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Running the 3100

Operating Instructions



PERKIN ELMER

Model 3100
Atomic Absorption Spectrophotometer

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0993-8434

Running the 3100 Atomic Absorption Spectrometer

Rev. August 1990

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ATTENTION

IMPORTANT: USE OF DRY AIR

To avoid possible damage to the Model 3100 Burner Control, the following precautions must be observed:

a. Only dry air may be used. Severe damage to the pneumatics will result unless the air supply is dry.

b. Compressor storage tanks should be bled (for water) at least weekly.

c. The air supply to the Burner Control should be turned off by an external valve whenever the Burner Control is not in use - even for a brief period.

IMPORTANT: Servicing of the Model 3100, including replacement or aligning of Background Corrector Sources, should only be performed by a Perkin-Elmer Service Representative. Servicing of the instrument by a person other than a Perkin-Elmer Service Representative will render the warranty null and void.

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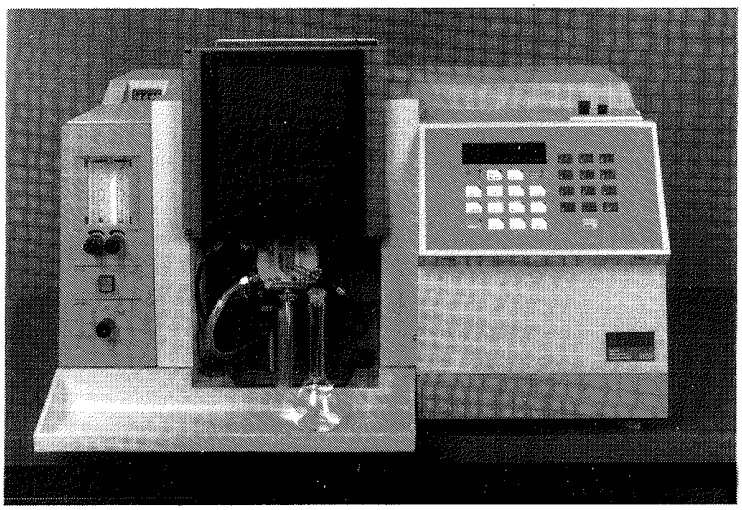
Introduction to the 3100 1

A Look at the System Components

The Model 3100 system consists of a Model 3100 Atomic Absorption Spectrometer and the following optional accessories:

- MHS-10 Mercury/Hydride System
- AS-51 Flame Autosampler
- AS-90 Flame Autosampler
- Background Correction Accessory
- Printer

Model 3100 Spectrometer



The Model 3100 Atomic Absorption Spectrometer is designed for use with flame absorption, flame emission, and mercury/hydride atomic absorption techniques.

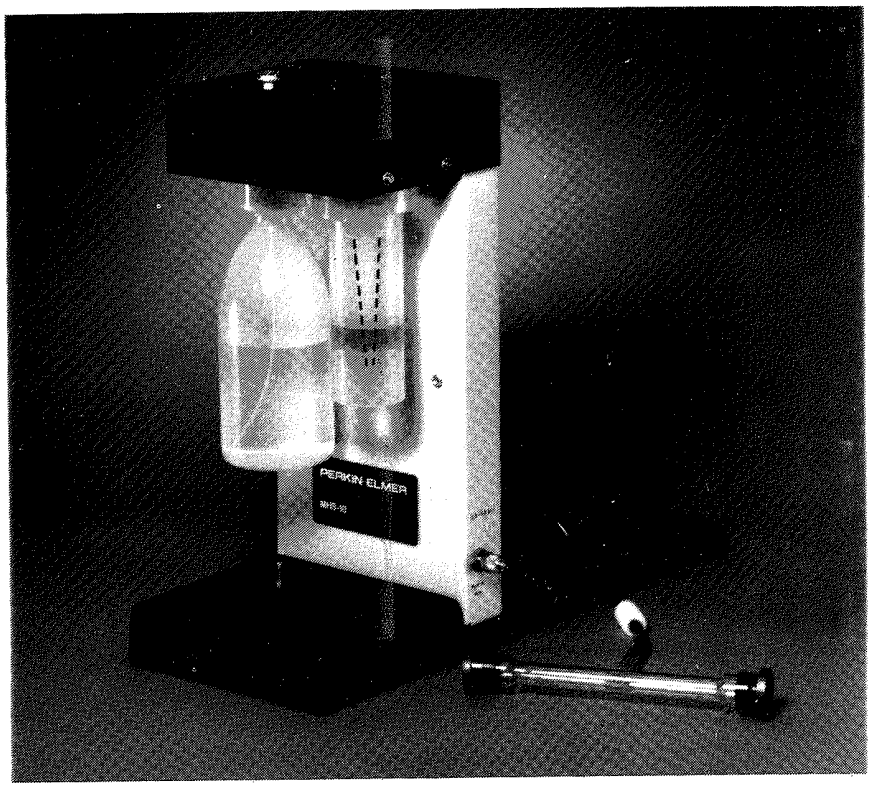
A high efficiency burner system offers both a flow spoiler and impact bead for optimal performance with all sample types.

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Built-in gas controls with safety interlocks, automatic pushbutton ignition, and automatic switching to nitrous oxide-acetylene flames simplify operation while insuring operator safety.

A built-in control panel with two-line alphanumeric display, numeric keypad, and function keys is used to access the comprehensive programming which is hard wired into the built-in microprocessor.

MHS-10 Mercury Hydride System



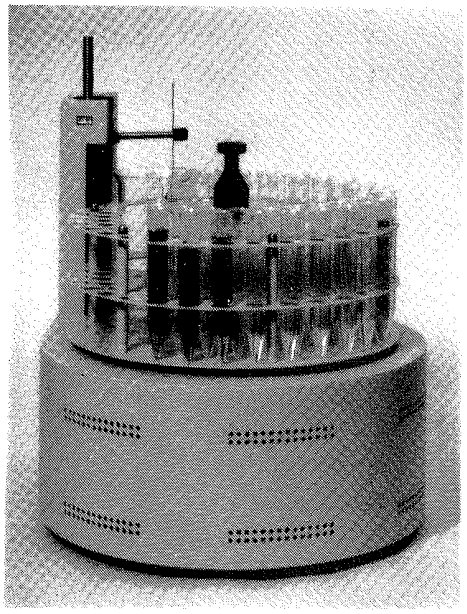
The MHS-10 Mercury/Hydride System is a manually operated accessory that includes an analyzer assembly and quartz cell assembly. The reductant is manually injected into the reaction vessel, and a flame-heated quartz cell is used for thermal dissociation of the resulting hydride.

The analyzer assembly is freestanding and is located next to the sample compartment of the 3100 Spectrometer. This assembly includes a reaction flask, a reservoir for the reducing agent, and all pneumatic components for control of the carrier gas and transport of metallic vapors to the quartz cell.

The cell assembly consists of a quartz cell and a mount. The mount installs on the 3100 Ignitor Assembly, positioned above the single slot burner head, permitting the quartz cell to be heated in the flame.

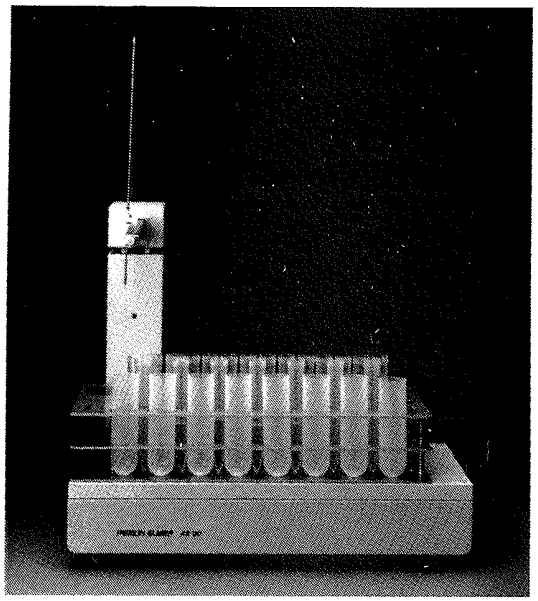
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AS-51 Autosampler



The AS-51 Autosampler, used for flame analyses, has a removable tray and can hold fifty-four 15 mL test or centrifuge tubes plus a 400 mL rinse vessel. The AS-51 is controlled by the 3100 software through the optional IEEE-488 interface.

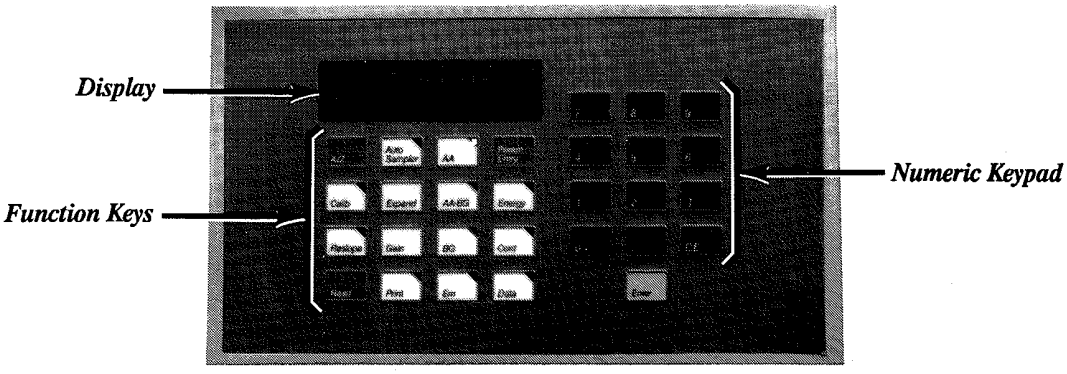
AS-90 Autosampler



The AS-90 Autosampler, used for flame analyses, can accommodate a 250 mL rinse vessel, eight 50 mL standard vessels, and ninety eight 15 mL sample vessels. The AS-90 is controlled by the 3100 software through the optional IEEE-488 interface.

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About The Control Panel



The Control Panel contains a 2-line by 20-character alphanumeric vacuum fluorescent display, function keys, and a numeric keypad.

The display not only shows analytical results, it also provides helpful information when errors are encountered, when setting up a method, aligning lamps, or optimizing wavelength settings.

Pressing a function key initiates the action associated with that key.

Function Key Descriptions

Param Entry Key

Commands the system to enter the Parameter Entry Mode. When this key is pressed, the first parameter screen appears.

```
LAMP CUR. (0-50mA):
        0.0
```

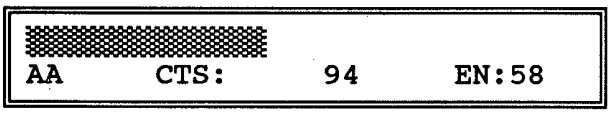
The 3100 display then guides you through the entry of the various parameters.

The parameters and their screens are described in Chapter 2, Setting Up Parameters.

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Energy Key

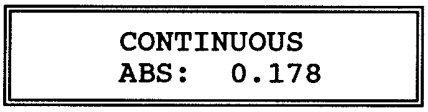
Displays the Energy screen.



This is a graphics screen used to facilitate optimizing a wavelength setting. This screen is also used when setting PMT (photomultiplier tube) voltages with the **[Gain]** key.

Cont Key

Displays the Continuous screen on which the measured absorbance is continuously updated at the rate set by the Integration Time parameter. For example,



The Continuous screen may be displayed at any time.

Data Key

Commands the system to enter the Data Acquisition Mode. Activates the **[A/Z]**, **[Calib]**, **[Reslope]**, and **[Read]** keys.

AA Key

Commands the system to enter the standard AA Flame Analysis Mode. In this mode, absorbance measurements are made without background correction.

AA-BG Key

Commands the system to enter the AA-BG mode.

> **NOTE:** This mode is available only if the optional background correction accessory is installed.

In this mode the system measures the total absorbance of the analyte and background, then measures the absorbance of the background, then subtracts the background from the total to give the net absorbance due to the sample.

Only the corrected signal is reported.

BG Key

> **NOTE:** This mode is available only if the optional background correction accessory is installed.

Pressing this key displays the background absorbance.

Em Key

Commands the system to enter the Flame Emission Analysis Mode.

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Auto Sampler Key

Commands the system to activate the autosampler and start a run.

NOTE: This key is active only if an IEEE-488 interface board has been installed, an autosampler attached to it, and the control switch (on the rear of the instrument), set to the down position (see Figure 1-2). For additional information please refer to Installing an Autosampler in Chapter 14, Installation Procedures.

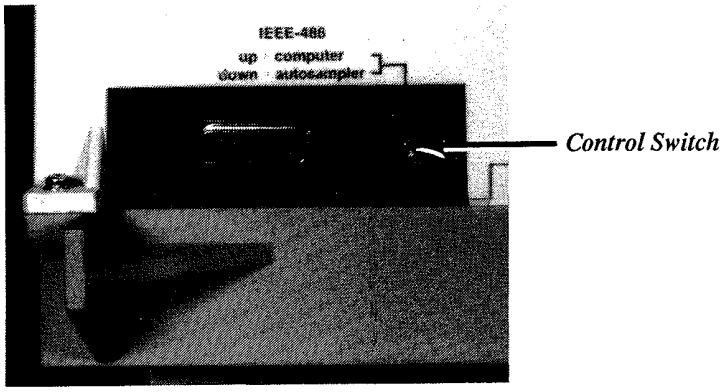


Figure 1-1. IEEE-488 Control Switch

Expand Key

Pressing this key displays the Expansion screen.

```
EXPANSION
  1.00
```

Entering a new value overrides any computed calibration curve. The system will multiply every subsequent absorbance reading by this value. Pressing **[1]** followed by pressing **[Expand]** gives you the original absorbance reading (see Changing the Expansion Factor in Chapter 17, Calibration).

Gain Key

Brings the bar chart on the Energy screen on scale. It is used in AA or AA-BG mode to automatically set the PMT (photomultiplier tube) voltage based on the lamp intensity. Also used in Flame Emission mode to set the PMT voltage based on the intensity of the highest standard. This key can also be used to set the PMT voltage manually.

Print Key

Activates a printer, if connected. In the Parameter Entry mode, pressing **[Print]** prints the operating parameters in effect. In the Data Acquisition mode, pressing **[Print]** prints the results for each standard or sample after its last replicate is measured. The correlation coefficient and slope of the calibration curve will be printed after the last replicate has been run for each standard. You can also have a sample sequence number printed for each sample, if you wish. See below.

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Other printer functions:

- Sample sequence numbers. Entering a number, then pressing **[Print]**, causes the printer to print a sequence number, from 1 to 9999, each time a sample is run, starting with the number entered.

  Examples: Pressing **[1] [0] [Print]** initiates sequence numbers starting at 10. **[1] [Print]** starts sequence numbers at 1; **[9] [7] [Print]** starts sequence numbers at 97.

  The sequence number automatically increments each time a sample (not a standard, blank, or reslope) reading is taken.

- Form feed. Pressing **[.] [Print]** causes the printer to form feed.

- Line Feed. Pressing **[.]**, a number, then **[Print]** causes the printer to line feed that number of times.

  Example: Pressing **[.] [1] [0] [Print]** causes the printer to line feed 10 times.

A/Z Key

Instructs the system to perform an autozero, in which the current absorbance reading is defined as 0.000 and all subsequent values are calculated relative to that baseline value.

Calib Key

Begins a measurement cycle for a standard.

Reslope Key

Begins a measurement cycle for a reslope standard.

Read Key

Begins a measurement cycle for a sample. The Read function also allows continuous readings to be taken in either the Absorbance or Concentration modes. To obtain continuous readings, enter **[0] [Read]**. When you no longer need continuous readings, press **[Read]** again.

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Setting Up Parameters 2

Overview

The 3100 software allows you to set up a single set of operating parameters called a method. To specify individual parameters (i.e., to set up a method) you enter the Parameter Entry Mode by pressing: **[Param Entry]**. The 3100 then guides you through the entry of the required information using the display.

To enter a parameter, type in the value using the numeric keypad, then press **[Enter]**. The next parameter screen appears. To accept a default value, press: **[Enter]**. To choose an option, type in the option number, then press: **[Enter]**.

The number of parameter screens varies depending on circumstances.

> **NOTE:** The allowable range of values that may be entered or numbers associated with options are shown in parentheses ( ) on each parameter screen. Default values (if any) appear on the bottom line.

> **NOTE:** When you turn off the instrument, parameters you have entered are erased.

Descriptions of the parameters, their associated screens, and instructions for specific screens follow. The numbers displayed with the screen titles correspond to those on the stick-on parameter template supplied and are shown between square brackets [ ].

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Parameter Descriptions

[1] Lamp Current

When you press **[Param Entry]**, the first parameter screen, Lamp Current, appears.

```
LAMP CUR. (0-50mA):
        0.0
```

Refer to the lamp label for the continuous lamp current to use for the lamp you have installed.

[2] Integration Time

```
INT. TIME (.1-60sec)
        0.10
```

NOTE: While the allowable range is 0.1 to 60 sec., typical values for normal flame analyses are 1 to 10 sec.

[3] Replicates

```
REPLICATES (1-99):
        01
```

The number of replicates you enter will be run for all samples and standards.

[4] Calibration Type

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

The 3100 allows three types of calibration: non-linear (NON LIN), linear (LIN), and method of additions (ADD). The default is non-linear. To select a calibration type, enter the number corresponding to the type of calibration desired, then press: **[Enter]**.

[5] AA Technique

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3): 1
```

Press: **[Enter]** if you are using flame. If you are using the Mercury/Hydride System, type 2 or 3 (usually 3), then press: **[Enter]**.

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[6] through [13] Standards 1-8

```
STD 1 (.0001-9999) :
      ------
```

On this screen you enter the concentration of the first standard. After pressing **[Enter]**, the screen for the second standard appears:

> **NOTE:** For all standards, the decimal point is automatically set based on the first standard entry.

```
STD 2 (.0001-9999) :
      ------
```

You can use up to eight standards, although three are usually adequate.

When you enter the concentration of a standard, the screen for the next standard appears.

If you press **[Enter]** without entering a standard concentration, the system skips over the remaining standard screens to the next parameter, Reslope Standard.

[14] Reslope Standard

```
RSLP (.0001-9999) :
      ------
```

If you want to run a Reslope Standard, type in its concentration, then press: **[Enter]**.

If you do not want to run a Reslope Standard, simply press: **[Enter]**.

In either case, the next screen that appears (Autosampler Type), depends on the position of the control switch on the IEEE-488 panel (see page 1-6, Autosampler Key) is attached. If you are not using an autosampler, the first parameter screen (Lamp Current) reappears.

[15] Autosampler Type

```
SELECT AUTOSAMPLER
AS51 (1)  AS90 (2):1
```

Enter the number corresponding to your autosampler, then press: **[Enter]**. One of two Last Sample Position screens appears.

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[16] Last Sample

If an AS-51 is selected on the previous screen, the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-54): 09
```

If an AS-90 is selected on the previous screen, the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-106): 09
```

In any event, type in the tray position which contains the last sample in your run, then press: **[Enter]**.

[17] Recalibrate

```
ASAMP RECAL POS:
__ __ __ __ __
```

Enter the position(s) before which the instrument is to be recalibrated by rerunning the standards. Up to five entries can be made. Each entry must consist of two digits. E.g., to enter position 9, press **[0] [9]**. The highest position number that can be entered is 99.

NOTE: Recalibration does not work for Method of Additions.

[18] A/Z Frequency

```
  A/Z FREQUENCY
ENTER (01-99) 99
```

The Autozero Frequency screen is specific for Method of Additions when using an autosampler.

This screen allows you to specify how often you want the system to aspirate the blank and perform an autozero.

For example, if you specify 1, the system will aspirate the initial blank, perform an Autozero, then repeat the blank and Autozero after each sample run (a Method of Additions sample run consists of an unspiked sample and spiked samples). If you specify 3, the system will aspirate the initial blank, perform an Autozero, then repeat the blank and Autozero after every three sample runs.

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Leaving the Parameter Entry Mode

Press: **[Energy]**, **[Cont]**, or **[Data]**.

Going Directly to a Parameter Screen

You can go to any parameter screen from the Energy, Continuous, or Data Modes by pressing the number of the parameter screen you wish to display, then pressing **[Param Entry]**. The numbers of the various screens are shown on the stick-on parameter template supplied (see below).

Stick-on Template

[1]	LAMP CURRENT
[2]	INTEGRATION TIME
[3]	REPLICATES
[4]	CALIBRATION TYPE
[5]	AA TECHNIQUE
[6]	STANDARD 1
[7]	STANDARD 2
[8]	STANDARD 3
[9]	STANDARD 4
[10]	STANDARD 5
[11]	STANDARD 6
[12]	STANDARD 7
[13]	STANDARD 8
[14]	RESLOPE STANDARD
[15]	AUTOSAMPLER TYPE
[16]	LAST SAMPLE
[17]	RECALIBRATE
[18]	A/Z FREQUENCY

Figure 2-1. Stick-on Parameter Template

NOTE: Certain screens may be unavailable depending on what has been previously entered in Parameter Entry. For example, the screens for standards 4 through 8 are unavailable if concentrations have been entered only for standards 1 and 2.

Printing the Parameters

* Press: **[Param Entry]** **[Print]**.

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Installing and Aligning Lamps 3

This chapter includes the following sections:

- Installing a Hollow Cathode Lamp
- Aligning a Hollow Cathode Lamp
- Installing an EDL Lamp
- Aligning an EDL in AA Mode
- Aligning an EDL in AA-BG Mode

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Installing a Hollow Cathode Lamp

Before you install a hollow cathode lamp, check the label for the Continuous Operating Lamp Current. Make a note of this value since you will have to enter it when you align the lamp.

There are two types of lamp labels you may encounter. These are illustrated below.

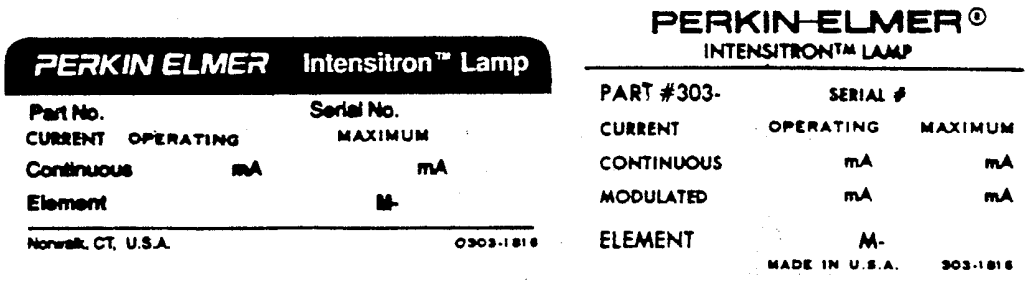


Figure 3-1. Hollow Cathode Lamp Labels

To install a hollow cathode lamp:

1. Locate the Lamp Alignment Screws on top of the instrument.
2. Make certain that the Lamp Alignment Screws are in the vertical position. If they are tilted, simply flip them into a vertical position.

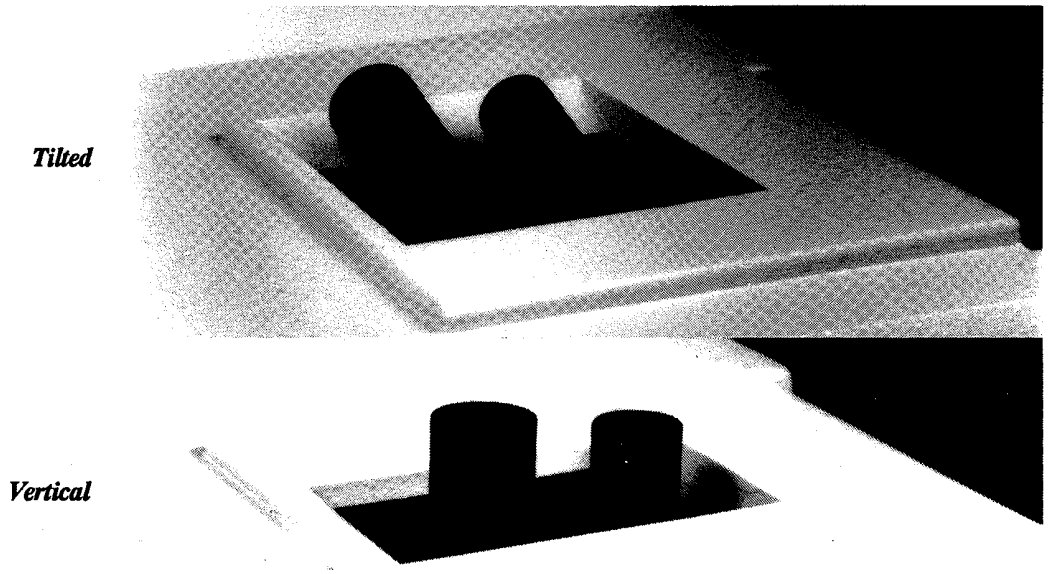


Figure 3-2. Lamp Alignment Screws

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3. Locate the lamp compartment on the right hand side of the instrument, near the front.

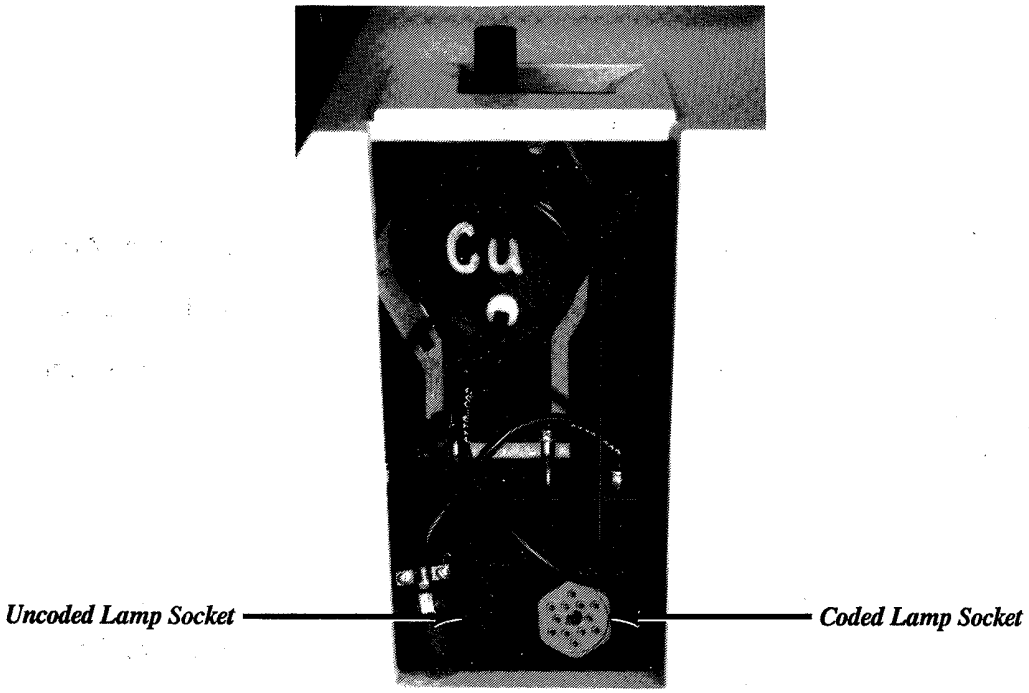


Figure 3-3. Lamp Compartment Side View

4. **Carefully** insert the lamp all the way, then pull it back about 5 mm or ¼ of an inch.

IMPORTANT: Be careful not to insert the lamp too forcefully to avoid damaging the front face of the lamp.

5. Connect the lamp plug to the appropriate socket type (see Figure 3-3).

NOTE: Two different lamp sockets are provided for your convenience, one for uncoded lamps (small socket) and one for coded lamps (larger socket). Only one lamp can be connected at a time.

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Aligning a Hollow Cathode Lamp

Carry out the following steps to align a lamp:

1 Set the wavelength*.

2 Set the slit width* and height**.

3 Set the parameters and turn on the lamp.

4 Maximize the energy.

* Obtain these values for the lamp being aligned from the AA Cookbook.

** For flame analysis the slit height is always set to the "High" position.

An example of how to align a copper lamp is given in the next section.

EXAMPLE: Aligning a Copper Lamp

1 Set the Wavelength

1. Locate the wavelength indicator on the top left side of the instrument.

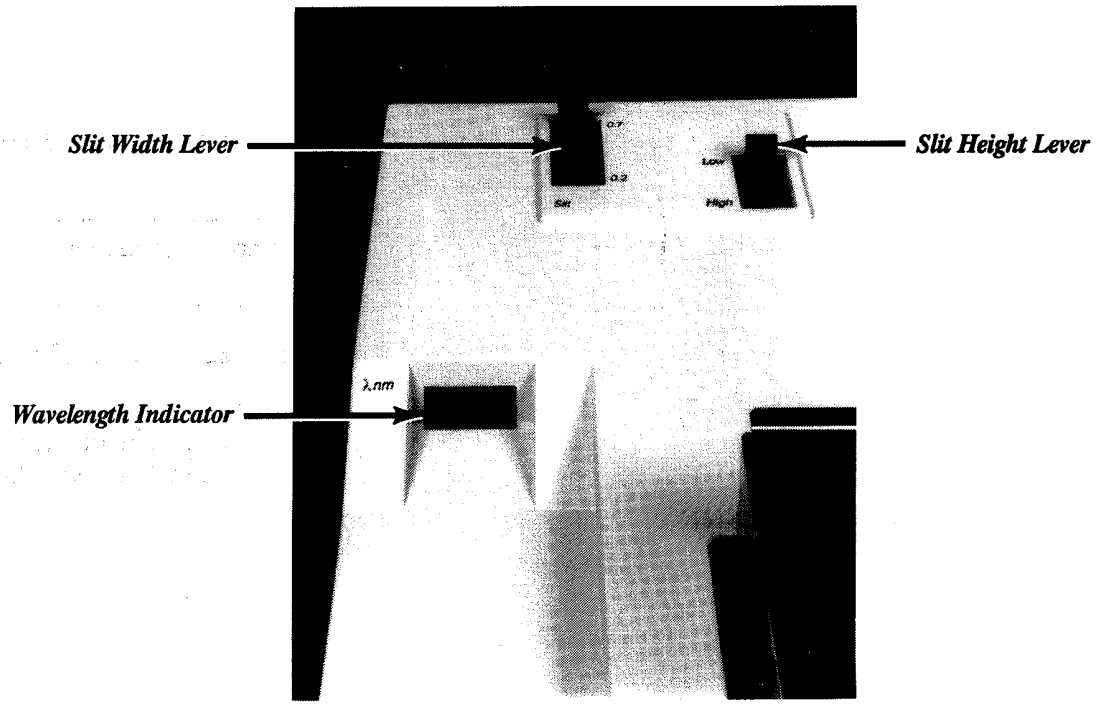


Figure 3-4. Wavelength Indicator and Slit Control Levers

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2. Locate the Wavelength Control Dial on the left side of the instrument.

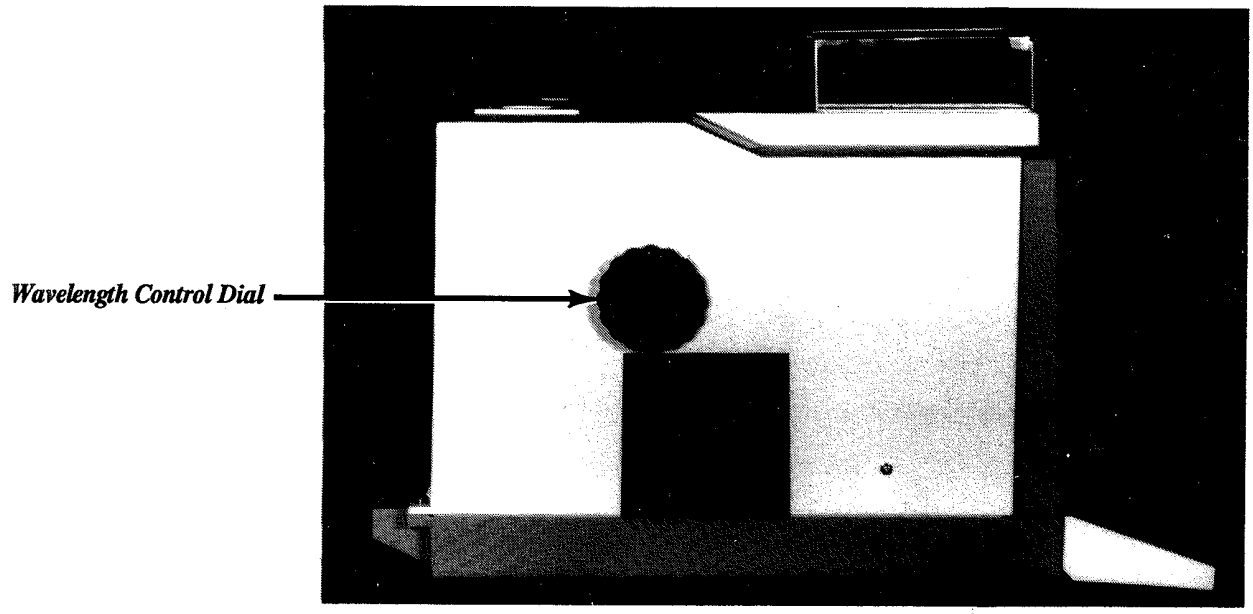


Figure 3-5. Wavelength Control Dial

3. Set the wavelength as close as possible to 324.8 nm with the Wavelength Control Dial.

2 Set the Slit Width and Height

1. Move the Slit Width Lever to the 0.7 position (see Figure 3-4).
2. Move the Slit Height Lever to the High position (see Figure 3-4).

3 Set Parameters and Turn on the Lamp

1. Turn on the instrument (if it is not already on) and wait for the following screen to appear.

```
PERKIN-ELMER
MODEL 3100
```

NOTE: For AA-BG operation, press: **[AA-BG]**.

2. Press: **[Param Entry]**. The following screen appears:

```
LAMP CUR. (0-50mA):
        0.0
```


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3. Press: **[1] [5]**. The screen changes to:

```
LAMP CUR. (0-50mA):
     15
```

4. <u>To turn on the lamp</u>, press: **[Enter]**.

   The lamp turns on, the message *AUTOMATIC GAIN APPLIED* flashes several times, then the following screen appears:

```
INT. TIME (.1-60sec)
     0.10
```

5. Press: **[0] [.] [3]**. The screen changes to:

```
INT. TIME (.1-60sec)
     0.3
```

6. Press: **[Enter] [Energy]**.

   A screen similar to the following <u>should</u> appear:

```
AA    CTS:  94   EN:58
```

7. If the bar chart is off scale,

```
AA    CTS: 276   EN:54
```

   or does not extend about halfway across the screen, press: **[Gain]** to bring the bar on scale.

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4 Maximize the Energy

1. Slowly rotate the Wavelength Control Dial about ½ nm to either side until you obtain maximum energy as shown by the bar and the number of counts (CTS).

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

2. Alternately, adjust the Lamp Alignment Screws (see Figure 3-2) until you have obtained maximum energy.

3. If the bar chart goes off scale, press: **[Gain]** to bring it on scale.

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

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Installing an Electrodeless Discharge Lamp

1. Turn on the EDL power supply
2. Set the switch on the rear of the EDL power supply to "CONTINUOUS."
3. Adjust the power using a Continuous power setting from Table 3-1. This is the same value shown on the lamp label.

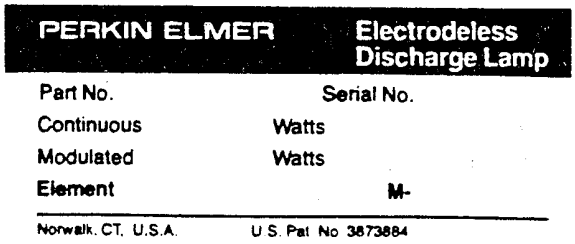


Figure 3-6. EDL Lamp Label

The lamp should turn on.

If the lamp does not turn on, point it at a UV source such as a fluorescent light, a flame, or the EDL Source Starter accessory.

Table 3-1

Recommended EDL Power Settings for the Model 3100

Element	3100(AA) Continuous	3100(AA-BG) Ext. Mod.
Antimony	8-8.5	7
Arsenic	8-8.5	7
Bismuth	8	7
Cadmium	5	4.5
Cesium	5	4
Germanium	7	5.5
Lead	10	8
Mercury	5	3.5
Phosphorus	8	7
Potassium	6	4.5
Rubidium	5	3.5
Selenium	6	4.5
Tellurium	8	6.5
Thallium	7	6
Tin	8-8.5	7
Titanium	9	7.5
Zinc	6	4

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NOTICE: The continuous power stated in Table 3-1 is a nominal value for most EDL lamps. However, due to variations in lamp components, a lamp may operate better at slightly higher or lower power settings than stated. Operate the lamp initially at the value stated. If the lamp intensity is low (Noisy signal), increase the power setting in increments of 0.5 to 1 watt until suitable intensity is obtained without oscillation (energy fluctuates up and down) or excessive loss of intensity. If the lamp oscillates, or if the absorption sensitivity is poorer than expected, reduce the power in increments of 0.5 to 1 watt until satisfactory operation is achieved.

The life of the lamp is relatively independent of power setting provided it is not operated at so high a setting that it oscillates, or at so low a setting that the lamp is only dimly lit, i.e., it is operating in the low brightness mode.

IMPORTANT: Running the EDL in the low brightness mode for an extended period of time will destroy the lamp and void the warranty.

NOTE: Some EDLs may have a coded lamp cable attached. It is not necessary to connect this cable to the 3100.

3. Locate the Lamp Alignment Screws on top of the instrument (see Figure 3-2).

4. Make certain that the Lamp Alignment Screws are in the vertical position. If they are tilted, simply flip them into a vertical position.

5. Locate the lamp compartment which is on the right hand side of the instrument (see Figure 3-3).

6. **Carefully** insert the lamp all the way, then pull it back about ¼ of an inch.

IMPORTANT: Be careful not to insert the lamp too forcefully to avoid damaging the front face of the lamp.

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7. If you have a Type B external modulation cable, connect the three pin connector to the three pin socket on the back of the power supply.

   If you have an Type A external modulation cable, connect the rounded connector to the back of the power supply.

8. Connect the modulation cable from the EDL power supply to the uncoded lamp socket on the instrument (see Figure 3-3).

9. Set the switch on the back of the EDL power supply to "CONTINUOUS" (see Figures 3-7 and 3-8).

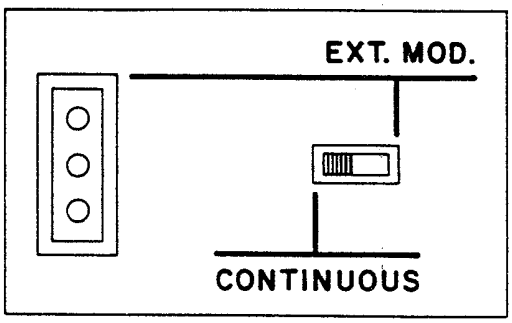


Figure 3-7. Rear View of EDL Power Supply With Type B External Modulation Cable.

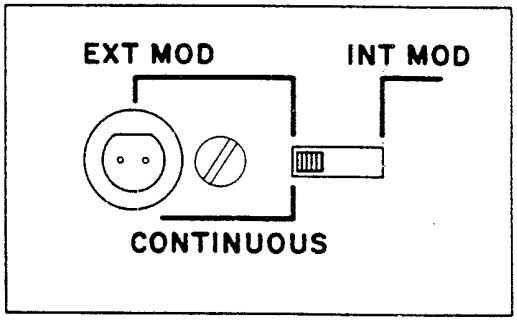


Figure 3-8. Rear View of EDL Power Supply With Type A External Modulation Cable.

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Aligning an Electrodeless Discharge Lamp in AA MODE

Carry out the following steps to align an electrodeless discharge lamp in AA Mode (lamp should be on):

1 Set the wavelength*.

2 Set the slit width* and height**.

3 Set parameters

4 Maximize the energy.

* Obtain these values for the lamp being aligned from the AA Cookbook.

** For flame analysis the slit height is always set to the "High" position.

Procedural details follow.

1 Set the Wavelength

1. Refer to the AA Cookbook and select a wavelength for the element of the installed lamp.
2. Set the wavelength as close as possible to this value with the Wavelength Control Dial located on the left side of the instrument (see Figure 3-5).

2 Set the Slit Width and Height

1. Locate the Slit Width and Slit Height Control Levers on top of the instrument (see Figure 3-4).
2. Move the Slit Width Lever to the position recommended in the AA Cookbook for the element of the lamp being aligned.
3. For flame operation, move the Slit Height Lever to the High position.

3 Set Parameters

1. Turn on the instrument (if it is not already on) and wait for the following screen to appear.

```
PERKIN-ELMER
MODEL 3100
```

2. Press: **[Param Entry]**. The following screen appears:

```
LAMP CUR. (0-50mA):
        0.0
```


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3. Press: **[5]**. The screen changes to:

```
LAMP CUR. (0-50mA):
        5
```

4. Press:**[Enter]**.

   The message *AUTOMATIC GAIN APPLIED* flashes several times, then the following screen appears:

   NOTE: The system will not automatically adjust the gain of the PMT unless a Lamp Current is entered.

```
INT. TIME (.1-60sec)
        0.10
```

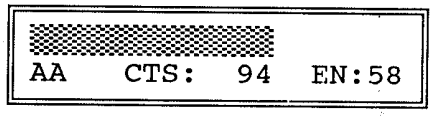
5. Press: **[0] [.] [3]**. The screen changes to:

```
INT. TIME (.1-60sec)
         0.3
```

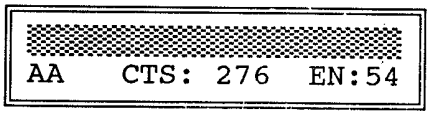
6. Press: **[Enter]**.

7. Press: **[Energy]**.

   A screen similar to the following should appear:



8. If the bar chart is off scale,



   or does not extend about halfway across the screen, press: **[Gain]** to bring the bar on scale.

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4 Maximize the Energy

1. Slowly rotate the Wavelength Control Dial in one direction, then the other, until you obtain maximum energy as shown by the bar and the number of counts (CTS).

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

2. Alternately adjust the Lamp Alignment Screws (see Figure 3-2) until you have obtained maximum energy.

3. If the bar chart goes off scale, press: **[Gain]** to bring it on scale.

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

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Aligning an Electrodeless Discharge Lamp in AA-BG MODE

Carry out the following steps to align an electrodeless discharge lamp in AA-BG Mode (lamp should be on):

NOTE: The lamp should be On and operating in Continuous Mode.

1 Set the wavelength*.

2 Set the slit width* and height**.

3 Set parameters and EDL Power Supply.

4 Maximize the energy.

* Obtain these values for the lamp being aligned from the AA Cookbook.

** For flame analysis the slit height is always set to the "High" position.

Procedural details follow.

1 Set the Wavelength

1. Refer to the AA Cookbook and select a wavelength for the element of the installed lamp.
2. Set the wavelength as close as possible to this value with the Wavelength Control Dial located on the left side of the instrument (see Figure 3-6).

2 Set the Slit Width and Height

1. Locate the Slit Width and Slit Height Control Levers on top of the instrument See Figure 3-5).
2. Move the Slit Width Lever to the position recommended in the AA Cookbook for the element of the lamp being aligned.
3. For Flame operation, move the Slit Height Lever to the High position.

3 Set Parameters and EDL Power Supply

1. Turn on the instrument (if it is not already on) and wait for the following screen to appear.

```
PERKIN-ELMER
MODEL 3100
```


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2. Press: **[AA-BG]**.

3. Press: **[Param Entry]**. The following screen appears:

```
LAMP CUR. (0-50mA):
        0.0
```

4. Press: **[5]**. The screen changes to:

```
LAMP CUR. (0-50mA):
        5
```

5. Press: **[Enter]**.

   The message *AUTOMATIC GAIN APPLIED* flashes several times.

   NOTE: This occurs because the instrument expects to see a modulated lamp signal in AA-BG Mode but did not.

6. Set the switch on the EDL power supply to "EXT MOD." The appearance of the switches are different depending on the type of modulation cable supplied (see Figures 3-7 and 3-8).

7. Adjust the power setting to the AA-BG value in Table 3-1.

8. Press: **[Energy] [Gain]**.

   The message *AUTOMATIC GAIN APPLIED* flashes several times, then the Energy screen (bar chart) is displayed.

```
▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒▒
AA   CTS:  94  EN:58
```

   If the bar chart is about halfway across the screen, proceed to Step 9.

   If the system cannot find the wavelength, the energy level remains at 0 or a very low level. Slowly rotate the Wavelength Control Dial about ½ nm on either side, until the bar chart appears on the top line of the above screen.

9. Press **[2] [Param Entry]**. The INT. Time screen reappears.

```
INT. TIME (.1-60sec)
        0.10
```


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10. Press: **[0] [.] [3]** .The screen changes to:

```
INT. TIME (.1-60sec)
          0.3
```

11. Press: **[Enter]**.

12. Press: **[Energy]**.

    A screen similar to the following should appear:

```
AA    CTS:  94   EN:58
```

13. If the bar chart is off scale

```
AA    CTS: 276   EN:54
```

    or does not extend about half-way across the screen, press: **[Gain]** to bring the bar on scale.

4 Maximize the Energy

1. Slowly rotate the Wavelength Control Dial about ½ nm on either side until you obtain maximum energy as shown by the bar and the number of counts (CTS).

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

2. Alternately adjust the Vertical and Horizontal Alignment Screws (see Figure 3-2) until you have obtained maximum energy.

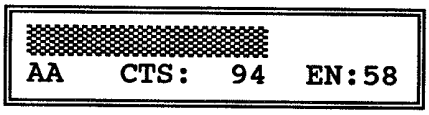
3. If the bar goes off scale, press: **[Gain]** to bring it on scale.

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

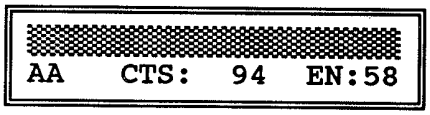
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12. Press: **[Energy]**.

    A screen similar to the following should appear:



13. If the bar chart is off scale



    or does not extend about half-way across the screen, press: **[Gain]** to bring the bar on scale.

4 Maximize the Energy

1. Slowly rotate the Wavelength Control Dial about ½ nm on either side until you obtain maximum energy as shown by the bar and the number of counts (CTS).

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

2. Alternately adjust the Vertical and Horizontal Alignment Screws (see Figure 3-2) until you have obtained maximum energy.

3. If the bar goes off scale, press: **[Gain]** to bring it on scale.

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

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Before You Ignite the Flame 4

Summary

Before you ignite the flame, make certain that:

1 The burner head is securely seated.

2 The burner head tie downs are in place.

3 The burner head pin is in place.

4 The drain and thermal shield interlock connectors are assembled.

5 The fuel/oxidant lines are securely attached to the burner and nebulizer.

6 The burner end cap is securely attached to the burner.

7 The nebulizer is securely attached to the end cap

8 The drain hose is securely attached to the burner end cap.

9 Water is present in the drain system float assembly.

10 The ignitor connector is attached to the ignitor assembly.

Procedural details follow.

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4 Connecting the Drain and Thermal Shield Interlock Connectors

The Drain and Thermal Shield Interlock Connectors are shown disassembled in Figure 4-5.

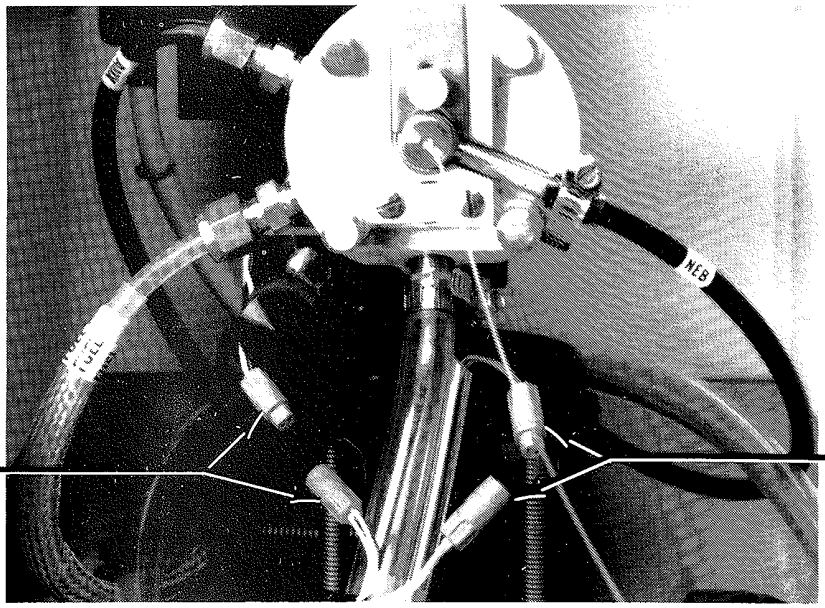


Figure 4-5. Drain and Thermal Shield Interlock Connectors Disconnected

* Make certain that both halves of the Drain and Thermal Shield Interlock Connectors are connected as shown in Figure 4-6.

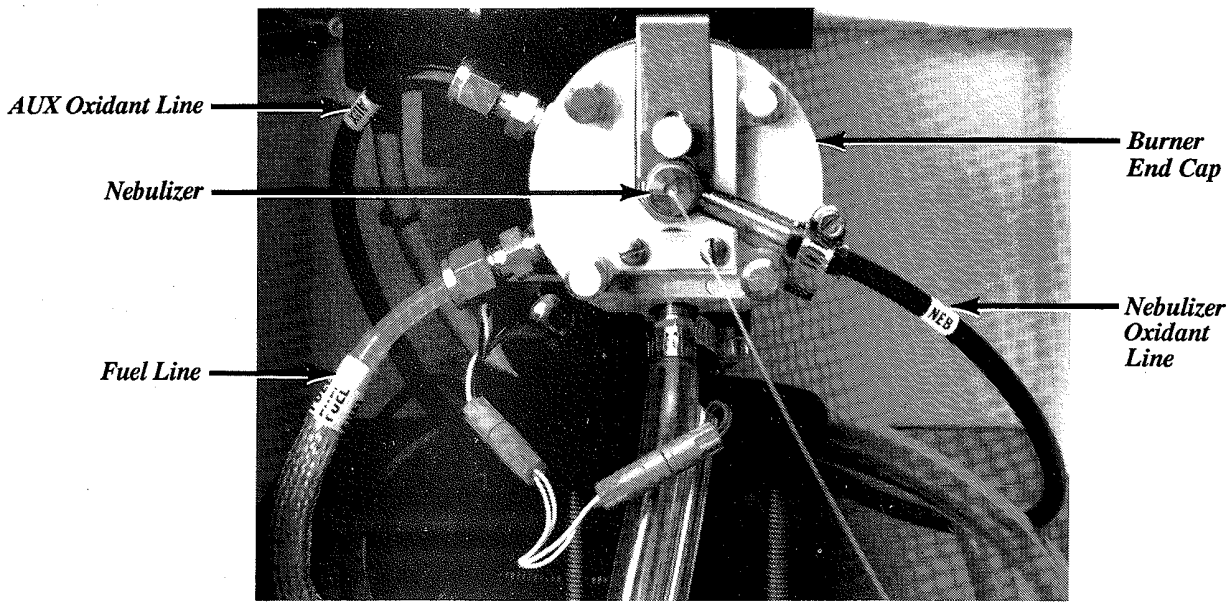


Figure 4-6. Drain and Thermal Shield Interlock Connectors Connected

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5 Securing the Gas Inlets

1. Make certain that the Fuel and Aux oxidant lines are attached to the Burner End Cap as shown in Figure 4-6, and that the connections are hand tight.

2. Make certain that the Neb oxidant line is attached to the nebulizer as shown in Figure 4-6. Tighten the hose clamp.

6 Securing the Burner End Cap

* Hand tighten the four knurled screws (see Figure 4-7).

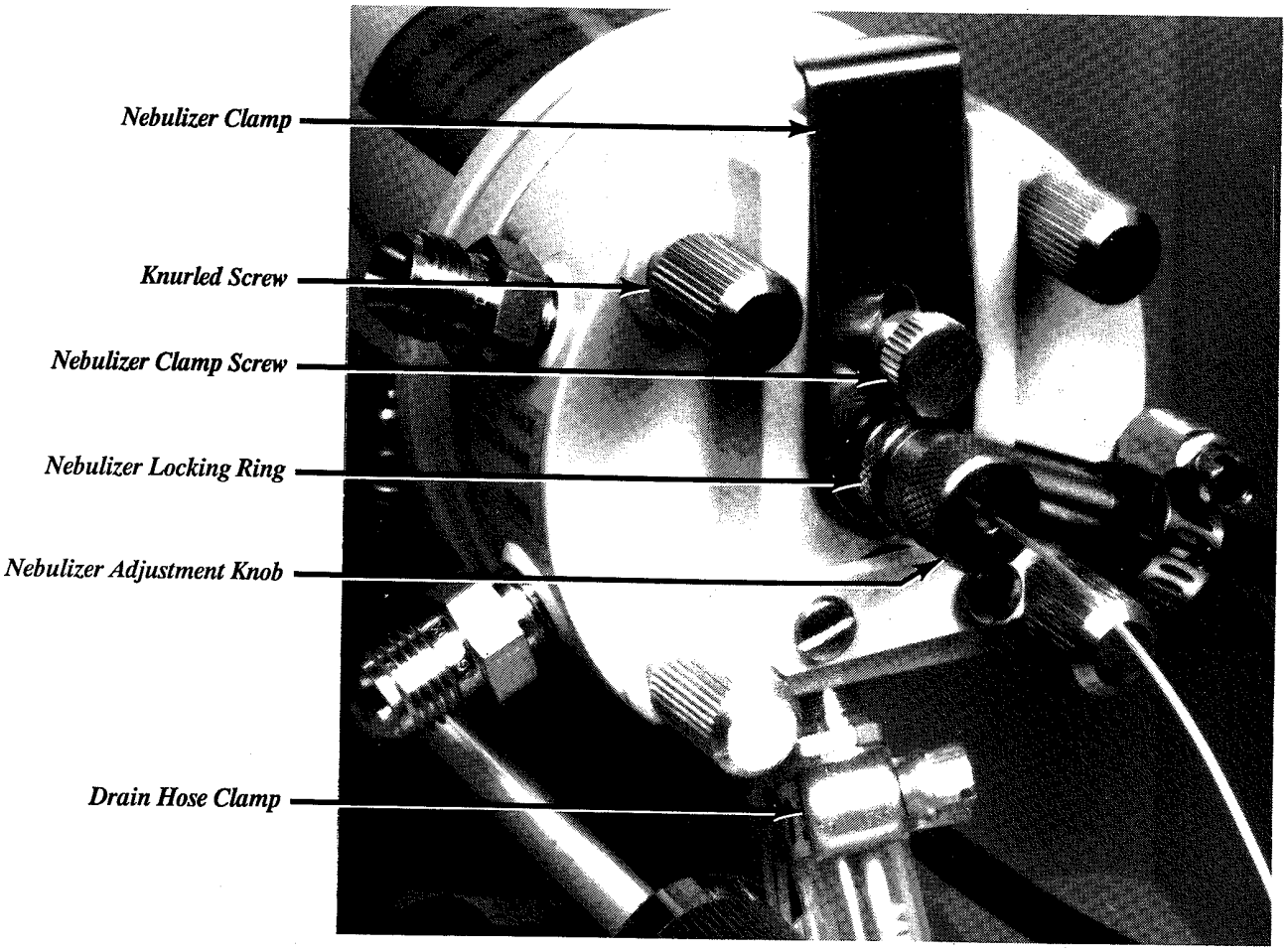


Figure 4-7. Burner End Cap Connections

7 Securing the Nebulizer

* Make certain that the nebulizer is in place (see Figure 4-7). Tighten the Nebulizer Clamp Screw.

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8 Securing the Drain Hose to the Burner End Cap

* Make certain the Drain Hose is attached to the Burner End Cap and that the Drain Hose Clamp is tightened securely (see Figure 4-7).

9 Checking for Water in the Drain Float Assembly

Enough water must be present in the Drain System to activate the Drain Float Assembly. If not, carry out the following procedure:

1. Loosen the Drain Hose Clamp and remove the drain tube from the drain outlet of the Burner End Cap (see Figure 4-7).
2. Pour about 250 mL of water into the drain tube.
3. Reconnect the drain tube to the Burner End Cap. Replace the Drain Hose Clamp and tighten securely.

10 Securing the Ignitor Assembly Connector

* Make certain that the Ignitor Assembly Connector is firmly connected to the Ignitor Assembly as shown in Figure 4-3 on page 4-3.

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Igniting and Optimizing the Flame 5

NOTE: Before igniting the flame, check that:

1. The burner head is securely seated.
2. The burner head tie downs are in place.
3. The burner head pin is in place.
4. The drain and thermal shield interlock connectors are assembled.
5. The burner end cap is securely attached to the burner.
6. The nebulizer is securely attached to the end cap
7. The fuel/oxidant lines are securely attached to the burner and nebulizer.
8. The drain hose is securely attached to the burner end cap.
9. Water is present in the drain system float Assembly.
10. The ignitor connector is attached to the ignitor assembly.

Procedures for performing the above are given in Chapter 4, Before You Ignite the Flame.

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Carry out the following steps to light and optimize the flame:

1 Turn on the gases at the source.

2 Adjust the burner height.

3 Ignite the flame.

4 Adjust the horizontal and rotational position of the burner.

5 Adjust the nebulizer.

6 Check the Characteristic Concentration.

The procedures which follow are for a 10 cm Burner Head using air-acetylene and assume that a flow spoiler has been installed and that you have just carried out the procedures in Chapters 3 and 4. The instrument will therefore be on and the Energy screen displayed.

Table 5-1 on page 5-10 gives recommended flowmeter gas settings for various burner heads.

A procedure for igniting the flame when using nitrous oxide is given on page 5-9.

Adjusting for organic solvents is discussed on page 5-7.

How to shut off the flame is described on page 5-10.

A graph of flowmeter divisions vs. gas flow rates is provided on page 5-10.

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1 Turn on the Gases

1. Turn on the vent.
2. Set the acetylene tank output pressure from 85 to 100 kPa (12 to 14 psig).

> **IMPORTANT:** Change the tank when the tank pressure falls to 520 kPa (75 psig). If the pressure falls below this level, acetone may be carried over into the burner regulator, possibly damaging valves and tubing. Such damage is not covered by the instrument warranty.

3. Set the air supply pressure to between 345 and 450 kPa (50 to 65 psig).

   If you are using nitrous oxide, set the nitrous oxide tank output pressure from 345 to 520 kPa (50 to 75 psig).

2 Adjust the Burner Height

With the instrument on and set up as described in Chapters 3 and 4, proceed as follows:

1. Place a piece of cardboard or white paper over the burner head slot and at right angles to it.
2. Adjust the Horizontal Adjustment Knob (see Figure 5-1.) until the light beam is approximately centered over the burner slot. Be sure that the beam passes over the head, i.e., that the burner head does not block the beam.

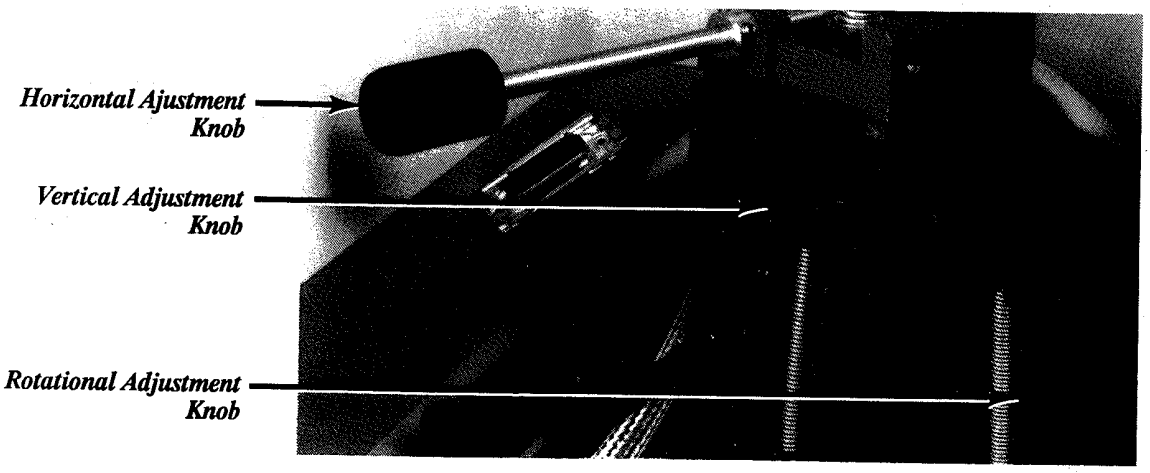


Figure 5-1. Burner Head Adjustment Controls

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3. Press: **[Cont]**.

   The Continuous screen appears and displays an absorbance value. For example:

```
CONTINUOUS
ABS = -0.394
```

4. Press: **[A/Z]**.

   Autozero is performed and a display similar to the following appears:

```
CONTINUOUS
ABS = 0.000
```

5. Raise the burner assembly by turning the Vertical Adjustment Knob counter-clockwise until you obtain a slight positive absorbance. For example:

```
CONTINUOUS
ABS = 0.003
```

6. Slowly rotate the Vertical Adjustment Knob clockwise until the absorbance returns to zero.

7. Rotate the knob an additional ¼ turn clockwise.

3 Ignite the Flame

WARNING: Never directly view the flame, hollow cathode lamps, electrodeless discharge lamps, or deuterium lamps without protective eyewear. Potentially hazardous ultraviolet radiation may be emitted from these sources. Ordinary safety glasses generally provide sufficient protection, but additional side shields insure a further margin of safety. Safety glasses also provide mechanical protection for the eyes.

1. Turn on the vent.

Please refer to Figure 5-2 on the following page for the next 3 steps.

2. Turn the Oxidant switch to Air.

3. Adjust the Oxidant flow to about 4 units on the flowmeter using the Oxidant Control Knob.

4. Adjust the acetylene flow to between 2 and 2.5 units on the flowmeter using the Fuel Control Knob.

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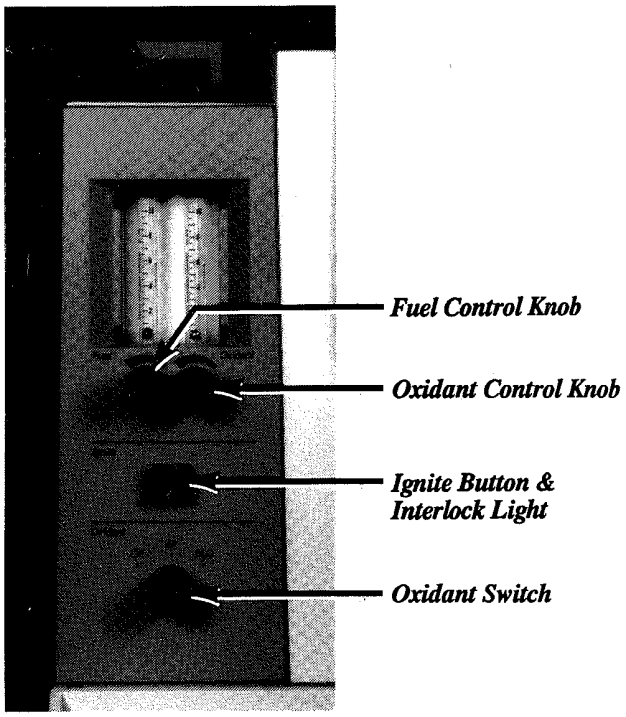


Figure 5-2. Pneumatics Control Panel

5. Press the Ignite Button.

   NOTE: If the gases are left on for 15 seconds without ignition, the red interlock light comes on and the system turns off the gas flows. If this happens, turn the gas control switch Off, then back to Air, then press the Ignite button.

   NOTE: When gases are first turned on, the flame may not light the first time due to air in the supply lines. If the flame does not ignite, turn the gas control switch Off, then back to Air and press the Ignite button.

   NOTE: If the interlocks are not satisfied, the flame will not ignite and the red interlock light turns on. If this occurs, refer to Chapter 15, Hardware Troubleshooting, page 15-6 for causes and cures.

6. Aspirate the blank and let the flame stabilize for about 30-60 seconds.

   NOTE: If you see bubbles coming out of the capillary tube, loosen the Nebulizer Locking Screw and turn the Nebulizer Adjustment Screw clockwise (see Figure 5-3) until the bubbling stops.

7. Press: **[A/Z]**.

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4 Adjust the Burner Position

1. Aspirate a standard.

   NOTE: Elements such as Cu, Mn, or Mg, which are at wavelengths where flame gases do not absorb, should be used for adjusting the Burner and Nebulizer.

2. Rotate the horizontal and rotational adjustment knobs (see Figure 5-1) in turn until you obtain maximum absorbance.

3. Leave the capillary in the standard for the next procedure.

5 Adjust the Nebulizer

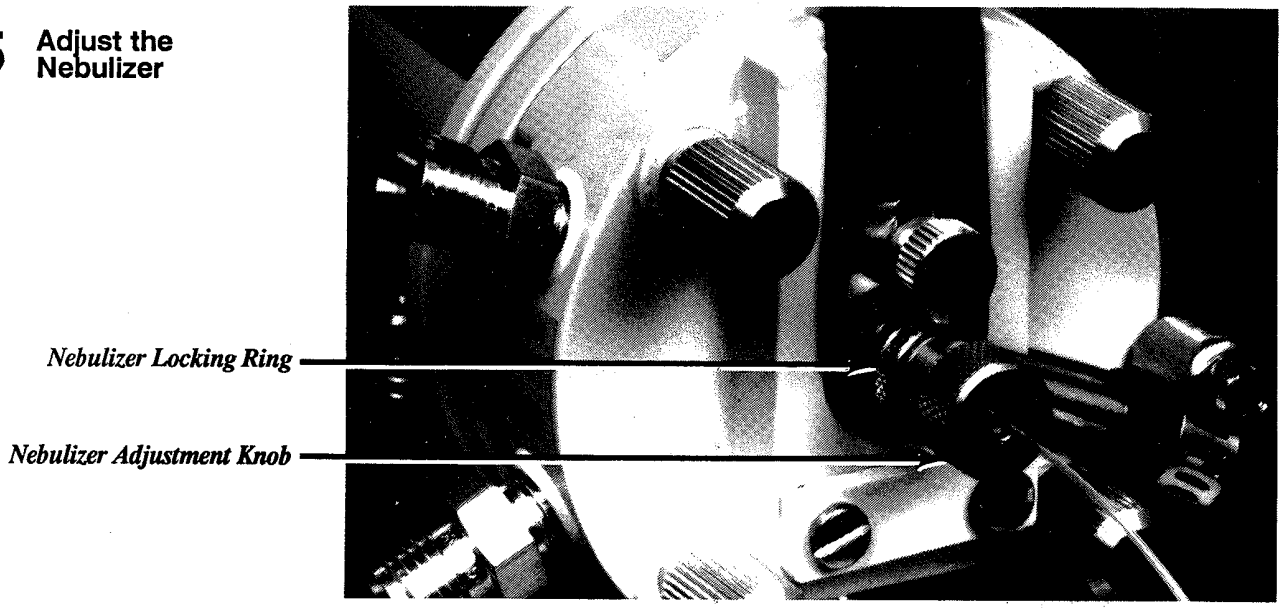


Figure 5-3 Nebulizer Adjustment Control

1. Loosen the Nebulizer Locking Ring by turning it clockwise until it is free of the Nebulizer Adjustment Knob.

2. Slowly turn the Nebulizer Adjustment Knob counter-clockwise until bubbles begin to appear at the end of the capillary tube in the sample solution.

3. Slowly turn the Nebulizer Adjustment Knob clockwise until the absorbance goes to a maximum and begins to decrease. (The inversion point at which absorbance changes marks the optimum setting). Slowly turn the Nebulizer Adjustment Knob counter-clockwise to obtain maximum absorbance.

4. Lock the Nebulizer Adjustment Knob in its final position by turning the Nebulizer Locking Ring counter-clockwise while holding the Nebulizer Adjustment Knob securely.

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6 Check the Characteristic Concentration

The Characteristic Concentration is the concentration that will give an absorbance of approximately 0.2 absorbance units. Typical Characteristic Concentration values are given in the AA Cookbook.

Some laboratories prefer to check the Characteristic Concentration daily before beginning a series of routine analyses.

Following is a suggested procedure that you can use after the flame has been ignited and optimized for the installed lamp.

1. Refer to the Standard Conditions section of the AA Cookbook and find the recommended Characteristic Concentration for the installed lamp. Prepare an appropriate standard solution of this element.
2. Aspirate a blank.
3. Press: **[Cont]**.
4. Press: **[A/Z]** to zero the display.
5. Aspirate the standard and note the absorbance.

The absorbance displayed should be within 20% of 0.2 absorbance units.

If not, refer to Chapter 15, Hardware Troubleshooting.

Adjusting for Organic Solvents

When aspirating organic solvents, you should lower the rate of nebulizer uptake, easily measured with a graduate cylinder. The normal rate is 7-10 mL/min. If you reduce this rate to about 2-4 mL/min, you will generally be able to operate with a leaner (bluer) flame compared to what you would get with the higher uptake rates. The leaner flame typically provides improved precision, particularly when aspirating xylene. A second advantage to using a lower flow rate is that it then is not necessary to aspirate the solvent continuously to keep the flow from extinguishing the flame.

Alternatively, or in combination with reducing the nebulizer flow, you may adjust the fuel/oxidant flow ratio to compensate for solvent flammability. Ignite the flame as for aqueous solutions, aspirate the solvent, and reduce the fuel flow to obtain desired flame conditions. After adjusting the flame, aspirate the solvent continuously to prevent the flame from extinguishing.

To adjust the nebulizer uptake rate, rotate the Knurled Nebulizer Adjustment Screw and locate a position providing reduced sample uptake.

You can easily measure the uptake rate by placing the nebulizer capillary tube into a graduate cylinder containing water. Simply measure the reduction in volume over a period of 1 minute.

For further information on organics, consult the AA Cookbook.

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Shutting Off the Flame

1. Aspirate water for a few minutes to clean the burner head.
2. Turn the gas control switch to Air and wait for 15 seconds.
3. Turn the gas control switch on the Pneumatics Control Panel to Off.
4. Close all gas source valves.

Recommended Flowmeter Settings

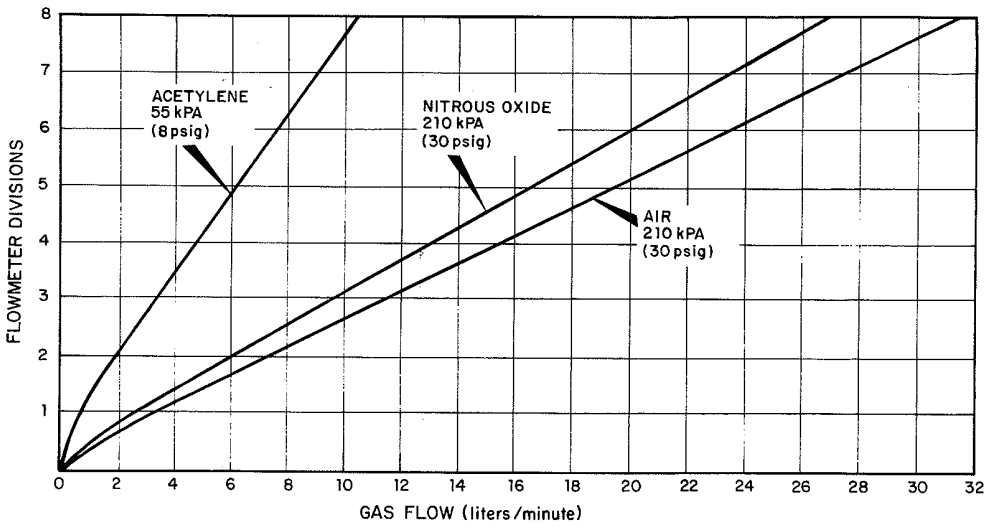
Table 5-1

Recommended Flowmeter Settings*

	5 cm Head		10 cm Head		3 Slot Head	
	Acet	Oxid	Acet	Oxid	Acet	Oxid
Air/Acet	2	3	2.5	4	4	6
N_20/Acet	3.5	4	-	-	-	-

* The above are approximate settings recommended for initial ignition.

Flowmeter Divisions vs. Gas Flow Rate



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Running Samples With Flame 6
Linear/Non-Linear Calibration, Manual Sampling

Overview

Once the system has been powered up and optimized, analyzing samples is simply a matter of entering parameters, then aspirating solutions of a blank, standards, and samples. An example of an aspirating sequence for Linear/Non-Linear calibration is shown below:

Blank
Standard 1
Standard 2
↓
Standard 8
Sample 1
Sample 2
↓
Sample 20
Recalibrate
Sample 21
Sample 22
etc.

For Non-Linear or Linear calibration, you can aspirate a Reslope Standard at any point after the first sample is run.

This chapter contains an example of how to run AA Flame analyses manually using Linear/Non-Linear calibration. The example takes you step by step through the required procedures and shows you how to display and print results.

To help you learn how to run samples manually with the 3100, it is recommended that you perform the set-up procedures in Chapters 3, 4, and 5, then duplicate the procedures given in the example.

> NOTE: Page 6-10 contains procedures for generating a new calibration curve and rerunning a single standard, if desired.

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EXAMPLE: Determining Copper Using Non-Linear Calibration

NOTE: The procedures in this example assume that the set-up procedures in Chapters 3, 4, and 5 have been performed, in which case the instrument will be on and the flame ignited.

In this example we ran a blank, 3 standards, 2 samples, a Reslope Standard, then 2 more samples.

Carry out the following steps to duplicate our example:

1. Prepare solutions
2. Enter parameters
3. Activate the printer
4. Enter the data collection mode
5. Run the blank
6. Run the standards
7. Run the samples
8. Run the reslope standard
9. Run the remaining samples

Detailed procedures follow.

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1 Prepare Solutions

We used a copper lamp and solutions of copper for our determinations. You don't have to use copper if you don't want to. For your convenience in making substitutions we have included a table of recommended concentrations for Ag, Mg, Mn, and Pb.

1. Prepare about 200-250 mL of 1% v/v HNO_3 to use as a blank.
2. Prepare about 200-250 mL each of three standards in 1% v/v HNO_3 of the element you want to determine. Suggested standard concentrations for five elements are shown in the following table:

Element	Std 1	Std 2	Std 3
Ag	2	6	12
Cu	4	15	30
Mg	0.25	0.75	1.5
Mn	1	3	6
Pb	10	30	60

3. For Samples 1 and 2 use Standards 1 and 2.
4. For the Reslope Standard use Standard 2.
5. For Samples 3 and 4, use Standards 2 and 3.

2 Enter Parameters

1. Press: **[Param Entry]**.

   If you have just aligned a copper lamp, the Lamp Current screen appears as shown below:

```
LAMP CUR. (0-50mA):
        15.0
```

   If you have aligned another lamp, its Lamp Current appears on the above screen.

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2. Press: **[Enter]**.

   If you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen.

```
INT. TIME (.1-60sec)
        0.3
```

3. Press: **[3]**. The screen changes to:

```
INT. TIME (.1-60sec)
        3
```

4. Press **[Enter]**. The following screen appears:

```
  REPLICATES (1-99):
        01
```

5. Press: **[3]**, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

6. To select Non-Linear calibration, press: **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```

7. Since the default AA technique is Flame (1), and that's what we want, Press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
       ------
```

8. If you are using Cu, press: **[4]** **[.]** **[0]** , then press: **[Enter]**. The screen for the next standard appears:

   If you are using another element, type in the concentration of Standard 1 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 2 (.0001-9999) :
       ------
```


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9. If you are using Cu, press: **[1] [5] [.] [0]** , then press: **[Enter]**. The screen for the next standard appears:

   If you are using another element, type in the concentration of Standard 2 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 3 (.0001-9999) :
      ------
```

10. If you are using Cu, press: **[3] [0] [.] [0]** , then press: **[Enter]**. The screen for the next standard appears:

    If you are using another element, type in the concentration of Standard 3 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 4 (.0001-9999) :
      ------
```

11. Press: **[Enter]**. The following screen appears:

```
RSLP (.0001-9999) :
     ------
```

12. When we ran our solutions, we aspirated a Reslope Standard after the second sample. For our Reslope Standard we actually used Standard 2 which contains 15 mg/L of Cu.

13. If you are using Cu, press: **[1] [5] [.] [0]**, then press **[Enter]**.

    If you are not using Cu, type in the concentration of your Reslope Standard, then press: **[Enter]**.

Parameter entry is now complete. Proceed to the next step.

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3 Activate the Printer (if one is attached)

* Press: **[Print]**.

  Since you are in the Parameter Entry mode, the parameters will be printed.

  Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

4 Enter the Data Collection Mode

1. Press: **[Data]**. The Data screen appears:

```
   ABS (MEAN):_______
SD:_____ RSD_____
```

As measurements are made, the results appear on the above screen.

5 Run the Blank

1. Aspirate the blank.
2. Press: **[A/Z]**.

The results are displayed and printed. For example,

```
   ABS(MEAN): 0.000
SD:0.000   RSD:16.29
```

```
AUTOZERO APPLIED
ABSORBANCE:
          -0.000  -0.000  -0.000
MEAN    0.000  SD: 0.0000  RSD(%): 16.29
```


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6 Run the Standards

1. Aspirate Standard 1, then press: **[Calib]**.

   As each replicate is read, its absorbance appears briefly on the screen and is printed. After the last replicate is read, the results are displayed and printed. For example,

```
  CONC (MEAN): 4.0
SD:0.0005   RSD:0.23
```

```
STANDARD 1
ABSORBANCE:
          0.215   0.214   0.215
CONC:   (STD APPLIED)
MEAN:    4.0    SD: 0.0005   RSD(%): 0.23
```

NOTE: If at any time you make a mistake and need to rerun a standard, simply enter the number of the standard then press: **[Calib]**. For example, if you wish to rerun Standard 1, aspirate standard 1, then press: **[1] [Calib]**. This does not erase previously run standards.

2. Aspirate Standard 2, then press: **[Calib]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 15.0
SD:0.0621   RSD:0.41
```

```
STANDARD 2
CONCENTRATION:
          14.0    14.1    14.0
CONC:   (STD APPLIED)
MEAN:    15.0    SD: 0.0621   RSD(%): 0.41
```

3. Aspirate Standard 3, then press: **[Calib]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 30.0
SD:0.1177   RSD:0.39
```

```
STANDARD 3
CONCENTRATION:
          28.4    28.4    28.2
CONC:   (STD APPLIED)
MEAN:    30.0   SD: 0.1177    RSD(%): 0.39
```


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7 Run the Samples

1. Aspirate Sample 1 (we used Standard 1 as our sample), then press: **[Read]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 4.0
SD:0.0161   RSD:0.40
```

```
CONCENTRATION:
          4.0    4.0    4.0
MEAN:    4.0  SD: 0.0161   RSD(%): 0.40
```

2. Aspirate Sample 2 (we used Standard 2 for our second sample), then press: **[Read]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 15.1
SD:0.0237   RSD:0.16
```

```
CONCENTRATION:
          15.0   15.1   15.1
MEAN:    15.1   SD: 0.0237  RSD(%): 0.16
```

8 Run the Reslope Standard

1. Aspirate the blank.
2. Press: **[A/Z]**.

   The results are displayed and printed. For example,

```
      (MEAN): 0.0
SD:0.000   RSD:16.29
```

```
AUTOZERO APPLIED
          -0.000  -0.000  -0.000
MEAN      0.0  SD: 0.0000  RSD(%): 16.29
```


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3. Aspirate the Reslope Standard (we used Standard 2 for our Reslope Standard), then press: **[Reslope]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 15.1
SD:0.0290   RSD:0.19
```

```
RESLOPE
CONCENTRATION:
          15.1    15.1    15.1
CONC:   (STD APPLIED)
MEAN:    15.0    SD: 0.0290   RSD(%): 0.19
```

9 Run the Remaining Samples

1. Aspirate Sample 3 (we used Standard 2), then press: **[Read]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 15.0
SD:0.0117   RSD:0.08
```

```
CONCENTRATION:
          15.0    15.0    15.0
MEAN:    15.0    SD: 0.0117   RSD(%): 0.08
```

2. Aspirate Sample 4 (we used Standard 3), then press: **[Read]**.

   The results are displayed and printed. For example,

```
  CONC(MEAN): 30.1
SD:0.1089   RSD:0.36
```

```
CONCENTRATION:
          29.9    30.1    30.1
MEAN:    30.1    SD: 0.1089   RSD(%): 0.36
```


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Generating a New Calibration Curve

Depending on circumstances, you may wish to generate a new calibration curve. If so, use the following procedure:

1. Aspirate the blank and press: **[A/Z]**.
2. Aspirate Standard 1.
3. Press: **[0] [Calib]**

   This erases the original calibration curve and applies Standard 1.
4. Aspirate Standard 2.
5. Press: **[Calib]**.
6. Aspirate Standard 3.
7. Press: **[Calib]**.

Rerunning a Single Standard

If you wish to rerun one of your standards, simply enter the number of the standard and press: **[Calib]**. For example, if you wish to rerun Standard 2, aspirate Standard 2, then press: **[2] [Calib]**.

RSD Calculations

NOTE: The calculated RSD is obtained using more significant digits than are displayed or printed.

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Running Samples With Flame 7
Linear/Non-Linear, Autosampler

Overview

Once the system has been powered up and optimized, analyzing samples is simply a matter of entering parameters, then aspirating solutions of a blank, standards, and samples. An example of an aspirating sequence for Linear/Non-Linear calibration is shown below:

Blank
Standard 1
Standard 2
↓
Standard 8
Sample 1
Sample 2
↓
Sample 20
Reslope Standard
Sample 21
Sample 22
etc.

For Non-Linear or Linear calibration, you can instruct the autosampler via a parameter entry to recalibrate at any point after the first sample is run. Up to 5 recalibrations can be performed.

This chapter contains an example of how to run AA Flame analyses using Non-Linear calibration with an autosampler. The example takes you step by step through the required procedures and shows you how to display and print results.

To help you learn how to run samples with an autosampler, it is recommended that you perform the setup procedures in Chapters 3, 4, and 5, then duplicate the procedures given in the example.

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EXAMPLE: Determining Copper Using Non-Linear Calibration

NOTE: The procedures in this example assume that the setup procedures in Chapters 3, 4, and 5 have been performed, in which case the instrument will be on and the flame ignited.

NOTE: Before starting an autosampler run, turn the autosampler on and make certain that the IEEE-488 control switch on the rear of the Model 3100 is set to the Down position.

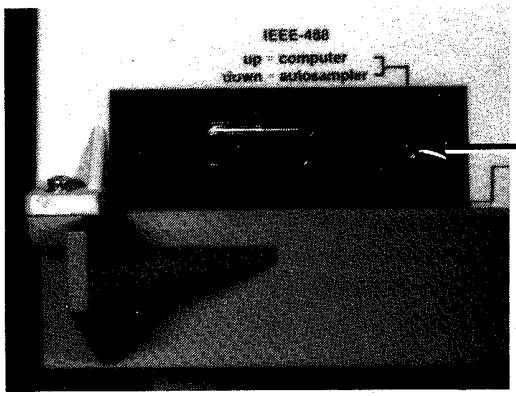


Figure 7-1. IEEE-488 Control Switch

In this example we ran a blank, 3 standards, and 3 samples. We instructed the autosampler to recalibrate after the second sample.

Carry out the following steps to duplicate our example:

1. Prepare solutions
2. Load the autosampler
3. Enter parameters
4. Activate the printer
5. Enter the data collection mode
6. Start the run

Detailed procedures follow.

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1 Prepare Solutions

We used a copper lamp and solutions of copper for our determinations. You don't have to use copper if you don't want to. For your convenience in making substitutions we have included a table of recommended concentrations for Ag, Mg, Mn, and Pb.

1. Prepare about 200-250 mL of 1% v/v HNO_3 to use as a blank.

2. Prepare about 200-250 mL each of three standards in 1% v/v HNO_3 of the element you want to determine. Suggested standard concentrations for five elements are shown in the following table:

Element	Std 1	Std 2	Std 3
Ag	2	6	12
Cu	4	15	30
Mg	0.25	0.75	1.5
Mn	1	3	6
Pb	10	30	60

3. For Samples 1, 2, and 3, use Standards 1, 2, and 3, respectively.

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2 Load the Autosampler

Each autosampler tray contains an opening for a large beaker (position 0). The AS-51 contains fifty four 15-mL positions, the first 8 are reserved for standards. The AS-90 contains eight 50-mL positions reserved for standards, and ninety eight 15-mL sample positions.

To load an autosampler tray:

1. Place the blank in position 0 (the large beaker).
2. Place Standards 1, 2, and 3 in positions 1, 2, and 3, respectively.
3. Place samples 1, 2, and 3 (we used Standards 1, 2, and 3 for our samples), in positions 9, 10, and 11, respectively.

3 Enter Parameters

1. Press: **[Param Entry]**.

   If you have just aligned a copper lamp, the Lamp Current screen appears as shown below: If you have aligned another lamp, its Lamp Current appears on the screen.

```
LAMP CUR. (0-50mA):
        15.0
```

2. Press: **[Enter]**.

   If you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen as shown below.

```
INT. TIME (.1-60sec)
        0.30
```

3. Press: **[3]**. The screen changes to:

```
INT. TIME (.1-60sec)
           3
```

4. Press **[Enter]**. The following screen appears:

```
  REPLICATES (1-99):
          01
```

5. Press: **[3]**, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```


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6. To select Non-Linear (1) press: **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```

7. To select Flame (1), press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
        _____
```

8. If you are using Cu, press: **[4] [.] [0]**, then press: **[Enter]**.

   If you are using another element, type in the concentration of Standard 1 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 2 (.0001-9999) :
        ------
```

9. If you are using Cu, press: **[1] [5] [.] [0]**, then press: **[Enter]**.

   If you are using another element, type in the concentration of Standard 2 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 3 (.0001-9999) :
        ------
```

10. If you are using Cu, press: **[30] [.] [0]**, then press **[Enter]**.

    If you are using another element, type in the concentration of Standard 3 for that element, then press: **[Enter]**. The screen for the next standard appears:

```
STD 4 (.0001-9999) :
        ------
```

11. Press **[Enter]** until the following screen appears

```
 SELECT AUTOSAMPLER
AS51 (1)  AS90 (2):1
```

12. Enter a number corresponding to the autosampler attached, then press: **[Enter]**.

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If an AS-51 is attached the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-54): 09
```

If an AS-90 is attached the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-106): 09
```

13. Press: **[1] [1]**, then press **[Enter]**. The following screen appears:

```
   ASAMP RECAL POS:
   __ __ __ __ __
```

14. Press: **[1] [1]**, then press **[Enter]**.

Parameter entry is now complete. Proceed to the next step.

4 Activate the Printer (if one is attached)

* Press: **[Print]**.

Since you are in the Parameter Entry mode, the parameters will be printed.

Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

5 Enter the Data Collection Mode

* Press: [Data]. The Data screen appears:

```
     ABS (MEAN):_____
SD:_____ RSD_____
```

As measurements are made the results appear on the above screen.

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6 Start the Run

* Press **[Auto Sampler]**.

The run starts and as each replicate is measured, its absorbance appears briefly on the above screen. After the last replicate is measured, the results are displayed and printed.

Running a Single Vial

The probe can be moved to any vial position (i.e., to run a single vial) by entering the desired vial number followed by pressing: **[Auto Sampler]**.

If the probe is down, the probe moves up, over to the specified position and down into that vial. Thus the vial in the specified position will be aspirated.

If the probe is up, it is moved to the specified position but stays up, i.e., the vial in the specified position will not be aspirated. To aspirate the sample in the specified position, move the probe down by pressing **[.] [Auto Sampler]**. Pressing **[.] [Auto Sampler]** again will raise the probe.

Recalibration

The Model 3100 allows periodic recalibration at any point during an autosampler run. To specify the position before which the recalibration is to occur, press **[17] [Param Entry]** to display the Recalibrate screen.

```
ASAMP RECAL POS:
__ __ __ __ __
```

Enter the position number(s) before which the instrument is to be recalibrated. Up to five entries can be made. Each entry must consist of two digits. E.g., to enter position 9, press **[0] [9]**. The highest position number that can be entered is 99.

RSD Calculations

NOTE: The calculated RSD is obtained using more significant digits than are displayed or printed.

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Running Samples With Flame 8

Using Method of Additions, Manual Sampling

Overview

Once the system has been powered up and optimized, analyzing samples is simply a matter of entering parameters in a prescribed sequence, then aspirating solutions of a blank, sample and standards in a prescribed sequence.

An example of an aspirating sequence for Method of Additions is shown below:

Blank

Sample 1 (Unspiked)
Standard 1 (Spiked Sample = Sample 1 + Spike 1)
Standard 2 (Spiked Sample = Sample 1 + Spike 2)

Sample 2
Standard 1 (Spiked Sample = Sample 2 + Spike 1)
Standard 2 (Spiked Sample = Sample 2 + Spike 2)

Sample #3
etc.

This chapter contains an example of how to run standard AA Flame analyses manually using Method of Additions. The example takes you step by step through the required procedures and shows you how to display and print results.

To help you learn how to run samples using Method of Additions, it is recommended that you perform the setup procedures in Chapters 3, 4, and 5, then duplicate the procedures given in the example.

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About Method of Additions

If you are familiar with Method of Additions, skip to the Example on page 8-3.

Method of Additions allows you to analyze samples in the presence of an interference and is particularly useful where the sample matrix varies from sample to sample. A typical example would be the determination of trace platinum metals in samples from a smelting operation.

In this technique, the standards are samples that are "spiked" with known amounts of the element of interest. Both the sample and the standards are then diluted to exactly the same volume. After aspirating a blank and autozeroing, these diluted solutions are run in the following sequence:

Sample

Standard 1 (Spiked Sample = Sample 1 + Spike 1)
Standard 2 (Spiked Sample = Sample 1 + Spike 2)

The system creates a calibration curve using these three solutions. It then extrapolates the calibration curve to the baseline and calculates the concentration of the element of interest in the original unspiked sample. For example:

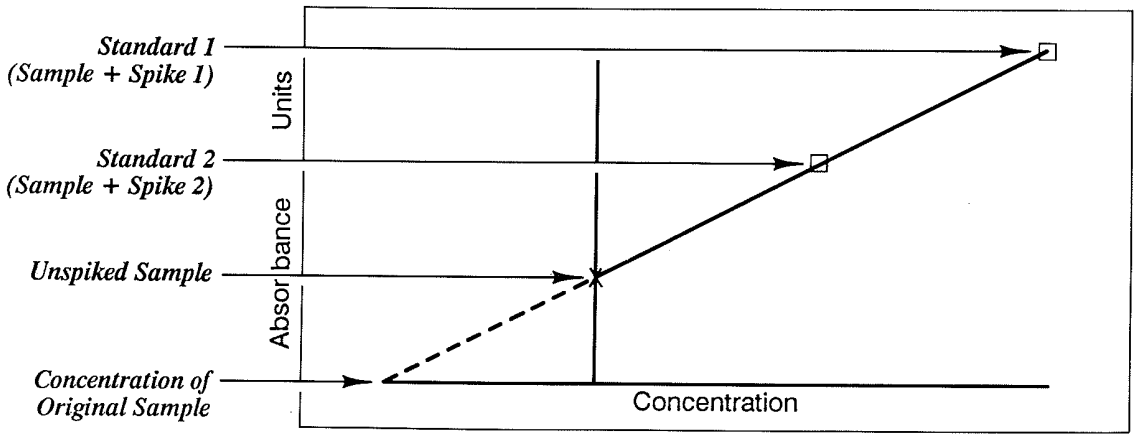


Figure 8-1. Example of Method of Additions Calibration Curve

NOTE: The Method of Additions calibration curve is recalculated whenever a blank, a first addition, or a last addition has been run. This allows a method of additions calibration curve to be aborted without affecting the sample sequence.

A more complete discussion of Method of Additions is given in Chapter 17, Calibration and in Section 7 (the yellow pages) of the AA Cookbook.

NOTE: All solution concentrations for Method of Additions must be in the linear range.

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EXAMPLE: Determining Copper Using Method of Additions

NOTE: The procedures in this example assume that the setup procedures in Chapters 3,4, and 5 have been performed, in which case the instrument will be on and the flame ignited.

In this example we ran a blank, one unspiked sample, and two standards (spiked samples).

Carry out the following steps to duplicate our example:

1. Prepare solutions
2. Enter parameters
3. Activate the printer
4. Enter the data collection mode
5. Run the blank
6. Run unspiked sample
7. Run the standards (spiked samples)

Detailed procedures follow.

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1 Prepare Solutions

We used a copper lamp and solutions of copper for our determinations. You don't have to use copper if you don't want to. For your convenience in making substitutions we have included a table of recommended concentrations for Ag, Mg, Mn, and Pb.

1. Prepare about 200 mL of 1% v/v HNO_3 to use as a blank and diluent.
2. Prepare about 200 mL of a Stock Standard of the element of interest.
3. Prepare about 200 mL of sample.

Recommended concentrations for Stock Standards (to be used as spiking solutions) and samples are shown below.

Element	Stock Standard Conc. (mg/L)	Original Sample Conc (mg/L)
Ag	2.0	1.0
Cu	2.0	1.0
Mg	0.2	0.1
Mn	1.0	0.5
Pb	10.0	5.0

Prepare the Unspiked Sample

* Dilute 50 mL of the sample to 100 mL with diluent.

Prepare Standard 1 (Addition 1)

* To 50 mL of sample, add 25 mL of Stock Standard and dilute to 100 mL with diluent.

  Since we diluted our stock standard by a factor of 2, we effectively spiked the sample with a solution containing 1 mg/L of Cu.

Prepare Standard 2 (Addition 2)

* To 50 mL of sample, add 50 mL of Stock Standard (effectively 50 mL of sample has been diluted to 100 mL with Stock Standard).

  Effectively, we have spiked the sample with a solution containing 2 mg/L of Cu.

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Calculating Standard Concentrations

In the next section, Entering Parameters, you will have to enter the concentrations for Standards 1 and 2. Use the following formula to calculate these concentrations:

$$\text{Conc. of Std} = \frac{\text{Vol of stock std. x Conc. of stock std.}}{\text{Vol. of sample}}$$

Therefore, for our example, the concentrations of Standards 1 and 2 are calculated as follows:

$$\text{Conc. of Std. 1} = \frac{25\ \text{mL} \times 2\ \text{mg/L}}{50\ \text{mL}} = 1\ \text{mg/L}$$

$$\text{Conc. of Std. 1} = \frac{50\ \text{mL} \times 2\ \text{mg/L}}{50\ \text{mL}} = 2\ \text{mg/L}$$

2 Enter Parameters

1. Press: **[Param Entry]**.

   If you have just aligned a copper lamp, the Lamp Current screen appears as shown below:

```
LAMP CUR. (0-50mA):
        15.0
```

   If you have aligned another lamp, its Lamp Current appears on the above screen.

2. Press: **[Enter]**.

   If you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen as shown below.

```
INT. TIME (.1-60sec)
        0.30
```

3. Press: **[3]**. The screen changes to:

```
INT. TIME (.1-60sec)
        3
```


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4. Press **[Enter]**. The following screen appears:

```
REPLICATES (1-99):
        01
```

5. Press: **[3]**, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

6. To select Method of Additions (3), press: **[3]** , then press: **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```

7. To select Flame (1), press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
        _____
```

8. If you are using Cu, press: **[1] [.] [0]**, then press: **[Enter]**.

   The following screen appears:

```
STD 2 (.0001-9999) :
       ------
```

   NOTE: If you are using another element, type in the concentration of Standard 1 for that element, then press: **[Enter]**.

9. If you are using Cu, press: **[2] [.] [0]**, then press: **[Enter]**. The following screen appears:

```
STD 3 (.0001-9999) :
       ------
```

   NOTE: If you are using another element, type in the concentration of Standard 2 for that element, then press: **[Enter]**.

10. Press:**[Enter]**.

Parameter entry is now complete. Proceed to the next step.

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3 Activate the Printer (if one is attached)

* Press: **[Print]**.

Since you are in the Parameter Entry mode, the parameters will be printed.

Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

4 Enter the Data Collection Mode

* Press: **[Data]**.

The Data screen appears:

```
   ABS (MEAN):_______
SD:_____ RSD_____
```

As measurements are made, the results appear on the above screen.

5 Run the Blank

1. Aspirate the blank.
2. Press: **[A/Z]**.

The following screen and printout appear:

```
   ABS(MEAN): 0.000
SD:0.000  RSD:16.29
```

```
AUTOZERO APPLIED
          -0.000  -0.000  -0.000
MEAN    0.000  SD 0.0000  RSD(%): 16.29
```


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6 Run the Unspiked Sample

* Aspirate the unspiked sample, then press: **[Read]**.

As each replicate is read, the absorbance appears briefly on the screen. After the last replicate is read, the results are displayed and printed. For example,

```
   ABS(MEAN): 0.029
SD:0.0005  RSD:1.70
```

```
ABSORBANCE:
          0.029   0.029   0.029
MEAN:    0.029    SD 0.0005  RSD(%): 1.70
```

7 Run the Standards (Spiked Samples)

1. Aspirate Standard 1 (Sample + Addition 1), then press: **[1] [Calib]**.

   The results are displayed and printed. For example,

```
  CONC (MEAN):  1.0
SD:0.0005  RSD:0.18
```

```
STANDARD 1
ABSORBANCE:
          0.057   0.057   0.057
MEAN:   (ADDITION  APPLIED)
SAMPLE CONC 1.0  SD 0.0001 RSD(%): 0.18
```

2. Aspirate Standard 2 (Sample + Addition 2), then press: **[2] [Calib]**.

   The results are displayed and printed. For example,

```
  CONC (MEAN): 1.0
SD:0.0005  RSD:0.24
```

```
STANDARD 2
ABSORBANCE:
          0.085   0.085   0.086
MEAN:   (ADDITION  APPLIED)
SAMPLE CONC 1.0 SD 0.0002  RSD(%): 0.24
```

RSD Calculations

NOTE: The calculated RSD is obtained using more significant digits than are displayed or printed.

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Running Samples With Flame 9
Using Method of Additions, Autosampler

Overview

Once the system has been powered up and optimized, analyzing samples with an autosampler is simply a matter of loading appropriate solutions into an autosampler tray in a prescribed sequence, entering parameters, then starting the autosampler.

An example of an aspirating sequence for Method of Additions is shown below:

Blank

Sample 1 (Unspiked)
Standard 1 (Spiked Sample = Sample 1 + Spike 1)
Standard 2 (Spiked Sample = Sample 1 + Spike 2)

Sample 2
Standard 1 (Spiked Sample = Sample 2 + Spike 1)
Standard 2 (Spiked Sample = Sample 2 + Spike 2)

etc.

NOTE: The combination of an unspiked sample and 2 standards (spiked sample), constitutes a single sample run. You do not have to load more than one blank. The system automatically runs the blank and performs an autozero before each sample run begins.

This chapter contains an example of how to run AA Flame analyses with an autosampler using Method of Additions. The example takes you step by step through the required procedures and shows you how to display and print results.

To help you learn how to run samples with an autosampler using Method of Additions, it is recommended that you perform the setup procedures in Chapters 3, 4, and 5, then duplicate the procedures given in the example.

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About Method of Additions

If you are familiar with Method of Additions, skip to the Example on page 9-3.

This technique allows you to analyze samples in the presence of an interference and is particularly useful where the sample matrix varies from sample to sample. A typical example would be the determination of trace platinum metals in samples from a smelting operation.

In this technique, the standards are samples that are "spiked" with known amounts of the element of interest. Both the sample and the standards are then diluted to exactly the same volume. After aspirating a blank and autozeroing, these diluted solutions are run in the following sequence:

Sample
Standard 1 (Spiked Sample = Sample 1 + Spike 1)
Standard 2 (Spiked Sample = Sample 1 + Spike 2)

The system creates a calibration curve using these three solutions. It then extrapolates the calibration curve to the baseline and calculates the concentration of the element of interest in the original unspiked sample. For example,

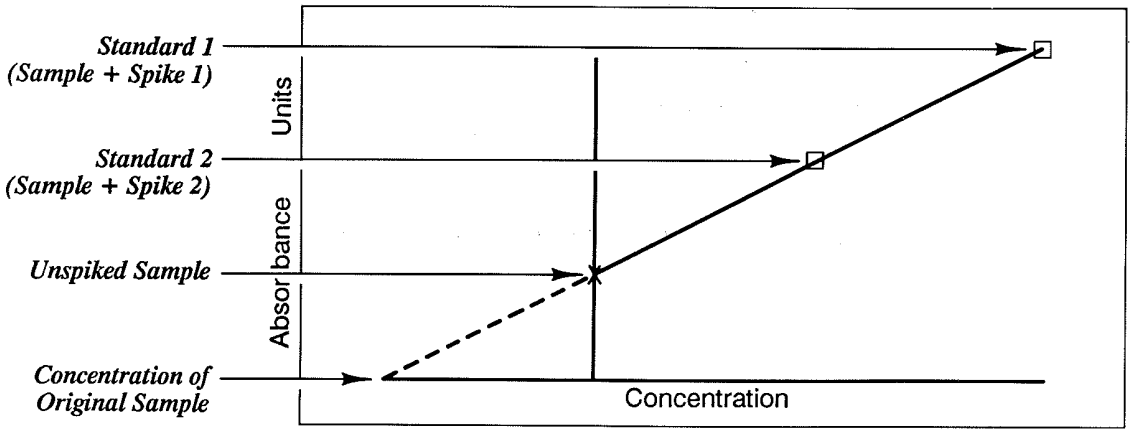


Figure 9-1. Example of Method of Additions Calibration Curve

NOTE: The Method of Additions calibration curve is recalculated whenever a blank, a first addition, or a last addition has been run. This allows a method of additions calibration curve to be aborted without affecting the sample sequence.

A more complete discussion of Method of Additions is given in Chapter 17, Calibration, and in Section 7 (the yellow pages) of the AA Cookbook.

NOTE: All solution concentrations for Method of Additions must be in the linear range.

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EXAMPLE: Determining Copper Using Method of Additions

NOTE: The procedures in this example assume that the setup procedures in Chapters 3, 4, and 5 have been performed, in which case the instrument will be on and the flame ignited.

NOTE: Before starting an autosampler run, turn the autosampler on and make certain that the IEEE- 488 control switch on the rear of the Model 3100 is set to the Down position.

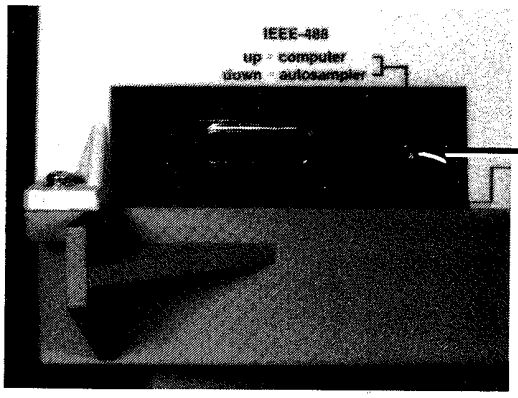


Figure 9-2. IEEE-488 Control Switch

In this example we ran a blank, two unspiked samples, and two standards (spiked samples).

Carry out the following steps to duplicate our example:

1. Prepare solutions
2. Load the autosampler
3. Enter parameters
4. Print the parameters
5. Enter the data collection mode
6. Start the run

Detailed procedures follow.

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1 Prepare Solutions

We used a copper lamp and solutions of copper for our determinations. You don't have to use copper if you don't want to. For your convenience in making substitutions we have included a table of recommended concentrations for Ag, Mg, Mn, and Pb.

1. Prepare about 200 mL of 1% v/v HNO_3 to use as a blank and diluent.
2. Prepare about 200 mL of a Stock Standard of the element of interest.
3. Prepare about 200 mL of sample.

Recommended concentrations for Stock Standards (to be used as spiking solutions) and samples are shown below.

Element	Stock Standard Conc. (mg/L)	Original Sample Conc (mg/L)
Ag	2.0	1.0
Cu	2.0	1.0
Mg	0.2	0.1
Mn	1.0	0.5
Pb	10.0	5.0

Prepare the Unspiked Sample

* Dilute 50 mL of sample to 100 mL with diluent. Our original sample contained 1.0 mg/L of Cu.

Prepare Standard 1 (Addition 1)

* To 50 mL of sample, add 25 mL of Stock Standard and dilute to 100 mL with diluent.

  Since we diluted our stock standard by a factor of 2, we effectively spiked the sample with a solution containing 1 mg/L of Cu.

Prepare Standard 2 (Addition 2)

* To 50 mL of sample, add 50 mL of Stock Standard (effectively 50 mL of sample has been diluted to 100 mL with Stock Standard).

  Effectively, we have spiked the sample with a solution containing 2 mg/L of Cu.

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Calculating Standard Concentrations

In the next section, Entering Parameters, you will have to enter the concentrations for Standards 1 and 2. Use the following formula to calculate these concentrations:

$$\text{Conc. of Std} = \frac{\text{Vol of stock std. x Conc. of stock std.}}{\text{Vol. of sample}}$$

Therefore, for our example, the concentrations of Standards 1 and 2 are calculated as follows:

$$\text{Conc. of Std. 1} = \frac{25\ \text{mL} \times 2\ \text{mg/L}}{50\ \text{mL}} = 1\ \text{mg/L}$$

$$\text{Conc. of Std. 2} = \frac{50\ \text{mL} \times 2\ \text{mg/L}}{50\ \text{mL}} = 2\ \text{mg/L}$$

2 Load the Autosampler

Each autosampler tray contains an opening for a large beaker (position 0). The AS-51 contains fifty four 15-mL positions. The AS-90 contains eight 50-mL positions and ninety eight 15-mL positions.

> **NOTE:** Locations 1-8 are not used for Method of Additions and should be left empty.

To demonstrate how to load the autosampler tray to run two analyses:

1. Place the blank in position 0 (the large beaker).
2. Place the unspiked sample in positions 9 and 12.
3. Place Standard 1 in positions 10 and 13.
4. Place Standard 2 in positions 11 and 14.

The above autosampler loading sequence is shown in the following table:

A/S Position	Contents
0	Blank
9	Sample 1 (Unspiked Sample)
10	Standard 1 (Sample 1 + Spike 1)
11	Standard 2 (Sample 1 + Spike 2)
12	Sample 2 (Unspiked Sample 2)
13	Standard 1 (Sample 2 + Spike 1)
14	Standard 2 (Sample 2 + Spike 2)

NOTE: It is only necessary to load the blank once (see A/Z Frequency on page 9-9).

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3 Enter Parameters

1. Press: **[Param Entry]**.

   If you have just aligned a copper lamp, the Lamp Current screen appears as shown below:

```
LAMP CUR. (0-50mA):
        15.0
```

   If you have aligned another lamp, its Lamp Current appears on the above screen.

2. Press: **[Enter]**.

   If you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen as shown below.

```
INT. TIME (.1-60sec)
        0.30
```

3. Press: **[3]**. The screen changes to:

```
INT. TIME (.1-60sec)
        3
```

4. Press **[Enter]**. The following screen appears:

```
REPLICATES (1-99):
        01
```

5. Press: **[3]**, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

6. To select Method of Additions (3), press: **[3]** , then press: **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```

7. To select Flame (1), Press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
        _____
```


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8. If you are using Cu, press: **[1] [.] [0]** , then press: **[Enter]**. The following screen appears:

```
STD 2 (.0001-9999) :
      ------
```

NOTE: If you are using another element, type in the concentration of Standard 1 for that element, then press: **[Enter]**.

9. If you are using Cu, press: **[2] [.] [0]**, then press: **[Enter]**. The following screen appears:

```
STD 3 (.0001-9999) :
      ------
```

NOTE: If you are using another element, type in the concentration of Standard 2 for that element, then press: **[Enter]**.

10. Press:**[Enter]** until the following screen appears:

```
 SELECT AUTOSAMPLER
AS51 (1)  AS90 (2):1
```

11. Enter a number corresponding to the autosampler attached, then press: **[Enter]**.

If an AS-51 is attached the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-54): 09
```

If an AS-90 is attached the following screen appears:

```
LAST SAMPLE POSITION
 ENTER (09-106): 09
```

12. Press: **[12]**, then press **[Enter]**. The following screen appears:

```
   A/Z FREQUENCY
 ENTER (01-99) 99
```

13. Press: **[1] [Enter]**. This will Autozero the instrument in between each sample run.

NOTE: An explanation of A/Z Frequency is given on page 9-9.

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4 Activate the Printer (if one is attached)

* Press: **[Print]**.

Since you are in the Parameter Entry mode, the parameters will be printed.

Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

5 Enter the Data Collection Mode

* Press: **[Data]**. The Data screen appears.

```
   ABS (MEAN):_______
SD:_____ RSD_____
```

As measurements are made, the results appear on the above screen.

6 Start the Run

* Press: **[Auto Sampler]**.

The run starts and as each replicate is measured, its absorbance appears briefly on the above screen. After the last replicate is measured, the results are displayed and printed.

```
 CONC (MEAN): 1.0
SD:0.0002  RSD:0.21
```

```
STANDARD 2
ABSORBANCE:
          0.085  0.085  0.086
MEAN:  (ADDITION  APPLIED)
SAMPLE CONC 1.0 SD:0.0002  RSD(%): 0.21
```


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Running a Single Vial

The probe can be moved to any vial position (i.e., to run a single vial) by entering the desired vial number followed by pressing: **[Auto Sampler]**.

If the probe is down, the probe moves up, over to the specified position and down into that vial. Thus the vial in the specified position will be aspirated.

If the probe is up, it is moved to the specified position but stays up, i.e., the vial in the specified position will not be aspirated. To aspirate the sample in the specified position, move the probe down by pressing **[.] [Auto Sampler]**. Pressing **[.] [Auto Sampler]** again will raise the probe.

Autozero Frequency

The Autozero Frequency screen show below is specific for Method of Additions when using an autosampler.

```
A/Z FREQUENCY
ENTER (01-99) 99
```

This screen allows you to specify how often you want the system to aspirate the blank and perform an autozero.

For example, if you specify 1, the system will aspirate the initial blank, perform an Autozero, then repeat the blank and Autozero after each sample run (a Method of Additions sample run consists of an unspiked sample and spiked samples). If you specify 3, the system will aspirate the initial blank, perform an Autozero, then repeat the blank and Autozero after every three sample runs.

RSD Calculations

NOTE: The calculated RSD is obtained using more significant digits than are displayed or printed.

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Running Samples in Emission Mode 10

Overview

The 3100 also performs flame emission measurements at a fixed wavelength. This option is useful when you do not have a lamp for the element you wish to determine or if you are determining elements such as Na, K, Ca, or Ba, which are sensitively determined by emission.

Prerequisites

The procedures in this chapter assume that you are familiar with the 3100 operating procedures. If not please read Chapters 2 through 5.

Setting Up for Emission

NOTE: Refer to Standard Conditions in the AA Cookbook to find the recommended settings for wavelength, slit width, and flame type. Use these values to make appropriate settings.

Carry out the following steps to set up for running analyses in Emission Mode:

1. Ignite the flame.
2. Enter parameters.
3. Set the wavelength.
4. Set the slit width and height.
5. Maximize the energy

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1 Ignite the Flame

1. Turn on the instrument and wait for the following screen to appear.

```
PERKIN-ELMER
MODEL 3100
```

2. Press **[Em]**.

3. Ignite the flame using the procedures on pages 5-4 and -5 in Chapter 5, Igniting and Optimizing the Flame.

2 Enter Parameters

1. Press: **[Param Entry]**. The following screen appears:

```
LAMP CUR. (0-50mA):
        0.0
```

2. Press: **[Enter]**. The INT. TIME screen appears:

```
INT. TIME (.1-60sec)
        0.10
```

3. Press: **[0.3]**, then press: **[Enter]**. The following screen appears:

```
REPLICATES (1-99):
        01
```

4. Enter the number of replicates you want to run, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

5. Enter the number corresponding to the calibration type desired, then press: **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```


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6. Press: **[Enter]** to select flame. The following screen appears:

```
STD 1 (.0001-9999) :
       ------
```

7. Enter the concentration of your first standard, then press: **[Enter]**. The screen for the next standard appears:

```
STD 2 (.0001-9999) :
       ------
```

8. Enter the concentration of additional standards (see Chapter 2, Entering Parameters).

   After you have entered the concentrations of all standards the following screen appears:

```
RSLP (.0001-9999) :
       ------
```

9. If you want to run a Reslope Standard, enter its concentration, then press **[Enter]**. If you don't want to run a Reslope Standard, just press: **[Enter]**.

3 Set the Wavelength

1. Locate the wavelength indicator on the top left side of the instrument (see Figure 3-4).
2. Locate the Wavelength Control Dial on the left side of the instrument (see Figure 3-5).
3. Set the wavelength as close as possible to the desired wavelength with the Wavelength Control Dial.

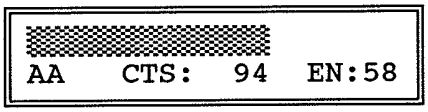
4 Set the Slit Width and Height

1. Move the Slit Width Lever to the position recommended in the AA Cookbook (see Figure 3-4).
2. Move the Slit Height Lever to the High position (see Figure 3-4).

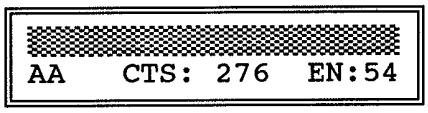
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5 Maximize the Energy

1. Aspirate the highest standard.
2. Press: **[Energy] [Gain]**. A screen similar to the following appears:



3. If the bar chart is off scale,



   or does not extend about half-way across the screen, press: **[Gain]** to bring the bar on scale. Proceed to Step 4.

   If the system cannot find the wavelength, the energy remains at 0 or a very low level and the bar shown on the screen above is absent.

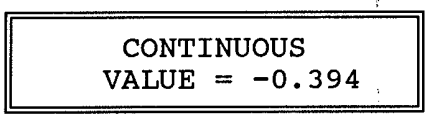
4. Slowly rotate the Wavelength Control Dial about ½ nm to either side until you obtain maximum energy and number of counts (CTS).

   NOTE: An energy setting within 3 or 4 counts of the maximum is satisfactory.

5. If the bar chart goes off scale, press: **[Gain]** to bring it on scale.

6. Press: **[Cont]**.

   The Continuous screen appears and displays an absorbance value. For example:



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7. Aspirate the blank, then press: **[A/Z]**.

   Autozero is performed and a display similar to the following appears:

```
CONTINUOUS
VALUE = 0.000
```

8. Aspirate the highest standard.

9. Adjust the burner with the Horizontal, Vertical, and Rotational Adjustment Knobs (see Figure 5-1) until you have obtained a maximum signal. If the displayed value is larger than 1.50, press **[Gain]** to bring the signal back on scale.

10. Adjust the nebulizer using the procedure on page 5-6.

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Running Samples

1. Press: **[Param Entry]**.
2. Get to the INT. TIME screen, enter the desired Integration Time, then press **[Enter]**.
3. Press: **[Print]** **[Data]**.

   NOTE: Do not press **[Print]** if a printer is not attached.

   The Data screen appears:

```
   ABS (MEAN):_____
SD:_____ RSD_____
```

   As measurements are made, the results appear on the above screen.

4. Aspirate the blank, then press: **[A/Z]**. The results are displayed and printed.
5. Aspirate Standard 1 and press **[Calib]**.

   After the last replicate is measured the results are displayed and printed.

6. Repeat step 5 for each additional standard.
7. Aspirate Sample #1 and press: **[Read]**.

   After the last replicate is measured the results are displayed and printed.

8. Repeat step 7 for each sample.

RSD Calculations

NOTE: The calculated RSD is obtained using more significant digits than are displayed or printed.

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Setting Up for MHS-10 11

This chapter contains the following set up procedures:

- Aligning the Quartz Cell.
- Igniting the Flame.

The procedures which follow assume that the MHS-10 accessory has been installed using the procedures that start on page 14-10 in Chapter 14, Installation Procedures.

> **NOTE:** MHS-10 analyses require the use of a 10 cm single slot air-acetylene burner head (PN 0040-0266). If another Burner Head is installed, it must be replaced by the correct burner. Refer to Chapter 14, Installation Procedures, for appropriate procedures.

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Aligning the Quartz Cell

The quartz cell should be aligned when first installed or whenever it is reinstalled after removal.

There are two alignment procedures involved in aligning the quartz cell. The first is a coarse alignment using a light source that gives a visible light beam, such as a copper hollow cathode lamp. This is followed by a fine alignment using the lamp for the element to be determined.

Coarse Alignment Procedure

1. Figure 11-1 shows a front view of the Quartz Cell out of the beam.

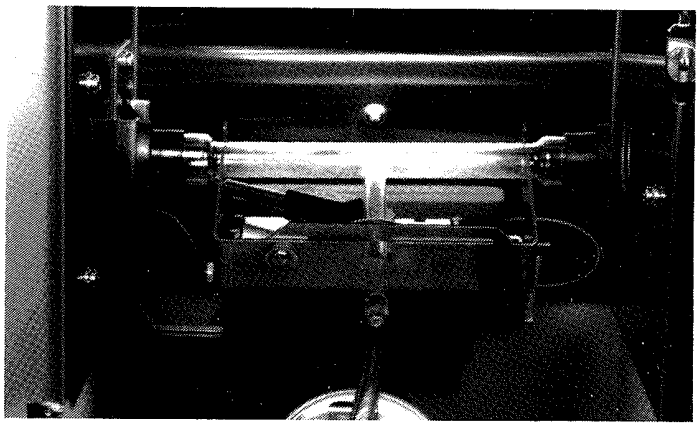


Figure 11-1. Quartz Cell out of the Beam

2. If the cell is out of the beam, move it into the beam by gently raising the Capture Arm Handle. Figure 11-2 shows a side view of the Quartz Cell in the beam.

3. Install and align a copper hollow cathode lamp using the procedures in Chapter 3, Installing and Aligning Lamps.

4. Holding a piece of matte white paper at the left end of the quartz cell, adjust the burner's vertical, horizontal, and rotational knobs (see Figure 5-1) until the light beam passes through the cell.

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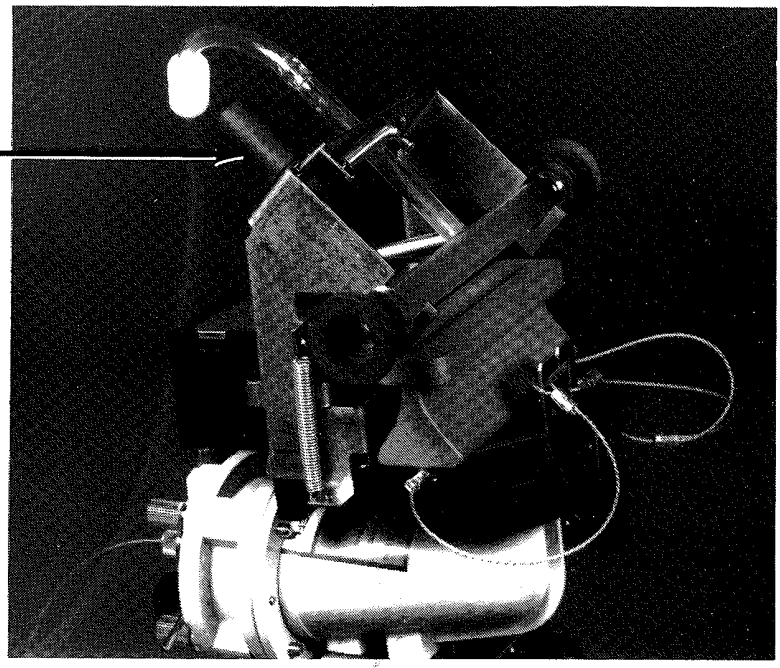


Figure 11-2. Quartz Cell in the Beam

5. Press: **[Cont]**, then **[A/Z]**.

6. Adjust the burner's vertical, horizontal, and rotational knobs until minimum absorbance is obtained.

Fine Alignment Procedure

IMPORTANT: This fine alignment procedure should only be performed with the proper light source for the element to be determined.

1. Install the lamp for the element to be determined. Make a note of the recommended continuous lamp current value on the lamp label.

   NOTE: If you are using an EDL, please refer to Chapter 3, pages 8 through 17.

2. Align the lamp using the procedures in Chapter 3, Installing and Aligning Lamps. On the Lamp Current screen, enter the continuous lamp current shown on the lamp label.

3. Align the cell using steps 5 and 6 in the Coarse Alignment Procedure above.

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Before you Ignite the Flame

Before you ignite the flame, make certain that:

1. A 10 cm single slot burner head (PN 0040-0266) is installed and securely seated by tightening the Burner Head Locking Ring.
2. The burner head tie downs are in place.
3. The burner head interlock pin is in place.
4. The burner end cap and nebulizer are securely attached to the burner.
5. The MHS-10 cell holder is in the "down" position, not directly above the burner head.
6. The fuel/oxidant lines are securely attached to the burner and nebulizer.
7. The drain interlock connector is connected.
8. Water is present in the Drain System Float Assembly.
9. The drain hose is securely attached to the burner end cap.
10. The thermal shield interlock connector is connected.
11. The ignitor connector is attached to the ignitor assembly.

If you are unfamiliar with procedures for performing the above, please refer to Chapter 4, Before You Ignite the Flame.

Igniting the Flame

1. Turn on the vent.
2. Set the acetylene tank output pressure from 85 to 100 kPa (12 to 14 psig).

> **IMPORTANT:** Change the tank when the tank pressure falls to 520 kPa (75 psig). If the pressure falls below this level, acetone may be carried over into the burner regulator, possibly damaging valves and tubing. Such damage is not covered by the instrument warranty.

3. Set the air supply pressure to between 345 and 450 kPa (50 to 65 psig).
4. Turn the Oxidant Switch on the Pneumatics Control Panel to Air (see Figure 5-2).
5. Adjust the air flow to about 4 units on the flowmeter using the Oxidant Control Knob on the Pneumatics Control Panel (see Figure 5-2).

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6. Adjust the acetylene flow to between 2 and 2.5 units on the flowmeter using the Fuel Control Knob on the Pneumatics Control Panel (see Figure 5-2).

7. Press the Ignite Button on the Pneumatics Control Panel.

   NOTE: If the gases are left on for 15 seconds without igniting them, the red interlock light will come on and the system will turn off the gas flows. If this happens, turn the gas control switch Off, then repeat steps 5 through 7 above.

   NOTE: When gases are first turned on, the flame may not light the first time due to air in the supply lines. If the flame does not ignite, turn the gas control switch Off, then back to Air and press the Ignite button.

   NOTE: If the interlocks are not satisfied, the flame will not ignite and the interlock light will turn on. If this happens, please refer to Chapter 15, Hardware Troubleshooting.

8. Once the flame has ignited, use the Fuel Control Knob to reduce the acetylene flow to about 1 unit on the flowmeter, and reset the Oxidant Control Knob to reduce the air flow to approximately 2 units.

9. Move the quartz cell into the flame once the burner gas flows have been reduced.

10. Allow the quartz cell to heat up for 10 to 15 minutes before performing an analysis.

11. Fill the reductant reservoir with enough reductant to cover the pickup tube.

12. Turn on the inert gas supply and adjust the pressure to 36 psi (250 kPa).

WARNING: Because the vapors vented from the MHS-10 are potentially toxic, and because hydrogen is liberated during the determination, continuous ventilation must be provided in the operating area. Details of suitable venting systems and safety procedures are provided in "Preparing Your Laboratory for Perkin-Elmer Atomic Spectroscopy Instruments," which is included with this product. See also Hazards and Safety Practices in this manual, for information on the handling of compressed gases.

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Running Samples With MHS-10 12

Overview

This chapter contains general procedures for running MHS-10 analyses using Linear/Non-Linear Calibration and Method of Additions.

Once the MHS-10 system has been set up, analyzing samples is simply a matter of entering parameters, then running solutions in a sequence that depends on the type of calibration specified, i.e., Linear/Non-Linear or Method of Additions.

An example of a running sequence when using Linear/Non-Linear calibration is shown below.

Blank
Standard 1
Standard 2
↓
Standard 8
Sample 1
Sample 2
↓
Sample 20
Reslope Standard
Sample 21
Sample 22
etc.

For Linear or Non-Linear calibration, you can aspirate a Reslope Standard at any point after the first sample is run.

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An example of a running sequence for Method of Additions is shown below:

Blank

Sample 1 (Unspiked)
Standard 1 (Spiked Sample = Sample 1 + Spike 1)
Standard 2 (Spiked Sample = Sample 1 + Spike 2)

Sample 2
Standard 1 (Spiked Sample = Sample 2 + Spike 1)
Standard 2 (Spiked Sample = Sample 2 + Spike 2)

Sample #3
etc.

Analyzer Assembly

The MHS-10 Analyzer Assembly is used to run samples with MHS-10. The individual components are illustrated in Figure 12-1.

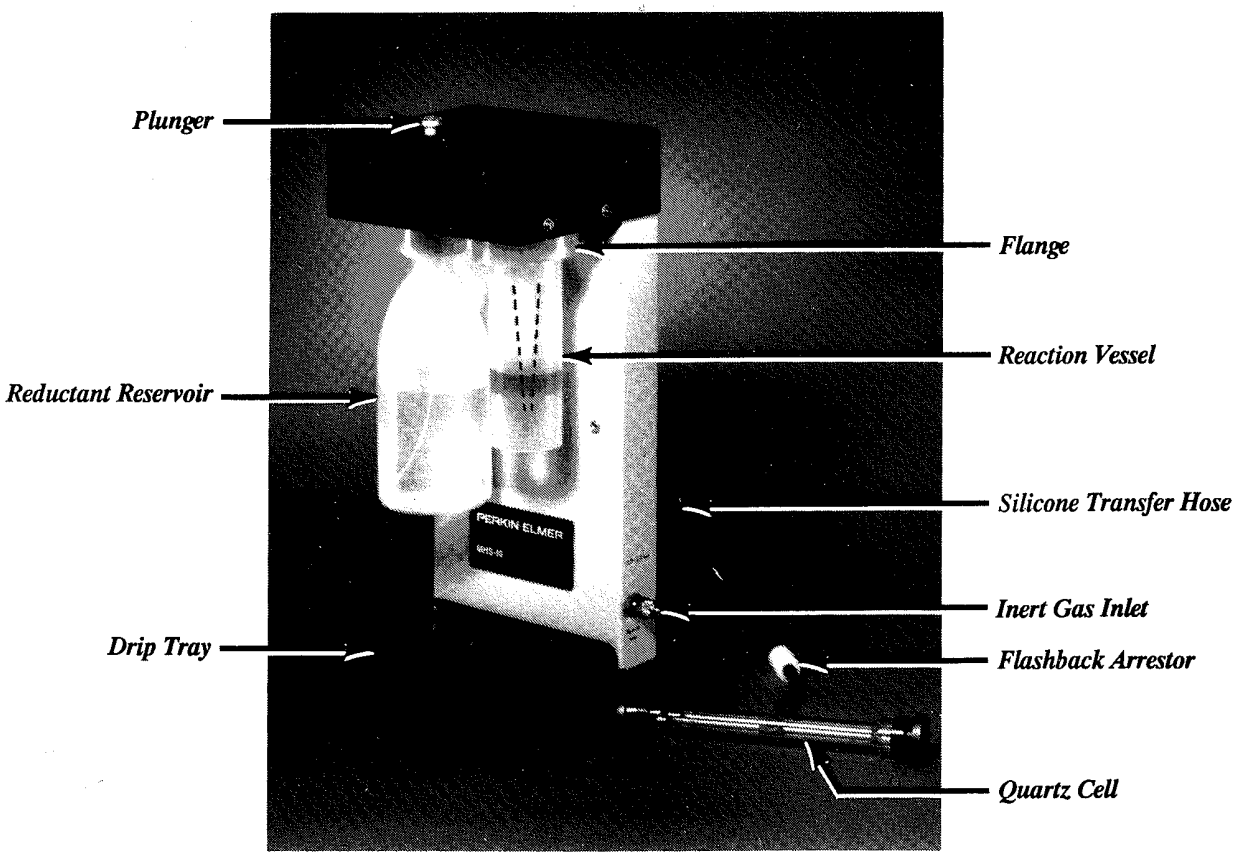


Figure 12-1. Analyzer Assembly

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Running Samples Using Linear/Non-Linear Calibration

The procedures which follow assume that you have set up the MHS-10 using the procedures in the previous chapter. The flame will therefore be on and the quartz cell will be in the beam.

Carry out steps **1** through **8** to run samples with MHS-10 using Linear or Non-Linear Calibration.

1 Enter Parameters

1. Press: **[Param Entry]**.

   Since you have just aligned a lamp, the Lamp Current screen appears and shows the lamp current you used. For example,

```
LAMP CUR. (0-50mA):
        15.0
```

2. Press: **[Enter]**.

   Since you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen. For example,

```
INT. TIME (.1-60sec)
        0.3
```

3. Enter an integration time of 20 to 30 seconds to insure detection of the absorbance peak maximum, then press: **[Enter]**.

   NOTE: If you are determining mercury using stannous chloride as the reductant, a longer integration time (40 to 50 seconds) may be necessary.

   The following screen appears:

```
REPLICATES (1-99):
        01
```

4. Enter the number of replicates you want to run, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

5. For Non-Linear or Linear calibration, press: **[1]** or **[2]**, then press **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```


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6. For a measurement type, enter a 2 or 3 depending on whether you want the system to make calculations using peak height (normally used), or area, then press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
      ------
```

7. Enter the concentration of the first standard then press: **[Enter]**.

   The screen for the second standard appears:

```
STD 2 (.0001-9999) :
      ------
```

You can use up to eight standards, although three are usually adequate.

When you have entered the concentration of the last standard you want to run, the screen for the next standard appears:

If you press **[Enter]** without entering a standard concentration, the system skips over the remaining standard screens to the next parameter, Reslope Standard.

```
RSLP (.0001-9999) :
      ------
```

8. If you will be running a Reslope Standard, enter its concentration , then press: **[Enter]**. If not, just press **[Enter]**.

Parameter entry is now complete. Proceed to the next step.

2 Activate the Printer (if one is attached)

1. Press: **[Print]**.

   Since you are still in the Parameter Entry mode, the parameters are printed.

   Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

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3 Enter the Data Collection Mode

1. Press: **[Data]**.

   The Data screen appears:

```
   ABS (MEAN):_______
SD:_____ RSD_____
```

   As measurements are made, the results appear on the above screen.

4 Run the Blank

1. Pipette a 10-50 mL aliquot of blank into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.

   As each replicate is measured, its absorbance appears on the screen.
4. After the last replicate is measured, press: **[A/Z]**.

5 Run the Standards

1. Pipette a 10-50 mL aliquot of standard into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.
4. After the last replicate is measured, press the number of the standard and **[Calib]**. For example, **[1] [Calib]**.

   The results are displayed and printed.
5. Repeat steps 1 through 4 for each additional standard.

   The results are displayed and printed.

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6 Run the Samples

1. Pipette a 10-50 mL aliquot of sample into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.
4. After the last replicate is measured, the results are displayed and printed.
5. Repeat steps 1 through 3 for each additional sample.

7 Run the Reslope Standard

You can run a Reslope Standard any time after the first sample is run.

To run a Reslope Standard:

1. Pipette a 10-50 mL aliquot of blank into the rinsed reaction vessel and attach to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly and press: **[Read]**.
3. Repeat steps 1 and 2 for each blank replicate.
4. After the last blank replicate is measured, press: **[A/Z]**.
5. Pipette a 10-50 mL aliquot of the reslope standard into the rinsed reaction vessel and attach to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
6. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds), and press: **[Read]**.
7. Repeat steps 1 and 2 for each reslope replicate.
8. After the last reslope replicate is measured, press: **[Reslope]**.

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8 Run the Remaining Samples

Use the procedures previously described on page 12-6.

Generating a New Calibration Curve

Depending on circumstances, you may wish to generate a new calibration curve. If so, use the following procedure:

NOTE: If desired, you may rerun your blank before continuing with the procedure below.

1. Pipette a 10-50 mL aliquot of Standard 1 into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly and press **[Read]**.
3. Repeat steps 1 and 2 for each replicate.
4. After the last replicate is measured, press: **[0] [Calib]**.

   This erases the original calibration curve and applies the first standard.
5. Pipette a 10-50 mL aliquot of Standard 2 into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
6. Simultaneously hold down the plunger on the analyzer assembly and press: **[Read]**.
7. Repeat steps 5 and 6 for each standard replicate.
8. After the last replicate is measured, press: **[2] [Calib]**.
9. Repeat steps 6 through 8 for each additional standard, incrementing the standard number by 1.

Rerunning a Single Standard

1. Pipette a 10-50 mL aliquot of the standard into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.
4. After the last replicate is measured, press the number of the standard and **[Calib]**. For example, **[3] [Calib]**.

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Running Samples Using Method of Additions

The procedures which follow assume that you have set up the MHS-10 using the procedures in the previous chapter. The flame will therefore be on and the quartz cell will be in the beam.

Carry out steps **1** through **7** to run samples with MHS-10 using Method of Additions.

Prerequisites

The following procedures assume that you are familiar with Method of Additions. If not, please read Chapter 8. It is suggested that you carry out the example shown.

1 Prepare Solutions

Please refer to Chapter 8, page 4 for an example of how to prepare Method of Addition solutions.

Another example would be the following:

1. Divide the sample into three equal parts.
2. Dilute one part of the sample with an equal volume of diluent. This is the Unspiked Sample.
3. To another part of the sample, add 1/2 the Unspiked Sample vloume of Stock Standard.and dilute to the same volume with diluent. This is Standard 1.
4. To the last part of the sample, add an equal volume of stock standard containing the element of interest. This is Standard 2.

   NOTE: All solution concentrations for Method of Additions must be in the linear range.

Calculating Standard Concentrations

In the next section, Entering Parameters, you will have to enter the concentrations for Standards 1 and 2. Use the following formula to calculate these concentrations:

$$\text{Conc. of Std} = \frac{\text{Vol of stock std. x Conc. of stock std.}}{\text{Vol. of sample}}$$

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2 Enter Parameters

1. Press: **[Param Entry]**.

   Since you have just aligned a lamp, the Lamp Current screen appears and shows the lamp current you used. For example,

```
LAMP CUR. (0-50mA):
        15.0
```

2. Press: **[Enter]**.

   Since you have just aligned a lamp, the Integration Time you used appears on the Integration Time screen. For example,

```
INT. TIME (.1-60sec)
        0.3
```

3. Enter an integration time of 20 to 30 seconds to insure detection of the absorbance peak maximum, then press: **[Enter]**.

   NOTE: If you are determining mercury using stannous chloride as the reductant, a longer integration time (40 to 50 seconds) may be necessary.

   The following screen appears:

```
REPLICATES (1-99):
        01
```

4. Enter the number of replicates you want to run, then press **[Enter]**. The following screen appears:

```
CAL: NON LIN (1),
LIN (2), ADD (3): 1
```

5. For Method of Additions, press: **[3]**, then press **[Enter]**. The following screen appears:

```
FLAME(1),PK AREA(2),
 PEAK HEIGHT(3), 1
```

6. For a measurement type, enter a 2 or 3 depending on whether you want the system to make calculations using peak height (normally used), or area, then press: **[Enter]**. The following screen appears:

```
STD 1 (.0001-9999) :
        ------
```


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7. Enter the concentration of Standard 1, then press: **[Enter]**. The following screen appears:

```
STD 2 (.0001-9999) :
      ------
```

8. Enter the concentration of Standard 2, then press: **[Enter]**. The following screen appears:

```
STD 3 (.0001-9999) :
      ------
```

9. Press:**[Enter]**.

3 Activate the Printer (if one is attached)

1. Press: **[Print]**.

   Since you are still in the Parameter Entry mode, the parameters are printed.

   Having activated the printer, results will be printed after the absorbance is measured for each aspirated solution.

4 Enter the Data Collection Mode

1. Press: **[Data]**.

   The Data screen appears:

```
   ABS (MEAN):_______
SD:_____ RSD_____
```

   As measurements are made, the results appear on the above screen.

5 Run the Blank

1. Pipette a 10-50 mL aliquot of blank into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.

   As each replicate is measured, its absorbance appears on the screen.
4. After the last replicate is measured, press: **[A/Z]**.

   The results are displayed and printed.

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6 Run the Unspiked Sample

1. Pipette a 10-50 mL aliquot of Unspiked Sample into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.

   As each replicate is measured, its absorbance appears on the screen.

   After the last replicate is measured, the absorbance for that sample is displayed and printed.

7 Run the Standards (Spiked Samples)

1. Pipette a 10-50 mL aliquot of Standard 1 into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
2. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
3. Repeat steps 1 and 2 for each replicate.

   As each replicate is measured, its absorbance appears on the screen.
4. After the last replicate is measured, press: **[1] [Calib]**.

   The results are displayed and printed.
5. Pipette a 10-50 mL aliquot of Standard 2 into the rinsed reaction vessel and attach the vessel to the analyzer assembly. Allow air to be purged from the flask for about 10-15 seconds.
6. Simultaneously hold down the plunger on the analyzer assembly (for no more than 15 seconds) and press: **[Read]**.
7. Repeat steps 5 and 6 for each replicate.

   As each replicate is measured, its absorbance appears on the screen.
8. After the last replicate is measured, press: **[2] [Calib]**.

   The results are displayed and printed.
9. Repeat steps 5 through 8 for any additional standards, incrementing the standard count by 1.

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Powering Down the System 13

Procedure

1. Aspirate water for a few minutes to clean the burner head.
2. Turn the gas control switch on the Pneumatics Control Panel to Off.
3. Close all gas source valves.
4. Turn the gas control switch to Air (or N_2O if used) to allow acetylene and oxidant to bleed. The red interlock light on the Ignite button should turn on.
5. Turn the gas control switch to Off.
6. Turn the instrument off.
7. Turn the vent off.

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Installation Procedures 14

This chapter includes the following procedures:

- Removing and installing a burner head
- Removing and installing the flow spoiler
- Removing and installing the impact bead
- Installing the MHS-10 quartz cell and holder
- Installing the MHS-10 analyzer assembly
- Installing an autosampler
- Installing a strip chart recorder
- Installing a serial printer

NOTE: Power requirements are given on page 14-14. A Shipping Check List is provided on Page 14-16.

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Removing a Burner Head

1. Extinguish the flame, turn off the gases, and let the burner head cool.
2. Open the burner compartment door to expose the Burner Assembly.

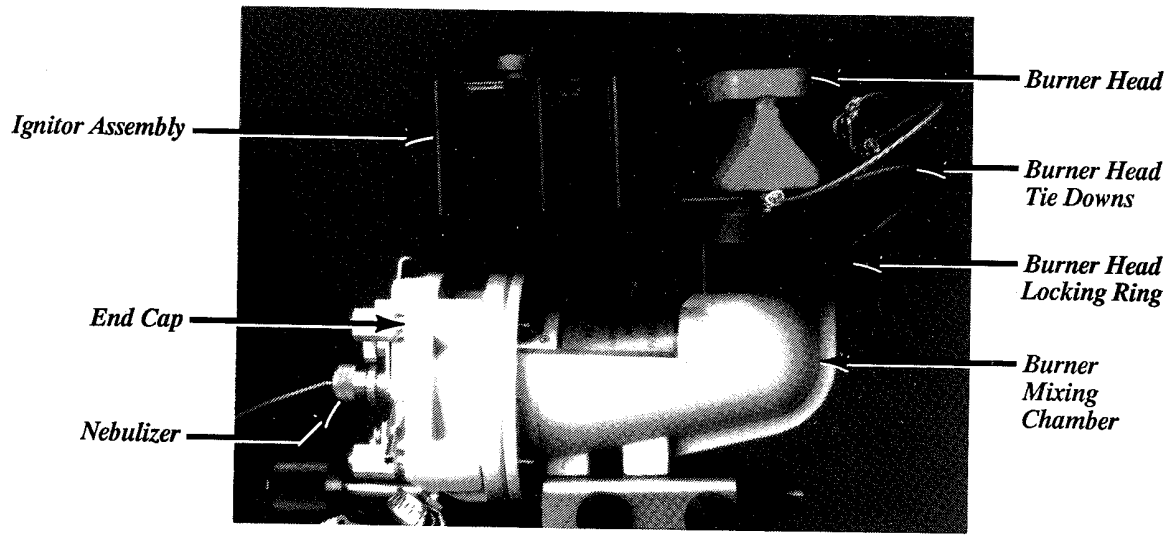


Figure 14-1. Burner Assembly

NOTE: The relative positions of the Burner Head Tie Downs, Locking Ring, and Mixing Chamber are the same for Flame and MHS-10.

3. Remove the Burner Head Tie Downs.
4. Remove the Burner Head Interlock Pin (located on the left side of the Ignitor Assembly) by pulling it up and out.

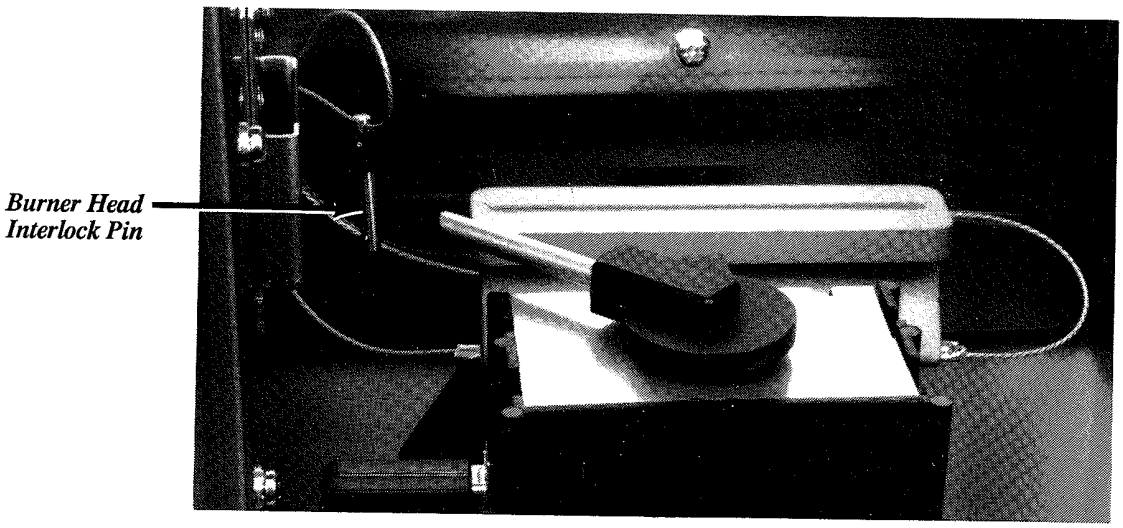


Figure 14-2. Burner Head Interlock Pin

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5. Loosen the Burner Head Locking Ring (see Figure 14-1) by turning it counter-clockwise. Hold the Ignitor Assembly in place and pull the Burner Head out of the Ignitor Assembly.

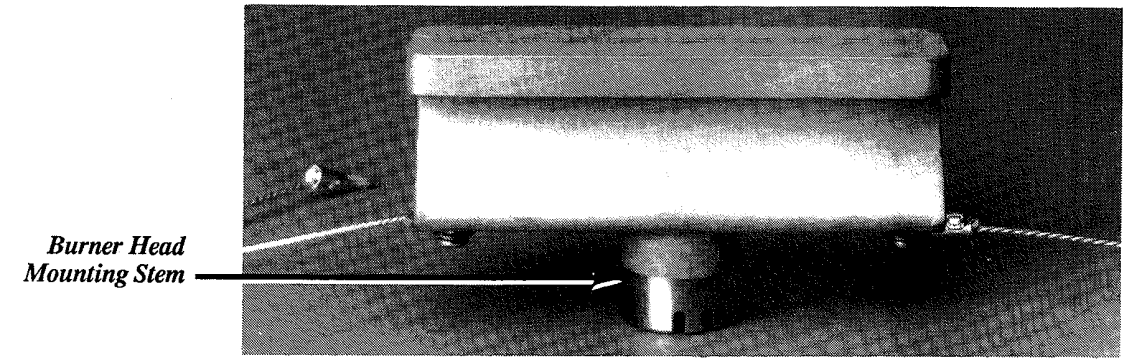


Figure 14-3. Burner Head

NOTE: Loosening the Burner Head Locking Ring may free the Ignitor Assembly. The Burner Head Locking Ring remains attached to the Ignitor Assembly (see Figure 14-4).

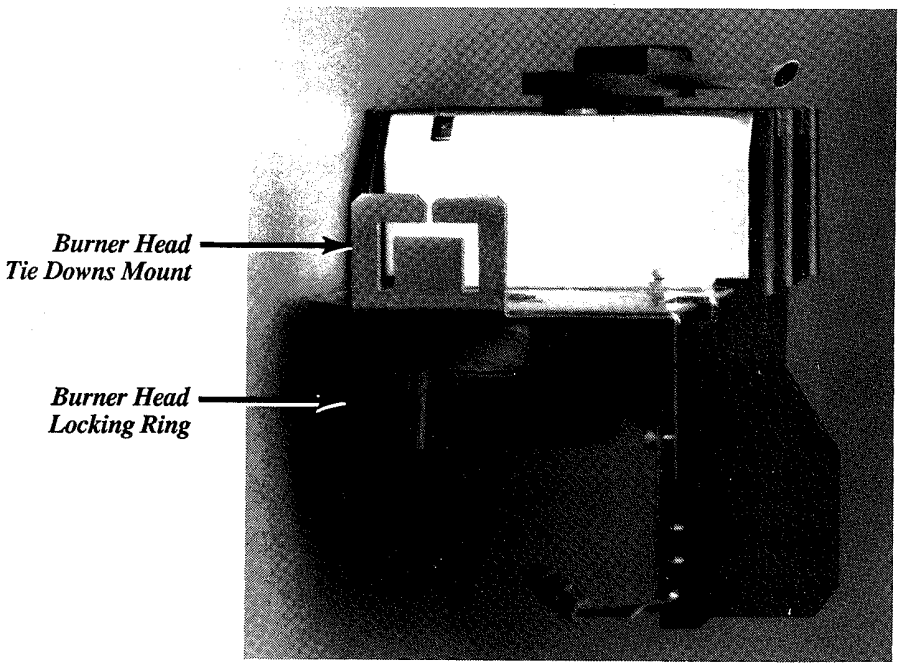


Figure 14-4. Ignitor Assembly -- Side View

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Installing a Burner Head

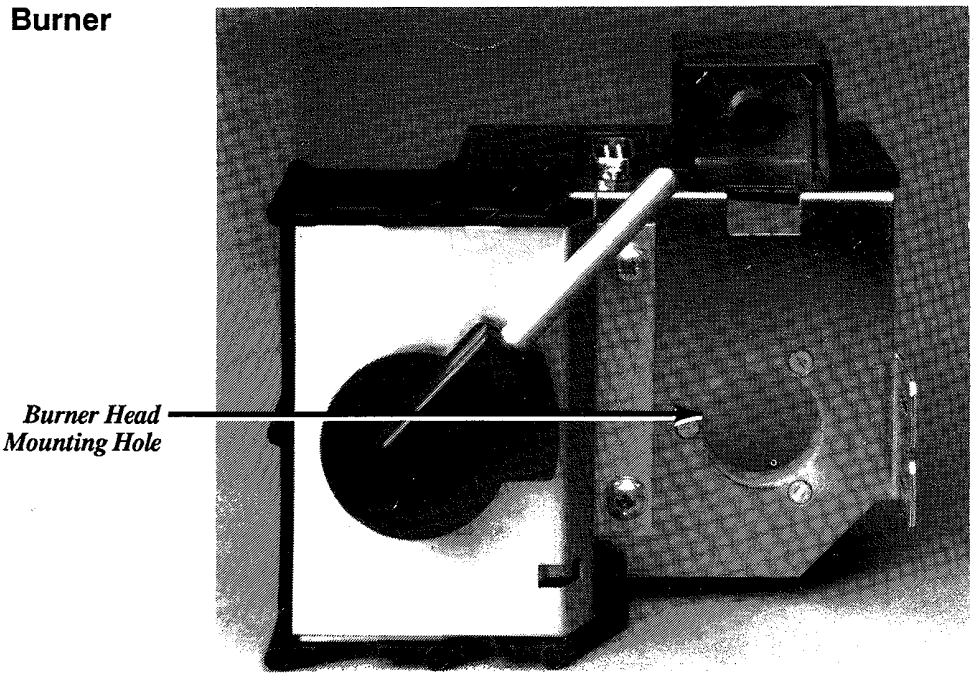


Figure 14-5. Ignitor Assembly --Top View

1. Examine the Burner Head Mounting Hole in the Ignitor Assembly (see Figure 14-5). Make certain that the O-ring is properly seated.

2. Firmly push the Burner Head Stem into the Burner Head Mounting Hole through the O-ring until it is down as far as it will go, making certain that you do not damage the O-ring.

3. If the Ignitor Assembly has been removed, reinstall it by placing the Burner Head Locking Ring on the Burner Mixing Chamber Threads and tightening the Burner Head Locking Ring securely.

4. Attach the Burner Head Tie Downs to the Burner Head Tie Downs Mount (see Figure 14-4).

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5. Place the Burner Head Interlock Pin fully into the hole at the left of the burner compartment (see Figure 14-6).

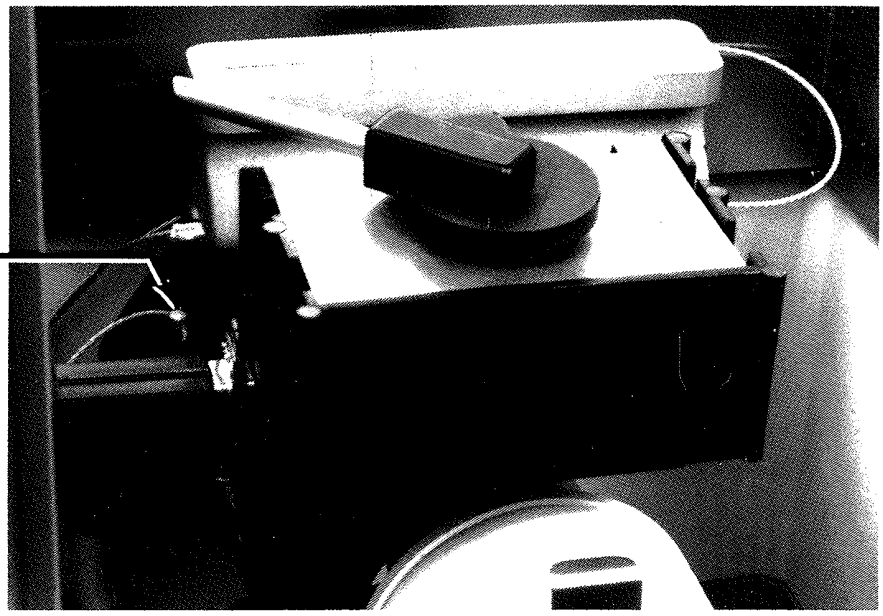


Figure 14-6. Burner Head Interlock Pin in Place

NOTE: The flame will not ignite unless this pin is fully inserted.

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Removing the Flow Spoiler

1. Extinguish the flame, turn off the gases, and let the burner head cool.
2. Remove the four knurled screws from the Burner End Cap (see Figure 14-7).

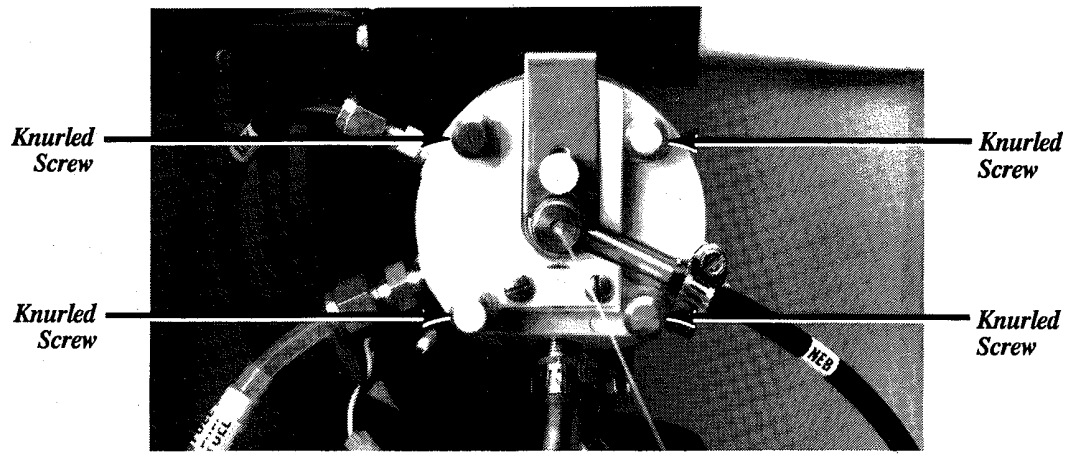


Figure 14-7. Burner End Cap

3. Remove the Burner End Cap exposing the Flow Spoiler (see Figure 14-8).

   NOTE: It is not necessary to disconnect the hoses from the end cap.

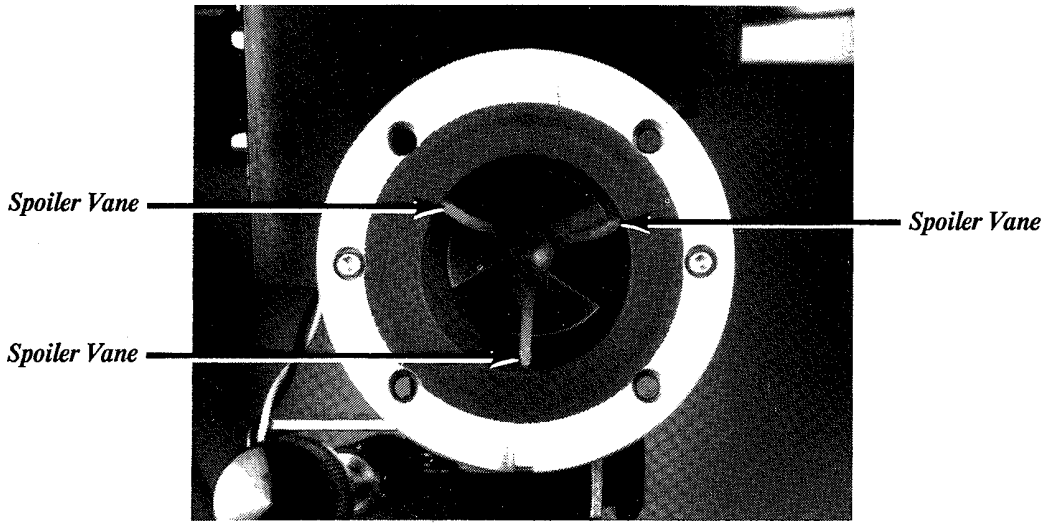


Figure 14-8. Flow Spoiler

4. Push sideways on one of the three vanes and pull the spoiler out of the spray chamber.

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Installing the Flow Spoiler

1. Position one of the spoiler vanes downward as shown in Figure 14-8.
2. Press the center of the Flow Spoiler with your index finger and push it into the spray chamber until firmly seated.
3. Examine the inside of the end cap (see Figure 14-9). Make certain that the end cap O-ring is properly seated in the end cap groove.

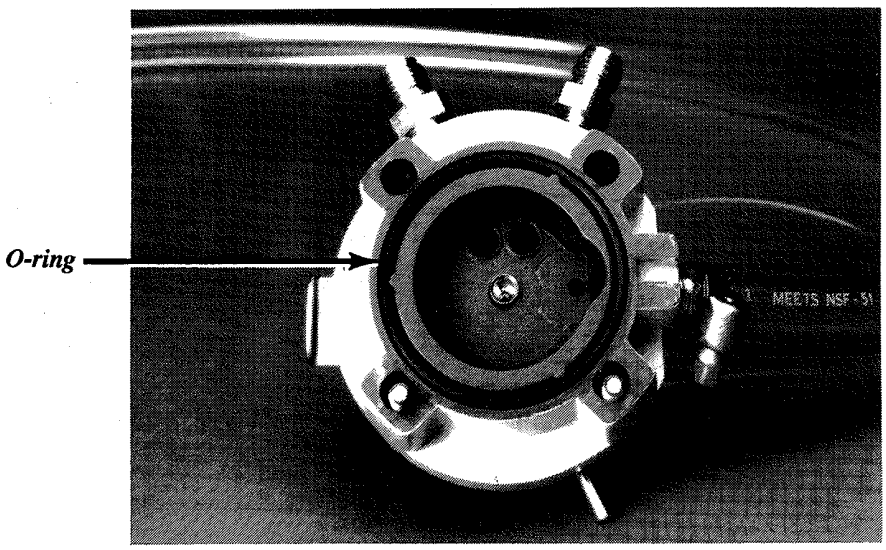


Figure 14-9. Inside of Burner End Cap

4. Install the Burner End Cap. Loosely tighten the four knurled screws.
5. Hand tighten the four knurled screws in the following sequence: top right-hand, bottom left-hand, bottom right-hand, top left-hand.

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Removing the Impact Bead

1. Extinguish the flame, turn off the gases, and let the burner head cool.
2. Remove the four knurled screws from the Burner End Cap (see Figure 14-7).
3. Remove the Burner End Cap. The Impact Bead is installed on the inner side of the end cap (see Figure 14-10).

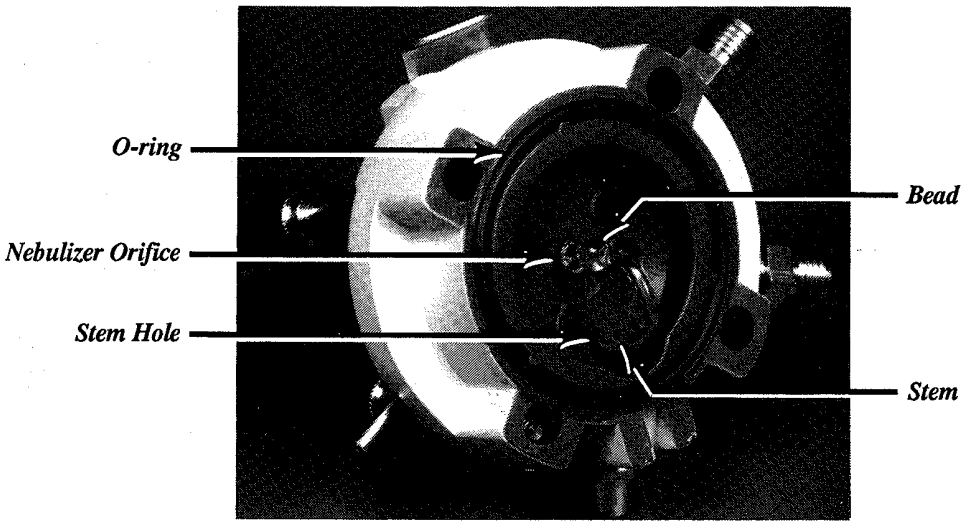


Figure 14-10. Impact Bead

NOTE: It is not necessary to disconnect the hoses from the end cap.

4. Grasp the bead by its stem and pull it straight out of the spray chamber.

Installing the Impact Bead

1. Moisten the stem with water.
2. Grasp the stem of the bead and press it firmly through the two O-rings in the Stem Hole, until it bottoms in the seat.
3. Carefully rotate the stem to center the bead on the nebulizer orifice.
4. Make certain that the end cap O-ring is properly seated in the end cap groove.
5. Install the end cap. Loosely tighten the four knurled screws.
6. Hand tighten the four knurled screws in the following sequence: top right-hand, bottom left-hand, bottom right-hand, top left-hand.

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NOTE: If sample residues have partially filled the stem hole, you may not be able to seat the impact bead. If this occurs:

1. Remove the two retaining plate screws and the retaining plate (see Figure 14-11). This allows you to push out any residue that may be present.
2. Carry out steps 1 through 6 on the previous page.
3. Replace the retaining plate and screws.

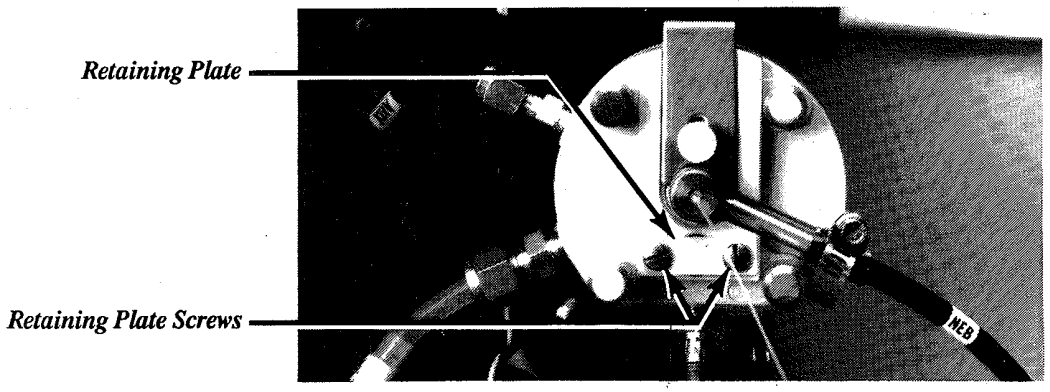


Figure 14-11. Burner End Cap

Removing a Broken Impact Bead

1. Remove the retaining plate screws and retaining plate (see Figure 14-11).
2. Using a small probe, push the broken stem out of the end cap.
3. Replace the retaining plate and screws.

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Installing the MHS-10 Quartz Cell Holder

1. Loosen the two phillips head screws between the Burner Head and the Ignitor Assembly.

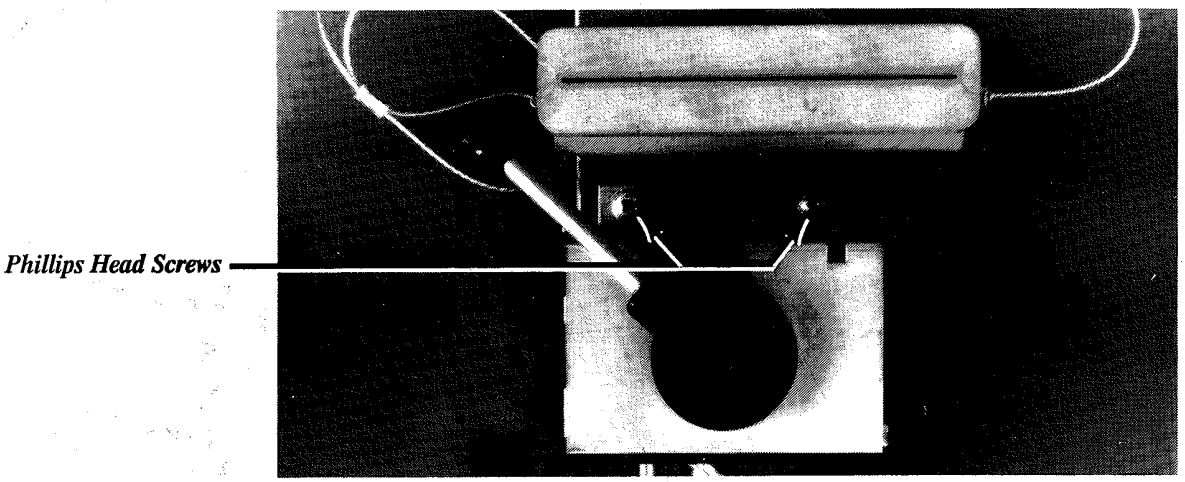


Figure 14-12. Top View of Burner Head and Ignitor Assembly

2. Slip the two identical slots on the front edge of the Cell Holder (see Figure 14-13) under the screws and tighten securely.

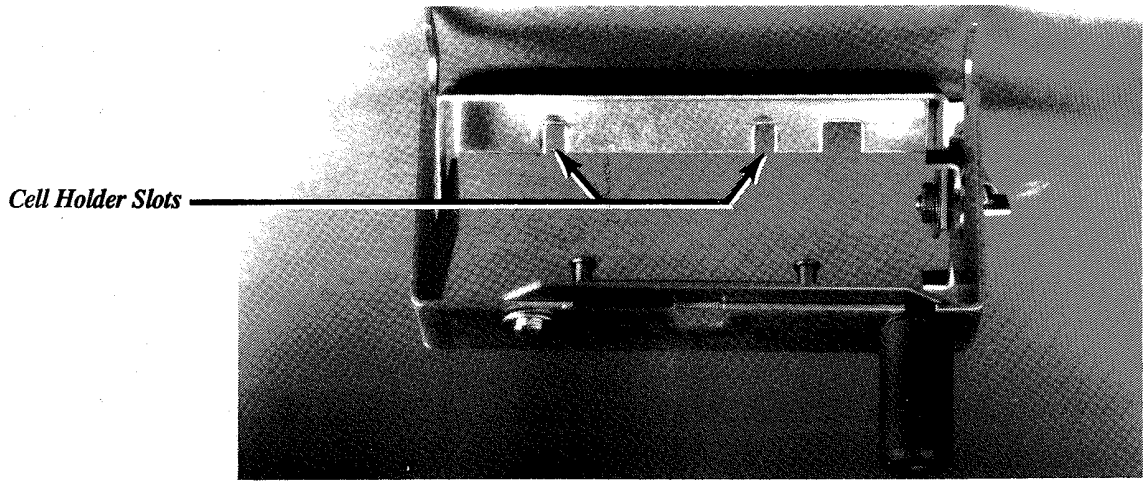


Figure 14-13. Quartz Cell Holder

The completed installation as seen from the top is shown on the next page in Figure 14-14.

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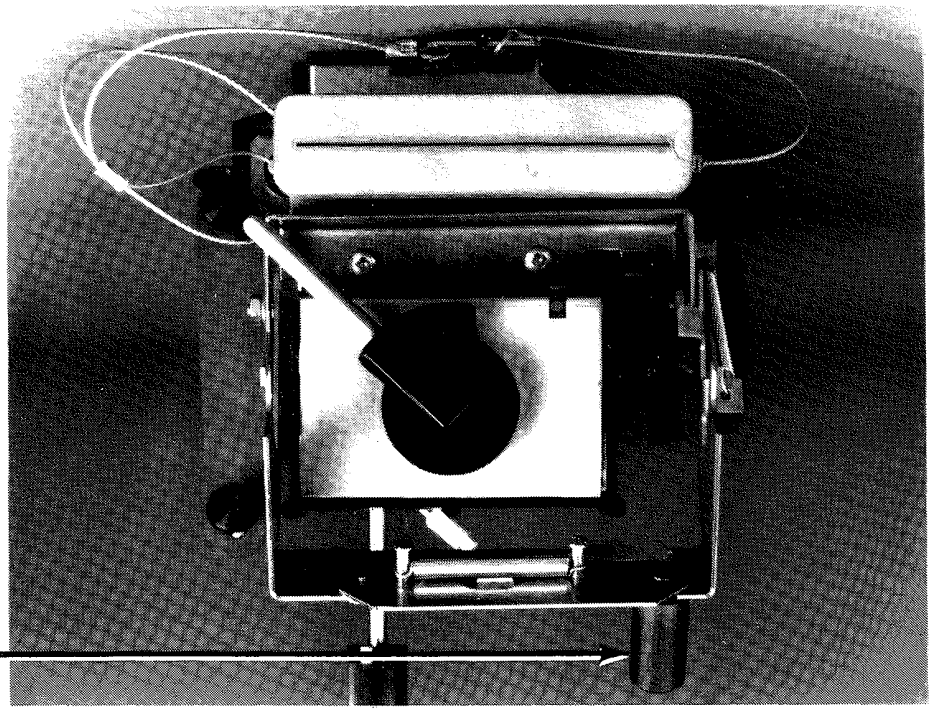


Figure 14-14. Quartz Cell Holder in Place

Installing the MHS-10 Quartz Cell

1. Turn the Capture Arm Handle (see Figure 14-14) 1/4 turn counter clockwise to loosen the Capture Arm.
2. Swing the Capture Arm up and to the left with the Capture Arm Handle.
3. Place the cell on the Cell Holder as shown in Figure 14-15.

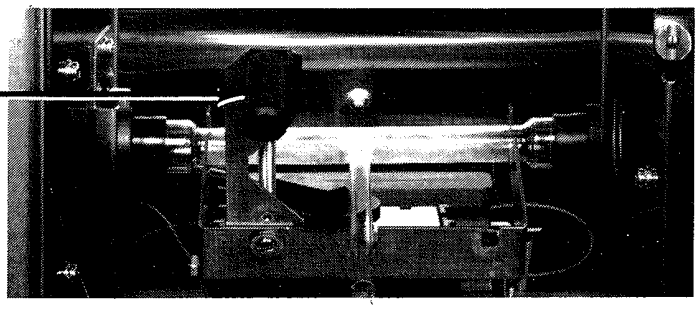


Figure 14-15. Quartz Cell in Place, Unlocked

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4. Swing the Capture Arm down and to the right to lock the cell in place as shown in Figure 14-16.

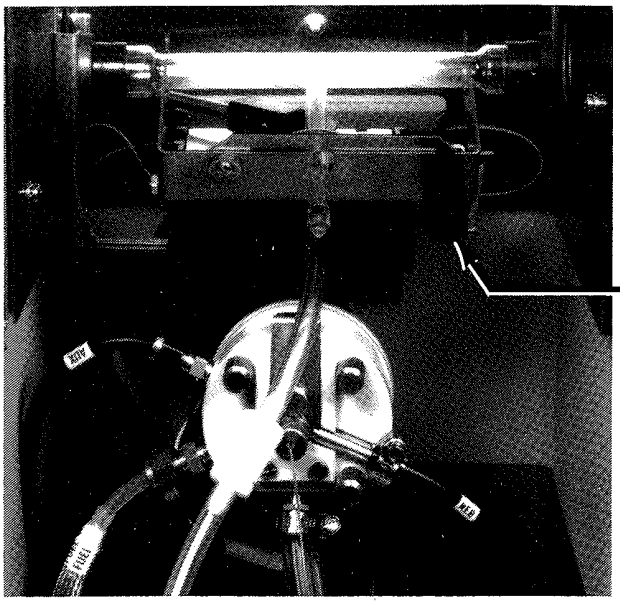


Figure 14-16. Quartz Cell in Place, Locked

5. Turn the Capture Arm Handle clockwise to secure the Capture Arm.

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Installing the Analyzer Assembly

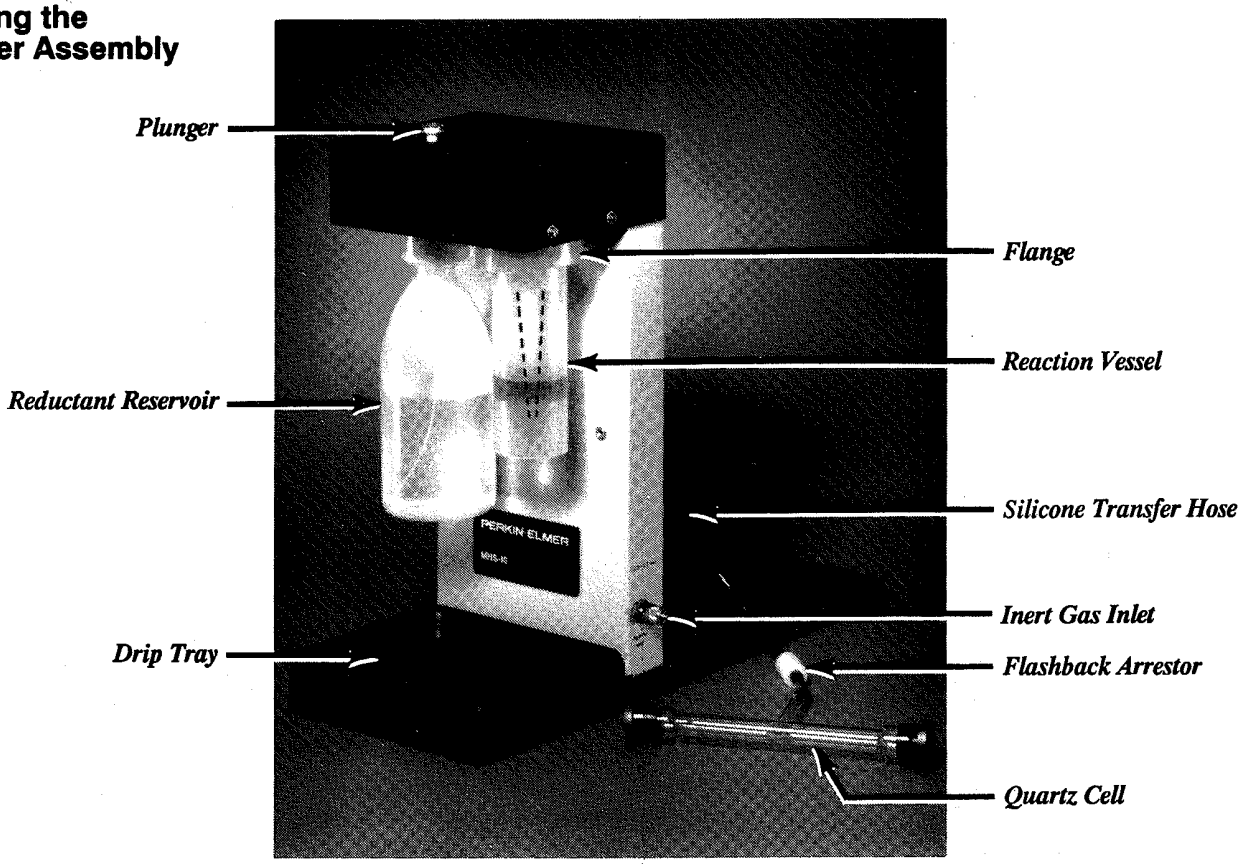


Figure 14-17. Analyzer Assembly

The analyzer assembly is a free standing unit. If there is sufficient bench space, you may position it in front of the spectrometer by sliding its base under the spectrometer. Alternatively, you can place it on a wheeled trolley in front of the spectrometer.

To install the analyzer assembly:

1. Unscrew the nut on the inert gas inlet nipple.
2. Push one end of the inert gas supply hose (2.5 m by 4.5 mm I.D.) through the nut.
3. Push the hose on the inert gas inlet nipple, then tighten the nut.
4. Connect the other end of the hose to a suitable supply of nitrogen or argon.
5. Push the reaction vessel onto the flange.
6. Place the drip tray on the base of the assembly.

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7. Locate the silicone transfer hose (with flashback arrestor) attached to the rear of the assembly (see Figure 14-17). Carefully attach the free end of this hose to the nipple on the quartz cell (see Figure 14-18).

> **WARNING:** Ensure that the transfer hose cannot become pinched or kinked in any way. A restriction in the hose may lead to a pressure buildup in the system.

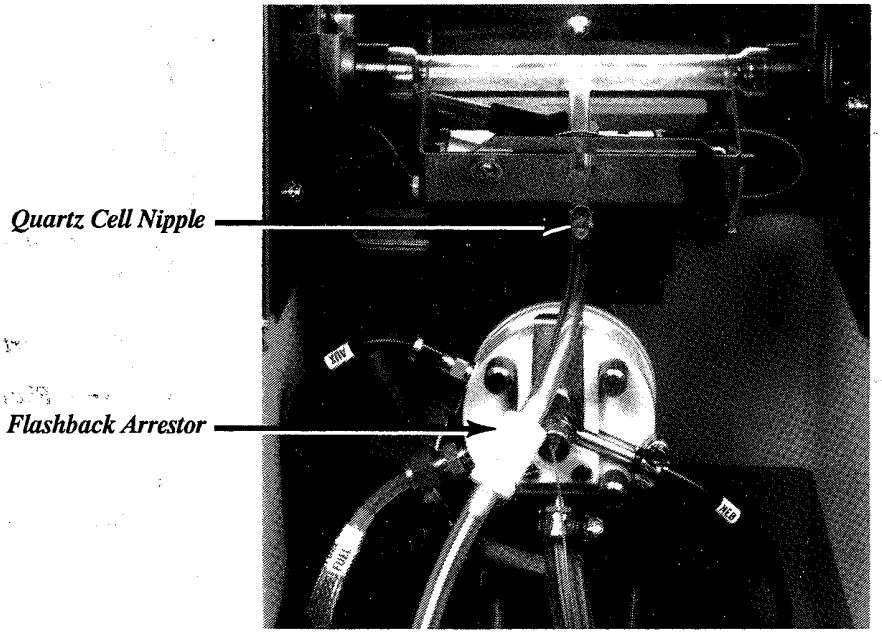


Figure 14-18. Transfer Hose Attached to Quartz Cell

> **NOTE:** The MHS-10 requires a supply of nitrogen or argon to operate the pneumatic system and to provide purging facilities. The necessary inlet pressure is 250 kPa (2 bar or 36 lb./sq. in) and the maximum consumption is approximately 1100 mL/min. To guarantee reproducible analytical results and safe operation, only nitrogen or argon having the following recommended minimum quality should be used:

NITROGEN (PURIFIED)	degree of purity	99.996%
	oxygen content	5 vpm*
	hydrogen content	1 vpm
	hydrocarbon	1 vpm
	moisture content	5 vpm
ARGON	degree of purity	99.996%
	oxygen content	5 vpm
	nitrogen content	20 vpm
	moisture content	4 vpm

* Volume, parts per million

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Installing Ancillary Devices

An autosampler, strip chart recorder, and serial printer may be attached to the 3100 by connecting these devices to the rear of the instrument.

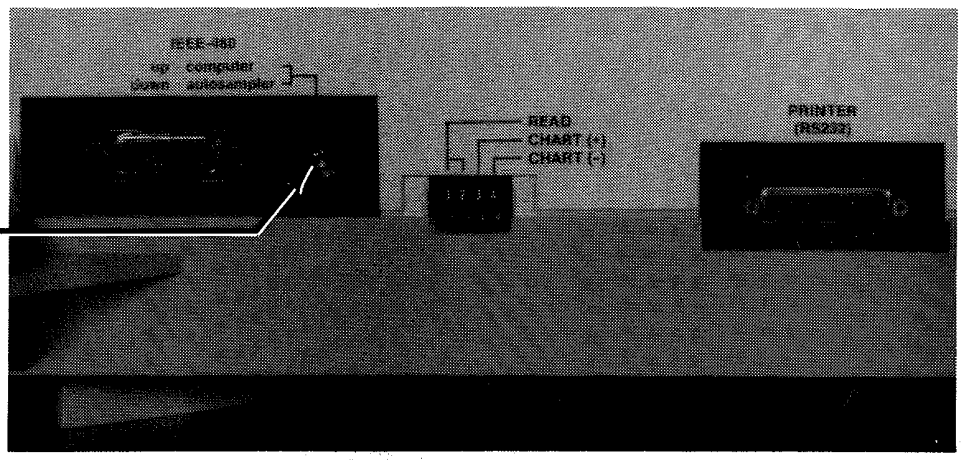


Figure 14-19 Ancillary Device Connections

Installing an Autosampler

1. Attach the IEEE-488 connector from the autosampler to the IEEE-488 port at the rear of the instrument.
2. Make certain the control switch is Down.

Installing a Strip Chart Recorder

1. Connect the positive lead from the strip chart recorder to the CHART (+) terminal on the terminal strip at the rear of the instrument.
2. Connect the negative lead from the strip chart recorder to the CHART (-) terminal on the terminal strip at the rear of the instrument.

NOTE: The full scale recorder output is 1V and corresponds to 3 Absorbance units or 3000 counts. The chart recorder output is expanded by the instrument's Expansion Factor.

Installing a Serial Printer

* Connect the RS-232 output cable from the printer to the RS-232 port on the rear of the instrument

Power Requirements

- 100, 120, 220, 240 Volts
- 50/60 Hertz
- 150 Watts
- 250 Watts with the optional Continuum Source Background Corrector

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Shipping Check List

Part No.	Part Name	Parts per Assy
N037-1078	MOLDING-PNY SAMPLE TRAY	1
N037-1079	COVER-PNY	1
0303-0152	AN. METHODS FOR AA	1
0303-0477	CABLE ASSY-RECORDER	1
0990-9390	AA NEWSLETTER SUB FORM	1
0993-8434	INSTRUCTION MANUAL	1
0993-9553	AA CONCEPTS MANUAL	1
0057-2825	SAFETY NOTES	1
0047-0556	CLAMP ASSY-DRAIN LOOP	1
0047-2014	GASKET-CORKPRENE END CAP	5
0303-0405	APIEZON "L" GREASE ASSY	1
0990-2147	"O" RING	1
0990-2236	"O" RING	2
0990-2240	"O" RING	2
0990-3193	FITTING NUT-FERRULE	4
0990-8265	POLY TUBING-10 FEET LONG	1
0047-3508	LABEL-ENCL LOCATION	2
0047-3509	LABEL-SURV NOTICE	2
0047-3510	LABEL-WASTE DISP NOTICE	2
0040-0266	BURNER HEAD ASSY	1
0057-2615	BEAD-IMPACT BEAD	1
0998-1615	FUSE-2.5A 250V 3AG SLOW	1
0998-1614	FUSE-2.0A 250V 3AG SLOW	1
0999-1641	FUSE-1.6A 250V SLOW-BLO	2
0999-1645	FUSE-1.0A 250V SLO-BLO	2
0998-1624	FUSE-1.5A 250V 3AG SLOW	1
0998-1611	FUSE-1.0A 250V 3AG SLOW	1
N037-0167	BURNER DOOR	1
0999-1400	220V LINE CORD	1
0789-1399	120V LINE CORD	1
N037-1093	DRAIN	1
0990-4991	WASH TRAP	1
L-310H	PREPARING YOUR LABORATORY	1
N037-1264	TEMPLATE CARD PARAMETER ENTRY INDEX	1

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Hardware Troubleshooting 15

Introduction

The Model 3100 has been designed for routine, trouble-free operation. Although the system is relatively simple to operate, occasional problems may occur.

We include this chapter of troubleshooting suggestions to help you determine systematically whether difficulties you experience with the spectrometer are due to improper analytical techniques, improper selection of instrument parameters, or malfunction of the instrument.

If, after checking the instrument against the symptoms described in this chapter, the problem appears to be due to malfunctioning of the instrument, we strongly recommend that you contact your local Perkin-Elmer Service Representative. Servicing of the instrument by a person other than a Perkin-Elmer Service Representative will render the warranty null and void.

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Problems With the Spectrometer

Problem	Possible Cause	Check and/or Remedy
Lamp will not fit mounting bracket.	Lamp diameter smaller than 2 inches.	Use adapter PN 0303-0870.
Lamp connector will not fit connection on the instrument.	Lamp is not manufactured by Perkin-Elmer.	Lamps other than Perkin-Elmer Intensitron Series require special connecting cables.
	Lamp is an EDL.	EDLs require a special power supply.
Hollow cathode lamp will not light.	Spectrometer is off or not plugged in.	Turn spectrometer on or plug spectrometer into line power.
	Lamp not plugged in.	Connect lamp
	Lamp worn out or damaged.	Try another lamp.
	Insufficient lamp current.	Use **[Param Entry]** to set the proper current.
EDL will not light.	EDL power supply not on.	Turn on power supply.
	Needs light source to initiate.	Point at flame, overhead fluorescent light or EDL source starting accessory.
Lamp energy too low.	Lamp starting to deteriorate.	Run lamp for several hours. Replace lamp.
	Insufficient current.	Use **[Param Entry]** to set the proper current.
	Wrong slit.	Check recommended settings in AA Cookbook.
	Lamp out of alignment.	Align the lamp.

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Problems With Flame Analysis

Problem	Possible Cause	Check and/or Remedy
Results erroneously high.	Corrections not made for reagent blank.	Zero instrument while aspirating a reagent blank.
	Ionization enhancement is present in sample or blank.	Add approximately 1000 ppm of an easily ionized salt to samples, standards and blank.
	Standard solutions deteriorated or have been improperly made.	Restandardize with a proven standard.
	Background absorption (molecular absorption, light scattering, etc.) is present.	Use background corrector or use a hotter flame.
Results erroneously low.	Chemical or matrix (bulk, viscosity) interferences present.	Remove interferent or match matrix.
	Standard solutions are improperly made.	Rerun proven standards.
	Blank solution contaminated.	Change blank.
Cannot get suggested detection limits.	Insufficient scale expansion and integration time are being used.	Try higher scale expansion with longer integration.
	Incorrect wavelength selected.	Use the most sensitive wavelength suggested in the AA Cookbook.
	Characteristic concentration for analysis is low.	Try changing flame conditions (hotter-cooler, lean-rich, nitrous oxide).
	Lamp current too low.	Increase lamp current.
Cannot get close to characteristic concentration.	Analysis being performed on wrong spectral line.	Check adjacent wavelengths for sensitivity.

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Problem	Possible Cause	Check and/or Remedy
Cannot get close to characteristic concentration.	Improper flame or flame conditions.	Use hotter or cooler flame or use nitrous oxide flame.
	Ionization suppressant not being used.	Add ionization suppressant.
	Slit width set too wide.	Check recommended settings in AA Cookbook.
	Nebulizer not adjusted properly.	Adjust nebulizer.
	Nebulizer partly blocked.	Clean nebulizer.
	Lamp current improperly set.	Reset lamp current.
	Liquid buildup in chamber.	Check for proper drainage.
	Burner skewed in light path.	Adjust burner position.
	Burner too high or low.	Adjust burner position.
	Sample aspiration during autozero.	Rezero instrument.
	Impact bead not in place and characteristic concentration using impact bead is being used for comparison.	Install the impact bead.
Data noisy with flame off.	Scale expansion set too high.	Reset scale expansion.
	Unstable or weak lamp.	Use wider slit width if possible or replace lamp.
	Lamp energy too low or unstable.	Use **[Param Entry]** to set the proper lamp current.

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Problem	Possible Cause	Check and/or Remedy
Data noisy when aspirating blank.	Contamination in burner.	Clean burner slot by washing with water and scraping slot carefully with a thin metal strip that does not have sharp edges. Clean burner chamber. Check for proper drainage.
	Highly absorbing flame in UV.	Use background correction. Vary flame gas flow rates to minimize flame absorption or maximize flame stability.
	Weak lamp emission with highly absorbing flame.	Increase lamp current to maximum rating. Use new lamp. Use a larger slit width if possible (sensitivity may be reduced). Use a different wavelength. Separate analyte element from highly emissive matrix by chemical means.
Continuum source background corrector does not provide adequate correction.	Background absorbance too large.	Use BG only mode to determine just the background. Do not correct for more than 2 absorbance units.
	Background corrector source lamp not aligned.	Have your PE Service Representative align the source lamp.
	Background is structured.	Change to another wavelength. Remove interferent by chemical means.
Continuum source gives excessively noisy results.	Background source intensity is too low.	Have the source lamp replaced or realigned by your PE Service Representative.
	Source lamp intensity too low or lamp not aligned properly.	Replace or realign source lamp.

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Problem	Possible Cause	Check and/or Remedy
Flame does not ignite (flame interlock light is on).	Fuel and oxidant not on or at proper pressure.	Check fuel and oxidant for flow and pressure.
	Burner head not correctly installed	See that the pin on the holddown cable is inserted into safety lock.
	Drain interlock not connected.	Connect interlock cable.
	Liquid level in drain vessel is excessively low.	Add about 250 mL of water to activate the float in the drain vessel
	Thermal Sensor	Not connected or shield too hot.
	Flame/sensor/ignitor assembly not connected.	Attach connector.
	Fuel/oxidant flow stopped after 15 seconds.	Turn Oxidant switch to Off, then back to Air and press the Ignite button.
Flame does not ignite in required 15 second interval.	Fuel/oxidant set too high or too low or to wrong ratio for ignition.	Turn Oxidant switch Off. Turn Oxidant switch to Air. Adjust fuel to 2-2.5 and oxidant to 4. Press the Ignite button.
Burner flashes back.	Gases connected to wrong inlet on burner or gas controls.	Check for proper connection of gases.
	Gas leakage.	Check connections and hoses for leaks with soap solution (if bubbles appear, fix leaks).
	Air supply contains excess oxygen.	Use compressed air, NOT breathing air. NEVER use oxygen.
	Break or leak in drain system.	Inspect tubing and replace if needed.
	Wrong gases used.	Check source of gases.

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Problems With the MHS-10 System

Problem	Possible Cause	Check and/or Remedy
Cannot obtain quoted characteristic mass; poor reproducibility.	Incorrect standards.	Prepare new standards.
	Reductant is not fresh.	Make up fresh reductant. Fresh reductant is essential for the determination of selenium.
	Reductant is not being transported properly to the reaction flask.	Check that the reductant is being transported properly into the reaction flask. Reductant transfer should continue until the maximum signal has been obtained.
	Sample transport hose is contaminated.	Flush the sample transport hose through with 1.5% HCl then with deionized water. Dry by purging with dry, dust-free air or inert gas (N_2, Ar).
	Quartz cell requires reconditioning.	Bathe the quartz cell as described in the MHS Analytical Methods Manual.
Excessive foaming.	Antifoaming agent not added to sample.	Add 1-2 drops of a silicone antifoaming agent.

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Problem	Possible Cause	Check and/or Remedy
Memory signal occurs during determination of antimony or tin.	Contamination.	Optimize the cell temperature. Flush the sample transfer hose with 1.5% HCl and then with deionized water. Dry by purging with dry, dust-free air or inert gas (N_2, Ar). Rinse the reaction flask and the flange on the Analyzer Assembly very thoroughly with 1.5% HCl. Bathe the quartz cell for 15 minutes in cold, 40% hydrofluoric acid. If tin chloride is used for mercury analysis as the reductant for the system, the analyzer assembly becomes contaminated and cannot be used for the hydride determination of tin.

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Error Messages

ERR:24 RAM ERR (U26) (DEPRESS CE KEY)

ERR:25 ROM ERR (U24) (DEPRESS CE KEY)

ERR:26 CHOPPER MOTOR (DEPRESS CE KEY)

ERR:27 R/W ERROR (U29) (DEPRESS CE KEY)

ERR:28 MPV ERR:(U15) (DEPRESS CE KEY)

ERR:47 NO A/D IRQ (DEPRESS CE KEY)

If any of the above error messages appear, power down the system, then power up again. If any of the above messages reappear, call your Perkin-Elmer Service representative.

ERR:30 LINE FREQ (DEPRESS CE KEY)

Line cycle drop out detected. You may need a line power conditioner.

ERR:31 VALUE OUT OF RANGE

Parameter entered is out of range.

ERR:32 TOO MANY DIGITS

Parameter entered has too many digits.

ERR:33 STANDARDS NOT IN ORDER

ERR:34 TOO MANY DECIMAL PLACES

Parameter entered has too many decimal places.

ERR:35 TOO MANY SIGNIFICANT DIGITS

You probably entered more digits for a standard concentration than you entered for the first standard. The concentration format is set by the first standard. Subsequent standards required more significant digits than are allowed by the instrument. Reduce the number of digits for the first standard (see "Significant Figures" on page 17-3 for more information).

ERR:36 AGC CANNOT ASSIGN CORRECT GAIN

Microprocessor cannot assign optimum voltage to PMT. Check wavelength, lamp, lamp current, slit, etc. If the error message persists, call your Perkin-Elmer Service representative.

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ERR:37 AIC CANNOT ASSIGN CORRECT GAIN

The deuterium arc cannot match the intensity of the hollow cathode lamp. Increasing the slit width may resolve this problem but could cause reduced sensitivity. Reducing the HCL lamp current may also correct this problem. If the error message persists, the deuterium arc lamp may need replacement. Call your PE Service Representative.

ERR:38 PMT VOLTAGE RANGE IS 250-850

You probably entered a PMT voltage out of range.

ERR:39 IEEE 488 BUS TIMEOUT

Autosampler is either not connected or not turned on.

ERR:40 DATA KEY MUST BE SELECTED

Autozero, Calibration, and Reslope must be run in Data Mode.

ERR:41 AUTOSAMPLER NOT SELECTED

Make sure that the control switch on the back panel is in the down position.

ERR:42 ENTRY MUST BE AN INTEGER

You entered a decimal. Enter a whole number.

ERR:43 D2 OPTION NOT AVAILABLE

Deuterium arc accessory probably not installed. Deuterium arc must be installed to run in AA-BG Mode.

ERR:45 SELECT GAIN WITH ENERGY OR EM

Press: **[Energy] [Gain]**.

ERR:46 CANNOT A/Z IN PARAM ENTRY OR ENERGY

You must be in Data or Cont Mode to Autozero.

ERR:48 PRINTER NOT SELECTED

Make certain that the printer is On Line and turned on. This error may also occur if the printer is turned on after the instrument is turned on.

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ERR:49 EMISSION TOO HIGH FOR PMT

Primary source (EDL or Hollow Cathode Lamp) may have drifted. Press [Gain] to rescale PMT voltage. Otherwise there may be excessive flame emission detected due to a high concentration or highly emissive sample. Dilute the sample and reanalyze.

ERR:50 CAN'T RESLOPE WITH METHOD OF ADDITIONS

Reslope cannot be performed with Method of Additions.

ERR:52 REPS HAVE NOT BEEN COMPLETED

You probably tried to perform an operation before the system read the last replicate

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Maintenance 16

This chapter contains routine maintenance procedures for the 3100 that you can do yourself. All other maintenance and service should be performed by a Perkin-Elmer Service Representative.

WARNING: The cover of the Model 3100 Spectrometer should only be removed by a Perkin-Elmer Service Representative.

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Cleaning the Burner Head

The nature of the samples aspirated determines the burner cleaning interval. In ordinary operation, the burner will need infrequent cleaning. Recondition the burner chamber after working with organic samples as described in the section entitled "Cleaning the Burner after Use with Organic Samples." When samples with high solids content are aspirated, clean the burner at least daily.

> **IMPORTANT:** If high concentrations of silver, copper, or mercury salts have been nebulized into an acetylene flame, clean the burner chamber immediately after use by following the procedure in this chapter (p. 17-4) entitled "Cleaning the Burner after Use of High Concentrations of Silver, Copper or Mercury Samples in an Acetylene Flame."

The burner should provide an even flame over the length of the burner slot. An uneven flame may indicate the slot needs cleaning. With the flame off and air on, carefully work along and through the slot using the thin metal strip provided*. Do not nick the edges of the slot.

Cleaning stubborn deposits from the burner head slot may require removing the burner head from the burner chamber. If so, carry out the procedures on pages 14-2 and 14-3 to remove the Burner Head, then proceed as follows:

1. Extinguish the flame, turn off the gases, and let the burner head cool.
2. Carefully work through the slot with the thin metal strip provided*. Remove scrapings from inside and outside the burner head.
3. The Burner Head can also be soaked overnight in a detergent solution and then rinsed with deionized water and blown dry with a clean air supply.
4. Reinstall the Burner Head using the procedures on pages 14-4 and 14-5 of Chapter 14.

* It is strongly recommended that you use the piece of shim stock shipped with the instrument to clean the burner head slot.

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Cleaning the Burner Mixing Chamber

Immediately After Using Organic Solvents

If aqueous samples are aspirated after aspirating organic samples such as oil or MIBK extracts, the absorption signal produced can be noisy and erratic. After aspirating organic samples, use the following procedure to prevent contamination of subsequent aqueous samples.

1. Aspirate for 5 minutes with an organic solvent miscible with the samples that have just been aspirated.
2. Aspirate methanol or another solvent miscible with both the organic solvent and water, for five minutes .
3. Aspirate 1% HNO_3 for five minutes.
4. Flush the waste drain tube thoroughly with water. Empty the collection vessel and refill with water. Dispose of hazardous or corrosive solutions properly and observe local codes concerning the effect of the waste on the environment.

Immediately After Aspirating High Concentrations of Ag, Cu, or Hg

IMPORTANT: Unstable acetylides, which are likely to explode when dry, may be formed when aspirating high concentrations of silver, copper, or mercury salts into an acetylene flame.

* To help prevent the build up of unstable acetylides, aspirate water for 5 minutes after each analysis.

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After Prolonged Use

To clean the Burner Mixing Chamber it must first be removed from the Burner Assembly. Use the following procedure to disassemble the Burner Assembly:

Disassembling the Burner Assembly

1. Extinguish the flame, turn off the gases and let the burner cool.

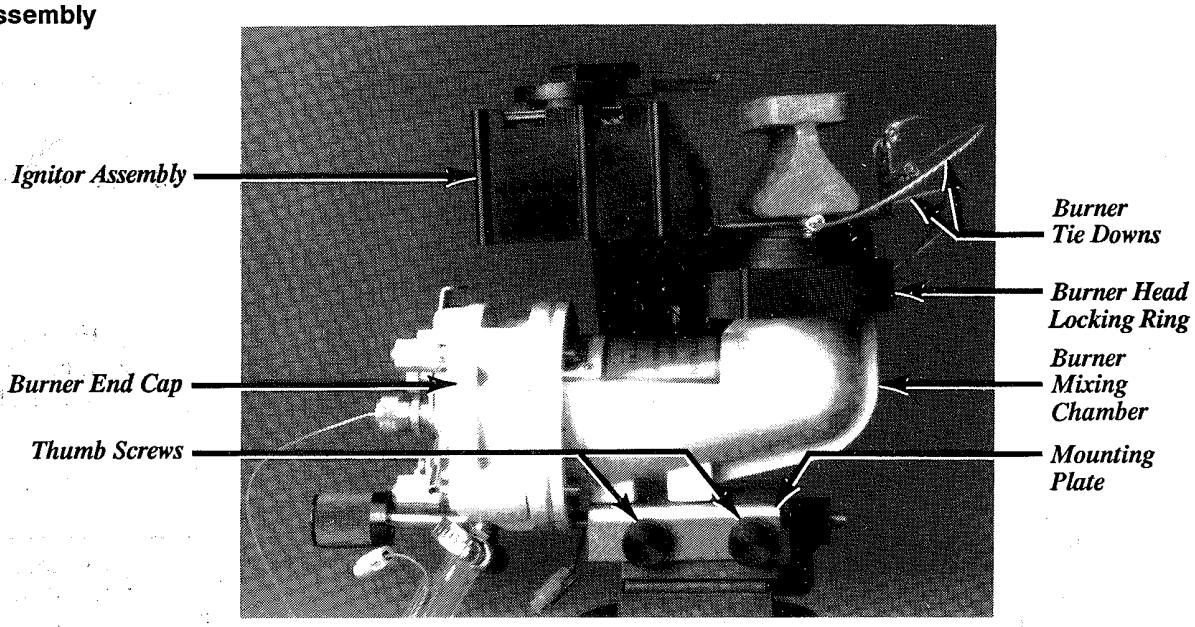


Figure 16-1. Burner Assembly

2. Remove the Burner Head Tie Downs.

3. Disconnect the Ignitor Assembly connector by first pushing down on the silver tab at the top of the connector and pulling the connector to the left.



Figure 16-2. Ignitor Assembly Connector

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4. Turn the Burner Head Locking Ring counter-clockwise until the Ignitor Assembly with the Burner Head attached is free of the Burner Mixing Chamber.

5. Remove the Burner End Cap and Flow Spoiler (see page 14-4) or Impact Bead (see page 14-6).

6. Remove the two Thumb Screws from the Burner Mounting Plate and remove the Burner Mixing Chamber.

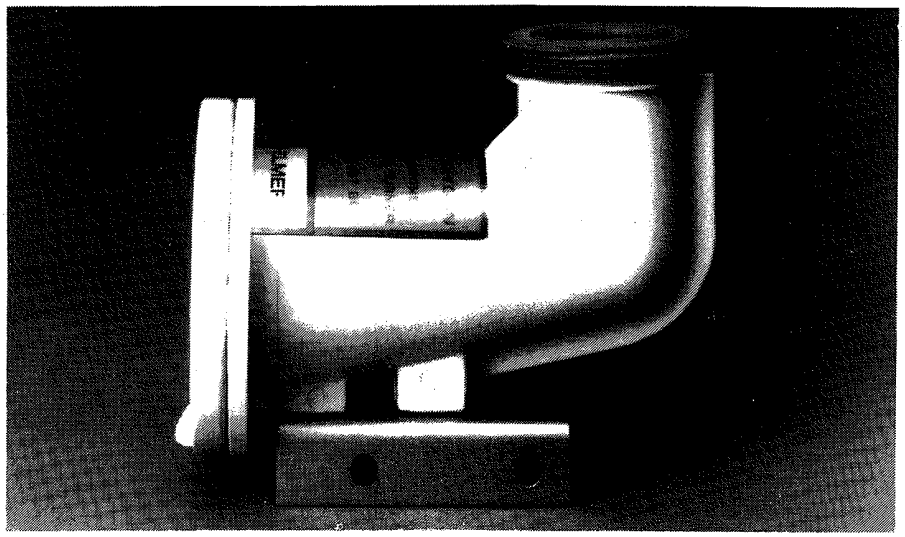


Figure 16-3. Burner Mixing Chamber

Cleaning the Burner Mixing Chamber

1. Remove stubborn deposits by gently scrubbing with a detergent solution and a bottle brush. Rinse with water and allow to dry.

IMPORTANT: To avoid damaging the plastic lining, do not use abrasive tools, kitchen type abrasive cleaners, or soak the Burner Mixing Chamber in acid.

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Reassembling the Burner Assembly

1. Replace the Burner Mixing Chamber on the Mounting Plate, insert and tighten the two Thumb Screws.
2. Place the Ignitor Assembly with the Burner Head attached on the threaded part of the Burner Mixing Chamber and tighten the Burner head Locking Ring.
3. Reconnect the Ignitor Assembly Connector and then push the silver tab from the bottom to lock the connector in place.
4. Reinstall the Flow Spoiler or Impact Bead using the procedure on page 14-4 or 14-6.
5. Replace the Burner End Cap using the procedure on page 14-5.
6. Check the burner alignment and align if necessary (see Chapter 3, Installing and Aligning lamps).

Flushing the Drain System

It is recommended that the drain system be flushed thoroughly with water at the end of each working day to remove caustic, corrosive, or organic waste materials that could otherwise damage the drain tubing and burner chamber.

To flush the system, remove the Burner Head and slowly pour about 500 mL of water into the Burner Mixing Chamber.

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Cleaning the Drain Float Assembly

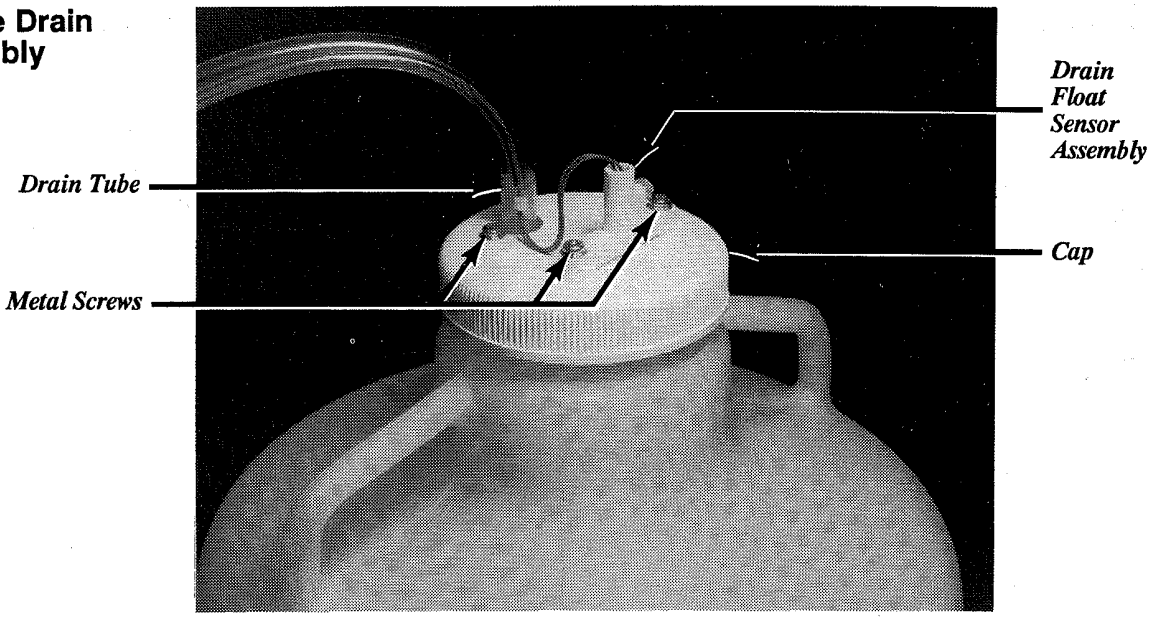


Figure 16-4. Drain System -- External View

1. With a screw driver, pry off the plastic Drain Tube.
2. Unscrew the cap.
3. Empty the Drain Loop attached to the underside of the cap.
4. Remove the three metal screws from the top of the cap.

   NOTE: Do not loosen the plastic screw in the Drain Float Sensor Assembly.

   This releases the Drain Float Assembly. Make a note of the orientation of the Drain Float Sensor Assembly.
5. Clean the Drain Float Assembly with soap and water using a test tube brush.
6. Rinse with water.
7. Reassemble the system in reverse order making certain the Drain Float Sensor Assembly is replaced with its original orientation.

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Maintaining the Nebulizer

Cleaning the Sample Capillaries

A low absorbance reading with a clean burner head slot may be due to an obstruction in the nebulizer or sample capillary tube. Aspirate pure solvent with the flame on until the absorbance reading is satisfactory for a subsequent standard.

> **NOTE:** If a longer sample capillary is used, as with the AS-51 autosampler, the rate of sample uptake and the sensitivity will decrease.

If solvent aspiration fails to clear the sample capillary or nebulizer capillary, push a copper wire (provided with the spectrometer) through the capillary from the inlet side. Move the wire in and out to dislodge any solid particles.

> **IMPORTANT:** Use only the copper cleaning wire provided (PN 0303- 0135) to clean the capillary. Other wire may be burred and may damage the nebulizer.

For deposits which do not yield to either of these measures, disassemble the nebulizer and clean it using the procedures starting on the next page.

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Disassembling and Cleaning the Nebulizer

IMPORTANT: The cleaning procedure on the preceding page usually suffices to restore the nebulizer to satisfactory working order. The disassembly described here is seldom needed. Performance characteristics of a nebulizer are likely to be modified by disassembly.

1. Remove the Nebulizer Oxidant Tubing from the Nebulizer.
2. Loosen the Nebulizer Clamp Screw.
3. Pull out the Nebulizer.

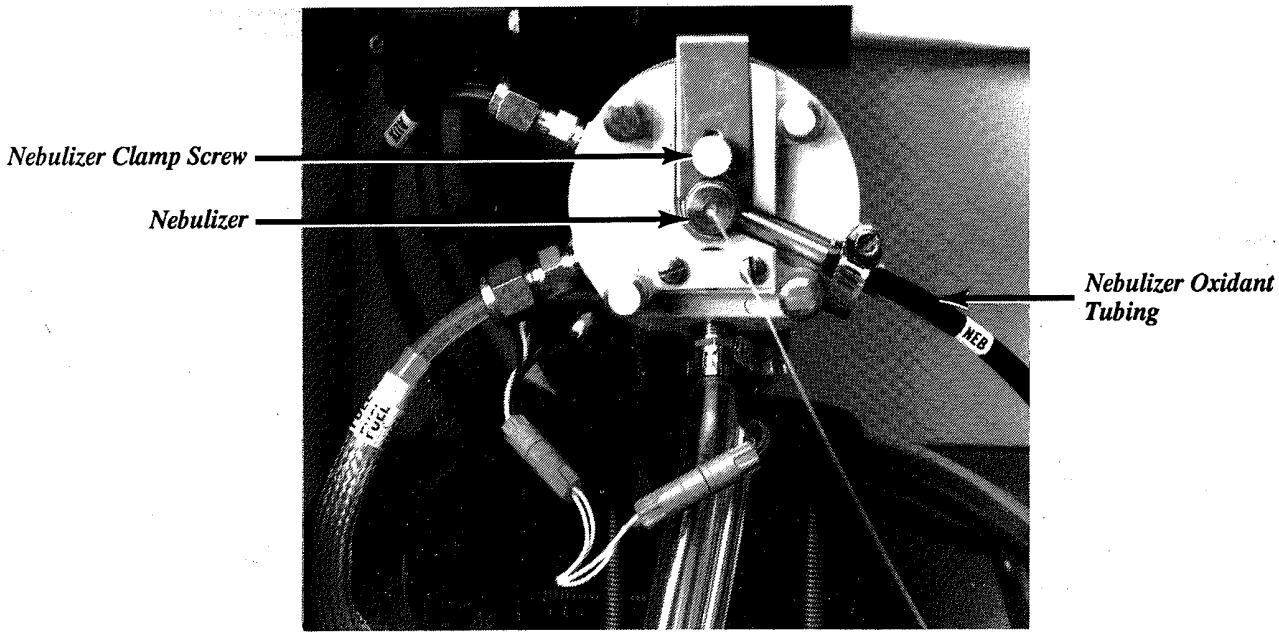
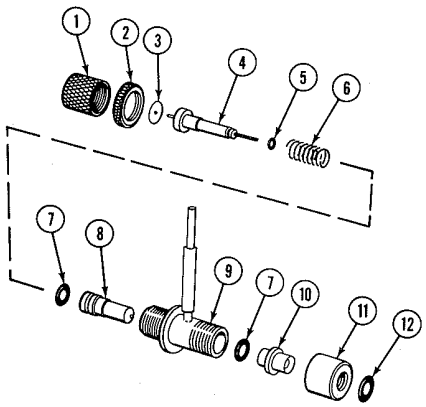


Figure 16-5. Nebulizer Connections

For the remaining steps refer to Figure 16-6 on the next page.

4. Unscrew and remove the knurled knob (item 1) and washer (item 3).
5. Carefully pull out the needle assembly (item 4). Inspect the inner end for deposits and clean if required.

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1 Knurled Knob (0303-1460)
2 Locking Ring (0303-1456)
3 Washer (0303-1802)
4 Needle Assembly (0303-0354)
5 O-ring (0990-2102)
6 Spring 0303-1806
7 O-Ring (2) (0990-2102
8 Insert (0303-1812)
9 Nebulizer Body (0303-0353)
10 Venturi (0303-1810)
11 Front End Cap (0303-1808)
12 O-ring (0990-2239)

Figure 16-6. Nebulizer -- Exploded View

6. Remove the O-ring (item 12), unscrew and remove the front end cap (item 11), and pull out the venturi section (item 10).

7. If necessary, remove the insert (item 8) by pushing it out with a wooden probe.

8. Clean components as required with a suitable solvent: O-rings may be cleaned with soap and water.

Reassembling the Nebulizer

1. Lubricate the O-ring on the insert (item 7) and the O-ring on the needle assembly (item 5) with a bit of grease such as Apiezon L (supplied with the nebulizer).

2. Slide the needle assembly into the insert as far as it will go; the needle tip should protrude from the small aperture in the insert end.

3. Insert these assembled components into the left end of the nebulizer body (as shown in Figure 16-6), and install the washer and knurled knob.

4. Install the venturi over the needle tip; put on and tighten the front end cap (item 11). Slip the O-ring (item 12) over the protruding end of the venturi.

5. Install the nebulizer in the burner and adjust the aspiration rate as described in Setting up for Flame in the Users Manual. Lock the setting by turning the locking ring counterclockwise against the knurled knob.

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AS-51 and AS-90 Autosampler Maintenance

Servicing

Servicing may only be performed by a Perkin-Elmer Service representative. Contact your local Perkin-Elmer sales office to schedule service.

Routine Maintenance

Minimal routine maintenance is required. Keep the carousel clean, and try to avoid spills. Do not use abrasives to clean the clear plastic carousel cover. Do not use organic solvents to clean the carousel cover.

Spare Parts

If corrosive solutions are being routinely analyzed, a corrosion-resistant, platinum-rhodium probe is available to replace the standard stainless-steel version. You may find it convenient to purchase additional sample carousels, so that samples may be loaded into a second carousel while the first is still in use. A list of spare parts is given on the following page.

MHS-10 Maintenance

For maintenance information regarding the MHS-10 refer to manual number B009-8670.

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Spare Parts and Accessories

Following is a partial list of common consumable parts and accessories for the Model 3100, along with their part numbers.

PE Part No.	Description
Background Correction Lamp	
N037-0051	Deuterium Arc Assembly
Burner Assemblies and Accessories	
0057-0988	Premix Burner Assembly
0057-0948	Burner Mixing Chamber for 0057-0988
0990-2219	Viton O-ring for 0057-0948
0047-0988	Burner Gasket Kit for 0057-0948
0057-0984	End Cap Assembly for 0057-0988
0057-2561	Flow Spoiler for 0057-0988
0056-2516	Impact Bead for 0057-0988
Burner Heads	
0040-0266	Flat 10 cm Single-Slot air/acetylene head
0040-0277	Titanium N20/acetylene burner head
Nebulizers and Components	
0303-0352	Stainless Steel Adjustable Nebulizer
0303-0354	Capillary Assembly for 0303-0352
0303-1810	Venturi Section of 0303-0352
0303-0404	Corrosion-Resistant Adjustable Nebulizer
0303-0402	Capillary Assembly for 0303-0404
0303-1920	Venturi Section of 0303-0404
0303-0299	Platinum/Rhodium Nebulizer Assembly
0303-0433	Capillary Assembly for 0303-0299
0303-3124	Venturi Section of 0303-0299
0303-0135	Nebulizer Cleaning Wires (pkg. of 5)
0990-8202	Sample Tubing, 0.015 in. i.d. x 10 feet
0990-8265	Wide Diameter Tubing 0.023 in. i.d. x 10 feet

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Calibration 17

The information in this chapter supplements the discussion of calibration in Section 7 (yellow pages) of the AA Cookbook.

This chapter describes how the 3100 converts absorbance or emission intensity values into concentration, the equations used, and the calibration options available. Error messages that may appear during calibration are described.

The 3100 offers three calibration choices: linear, non-linear, and method of additions. All of these are based on the same basic equation and each has been designed to solve a particular type of analytical problem.

Calibration Equation

Quantitative measurements in atomic absorption are based on Beer's Law, that concentration is proportional to absorbance, as shown in the following expression:

$$C = KA = K \log (I_0 / I) \qquad \text{[Equation 1]}$$

where C is the concentration, A is the absorbance, I_0 and I are the incident and transmitted source intensities, and K is a proportionality constant.

It is well known, however, that the observed relationship between concentration and absorbance is not linear at higher concentrations.

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To correct for this non-linearity, the 3100 uses the equation:

$$C = K_0 \left(\frac{K_1 A + K_3 A^2}{K_2 A - 1} \right) \qquad \text{[Equation 2]}$$

In this expression C is the concentration, A is the observed absorbance, K_1, K_2, and K_3 are coefficients determined during the calibration procedure, and K_0 is the reslope coefficient that is set equal to 1.0 during the initial calibration. In applying this equation a blank is always measured first. The blank value is automatically subtracted from all subsequent readings. The use of this equation for calibration in atomic absorption has been described by Unvala (1) and by Barnett (2).

This equation belongs to a class of functions known as "rational," a term applied to functions that are the quotient of two polynomials. Functions of this type can approximate straight lines, curves that approach an asymptote, or anything in between depending on the values of the empirical coefficients. This type of behavior approximates that typical of the atomic absorption calibration curves of most elements over a wide range of concentrations. These functions can also approximate the flame emission calibration curves for most elements over a more restricted concentration range.

The 3100 uses Equation 2 in three forms: as shown; with K_3 being set equal to zero; or with K_2 and K_3 both being set equal to zero. This latter case is, of course, the equation for a straight line that passes through the origin. When the number of reference standards matches the number of coefficients to be determined, it is a simple matter to determine the coefficients by solving a system of simultaneous equations. When the number of reference standards exceeds the number of coefficients, the 3100 determines the coefficients by the method of least squares. To prevent the more concentrated reference standards from dominating the fitting procedure, 1/C weighting is used except for the linear equation. By fitting a curve using least squares, the 3100 is able to average small errors in the standardization process. However, three carefully chosen and prepared reference standards will usually yield acceptable results, even over a fairly long dynamic range.

The reslope feature of the 3100 allows you to make small corrections to the calibration curve using a single reference standard following small changes in instrumental conditions. You might also want to use a reslope standard with a matrix similar to your samples, for example an NBS Standard Reference Material, to compensate for small matrix effects after you have performed the initial calibration with aqueous reference standards. When a reslope recalibration is performed, coefficient K_0 is adjusted to make the concentration calculated by Equation 2 equal to the actual concentration of the reslope standard. A reslope standard near the middle of the calibration range will usually give the best results.

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A single reslope standard, however, will not adequately compensate for major changes in instrument parameters. You should do a complete recalibration following parameter changes that are likely to alter the shape of the calibration curve. For example, large changes in flame composition or wavelength changes will likely require recalibration of the instrument.

Significant Figures

The 3100 software permits you to enter up to four significant figures for each reference standard concentration. This is more significant figures than are justified by atomic absorption data in most circumstances. The placement of the decimal point for Standard 1 fixes the position for all the standards. This means that values between 0.0001 and 9999 can be entered for Standard 1. The entries for the remainder of the standards are then fixed by that entry. If "5.000" is entered for Standard 1, then it would not be possible to enter "10" for Standard 2 since that entry would be formatted to match Standard 1 and would become "10.000," which is five significant figures--one too many. Rather, the entry for Standard 1 should be "5.00" or "5.0," in which case the value of 10 would be formatted as "10.00" or "10.0," both of which are acceptable.

The format of reference standard concentrations 2 through 8 and the reslope standard concentration will be formatted to match reference standard 1. Also the reference standards must be applied in sequence (though the concentrations can be in any order). Because of these restrictions you must enter the reference standard concentrations starting with Reference Standard 1 followed by Reference Standard 2, continuing up to Reference Standard 8. The concentration for the reslope standard can be entered at any time after Reference Standard 1 is defined. In the method of additions case, the entries for the standards represent concentrations resulting from the addition of standard to the sample.

Expansion Factor

The displayed result of a determination is the absorbance reading multiplied by an expansion factor. This expansion factor is computed automatically by the system and can be changed if desired by entering the Expansion Factor, the pressing: **[Expand]** (see Changing the Expansion Factor on page 17-4). Pressing **[CE] [Expand]** displays the Expansion Factor. It is important to differentiate between the two types of expansion. Entering an Expansion Factor erases the calibration curve.

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Computed Expansion During Calibration

When you run samples, the concentration is computed using Equation 2. This computation must take into account the position of the decimal point. However, when computing the expansion factor, the decimal point is ignored. The maximum permitted expansion is 100X. The minimum factor is 0.01X.

For example, if Standard 1 has a concentration of 40 mg/L (as entered in Parameter Entry for S1) and gives a measured absorbance of 0.008, then the instrument will compute an expansion factor of 40/8 = 5 and display the result as 40 mg/L. If the concentration for S1 were entered as 40.0, the system would compute an expansion factor of 400/8 = 50. In this case, the result would be displayed as 40.0 mg/L. Thus, entering 40.00 as the concentration of S1 would produce an expansion factor of 500, and the system would display an error message since the expansion must be less than 100X. The computed expansion factor can be obtained by pressing **[CE] [Expand]**.

The expansion factor is only meaningful if the plot is linear (i.e., one expansion factor applies throughout the calibration range). This implies that only a single-standard non-linear calibration or a linear calibration with any number of standards will produce a useful expansion factor. If the calibration curve is non-linear, the system will display the expansion factor computed for the standard having the lowest concentration.

Changing the Expansion Factor

You can enter a scale expansion factor directly by entering a number, then pressing **[Expand]**. Such an entry overrides any computed calibration curve. The system will multiply every subsequent absorbance reading by this value. To set the expansion:

- Enter the expansion factor, then press: **[Expand]**.

  The following screen appears:

```
EXPANSION
x.xx
```

Resetting the Expansion Factor

To reset the expansion to its default, 1X, press: **[1] Expand]**.

Displaying the Expansion Factor

Press: **[CE] [Expand]**.

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Calibration Options

The 3100 has three calibration options designed to accommodate most analytical problems. The following sections describe the options and provide suggestions regarding the situations where each is appropriate.

Linear

Most atomic absorption calibration curves are approximately linear at low concentration values, usually those giving absorbance values below 0.2. For the flame technique, the Standard Conditions section of the AA Cookbook specifies an approximate linear range for the more sensitive wavelengths of most elements with the flow spoiler. If you are making determinations within the linear range, you should choose this option as it allows the use of multiple standards with the single coefficient being determined by least squares. A linear calibration is also assumed when only the first standard is applied, even if the non-linear calibration method with multiple standards has been specified.

When you select "Linear," the 3100 uses Equation 3.

$$As = K_1 + K_2C \qquad \text{[Equation 3]}$$

where K_1 represents the intercept and K_2 represents the slope of the calibration curve.

K_1 and K_2 are determined using a least squares fit through the data points.

K_2 is determined using the standard concentration and absorbance values for the reference standards and represents the slope of the calibration function.

Up to eight reference standards can be used with this procedure, though one to three are usually adequate. One reference standard should always be located at the top of the analytical range to be covered and any other reference standards should be spread approximately equally along the calibration curve.

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Non-linear

You are likely to choose the "Non-linear" calibration option more frequently than any other available on the 3100. This option allows you to cover the widest possible concentration range, and you do not have to be concerned if the calibration relationship is truly linear. A typical non-linear calibration curve is shown in Figure 6-1.

The non-linear calibration algorithm of the 3100 employs Equation 2 with K_2 and K_3 being set equal to zero after the first calibration standard has been analyzed. As soon as the second standard is applied only K_3 is set equal to zero. As soon as three or more standards have been analyzed the full equation is used. The only exception occurs when an S shaped calibration curve is detected with the 3-coefficient version of the equation. In that case the calibration is recalculated using the 2-coefficient version of Equation 2. An S-shaped calibration situation occurs most commonly when the calibration curve is nearly linear, but when there is sufficient instrumental noise to make it appear S-shaped. In that case the 2-coefficient version of Equation 2 is an excellent choice. In all cases when the number of standards exceeds the number of coefficients, the method of least squares is used to determine the coefficients.

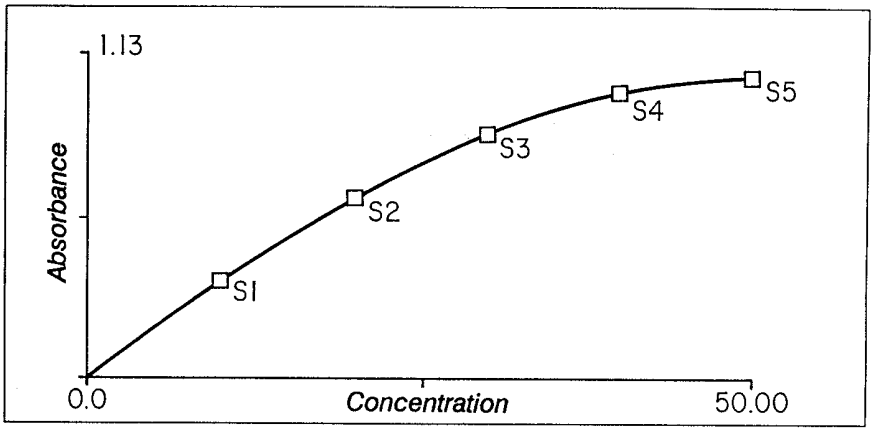


Figure 17-1. A Typical Multistandard, Non-linear Calibration Curve

Equation 2 can be used to cover a dynamic range greater than 100-fold. The following rules will help you to determine the optimum calibration range for your samples and to choose the required reference standard concentrations.

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1. The practical lower limit for a wide-ranging calibration curve is approximately 20 to 100 times the detection limit specified for the analyte in Analytical Methods for Atomic Absorption Spectrophotometry provided with your instrument. It is certainly possible to do analyses below that limit, but usually more replicates or longer integration times will be required, or, at very low concentrations, you may have to measure a blank between each sample.

2. The upper end of the calibration range will frequently be limited to about 6 times the linear range. Standard Conditions in the AA Cookbook list the linear range for the more sensitive lines of most elements when determined with a flame. This maximum concentration assumes that you are using at least three reference standards for calibration. If you are using only two reference standards, then the range covered will normally be no more than three times the linear range, sometimes less.

3. When using three reference standards, you should select them so that the concentration of the first is near the middle or upper end of the linear range, the concentration of the second is 3X the first, the concentration of the third is 6X the first (the 6:3:1 rule). The concentrations do not have to be exactly at these concentrations, but these values provide a good starting point. If the concentration range being covered is particularly wide, slightly greater spacing may produce better results; if the range is narrow, then the spacing is much less critical.

4. While three reference standards are adequate for most determinations performed using Equation 2, the 3100 allows up to eight reference standards to be used. If you are using more reference standards, you should insert them between the standards determined using rule 3 above so that they are spaced along the calibration curve.

You should consider these four rules as a starting point; based on them, prepare a set of standards covering the concentration range of interest and test to be sure that adequate performance is being provided under your operating conditions. In some cases it will be possible to calibrate above or below the predicted range, while in others the observed range of satisfactory calibration may be smaller. For many elements it will be possible to calibrate to a higher concentration value at the expense of a poorer detection limit by choosing a less sensitive line. The Standard Conditions section of the AA Cookbook lists several lines for most elements.

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Method of Additions

The "Method of Additions" option allows you to analyze samples where the sample matrix has a significant effect on the slope of the calibration curve, an effect that is different for each sample. To perform determinations using this method, the sample and aliquots of the sample that have been diluted equally and to which known amounts of the analyte have been added are analyzed. The absorbances of the sample and of the samples to which additions have been added are used to determine the slope of the calibration curve. This slope plus the absorbance of the sample allow the concentration of analyte in the sample to be calculated. An example of a method of additions calibration curve is shown in Figure 17-2.

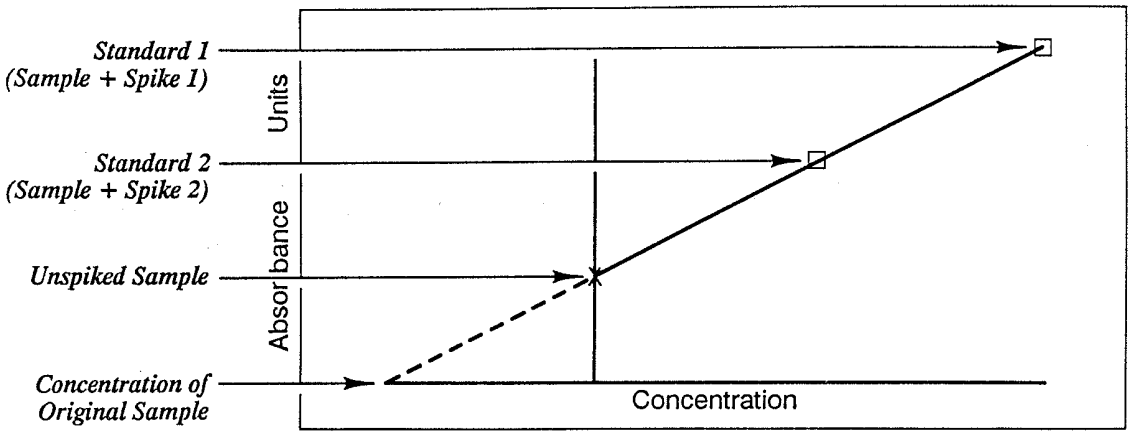


Figure 17-2. Example of a Method of Additions Calibration Curve

You should remember, however, that the method of additions is an indirect procedure that will never be as accurate as the direct determination of concentration from a calibration curve. It is always better to eliminate interferences by proper choice of atomizer conditions or by chemical treatment of your sample, if possible, than to use the method of additions. The Method of Additions should always be considered as a last resort rather than as the method of choice.

The method of additions makes two assumptions that must be true if the method is to yield accurate results:

1. The calibration curve must be linear up to the concentration of the sample plus the most concentrated addition.

2. The matrix must have the same effect on the added analyte as it has on the analyte present naturally in the sample.

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If either of these assumptions is not correct for your samples, you should not use "Method of Additions." The 3100 will display and print an error message if more than one addition has been used and if the resulting calibration curve is significantly non-linear.

For best results with the Method of Additions, you should use at least two additions, though one is sufficient to produce a result. It is also recommended that the largest addition be approximately equal to the expected concentration of analyte in the sample. As a test the 3100 calculates an m-ratio value using the following equation:

$$\text{m-ratio} = \frac{A_{add} - A_{samp}}{A_{samp}} \qquad \text{[Equation 4]}$$

In this equation A_{add} is the absorbance of the sample plus the largest addition and A_{samp} is the absorbance of the sample. If the m-ratio is less than 0.3, the 3100 displays an error message indicating that the calculated concentration may not be very accurate because the concentration of the addition was too small. Katzlaff (3) has discussed the question of addition concentration relative to accuracy of determination.

Method of Additions assumes that the concentration values entered in Parameter Entry are expressed in terms of the same volume as the original sample.

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Calibration Error Messages

Following is a list of error messages that may be displayed and printed if an error occurs during calibration. Explanations are included.

ERR:1 RESULT > ALLOWED FORMAT

The display format of the Model 3100 can accommodate four significant figures. That means that values between 0.0001 and 9999 can be displayed.

ERR:2 EXPANSION > 100

Expansion must be less than 100.

ERR:3 STD ABS NOT IN SEQUENCE

The calibration function of the Model 3100 checks to be sure that the standard concentration values and the measured absorbance values are in the same sequence. If they are not, the calibration curve is still calculated, but this message is displayed. The most probable causes are measuring the standards in the wrong sequence or, if the standards are close together, noise in the system may make them appear to have been measured in the wrong order.

ERR:4 RESLP STD ABS > THAN HI STD

The calibration function of the Model 3100 requires that the absorbance of the reslope standard be less than or equal to that of the highest standard used for the original calibration. If that is not true, no reslope operation is performed and the concentration of the reslope standard is displayed in terms of the existing calibration curve. The most likely cause of this message is measurement of an incorrect standard. It is suggested that the concentration of the reslope standard be near the middle of the calibration curve for the best results.

ERR:5 ABS & CONC SIGNS DO NOT MATCH

There are obscure situations, usually when the standards are incorrectly prepared, where a calibration curve is calculated which can give positive concentration values for negative absorbance values or negative concentration values for positive absorbance values. When this happens, you should prepare your standards again and be sure that the blank value is reasonable.

ERR:6 INVALID CONCENTRATION CURVE

There are obscure situations, usually when the standards are incorrectly prepared, where the calibration curve will roll over near or above the highest standard. When this happens, you should prepare your standards again and be sure that the blank value is reasonable.

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ERR:7 S SHAPED CURVE - 2 COEF USED

It is possible, particularly when the calibration is nearly linear, for the calibration curve to appear to be S-shaped. This can only happen when there are three or more standards. The 3-coefficient version of the calibration equation is not able to follow an S-shaped curve. When this happens, the software automatically uses the 2-coefficient equation with the coefficients determined by least squares, an excellent alternative when the calibration is nearly linear.

ERR:8 SAMPLE CONC > THAN HIGHEST STANDARD

It is good analytical practice to prepare the calibration curve with standards which cover the expected concentration range of the samples. Extrapolating the calibration curve to concentrations higher than the highest standard provides only an estimate of concentration. This error message indicates that the displayed concentration is only an estimate and that you should dilute and rerun the sample.

ERR:9 ABS BEYOND RANGE OF CALIB

As explained previously in this chapter, the function used by the Model 3100 software approaches an asymptote at some absorbance value. However, it may be possible for a very concentrated sample to produce an absorbance greater than that absorbance value. In those situations no concentration can be calculated.

ERR:10 SAMPLE CONC > MAXIMUM STD

These are obscure situations, usually when the standards are incorrectly prepared, where the calibration curve will roll over near or above the highest standard. If you choose to use this calibration curve to analyze samples, it will not be possible to calculate concentration values for those samples which fall in the rollover region of the calibration curve.

ERR:11 NO CONC VALUE ENTERED FOR STD

Enter a concentration for the standard.

ERR:12 NO CONC CURVE FOR RESLOPE

Before you attempt to reslope a calibration curve, it must have been generated using a blank and one or more standards.

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ERR:13 CURVE FIT

ERR:14 CURVE FIT

ERR:15 CURVE FIT

The calibration function of the Model 3100 uses a least squares routine which, in obscure situations, could fail to operate correctly. If this happens, it is likely that the standards have been prepared incorrectly or the standard concentrations are incorrect.

ERR:16 CALIB CURVE MAY NOT BE LINEAR

When you use more than one addition to perform the method of additions, the software makes a check to determine if the calibration curve is non-linear. The check procedure uses the absorbance of the sample with the least concentrated addition to calculate the expected absorbance of the sample with the most concentrated addition. If the actual absorbance of the sample with the most concentrated addition differs by more than 10% from the calculated value, this error message is displayed.

ERR:17 METHOD OF ADDITIONS TOO SMALL

For best results in performing the method of additions, the concentrations of the additions should be comparable to the concentration of analyte expected in the sample. The Model 3100 software performs an m-ratio check using the equation:

m-ratio - $(A_{max} - A_{samp}) / A_{samp}$

where A_{max} is the absorbance of the sample plus maximum addition and A_{samp} is the absorbance of the sample alone. If the m-ratio is less than 0.3, this error message is displayed.

ERR:20 RSLP STD > 20% IN ERROR

The measured concentration of the reslope standard based on the original calibration differs by more than 20% from the entered value. This indicates that an incorrect standard was used. No reslope operation was performed.

ERR:21 CORR COEFF ERROR

Error in the calculation of the correlation coefficient.

ERR:23 HI STD > THAN 2 COEF ASYMP

There are obscure situations, usually when the standards are incorrectly prepared, where the calibration curve will roll over near or above the highest standard. When this happens, you should prepare your standards again and be sure that the blank value is reasonable.

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2 Barnett, W. B. A calibration algorithm for atomic absorption. Spectrochim. Acta. 39B(6) 829-836; 1984.

3 Katzlaff, K. L. Anal. Chem. 51 232; 1979.

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