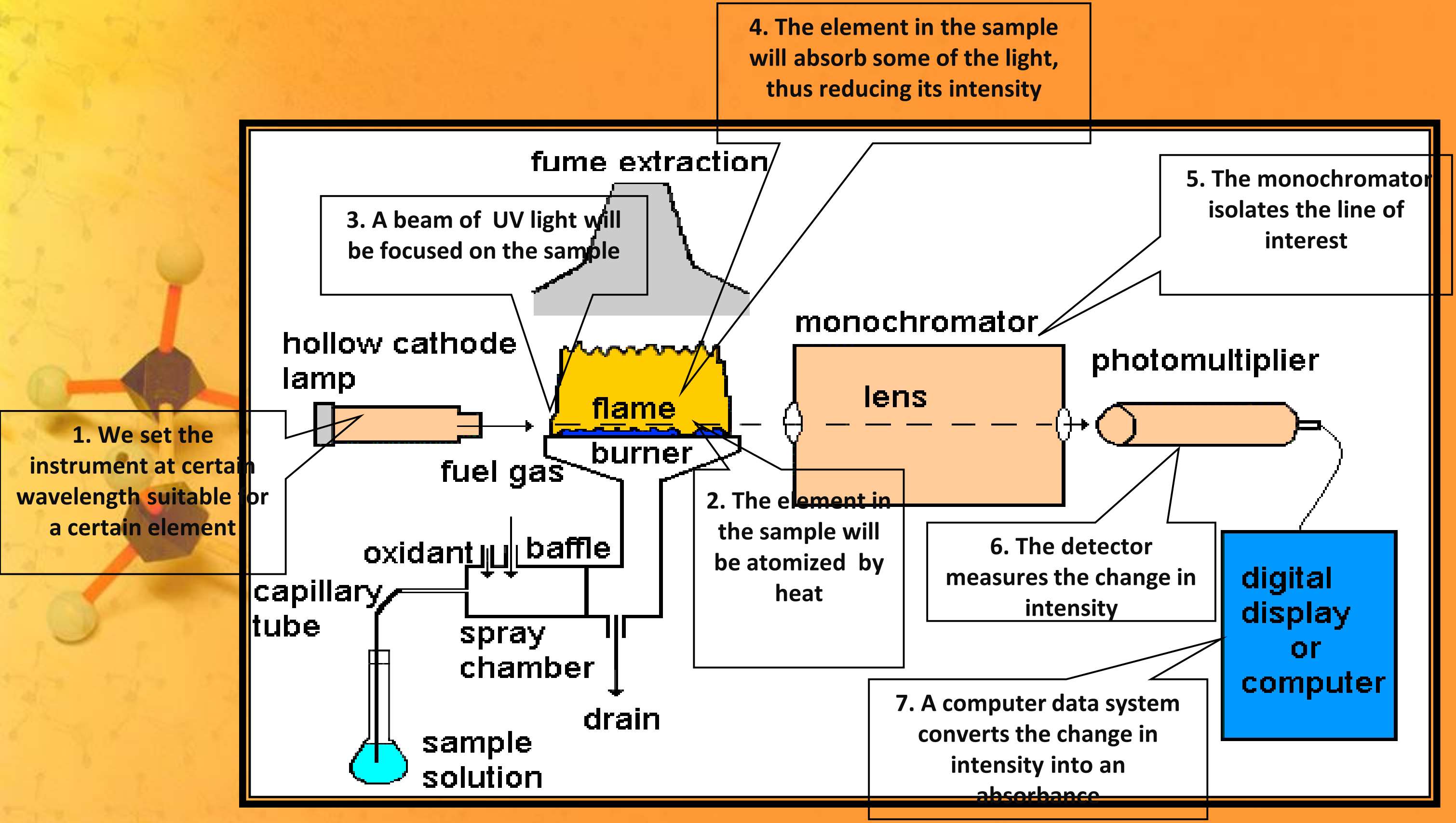
# Basic SPECTROMETER Design

**All Analytical Spectrometers have several BASIC design components in common:**

- **Light Source(HCL in FAAS)**
- **Sample “Chamber” or Atomizer (atom cell)**
- **Optical System (Monochromator)**
- **Detector(E.g. Photomultiplier)**
- **Output (Read out)**

By:A.Sorkheh@yahoo.com

# The simple  diagram for the AAS



1. We set the instrument at certain wavelength suitable for a certain element

2. The element in the sample will be atomized  by heat

3. A beam of  UV light will be focused on the sample

4. The element in the sample will absorb some of the light, thus reducing its intensity

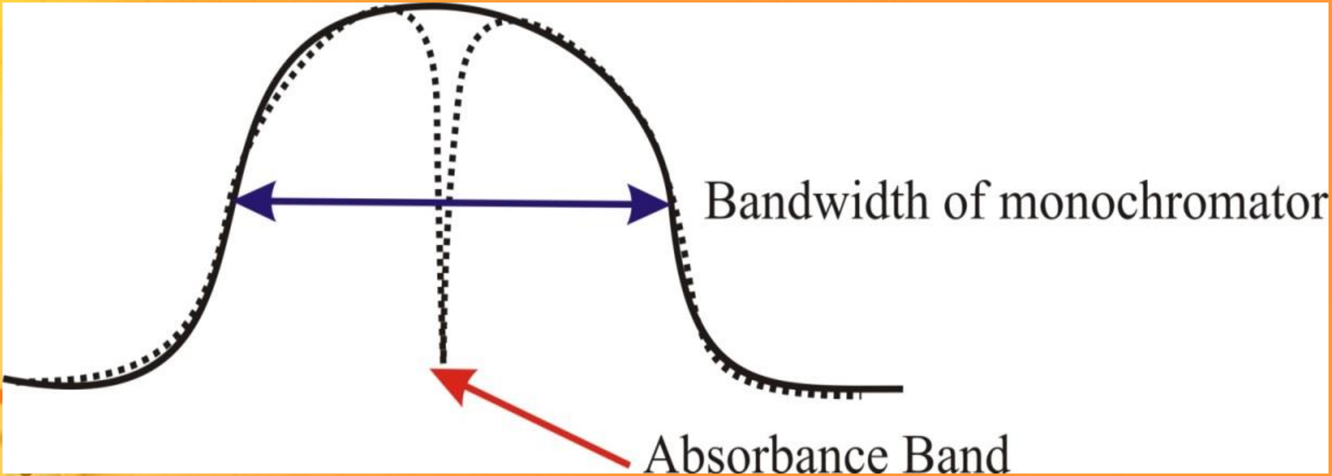
5. The monochromator isolates the line of interest

6. The detector measures the change in intensity

7. A computer data system converts the change in intensity into an absorbance

# A) Light source

- ✓ Need light source with a narrow bandwidth for light output
- ✓ AA lines are remarkably narrow (0.002 to 0.005 nm)
- ✓ Separate light source and filter is used for each element

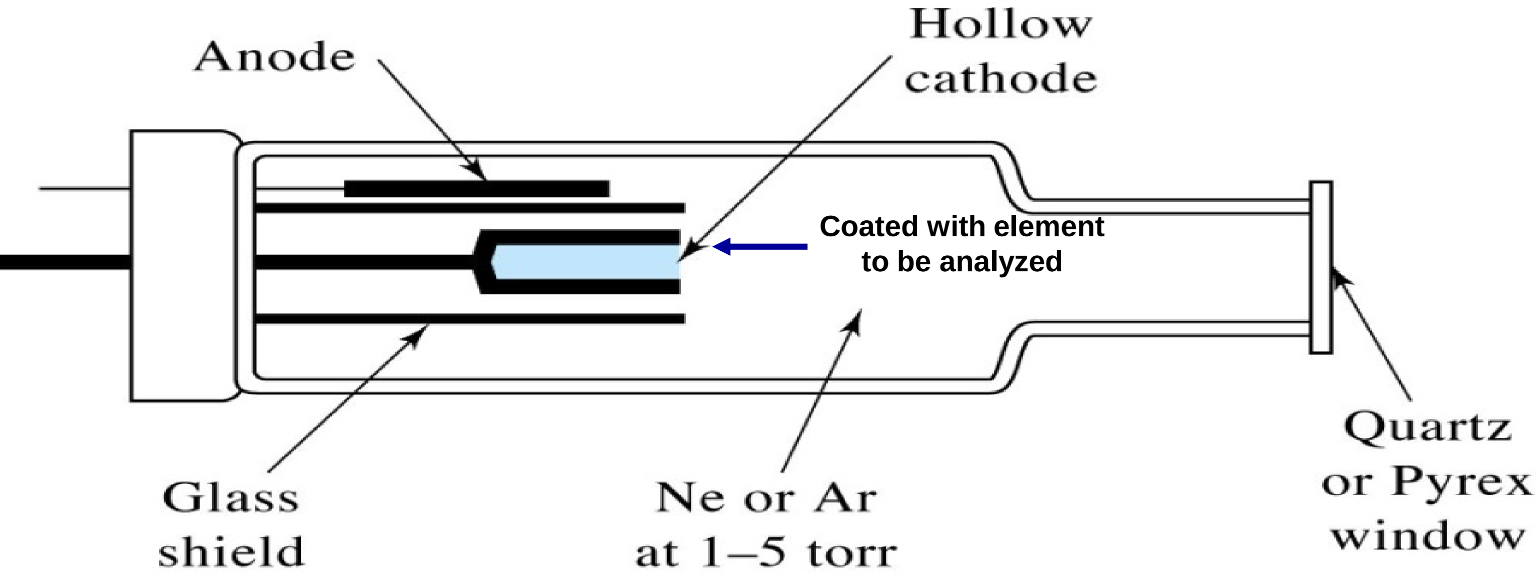


1) problem with using typical UV/Vis continuous light source:

- ➢ have right l, but also lots of others (non-monochromatic light)
- ➢ hard to see decrease in signal when atoms absorb in a small bandwidth
- ➢ only small decrease in total signal area
- ➢ with large amount of elements → bad sensitivity

2) Solution is to use light source that has line emission in range of interest

- ✓ laser – but hard to match with element line of interest
- ✓ hollow cathode lamp (HCL) is common choice



By:A.Sorkheh@yahoo.com

# Light source

**The main sources used for atomic absorption:**

**1.Hollow cathode lamp (HCL): low intensity and short lamp lifetime**

**2.Electrodeless discharge lamp (EDL):more intense than hollow cathode lamps**

- ✓ **The hollow cathode lamp is an excellent, bright, stable line source for most elements.**
- ✓ **EDLs are typically more intense than hollow cathode lamps and, therefore, may offer better precision and lower detection limits for some elements.**

By:A.Sorkheh@yahoo.com

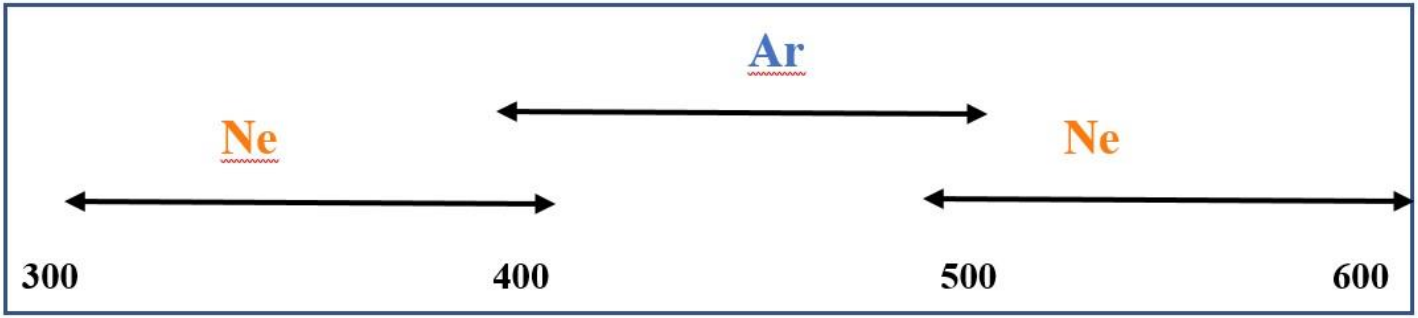
- **The cathode is a hollowed-out cylinder constructed entirely or in part of the metal whose spectrum is to be produced.**

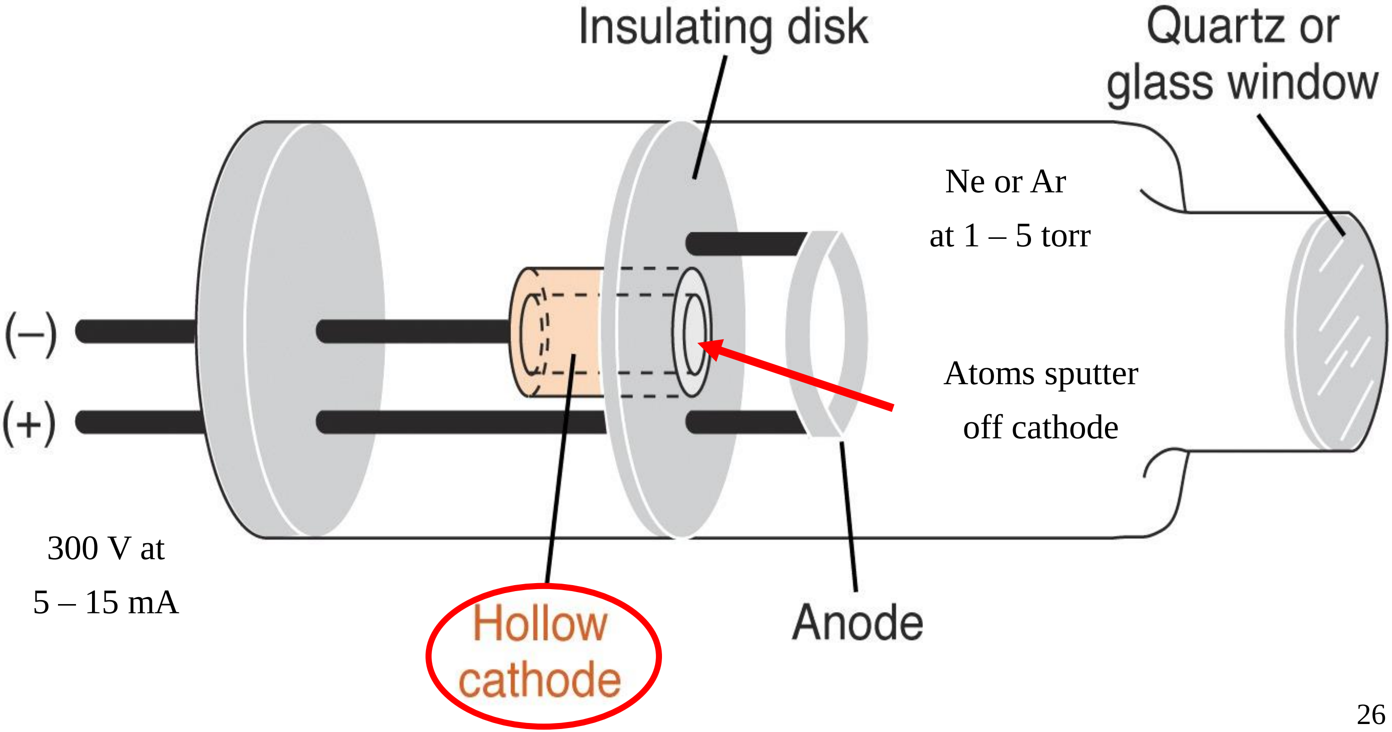
- **The anode and cathode are sealed in a glass cylinder filled with neon or argon.**

- **The glass cylinder has a quartz or UV glass window for optimum transmittance of the emitted radiation.**

- **The optimum fill gas is selected that gives the best lamp intensity while taking into consideration spectral interferences from either neon or argon.**

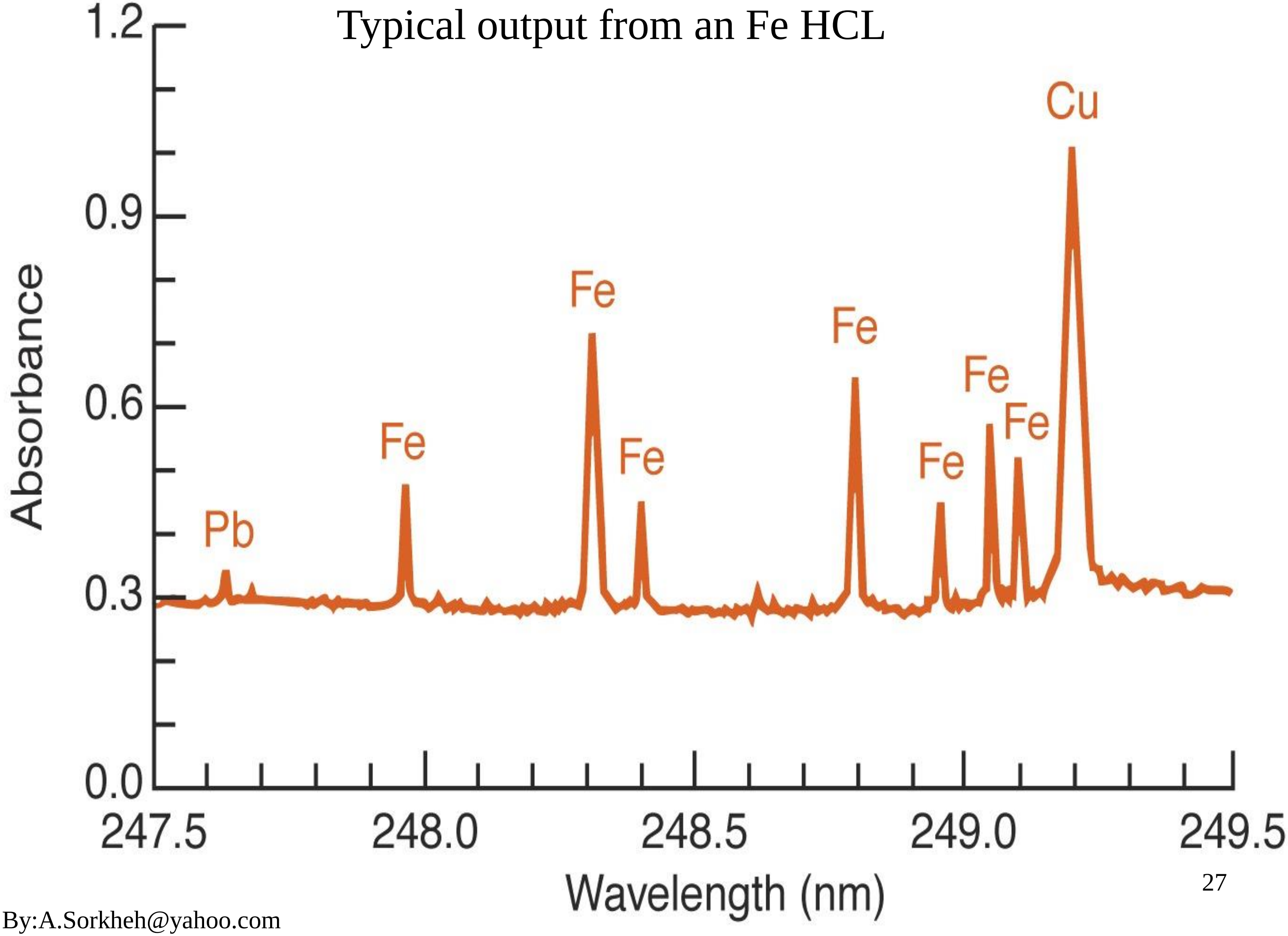
- **A *red glow* is observed in lamps filled with *neon*, while *argon* filled lamps have a *blue glow*.**





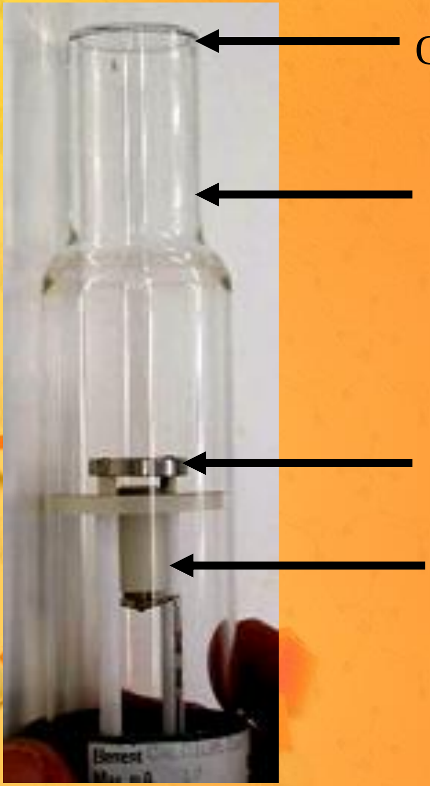
By:A.Sorkheh@yahoo.com

Typical output from an Fe HCL



By:A.Sorkheh@yahoo.com

# Hollow Cathode Lamp

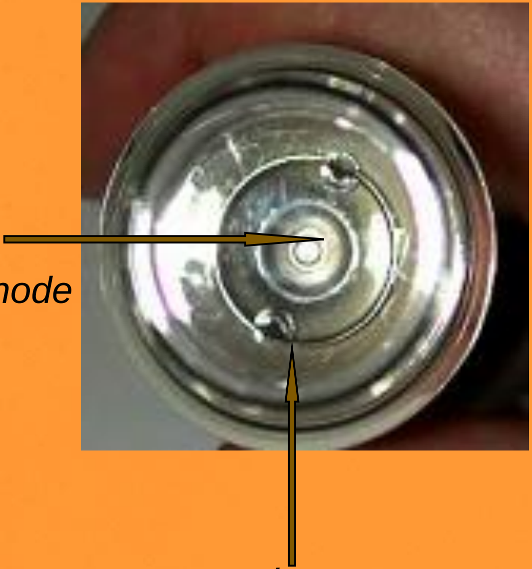


Quartz window

Pyrex body

Anode

Cathode



*cathode*

*Anode*

- **An electrical potential is applied between the anode and cathode and some of the fill gas atoms are ionized.**

- ***Sputtering*: The positively charged ions collide with the negatively charged cathode and dislodge metal atoms in a process called "sputtering."**

- ***Excitation & Emission*: Sputtered metal atoms are further excited to emission through impact with the fill gas.**

- **Hollow cathode lamps have a finite lifetime. With extended use, the sputtering process removes some of the metal atoms from the cathode and these are deposited elsewhere.**

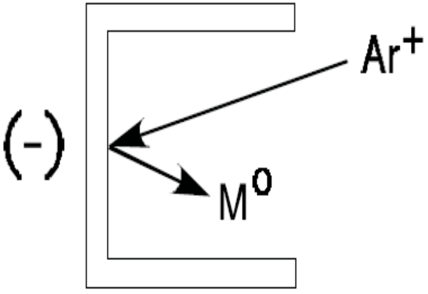
- **Fill gas is absorbed in the sputtered metal, on the glass walls and also absorbed into the glass from bombardment.**

- **Lamps for volatile elements age faster due to more rapid sputtering of the cathode.**

By:A.Sorkheh@yahoo.com

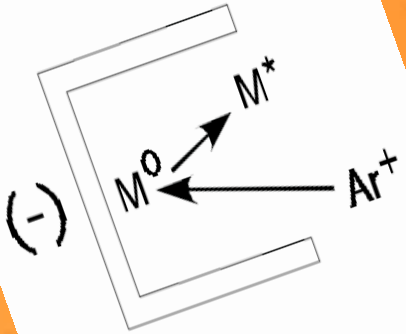
**Process:** 1. ionizes inert gas to high potential (300V)

$Ar \rightarrow Ar^+ + e^-$



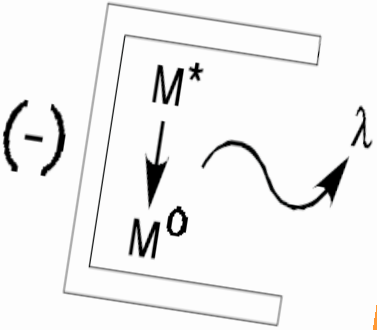
1. Sputtering

2. $Ar^+$ go to “-” cathode & hit surfaces



2. Excitation

3. As $Ar^+$ ions hit cathode, some of deposited element is excited and dislodged into gas phase (sputtering)



3. Emission

4. Excited element relaxes to ground state and emits characteristic radiation

- ➢ ***Advantage:*** **sharp lines specific for element of interest**
- ➢ ***Disadvantage:*** **can be expensive, need to use different lamp for each element tested.**

By:A.Sorkheh@yahoo.com

# Multielement Lamps

- ✓ The cathode of a hollow cathode lamp is generally constructed from a very pure metal resulting in a very pure emission spectrum.

- ✓ It is possible to construct a cathode from a mixture or alloy of several metals. The resulting "multielement" lamp can be used as a source for all the metals contained in the cathode.

- ✓ There is a wide variety of multielement lamp combinations available.

- ✓ Not all metals can be used in combination due to metallurgical properties or spectral limitations.

By:A.Sorkheh@yahoo.com

# Single-Element or Multielement Lamp?

- The emission intensity for a particular element in a multielement lamp is not as great as that for the same element in a single-element lamp. This can result in a poorer signal/noise ratio which can influence the precision of analyses and the detection limit.

- When working close to the detection limit or when the best precision is required, select a single-element lamp.

- If economics is a problem, one multielement lamp can take the place of several single-element lamps.

- A multielement lamp is adequate for routine analyses that are well above the detection limit.
- If a backup lamp is necessary for a specific determination, a multielement lamp is ideal.

- If an infrequent analytical determination is required for several elements, a multielement lamp can offer considerable savings.

# Hollw cathod lamp SPECIFICATIONS

## Windows

High purity fused quartz (Spectral range 200 - 5000nm).

## Cathode

Most of the cathode materials used in standard Cathodeon hollow cathode lamps may be used in the "see through“ design. (Unavailable elements are Rb, Cs, K). Cathode length is 19mm and bore diameter is in the range 3 to 6mm. Where material is not critical Iron is recommended for its high intensity operation.

## Fill Gas

Neon, Argon, Xenon, Krypton, Helium, Deuterium, Hydrogen.

Gas mixtures and other gases may be specified provided they are non-corrosive and non-radioactive.

Where the gas is not critical Neon or Argon are recommended based on Cathodeon standard hollow cathode lamp practice.

Fill pressure must normally be in the range of 8 to 15mbar.

## ELECTRICAL

Striking volts 350—400V

Running volts 200—320V

Maximumcontinuous current 5 - 20mA(Each cathode material has a maximum value within this range beyond which life and/or performance is reduced).

# Lamp Current

- The label on each hollow cathode lamp gives an operating and maximum current.

- The recommended operating currents are those which have been found through experience to be appropriate for most analytical situations.

- However, you may find by making tests that it is possible to get satisfactory analytical results with a particular lamp run at less than the recommended current.

- Running at lower current reduces the light output and therefore requires a higher gain setting of the instrument, but it does not harm the lamp in any way.

- On the contrary, it will extend the lamp life. Therefore, if you find that the performance (precision) is not poorer when using slightly lower currents than recommended, by all means do so.

- As a hollow cathode lamp ages, it may be necessary to increase the lamp current to the maximum current rating to give additional element light emission to equal that originally obtained when the lamp was new.

- While this will somewhat accelerate the aging of the lamp, it is a necessary compromise to obtain enough element emission for good performance.

- Analytical sensitivity may be poorer at higher current settings.
- Lamps should never be operated above the maximum current rating, as this will dramatically shorten lamp life.

- Hollow cathode lamp failure occurs when the fill gas is gradually captured on the inner surfaces of the lamp and, finally, the lamp will no longer light.

- Higher lamp currents accelerate gas depletion and cathode"sputtering" and should be avoided until the age of the lamp requires a current increase.

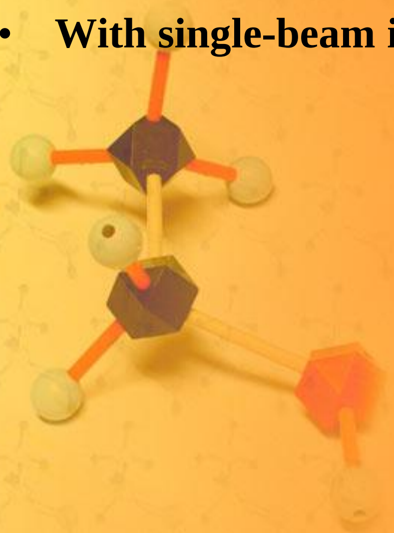
By:A.Sorkheh@yahoo.com

# Lamp Power effects on Line Signal quality

# Warm-Up Time

- **The time needed for a hollow cathode lamp to reach a constant emission intensity is short, and with a double-beam instrument no warm-up is required.**

- **With single-beam instruments, a short warm-up period is recommended.**



By:A.Sorkheh@yahoo.com

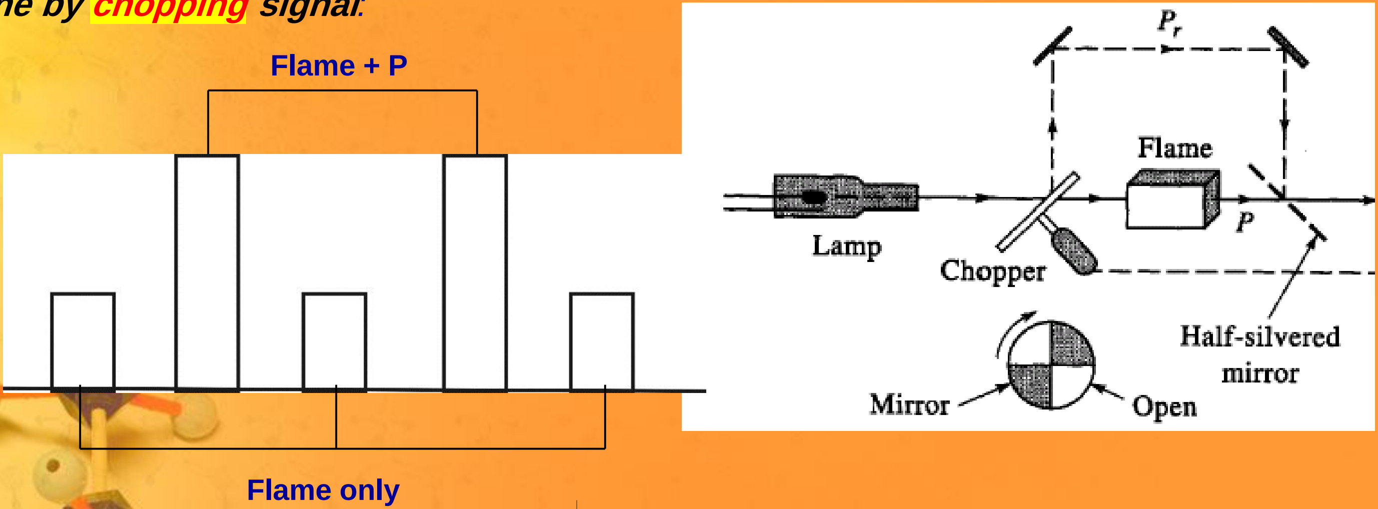
# Source Modulation (spectral interference due to flame)

- problem with working with flame in AA is that light from flame and light source both reach detector

- measure small signal from large background
- need to subtract out flames to get only light source signal ($P/P_o$)

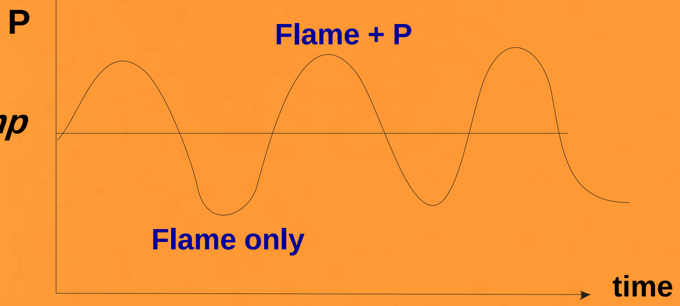
## *i. Mechanical Modulation*

*Done by chopping signal:*



## *ii. Electrical Modulation*

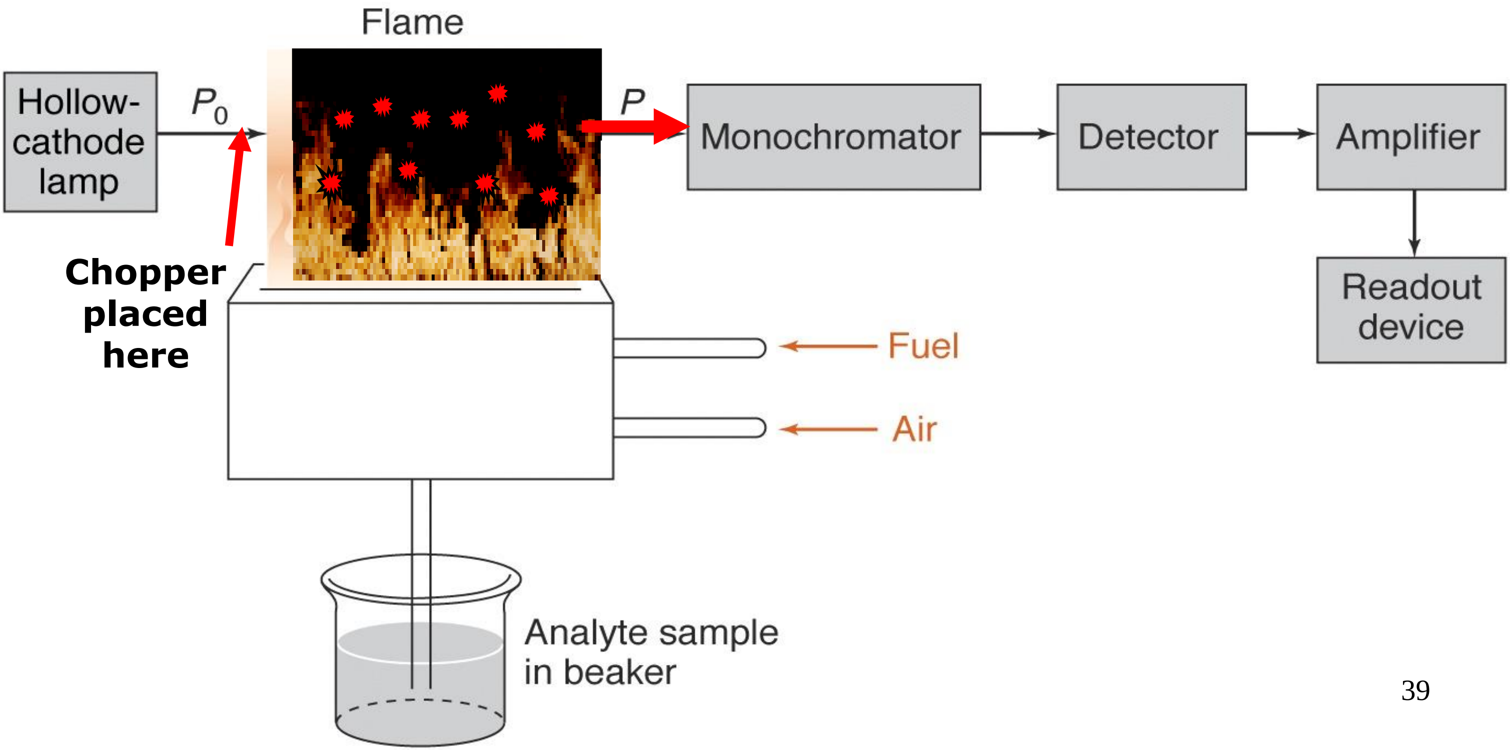
*Use an AC electrical source for lamp modulating P from lamp:*



By:A.Sorkheh@yahoo.com

# AA Source Modulation

- Need to **eliminate emission** from **analyte atoms**
- Source beam is **chopped**



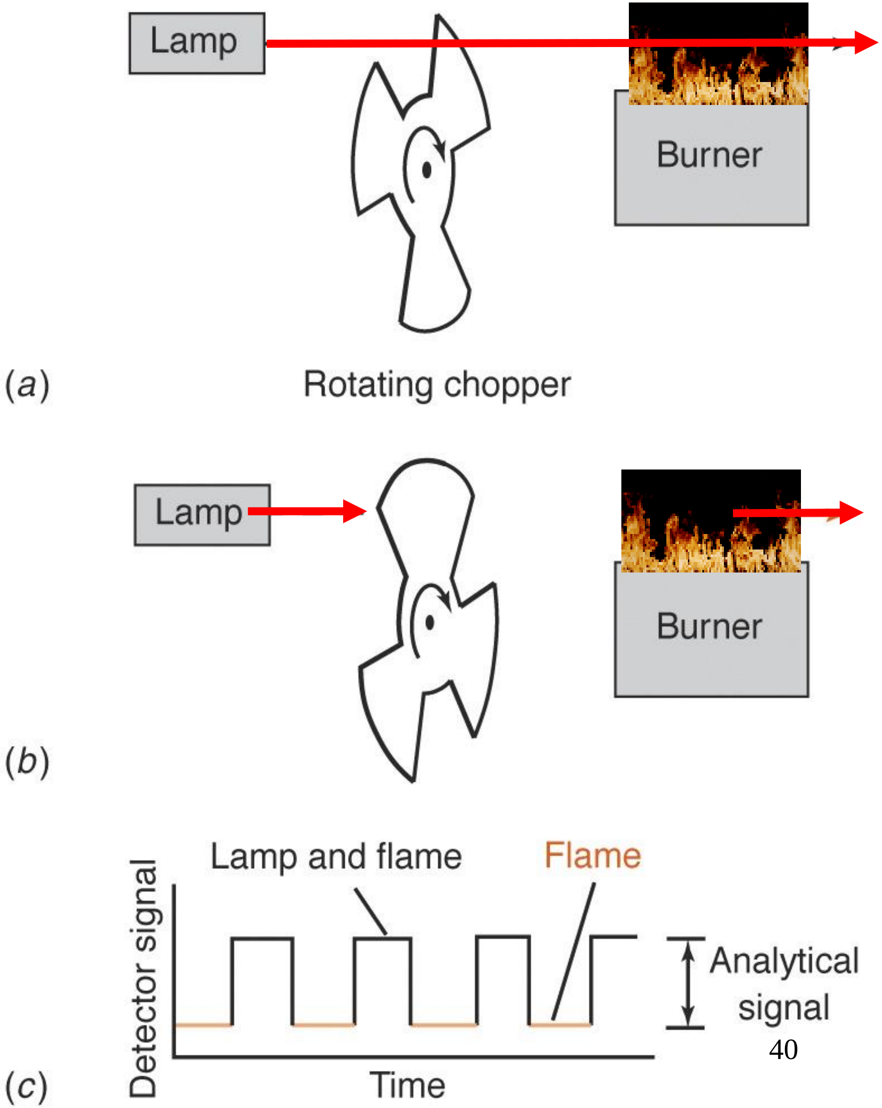
**Chopper placed here**

# Beam chopper for subtracting flame background emission

**(a)** **Lamp and flame** emission reach detector

**(b)** Only **flame** emission reaches detector

**(c) Resulting signal**

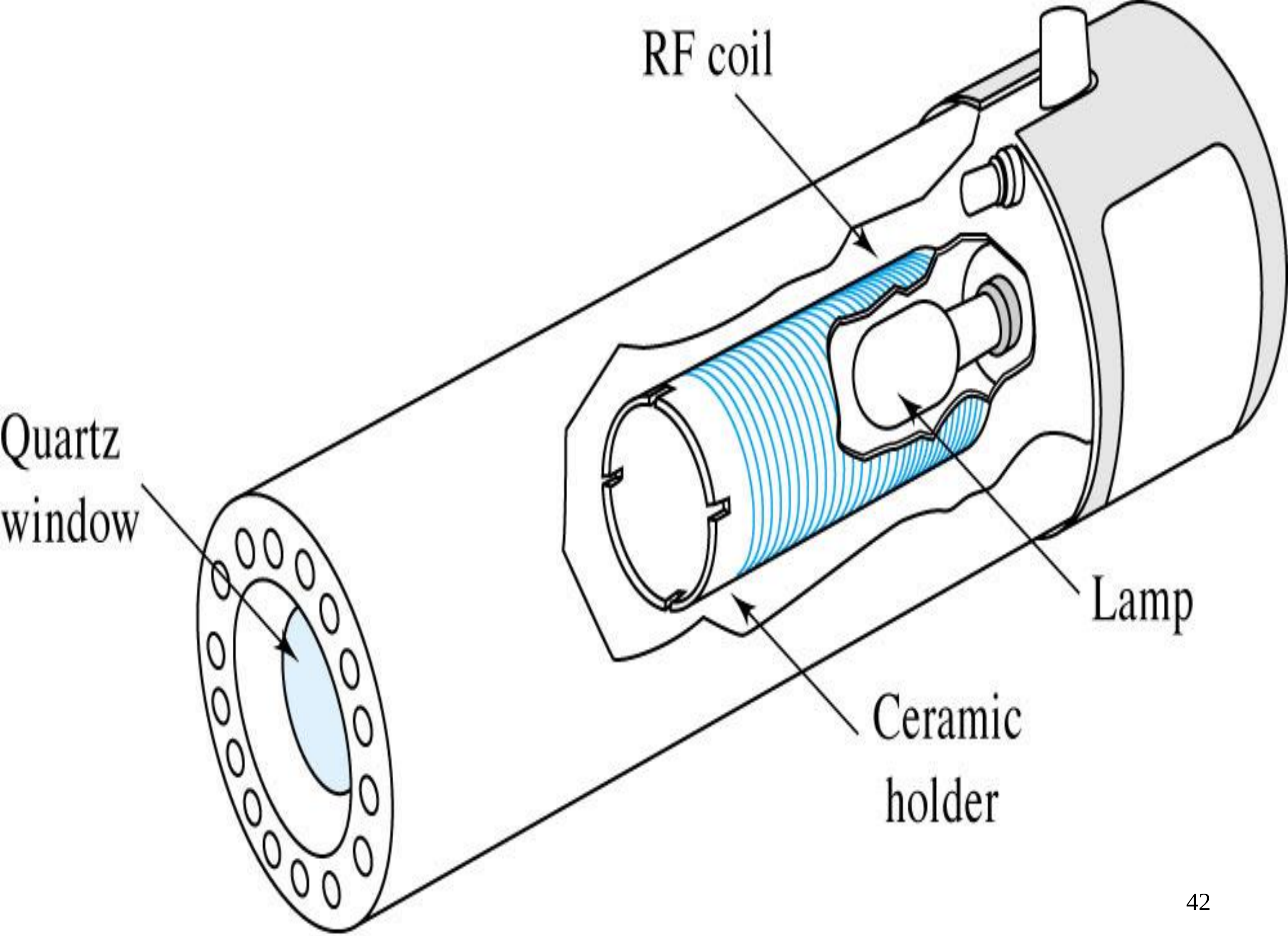


By:A.Sorkheh@yahoo.com

# Electrodeless Discharge Lamps (EDLs):

These provide radiant intensities that are usually one to two orders of magnitude greater than hollow cathode lamps. A typical lamp is constructed from a sealed quartz tube containing a few torr of an inert gas such as argon and a small quantity of the metal (or its salt) whose spectrum is of interest.

By:A.Sorkheh@yahoo.com

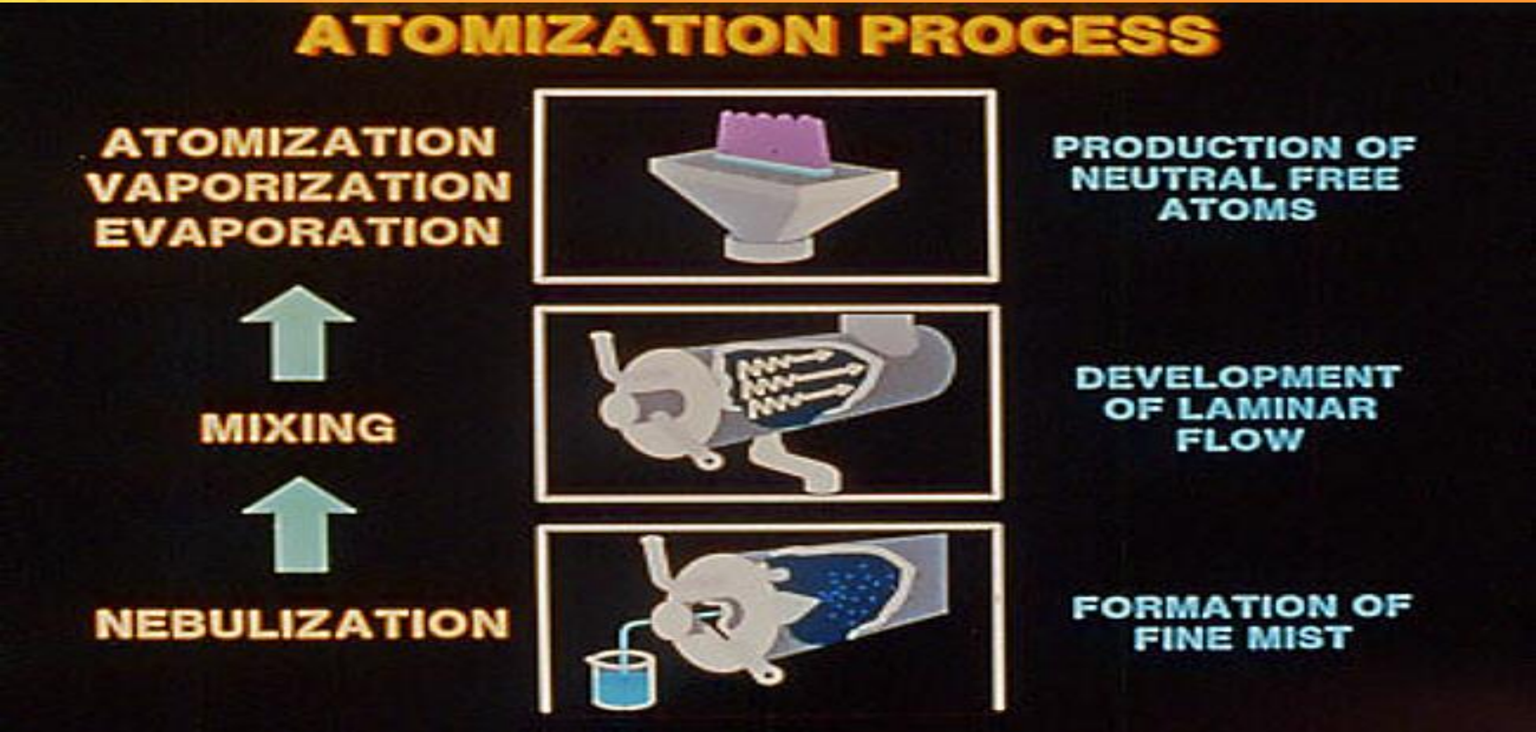


© 2007 Thomson Higher Education

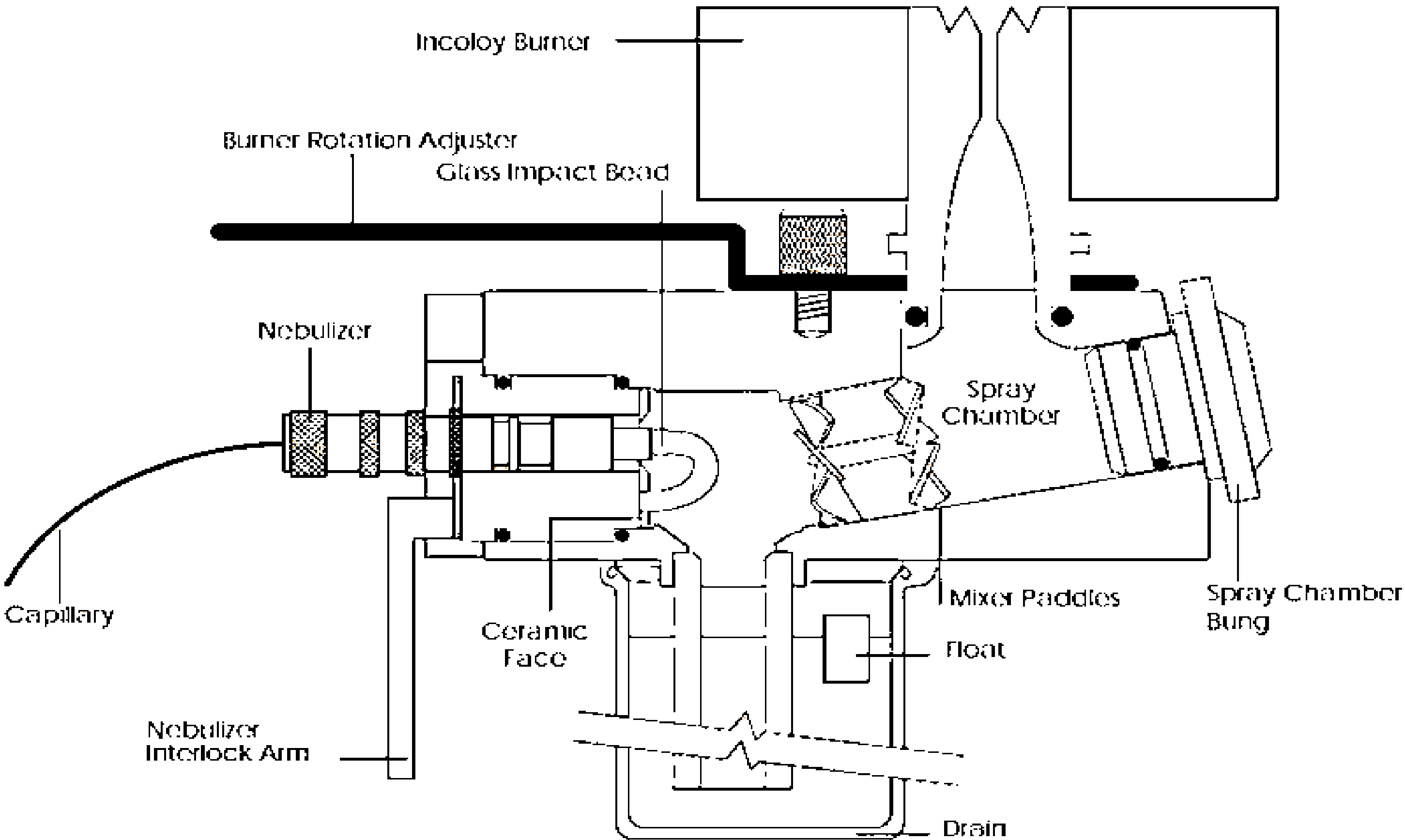
By:A.Sorkheh@yahoo.com

**“SAMPLE” chamber or Atomizer to deliver the Solution is made of :**

1. **DYNAMIC NEBULIZER**
2. **SPRAY CHAMBER**
3. **BURNER HEAD**

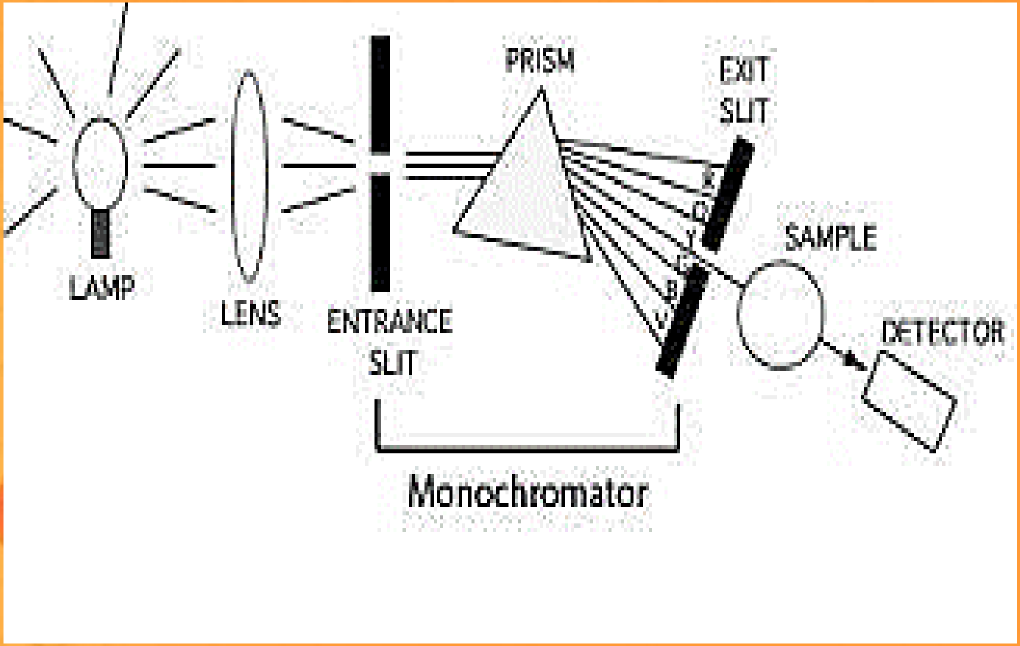


**BURNER HEAD** uses ***Dynamic Impact-Bead*** **NEBULIZER for maximum Sample Aerosol delivery to Flame for ALL types of Solutions (high salt, organic)**



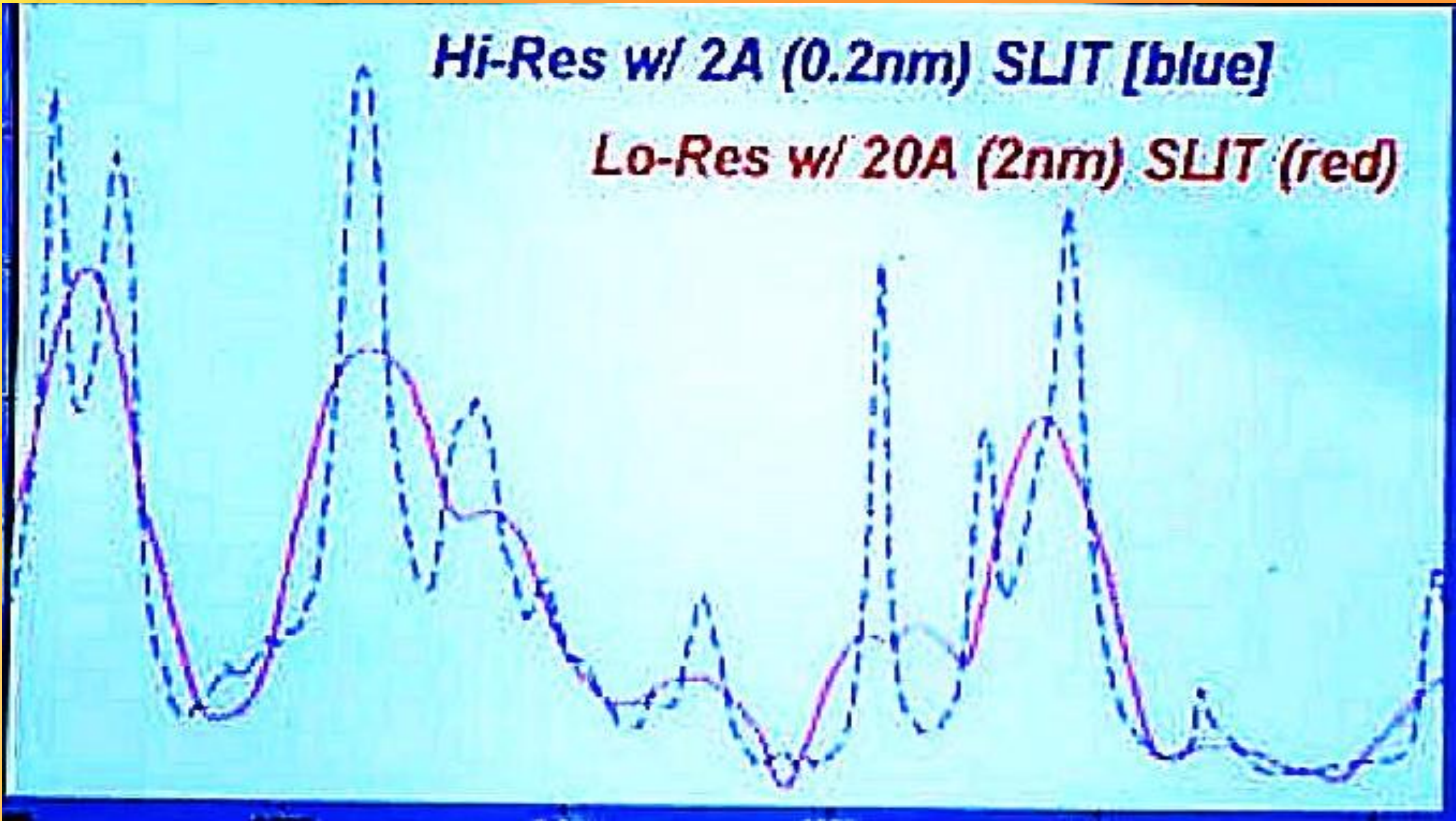
# OPTICAL SYSTEM

**For the BUCK AAS consists of a compact, med-high Resolution, *SINGLE-BEAM, 1/4M Ebert* Monochromator with variable Slits to maximize separation of Resonance Lines that come from the Hollow-Cathode Lamps**



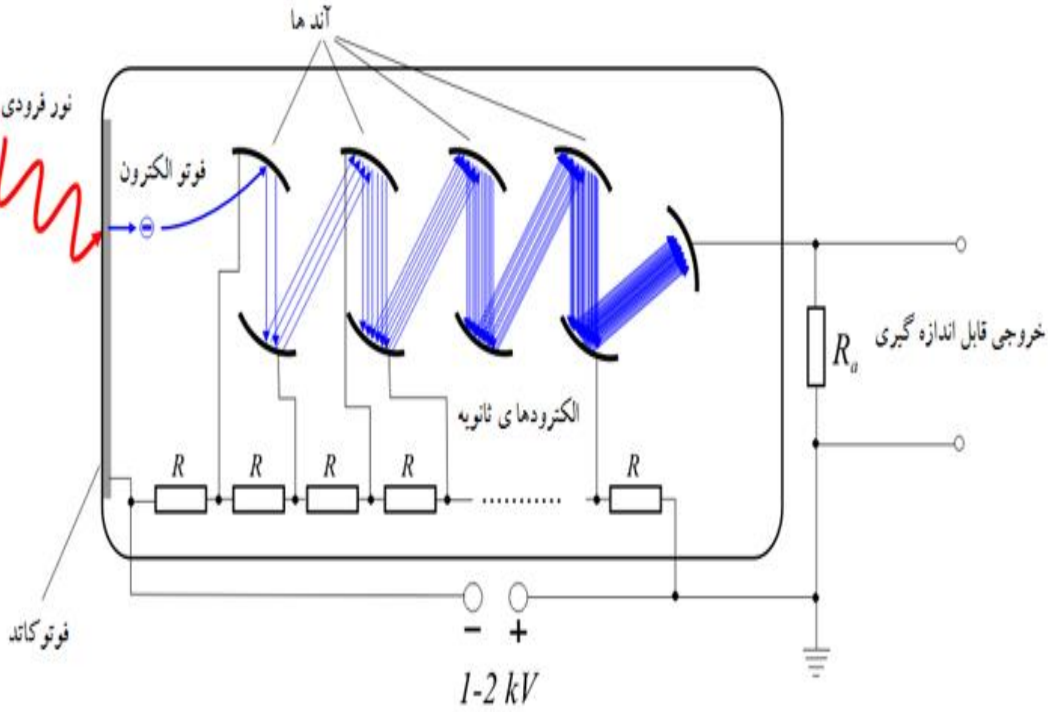
By:A.Sorkheh@yahoo.com

**Adjustable *SLITS* are used to increase the Resolution / Separation of HCL signals from Metals with "rich" Spectra (many lines); such as Fe, Ni, Mn & Co**



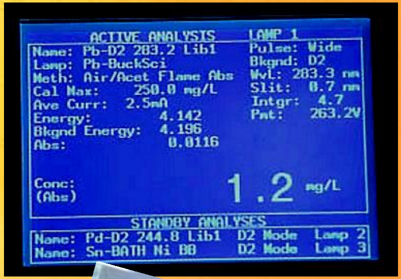
# DETECTOR

**The DETECTOR used to measure the Absorbance SIGNAL is a high-Gain / low-Noise *Photo-Multiplier Tube* (PMT) that is OPTIMIZED for the UV region**



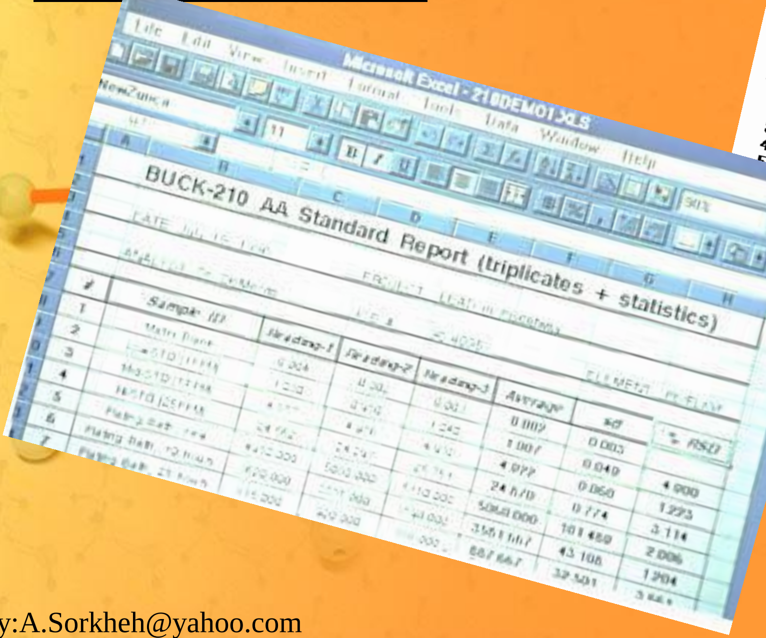
# DATA OUTPUT

**DATA OUTPUT** can be read directly from the Graphics LCD, sent to a Printer or be transferred to a Computer as an ASCII file to process in Excel, Lotus, etc.

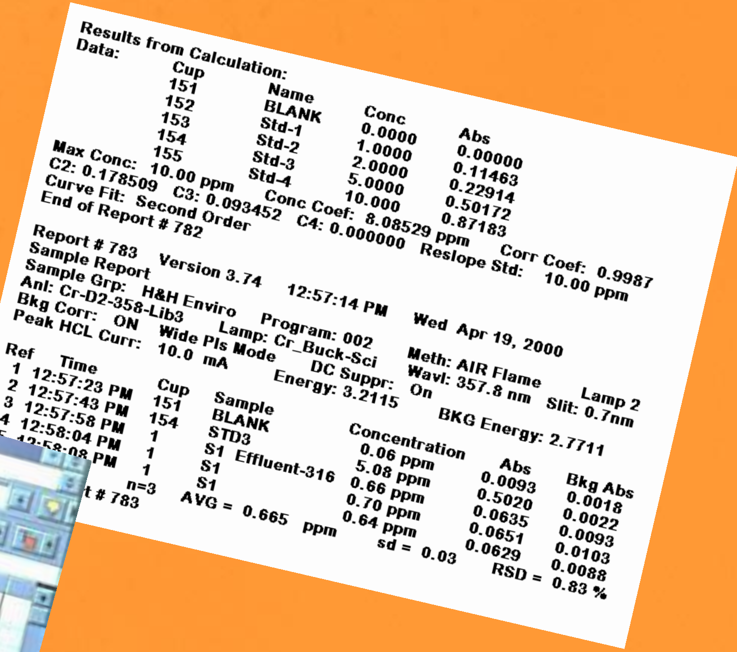


```
ACTIVE ANALYSIS                  LAMP 1
Name: Pb-D2 283.2 Lib1           Pulse: Wide
Lamp: Pb-BuckSci                 Bkgnd: D2
Meth: Air/Acet Flame Abs         WvL: 283.3 nm
Cal Max:      250.0 mg/L         Slit:  0.7 nm
Ave Curr:  2.5mA                 Intgr:  4.7
Energy:           4.142          Pmt:   263.2V
Bkgnd Energy:     4.196
Abs:               0.0116

Conc:
(Abs)                      1.2 mg/L
             STANDBY ANALYSES
Name: Pd-D2 244.8 Lib1    D2 Mode   Lamp 2
Name: Sn-BATH Ni BB       D2 Mode   Lamp 3
```



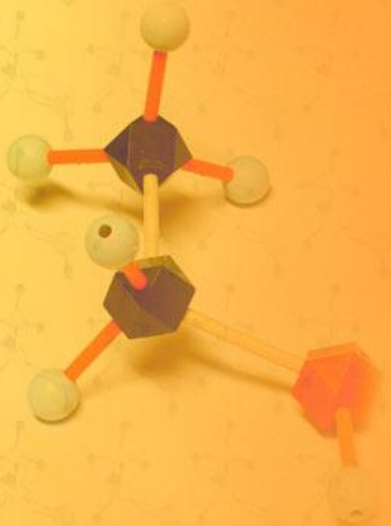
BUCK-210 AA Standard Report (triplicates + statistics)



```
Results from Calculation:
Data:
        Cup     Name     Conc      Abs
        151     BLANK    0.0000    0.00000
        152     Std-1    1.0000    0.11463
        153     Std-2    2.0000    0.22914
        154     Std-3    5.0000    0.50172
        155     Std-4    10.000    0.87183
Max Conc:  10.00 ppm   Conc Coef:  8.08529 ppm    Corr Coef:  0.9987
C2: 0.178509  C3: 0.093452  C4: 0.000000  Reslope Std:  10.00 ppm
Curve Fit:  Second Order
End of Report # 782

Report # 783    Version 3.74    12:57:14 PM    Wed  Apr 19, 2000
Sample Report
Sample Grp:  H&H Enviro   Program: 002
Anl: Cr-D2-358-Lib3    Lamp: Cr_Buck-Sci    Meth: AIR Flame      Lamp 2
Bkg Corr:  ON   Wide Pls Mode    DC Suppr:  On   Wavl: 357.8 nm   Slit: 0.7nm
Peak HCL Curr:  10.0  mA    Energy: 3.2115       BKG Energy: 2.7711
Ref   Time
 1 12:57:23 PM   Cup   Sample
 2 12:57:43 PM   151   BLANK               Concentration
 3 12:57:58 PM   154   STD3                  0.06 ppm     Abs      Bkg Abs
 4 12:58:04 PM   1     S1  Effluent-316      5.08 ppm    0.0093    0.0018
 5 12:58:08 PM   1     S1                    0.66 ppm    0.5020    0.0022
                 1     S1                    0.70 ppm    0.0635    0.0093
              n=3    AVG =  0.665  ppm       0.64 ppm    0.0651    0.0103
t # 783                                       sd =  0.03 0.0629    0.0088
                                                       RSD =  0.83 %
```

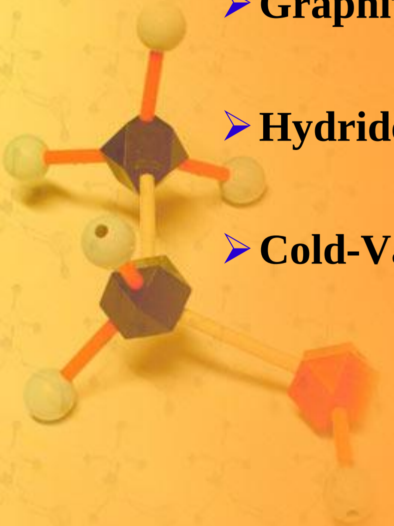
# فیلم یا عکس



By:A.Sorkheh@yahoo.com

# AA Techniques(Atomizers)

**There are several MODES of Analysis for Atomic Absorption Spectroscopy:**



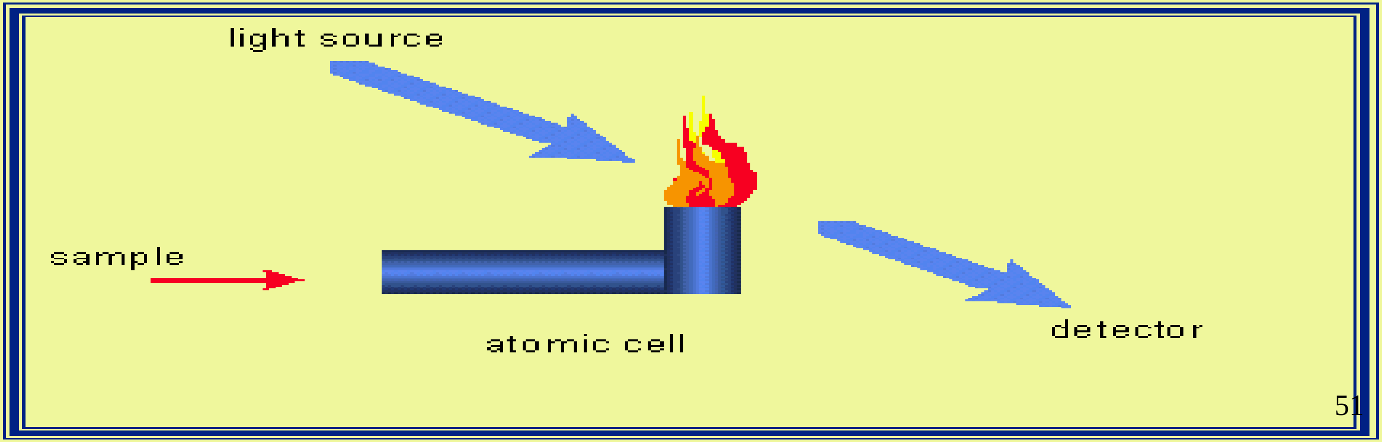
- **Flame AAS**
- **Graphite Furnace AAS**
- **Hydride Generation AAS**
- **Cold-Vapor Mercury AAS**

By:A.Sorkheh@yahoo.com

# AA Techniques

## ➢ Flame AAS

- ✓ Air-Acetylene (~2100oC) or Nitrous Oxide (~2300oC) Flames allow the determination of most Metals with high-PPB / low-PPM Detection Limits
- ✓ Simplest atomization of gas/solution/solid
- ✓ Laminar flow burner - stable "sheet" of flame
- ✓ Flame atomization best for reproducibility (precision) (<1%)
- ✓ Relatively insensitive - incomplete volatilization, short time in flame



# Flame Atomizers (Continuous Atomizers)

## Burner Assembly:

**Design:** **Polyethylene Pre-mix chamber, glass impact bead dispersion**

**Burner Head: Titanium; air-acetylene head - 4" x 0.026" single slot(nitrous oxide head - 2" x 0.019" single slot)**

**Adjustments: Horizontal g**

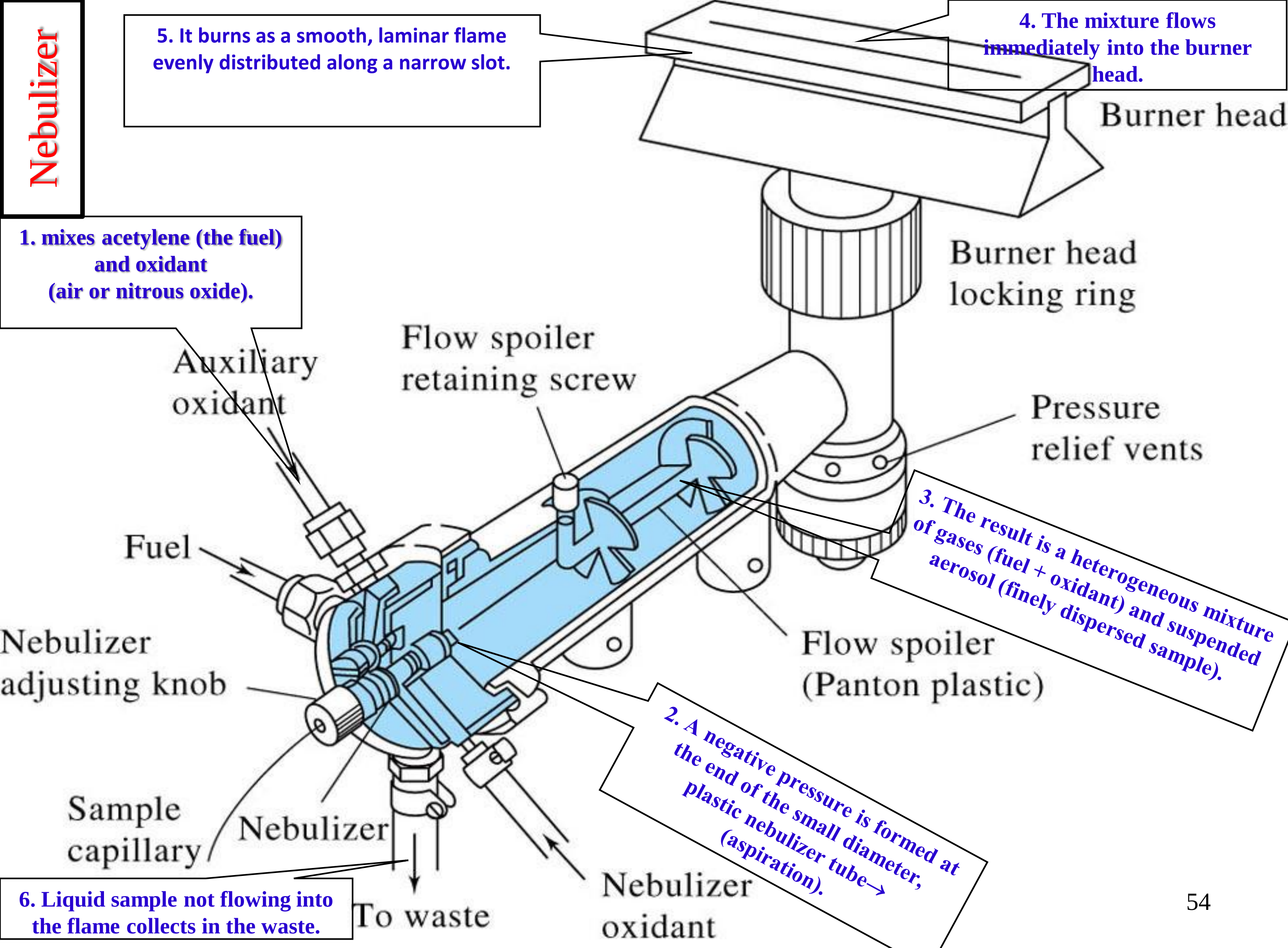
1. **The simplest is a *turbulent flow burner* that is very similar to conventional Bunsen burner. This type of burner suffers from fluctuations in temperature since there is no good mechanism for homogeneous mixing of fuel and oxidant. The drop size of nebulized sample is also inhomogeneous which adds to fluctuations in signal. The path length of radiation through the flame is small which suggests a lower sensitivity of the technique.**

By:A.Sorkheh@yahoo.com

- **Turbulent flow burners** are also susceptible to flashback. These drawbacks were overcome using the most widely used laminar flow burner where quite flames and long path length are obtained. Flashback is avoided and very homogeneous mixing between fuel, oxidant, and droplets take place. Larger droplets are excluded and directed to a waste container. A schematic representation of the burner is shown below:

By:A.Sorkheh@yahoo.com

# Nebulizer



1. mixes acetylene (the fuel) and oxidant (air or nitrous oxide).

2. A negative pressure is formed at the end of the small diameter, plastic nebulizer tube→ (aspiration).

3. The result is a heterogeneous mixture of gases (fuel + oxidant) and suspended aerosol (finely dispersed sample).

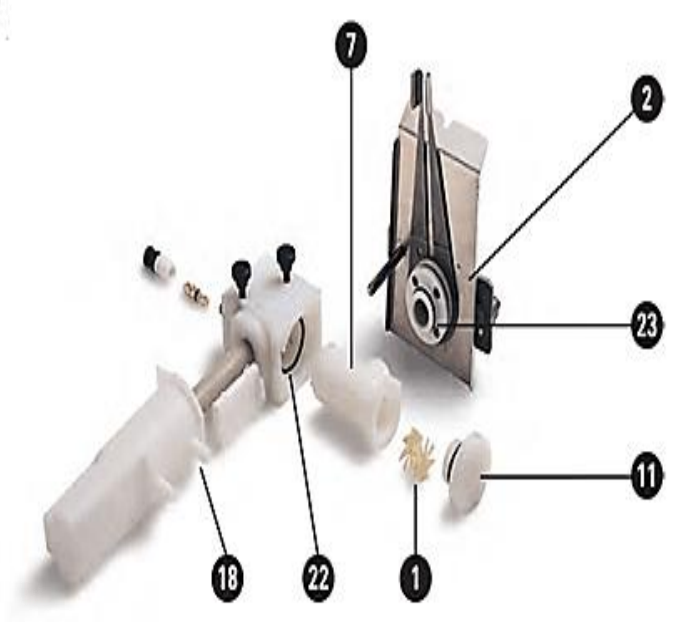
4. The mixture flows immediately into the burner head.

5. It burns as a smooth, laminar flame evenly distributed along a narrow slot.

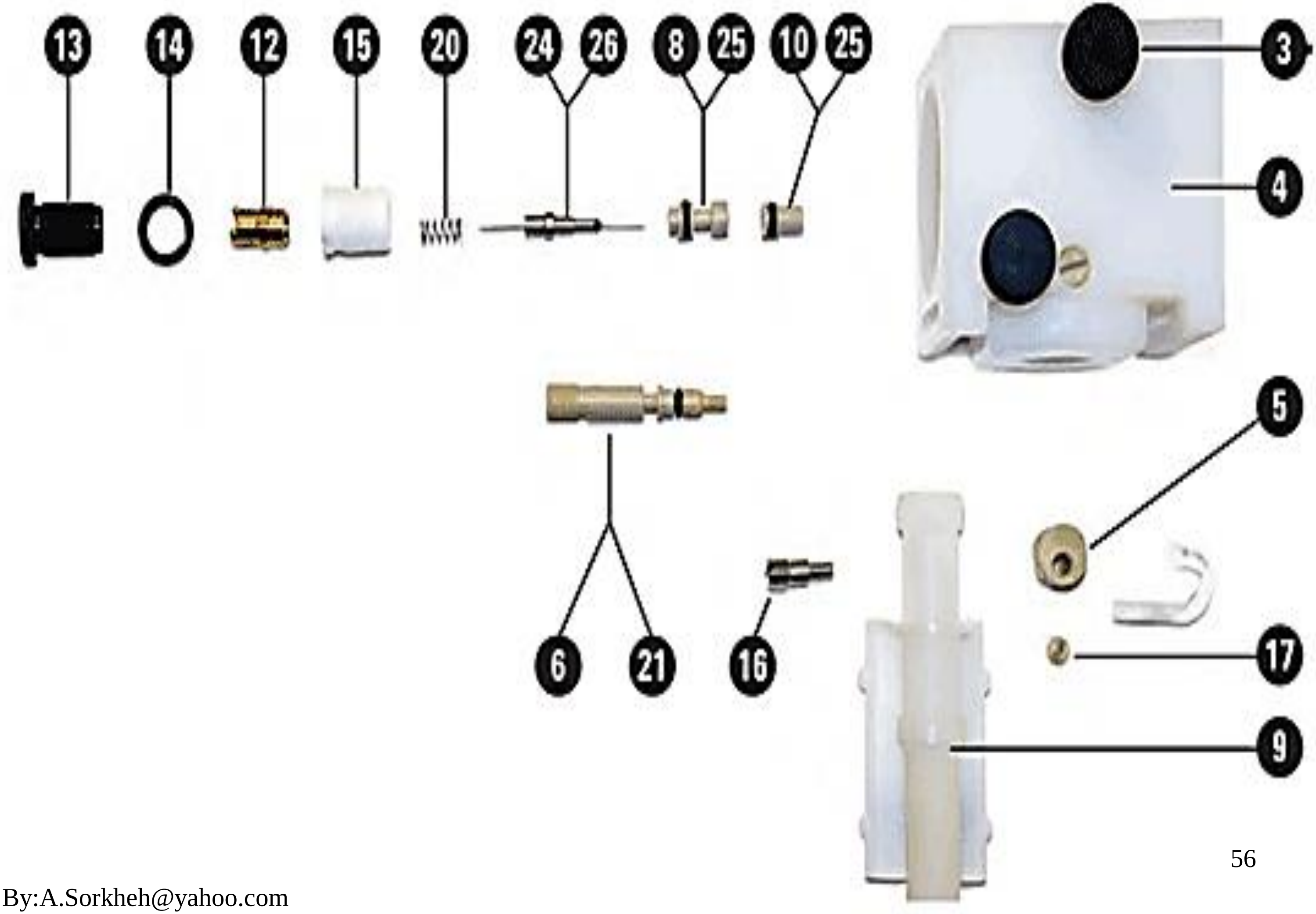
6. Liquid sample not flowing into the flame collects in the waste.

# Spray Chamber

| Item | Description |
|---|---|
| 1 | Mixing paddle, fluorinated, 1/pk |
| | Mixing paddles, fluorinated, 5/pk |
| 2 | Mark 7 air/acetylene burner |
| | Mark 7 nitrous oxide/acetylene burner |
| Not Shown | Burner cleaning and alignment card, 100/pk |
| 7 | Spray chamber body, fluorinated |
| 11 | Pressure relief bung, fluorinated |
| | O-ring for spray chamber bung, nitrile for aqueous solutions |
| | O-ring for spray chamber bung, organic solvent compatible |
| 18 | Kit, fluorinated liquid trap, drain tube with integrated float and magnet |
| 22 | O-ring spray chamber, nitrile |
| | O-ring spray chamber, organic solvent compatible |
| 23 | O-ring burner base, nitrile for aqueous solutions |
| | O-ring burner base, organic solvent compatible |



# Exploded view of nebulizer block from the spray chamber



By:A.Sorkheh@yahoo.com

| Item | Description |
|---|---|
| 3 | Spray chamber screw mounting |
| 4 | Nebulizer block, fluorinated (excludes integrated nebulizer) |
| 5 | Clamp bead adjuster |
| 6 | Screw bead adjuster |
| 8 | Nebulizer capillary guide |
| 9 | Kit, fluorinated liquid trap, drain tube with integrated float and magnet |
| 10 | Venturi nebulizer, PEEK |
| 12 | Clamp bush guide |
| 13 | Screw capillary adjuster |
| 14 | Nut lock  M12 x 0.5 |
| 15 | Bearing bush nebulizer |
| 16 | Screw lock dog point M8 |
| 17 | Screw M6 x 6 Ertalyte |
| 18 | Kit, fluorinated liquid trap, drain tube with integrated float and magnet |
| 20 | Spring nebulizer |
| 21 | O-ring screw bead adjuster, nitrile |
| 21 | O-ring screw bead adjuster organic solvent compatible |
| 24 | O-ring 1/32 in id x 3/32 in od x 1/32 in nitrile |
| 25 | O-ring 3/16 in id x 5/16 in od x 1/16 in nitrile |
| 25 | O-ring 3/16 in id x 5/16 in od x 1/16 in organic solvent compatible |
| 26 | Nebulizer Capillary Kit Includes capillary assembly, capillary guide, nebulizer spring, nebulizer cleaning wire ,Hi-Vac capillary tubing and standard capillary tubing |
| Not Shown | Glass impact beads, 5/pk |
| Not Shown | PTFE impact beads (for use with HF digests), 5/pk |
| Not Shown | Mark 7 O-ring kit, for aqueous samples. Includes O-rings to suit screw bead adjuster (1 ea), nebulizer capillary assembly (1 ea), capillary guide and venturi (7 ea), spray chamber bung and burner (3 ea), and nebulizer block (1 ea). |
| Not Shown | Mark 7 O-ring kit, for organic solvents. Includes O-rings to suit screw bead adjuster (1 ea), nebulizer capillary assembly (1 ea), capillary guide and venturi (2 ea), spray chamber bung and burner (2 ea), and nebulizer block (1 ea). |
| Not Shown | Drain tubing for aqueous solutions, per meter, 2 m length required |
| Not Shown | Drain tubing for organic solvents, nitrile rubber, per meter, 2 m length required |
| Not Shown | Venturi extraction tool |
| Not Shown | Nebulizer capillary extraction tool |

# Advantages:

1. Uniform dropsize
2. Homogeneous flame
3. Quiet flame and a long path length

# Disadvantages:

1. Flash back if $V_{burning} > V_{flow}$
2. ~90% of sample is lost
3. Large mixing volume

# **Types of Flame/Flame Structure** – *selection of correct flame region is important for optimal performance*

**a) primary combustion zone – blue inner cone (blue due to emission from $C_2$, CH & other radicals)**

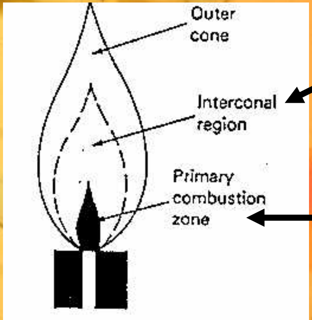
- **not in thermal equilibrium and not used**

**b) interconal region**

- **region of highest temperature (rich in free atoms)**
- **often used in spectroscopy**
- **can be narrower in some flames (hydrocarbon) tall in others (acetylene)**

**c) outer cone**

- **cooler region**
- **rich in $O_2$ (due to surrounding air)**
- **gives metal oxide formation**

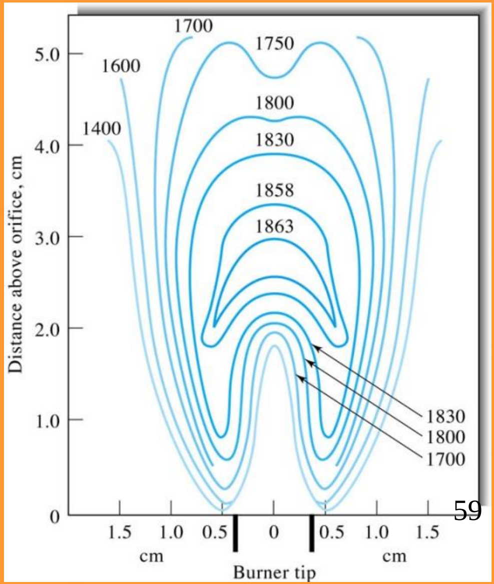


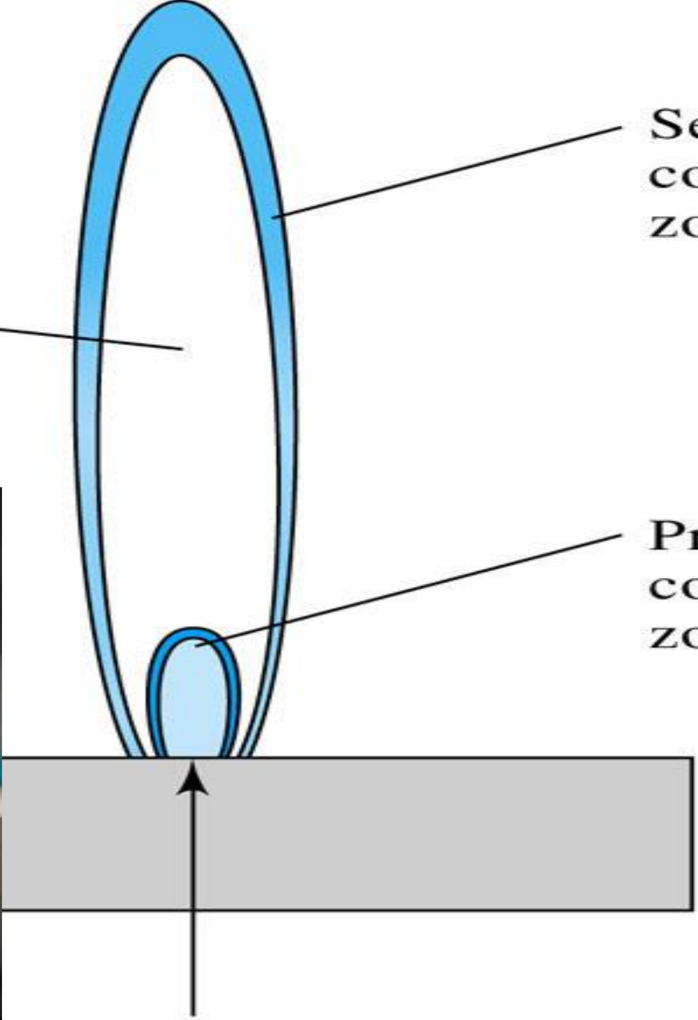
**Primary region for spectroscopy**

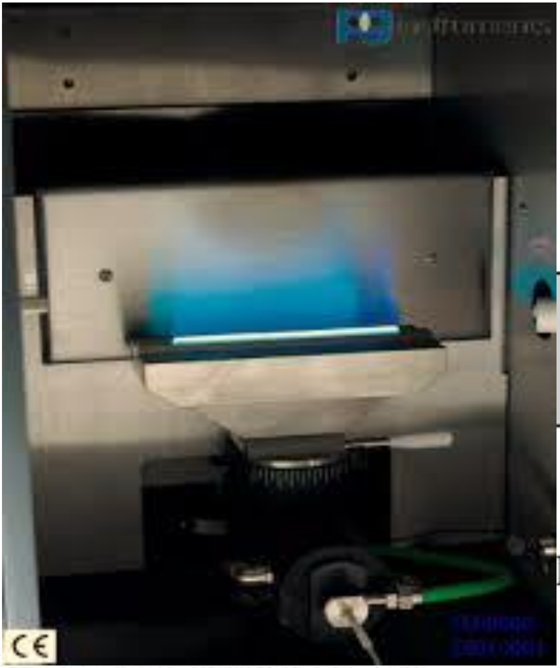
**Not in thermal equilibrium and not used for spectroscopy**

***Flame profile: depends on type of fuel and oxidant and mixture ration***

**Temperature varies across flame – need to focus on correct part of flame**



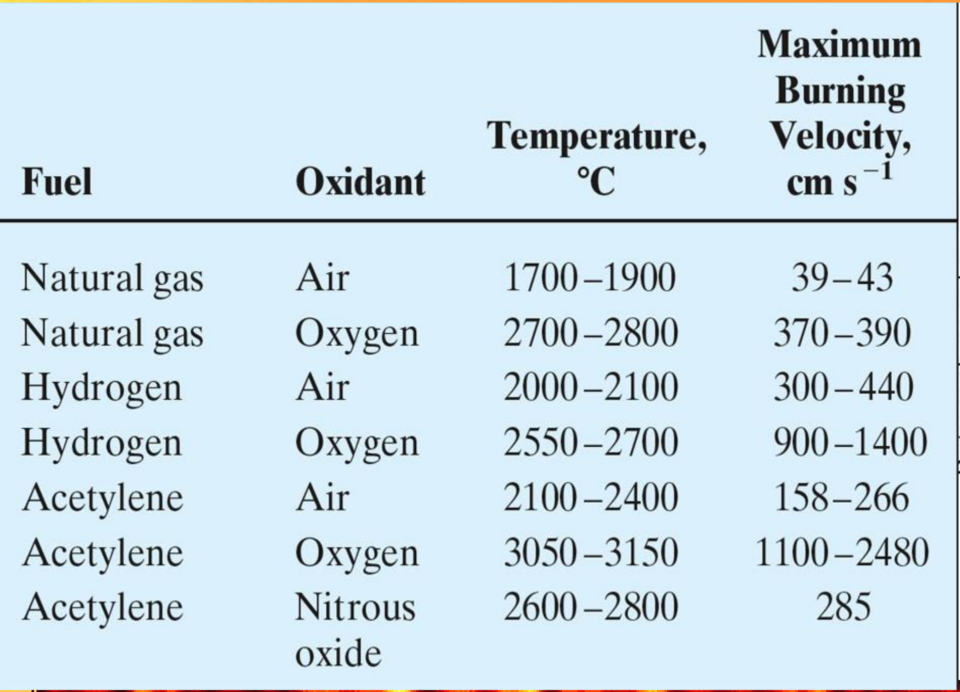

Secondary combustion zone
Interzonal region
Primary combustion zone
Fuel-oxidant mixture



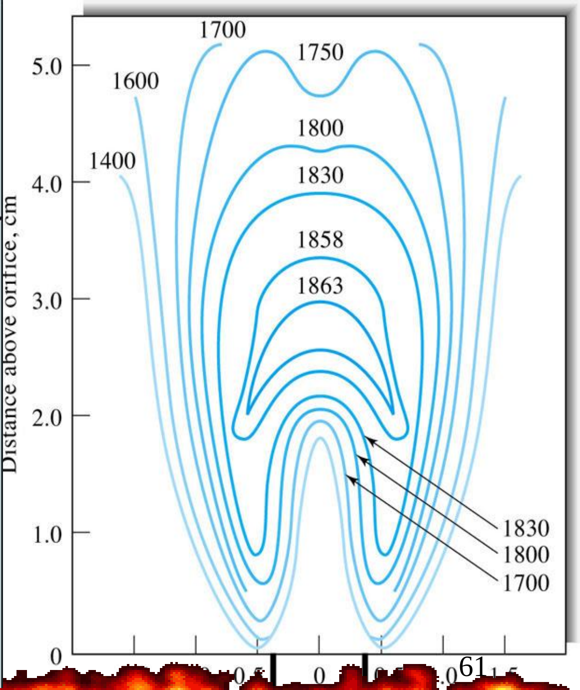
# ➢FLAMES in AAS

i. Different mixes and flow rates give different temperature profile in flame

- gives different degrees of excitation of compounds in path of light source



| Fuel | Oxidant | Temperature, °C | Maximum Burning Velocity, cm s$^{-1}$ |
|---|---|---|---|
| Natural gas | Air | 1700–1900 | 39–43 |
| Natural gas | Oxygen | 2700–2800 | 370–390 |
| Hydrogen | Air | 2000–2100 | 300–440 |
| Hydrogen | Oxygen | 2550–2700 | 900–1400 |
| Acetylene | Air | 2100–2400 | 158–266 |
| Acetylene | Oxygen | 3050–3150 | 1100–2480 |
| Acetylene | Nitrous oxide | 2600–2800 | 285 |

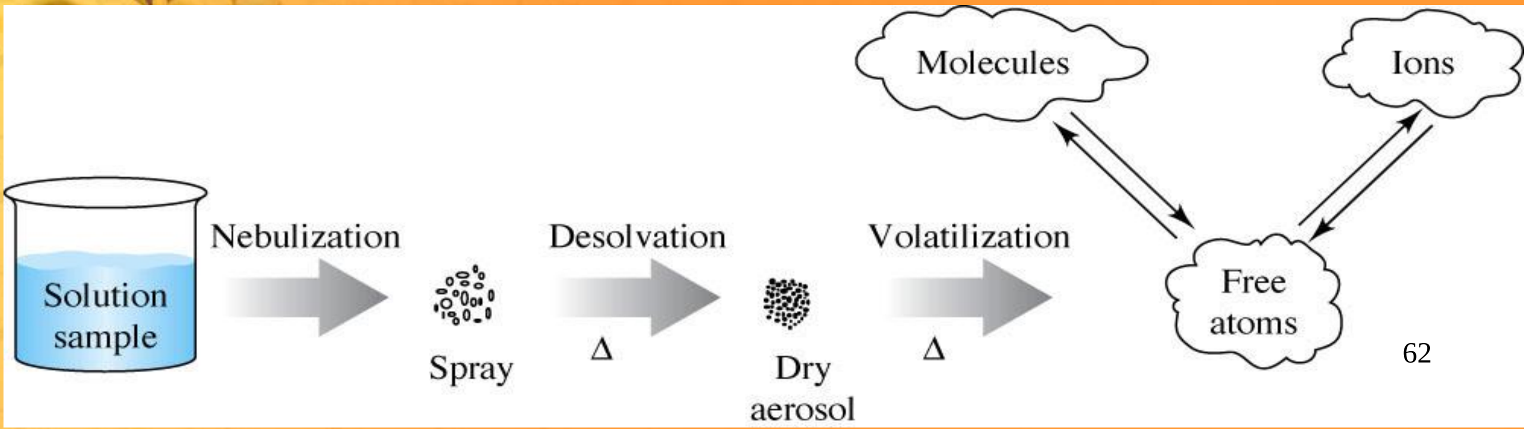


# ***Sample Atomization*** *– expose sample to flame or high-temperature*

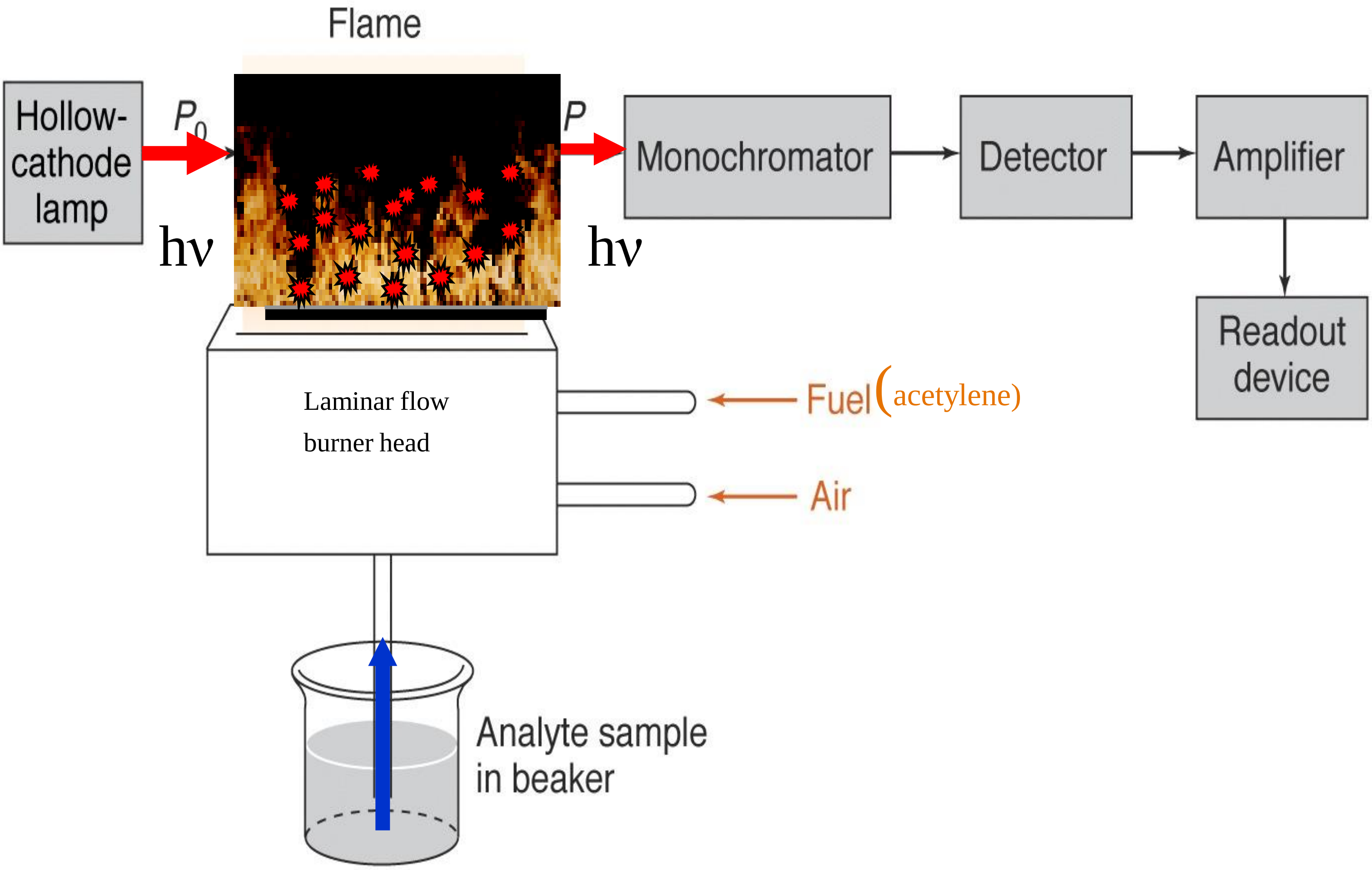
i. **Need to break sample into atoms to observe atomic spectra**

ii. **Basic steps:**

- **a) <u>Nebulization</u> – solution sample, get into fine droplets by spraying thru thin nozzle or passing over vibrating crystal.**
- **b) <u>Desolvation</u> - heat droplets to evaporate off solvent just leaving analyte and other matrix compounds**
- **c) <u>Volatilization</u> – convert solid analyte/matrix particles into gas phase**
- **d) <u>Dissociation</u> – break-up molecules in gas phase into atoms.**
- **e) <u>Ionization</u> – cause the atoms to become charged**
- **f) <u>Excitation</u> – with light, heat, etc. for spectra measurement.**



- **Flame Atomization**



# Sample Introduction Methods

- **The sample is in solution or solid form??**

- **The method for sample introduction in atomic spectroscopy affects the:**

- ✓ **precision**
- ✓ **accuracy**
- ✓ **detection limit**

By:A.Sorkheh@yahoo.com

# Introduction of Solution Samples

## 1. Pneumatic Nebulizers

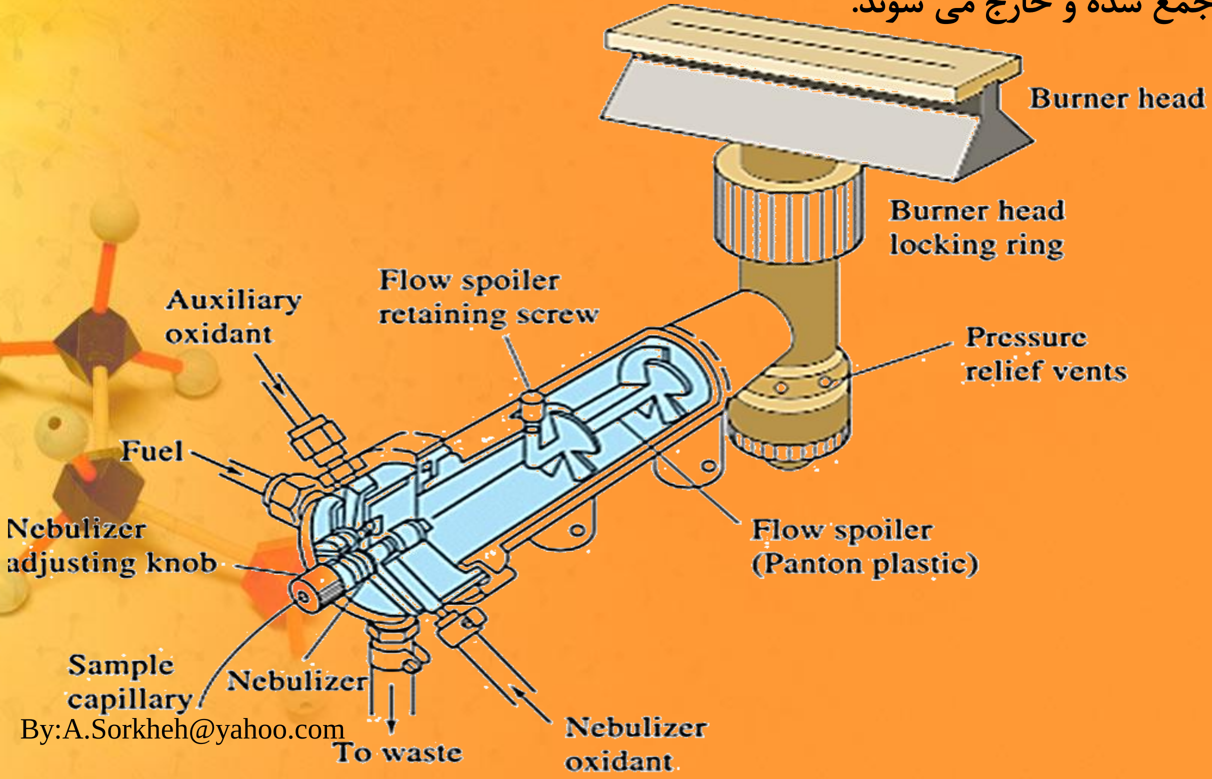
- ✓ For Samples in solution
- ✓ Nebulization converts the ***solution*** into an ***aerosol*** of very fine droplets using a ***jet of compressed gas***.
- ✓ The flow of gas carries the aerosol droplets to the atomization chamber or region.
- ✓ ***Several versions of nebulizers*** are available and few are shown in the figure below:

By:A.Sorkheh@yahoo.com

# 1. Pneumatic Nebulizers

## مشعل پیش مخلوط کن

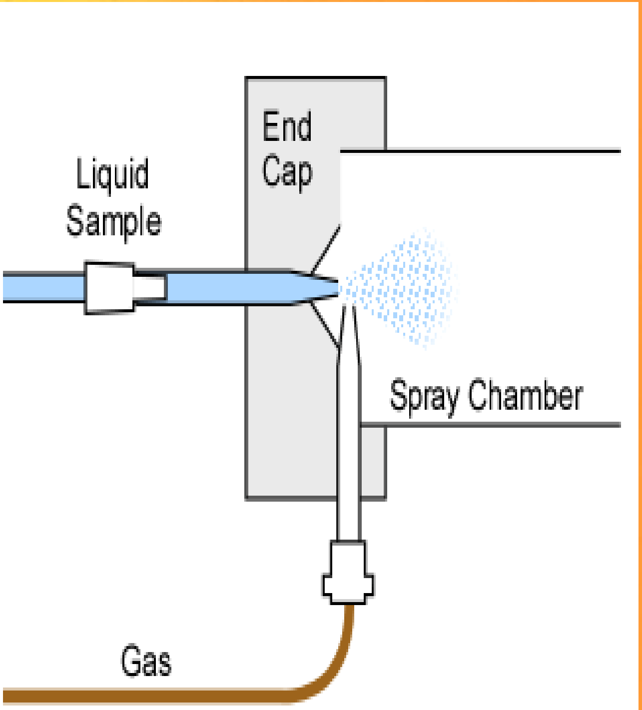
- نمونه به داخل یک محفظه بزرگ به وسیله جریانی از اکسنده مکیده می شود؛ در اینجا مه رقیق نمونه اکسنده و سوخت مخلوط می گردند و سپس به طرف دهانه مشعل رانده می شوند. قطرات بزرگتر نمونه در ته محفظه جمع شده و خارج می شوند.

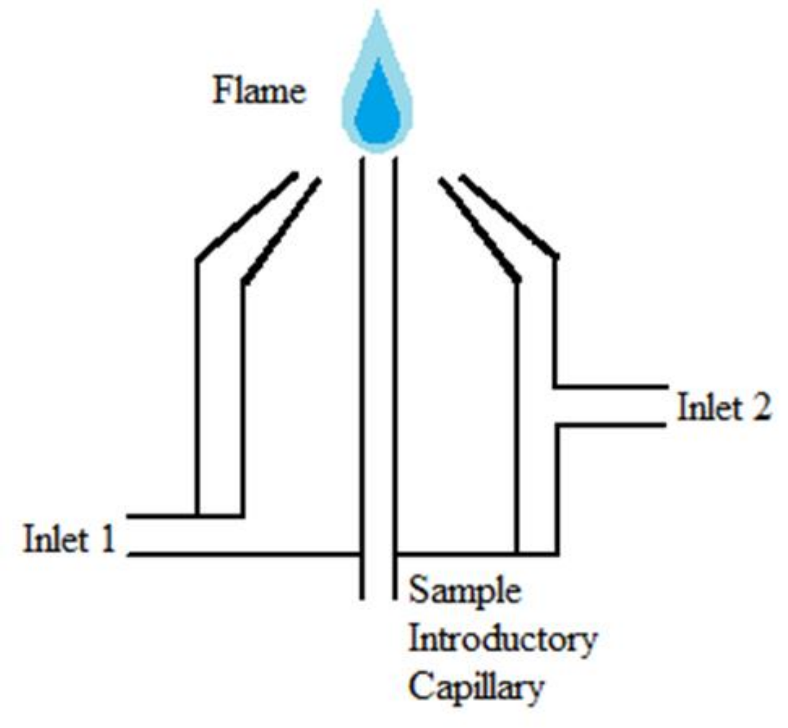


By:A.Sorkheh@yahoo.com

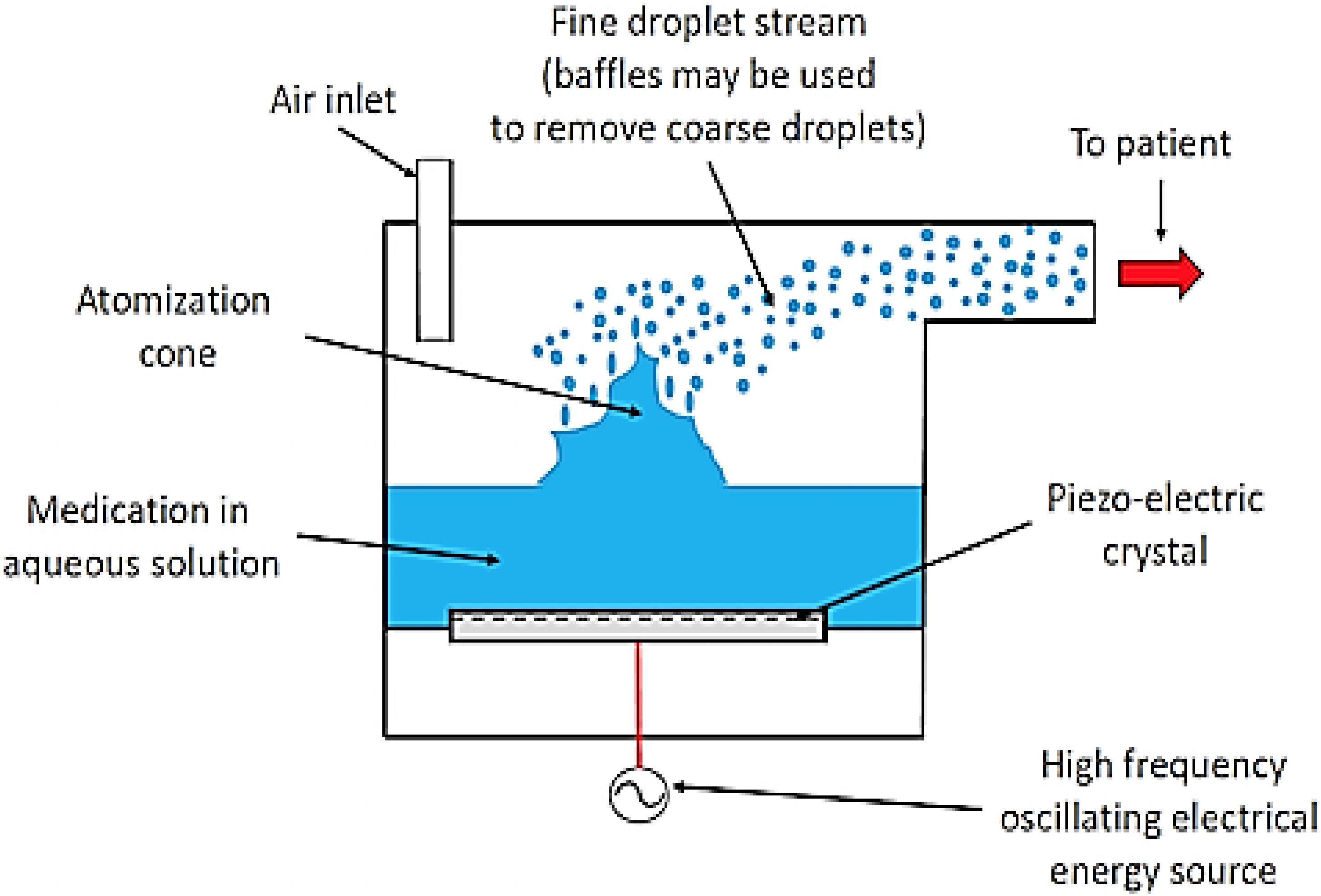
# مشعل تمام مصرف کن

- محلول، سوخت و گاز اکسنده از راهروهای جداگانه انتقال داده شده و د دهانه ای در پایه شعله با یکدیگر مخلوط می گردند.





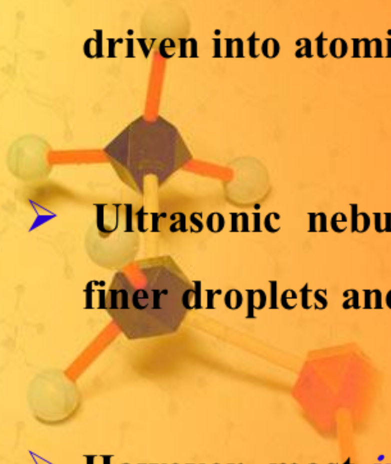
# 2. Ultrasonic Nebulizers



# Ultrasonic Nebulizers

- **Samples are pumped onto the surface of a piezoelectric crystal that vibrates in the kHz to MHz range.**

- **Such vibrations *convert* samples into *homogeneous aerosols* that can be driven into atomizers.**



- **Ultrasonic nebulization is preferred over pneumatic nebulization since finer droplets and more homogeneous aerosols are usually achieved.**

- **However, most *instruments use pneumatic nebulization*.**

By:A.Sorkheh@yahoo.com

# 3. Electrothermal Vaporization

- An accurately measured quantity of sample (few mL) is introduced into an electrically heated cylindrical chamber through which an inert gas flows.

- Usually, the cylinder is made of pyrolytic carbon but tungsten cylinders are now available.

- The signal produced by instruments which use electrothermal vaporization (ETV) is a discrete signal for each sample injection.

- Electrothemal vaporizers are called discrete atomizers to differentiate them from nebulizers which are called continuous atomizers

By:A.Sorkheh@yahoo.com

# AA Techniques

## ➢ GRAPHITE FURNACE(Flameless atomizer)

- ✓ **(GFAAS) Graphite Furnace Atomic Absorption Spectroscopy**
- ✓ **(ETAAS) Electro thermal Atomic Absorption Spectrometry**

**Uses extremely SMALL sample volumes (20µL) .**

## GFAAS Mechanism

**i.Runs a Temperature PROGRAM to DRY any liquid**

**ii.CHAR or ASH away any organic or matrix**

**iii.ATOMIZE the Analyte for PPB levels**

- ➢ **GFAAS requires a source of Cooling Water and Argon Gas to operate**

By:A.Sorkheh@yahoo.com

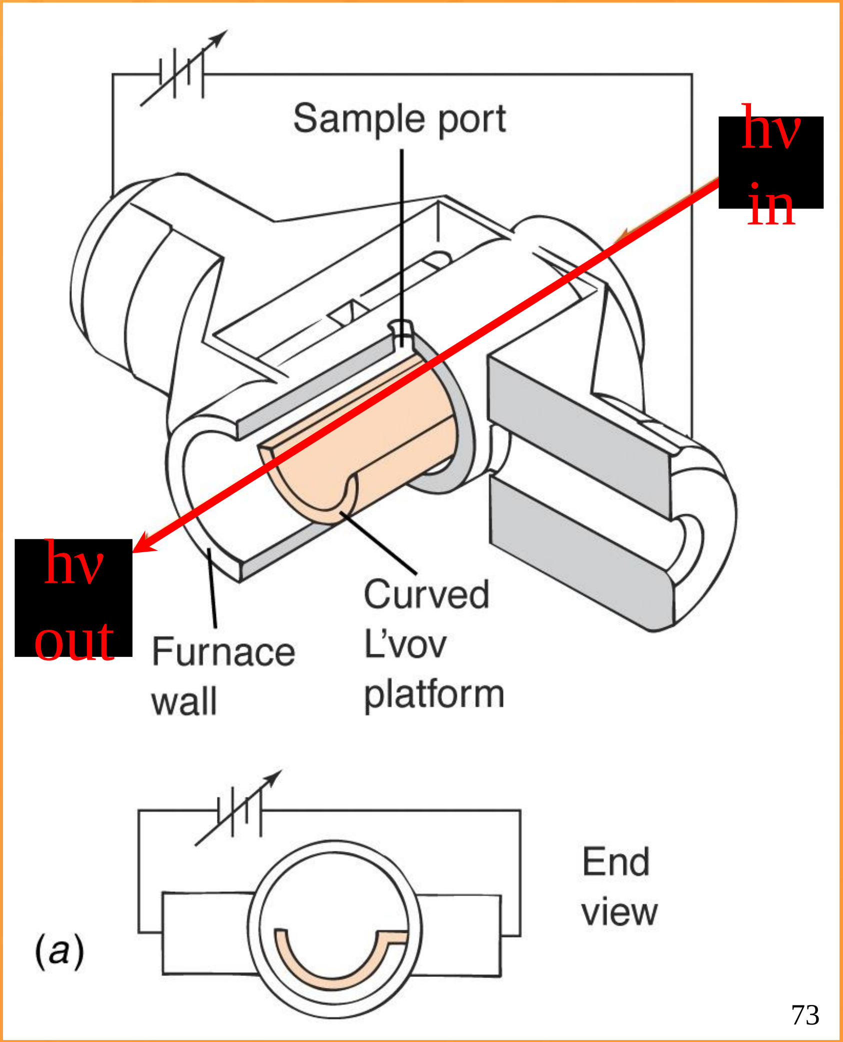
# Graphite furnace tube

## Advantages:

- highly sensitive down to pg of analyte
- long residence time
- more efficient than flame
- use with solid samples

## Disadvantages:

- poor reproducibility
- small analytical range

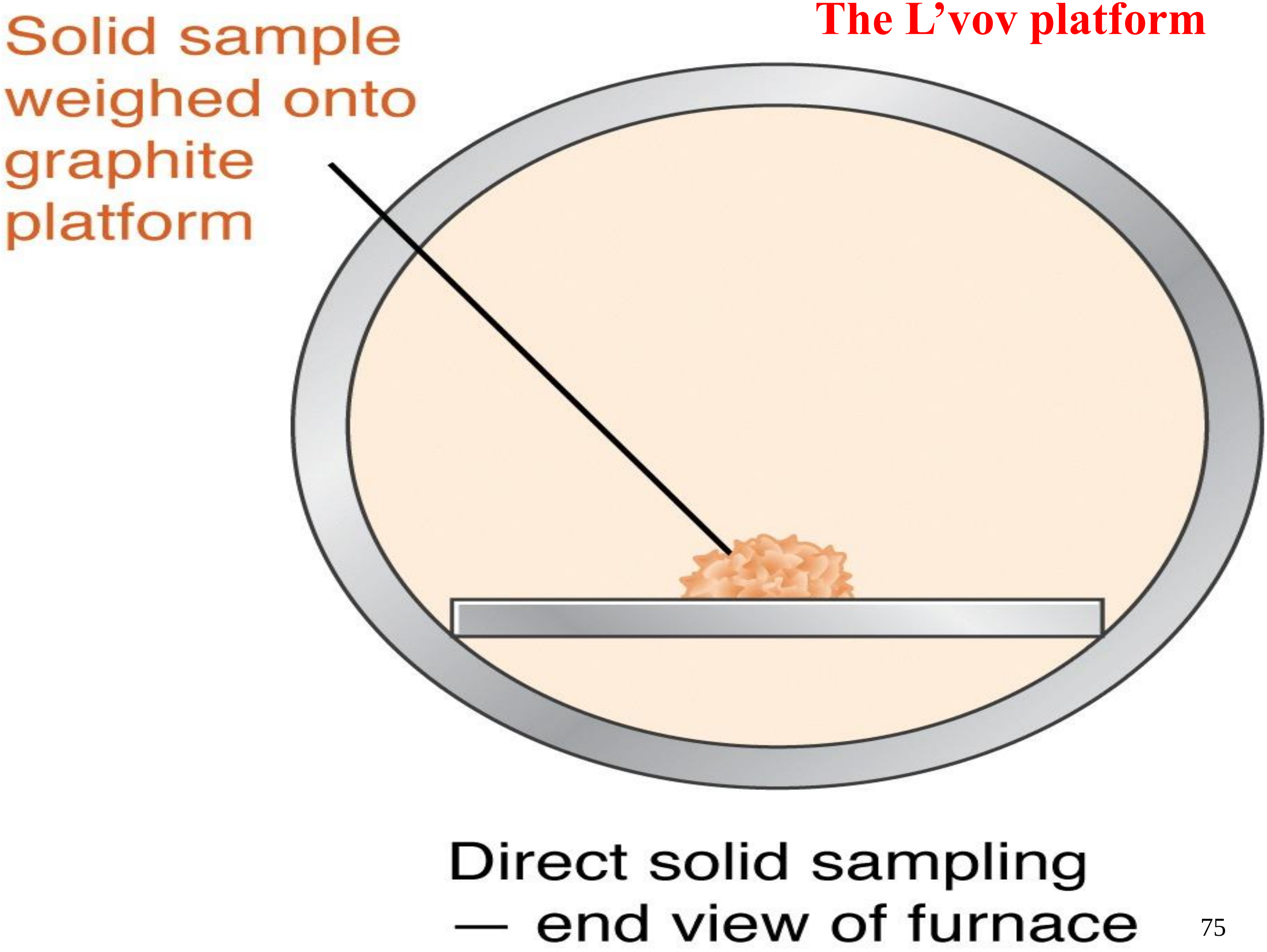


By:A.Sorkheh@yahoo.com

- **Small sample**
- **Formation of stable Thermal oxide (Al, Mo, Ti)**
- **Residual elements**
- **Biological samples**
- **Effective atomization**
- **Sample stay for Long time in graphite tube>>>high sensetivety**
- **Suitable for solid samples**
- **Ionic interferase rather than chemical or physical due to high temp.**

By:A.Sorkheh@yahoo.com

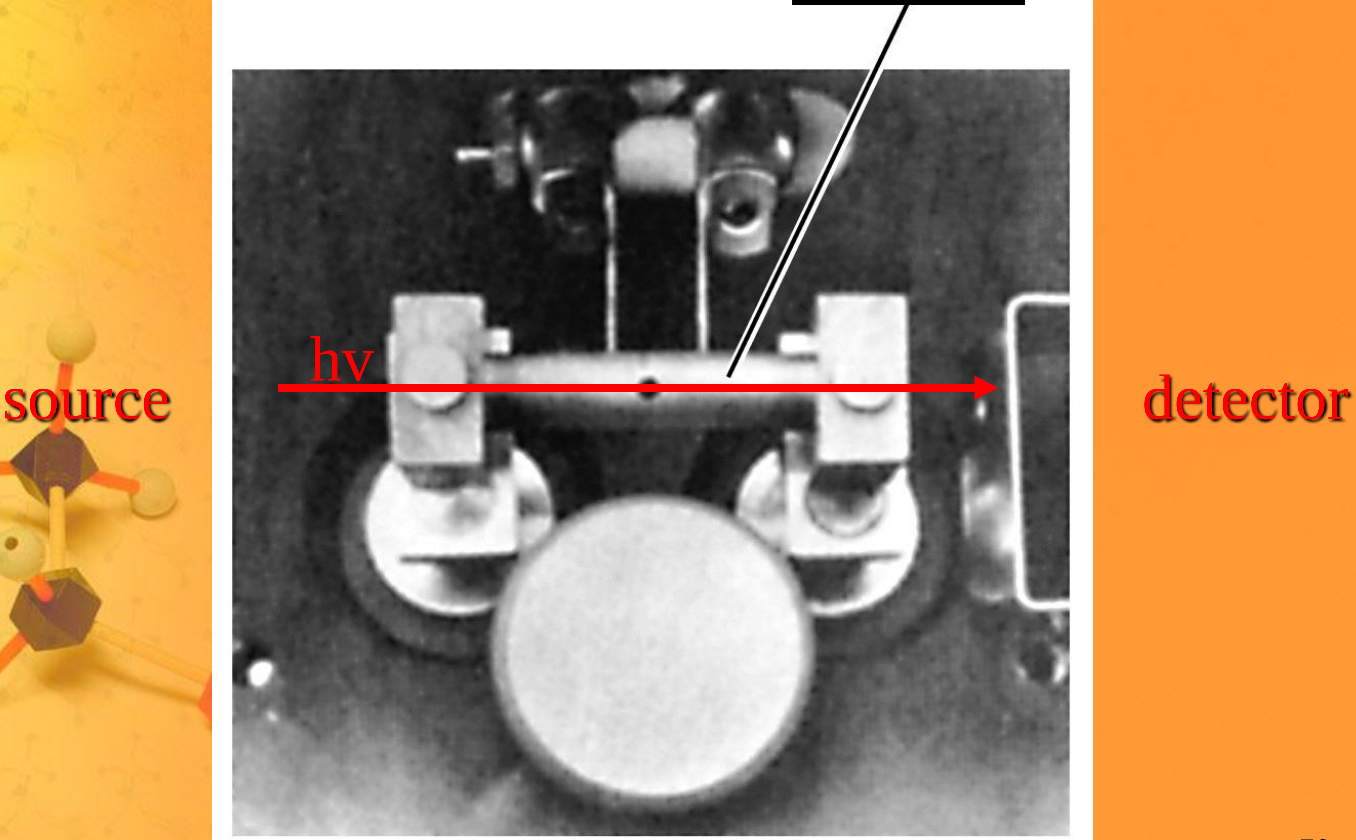
# The L'vov platform



By:A.Sorkheh@yahoo.com

# Graphite Furnace

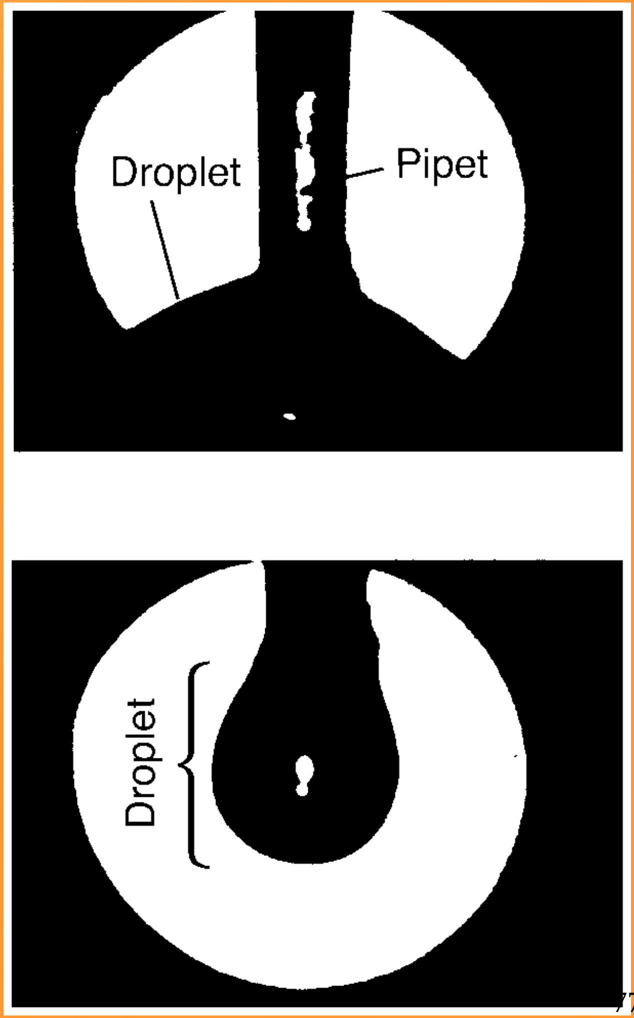
[redacted]



By:A.Sorkheh@yahoo.com

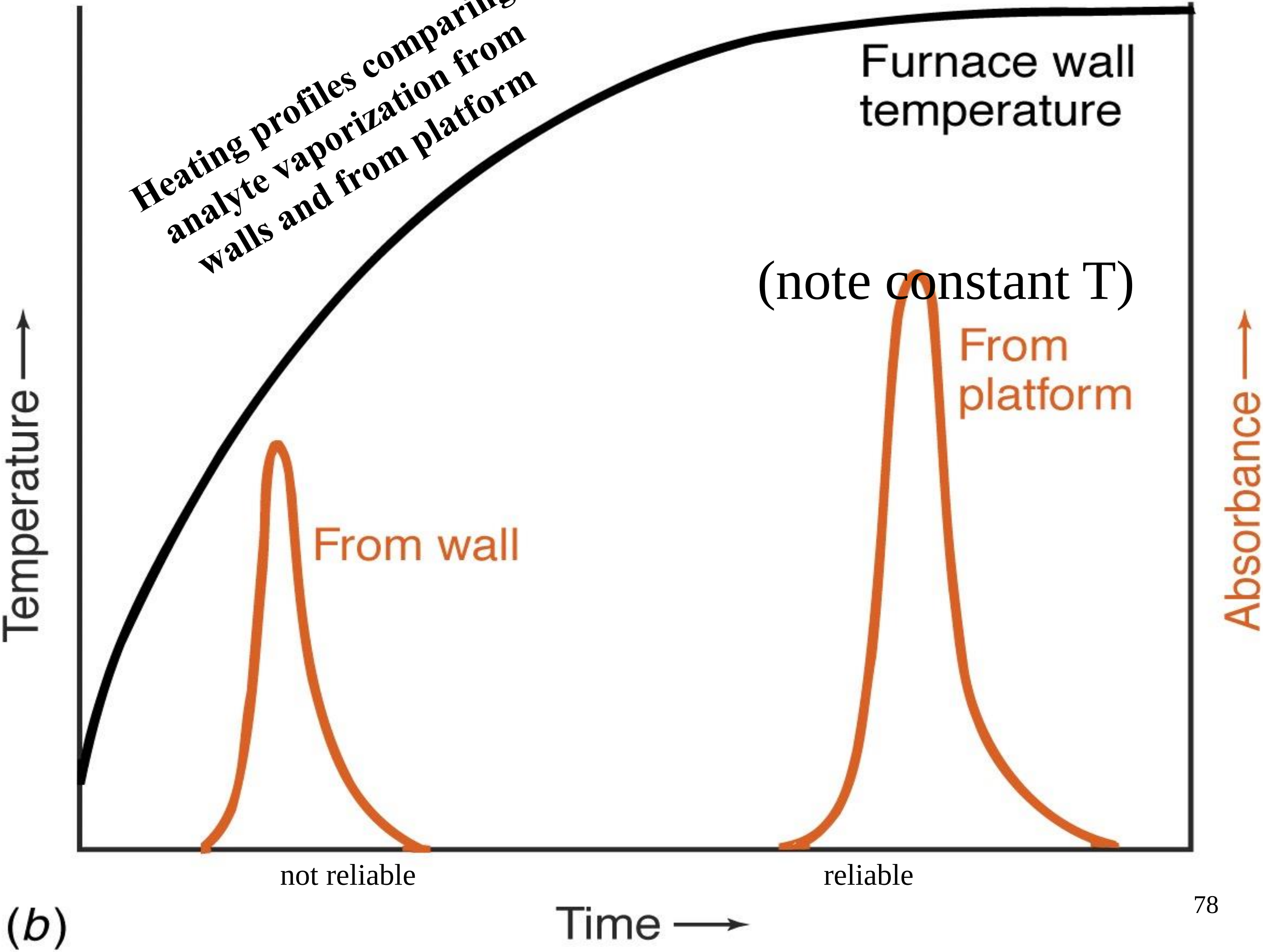
- ✓ Correct position for injecting sample into graphite furnace

- ✓ If injection is too high,sample splatters and precision is poor



By:A.Sorkheh@yahoo.com

**Heating profiles comparing analyte vaporization from walls and from platform**



(note constant T)

not reliable

reliable

# The Graphit furnace temperature program

1.Drying

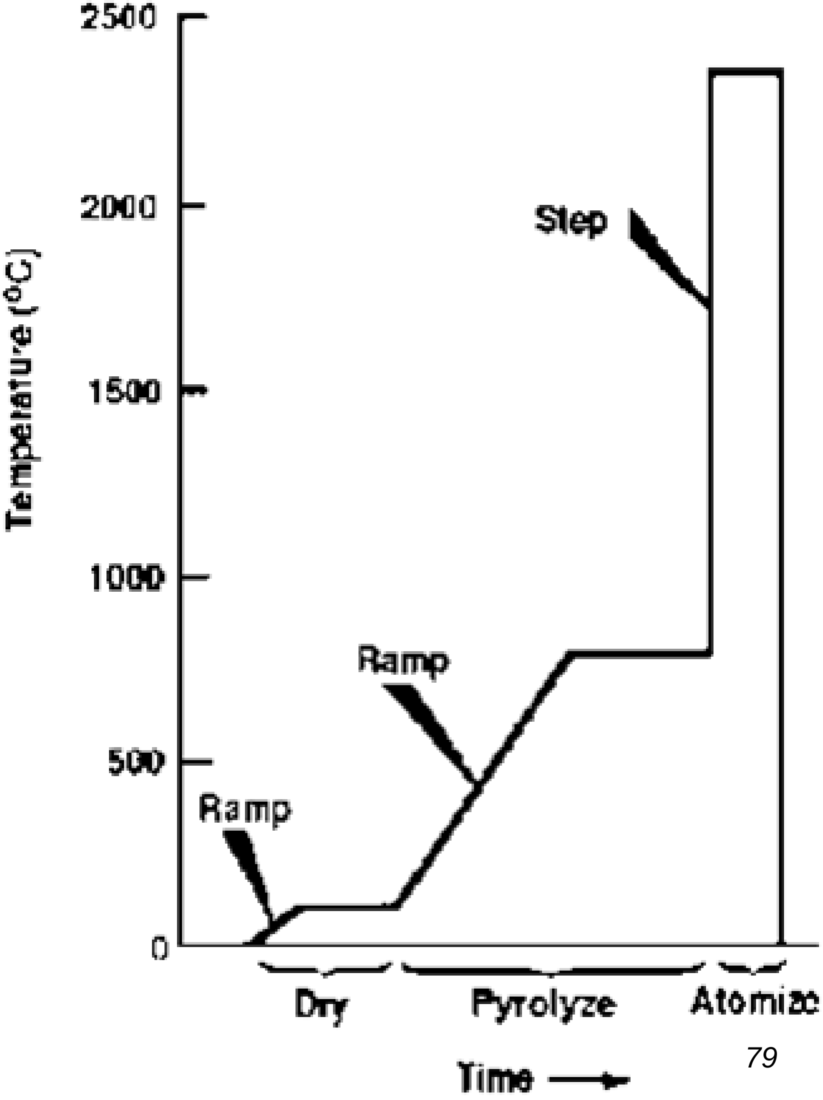
2.Pyrolysis

3.Cool own(Optional)

4.Atomization

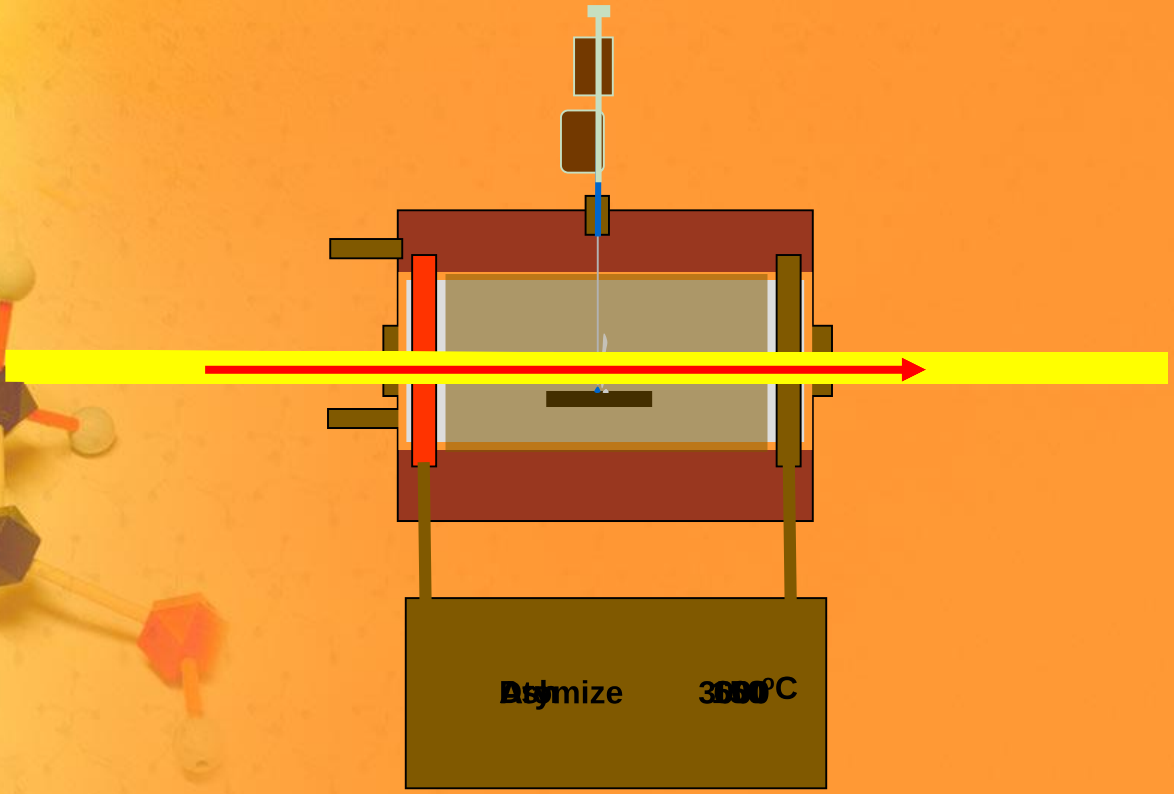
5.Clean Out

6.Cool Down

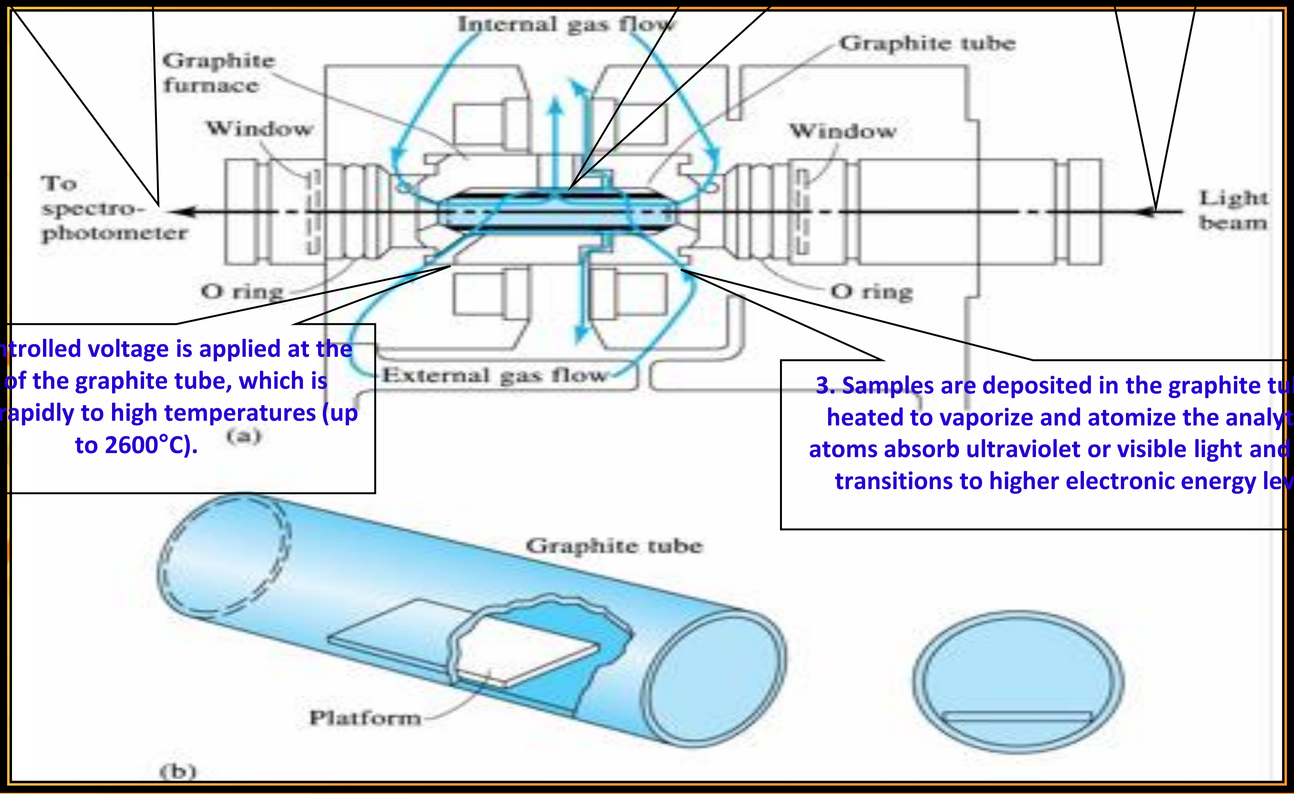


By:A.Sorkheh@yahoo.com

# Graphite Furnace



By:A.Sorkheh@yahoo.com



1. The source of light (lamp) emits light with a wavelength specific to the element of interest

2. A controlled voltage is applied at the ends of the graphite tube, which is heated rapidly to high temperatures (up to 2600°C).

3. Samples are deposited in the graphite tube → heated to vaporize and atomize the analyte → atoms absorb ultraviolet or visible light and make transitions to higher electronic energy levels.

4. The graphite tube is permanently flushed with argon while it is in operation

5. The monochromator isolates the light of the element of interest from the background lights to the PMT. The PMT tube measures the change intensity.

# Chemical Modifiers for Specific Elements in GFAAS

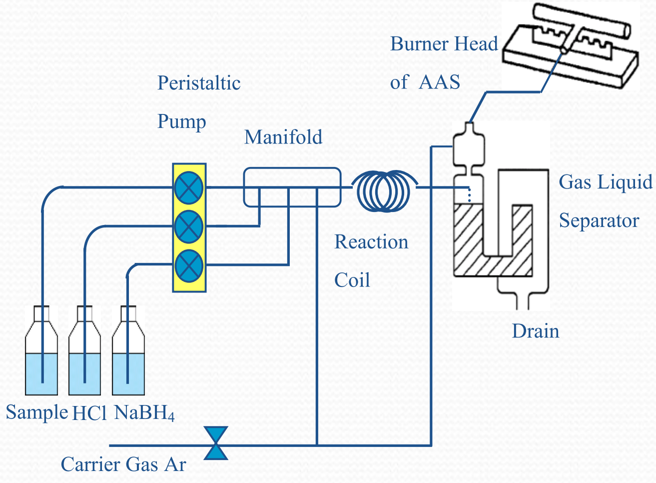
| Analyte | Modifier | Effect |
|---|---|---|
| As | Ni<br>Pd | Permits a higher ashing temperature and enhances the signal |
| Cd | $H_3PO_4$+ $Mg(NO_3)_2$<br>$NH_4H_2PO_4$<br>Pd | Conversion to less volatile phosphate which atomizes at a higher temperature |
| Pb | $H_3PO_4$+ $Mg(NO_3)_2$<br>$NH_4H_2PO_4$<br>EDTA<br>citrate<br>oxalate | Permits a higher ashing temperature and stabilizes the signal |

# Hydride Vapor Generation Atomic Absorption Spectroscopy (HGAASC)

- **As, Se, Sb, Sn, Te, Bi, Hg and other metals produce a metal hydride by this method**

| Elements | Concentration (ppb) |
|---|---|
| As | 5~20 |
| Sb | 5~20 |
| Te | 5~20 |
| Bi | 5~20 |
| Se | 10~40 |
| Hg | 20~80 |
| Sn | 30~90 |

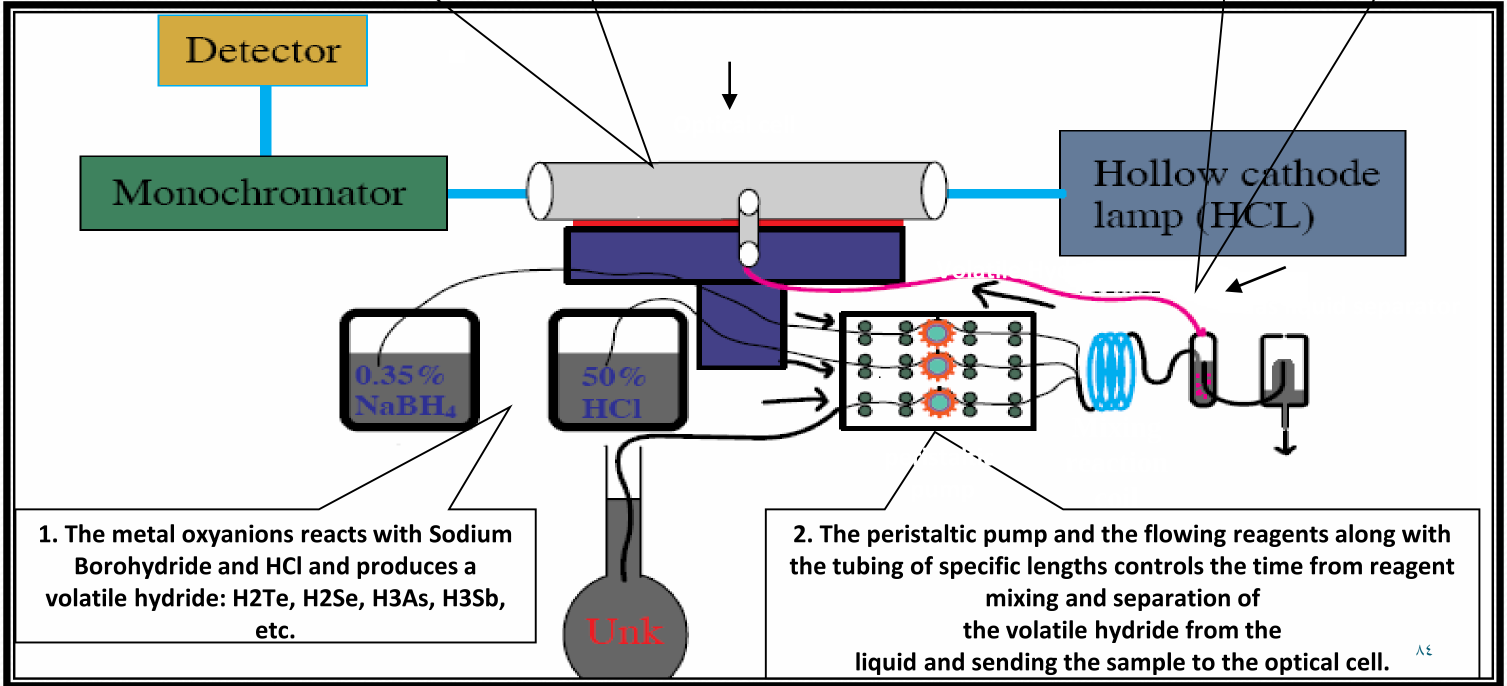
$$6BH_4^- + As^{3+} + 3H^+ \longrightarrow 3B_2H_6 + 3H_2 + AsH_3 \text{ (gas)}$$



**Structural Diagram of Hydride Vapor Generator**

**(HAAS) is very useful in case of interferences, poor reproducibility, and poor detection limits.**

# Sample flow in the (HGAAS)



4. In the optical cell the flame is ignited automatically by the air/$C_2H_4$ and the gaseous metal hydride form decomposes into the elemental form which can absorb the HCL's beam. The light passes to the Mon. and then to the PMT

3. The liquid mixture flows into a gas/liquid separator where the hydride and some gaseous hydrogen are purged (via a high purity inert gas) into the optical cell via a gas transfer line

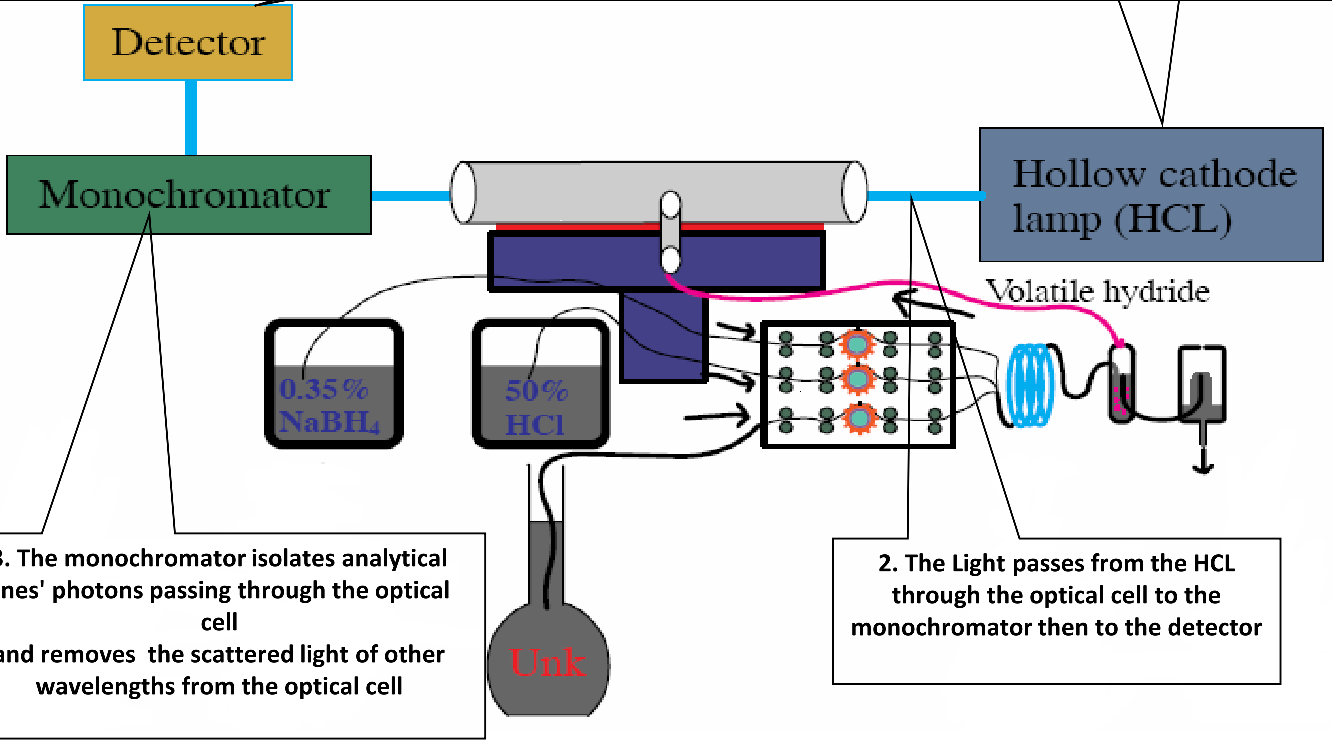
1. The metal oxyanions reacts with Sodium Borohydride and HCl and produces a volatile hydride: $H_2Te$, $H_2Se$, $H_3As$, $H_3Sb$, etc.

2. The peristaltic pump and the flowing reagents along with the tubing of specific lengths controls the time from reagent mixing and separation of the volatile hydride from the liquid and sending the sample to the optical cell.

# Mechanism of the (HGAAS)

4. The PMT detects the amount of reduction of the light intensity due to absorption by the analyte and can be directly related to the amount of the element in the sample

1. The HCL emits the light with a wavelength characteristic to the element of interest



3. The monochromator isolates analytical lines' photons passing through the optical cell
and removes the scattered light of other wavelengths from the optical cell

2. The Light passes from the HCL through the optical cell to the monochromator then to the detector

# COLD-VAPOR or Flameless AAS
# (Mercury Cold Vapor)

- **The Metal, *MERCURY*, has the unique ability to ABSORB light in the Vapor state at ROOM Temperature. Since it does not require any HEAT to generate an ABS signal, this method is called COLD-VAPOR or Flameless AAS**

- **Free mercury atoms can exist at room temperature can be analysed using atomic absorption without employing flame and graphite furnace techniques at high temperatures.**

By:A.Sorkheh@yahoo.com

# COLD-VAPOR Mechanism

a) **Mercury is reduced in solution using stannous chloride or sodium borohydride in a closed system.**

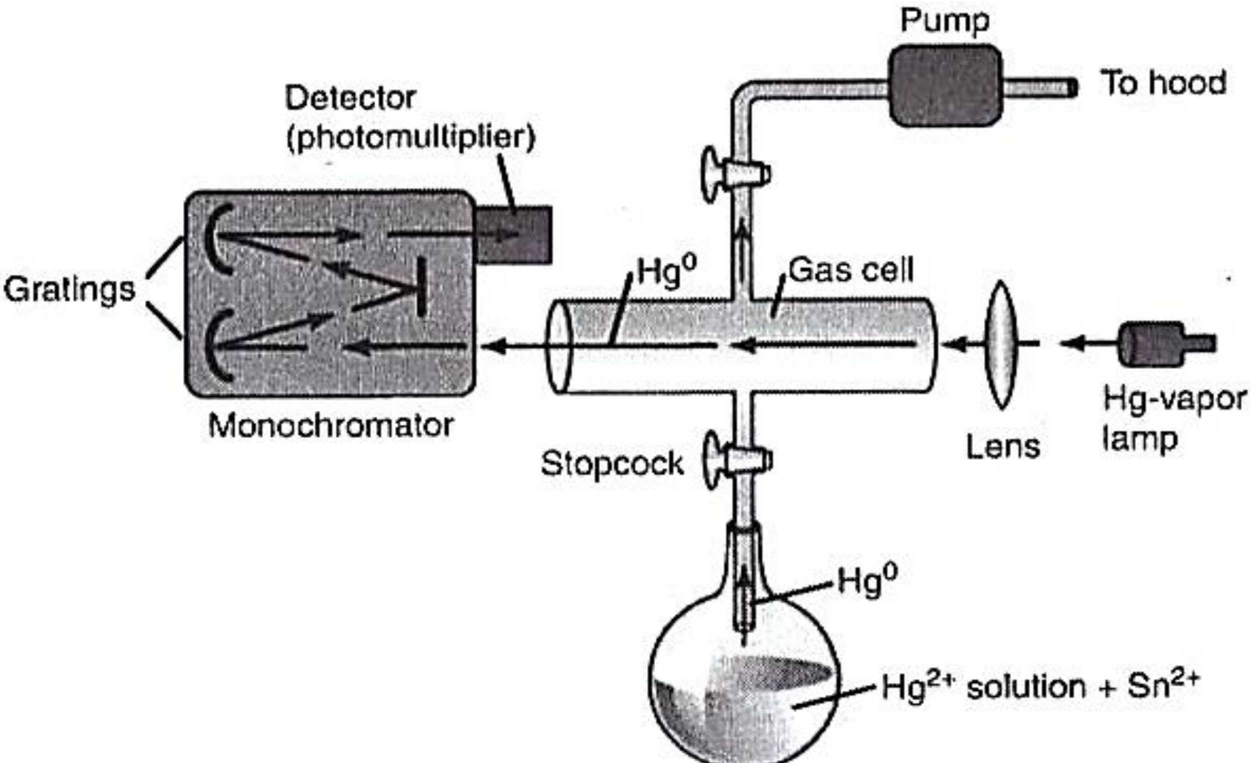
b) **The reaction quantitatively releases mercury (from the sample solution) and is carried by a stream of air or argon through a quartz sample sell placed in the light path of an AA instrument for analysis.**

✓ **The detection limit for mercury by this cold vapor technique is approximately 0.02 mg/L.**

By:A.Sorkheh@yahoo.com

# Cold vapor generation

$$Sn^{2+} + Hg^{2+} \rightarrow Sn^{4+} + Hg_{(L)}$$



# AAS Interferences

➢ Any factor that affects the ground state population of the analyte element.

➢ Factors that may affect the ability of the instrument to read this parameter.

## A. Spectral interferences(Back ground Absorption of Source Radiation ):

✓ Due to radiation overlapping than of the light source.

✓ Element other than the one of interest may absorb the wavelength being used

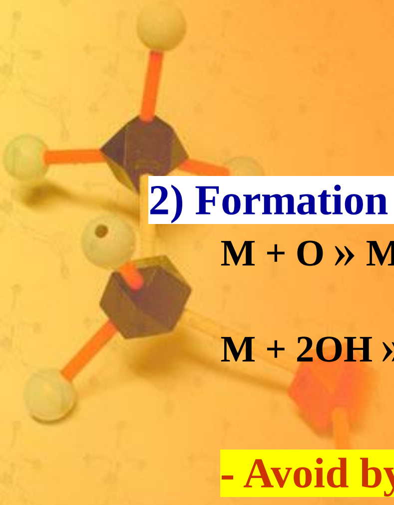
### Back ground Absorption of Source Radiation Reasons

➢ This is caused by the presence of a particle from incomplete atomization .

✓ This problem is overcome by increasing the flame temperature .

✓ Interferences are addressed by using a good quality HCL.

# B) Chemical Interference - more common than spectral interference



## 1) Formation of Compounds of Low Volatility

- Anions + Cations → Salt

$Ca^{2+} + SO_4^{2-} \rightarrow CaSO_4\ (s)$

- Decreases the amount of analyte atomized → decreases the absorbance signal

**- Avoid by:**

> increase temperature of flame (increase atom production)

> add **“releasing agents”** – other items that bind to interfering ions

eg. For $Ca^{2+}$ detection add $Sr^{2+}$

$Sr^{2+} + SO_4^{2-} \rightarrow SrSO_4\ (s)$

increases Ca atoms and Ca absorbance

> add **“protecting agents”** – bind to analyte but are volatile

eg. For $Ca^{2+}$ detection add $EDTA^{4-}$

$Ca^{2+} + EDTA^{4-} \rightarrow CaEDTA^{2-} \rightarrow$ Ca atoms

## 2) Formation of Oxides/Hydroxides

M + O » MO A *non-volatile & intense molecular absorbance*

$M + 2OH$ » $M(OH)_2$

- M is analyte

**- Avoid by:**

> increase temperature of flame (increase atom production)

> use less oxidant

By:A.Sorkheh@yahoo.com

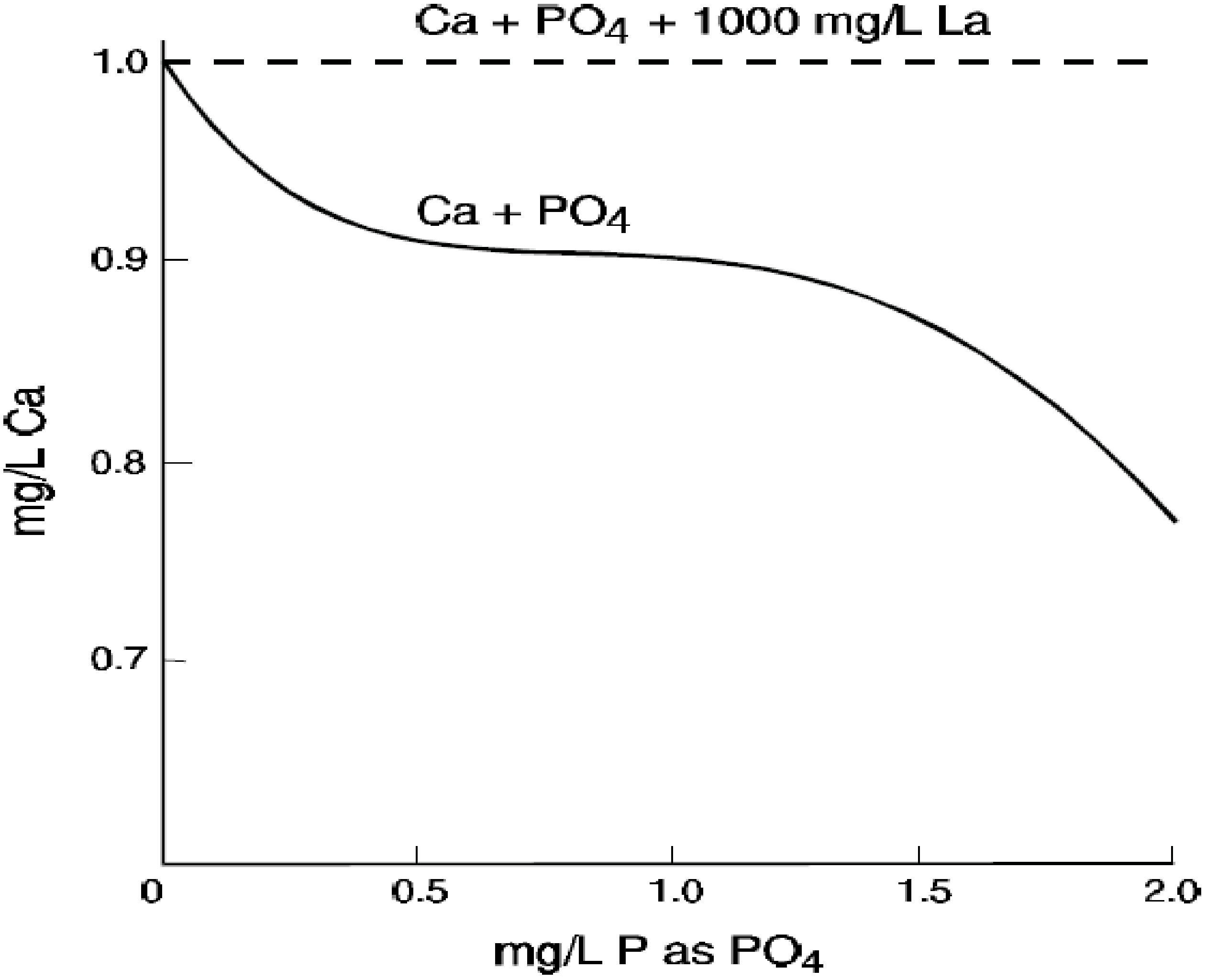


Figure 15. Interference of Phosphate on Calcium

**3) Ionization** Ionization of the analyte reduces the signal  M » $M^+ + e^-$

- M is analyte
- Avoid by:
  - > lower temperature
  - > add ionization suppressor – creates high concentration of $e^-$

suppresses $M^+$ by shifting equilibrium.

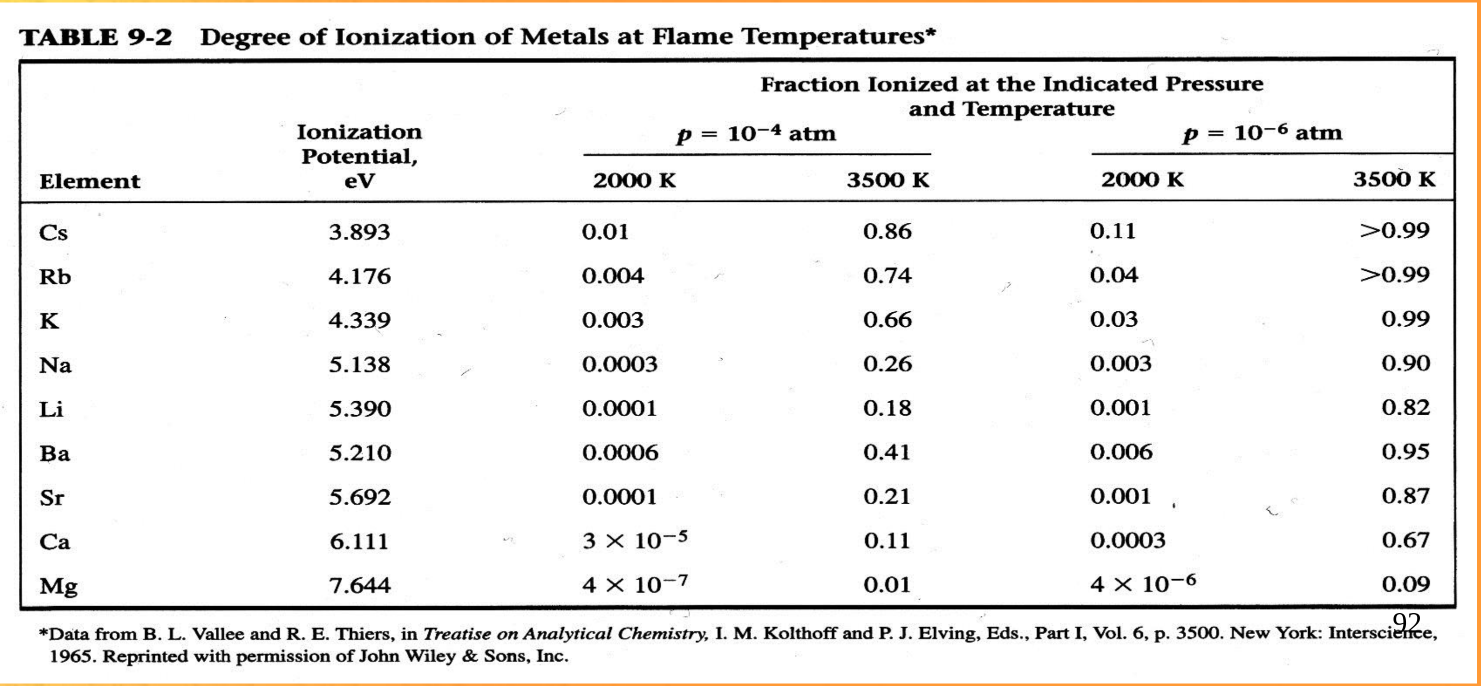


**TABLE 9-2** Degree of Ionization of Metals at Flame Temperatures*

| Element | Ionization Potential, eV | Fraction Ionized at the Indicated Pressure and Temperature | | | |
|---|---|---|---|---|---|
| | | $p = 10^{-4}$ atm | | $p = 10^{-6}$ atm | |
| | | 2000 K | 3500 K | 2000 K | 3500 K |
| Cs | 3.893 | 0.01 | 0.86 | 0.11 | >0.99 |
| Rb | 4.176 | 0.004 | 0.74 | 0.04 | >0.99 |
| K | 4.339 | 0.003 | 0.66 | 0.03 | 0.99 |
| Na | 5.138 | 0.0003 | 0.26 | 0.003 | 0.90 |
| Li | 5.390 | 0.0001 | 0.18 | 0.001 | 0.82 |
| Ba | 5.210 | 0.0006 | 0.41 | 0.006 | 0.95 |
| Sr | 5.692 | 0.0001 | 0.21 | 0.001 | 0.87 |
| Ca | 6.111 | $3 \times 10^{-5}$ | 0.11 | 0.0003 | 0.67 |
| Mg | 7.644 | $4 \times 10^{-7}$ | 0.01 | $4 \times 10^{-6}$ | 0.09 |

*Data from B. L. Vallee and R. E. Thiers, in *Treatise on Analytical Chemistry,* I. M. Kolthoff and P. J. Elving, Eds., Part I, Vol. 6, p. 3500. New York: Interscience, 1965. Reprinted with permission of John Wiley & Sons, Inc.

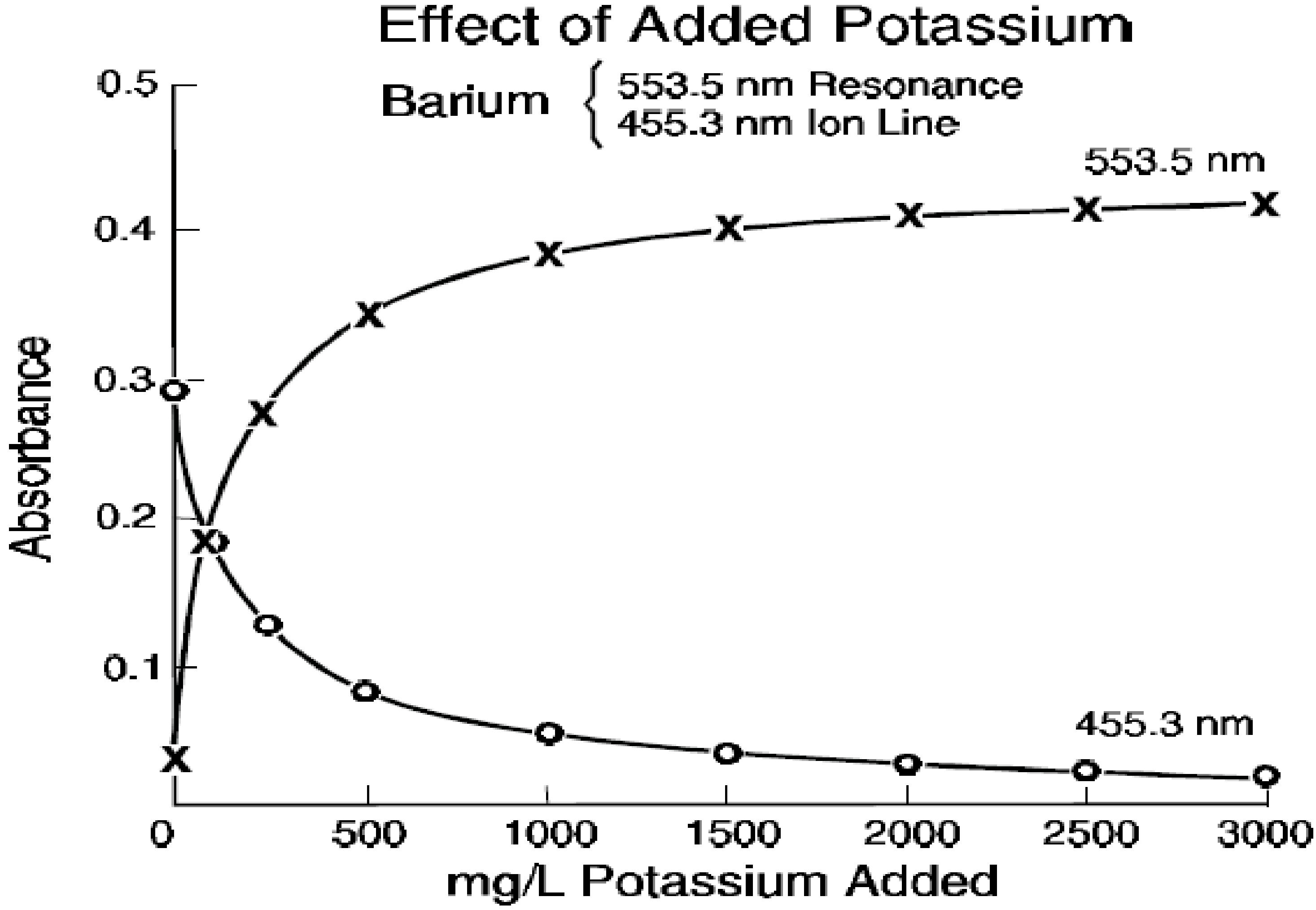


Figure 16. Ionization Interference Effect of Added Potassium

## C) Matrix interferences:

- Due to differences between surface tension and viscosity of test solutions and standards.

- ✓ Errors are minimized by using Matrix Modifiers and Reagents…

## D) Transport Interference :

- Rate of aspiration, nebulization, or transport of the sample ( e g viscosity, surface tension, vapor pressure , and density ).

By:A.Sorkheh@yahoo.com

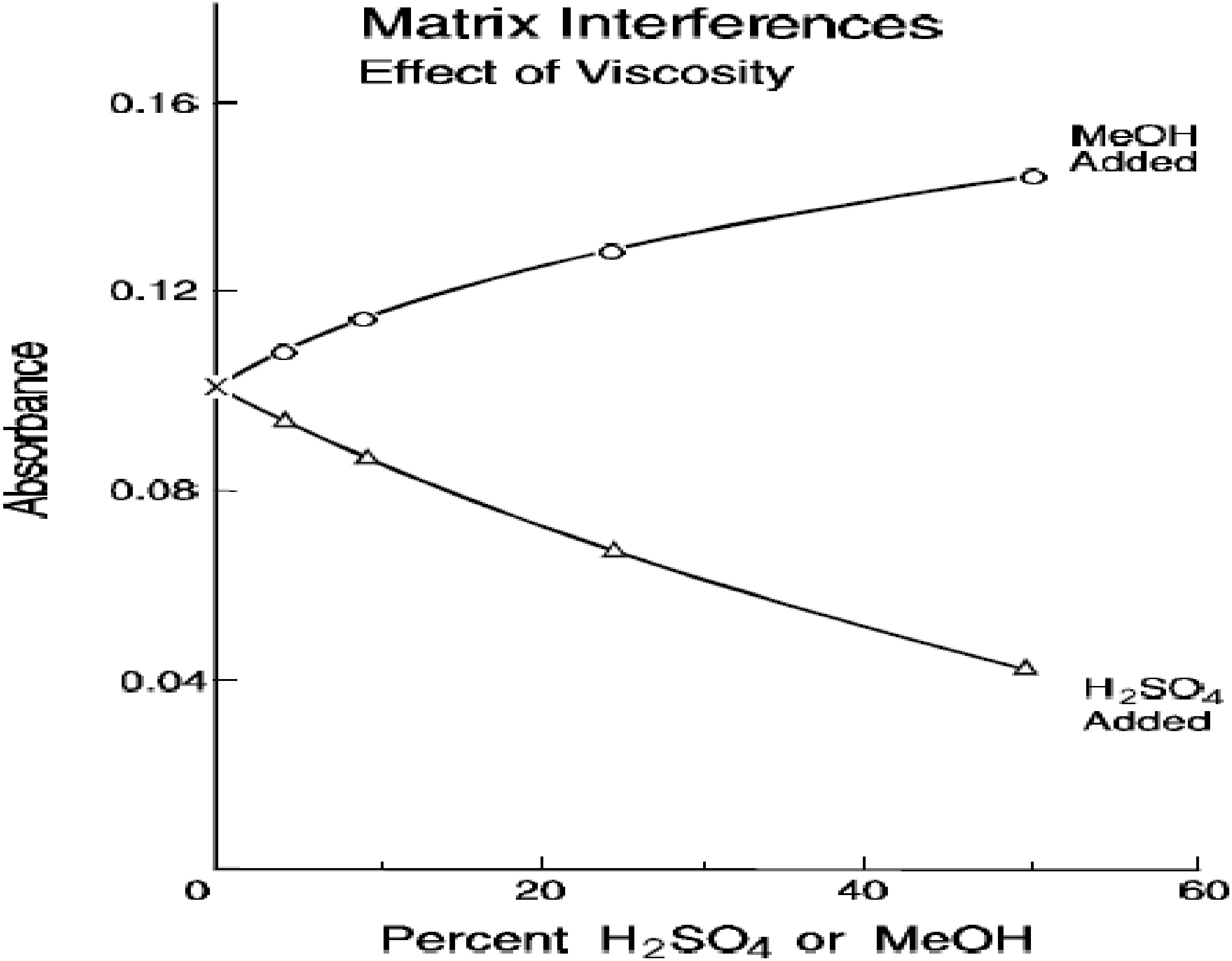


Figure 17. Matrix Interferences

# E) Broadening of a spectral line

## 1) Doppler effect:

- ✓ This effect arises because atoms will have different components of velocity along the line of observation.

## 2) Lorentz effect:

- ✓ This effect occurs as a result of the concentration of foreign atoms present in the environment of the emitting or absorbing atoms. The magnitude of the broadening varies with the pressure of the foreign gases and their physical properties.

## 3) Quenching effect :

- ✓ In a low-pressure spectral source, quenching collision can occur in flames as the result of the presence of foreign gas molecules with vibration levels very close to the excited state of the resonance line.

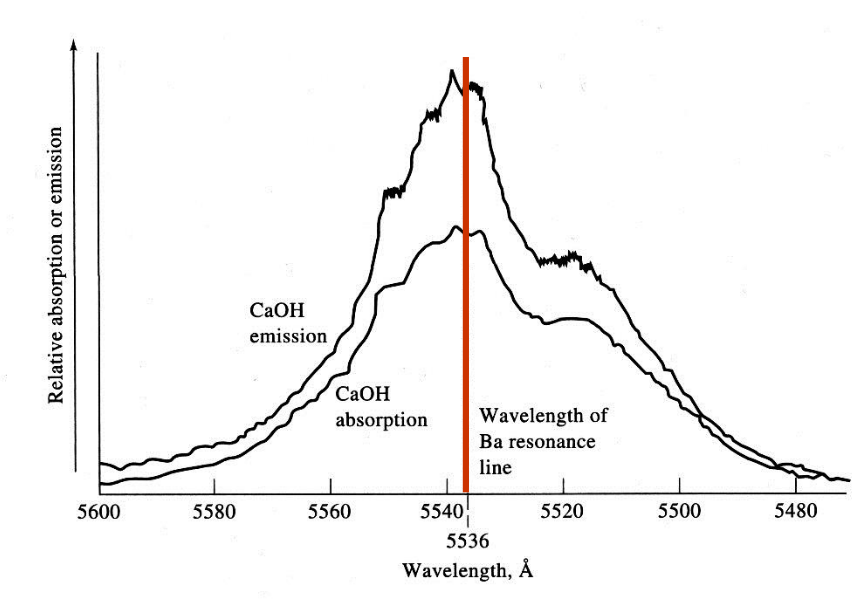
## 4) Self absorption or self-reversal effect

- ✓ Self absorptionthe atoms of the same kind that are absorbing radiation will absorb more at the center of the line than at the wings ,and thus resulting in the change of shape of the line as well as its intensity

# BACKGROUND Correction

## A)Corrections For Spectral Interferences Due to Matrix

- ✓ Molecular species may be present in flame
- ✓ Problem if absorbance spectra overlap since molecular spectrum is much broader with a greater net absorbance
- ✓ Need way of subtracting these factors out



# *Methods for Correction*

## 1) Two-line method

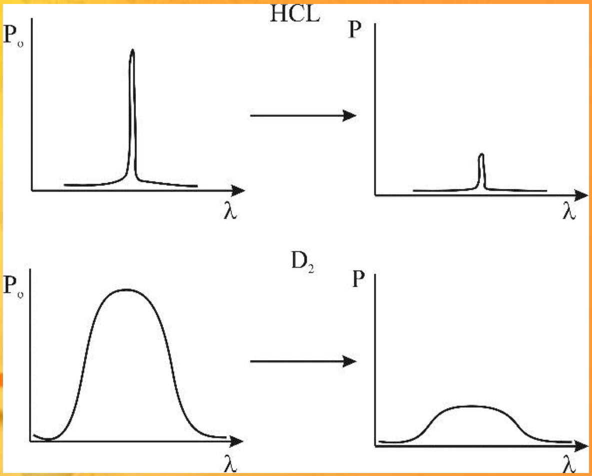
- monitor absorbance at two line close together
    > One line from sample one from light source
    > Second l from impurity in HCL cathode, Ne or Ar gas in HCL, etc
- second line must not be absorbed by analyte
    > absorbed by molecular species, since spectrum much broader
- A & e are ~ constant if two line close
- comparing $A_{l1}$, $A_{l2}$ allows correction for absorbance for molecular species

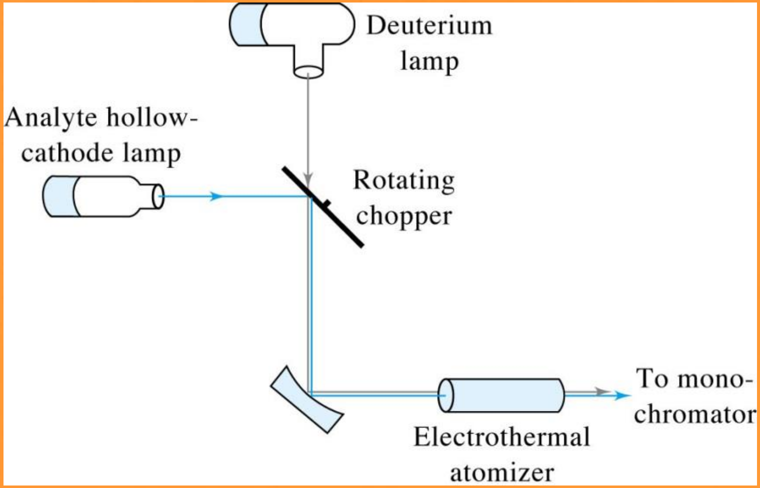
$$A_{l1}\ \text{(atom\&molecule)} - A_{l2}\ \text{(molecule)} = A\ \text{(atom)}$$

***Problem:*** **Difficult to get useful second l with desired characteristics**

# 2) Continuous source method

- alternatively place light from HCL or a continuous source $D_2$ lamp thru flame
- HCL → absorbance of atoms + molecules
- $D_2$ → absorbance of molecules





**Advantage:**

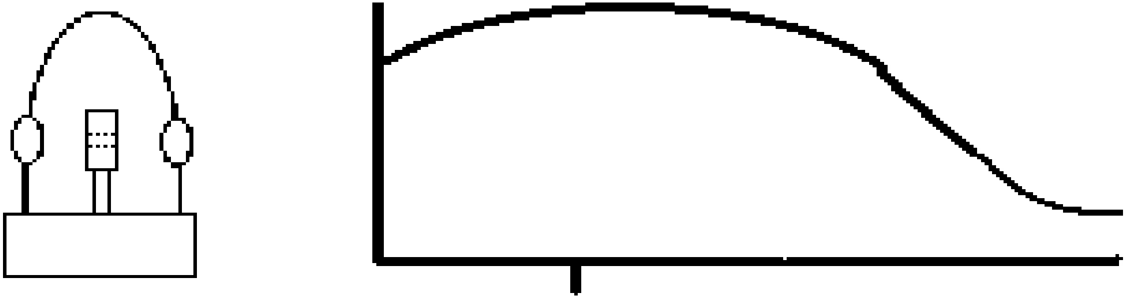
-available in most instruments

-easy to do

**Disadvantage:**

-difficult to perfectly match lamps (can give + or – errors)

*HCL generates a "narrow" band radiation, that "sees" BOTH Element-Specific AND Background Non-Specific Absorbances*

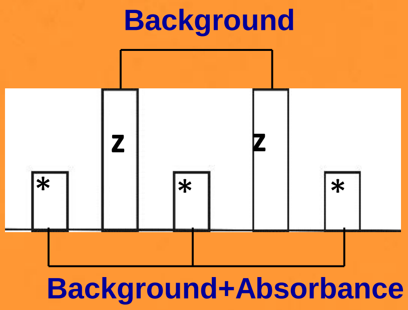


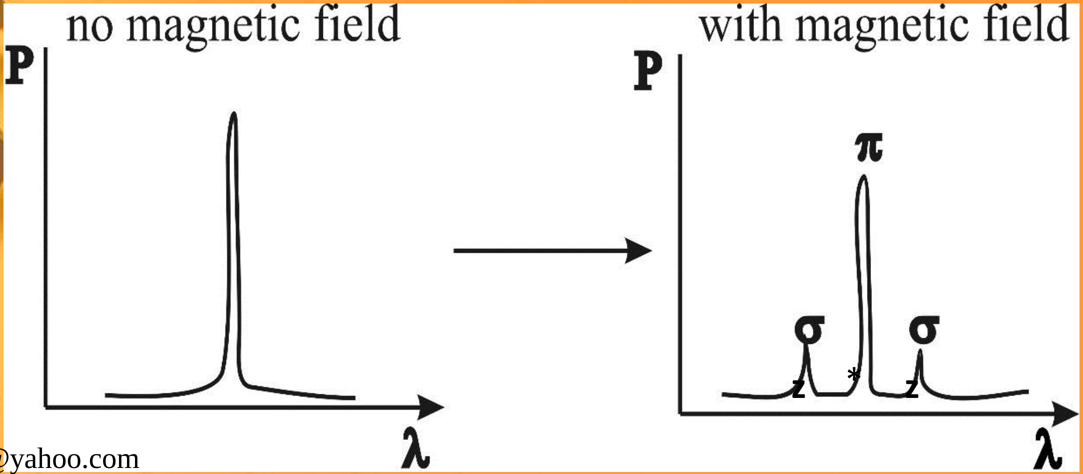
*D2 Lamp creates a "continuum" of "BROAD band radiation, that ONLY "sees" other BROAD band signals, such as BACKGROUND*

# 3) Zeeman Effect

- Placing gaseous atoms in magnetic field causes non-random orientation of atoms
- Not apparent for molecules
- Splitting of electronic energy levels occurs (~ 0.01 nm)
- Sum of split absorbance lines → original line
- Only absorb light with same orientation
- Can use Zeeman effect to remove background

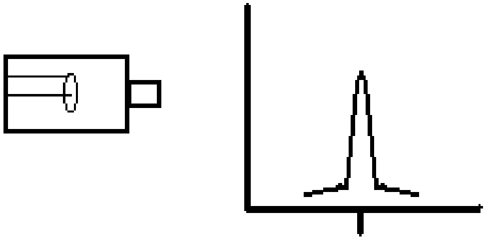
> place flame polarized light through sample in magnetic field get absorbance (atom+molecule) or absorbance (molecule) depending on how light is polarized





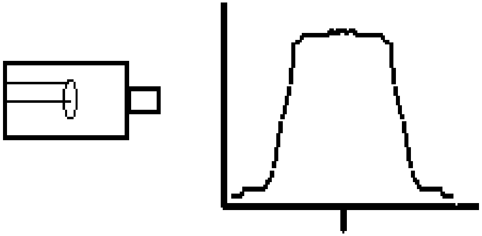
By:A.Sorkheh@yahoo.com

# BKG Correction: VGP



Pb-283nm.
@ 5mA

**Low CURRENTS for Analytical Measurements create a “narrow” band source of radiation**



Pb-283nm.
@500mA

***High CURRENTS (>500mA) are used to form the GIANT PULSE as a “broad”, wide band; to make it SIMILAR to the D2 Continuum for reading ONLY signals from the BACKGROUND***

**For any Analytical wavelengths outside the 180-400nm. Range for the D2 Lamp, Variable *GIANT PULSE* Correction will work for many applications**

# Sample preparation Methods

- Careful techniques must be used to Collect, Preserve and then PREPARE the Sample so it can be run on the AAS Instrument.
- Samples MUST be in the form of a clear, particle-free Solution
- These Solutions can be based on an Aqueous (water) or Organic (solvent) matrix
- Calibration materials (Blank and Standards) must be prepared with the SAME procedures as the Samples

By:A.Sorkheh@yahoo.com

# Sample PREPARATION

- **Some of the common procedures for AAS:**
  - ✓ **Open Beaker Acid Digestion**
  - ✓ **Closed Vessel Pressure Digestion**
  - ✓ **Soxhlet Solvent Extraction**
  - ✓ **Muffle-Furnace Ashing**
  - ✓ **Sonication**
  - ✓ **Centrifugation**
  - ✓ **Simple Filtration**

By:A.Sorkheh@yahoo.com

# Wet Digestion Methods

- The common methods used for dissolving samples for metals analysis are :
  - Digestion in open flask
  - Digestion in a pressurized sealed container
  - Microwave assisted decomposition

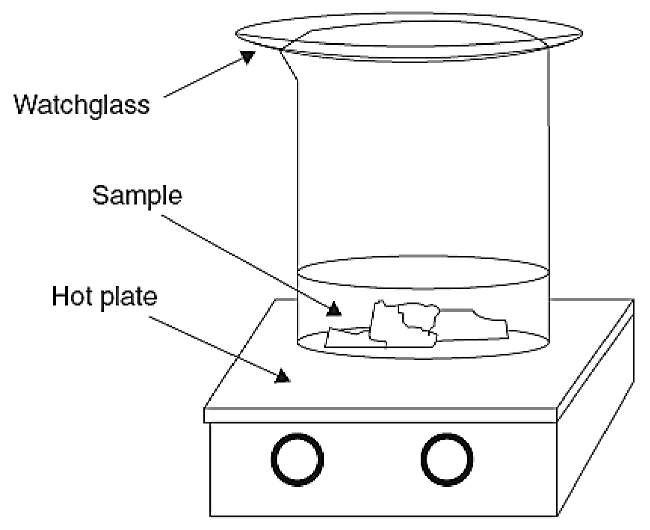
By:A.Sorkheh@yahoo.com

# Reagents Commonly used in Sample Dissolution or Digestion

| Reagent | Sample Type |
|---|---|
| Water | Soluble salt |
| Dilute acids | Dry-ashed sample residues, easily oxidized metals and alloys, salts |
| Concentrates acid (e.g., $HNO_3$) | Less readily oxidized metals and alloys, steels, metal oxides |
| Concentrates acid + oxidizing agent | Metals, alloys, soils, particulates from air, refractory minerals, vegetable matter |
| Hydrofluoric acid | Silicates and other rock samples |

# Acid Digestion-Wet Ashing

- The simplest method for wet digestion is carried out in an open container.
- Samples are dried, weighed, and placed in a beaker. The digestion reagent is added.
- The beaker is covered with a watch glass and placed on a hot plate.



By:A.Sorkheh@yahoo.com

# Aggressive acid digestion

Water

↓

Dilute nitric acid

↓

$HNO_3 + HCl$

↓

$HNO_3 + H_2SO_4$

↓

$HNO_3 + HClO_4$

↓

$HNO_3 + HClO_4$ then HF

↓

Aqua regia

By:A.Sorkheh@yahoo.com

- For samples that show significant losses of analyte due to the retention of metals in silica residues, the sample is first digested thoroughly with $HNO_3$ in a PTFE beaker. Then concentrated $HClO_4$ and a small amount of HF are added.

- A mixture of an oxidizing acid with HF provides acidity, oxidizing power, and complexation to dissolve all metals and alloys and most refractory minerals, soils, rocks, and sediments.

By:A.Sorkheh@yahoo.com

# Microwave Digestion

- **Advantages of digestion in a closed container :**
  - The containers are fabricated of high-temp polymers, which are less likely to contain metal contaminants than are glass or ceramic beakers or crucibles.
  - The sealed container eliminates the chance of airborne dust contamination.
  - The sealed container reduce evaporation, so that less acid digestion solution is required.
  - The sealed container also eliminates losses of more volatile metal species, which can be a problem in open container.
  - The electronic controls on modern microwave digesters allow very reproducible digestion conditions.

By:A.Sorkheh@yahoo.com

# Dry Ashing

- For samples that contain much organic matter, which are analyzed for nonvolatile metals, dry ashing is a relatively simple method of removing the organic matter.
- A general procedure is to place the weighed sample into a platinum or glass crucible and heat it in a furnace to a white ash. The temperature should be kept at 400 to 450°C if any of the more volatile metals are being determined. Salts or sulfuric acid may be added, if needed, and a final ashing step can be done with HF if required. The residue is then dissolved in concentrated nitric acid and water, and diluted to volume. The final concentration of acid should be between 1 and 5%.

By:A.Sorkheh@yahoo.com

# Extraction, Separation, and concentration

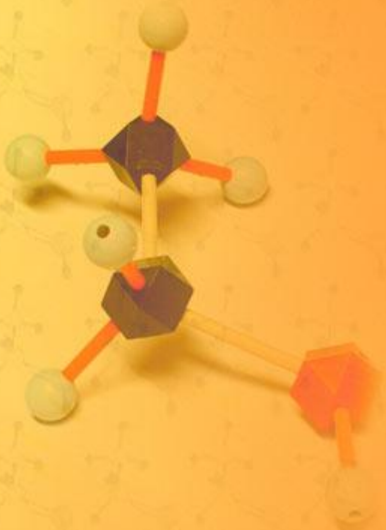
- It is not always necessary or required to digest the entire sample in order to free the metal for analysis. The analyte of interest may be present as a soluble salt from a pollution source or being present in the structure of the mineral crystals.

  - ✓ Organic extraction of metals (LLE)
  - ✓ Extraction with Supercritical Fluids
  - ✓ Ultrasonic sample preparation
  - ✓ Solid-Phase Extraction for preconcentration

By:A.Sorkheh@yahoo.com

# Calibration Equipment

- **STANDARD Solutions for Calibration of all Atomic Absorption instrumentation should be NIST/NBS or ISO / IUPAC Certified for true Accuracy**



# Selecting the Number of Standards

❖ **How can we select our calibration standards properly?**

1. **One Cal Standard: If all our samples are in the linear range (less than 0.20–0.30Absorbance), then one calibration standard (S1) can be used.**

2. **Two calibration standards: are used when we exceed the linear range.**

- **Usually S2 should be 3× the concentration of S1.**

By:A.Sorkheh@yahoo.com

# If we know we are exceeding the linear range, how can we decide whether to use two or three calibration standards?

## There is an easy procedure to follow:

1. **Make up three standards. The first one should be at the top of the linear range. The concentration of the second standard should be approximately 3× the concentration of the first. The concentration of the third standard should be approximately 6× the concentration of the first standard (6:3:1 rule).**

2. Set up your atomic absorption instrument in the concentration mode. Autozero on the blank. Set your first standard to be S1. Calibrate with your second standard to S2. READ your third standard. If its value is within 10% of the expected value, make your third standard = S2 and drop your second standard. You have slight curvature and should use only two calibration standards.

3. If your third standard deviates more than 10% from the expected value, your curvature is more severe and three calibration standards are necessary. Keep S2, and enter your third standard as S3. Seldom are more than three standards required for accurate calibration. However, when you are using more than three calibration standards, insert them between the standards that you determined using the 6:3:1 rule so that they are spaced along the calibration curve.

By:A.Sorkheh@yahoo.com

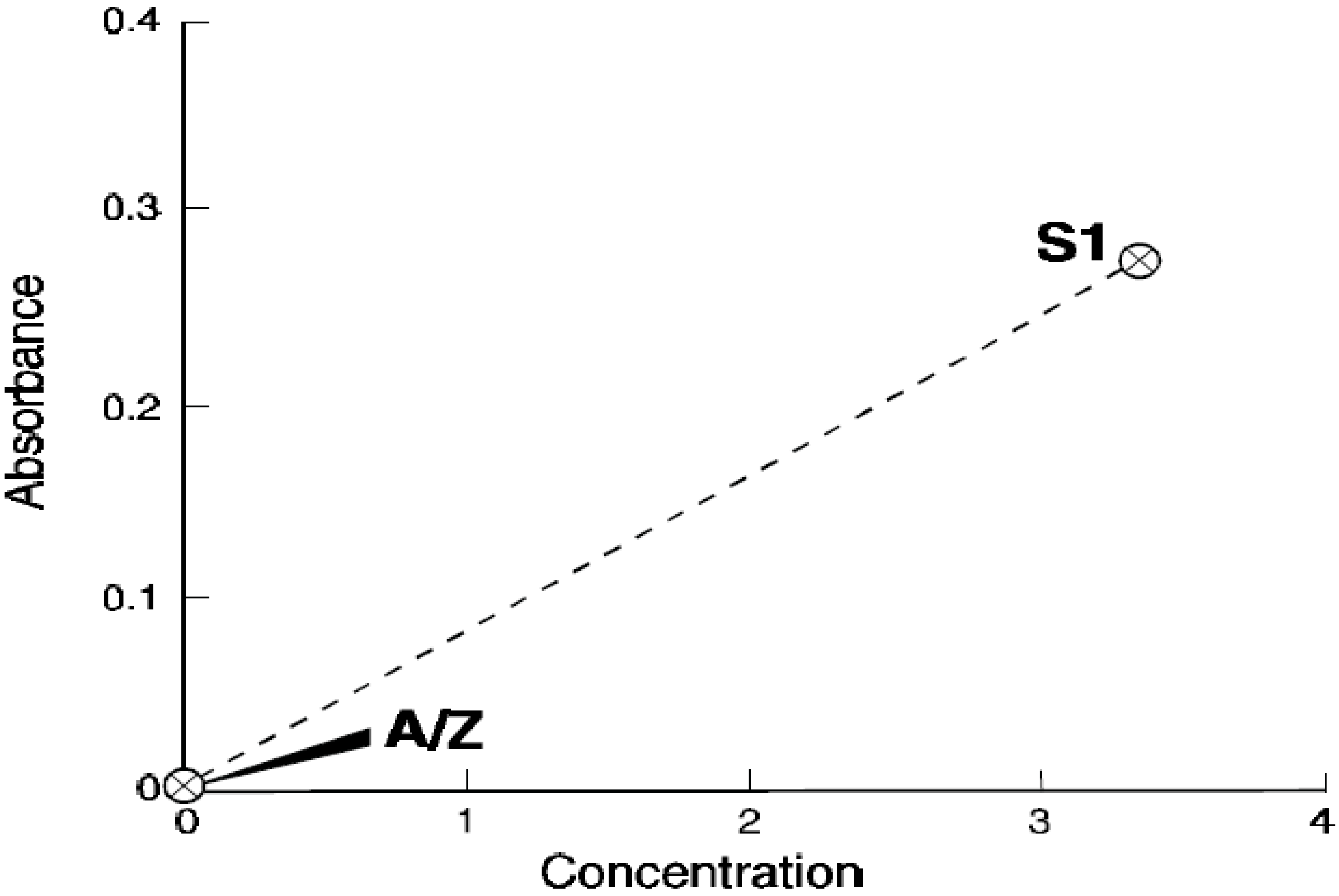


Figure 10. One Calibration Standard (Linear Plot)

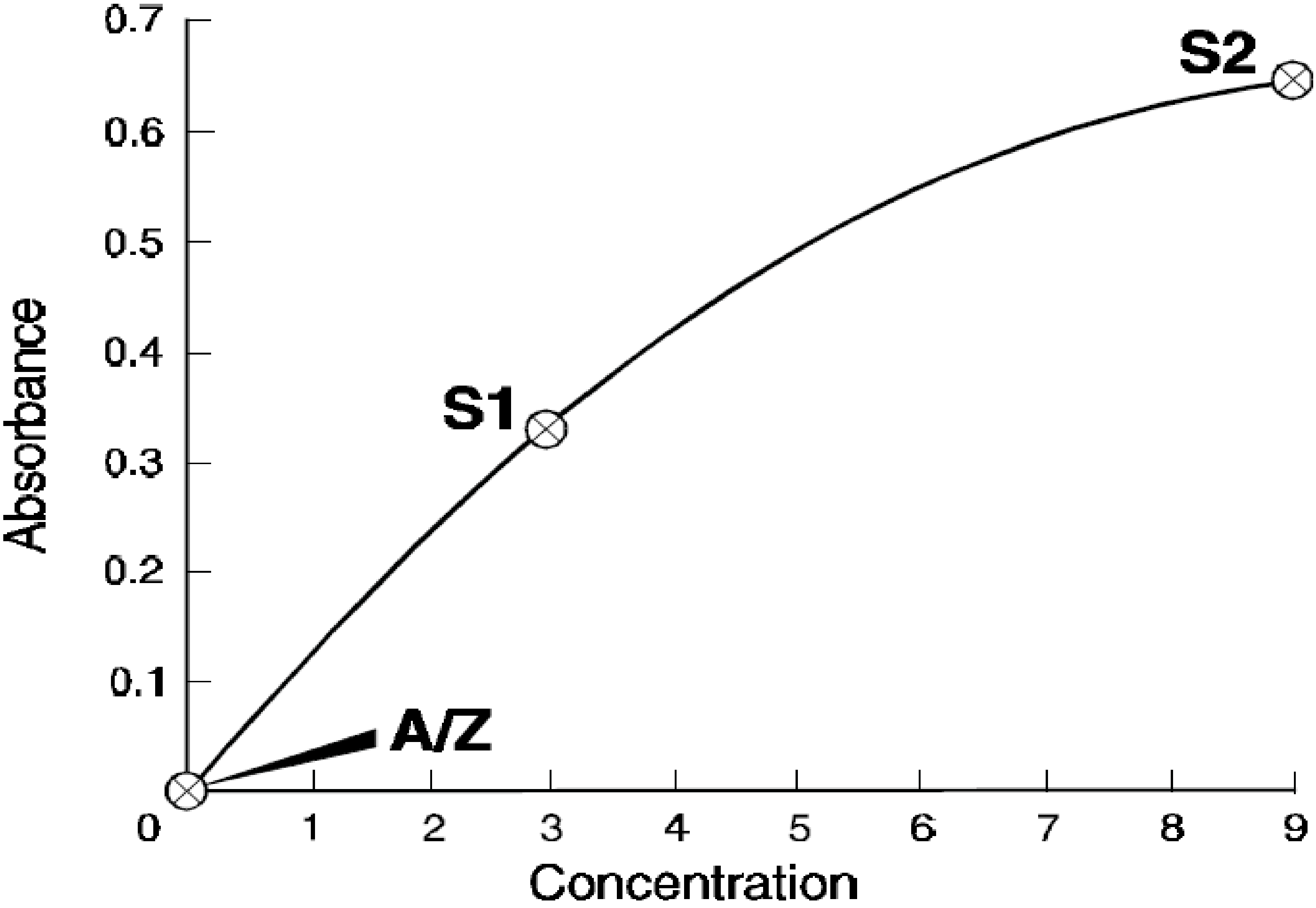
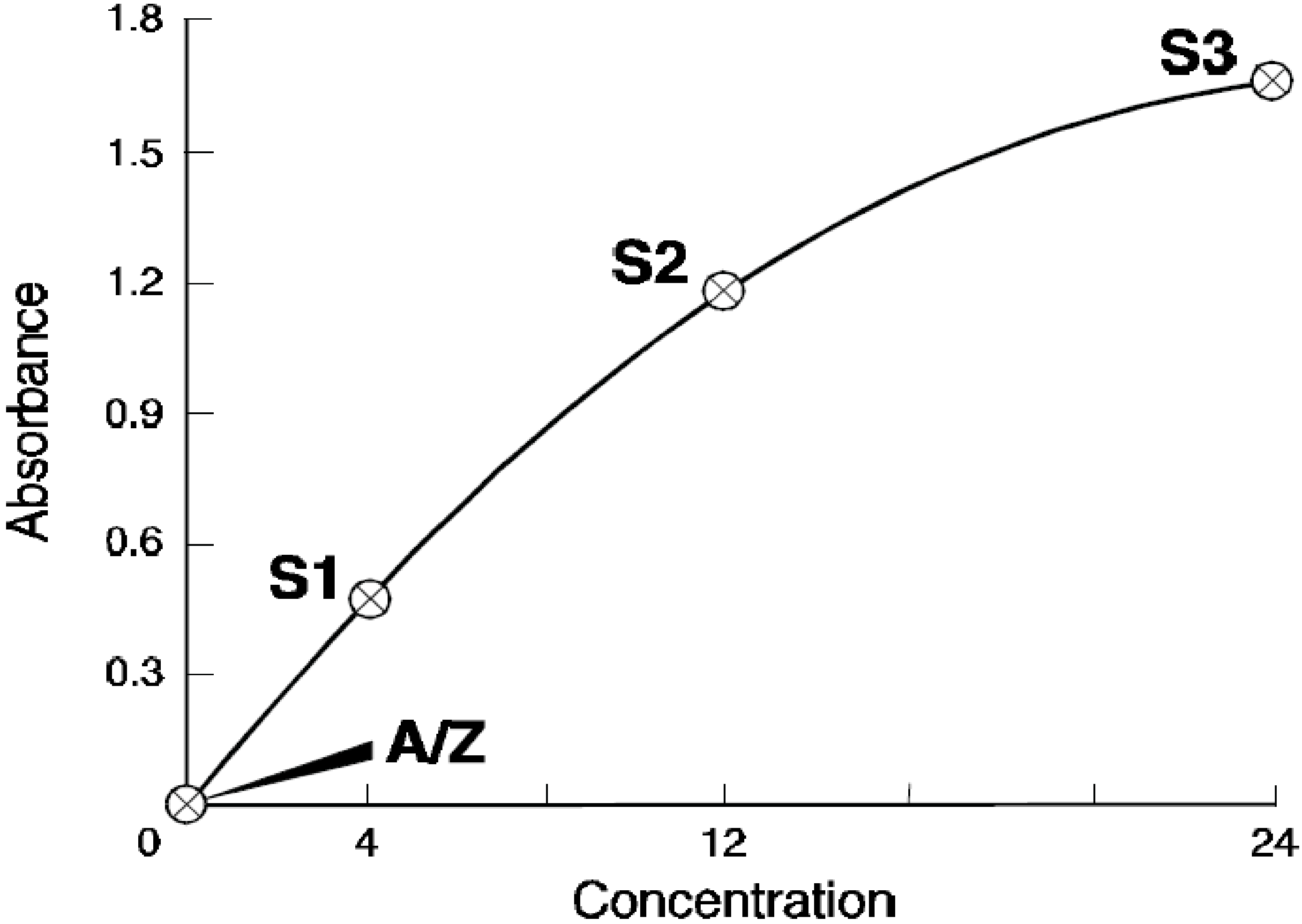


**Figure 11.** Calibration Curve for 2 Calibration Standards



By:A.Sorkheh@yahoo.com

**Figure 12.** Calibration Curve for 3 Calibration Standards

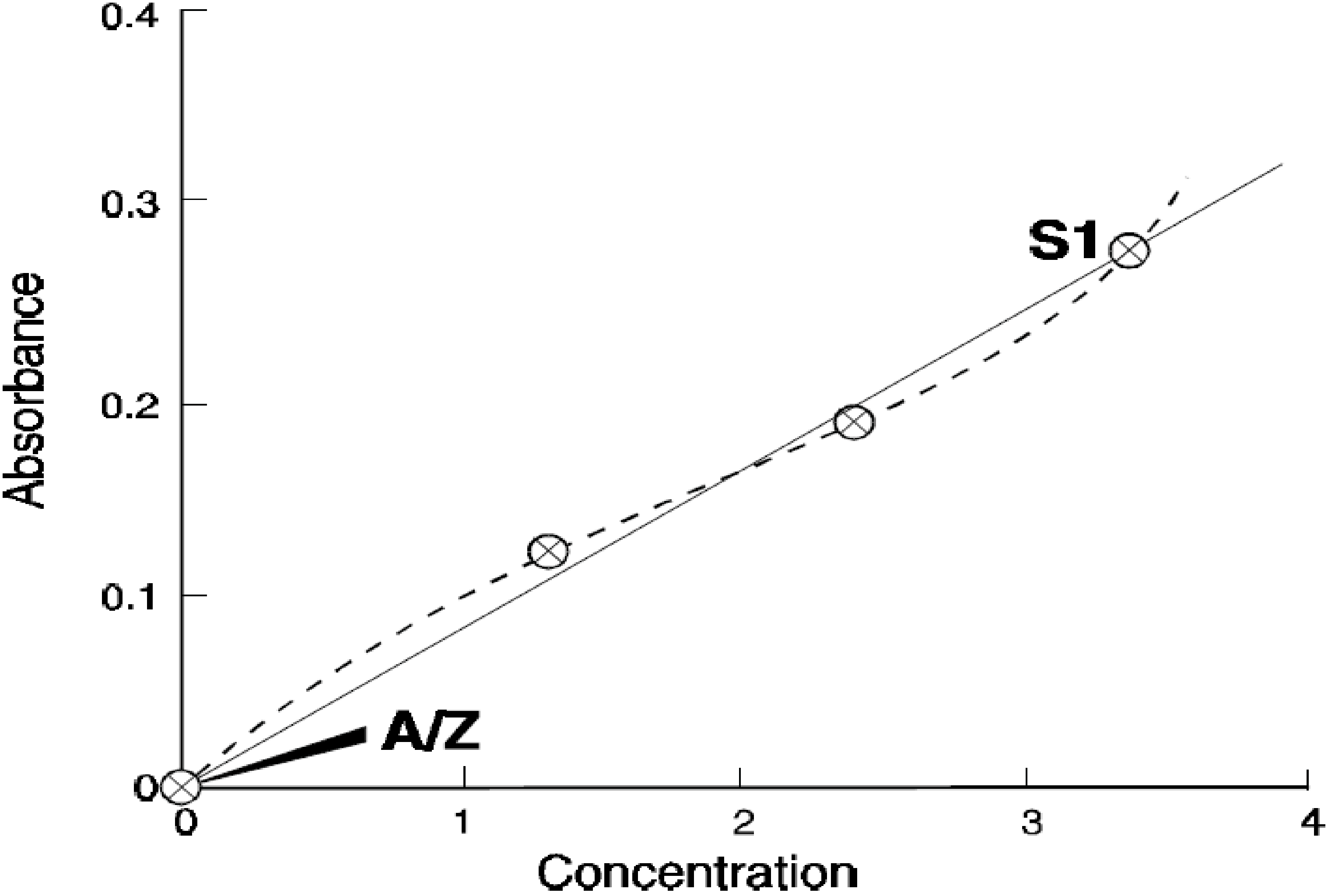
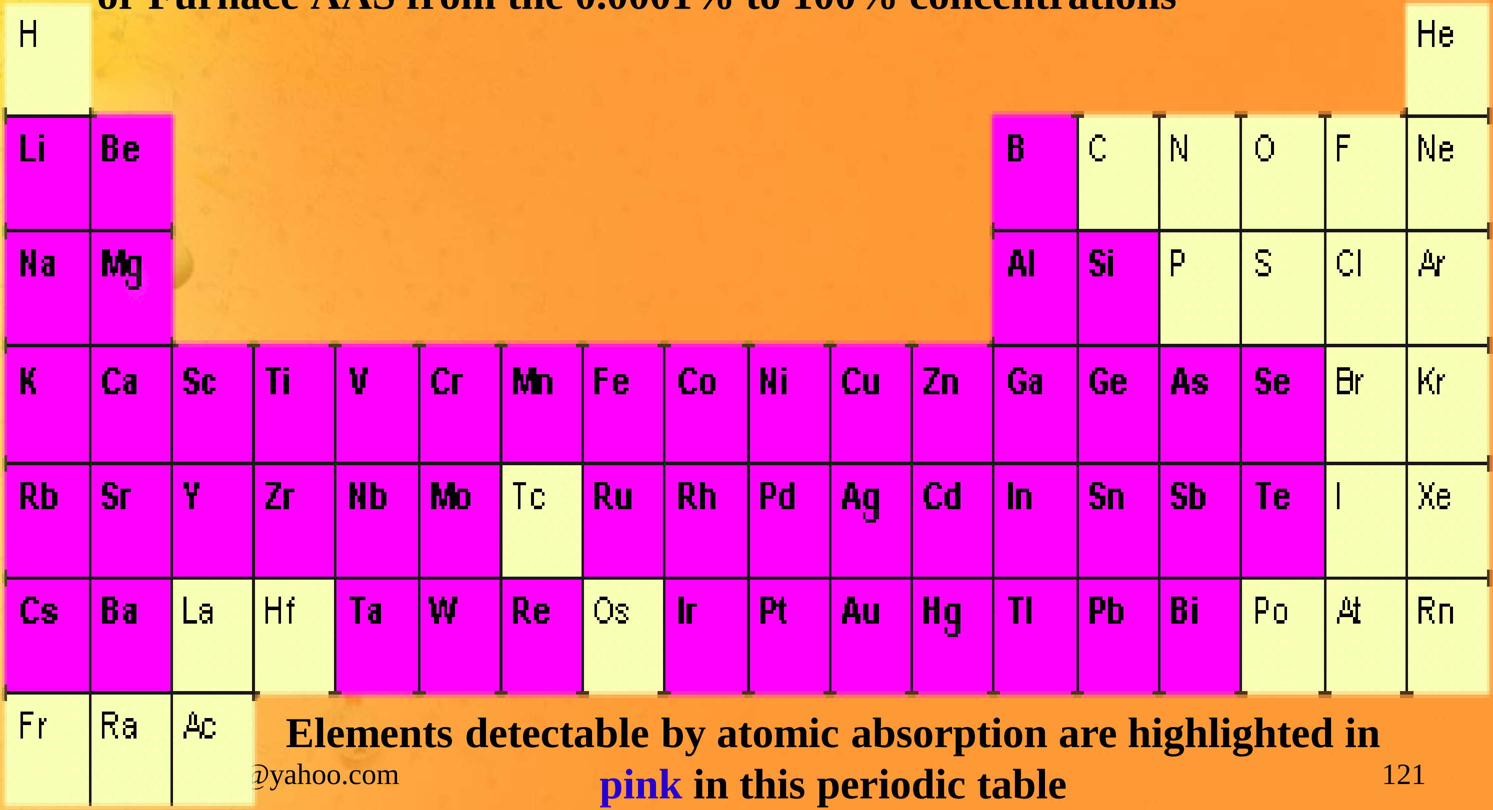


Figure 13. Three Calibration Points in Linear Range - S-Shaped Curve

# APPLICATIONS

**50+ Metallic Elements of the Periodic Table can be determined by Flame or Furnace AAS from the 0.0001% to 100% concentrations**



| | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| H | | | | | | | | | | | | | | | | | He |
| **Li** | **Be** | | | | | | | | | | | **B** | C | N | O | F | Ne |
| **Na** | **Mg** | | | | | | | | | | | **Al** | **Si** | P | S | Cl | Ar |
| **K** | **Ca** | **Sc** | **Ti** | **V** | **Cr** | **Mn** | **Fe** | **Co** | **Ni** | **Cu** | **Zn** | **Ga** | **Ge** | **As** | **Se** | Br | Kr |
| **Rb** | **Sr** | **Y** | **Zr** | **Nb** | **Mo** | Tc | **Ru** | **Rh** | **Pd** | **Ag** | **Cd** | **In** | **Sn** | **Sb** | **Te** | I | Xe |
| **Cs** | **Ba** | La | Hf | **Ta** | **W** | **Re** | Os | **Ir** | **Pt** | **Au** | **Hg** | **Tl** | **Pb** | **Bi** | Po | At | Rn |
| Fr | Ra | Ac | | | | | | | | | | | | | | | |

**Elements detectable by atomic absorption are highlighted in pink in this periodic table**

@yahoo.com

# LOD & LOQ

- **Limit of detection (LOD) and limit of quantification (LOQ) are two important performance characteristics in method validation. LOD and LOQ are terms used to describe the smallest concentration of an analyte that can be reliably measured by an analytical procedure.**

- **In analytical chemistry, the detection limit, lower limit of detection, or LOD (limit of detection), is the lowest quantity of a substance that can be distinguished from the absence of that substance (a *blank value*) within a stated confidence limit (generally 1%)**

## Sample Variance

S2 or SV

$$s^2 = \frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}$$

## Sample Standard Deviation

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}}$$

**How can I calculate LOD in Hg analysis by AAS (sample type: Fish)?**

**I need to calculate LOD in Hg analysis of fish sample. My method is;**

**Blank: put 10 mL nitric in microwave vessels and run the program and volume up to 50 mL and read (result received in microgram/L)**

**Sample: take 1 g sample+10 mL nitric acid in microwave vessels, then run the program,**

**My equation is for LOD=mean+ 3*standard deviation**

**But I receive blank reading in microgram/L unit (See attached excel sheet), then how I calculate LOD? Can you give the example or correct my excel sheet**

| Sample type : Fish | |
|---|---|
| Sample No | Blank reading (µg/L) |
| 1 | 2.24 |
| 2 | 0.00 |
| 3 | 0.92 |
| 4 | 1.58 |
| 5 | 0.67 |
| 6 | 0.55 |
| 7 | 0.35 |
| 8 | 0.76 |
| 9 | 0.19 |
| 10 | 0.00 |
| 11 | 0.73 |
| 12 | 0.00 |
| 13 | 0.36 |
| 14 | 0.39 |
| 15 | 0.14 |
| Mean | 0.59 |
| Standard deviation | 0.62 |
| LOD | 2.46 |

**LOD=mean+ 3*standard deviation**

**LOD=mean+ 3*standard deviation**

**OR**

$$LOD = \frac{SV}{R^\wedge 2} * 3$$

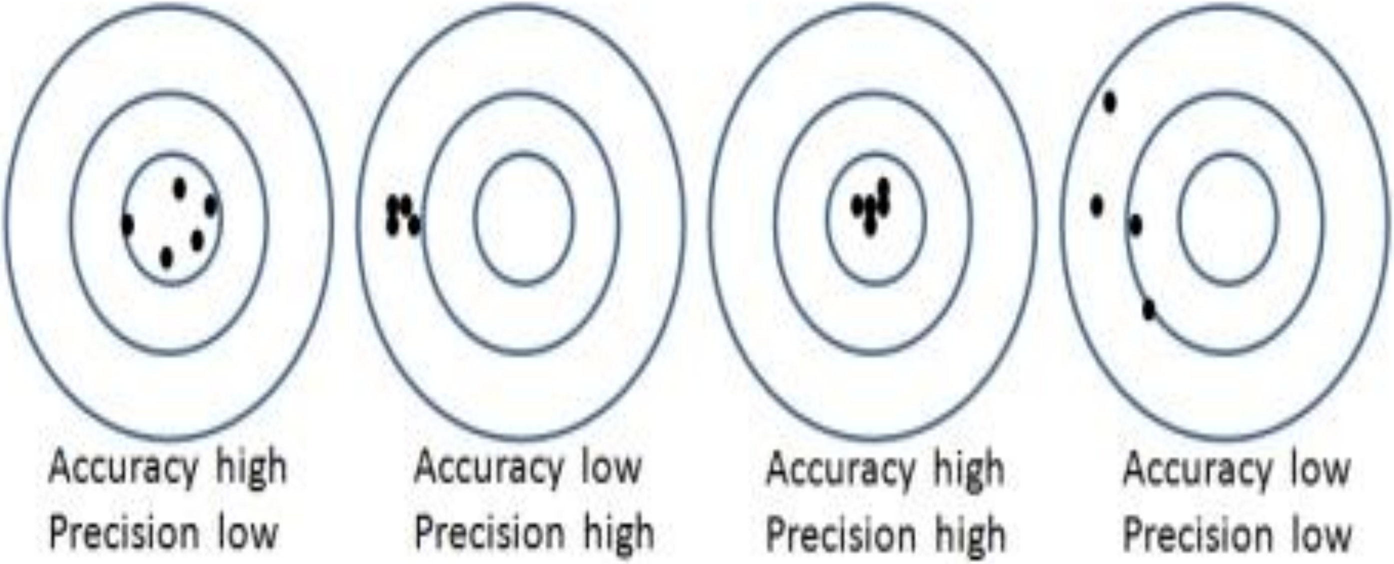
LOD is Theoryic

LOD is Practical

$$LOQ = \frac{SV}{R^\wedge 2} * 10$$

R^2 of calibration curve

# Accuracy & Precision



Accuracy high
Precision low

Accuracy low
Precision high

Accuracy high
Precision high

Accuracy low
Precision low

# Techniques for elemental analysis

| | ICP-MS | ICP-AES | FAAS | GFAAS |
|---|---|---|---|---|
| Detection Limits | Excellent | Good | Good | Excellent |
| Productivity | Excellent | Very good | Good | Low |
| LDR | $10^5$ | $10^6/10^{10}$ HDD | $10^3$ | $10^2$ |
| Precision | 1-3 % | 0.3-2 % | 0.1-1 % | 1-5 % |
| Spectral interference | Few | Common | Almost none | Very few |
| Chemical interference | Moderate | Few | Many | Many |
| Ionization | Minimal | Minimal | Some | Minimal |
| Mass efffects | High on low | none | none | none |
| Isotopes | Yes | none | none | none |
| Dissolved solids | 0.1-0.4 % | up to 30 % | 0.5-3 % | up to 30 % |
| No. of elements | ~75 | ~73 | ~68 | ~50 |
| Sample usage | low | medium | high | very low |
| Semi-quantitative | yes | yes | no | no |
| Isotope analysis | yes | no | no | no |
| routine operation | Skill required | easy | easy | skill required |
| Method development | skill required | skill required | easy | skill required |
| Running costs | high | high | low | medium |
| Capital costs | very high | high | low | medium |

| Parameters | AAS | GFAAS | ICP – MS |
|---|---|---|---|
| Temperature | 2300°C – 2700°C | 3000°C | 6000°C |
| Radiation used | UV ,VISIBLE | UV, VISIBLE | ---- |
| Detection limit | Ppm | Ppb | Ppt |
| Elements applicable to | 68+ | 50+ | 82 |
| Sample throughput | 10-15 sec per element | 3-4 min per element | All elements <1 minute |
| Sample volume required | Large | Very small | Very small to medium |
| Isotopic analysis | No | No | Yes |
| typical consumable items and utilities required | acetylene/nitrous oxide gases (compressed air source)<br>hollow cathode lamps<br>reagents and standards<br>power | argon gas<br>hollow cathode lamps<br>graphite tubes and cones<br>reagents and standards<br>power<br>cooling water | argon gas<br>quartz torches<br>sampling and skimmer cones<br>reagents and standards<br>pump tubing<br>power<br>cooling water |

# Operating instructions for VARIAN 280FS Spectrophotometer

## Exhaust Vent

**Exhaust venting is important for a number of reasons:**

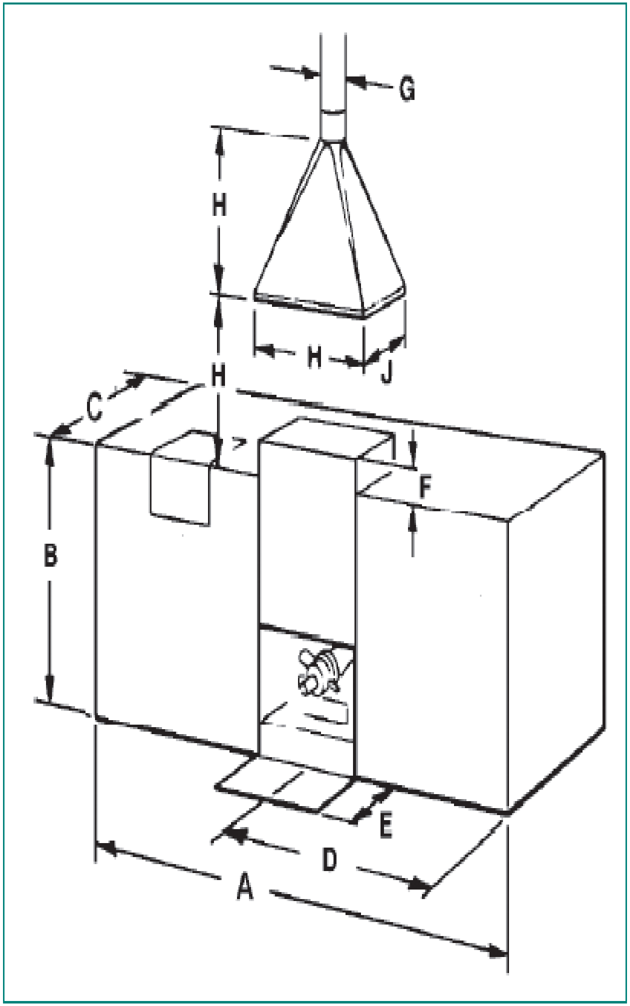
**1.It will protect laboratory personnel from toxic vapors which may be produced by some samples.**

**2.It will tend to remove the effects of room drafts and the laboratory atmosphere on flame stability.**

**3. It will help to protect the instrument from corrosive vapors which may originate from the samples.**

**4.It will remove dissipated heat which is produced by the flame or furnace.**

By:A.Sorkheh@yahoo.com



A = length

B = height to top of cover

C = depth

D = distance from center of atomizer compartment to right hand edge of instrument

E = protuberance of sample tray in front of instrument

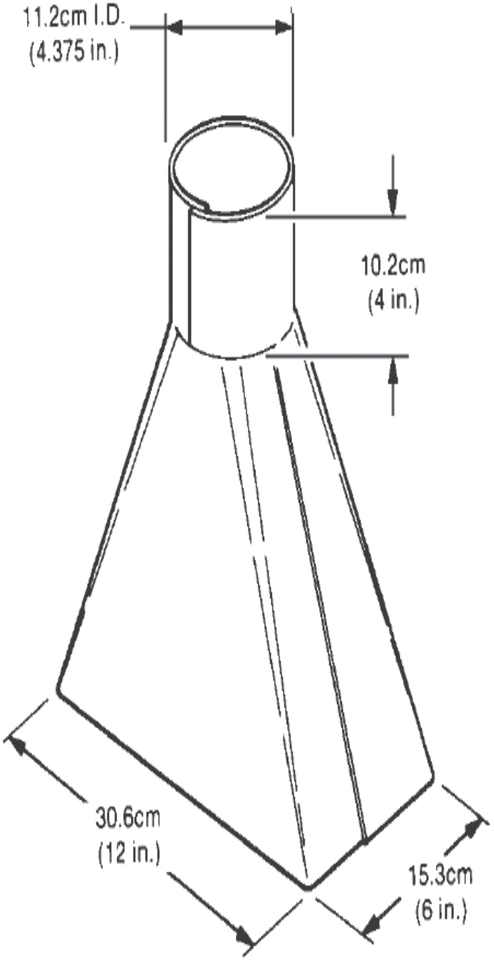
F = height of flame shield above top of instrument cover

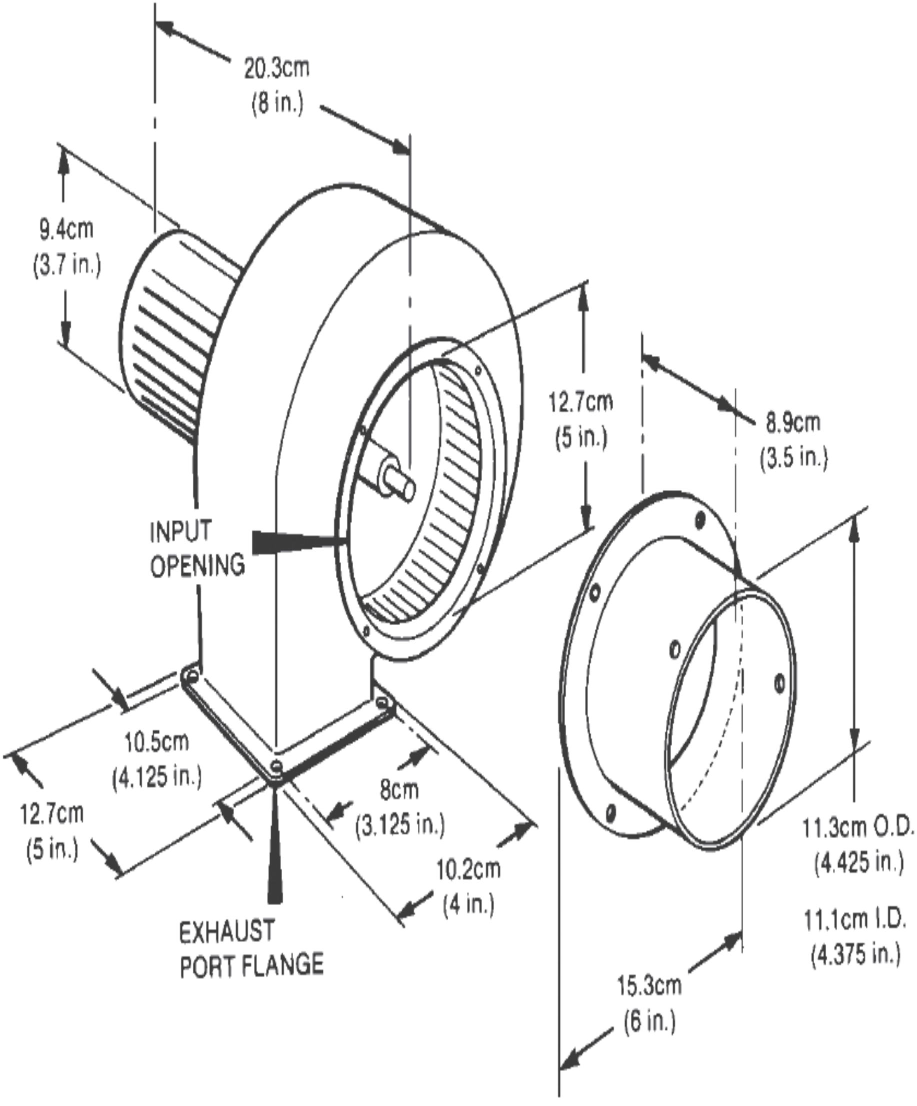
G = 10 cm (4 inches) in all cases

H = 30 cm (12 inches) in all cases

J = 15 cm (6 inches) in all cases

*Figure 3.* Spectrometer and accessories dimensions.


11.2cm I.D.
(4.375 in.)
10.2cm
(4 in.)
30.6cm
(12 in.)
15.3cm
(6 in.)


20.3cm
(8 in.)
9.4cm
(3.7 in.)
12.7cm
(5 in.)
8.9cm
(3.5 in.)
INPUT
OPENING
10.5cm
(4.125 in.)
12.7cm
(5 in.)
8cm
(3.125 in.)
10.2cm
(4 in.)
EXHAUST
PORT FLANGE
11.3cm O.D.
(4.425 in.)
11.1cm I.D.
(4.375 in.)
15.3cm
(6 in.)

# Operating instructions for VARIAN 280FS Spectrophotometer

## Lamp installment

1. Install the recommended **light source**, position the lamp so that is held by two springs in the lamp holder.
2. Press the ***POWER*** button on the ***control panel*** to turn on the instrument.
3. Turn the ***SIGNAL*** control knob to LAMP, the AA/BG switch to AA, and turn GAIN fully counterclockwise.
4. Turn the LAMP CURRENT CONTROL (F) clockwise until the ENERGY/LAMP METER (E) shows the proper value indicated on the lamp label.
5. Change the ***SIGNAL*** control knob to SET UP. Set the SLIT (A) to the proper value and adjust the COARSE ADJUST WAVELENGTH CONTROL (D) to the correct setting on the WAVELENGTH COUNTER (B) (refer to the Analytical Methods Manual for proper settings for the element of interest).�
6. Turn the FINE ADJUST WAVELENGTH CONTROL (C) slowly to obtain maximum needle deflection to the right on the ENERGY/LAMP meter (E).� With the GAIN control knob (G) keep the needle on scale.
7. Turn the two alignment knobs on the lamp holder to maximize the ENERGY/LAMP meter (E) reading.
8. Close the lamp compartment.
9. Change the ***SIGNAL*** control knob to ABS and proceed to ignite the flame.

**A � Slit Selector**

**B � Wavelength Counter**

**C � Fine Adjust Wavelength  Control**

**D � Coarse Adjust Wavelength Control**

**E � Energy/Lamp Meter**

**F � Lamp Current Control**

**G � Gain Control**

By:A.Sorkheh@yahoo.com

# Gas ignition

1. Turn the *POWER switch* of the gas control (K) to the ON position.

2. Turn on the vent hood to remove fumes and vapors from the flame.

3. Switch the OXIDANT SELECTOR VALVE (I) to air. The air supply for the AA in room 263 comes from a central source and an oil-filter is used to maintain the air free of oil and dust .

4. Switch the FUEL VALVE (C) to the on position (For most of the analyses acetylene is the fuel).

5. Once the fuel and oxidant flow meter are set at 30 and 55 (arbitrary units) respectively, ignite the burner by pressing the IGNITE button (E).

By:A.Sorkheh@yahoo.com

**A � Fuel Pressure Gauge**

**B � Oxidant Pressure Gauge**

**C � Fuel Toggle Valve**

**D � Fuel and Oxidant Flowmeters**

**E � Ignite Button**

**F � Fuel Pressure Regulator**

**G � Oxidant Pressure Regulator**

**H � Fuel Flow Needle Valve**

**I � Oxidant Selector Valve**

**J � Oxidant Flow Needle Valve**

**K � Power ON/OFF Switch**

By:A.Sorkheh@yahoo.com

# Gas Flow Adjustment

- ➢ For many elements, the fuel/oxidant ratio must be adjusted for maximum sensitivity.

**Actylene Gauge**

# Acetylene

- For most air-acetylene flames, the acetylene flow used is about 4 liters/min or 0.14 cubic feet/min.

- Acetylene is dissolved in acetone in order to prevent explosive decomposition when compressed to greater than about 30 psi.

- *Failure to change the acetylene cylinder before the cylinder pressure drops below 600 kPa (85 psig) may cause damage to valves or tubing within the burner gas control system due to high acetone carryover*

- To provide a margin of safety, acetylene should not be used above 15 psi line pressure.

- For some elements, absorption sensitivity will change as the amount of acetone increases, so it is a good practice to replace the cylinder when the pressure falls to between 75 and 100 psi.

➢ Minimize the amount of acetone which is carried over with the acetylene by:

✓ ‘Cracking’ the bottle before use by gently opening the valve to check for any drops or spray of acetone. Any bottle showing acetone should be returned to the supplier for replacement.

✓ Storing and using the bottles in the vertical position.

✓ Using only one instrument per bottle.

✓ Using only ‘instrument’ grade acetylene.

✓ Replacing bottles when the pressure drops to 750 kPa (100 psi).

By:A.Sorkheh@yahoo.com

# *Nitrous Oxide*

- ✓ **The N2O is in the liquid state, at an initial pressure of about 750 psi.**

- ✓ **A nitrous oxide-acetylene flame consumes about 20 liters per minute of N2O at 50 psi;**

- ✓ **When N2O is removed from the cylinder at this rate the expanding gas cools the diaphragm of the regulator so that sometimes it freezes, causing loss of regulation.**

- ✓ **All lines carrying N2O should be free of grease, oil or other organic material, as it is possible for spontaneous combustion to occur.**

- ✓ **For the nitrous oxide-acetylene flame, the acetylene flow is about 14 liters/min or 0.5 cubic feet per min.**

# Air

- **Air is the most common oxidant and can be obtained from either a compressed air cylinder or from a compressor unit**

- **The premix burner-Nebulizer will use about 20 liters per minute**

- **General Purpose Compressed Air is suitable instrument use.**

- **Air cylinders are operated at 50psi utilizing a two-stage regulator**

- **The compressor should provide at least 30 liters per minute at 50 psi, with a water and oil trap installed between the compressor**

By:A.Sorkheh@yahoo.com

# Hydrogen

- In certain applications hydrogen is used as the
- The extra dry grade (99.9+% purity) is suitable for atomic absorption work.
- An air-hydrogen flame requires about 15 liters per minute
- The air-hydrogen flame is invisible under normal circumstances, and extra caution should be excersized when using this flame.
- Do not place hand on or lean over a burner where hydrogen is being used in case the flame is actually lit.
- Always test first by placing a shiny surface above the region of combustion; the formation of water vapor indicates that the flame is lit.
- Hydrogen cylinders are used at high pressure and should be handled with care at all times.
- They are operated at 40psi using a two-stage regulator

# *Argon*

- **Argon is generally used in conjunction with hydrogen :**

- ✓ **As a flame diluent to provide a cool flame**
- ✓ **As a purge gas in the analysis of hydride-forming metals**
- ✓ **As a sheathing gas in graphite furnace work.**

- **High purity grade (99.998%) Grade Argon is good for most analyses.**

- **Argon in the prepurified grade (99.995%) can also be used but is not recommended for the Graphite Furnace since the grade often contains a small amount of oxygen which may shorten tube life.**

By:A.Sorkheh@yahoo.com

# ***Nitrogen***

- **Nitrogen is used similarly to argon, and therefore the consumption rates vary widely with the application.**

- **The high purity grade (99.9%) or the extra dry grade (99.7%) is suitable for atomic absorption work.**

- **Nitrogen cylinders are used at high pressure and should be handled with care at all times.**

By:A.Sorkheh@yahoo.com

# Burner alignment

- In order to make sure that burner is correctly aligned check that the slot in the burner head is parallel to, and slightly below the sample beam.� This procedure will only need to be carried out when changing burner heads.

1. In the control panel, set the ***SIGNAL*** control knob to ABS and the **MODE** control to continuous.� Press the **AZ** (auto zero) key to set the display to zero.

**2.** Raise the burner head with the **VERTICAL ADJUST KNOB** until an absorbance is indicated on the display.

**3.** Slowly lower the burner head with the vertical adjust until it�s just below the source beam. The display must be at zero.

**4.** Ignite gases as describe in the **GAS IGNITION** section.

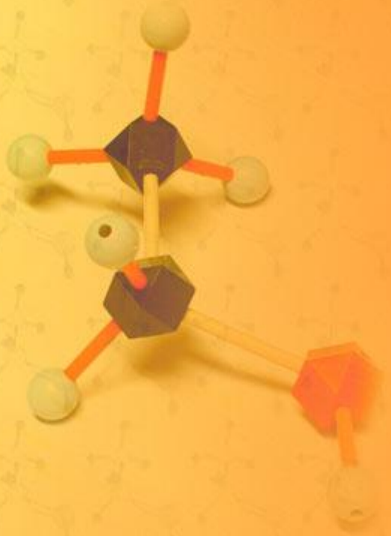
**5.** Aspirate a standard that provides an absorbance of 0.2 to 0.6 units and adjust the position of the burner head by turning the **HORIZONTAL ADJUST KNOB** until maximum absorbance is indicated.

**6.** Turn the **ROTATIONAL ADJUST KNOB** to obtain maximum absorbance.

# *Burner Adjustment*

- **Adjustment of the burner head relative to the light path of the instrument is necessary to obtain maximum sensitivity.**

- **Clean the burner frequently — never allow the burner to clog, and wash it thoroughly both inside and out.**

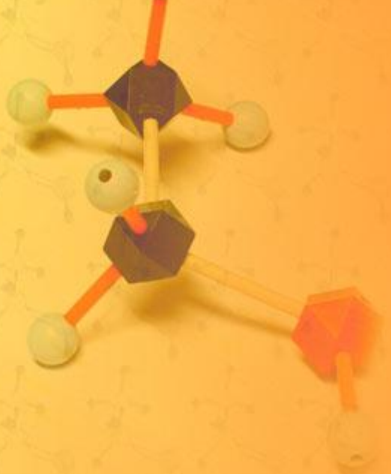


By:A.Sorkheh@yahoo.com

# Nebulizer Adjustment

- **Set up the instrument for the determination of copper, magnesium, or other elements which have an absorbing wavelength above 250 nm and whose sensitivity is not dependent on the fuel/oxidant ratio.( in 90º (Vertical) positon toward the HCL)**

- ✓ **Do not select an element requiring a nitrous oxide-acetylene flame.**



By:A.Sorkheh@yahoo.com

# Calibration

1. Set the SIGNAL control to concentration.

2. Through the keyboard enter the value for the lowest standard then press the S1 key, repeat for standard two and three.� Model 460 measures the absorbance of each standard, equates it to the concentration and constructs an appropriate calibration curve.

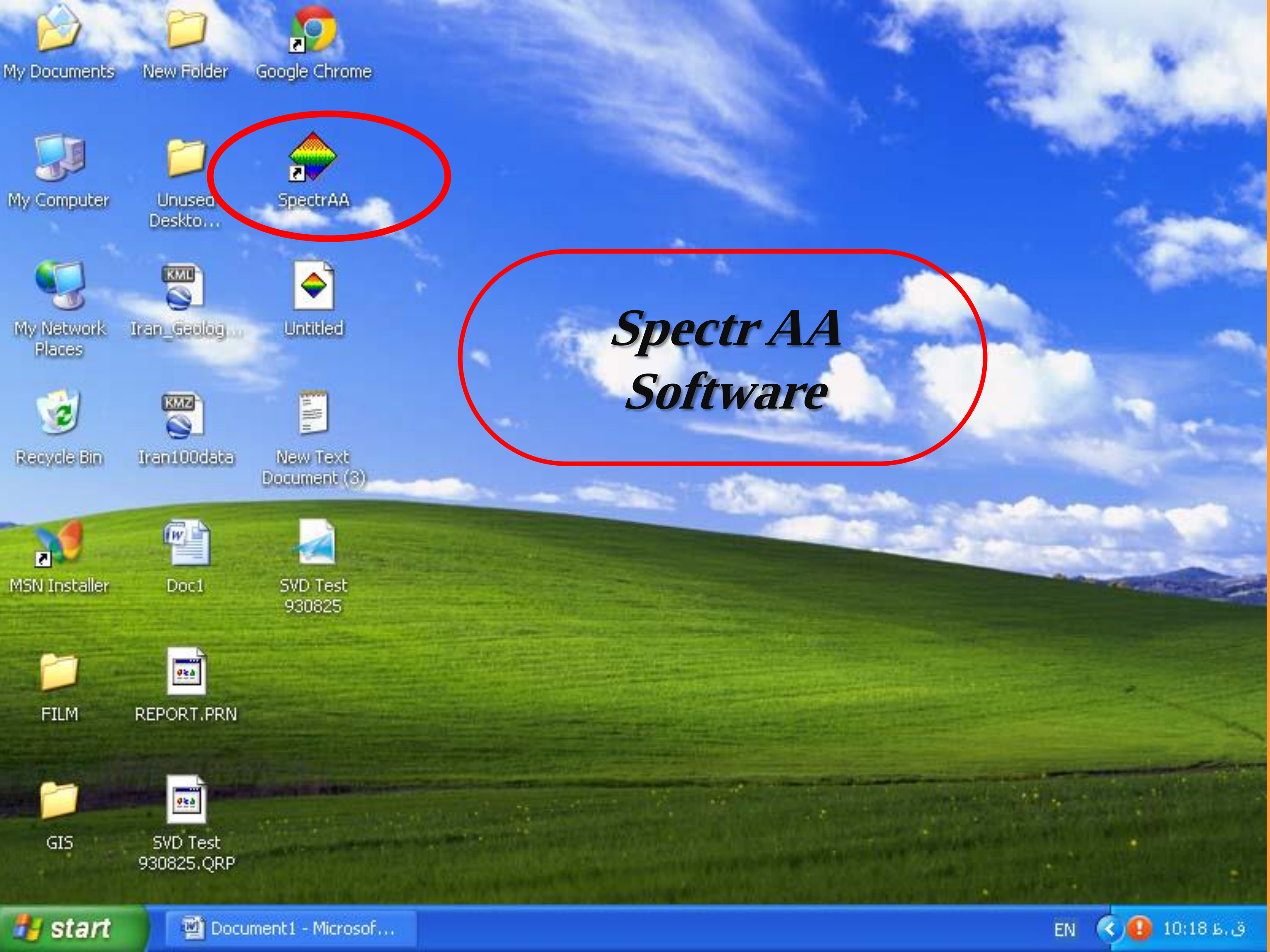
3. Enter the integration time through the keyboard and press the INT button to enter the interval (0.2 � 60 seconds).

4. Aspirate a blank solution and press the AZ key (auto zero).

5. Aspirate the standard representing S1 and press the S1 key.� Repeat for the remaining standards in order of increasing concentration.

6. Since some degradation in calibration may occur the operator is urge to recalibrate after reading various samples.

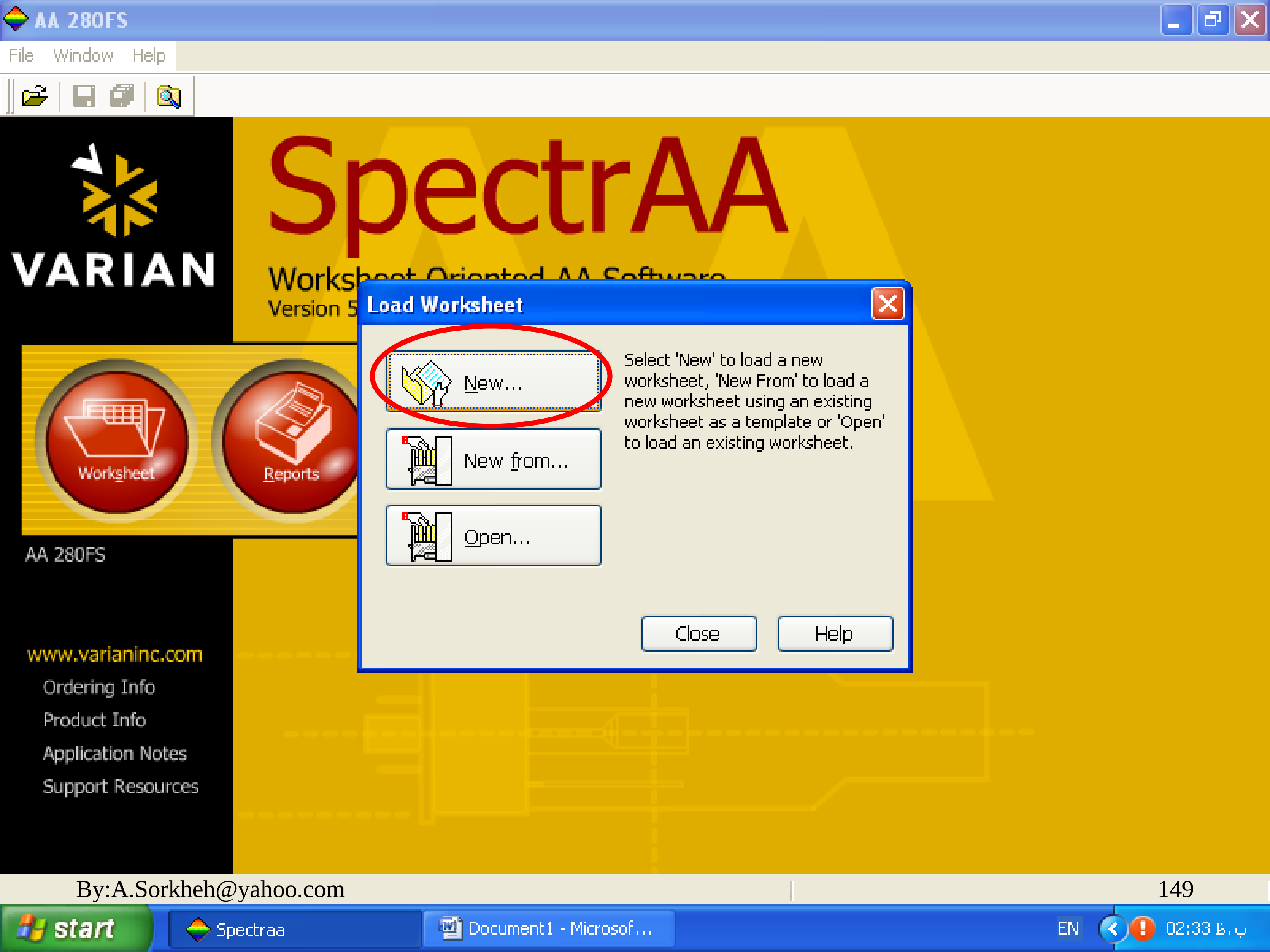
7. For Flame Emission analyses remove or unplug any hollow cathode lamp is in the lamp compartment.� Set the SIGNAL switch to EM CHOP with the appropriate slit and wavelength for the element of interest and repeat steps 2 through 6.���



***Spectr AA Software***

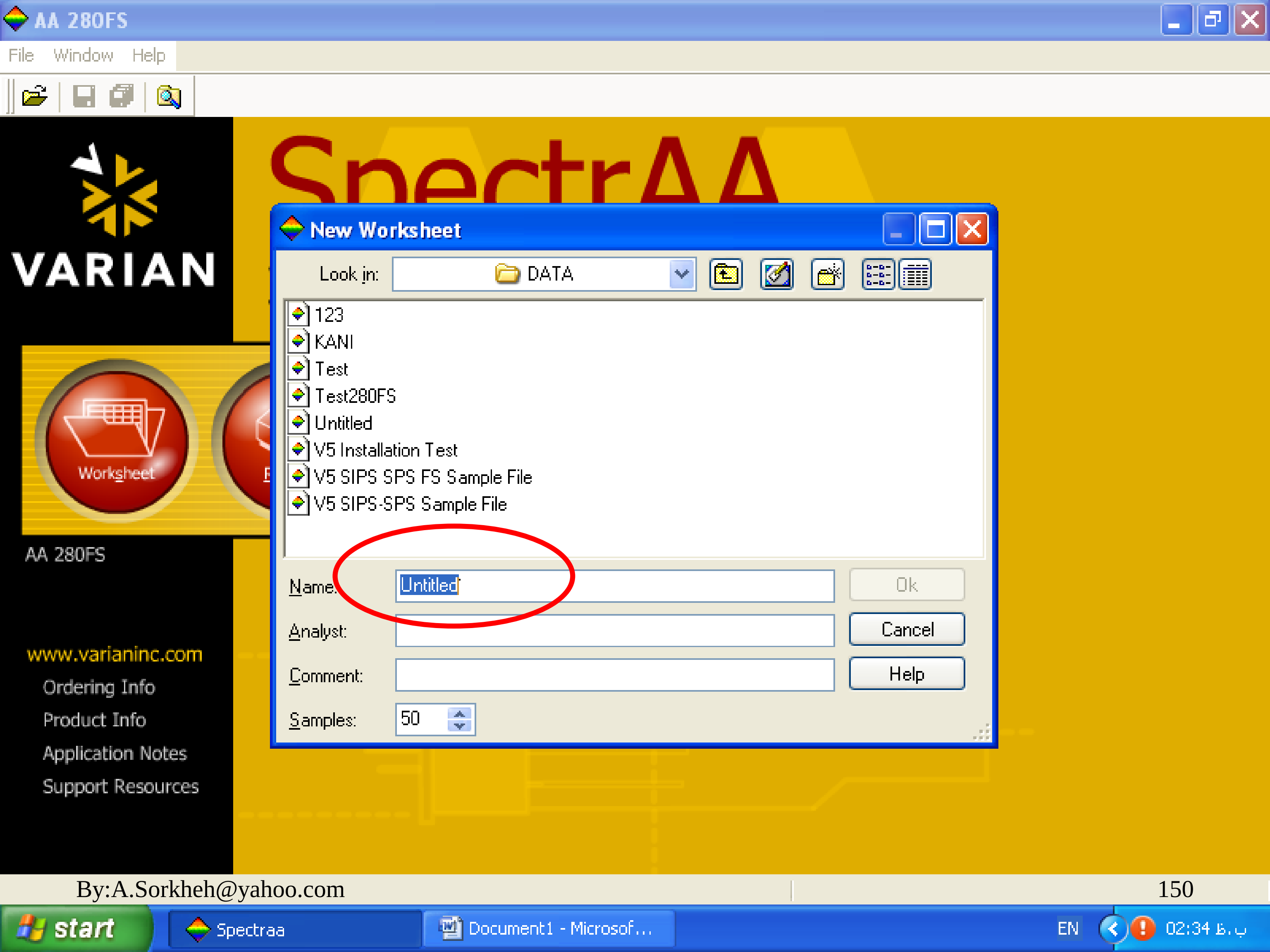
# ADDING METHOD

By:A.Sorkheh@yahoo.com



Select 'New' to load a new worksheet, 'New From' to load a new worksheet using an existing worksheet as a template or 'Open' to load an existing worksheet.

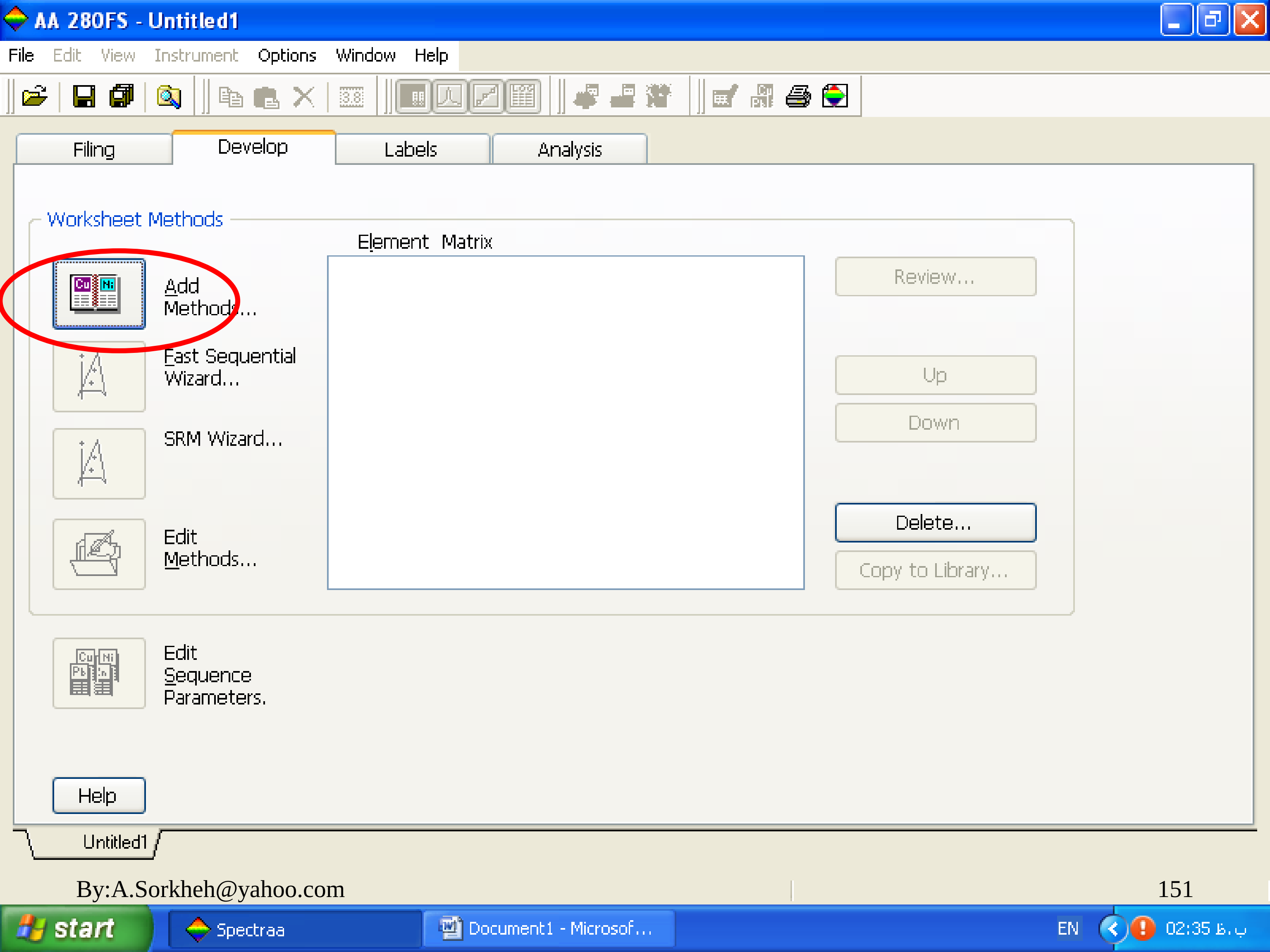
By:A.Sorkheh@yahoo.com



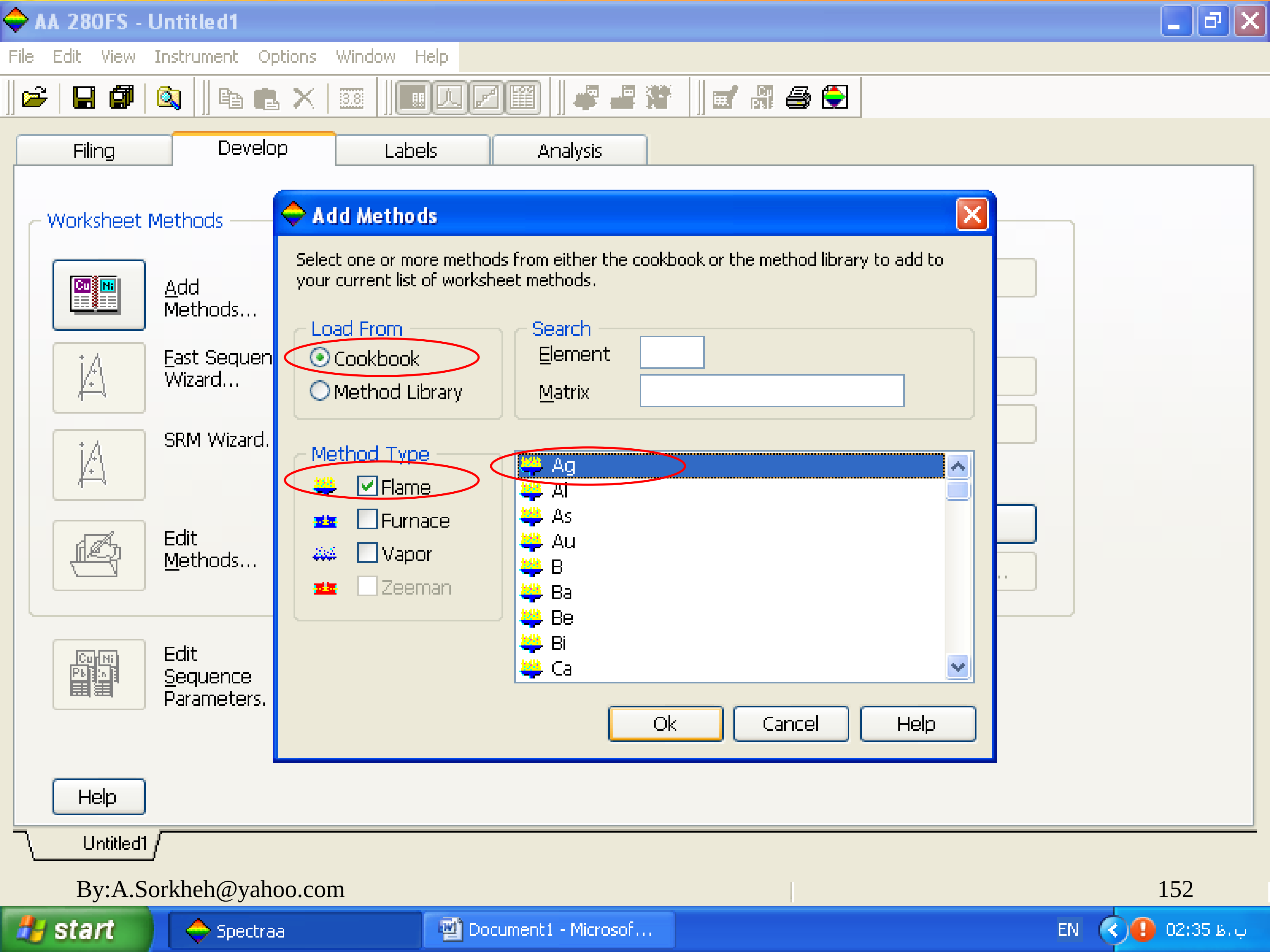
Look in: DATA

123
KANI
Test
Test280FS
Untitled
V5 Installation Test
V5 SIPS SPS FS Sample File
V5 SIPS-SPS Sample File

Name: Untitled
Analyst:
Comment:
Samples: 50



By:A.Sorkheh@yahoo.com



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

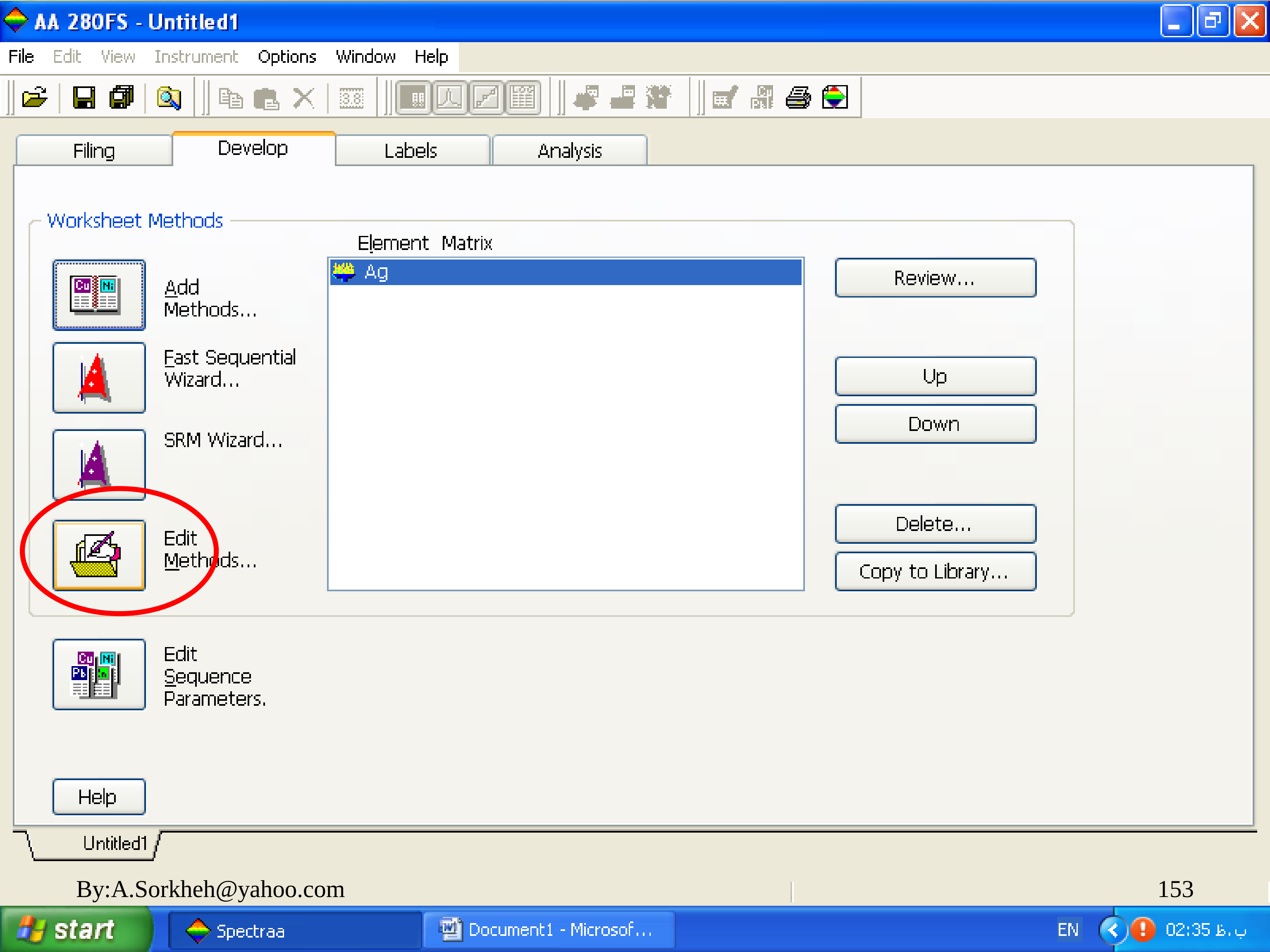
Load From
● Cookbook
○ Method Library

Search
Element
Matrix

Method Type
☑ Flame
☐ Furnace
☐ Vapor
☐ Zeeman

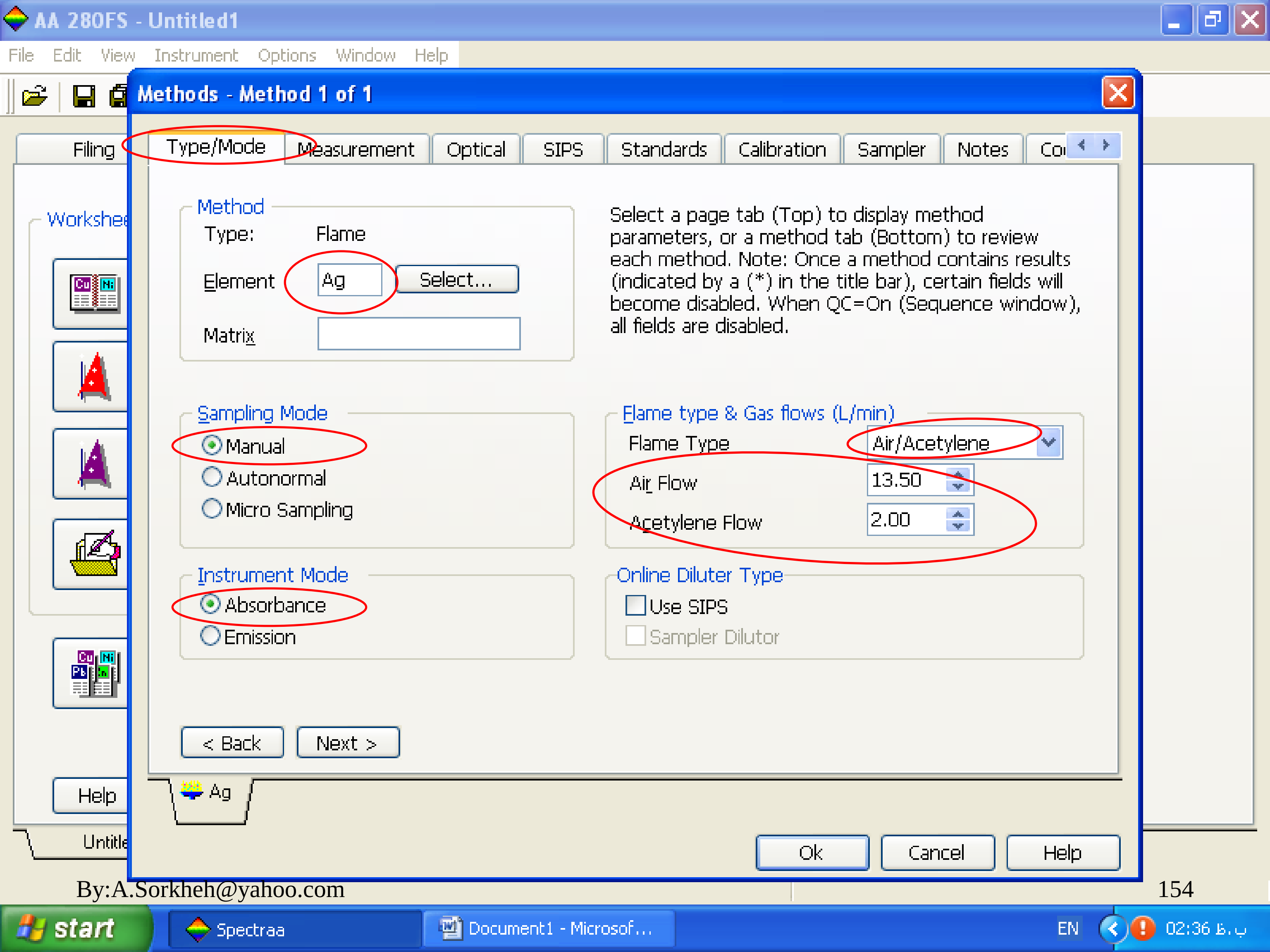
Ag
Al
As
Au
B
Ba
Be
Bi
Ca

By:A.Sorkheh@yahoo.com



| Element | Matrix |
|---|---|
| Ag | |

By:A.Sorkheh@yahoo.com



**Methods - Method 1 of 1**

**Method**

Type: Flame

Element: Ag

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 Dilutor

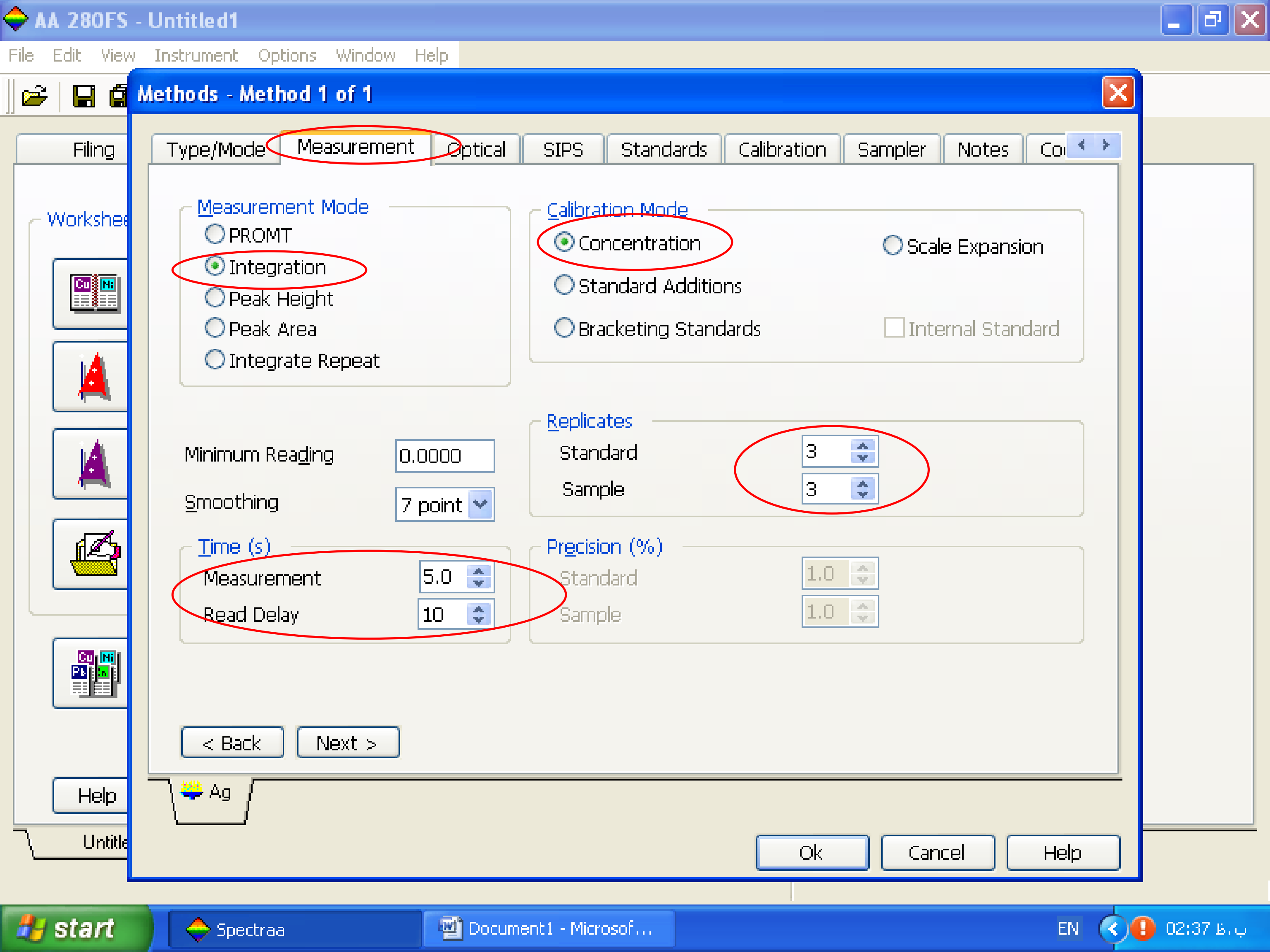
By:A.Sorkheh@yahoo.com

For the air-acetylene flame, the acetylene flow is about 2 liters/minute,

.

For the nitrous oxide-acetylene flame, the acetylene flow is about 14 liters per minute or 30 cubic feet per hour.

By:A.Sorkheh@yahoo.com



Methods - Method 1 of 1

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

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

Minimum Reading 0.0000

Smoothing 7 point

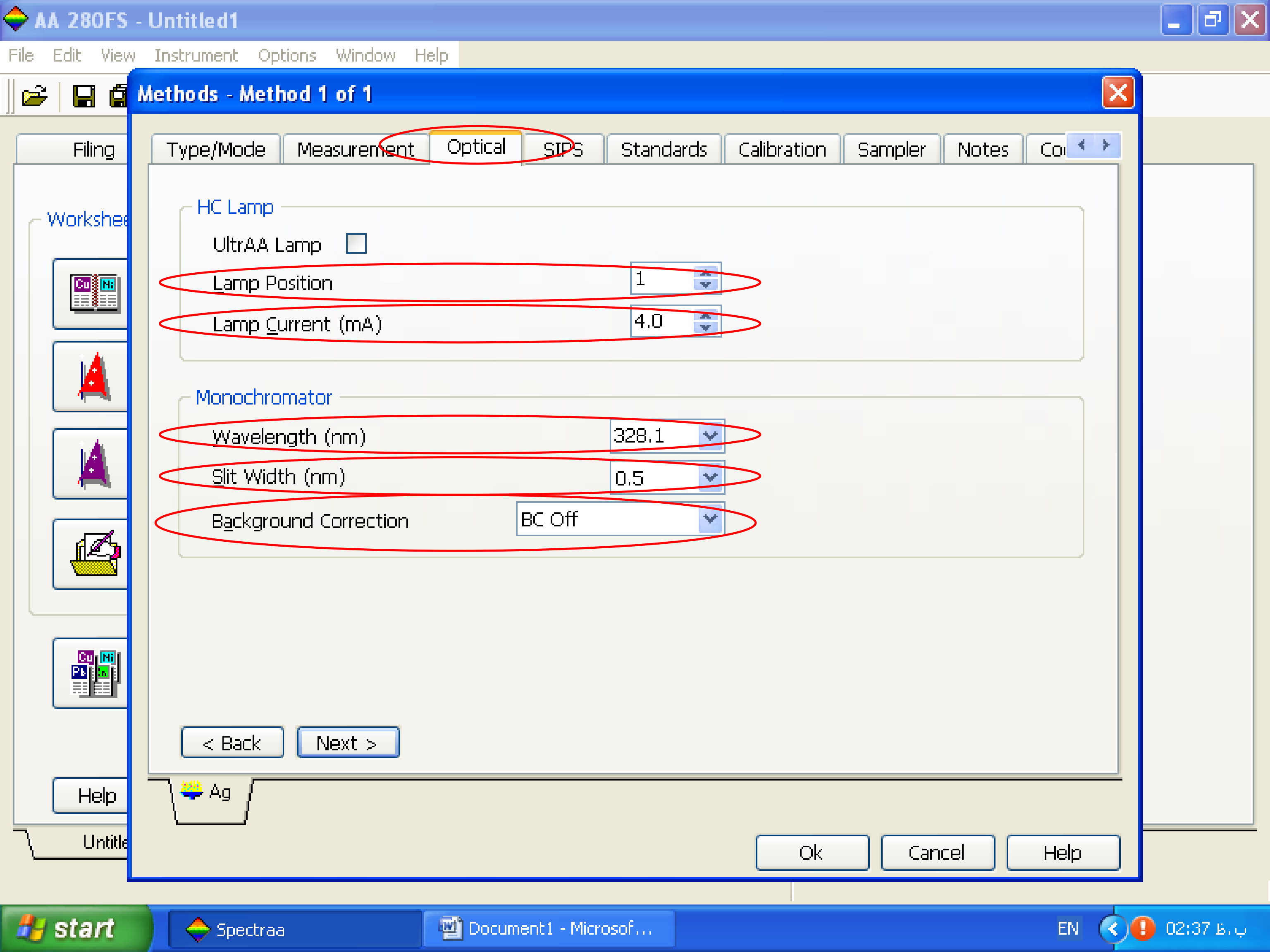
Replicates
Standard 3
Sample 3

Time (s)
Measurement 5.0
Read Delay 10

Precision (%)
Standard 1.0
Sample 1.0

For routine analyses, integration times of 1 to 3 seconds are generally used. Precision can be improved by using longer integration times, up to a maximum of roughly 10 seconds. Integration times longer than 10 seconds provide no further improvement and are generally not recommended.

By:A.Sorkheh@yahoo.com



Methods - Method 1 of 1

HC Lamp

UltrAA Lamp ☐

Lamp Position 1

Lamp Current (mA) 4.0

Monochromator

Wavelength (nm) 328.1

Slit Width (nm) 0.5

Background Correction BC Off

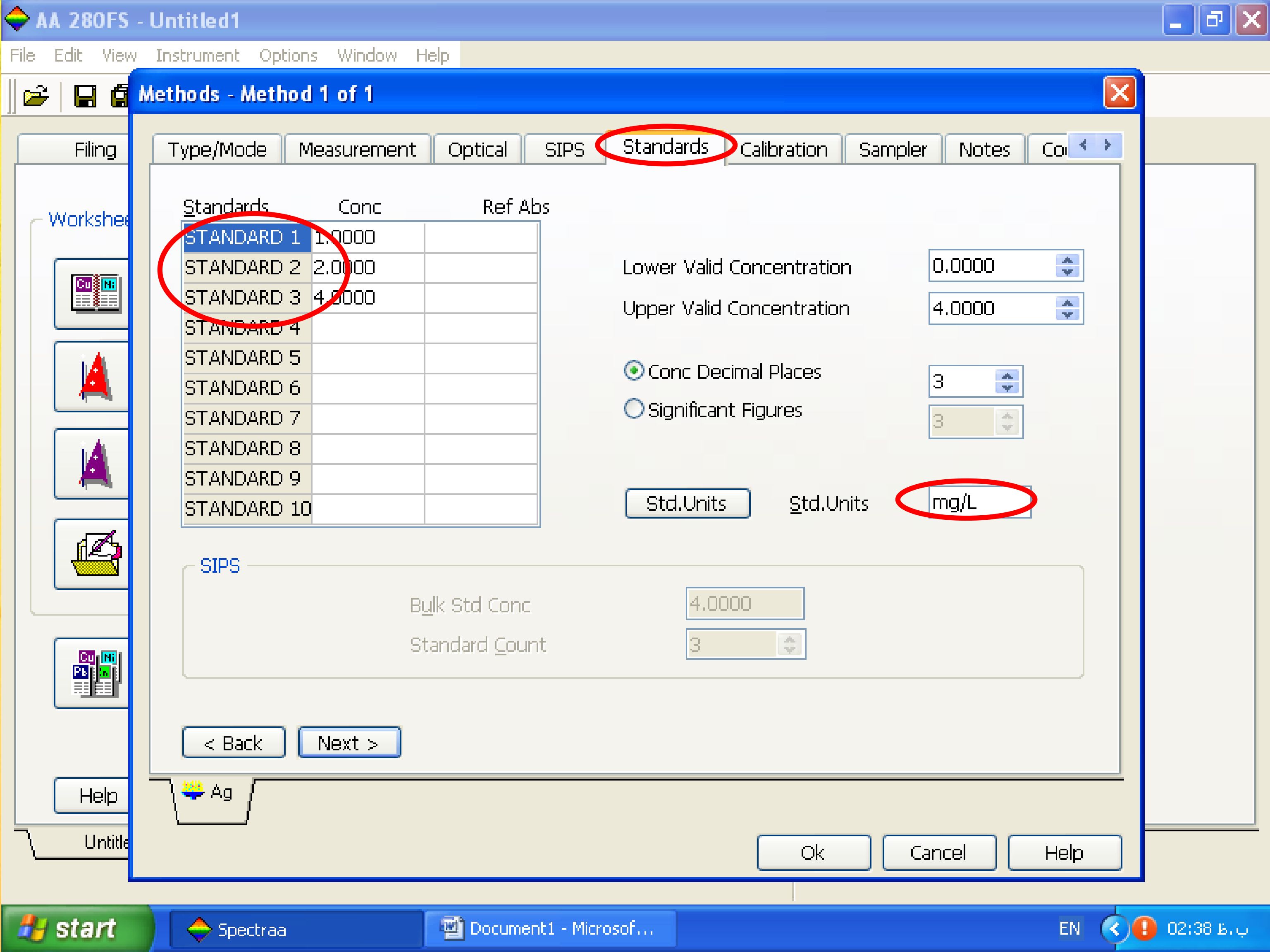
# Wavelengths

Several possible wavelengths are listed for each element. The most commonly used primary wavelength is always listed first. Select the best wavelength for your analysis based on the concentration range of the samples. If the samples are at a very low concentration, the most sensitive wavelength should be selected; if the samples are very concentrated, a less sensitive wavelength should be used.

# Slit Width

The slit width listed for each element is the one found to be optimum for that element at the particular wavelength. Other slits can be used, but the signal to noise ratio and characteristic concentration will vary.

For wavelengths in the UV range, a single recommended slit setting (0.2 nm, 0.7 nm, or 2.0 nm) is listed. Two recommended slit positions (e.g., 0.2/0.4 nm) are listed for wavelengths in the visible region. The 0.2 nm, 0.7 nm, and 2.0 nm positions are for those spectrometer models utilizing a single grating, while the 0.4 nm and 1.4 nm positions are for spectrometer models that have dual gratings.



| Standards | Conc | Ref Abs |
|---|---|---|
| STANDARD 1 | 1.0000 | |
| STANDARD 2 | 2.0000 | |
| STANDARD 3 | 4.0000 | |
| STANDARD 4 | | |
| STANDARD 5 | | |
| STANDARD 6 | | |
| STANDARD 7 | | |
| STANDARD 8 | | |
| STANDARD 9 | | |
| STANDARD 10 | | |

Lower Valid Concentration 0.0000

Upper Valid Concentration 4.0000

● Conc Decimal Places 3

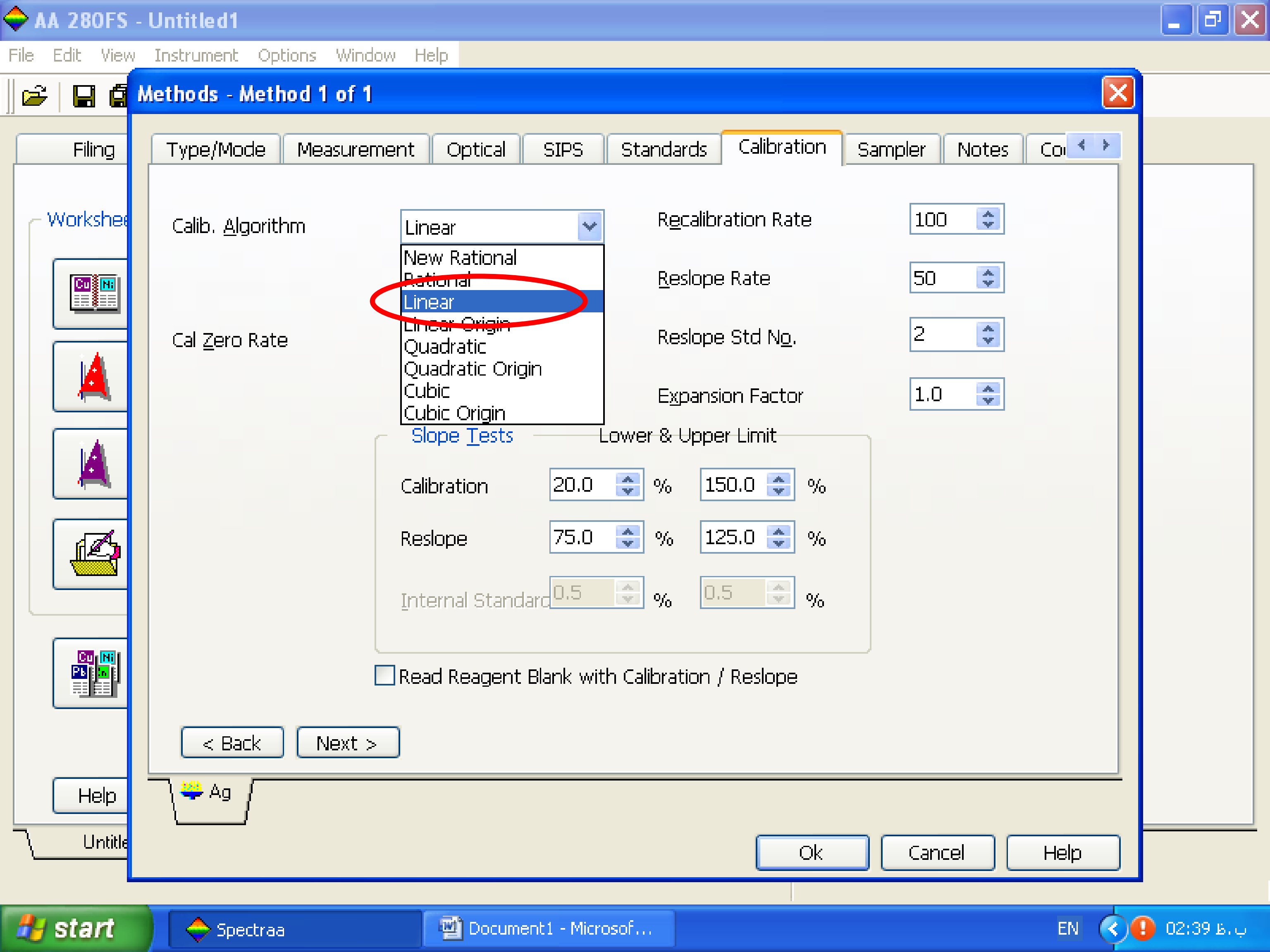
○ Significant Figures 3

Std.Units mg/L

SIPS

Bulk Std Conc 4.0000

Standard Count 3



Calib. Algorithm: Linear

Cal Zero Rate

Recalibration Rate: 100

Reslope Rate: 50

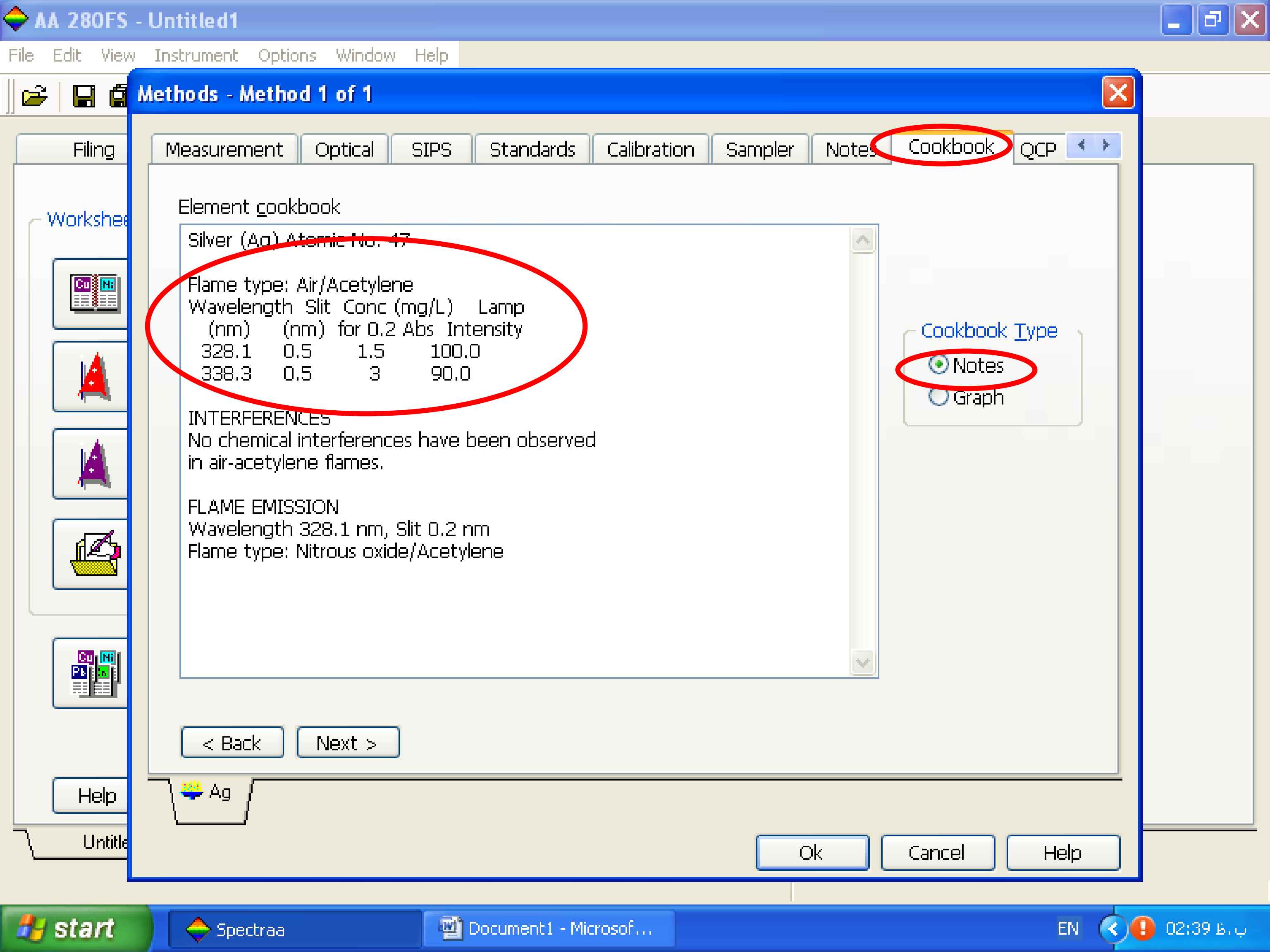
Reslope Std No.: 2

Expansion Factor: 1.0

Slope Tests — Lower & Upper Limit

| | Lower | | Upper | |
|---|---|---|---|---|
| Calibration | 20.0 | % | 150.0 | % |
| Reslope | 75.0 | % | 125.0 | % |
| Internal Standard | 0.5 | % | 0.5 | % |

☐ Read Reagent Blank with Calibration / Reslope



Element cookbook

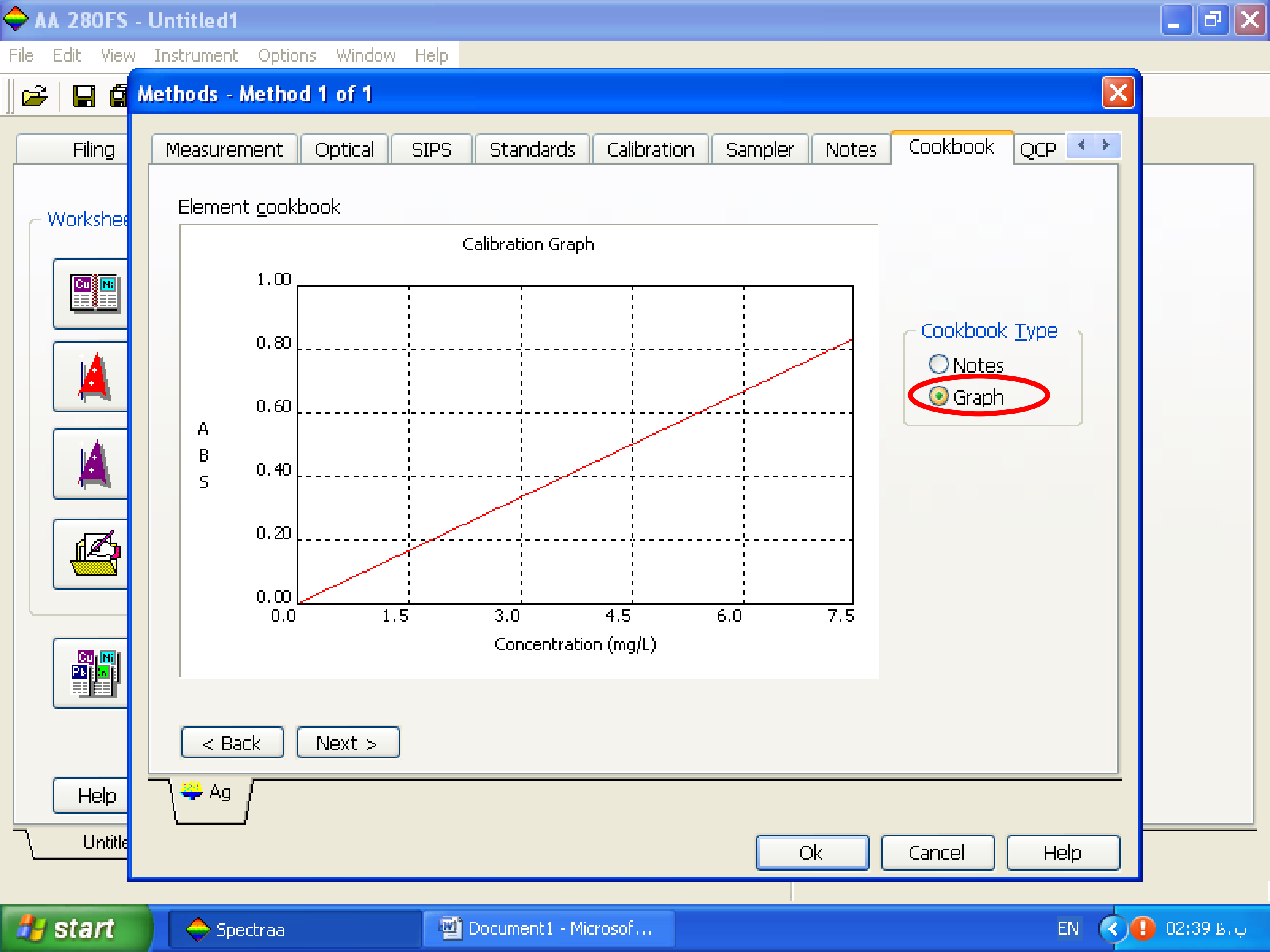
```
Silver (Ag) Atomic No. 47

Flame type: Air/Acetylene
Wavelength  Slit  Conc (mg/L)    Lamp
  (nm)      (nm)  for 0.2 Abs  Intensity
  328.1     0.5       1.5       100.0
  338.3     0.5        3         90.0

INTERFERENCES
No chemical interferences have been observed
in air-acetylene flames.

FLAME EMISSION
Wavelength 328.1 nm, Slit 0.2 nm
Flame type: Nitrous oxide/Acetylene
```

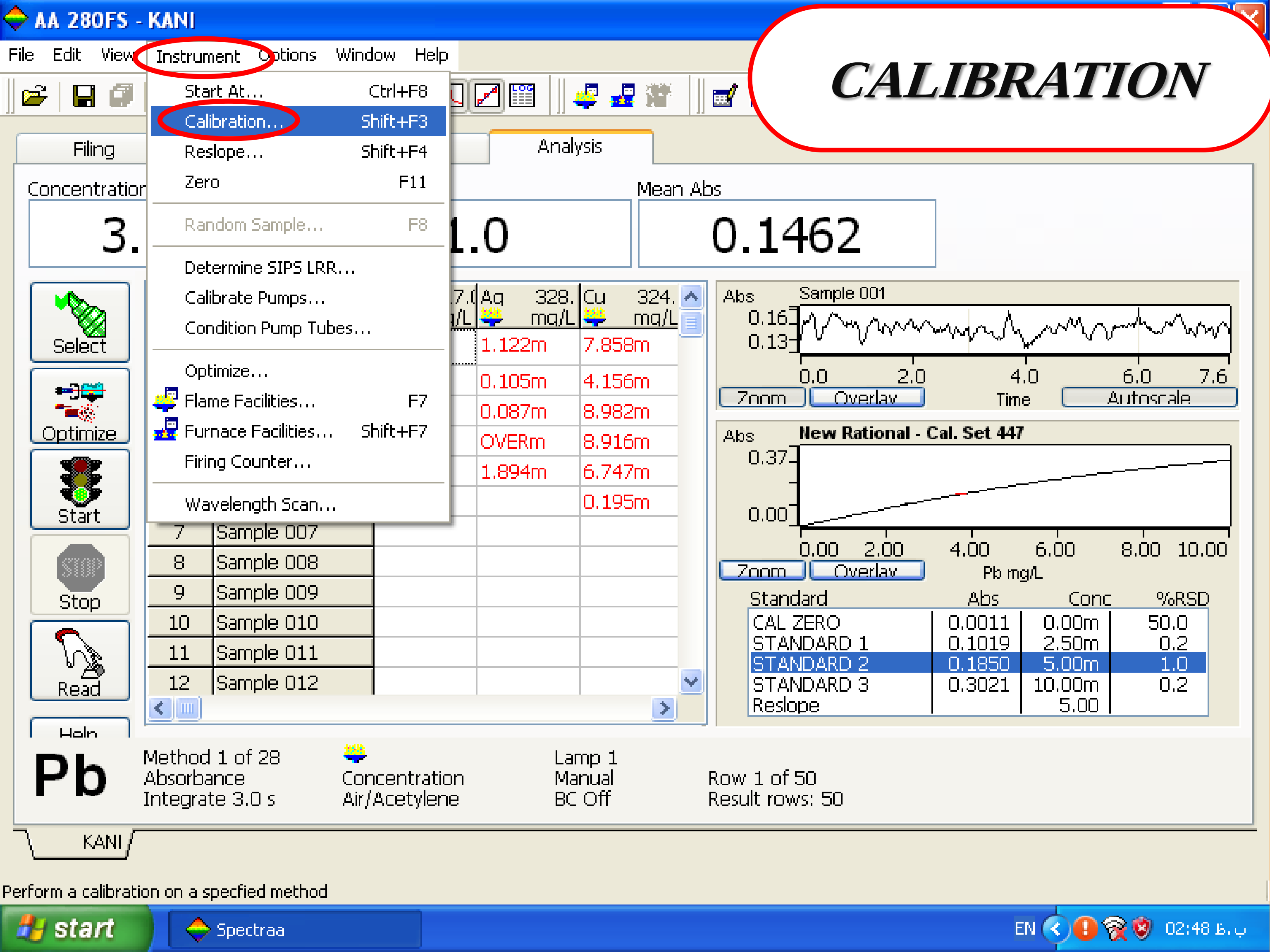
Cookbook Type
● Notes
○ Graph



Cookbook Type

○ Notes
● Graph

# CALIBRATION



Concentration 3.

1.0

Mean Abs 0.1462

| | | | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| | | | 1.122m | 7.858m |
| | | | 0.105m | 4.156m |
| | | | 0.087m | 8.982m |
| | | | OVERm | 8.916m |
| | | | 1.894m | 6.747m |
| | | | | 0.195m |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0011 | 0.00m | 50.0 |
| STANDARD 1 | 0.1019 | 2.50m | 0.2 |
| STANDARD 2 | 0.1850 | 5.00m | 1.0 |
| STANDARD 3 | 0.3021 | 10.00m | 0.2 |
| Reslope | | 5.00 | |

**Pb**

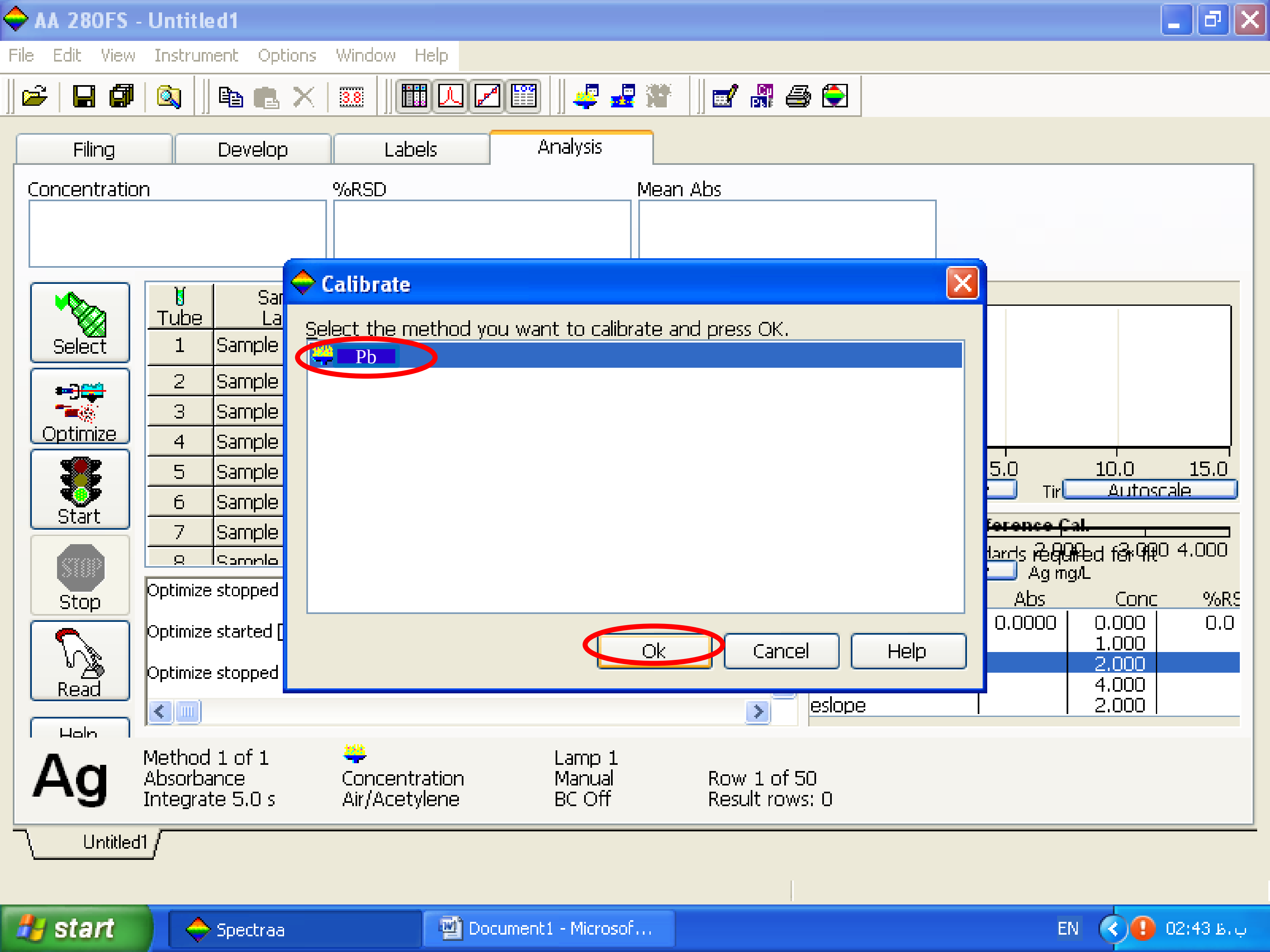
Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Row 1 of 50
Result rows: 50

Perform a calibration on a specfied method



Calibrate

Select the method you want to calibrate and press OK.

Pb

Method 1 of 1
Absorbance
Integrate 5.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

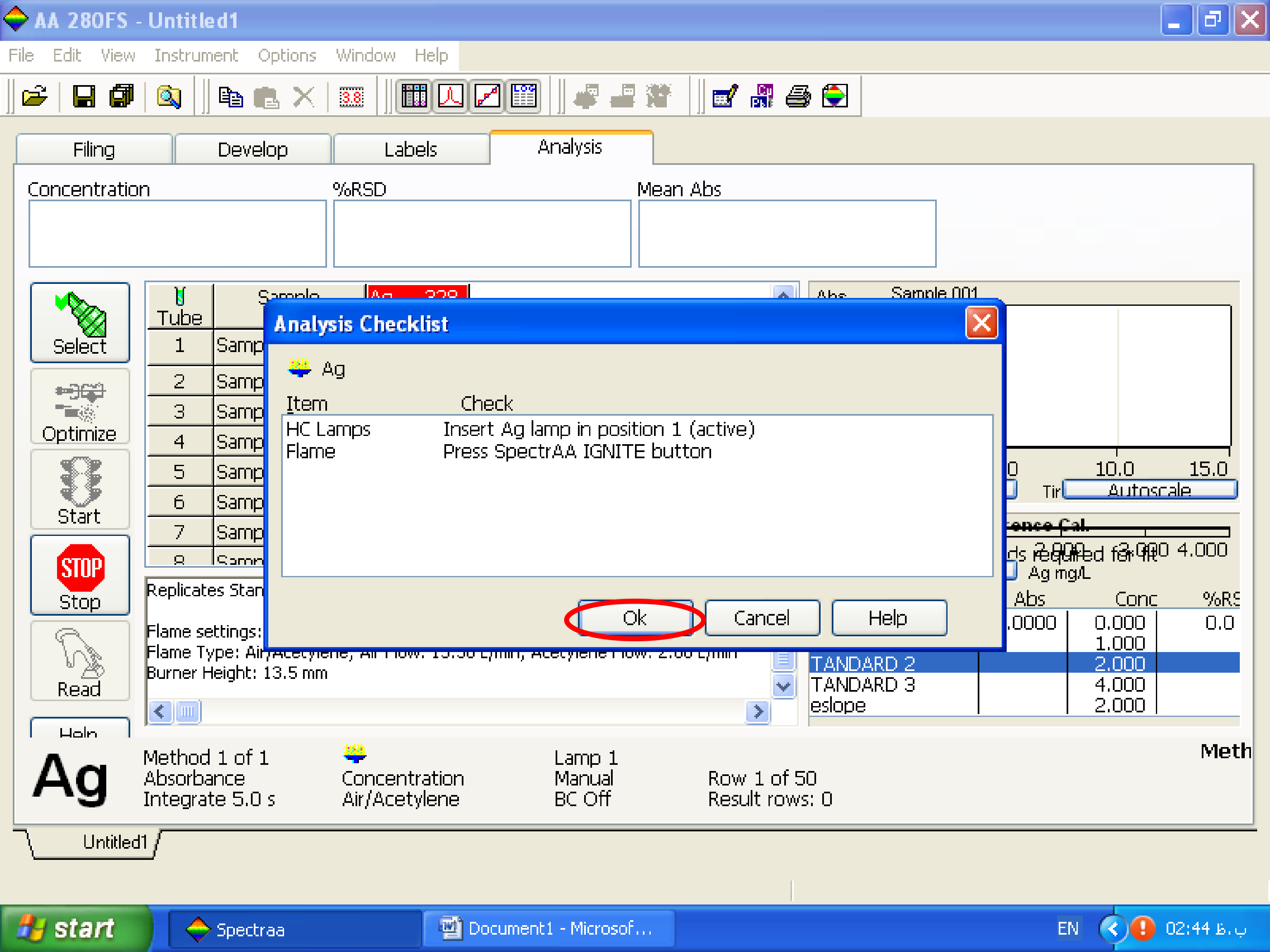
Row 1 of 50
Result rows: 0

Optimize stopped

Optimize started

Optimize stopped

| Abs | Conc | %RS |
|---|---|---|
| 0.0000 | 0.000 | 0.0 |
| | 1.000 | |
| | 2.000 | |
| | 4.000 | |
| eslope | 2.000 | |



## Analysis Checklist

Ag

| Item | Check |
|---|---|
| HC Lamps | Insert Ag lamp in position 1 (active) |
| Flame | Press SpectrAA IGNITE button |

Replicates Stan

Flame settings:
Flame Type: Air/Acetylene
Burner Height: 13.5 mm

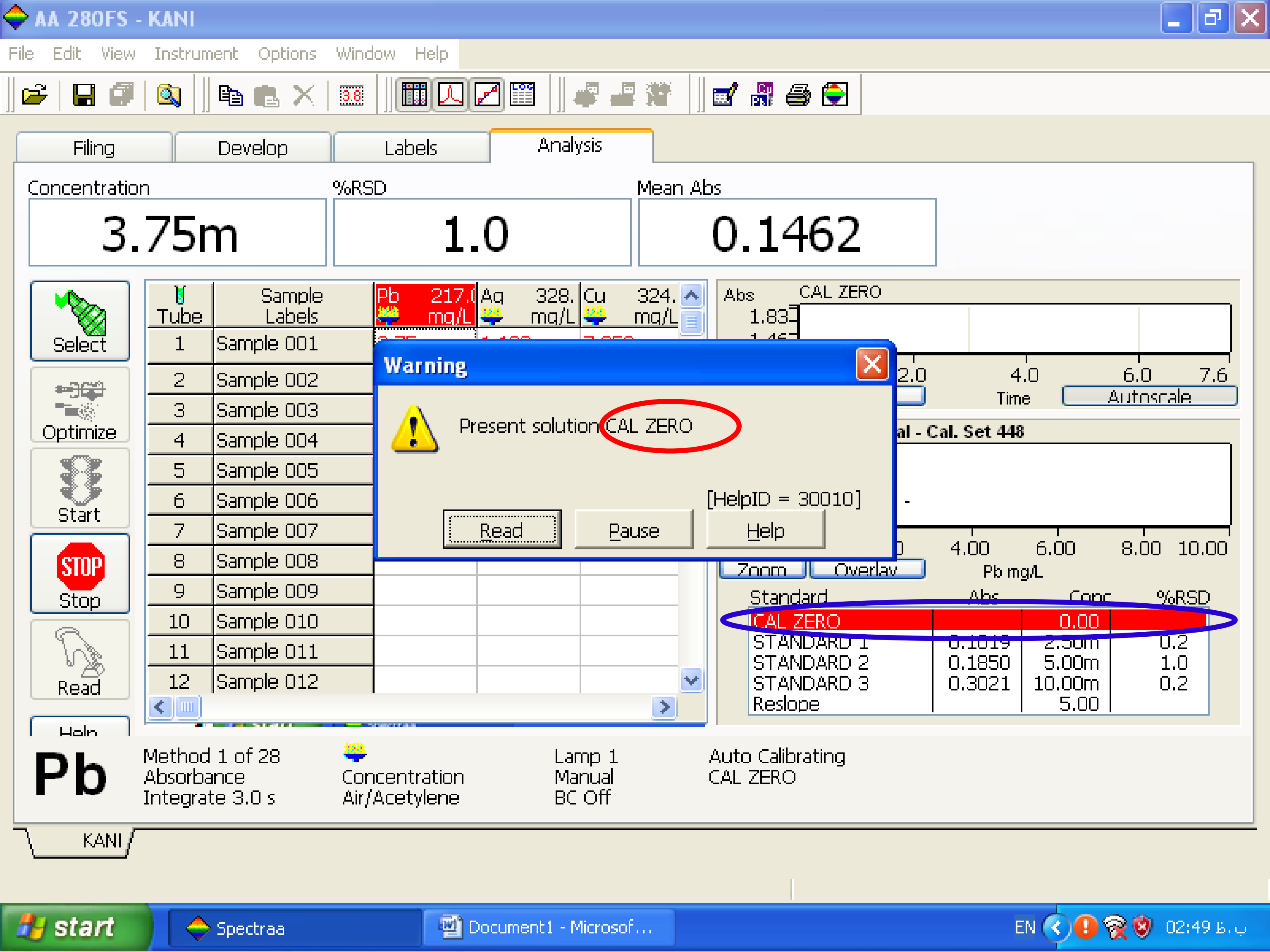
| | Abs | Conc | %RS |
|---|---|---|---|
| | .0000 | 0.000 | 0.0 |
| | | 1.000 | |
| TANDARD 2 | | 2.000 | |
| TANDARD 3 | | 4.000 | |
| eslope | | 2.000 | |

Ag
Method 1 of 1
Absorbance
Integrate 5.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Row 1 of 50
Result rows: 0



Concentration: 3.75m

%RSD: 1.0

Mean Abs: 0.1462

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | | | |
| 2 | Sample 002 | | | |
| 3 | Sample 003 | | | |
| 4 | Sample 004 | | | |
| 5 | Sample 005 | | | |
| 6 | Sample 006 | | | |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

**Warning**

Present solution CAL ZERO

[HelpID = 30010]

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | | 0.00 | |
| STANDARD 1 | 0.1019 | 2.50m | 0.2 |
| STANDARD 2 | 0.1850 | 5.00m | 1.0 |
| STANDARD 3 | 0.3021 | 10.00m | 0.2 |
| Reslope | | 5.00 | |

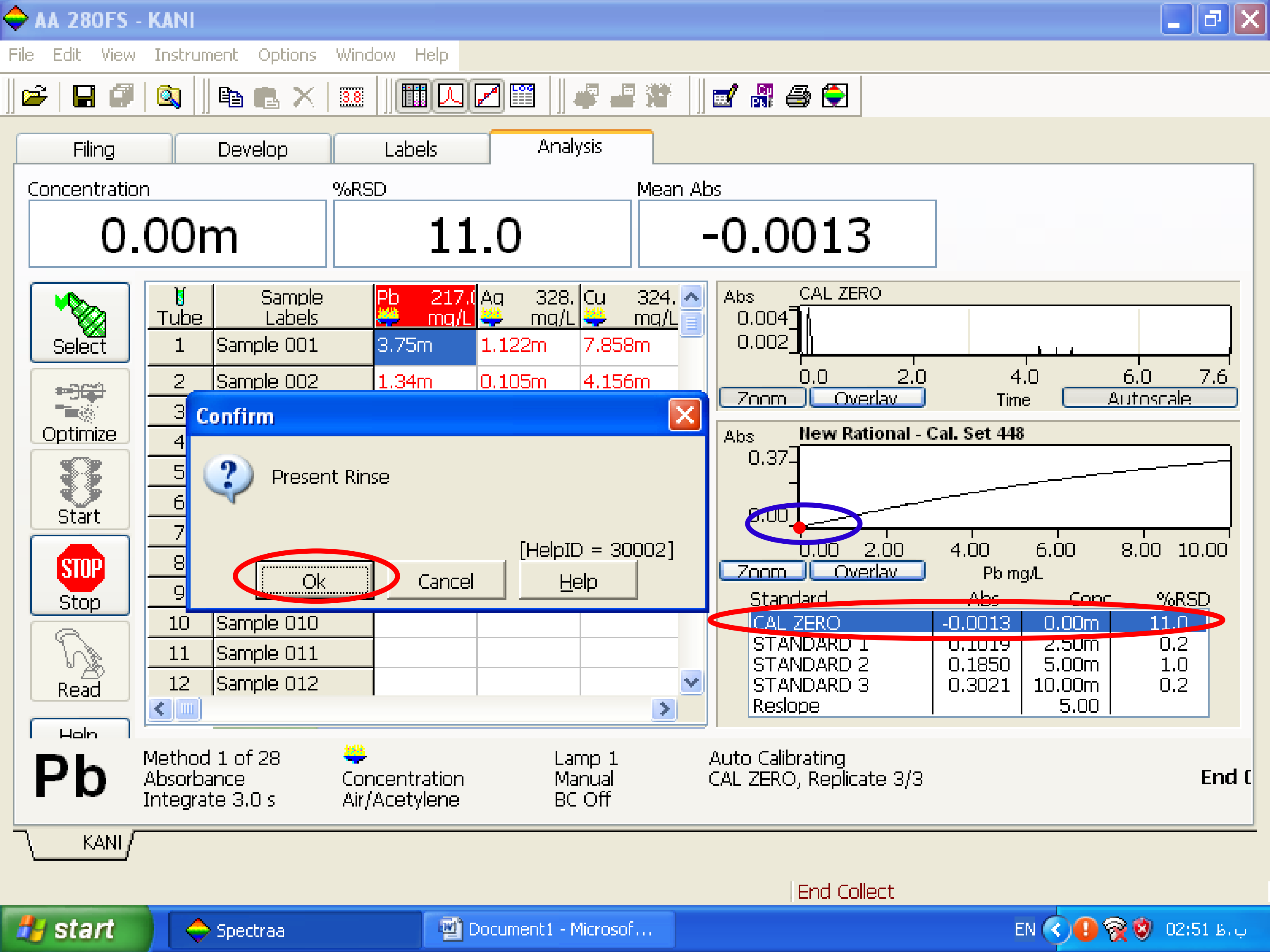
**Pb**

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
CAL ZERO



Concentration: 0.00m

%RSD: 11.0

Mean Abs: -0.0013

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | 3.75m | 1.122m | 7.858m |
| 2 | Sample 002 | 1.34m | 0.105m | 4.156m |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | | | | |
| 9 | | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

**Confirm**

Present Rinse

[HelpID = 30002]

Ok Cancel Help

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | -0.0013 | 0.00m | 11.0 |
| STANDARD 1 | 0.1019 | 2.50m | 0.2 |
| STANDARD 2 | 0.1850 | 5.00m | 1.0 |
| STANDARD 3 | 0.3021 | 10.00m | 0.2 |
| Reslope | | 5.00 | |

**Pb**

Method 1 of 28
Absorbance
Integrate 3.0 s

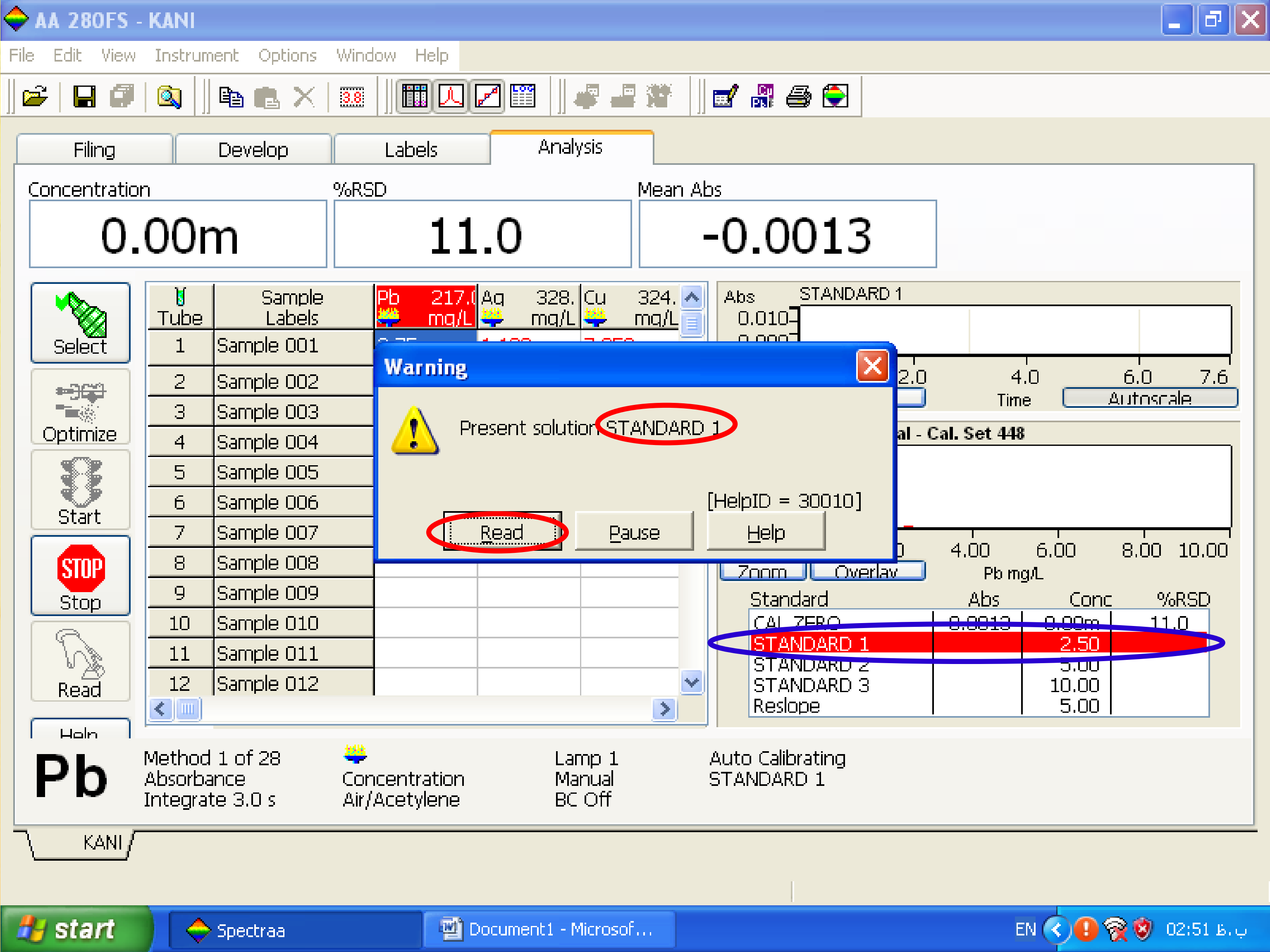
Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
CAL ZERO, Replicate 3/3

End (

End Collect



Concentration 0.00m

%RSD 11.0

Mean Abs -0.0013

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | | | |
| 2 | Sample 002 | | | |
| 3 | Sample 003 | | | |
| 4 | Sample 004 | | | |
| 5 | Sample 005 | | | |
| 6 | Sample 006 | | | |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

**Warning**

Present solution STANDARD 1

[HelpID = 30010]

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | -0.0013 | 0.00m | 11.0 |
| STANDARD 1 | | 2.50 | |
| STANDARD 2 | | 5.00 | |
| STANDARD 3 | | 10.00 | |
| Reslope | | 5.00 | |

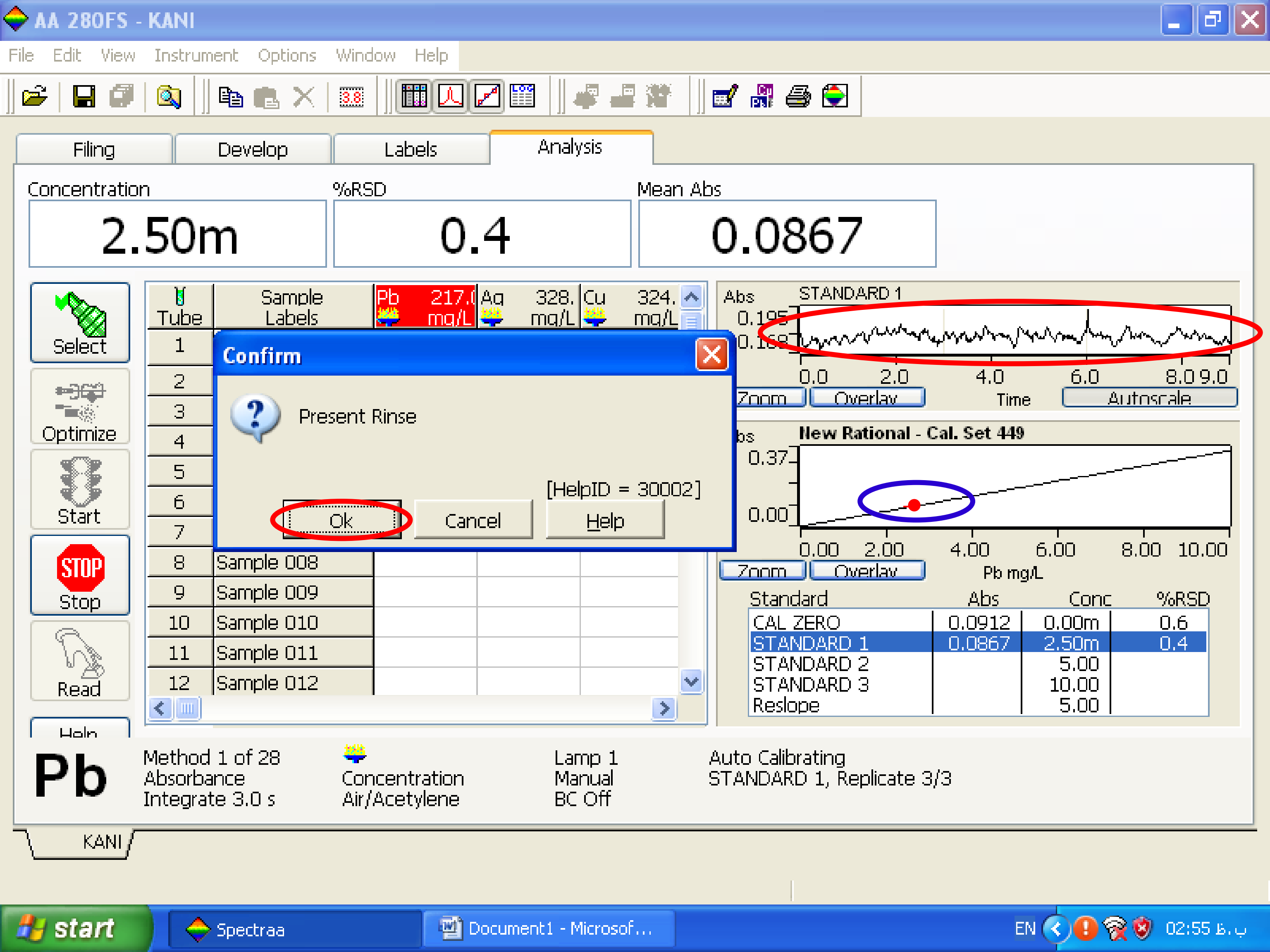
Pb

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 1



Concentration 2.50m

%RSD 0.4

Mean Abs 0.0867

**Confirm**

Present Rinse

[HelpID = 30002]

Ok Cancel Help

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | | | | |
| 2 | | | | |
| 3 | | | | |
| 4 | | | | |
| 5 | | | | |
| 6 | | | | |
| 7 | | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | | 5.00 | |
| STANDARD 3 | | 10.00 | |
| Reslope | | 5.00 | |

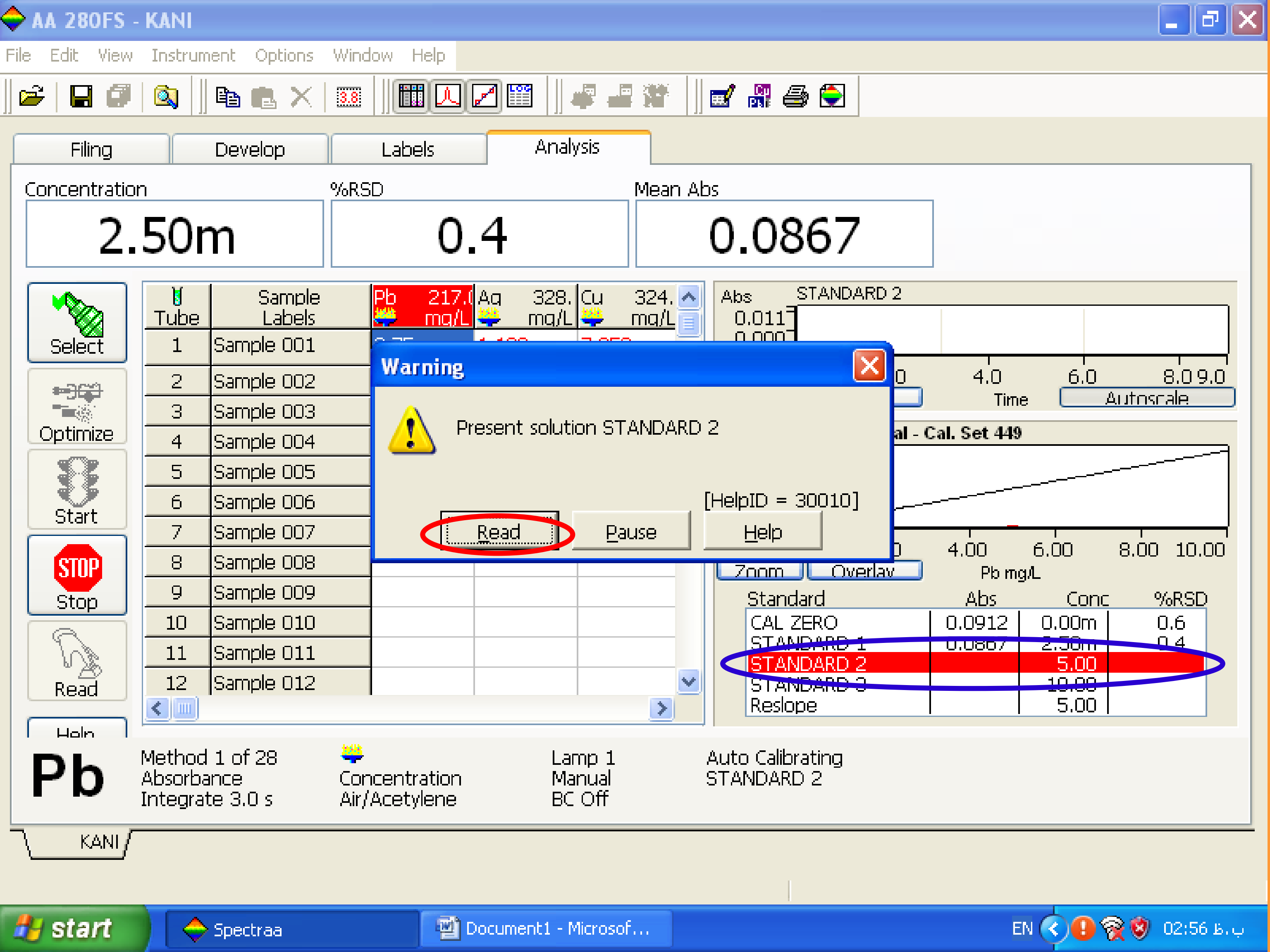
**Pb**

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 1, Replicate 3/3



Concentration: 2.50m

%RSD: 0.4

Mean Abs: 0.0867

**Warning**

Present solution STANDARD 2

[HelpID = 30010]

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | | | |
| 2 | Sample 002 | | | |
| 3 | Sample 003 | | | |
| 4 | Sample 004 | | | |
| 5 | Sample 005 | | | |
| 6 | Sample 006 | | | |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | | 5.00 | |
| STANDARD 3 | | 10.00 | |
| Reslope | | 5.00 | |

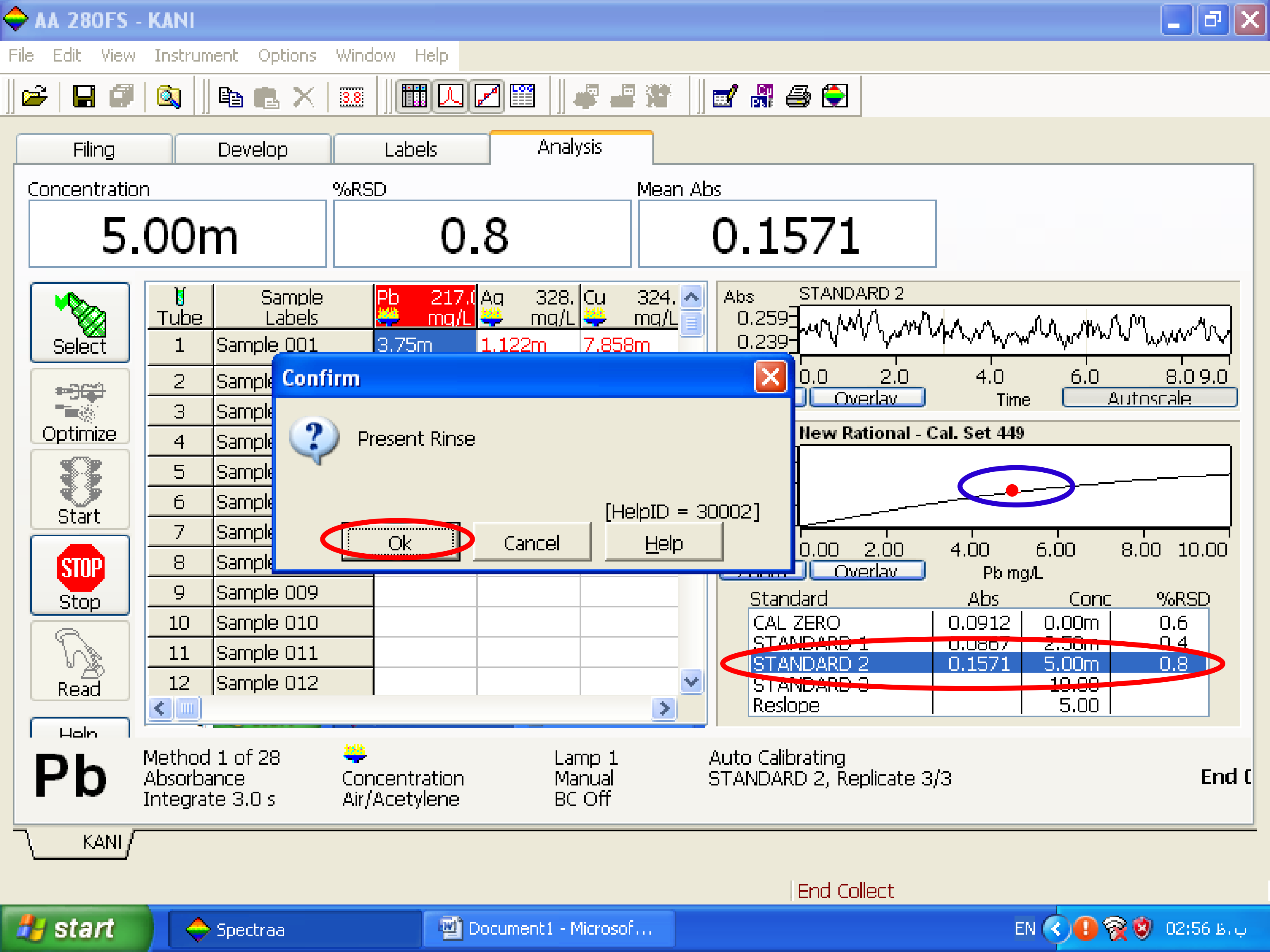
**Pb**

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 2



Concentration: 5.00m

%RSD: 0.8

Mean Abs: 0.1571

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | 3.75m | 1.122m | 7.858m |
| 2 | Sample | | | |
| 3 | Sample | | | |
| 4 | Sample | | | |
| 5 | Sample | | | |
| 6 | Sample | | | |
| 7 | Sample | | | |
| 8 | Sample | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

**Confirm**

Present Rinse

[HelpID = 30002]

Ok  Cancel  Help

STANDARD 2

New Rational - Cal. Set 449

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | 0.1571 | 5.00m | 0.8 |
| STANDARD 3 | | 10.00 | |
| Reslope | | 5.00 | |

**Pb**

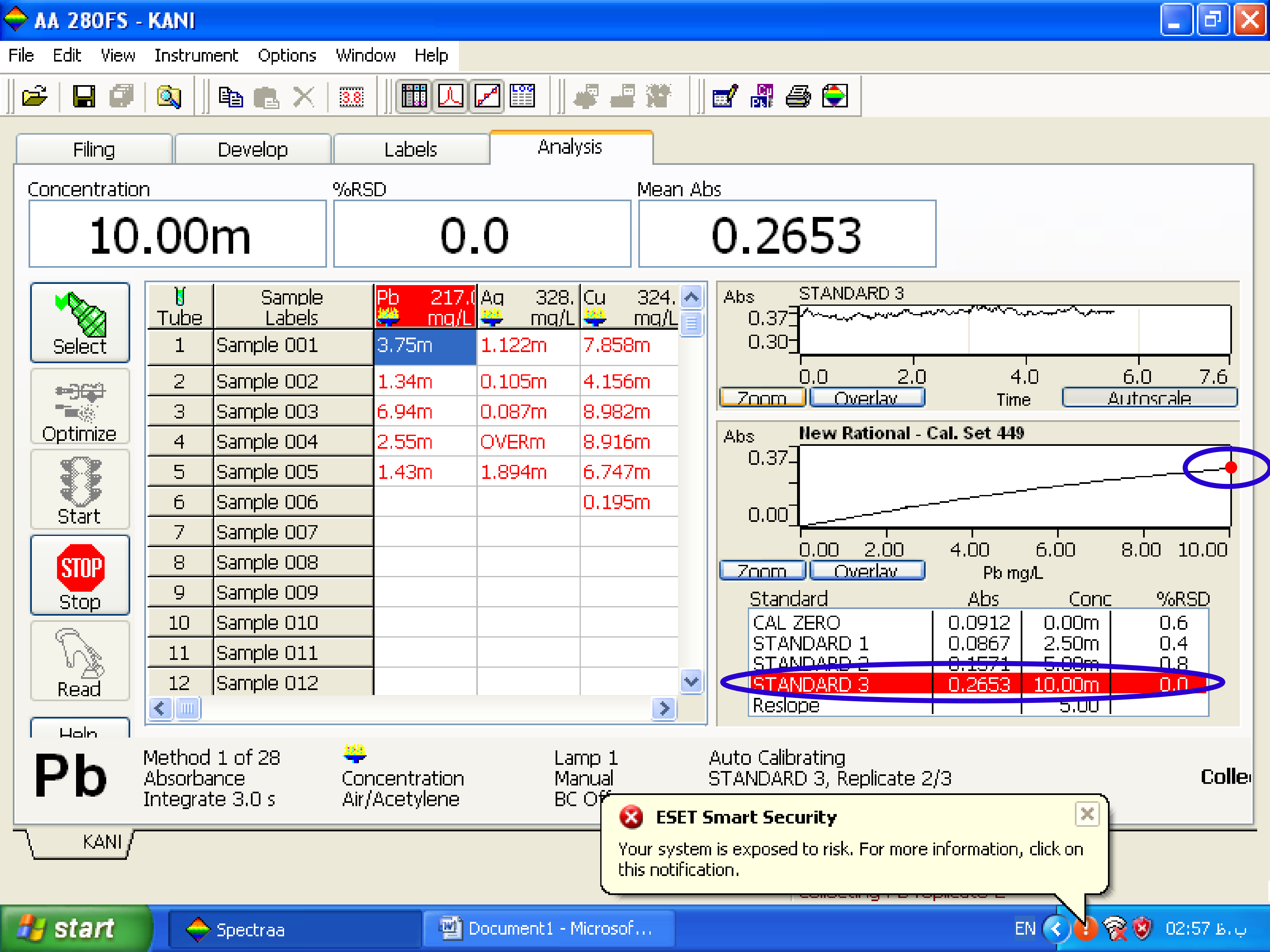
Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 2, Replicate 3/3

End C



Concentration: 10.00m

%RSD: 0.0

Mean Abs: 0.2653

| Tube | Sample Labels | Pb 217. mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | 3.75m | 1.122m | 7.858m |
| 2 | Sample 002 | 1.34m | 0.105m | 4.156m |
| 3 | Sample 003 | 6.94m | 0.087m | 8.982m |
| 4 | Sample 004 | 2.55m | OVERm | 8.916m |
| 5 | Sample 005 | 1.43m | 1.894m | 6.747m |
| 6 | Sample 006 | | | 0.195m |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |
| 9 | Sample 009 | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | 0.1571 | 5.00m | 0.8 |
| STANDARD 3 | 0.2653 | 10.00m | 0.0 |
| Reslope | | 5.00 | |

**Pb**

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

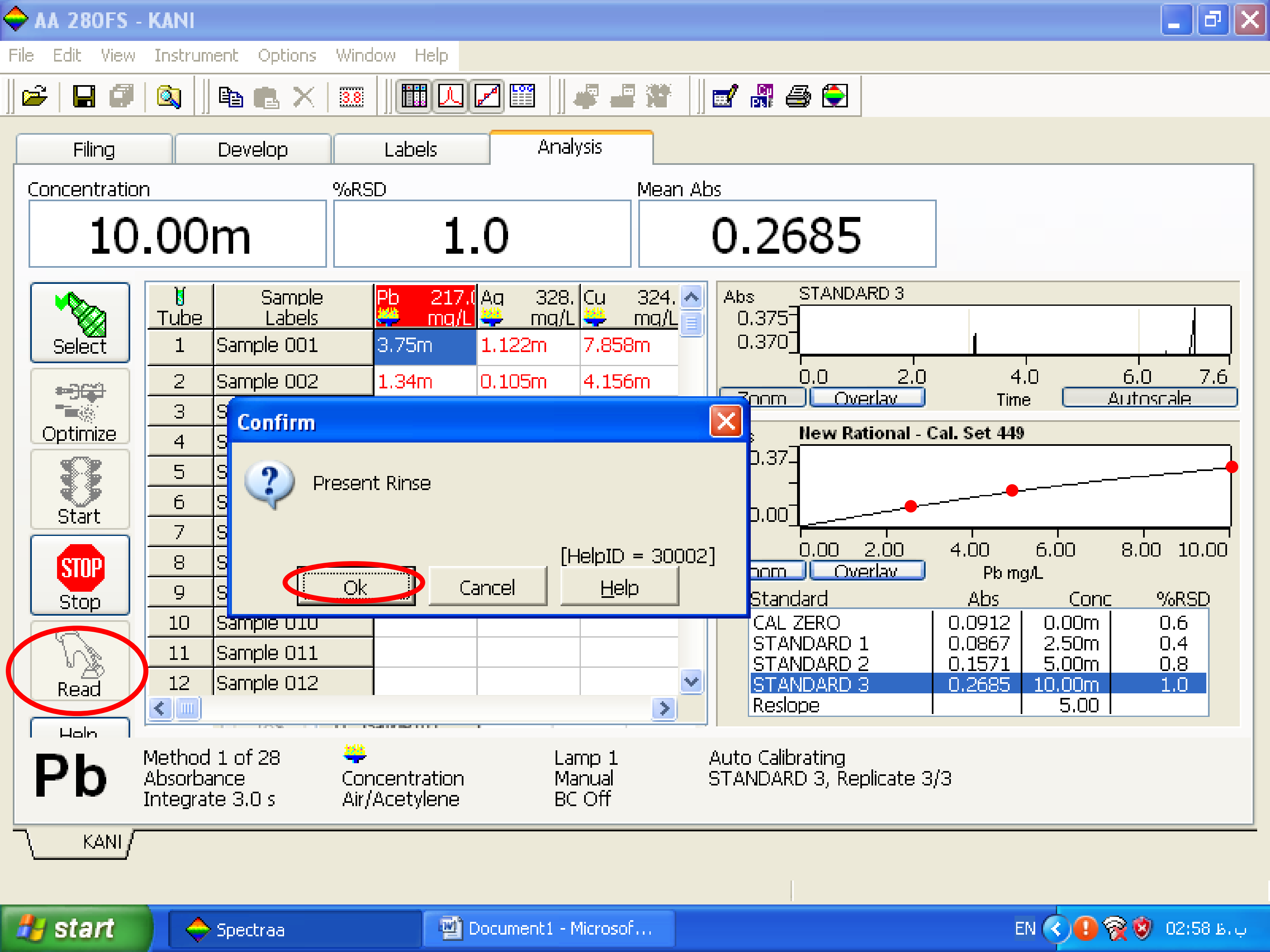
Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 3, Replicate 2/3

**Colle**

**ESET Smart Security**

Your system is exposed to risk. For more information, click on this notification.



Concentration 10.00m

%RSD 1.0

Mean Abs 0.2685

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | 3.75m | 1.122m | 7.858m |
| 2 | Sample 002 | 1.34m | 0.105m | 4.156m |
| 3 | S | | | |
| 4 | S | | | |
| 5 | S | | | |
| 6 | S | | | |
| 7 | S | | | |
| 8 | S | | | |
| 9 | S | | | |
| 10 | Sample 010 | | | |
| 11 | Sample 011 | | | |
| 12 | Sample 012 | | | |

**Confirm**

Present Rinse

[HelpID = 30002]

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | 0.1571 | 5.00m | 0.8 |
| STANDARD 3 | 0.2685 | 10.00m | 1.0 |
| Reslope | | 5.00 | |

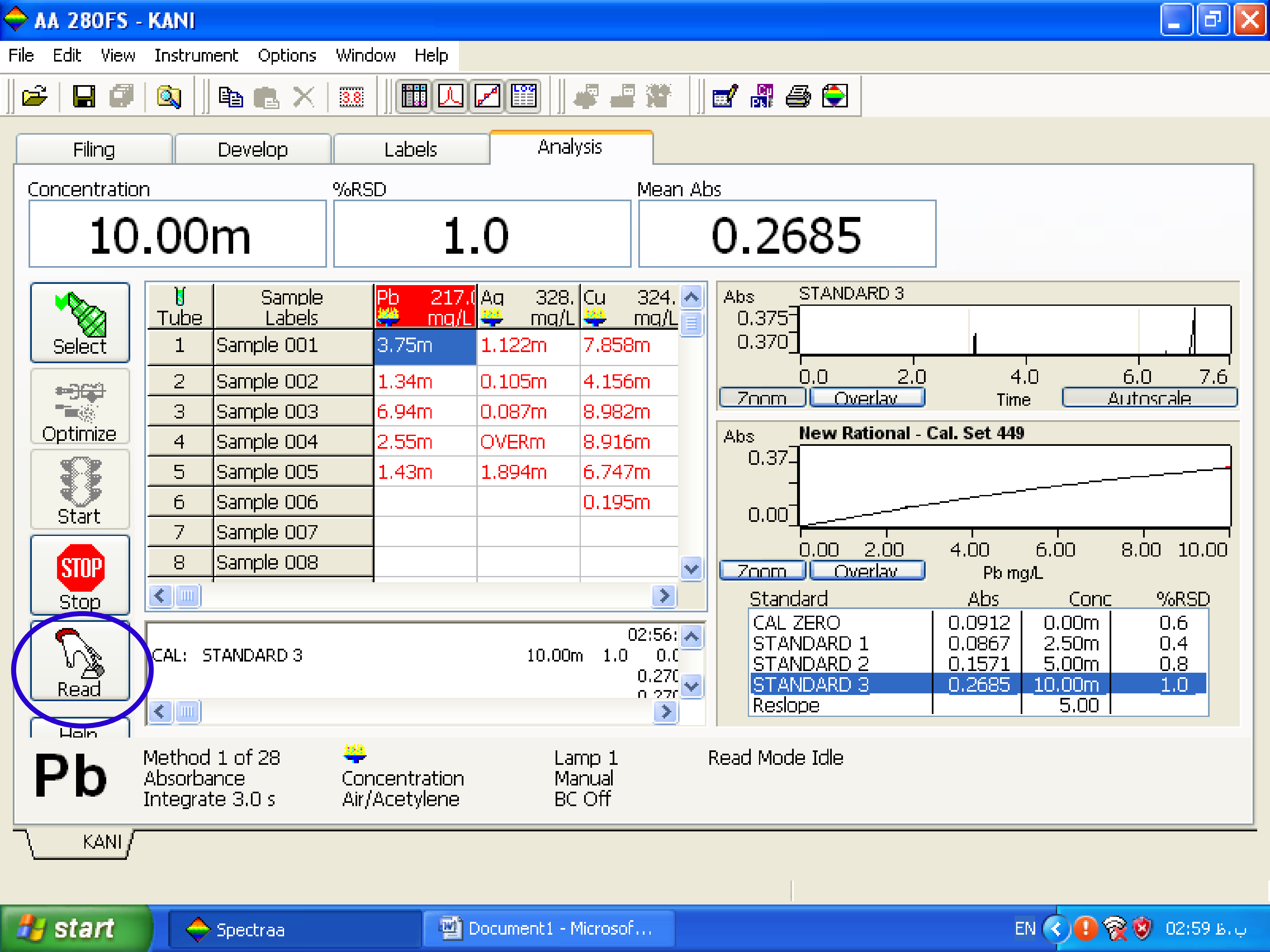
Pb

Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Auto Calibrating
STANDARD 3, Replicate 3/3



Concentration: 10.00m

%RSD: 1.0

Mean Abs: 0.2685

| Tube | Sample Labels | Pb 217.0 mg/L | Ag 328. mg/L | Cu 324. mg/L |
|---|---|---|---|---|
| 1 | Sample 001 | 3.75m | 1.122m | 7.858m |
| 2 | Sample 002 | 1.34m | 0.105m | 4.156m |
| 3 | Sample 003 | 6.94m | 0.087m | 8.982m |
| 4 | Sample 004 | 2.55m | OVERm | 8.916m |
| 5 | Sample 005 | 1.43m | 1.894m | 6.747m |
| 6 | Sample 006 | | | 0.195m |
| 7 | Sample 007 | | | |
| 8 | Sample 008 | | | |

```
                                               02:56:
CAL:   STANDARD 3                  10.00m   1.0   0.0
                                                0.270
                                                0.270
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0912 | 0.00m | 0.6 |
| STANDARD 1 | 0.0867 | 2.50m | 0.4 |
| STANDARD 2 | 0.1571 | 5.00m | 0.8 |
| STANDARD 3 | 0.2685 | 10.00m | 1.0 |
| Reslope | | 5.00 | |

**Pb**

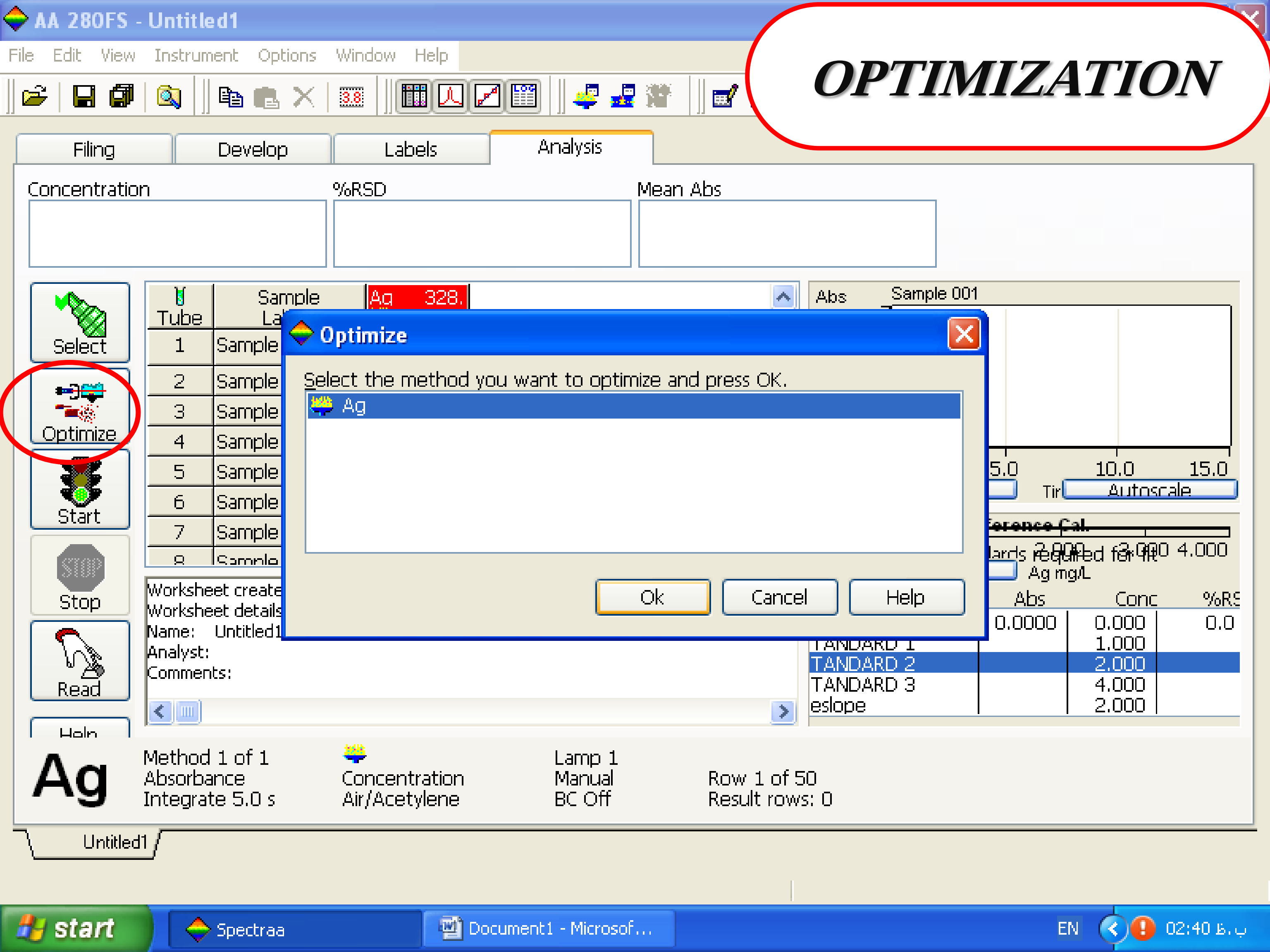
Method 1 of 28
Absorbance
Integrate 3.0 s

Concentration
Air/Acetylene

Lamp 1
Manual
BC Off

Read Mode Idle

# OPTIMIZATION



**Optimize**

Select the method you want to optimize and press OK.

Ag

Ok | Cancel | Help

| Tube | Sample Label |
|---|---|
| 1 | Sample |
| 2 | Sample |
| 3 | Sample |
| 4 | Sample |
| 5 | Sample |
| 6 | Sample |
| 7 | Sample |
| 8 | Sample |

Worksheet create
Worksheet details
Name: Untitled1
Analyst:
Comments:

| | Abs | Conc | %RS |
|---|---|---|---|
| | 0.0000 | 0.000 | 0.0 |
| TANDARD 1 | | 1.000 | |
| TANDARD 2 | | 2.000 | |
| TANDARD 3 | | 4.000 | |
| eslope | | 2.000 | |

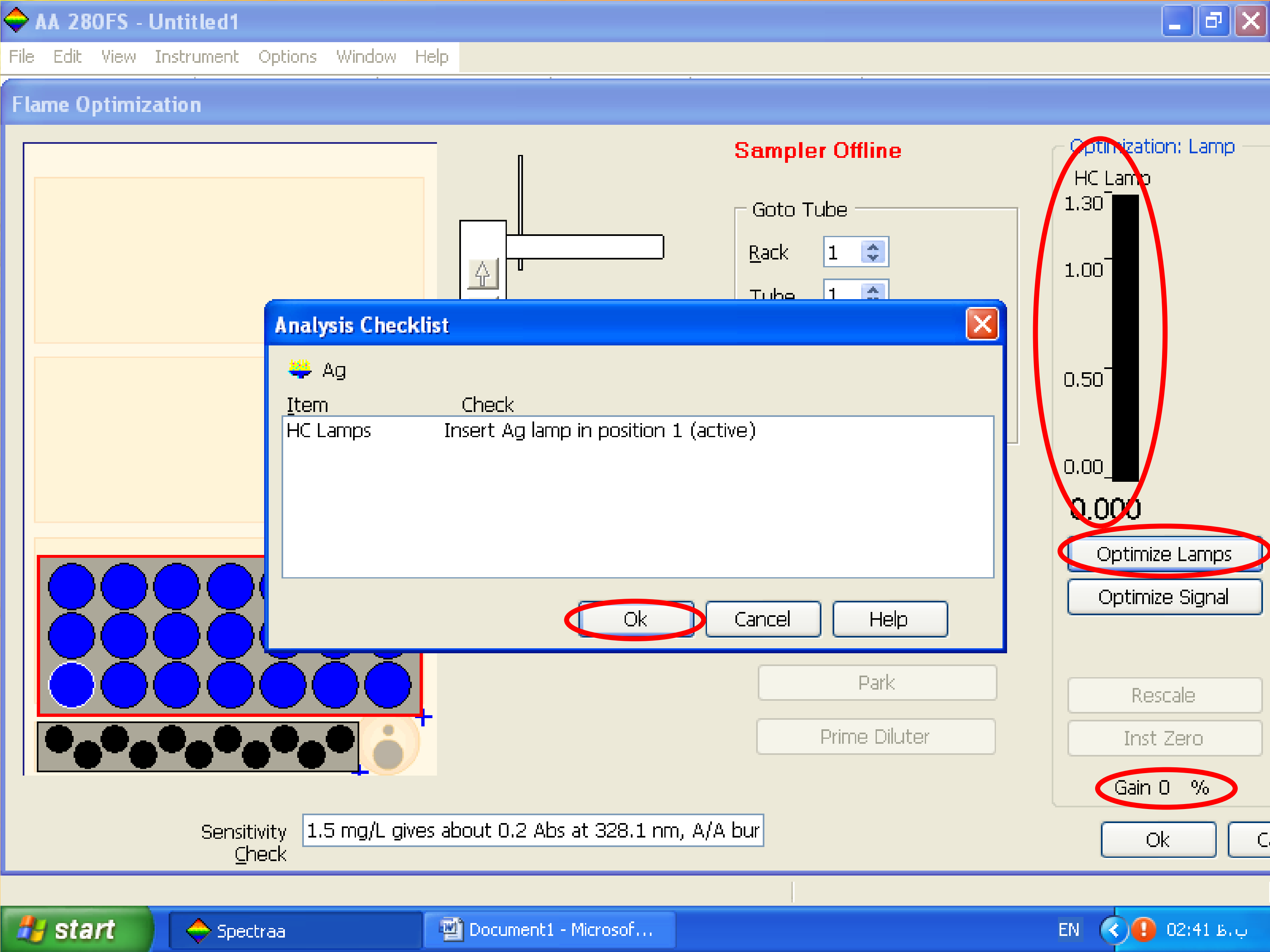
Ag

Method 1 of 1
Absorbance
Integrate 5.0 s

Concentration
Air/Acetylene

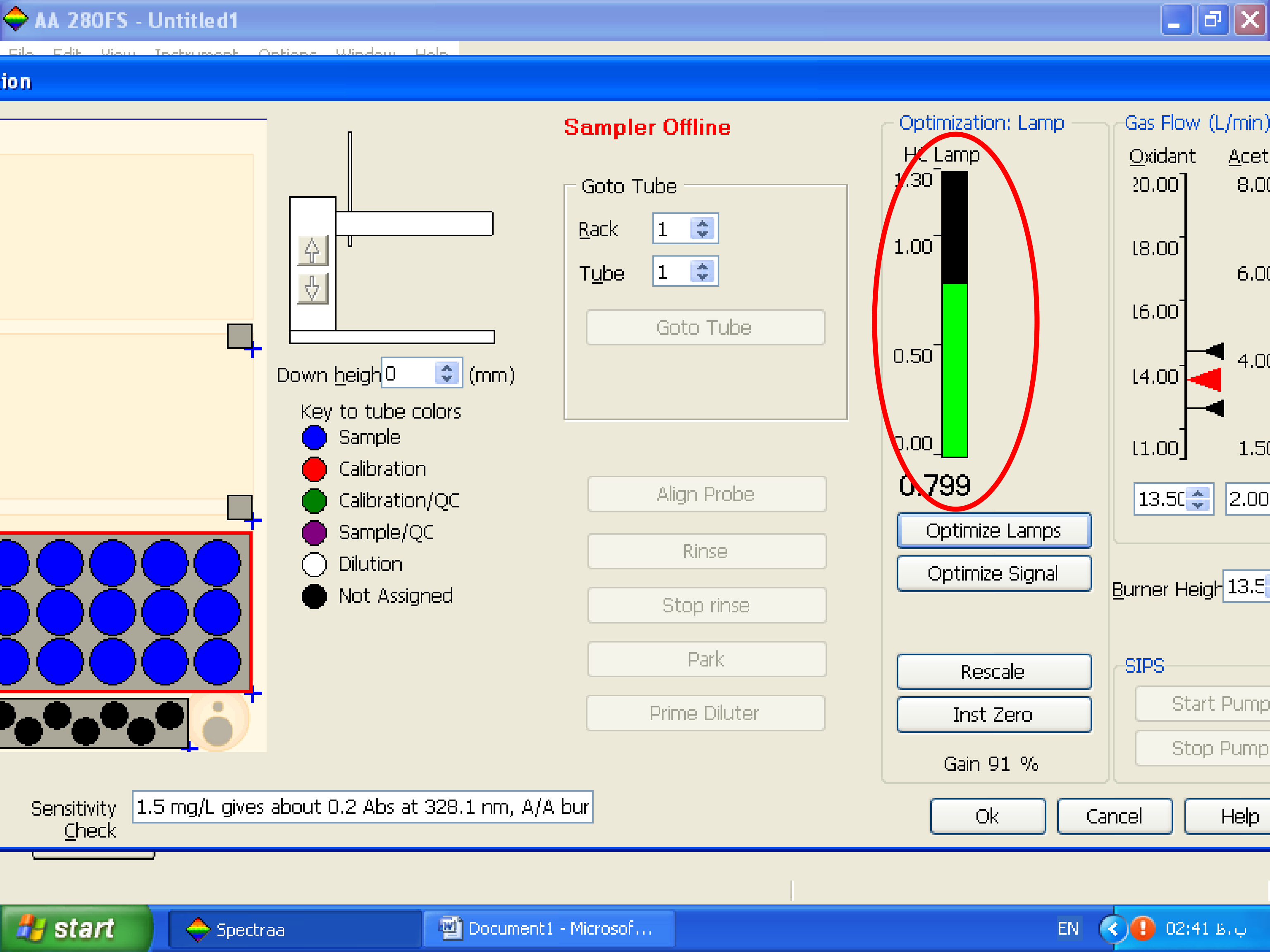
Lamp 1
Manual
BC Off

Row 1 of 50
Result rows: 0



| Item | Check |
|---|---|
| HC Lamps | Insert Ag lamp in position 1 (active) |

Sensitivity Check: 1.5 mg/L gives about 0.2 Abs at 328.1 nm, A/A bur



AA 280FS - Untitled1

**Sampler Offline**

Rack 1

Tube 1

Down heigh 0 (mm)

Key to tube colors
- ● Sample
- ● Calibration
- ● Calibration/QC
- ● Sample/QC
- ○ Dilution
- ● Not Assigned

HC Lamp 0.799

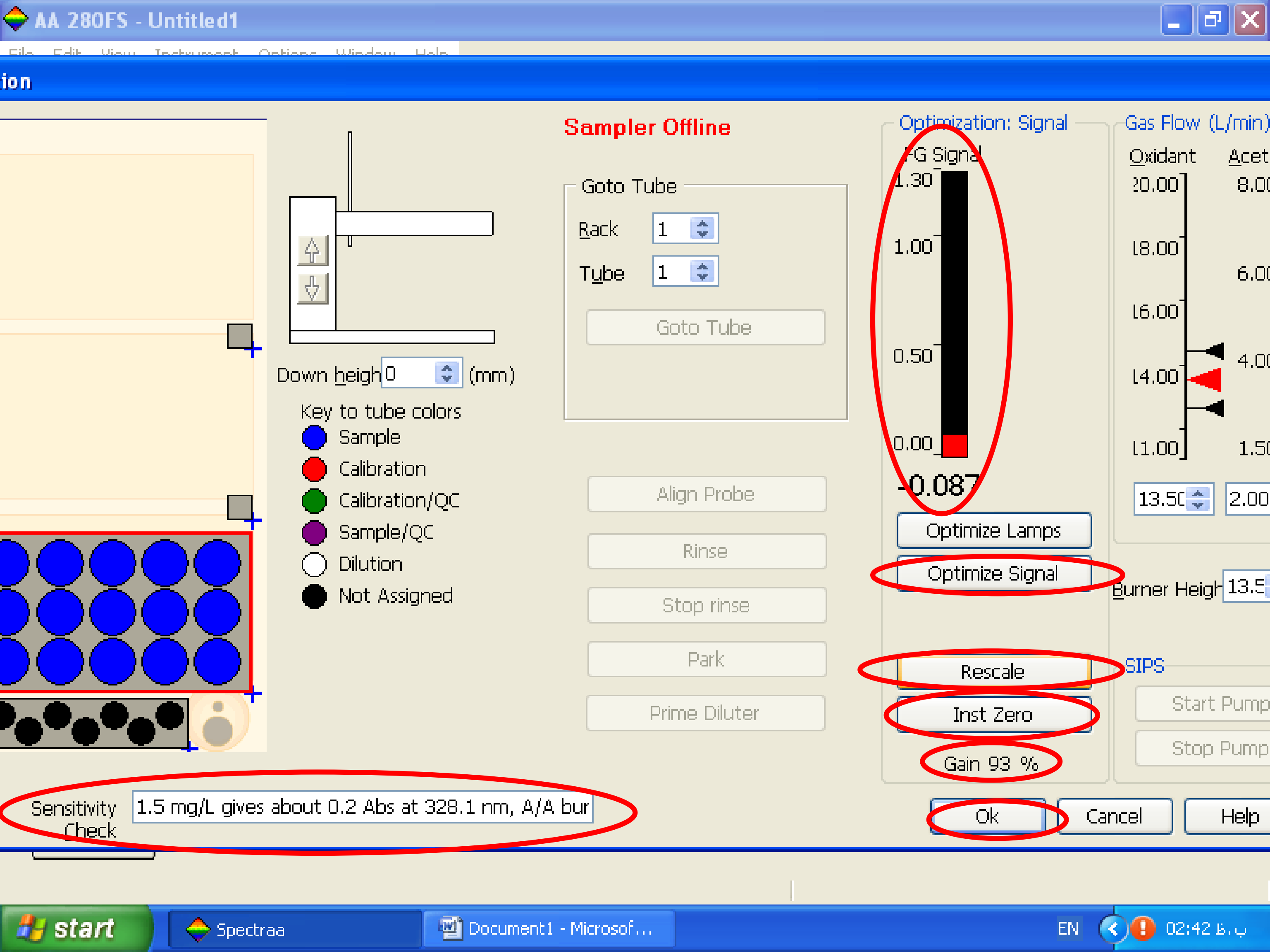
Oxidant 13.50

Acet 2.00

Burner Heigh 13.5

Gain 91 %

Sensitivity Check: 1.5 mg/L gives about 0.2 Abs at 328.1 nm, A/A bur



AA 280FS - Untitled1

Sampler Offline

Rack 1

Tube 1

Down heigh 0 (mm)

Optimization: Signal

G Signal -0.087

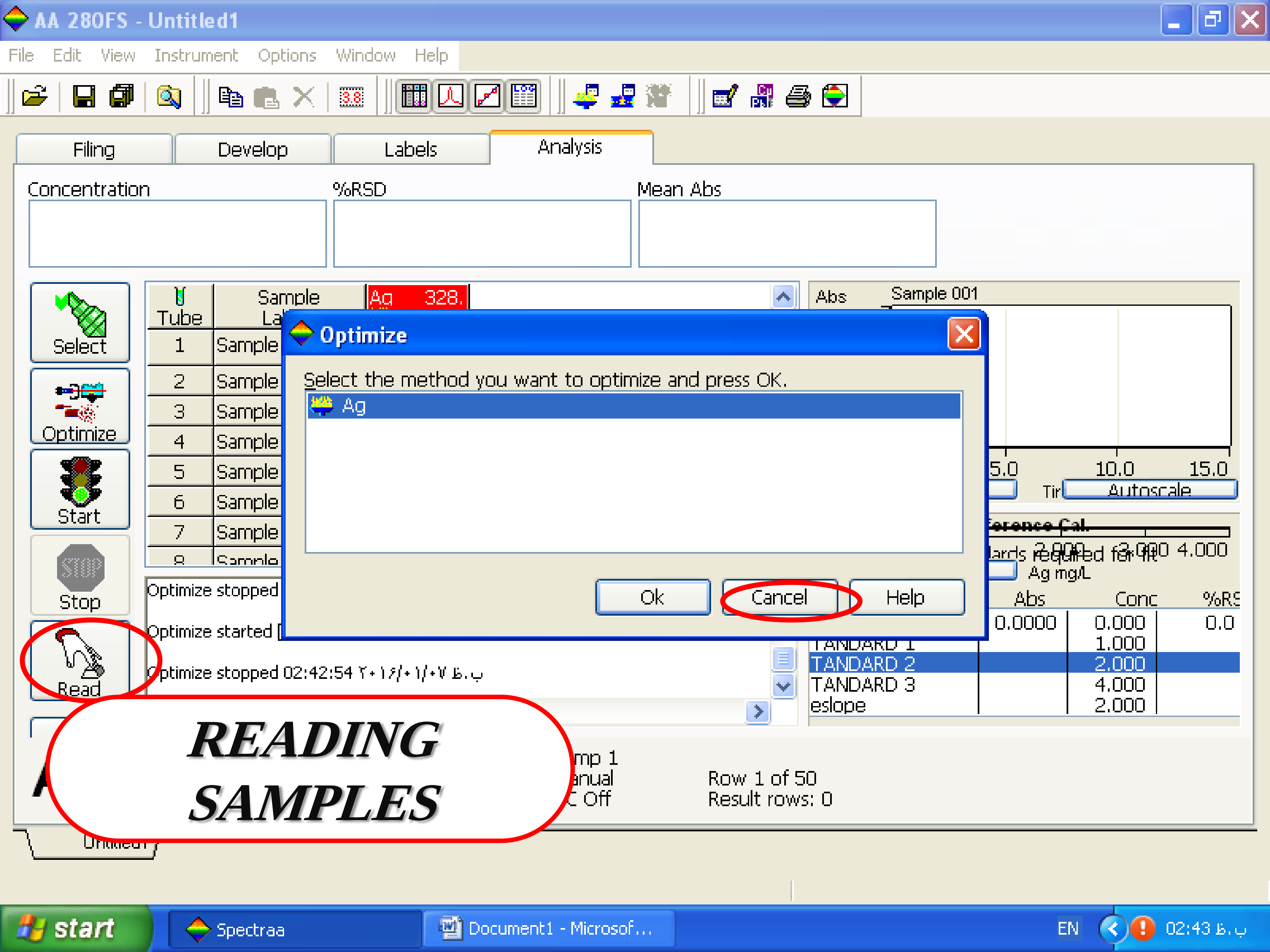
Oxidant 13.50

Acet 2.00

Burner Heigh 13.5

Gain 93 %

Sensitivity Check 1.5 mg/L gives about 0.2 Abs at 328.1 nm, A/A bur



| Tube | Sample Label |
|---|---|
| 1 | Sample |
| 2 | Sample |
| 3 | Sample |
| 4 | Sample |
| 5 | Sample |
| 6 | Sample |
| 7 | Sample |
| 8 | Sample |

**Optimize**

Select the method you want to optimize and press OK.

Ag

Ok | Cancel | Help

Optimize stopped

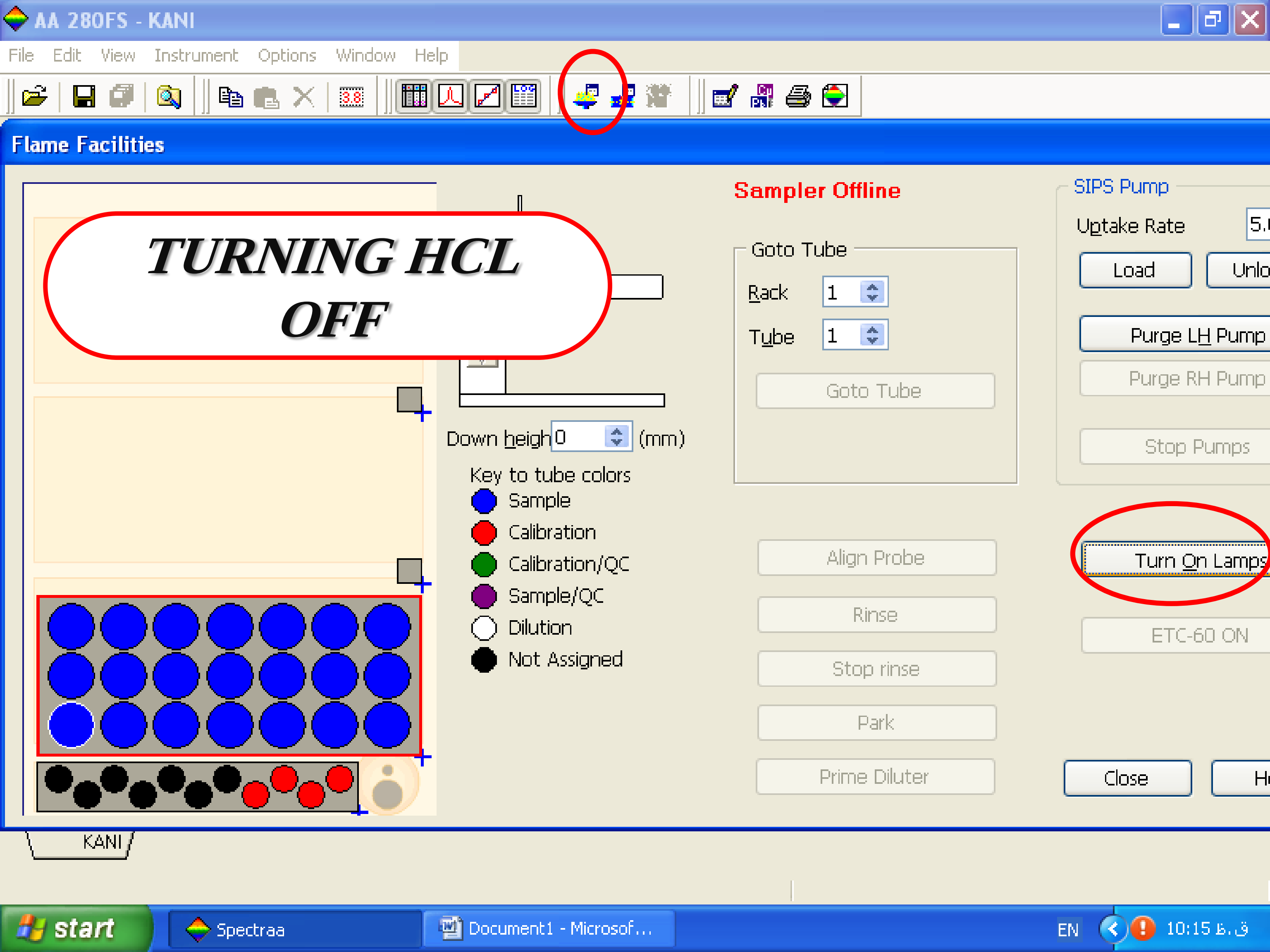
Optimize started

Optimize stopped 02:42:54 ۲۰۱۶/۰۱/۰۷ ب.ظ

| | Abs | Conc | %RS |
|---|---|---|---|
| | 0.0000 | 0.000 | 0.0 |
| TANDARD 1 | | 1.000 | |
| TANDARD 2 | | 2.000 | |
| TANDARD 3 | | 4.000 | |
| eslope | | 2.000 | |

Row 1 of 50
Result rows: 0

# READING SAMPLES



# TURNING HCL OFF

AA 280FS - KANI

Flame Facilities

Sampler Offline

Goto Tube

Rack 1

Tube 1

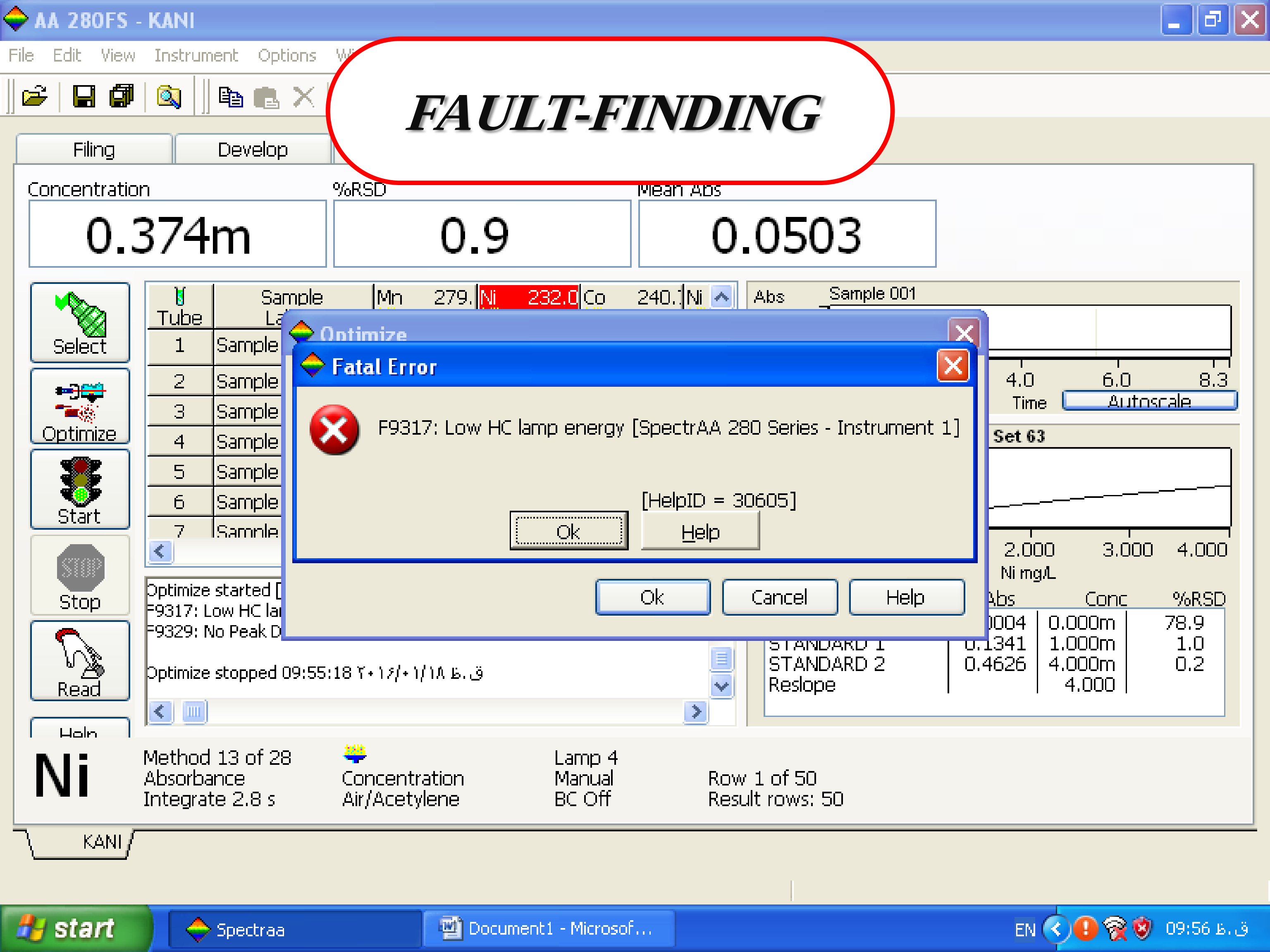
Down heigh 0 (mm)

Key to tube colors
- 🔵 Sample
- 🔴 Calibration
- 🟢 Calibration/QC
- 🟣 Sample/QC
- ○ Dilution
- ● Not Assigned

SIPS Pump

Uptake Rate 5.

# FAULT-FINDING



Concentration: 0.374m

%RSD: 0.9

Mean Abs: 0.0503

| Tube | Sample La | Mn 279. | Ni 232.0 | Co 240.1 | Ni |
|---|---|---|---|---|---|
| 1 | Sample | | | | |
| 2 | Sample | | | | |
| 3 | Sample | | | | |
| 4 | Sample | | | | |
| 5 | Sample | | | | |
| 6 | Sample | | | | |
| 7 | Sample | | | | |

F9317: Low HC lamp energy [SpectrAA 280 Series - Instrument 1]

[HelpID = 30605]

```
Optimize started [
F9317: Low HC la
F9329: No Peak D

Optimize stopped 09:55:18 ۲۰۱۴/۰۱/۱۸ ق.ظ
```

Abs Sample 001

4.0 6.0 8.3

Set 63

2.000 3.000 4.000

| | Abs | Conc | %RSD |
|---|---|---|---|
| | 0004 | 0.000m | 78.9 |
| STANDARD 1 | 0.1341 | 1.000m | 1.0 |
| STANDARD 2 | 0.4626 | 4.000m | 0.2 |
| Reslope | | 4.000 | |

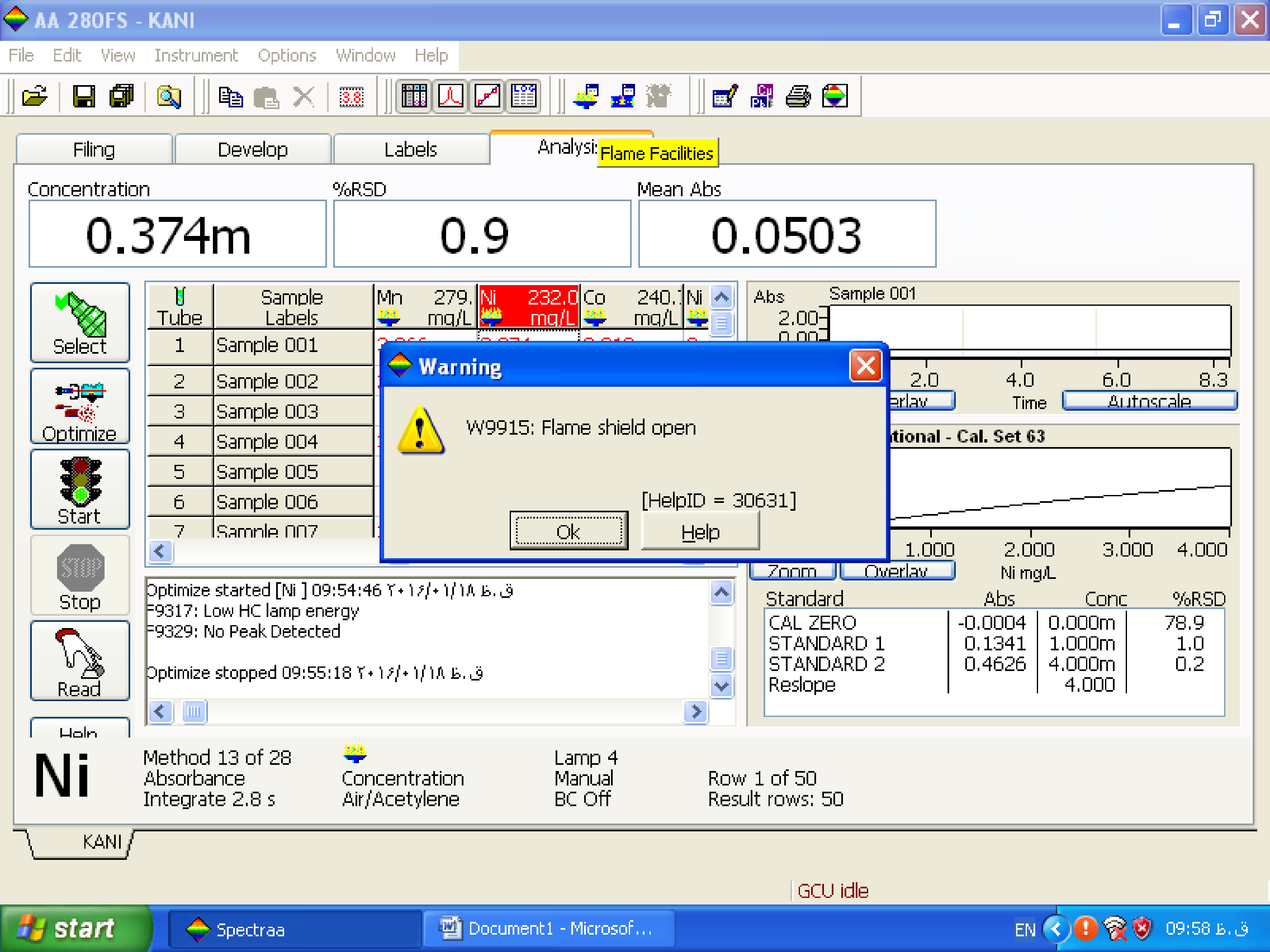
**Ni**

Method 13 of 28
Absorbance
Integrate 2.8 s

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 50
Result rows: 50



Concentration: 0.374m

%RSD: 0.9

Mean Abs: 0.0503

| Tube | Sample Labels | Mn 279. mg/L | Ni 232.0 mg/L | Co 240.1 mg/L | Ni |
|---|---|---|---|---|---|
| 1 | Sample 001 | | | | |
| 2 | Sample 002 | | | | |
| 3 | Sample 003 | | | | |
| 4 | Sample 004 | | | | |
| 5 | Sample 005 | | | | |
| 6 | Sample 006 | | | | |
| 7 | Sample 007 | | | | |

**Warning**

W9915: Flame shield open

[HelpID = 30631]

Ok Help

```
Optimize started [Ni ] 09:54:46 ۲۰۱۴/۰۱/۱۸ ق.ظ
F9317: Low HC lamp energy
F9329: No Peak Detected

Optimize stopped 09:55:18 ۲۰۱۴/۰۱/۱۸ ق.ظ
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | -0.0004 | 0.000m | 78.9 |
| STANDARD 1 | 0.1341 | 1.000m | 1.0 |
| STANDARD 2 | 0.4626 | 4.000m | 0.2 |
| Reslope | | 4.000 | |

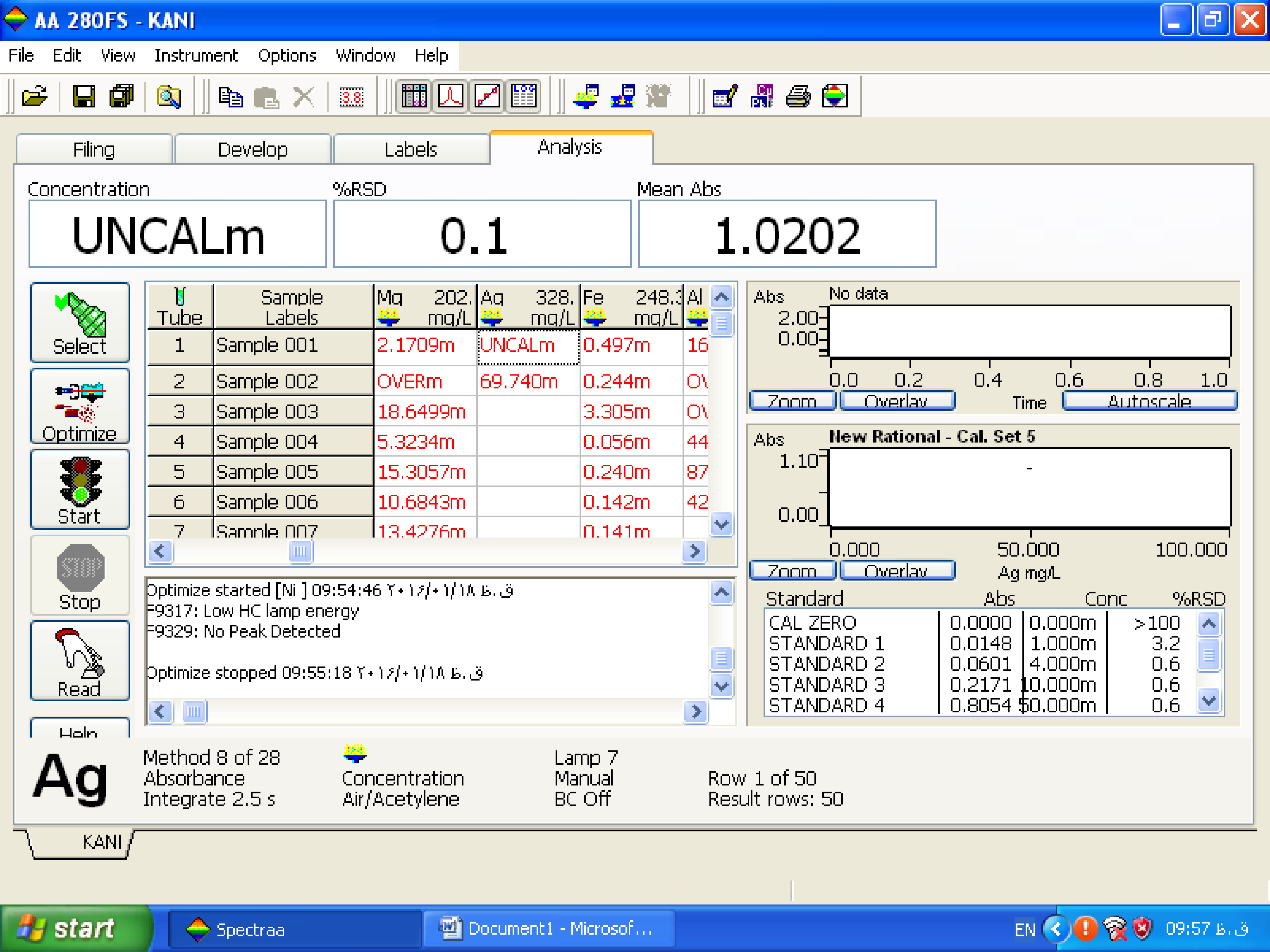
Ni

Method 13 of 28
Absorbance
Integrate 2.8 s

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 50
Result rows: 50



Concentration: UNCALm

%RSD: 0.1

Mean Abs: 1.0202

| Tube | Sample Labels | Mg 202. mg/L | Ag 328. mg/L | Fe 248.3 mg/L | Al |
|---|---|---|---|---|---|
| 1 | Sample 001 | 2.1709m | UNCALm | 0.497m | 16 |
| 2 | Sample 002 | OVERm | 69.740m | 0.244m | O\ |
| 3 | Sample 003 | 18.6499m | | 3.305m | O\ |
| 4 | Sample 004 | 5.3234m | | 0.056m | 44 |
| 5 | Sample 005 | 15.3057m | | 0.240m | 87 |
| 6 | Sample 006 | 10.6843m | | 0.142m | 42 |
| 7 | Sample 007 | 13.4276m | | 0.141m | |

```
Optimize started [Ni ] 09:54:46 ۲۰۱۶/۰۱/۱۸ ق.ظ
F9317: Low HC lamp energy
F9329: No Peak Detected

Optimize stopped 09:55:18 ۲۰۱۶/۰۱/۱۸ ق.ظ
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0000 | 0.000m | >100 |
| STANDARD 1 | 0.0148 | 1.000m | 3.2 |
| STANDARD 2 | 0.0601 | 4.000m | 0.6 |
| STANDARD 3 | 0.2171 | 10.000m | 0.6 |
| STANDARD 4 | 0.8054 | 50.000m | 0.6 |

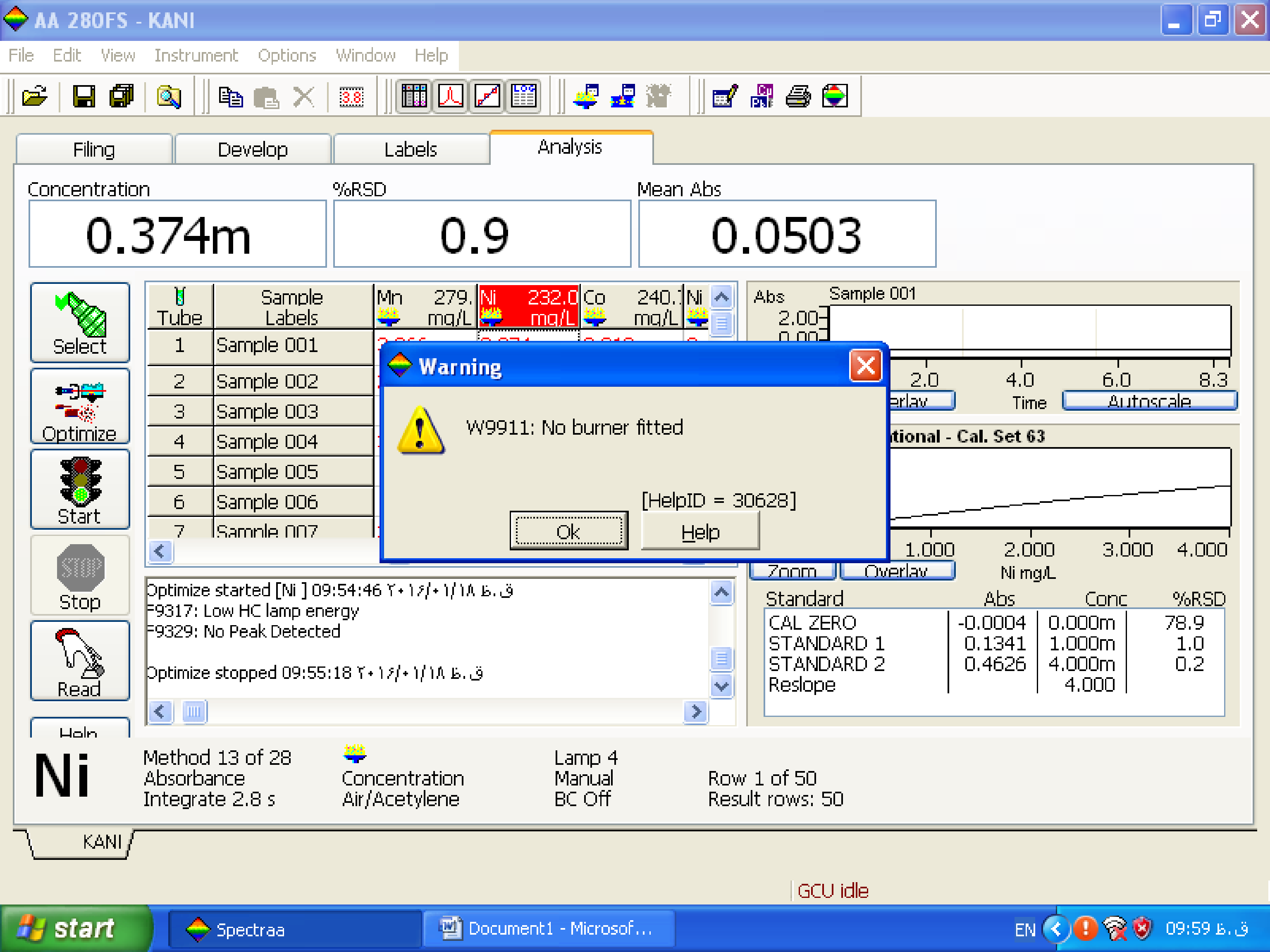
**Ag**

Method 8 of 28
Absorbance
Integrate 2.5 s

Concentration
Air/Acetylene

Lamp 7
Manual
BC Off

Row 1 of 50
Result rows: 50



Concentration: 0.374m

%RSD: 0.9

Mean Abs: 0.0503

| Tube | Sample Labels | Mn 279. mg/L | Ni 232.0 mg/L | Co 240.1 mg/L | Ni |
|---|---|---|---|---|---|
| 1 | Sample 001 | | | | |
| 2 | Sample 002 | | | | |
| 3 | Sample 003 | | | | |
| 4 | Sample 004 | | | | |
| 5 | Sample 005 | | | | |
| 6 | Sample 006 | | | | |
| 7 | Sample 007 | | | | |

**Warning**

W9911: No burner fitted

[HelpID = 30628]

Ok Help

```
Optimize started [Ni ] 09:54:46 ۲۰۱۶/۰۱/۱۸ ق.ظ
F9317: Low HC lamp energy
F9329: No Peak Detected

Optimize stopped 09:55:18 ۲۰۱۶/۰۱/۱۸ ق.ظ
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | -0.0004 | 0.000m | 78.9 |
| STANDARD 1 | 0.1341 | 1.000m | 1.0 |
| STANDARD 2 | 0.4626 | 4.000m | 0.2 |
| Reslope | | 4.000 | |

Ni

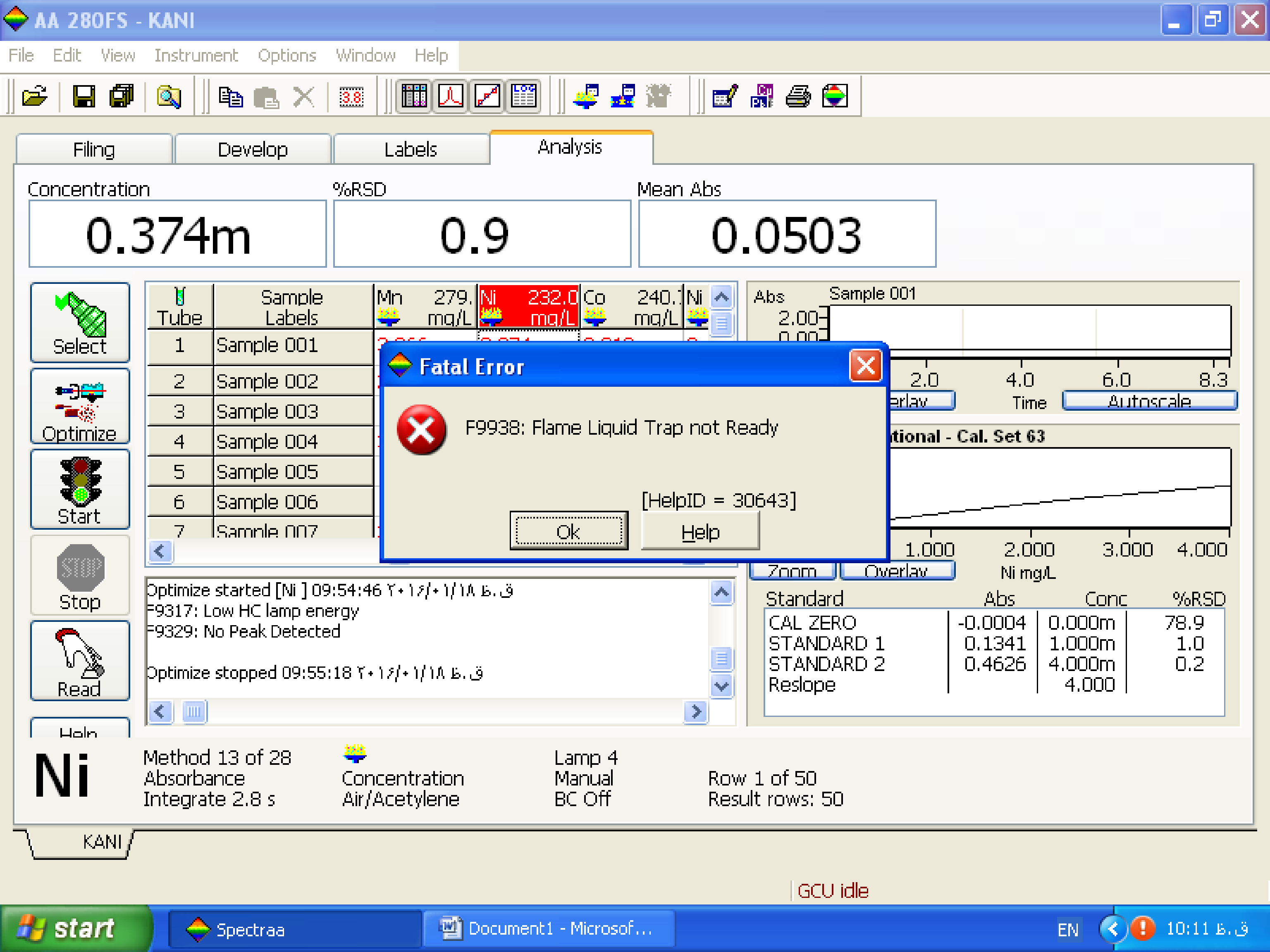
Method 13 of 28
Absorbance
Integrate 2.8 s

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 50
Result rows: 50

GCU idle



Concentration: 0.374m

%RSD: 0.9

Mean Abs: 0.0503

| Tube | Sample Labels |
|---|---|
| 1 | Sample 001 |
| 2 | Sample 002 |
| 3 | Sample 003 |
| 4 | Sample 004 |
| 5 | Sample 005 |
| 6 | Sample 006 |
| 7 | Sample 007 |

**Fatal Error**

F9938: Flame Liquid Trap not Ready

[HelpID = 30643]

```
Optimize started [Ni ] 09:54:46 ۲۰۱۶/۰۱/۱۸ ق.ظ
F9317: Low HC lamp energy
F9329: No Peak Detected

Optimize stopped 09:55:18 ۲۰۱۶/۰۱/۱۸ ق.ظ
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | -0.0004 | 0.000m | 78.9 |
| STANDARD 1 | 0.1341 | 1.000m | 1.0 |
| STANDARD 2 | 0.4626 | 4.000m | 0.2 |
| Reslope | | 4.000 | |

Ni

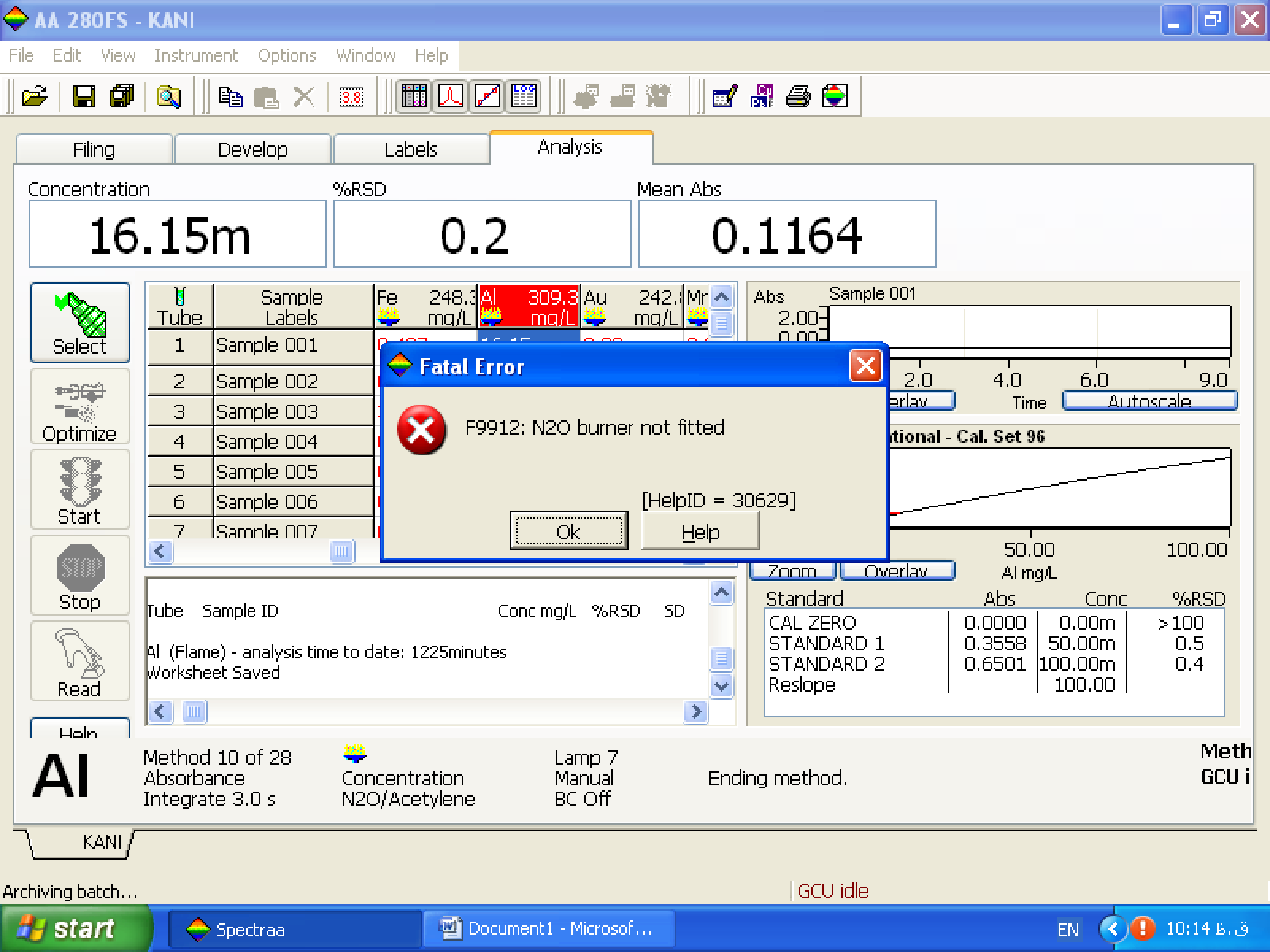
Method 13 of 28
Absorbance
Integrate 2.8 s

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 50
Result rows: 50

GCU idle



Concentration: 16.15m

%RSD: 0.2

Mean Abs: 0.1164

| Tube | Sample Labels | Fe 248.3 mg/L | Al 309.3 mg/L | Au 242. mg/L | Mr |
|---|---|---|---|---|---|
| 1 | Sample 001 | | | | |
| 2 | Sample 002 | | | | |
| 3 | Sample 003 | | | | |
| 4 | Sample 004 | | | | |
| 5 | Sample 005 | | | | |
| 6 | Sample 006 | | | | |
| 7 | Sample 007 | | | | |

**Fatal Error**

F9912: N2O burner not fitted

[HelpID = 30629]

Ok Help

Tube Sample ID Conc mg/L %RSD SD

Al (Flame) - analysis time to date: 1225minutes
Worksheet Saved

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0000 | 0.00m | >100 |
| STANDARD 1 | 0.3558 | 50.00m | 0.5 |
| STANDARD 2 | 0.6501 | 100.00m | 0.4 |
| Reslope | | 100.00 | |

Al

Method 10 of 28
Absorbance
Integrate 3.0 s

Concentration
N2O/Acetylene

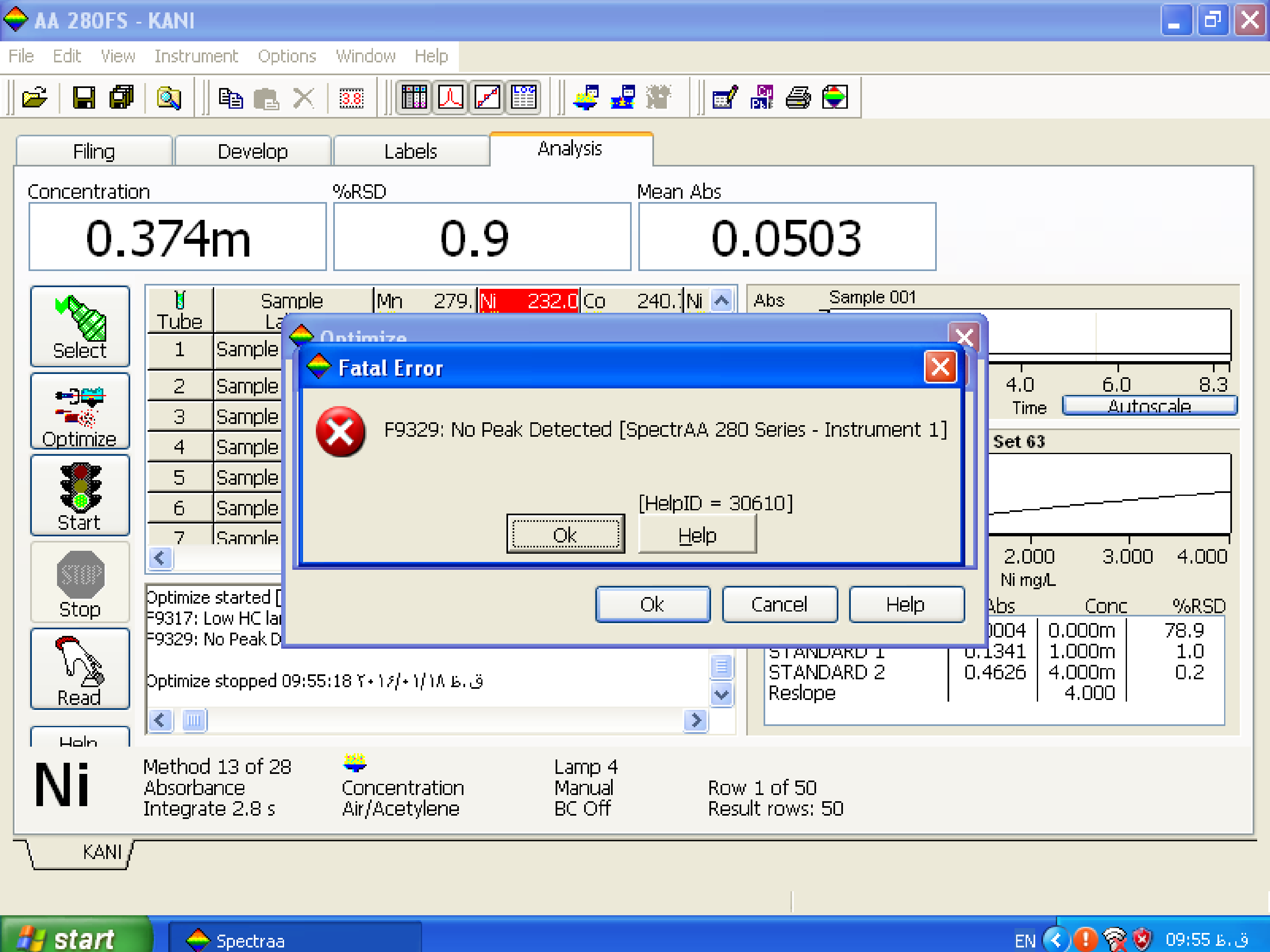
Lamp 7
Manual
BC Off

Ending method.

Meth
GCU i

Archiving batch...

GCU idle



Concentration: 0.374m

%RSD: 0.9

Mean Abs: 0.0503

| Tube | Sample La | Mn 279. | Ni 232.0 | Co 240.1 | Ni |
|---|---|---|---|---|---|
| 1 | Sample | | | | |
| 2 | Sample | | | | |
| 3 | Sample | | | | |
| 4 | Sample | | | | |
| 5 | Sample | | | | |
| 6 | Sample | | | | |
| 7 | Sample | | | | |

**Fatal Error**

F9329: No Peak Detected [SpectrAA 280 Series - Instrument 1]

[HelpID = 30610]

Ok Help

```
Optimize started [
F9317: Low HC la
F9329: No Peak D

Optimize stopped 09:55:18 ۲۰۱۶/۰۱/۱۸ ق.ظ
```

Abs Sample 001

4.0 6.0 8.3

Time

Set 63

2.000 3.000 4.000

Ni mg/L

| | Abs | Conc | %RSD |
|---|---|---|---|
| | 0004 | 0.000m | 78.9 |
| STANDARD 1 | 0.1341 | 1.000m | 1.0 |
| STANDARD 2 | 0.4626 | 4.000m | 0.2 |
| Reslope | | 4.000 | |

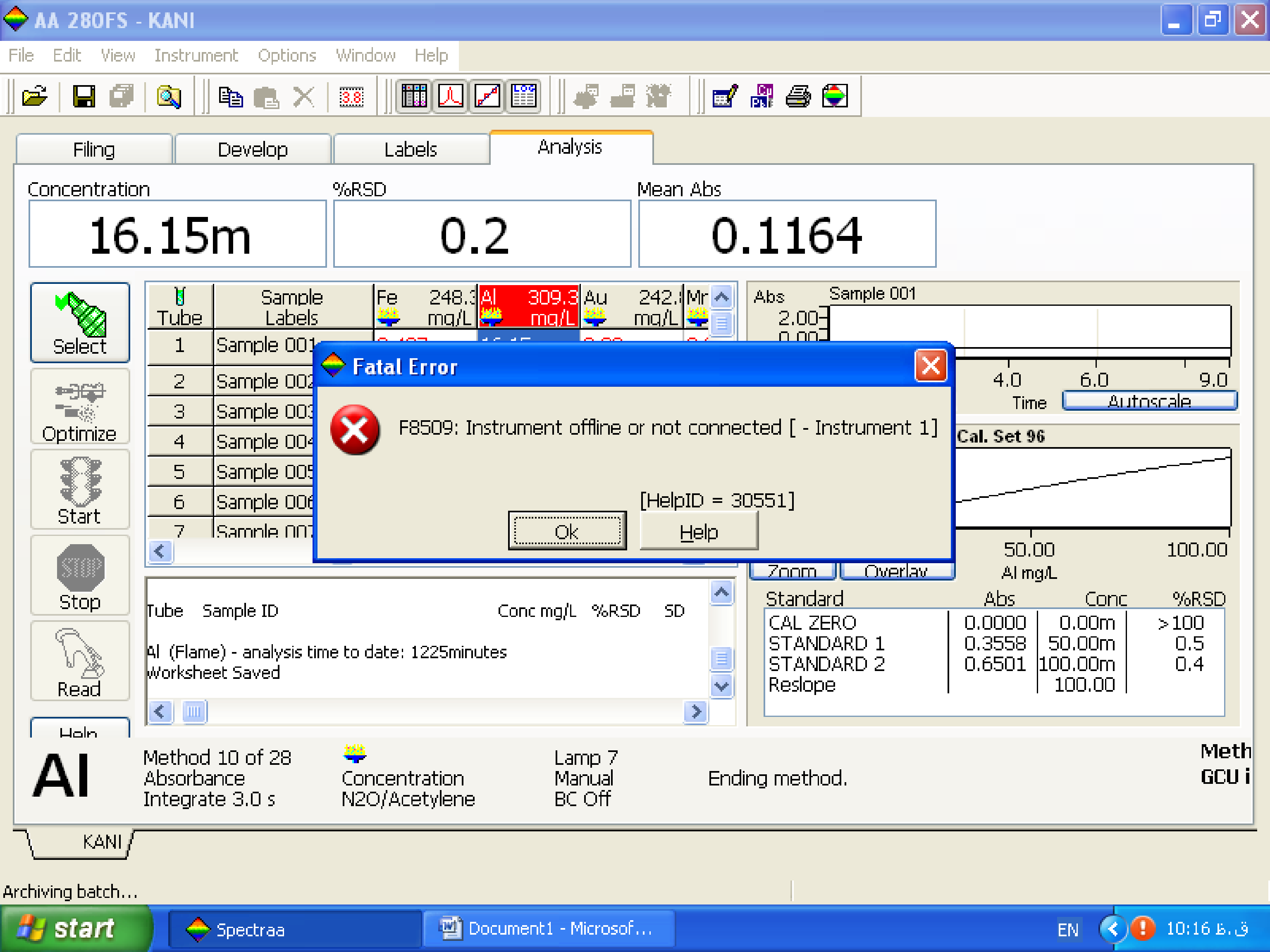
**Ni**

Method 13 of 28
Absorbance
Integrate 2.8 s

Concentration
Air/Acetylene

Lamp 4
Manual
BC Off

Row 1 of 50
Result rows: 50



Concentration: 16.15m

%RSD: 0.2

Mean Abs: 0.1164

| Tube | Sample Labels | Fe 248.3 mg/L | Al 309.3 mg/L | Au 242.8 mg/L | Mn |
|---|---|---|---|---|---|
| 1 | Sample 001 | | | | |
| 2 | Sample 002 | | | | |
| 3 | Sample 003 | | | | |
| 4 | Sample 004 | | | | |
| 5 | Sample 005 | | | | |
| 6 | Sample 006 | | | | |
| 7 | Sample 007 | | | | |

**Fatal Error**

F8509: Instrument offline or not connected [ - Instrument 1]

[HelpID = 30551]

```
Tube   Sample ID                      Conc mg/L  %RSD   SD

Al  (Flame) - analysis time to date: 1225minutes
Worksheet Saved
```

| Standard | Abs | Conc | %RSD |
|---|---|---|---|
| CAL ZERO | 0.0000 | 0.00m | >100 |
| STANDARD 1 | 0.3558 | 50.00m | 0.5 |
| STANDARD 2 | 0.6501 | 100.00m | 0.4 |
| Reslope | | 100.00 | |

**Al**

Method 10 of 28
Absorbance
Integrate 3.0 s

Concentration
N2O/Acetylene

Lamp 7
Manual
BC Off

Ending method.

**Meth**
**GCU i**

Archiving batch...