

DRAFT
LUMEX RP-91C
MERCURY BY ATOMIC
ABSORPTION SPECTROMETRY

Standard Operating Procedure

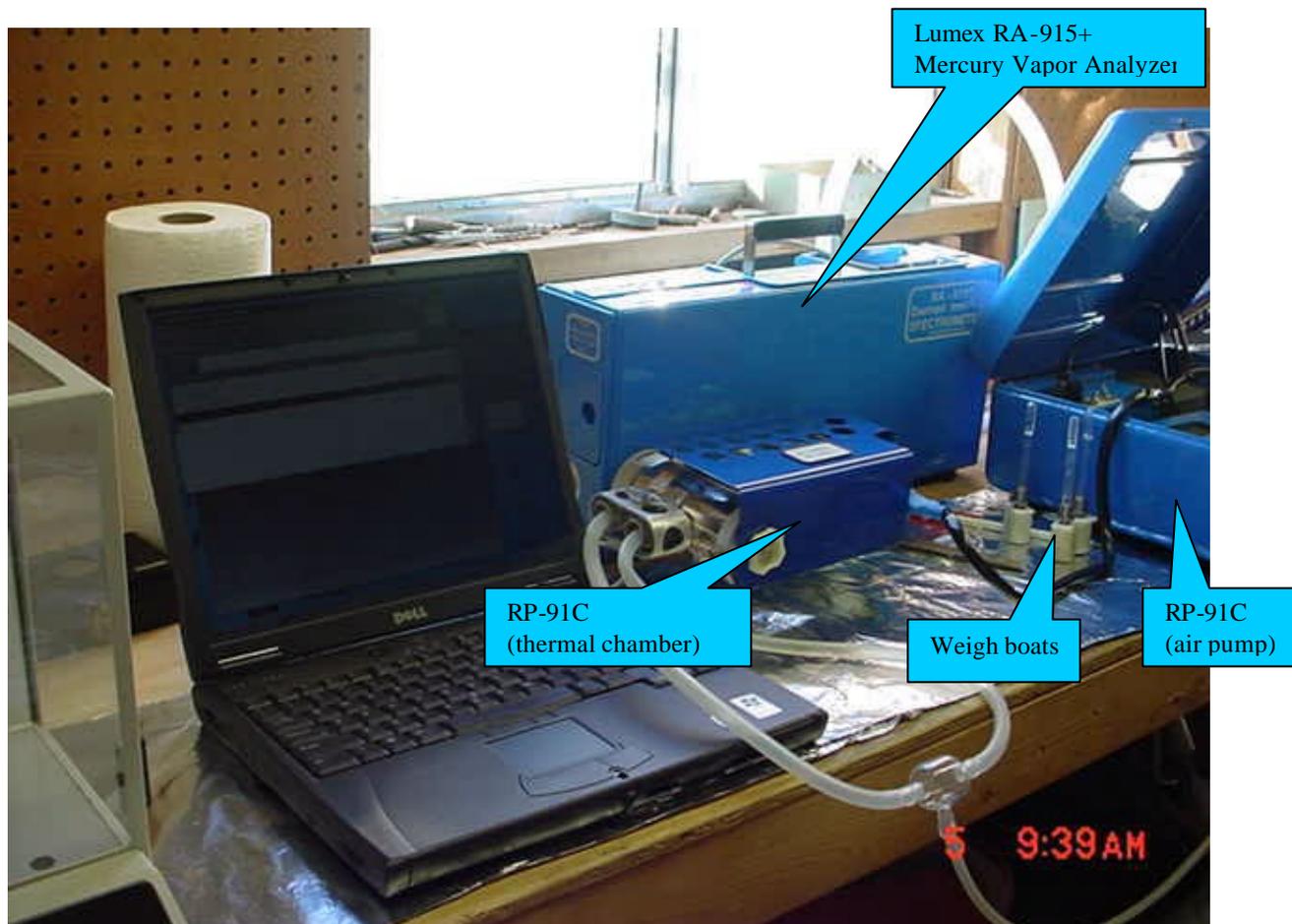


Table of Contents

| | | |
|------------|---|-----------|
| I | PRE-OPERATIONAL PROCEDURES | 1 |
| 1) | LAPTOP WITH 9-PIN PORT | 1 |
| 2) | INSTALL SOFTWARE | 1 |
| 3) | POWER | 1 |
| 4) | PERFORM LUMEX AIR MODE OPERATIONS CHECK | 1 |
| 5) | CONNECTIONS | 1 |
| 6) | WARM-UP | 1 |
| 7) | CALIBRATE/ CALIBRATION CHECK FOR BALANCE | 2 |
| 8) | CONNECT PC | 2 |
| 9) | DECONTAMINATE WEIGH BOATS | 4 |
| 10) | BEGIN DAILY RUN | 4 |
| II | OPERATIONS CHECK | 5 |
| 1) | CHECK RSD | 5 |
| 2) | CHECK BLUE (PMT) LINE | 5 |
| 3) | CHECK AIR FLOW SEALS | 5 |
| III | OBTAINING THE CALIBRATION | 6 |
| 1) | 1 ST POINT ON CALIBRATION CURVE (STANDARD) | 6 |
| A) | WEIGH STANDARD | 6 |
| B) | SET BASELINE | 6 |
| C) | ANALYZE STANDARD | 7 |
| D) | DECONTAMINATION OF WEIGH BOAT | 8 |
| E) | ENTER DATA IN TABLE | 8 |
| 2) | 2 ND THROUGH 7 TH POINTS ON CALIBRATION CURVE | 9 |
| 3) | CALCULATE CALIBRATION CURVE | 10 |
| 4) | CHECK CURVE, SAVE BITMAP, AND APPLY COEFFICIENTS | 11 |
| IV | QUALITY CONTROL CHECKS | 13 |
| 1) | PRECISION SAMPLE | 13 |
| 2) | ICV/CCV | 14 |
| 3) | METHOD BLANK | 14 |
| 4) | INSTRUMENT BLANK | 14 |
| 5) | CONTROL STANDARD | 15 |
| 6) | DUPLICATE SAMPLES | 15 |
| 7) | MONITORING QC RESULTS | 15 |
| V | ANALYZING A SAMPLE | 16 |
| 1) | WEIGH SAMPLE | 16 |
| 2) | SET BASELINE | 16 |
| 3) | ANALYZE SAMPLE | 16 |
| 4) | DECONTAMINATION OF WEIGH BOAT | 16 |
| 5) | ENTER DATA IN TABLE AND CALCULATE RESULTS | 16 |
| A) | ENTER SAMPLE ID | 16 |
| B) | ENTER SAMPLE WEIGHT | 17 |
| C) | CALCULATE RESULT | 17 |
| D) | CONTINUE ANALYZING SAMPLES | 17 |
| VI | DETECTION LIMIT STUDY | 18 |
| 1) | SITE SPECIFIC | 18 |
| 2) | SAMPLE SPECIFIC | 18 |
| VII | CONSIDERATIONS IN WHEN USING THE RP-91C | 19 |
| 1) | WORK STATION | 19 |
| 2) | INTERFERENCES | 19 |

| | | |
|-------------|---|-----------|
| 3) | SHIPPING | 19 |
| 4) | MATERIALS NEEDED | 19 |
| VIII | DOCUMENTATION OF RP-91C ACTIVITIES | 20 |
| 1) | INSTRUMENT LOGBOOKS | 20 |
| 2) | FIELD LABORATORY NOTEBOOKS | 20 |
| IX | ELECTRONIC DATA MANAGEMENT | 21 |
| 1) | EXPORTING RP-91C DATA TO EXCEL | 21 |
| 2) | ANALYST TO CHECK EXPORTED DATA AT THE END OF EACH DAY | 21 |
| X | DATA QUALITY/LABORATORY CONFIRMATION | 22 |
| 1) | FIELD SCREENING DATA | 22 |
| 2) | QUANTITY OF SAMPLES SENT TO LABORATORY | 22 |
| 3) | MASS OF SAMPLE REQUIRED FOR LABORATORY ANALYSIS | 22 |
| 4) | DATA REVIEW | 22 |

List of Figures

| | |
|--|----|
| Figure I - 1 - Lumex software start-up screen..... | 3 |
| Figure I - 2 - Two windows after software start-up..... | 4 |
| Figure III - 1- Set Baseline | 7 |
| Figure III - 2 – Click Start in Integration Window..... | 8 |
| Figure III - 3 – Go To Data Table from Analysis Graph | 9 |
| Figure III - 4 – Enter Sample Information in Table | 9 |
| Figure III - 5 – Creating Calibration Graph..... | 10 |
| Figure III - 6 – Example Table For Calibration..... | 10 |
| Figure III - 7 – Example Calibration Graph | 11 |
| Figure III - 8 – Apply Calibration Graph and Save as Bitmap..... | 12 |
| Figure V - 1 – Calculate the Result | 17 |
| Figure IX - 1 - Exporting Data to Excel..... | 21 |

Attachment Software Installation Troubleshooting (on CD)

I PRE-OPERATIONAL PROCEDURES

1) LAPTOP WITH 9-PIN PORT

Before departing for the field make sure your laptop has a 9-pin port. See “connections” below.

2) INSTALL SOFTWARE

Prior to departing for field install software on laptop PC according to manufacturer’s instructions. Note that the software has incompatibility issues with Borland databases. Additional instruction and software to remove the conflicting programs from Ohio Lumex is attached to this SOP.

3) POWER

The MVA has an internal battery that is charged via an AC adapter. The unit should be plugged in while using the RP-91C. The MVA also has an external battery as a back up. If needed, it may be possible to use the MVA + RP-91C with the internal/external batteries fully charged. However, it is preferred to use a stable power source, such as a wall outlet.

Plug balance into surge protector. Note: Set-up and calibrate analytical balance at least **2 hours prior to sample analysis** to allow for stabilization.

4) PERFORM LUMEX AIR MODE OPERATIONS CHECK

Prior to connecting RP-91C, set Lumex RA-915+ Mercury Vapor Analyzer (MVA) for air mode (optical bridge position III) without the PC and check %R according to manufacturer’s instructions. If air mode working properly, go to step 3. Otherwise stop and troubleshoot MVA operation. After ensuring the MVA is in working order, turn off the MVA and switch the optical bridge on the back of the unit to position I.

5) CONNECTIONS

Before turning on the MVA, make sure that the external thermal chamber, the power source for the thermal chamber and compressor, and the PC cable are properly connected. Use a heavy-duty surge protector and/or a stable power source for best results. Additionally, best results have been achieved by connecting the 9-pin portion of the PC cable to the laptop computer after the MVA + RP-91C system and laptop are running.

6) WARM-UP

Once all connections have been made, with the exception of the 9-pin port to the laptop PC, turn on the external power source (black box) and begin warming the thermal unit. Turn on the air pump (attached to the blue RP-91C case) with the lever switched to 2 and adjust flow rate to 2 liters per minute (l/min) using the black knob. While waiting for the thermal unit to warm-up and the air flow rate to stabilize (APPROXIMATELY 45 MINUTES), complete steps six (6) and seven (7) below.

Note about flow rate: that the manufacturer’s instruction manual states 1 l/min and the manufacturer’s internal SOP received after publishing the instruction manual states 3 l/min.

Therefore, flow rates between 1 and 3 liters per minute are acceptable. For the START Region 9 SOP, we will use 2 l/min. If the analyst feels that the peak time is too small and they are unable to determine matrix interference, they can reduce the flow rate to 1 l/min. A deviation from the 2 l/min flow rate should be discussed with the manufacturer and START project manager prior to implementation.

7) CALIBRATE/ CALIBRATION CHECK FOR BALANCE

Analytical balance should be calibrated following manufacturer's instructions after being moved to a new location. Also, after moving the balance, it will be necessary to wait 2 hours before calibration (see "power" section above).

Verify that the balance is working appropriately by testing weights for the range of sample you plan to use (typically 5 mg to 5 grams). If the balance is not within 0.5 mg of the correct weight, then:

- a) double-check that it is level and adjust screws at back of balance as necessary, and/or
- b) conduct an internal calibration by pressing and holding the "mode" section of the black bar of the balance and re-check the weights after the internal calibration is complete. Record the results of the weight check in the field laboratory notebook.

8) CONNECT PC

Connect 9-pin cable to PC and start Lumex RA-915+ software. Choose complex from the pop-up screen (see Figure I - 1). Two windows will appear (see Figure I - 2)

Figure I - 1 - Lumex software start-up screen.

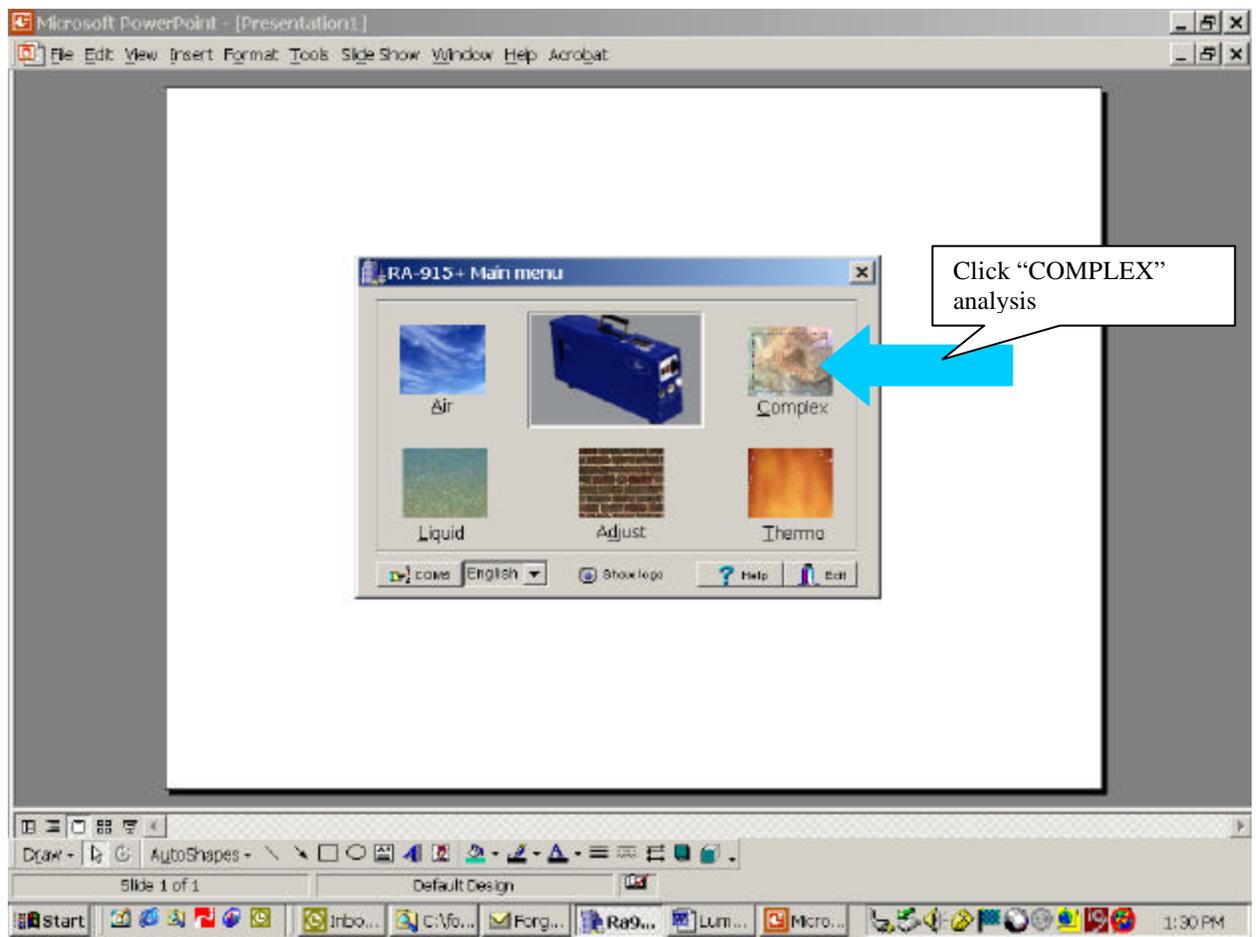
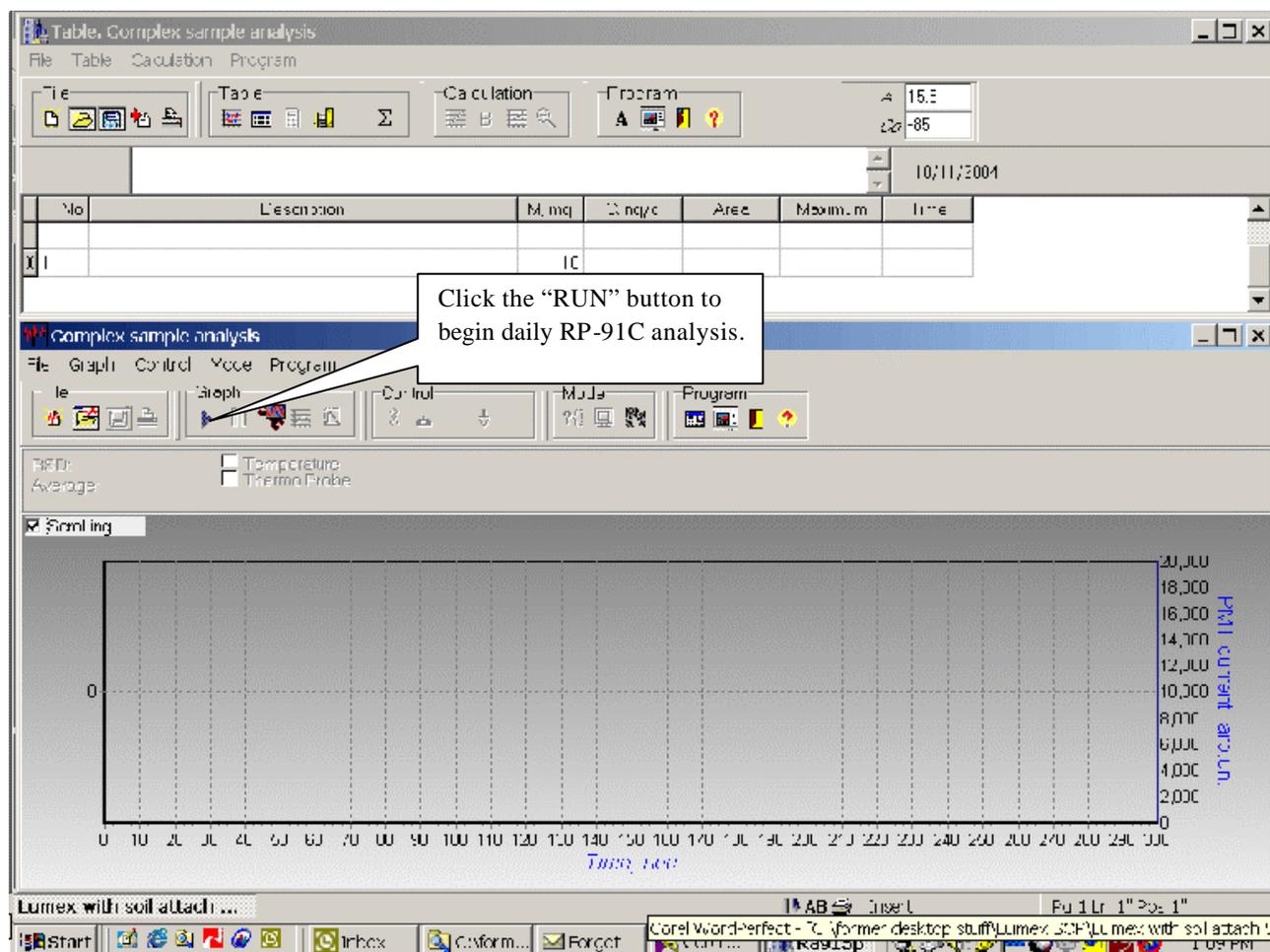


Figure I - 2 - Two windows after software start-up.



9) DECONTAMINATE WEIGH BOATS

Once warm-up is complete (thermal unit is red hot on the inside and air flow is stable at 3 l/min), decontaminate weigh boats by inserting each weigh boat into thermal unit for approximately 30 seconds.

10) BEGIN DAILY RUN

Go to the graph window of the complex sample analysis and click run (arrow) on toolbar (see Figure I - 2). Two lines will appear. The dark red line on the bottom part of the screen is the baseline analytical signal, and the blue line in the middle of the graph is the total intensity (the photo multiplier (PMT) current) signal

II OPERATIONS CHECK

The operations check is to be completed after a 45-minute warm-up and after items specified in Section I have been completed.

1) CHECK RSD

After the system has been running for a minute, click the statistics button on the toolbar of the graph window. The RSD will appear in the upper left of the screen. RSD should be less than 15% according to manufacturer's instructions, but it is typically less than 5%. Record the value in the field logbook.

2) CHECK BLUE (PMT) LINE

The current value of the blue line should be less than 8,000 based on the arbitrary units on the left side of the graph. If it is not, stop operation and clean the windows of the analytical cell of the RP-91C (see user's manual for instructions). Record the value in the field logbook.

3) CHECK AIR FLOW SEALS

Check the seal of the gas duct by pinching the silicone tube that connects the gas tee-branch to the absorption filter. The flow rate on the rotameter should be less than 2 l/min. Record the value in the field logbook.

III OBTAINING THE CALIBRATION

There are two ways to obtain a calibration curve. The first method, the one described below, uses one control standard. The alternate calibration method is using several control standards.

1) 1ST POINT ON CALIBRATION CURVE (STANDARD)¹

A) WEIGH STANDARD

Insert room temperature weigh boat onto center of balance and tare.

*** The doors of the balance must be close to ensure accurate analysis.**

Current experience using the Mettler AE200 shows that the analyst will have to put the white base of the weigh boat off of the center of the balance tray or center the white base of the weigh boat on the balance, but angle the glass weigh boat portion so that the doors of the balance will close. The analyst should try to achieve the same placement of the weigh boat with each analysis.

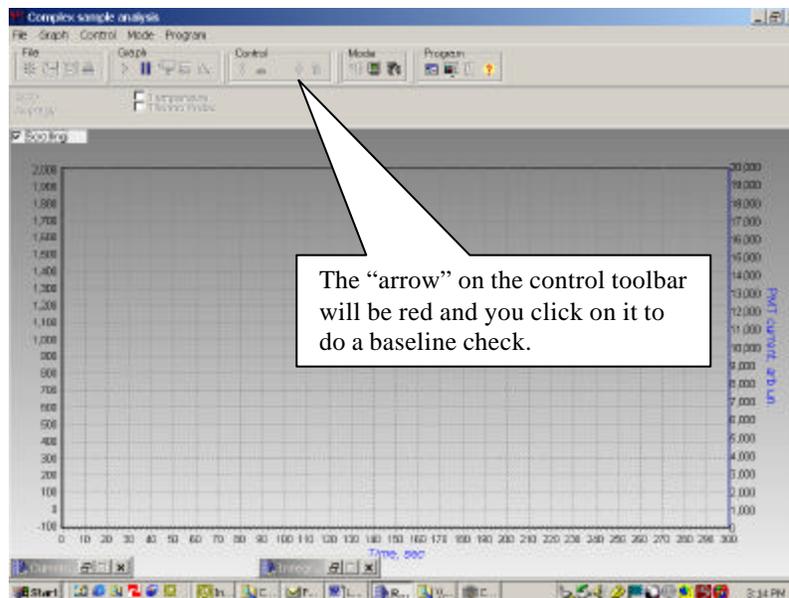
Insert a known amount of a standard into the weigh boat while it sits on the balance. (Note: manufacturer recommends starting with 20 mg, but it is ultimately up to the analyst to establish a curve that will encompass all potential mercury concentrations.) Record the mass of standard used in the field logbook.

B) SET BASELINE

Click baseline check (the red arrow) on the control toolbar. Click baseline check on the control toolbar while the thermal chamber is empty. Wait about 10 seconds and then click baseline check button again to establish the baseline (see **Figure III - 1**).

¹ Prior to writing this SOP, START was using an empty weigh boat as a blank. However, the manufacturer has indicated that this is not a necessary step and the software automatically places the calibration curve through zero.

Figure III - 1– Set Baseline



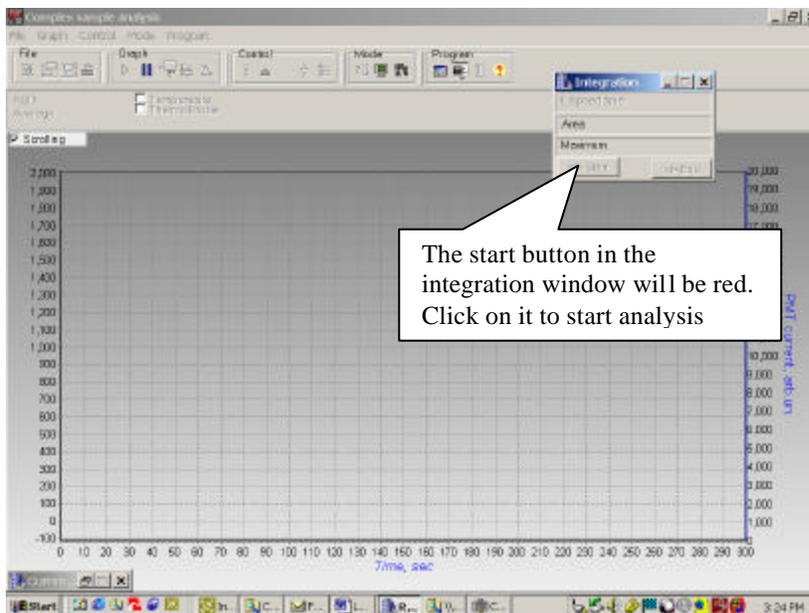
C) ANALYZE STANDARD

Click start in the integration window (see **Figure III - 2**), remove weigh boat from balance and insert into thermal chamber. When the sample peak (orange line) returns to the baseline (dark red line), usually after 45-90 seconds, click end in the integration window.

Note: While the standard is being analyzed, you can set up balance/ weigh boat for the next standard.

Note: To assist in promoting consistency between analysts, you can watch the current value window. When the number in the current value reaches the same number it was during the baseline check, then click stop in the integration window.

Figure III - 2 – Click Start in Integration Window



D) DECONTAMINATION OF WEIGH BOAT

After analyzing the standard, remove it from the thermal chamber. Empty remaining contents into glass or plastic jar for later disposal. Re- insert the weigh boat into the thermal chamber for decontamination (about 30 seconds).

E) ENTER DATA IN TABLE

a) Click the table button on the program toolbar (see **Figure III - 3**). In row 1, double click the description field and click on standard in the pop-up window (see **Figure III - 4**).

b) Enter the amount of standard in the weigh boat in the “M, mg” field. After entering data in the table, click the graph button on the table toolbar to continue analyzing standards.

Figure III - 3 – Go To Data Table from Analysis Graph

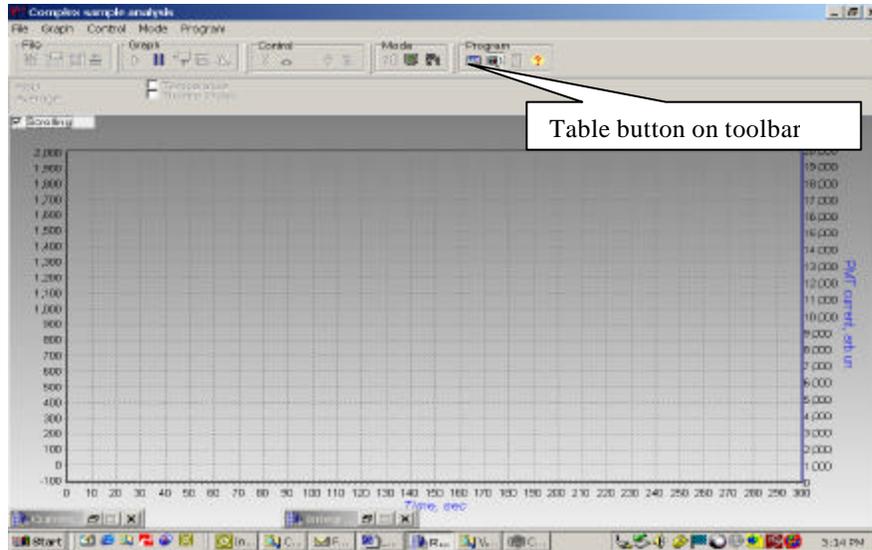
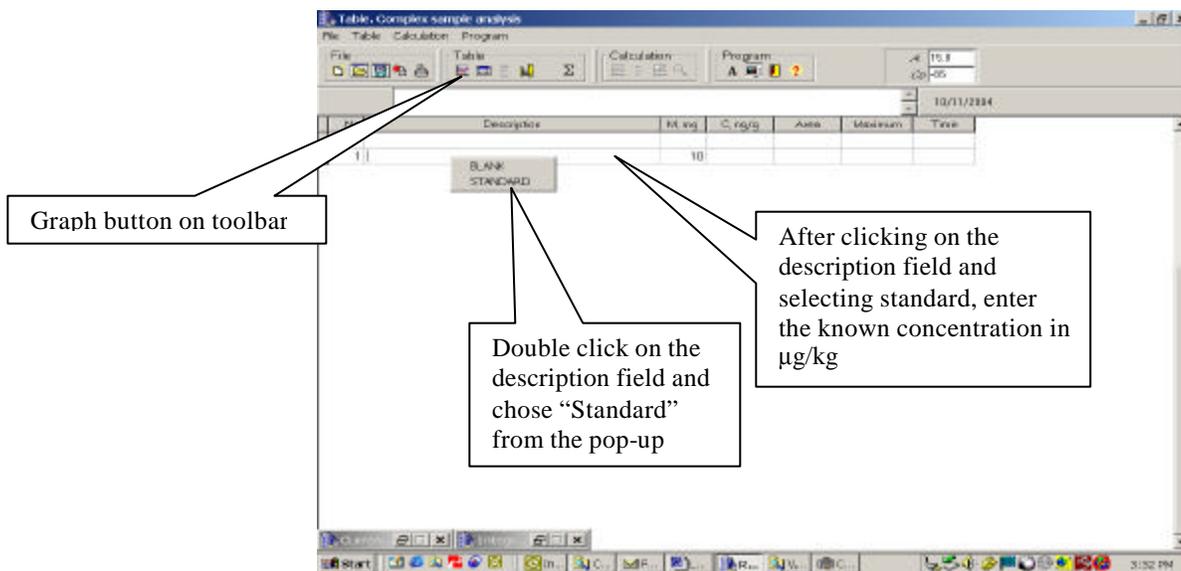


Figure III - 4 – Enter Sample Information in Table



2) 2nd through 7th POINTS ON CALIBRATION CURVE

Repeat step III-1 six times with varying weights of standard (e.g. double and triple weights or additional 10 mg each time). For example:

1st point on calibration curve = 10 mg of standard

2nd point = 20 mg of standard

3rd point = 30 mg of standard

4th point = 40 mg of standard

etc...

3) CALCULATE CALIBRATION CURVE

Click the select button on the table toolbar. Click on the first line, the one with the blank, and hold down the shift button while pressing the down arrow to highlight the blank and all the standards (see **Figure III - 5** and **Figure III - 6**). On the Calculation toolbar click the Cal Graph button (see **Figure III - 7**).

Figure III - 5 – Creating Calibration Graph

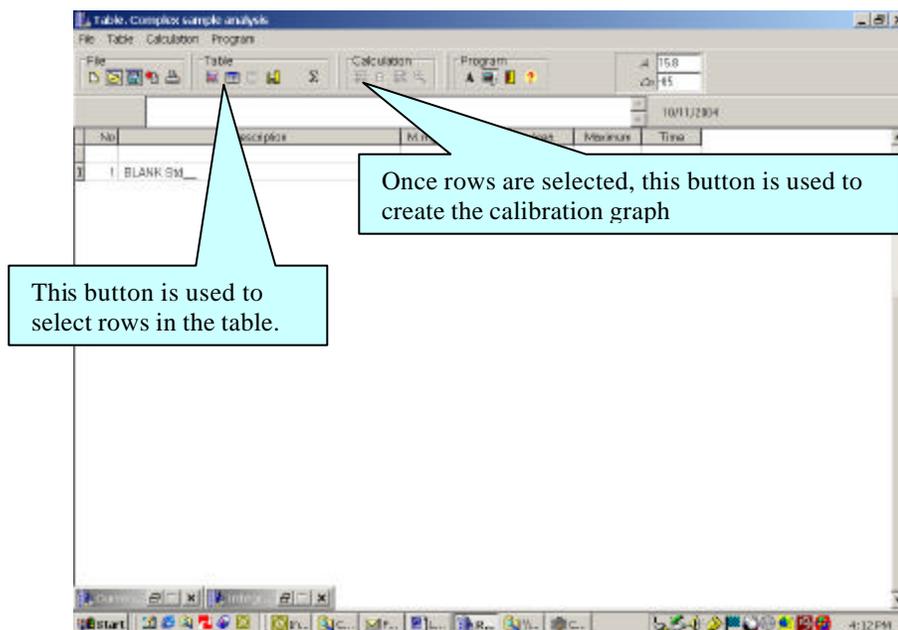
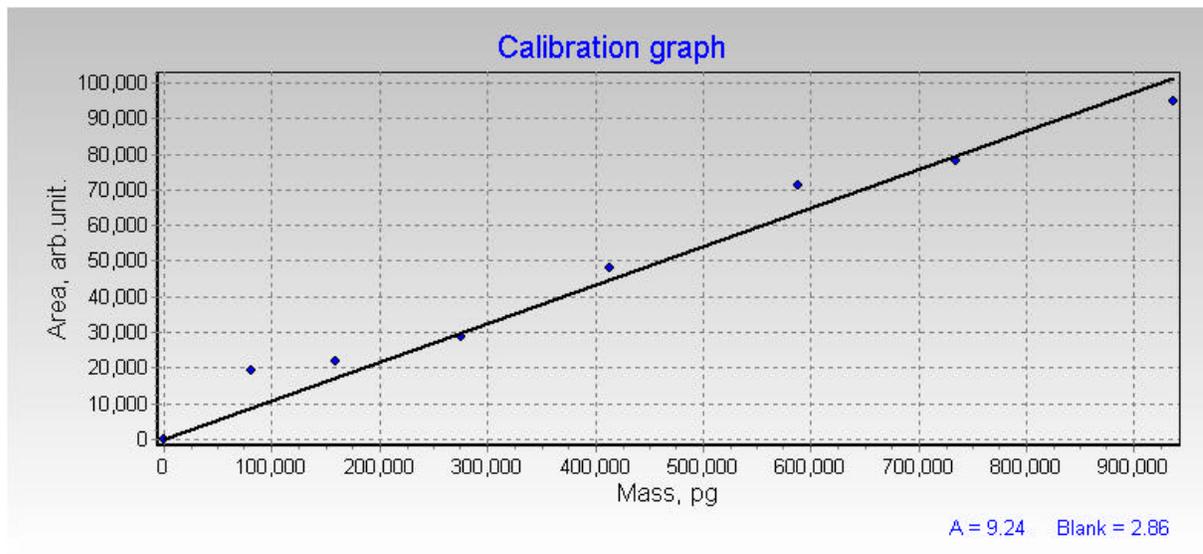


Figure III - 6 – Example Table For Calibration

| No | Description | M, mg | C, ng/g | Area | Maximum | Time |
|----|---------------|-------|---------|-------|---------|------------|
| 1 | BLANK | 1.4 | | | | 8:19:30 AM |
| 2 | Std_16900 | 4.8 | 18100 | 48200 | 11900 | 8:23:34 AM |
| 3 | Std_16900 | 9.4 | 18900 | 71500 | 15000 | 8:26:57 AM |
| 4 | Std_16900 | 16.3 | 16500 | 78200 | 17100 | 8:30:18 AM |
| 5 | Std_16900 | 24.5 | 15700 | 94800 | 14600 | 8:33:35 AM |
| 6 | Std_16900 | 31.7 | 29400 | 29700 | 8040 | 8:46:33 AM |
| 7 | Std_16900 | 39.1 | 11500 | 14600 | 4470 | 8:50:30 AM |
| 8 | Std_16900 | 46.5 | 3910 | 6310 | 1830 | 8:56:33 AM |
| 9 | LCS-NIST 2710 | 9.3 | 29400 | 29700 | 8040 | 8:46:33 AM |
| 10 | CCV-ERA 540 | 11.7 | 11500 | 14600 | 4470 | 8:50:30 AM |
| 11 | B3-150-0 | 14.8 | 3910 | 6310 | 1830 | 8:56:33 AM |

Callout: Lines 1 through 8 of this table are highlighted to create the calibration graph.

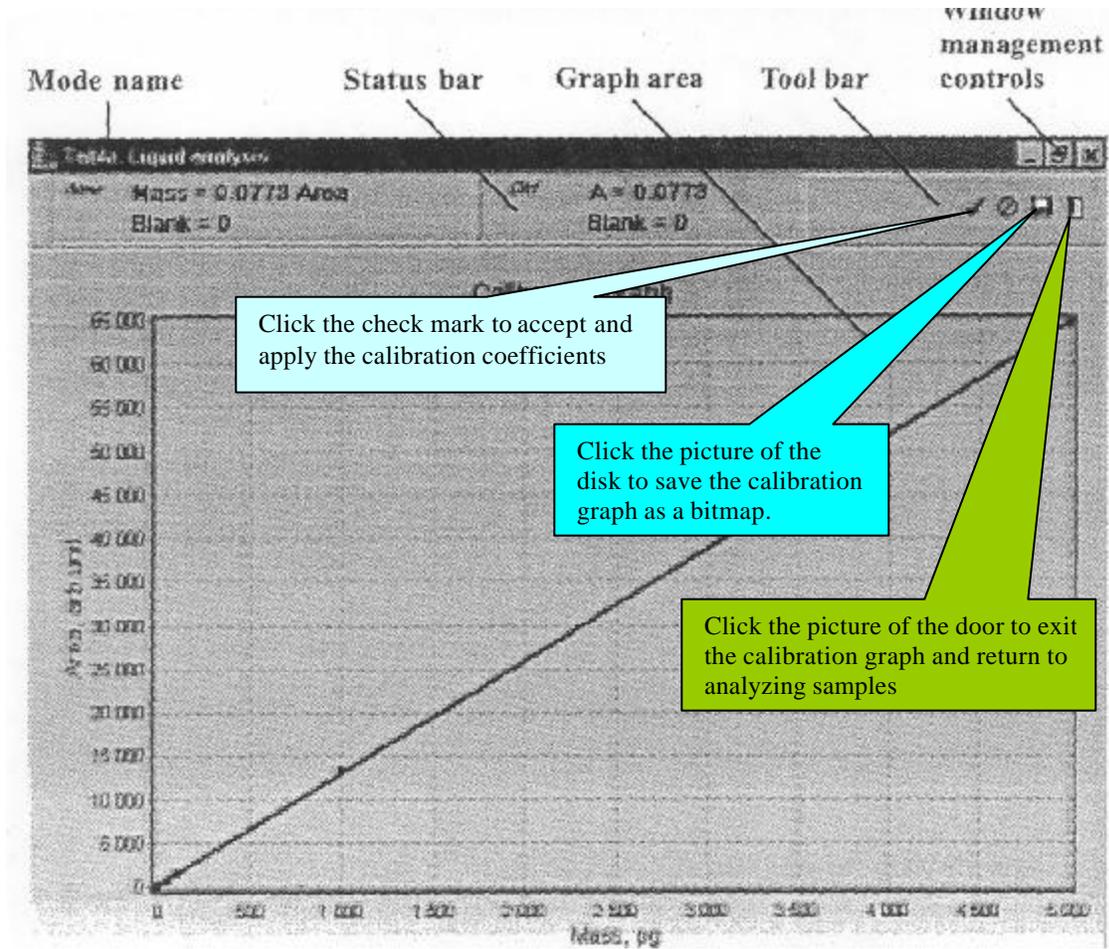
Figure III - 7 – Example Calibration Graph



4) CHECK CURVE, SAVE BITMAP, AND APPLY COEFFICIENTS

The analyst should check that the points along the curve are relatively linear and no large outliers are present. Once satisfied with the curve, the analyst should save the calibration curve as a bitmap for future data validation and reference. Lastly, click the red check mark to apply the calibration coefficients and begin analyzing samples (see Figure III - 8).

Figure III - 8 – Apply Calibration Graph and Save as Bitmap



IV QUALITY CONTROL CHECKS

At this point you should have a linear calibration curve based on 7 standard points and a blank. At the beginning of each project, you also should perform a precision sample to determine the approximate detection limits. Additional quality control checks will also be performed throughout the day, as described below:

| When | Frequency | QC Type |
|------------------|--|---|
| First Day | Once | Precision Sample |
| | As often as necessary | Balance calibration |
| | | |
| Everyday | At the beginning of each day | Balance calibration check |
| | At the beginning of each day and as necessary | 7 point calibration |
| | At the beginning of each day, after calibration | Method Blank (sand) |
| | At the beginning of each day, after calibration | Initial Calibration Verification (ICV) |
| | At the beginning of each day, after calibration and at the end of each day | Laboratory Control Sample (LCS) – 2 nd source standard |
| | Every 10 samples | Continuing Calibration Verification (CCV) |
| | Every 10 samples after CCV | Instrument blank (aka continuing calibration blank [CCB]) |
| | Alternate types of duplicates after every 10 samples | Duplicate samples (re-run and preparation duplicates) |

1) PRECISION SAMPLE

The precision of the method is monitored by analyzing a sample with concentrations near the action level for mercury. The precision sample should be a site-specific field sample analyzed by definitive methods or a prepared standard such as the sample used for the ICV/CCV (if a site-specific standard is not available). At least one precision sequence should be run initially. If the initial precision sequence is based on a prepared standard, a second precision sequence should be performed if an acceptable, homogenized site-specific sample can be identified. Each precision sample should be analyzed seven times in replicate using approximately the same sample mass within +/- 2mg.

After analysis, the relative standard deviation (RSD) should be calculated. Usually it is easier to calculate the RSD using a calculator with statistical functions or a spreadsheet such as Excel or Lotus; however, the RSD may be calculated without these items using the following equation:

$$RSD = (SD/\bar{x}) * 100$$

Where :

SD =Standard deviation of the seven concentrations for mercury.

Given by:

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

Where:

x_i is the mercury concentration

\bar{x} is the mean concentration of the seven samples

N is the number of samples

If the RSD is found to be greater than twenty percent, then the analyst should consider recalibration, ensuring weigh boats are at room temperature, and more precise balance technique.

NOTE: The RSD range listed above is recommended as a general guideline. Acceptable RSD ranges should be updated based on performance data gathered to demonstrate the capabilities of the RP-91C and based on the data quality objectives.

2) ICV/CCV

The ICV/CCV is the same sample. The ICV/CCV measures the instrument drift during the day and should ideally be representative of the samples which are to be analyzed with respect to mineralogy, particle size, and homogeneity and should contain contaminants at concentrations near the action level for mercury. In theory, the ideal ICV/CCV will consist of a soil sample from the site that has been analyzed by definitive methods and has a known value near the action level. If such an ICV/CCV sample is not available, then a pre-prepared NIST or other acceptable standard containing the contaminants of interest near the action level should be used. The percent difference between the known value of the ICV/CCV and the value returned by the RP-91C should be less than 30 percent for screening level data. Some type of corrective action, such as cleaning the detector or recalibration, should be taken if the CCV values vary more than fifty percent from the ICV. The percent difference may be calculated as follows:

$$\%D = ((C_s - C_k) / C_k) * 100$$

where:

%D = Percent difference

C_k = Certified concentration of standard sample

C_s = Measured concentration of standard sample

3) METHOD BLANK

A method blank (sand blank) should be analyzed initially. In order for the RP-91C results to be acceptable for the sand blank, no mercury may be present. Ideally the method blank is acid-washed sand prepared in the field exactly as samples are being prepared.

4) INSTRUMENT BLANK

An instrument blank must be analyzed initially, after every ten samples (after the CCV), and at the end of the day. This QC sample is also known as a continuing calibration blank

(CCB). The instrument blank can be either an empty weigh boat or a weigh boat with acid washed sand.

5) CONTROL STANDARD

A control standard should be analyzed at least initially and at the end of the day. Until the RP-91C has had more field testing, it is ideal to analyze the control standard with all other QC samples, after every 10 investigative samples. The control standard is a pre-prepared NIST standard or other traceable standard. It can not be the same sample as the CCV. Control standard data is used to help evaluate the overall quality of the data. Ideally the percent difference between the known value of the control standard and the value returned by the RP-91C should be less than 30 percent. However, no action is required if value is outside those parameters.

6) DUPLICATE SAMPLES

Duplicate samples should be analyzed every 10 samples. Preparation duplicates should be within 35% RPD of each other and analyzed every 20 samples. Instrument duplicate analyses on the same sample should be performed every 20 samples. An instrument duplicate analysis should be within 20% RPD of each other. Corrective actions are not required for RPDs outside specified parameters, however failure to meet those RPD parameters, may indicate inconsistencies in sample preparation or analyses.

The relative percent difference (RPD) is calculated as:

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} * 100$$

where:

| | | |
|----------------|---|---|
| RPD | = | the relative percent difference |
| X ₁ | = | the concentration of the sample |
| X ₂ | = | the concentration of the duplicate sample |

7) MONITORING QC RESULTS

The monitoring of QC data as they are generated is essential to the effective use of the RP-91C. Corrective actions should be initiated if RP-91C data is deviating from acceptance ranges. Results should not be reported if monitoring indicates that the RP-91C data is significantly deviating from acceptance ranges. Reported data must be qualified if monitoring indicates QC deviations.

V ANALYZING A SAMPLE

The procedure for sample analysis is very much the same as analyzing the standards, but you won't enter a known concentration in the table, you will enter the sample ID instead. The concentration range for the MVA is 5 to 100 mg/kg mercury.

1) WEIGH SAMPLE

Insert room temperature weigh boat onto center of balance and tare. Insert a known amount of a standard into the boat (Note: Previously in the field we were using 20 to 40 mg of sample for the first analysis of a sample.) At least 5 mg should be used for each sample. Measuring sample masses less than 5 mg can introduce error.

2) SET BASELINE

Click baseline check (see Click baseline check on the control toolbar while the thermal chamber is empty. Wait about 10 seconds and then click baseline check button again to establish the baseline (see **Figure III - 1**).

Figure III - 1

3) ANALYZE SAMPLE

Click start in the integration window (see Figure III - 2), remove weigh boat from balance and insert into thermal chamber. When the sample peak (orange line) returns to the baseline (dark red line), usually after 45-90 seconds, click end in the integration window.

Note: Analyst should watch the peak develop. If the maximum concentration of the blue, PMT line exceeds 15,000 (numbers on left side of screen) or if the blue, PMT line changes by more or less than 2,000 a matrix interference is most likely present and the sample will have to be re-analyzed using less mass.

Note: While the sample is being analyzed, you can set up balance/ weigh boat for the next sample.

4) DECONTAMINATION OF WEIGH BOAT

After analyzing the sample, remove it from the thermal chamber. Empty remaining contents into glass or plastic jar for later disposal. Re- insert the weigh boat into the thermal chamber for decontamination (about 30 seconds). Let it cool before re-using. Cooling of the weigh boat may take up to 5 minutes so it is best to use the weigh boats in a rotation.

5) ENTER DATA IN TABLE and CALCULATE RESULTS

Click the table button on the program toolbar.

A) ENTER SAMPLE ID

In the row of the table that has an area, but no description, enter the sample ID. Also, enter the sample ID in the field laboratory notebook.

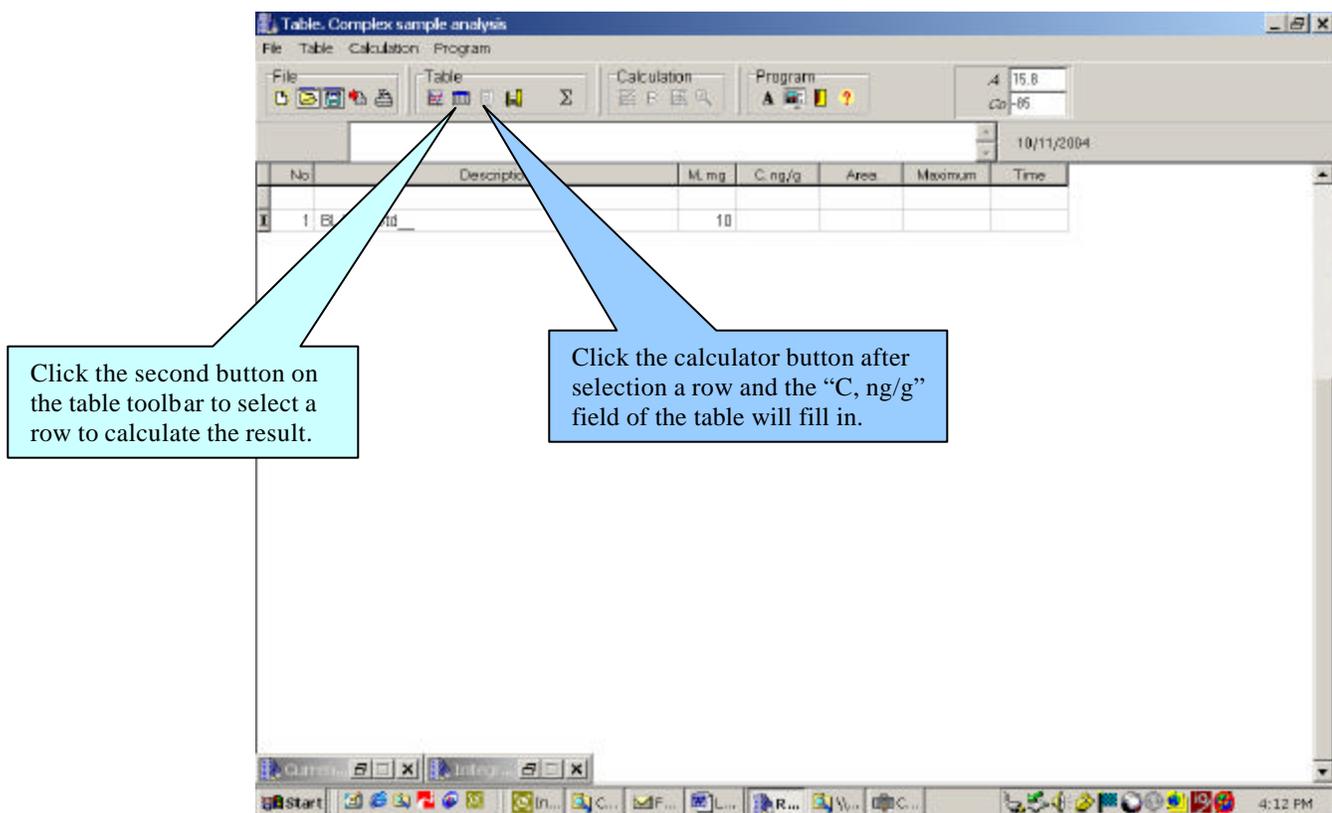
B) ENTER SAMPLE WEIGHT

Enter the amount of sample in the weigh boat in the “M, mg” field of the software table and in the field laboratory notebook.

C) CALCULATE RESULT

Calculate the result by selecting the row in the table and clicking the calculation button on the table toolbar (see **Figure V - 1**). Enter the result in the field laboratory notebook.

Figure V - 1 – Calculate the Result



D) CONTINUE ANALYZING SAMPLES

After entering data in the table, click the graph button on the table toolbar to continue analyzing samples.

VI DETECTION LIMIT STUDY

The manufacturer's detection limit is provided in the equipment manual as 0.5 µg/kg using 200 mg of sample. This detection limit should be considered as unlikely to reach for several reasons. The first reason is that it is difficult to get 200 mg of sample into the weigh boats. The second reason is that using 200 mg of sample at a previous site (e.g. Rinconada Mine Removal Action) yielded irregular peaks in which the sample volume had to be reduced to obtain acceptable data. Until more site-specific data is available, the detection limit used in the planning stages for the RP-91C should be approximately 5 mg/kg. Site-specific detection limits must then be determined while in the field by either of the following two procedures.

1) SITE SPECIFIC

A site-specific sample should be analyzed seven times. The method detection limit (MDL) for mercury will be defined as three times the standard deviation from the seven replicate analyses. The practical quantitation limit (PQL) will be defined as ten times the standard deviation.

2) SAMPLE SPECIFIC

A second method that may be used to determine the detection limit involves using the standard deviation value given for each element in an individual sample. Three times the given standard deviation for each element's individual reading is the MDL. Ten times this standard deviation is the PQL.

Note: The range between three and ten times the standard deviation for each element is a gray area not addressed. Ideally one would like to only use the RP-91C when the action level is above the estimated PQL; however, this often is not plausible. To justify generating quantitative data with concentrations between three and ten times the standard deviation, the analyst must document that the RP-91C passes all the pertinent Quality Control measures listed in Sections II, III and IV of this SOP for those concentrations.

Note: Sample specific detection limits should always be determined in order to monitor potential increases in detection limits. Detection limits may change with variations in mercury concentrations.

VII CONSIDERATIONS IN WHEN USING THE RP-91C

1) WORK STATION

Keep the RP-91C out of direct sunlight and try to maintain a constant cool temperature in the workspace. The balance, sensitive to the 0.5 mg level, will need the most stable environment possible. (For example: it is likely that a field trailer will be unacceptable for the balance because constant small movements will cause the balance to lose its calibration).

2) INTERFERENCES

No known interferences

3) SHIPPING

If available, a shock and heat sensor shall be attached to case prior to shipping, in order to monitor shipping conditions. The balance and weigh boats are especially sensitive when shipping. Ideally, the balance will be driven to the field location from the San Francisco Eagle Instrument Warehouse. However, when driving the balance is not possible, the balance should be shipped days in advance to allow for the possibility of breakage and time for repair prior to fieldwork.

4) MATERIALS NEEDED

The following is a list of items to be used in the field laboratory.

- 2 soil attachments (for extra weigh boats)
- 1 Lumex RA-915+ Mercury Vapor Analyzer
- 1 balance (accuracy to below 1 mg)
- non-slip surface for balance operation
- nitrile gloves
- spatulas (for standards and samples)
- 2 soil standards (at least one should be at the mercury action level) (can be procured from either EPA QATS or outside firm such as ERA, consult START QA manager or START project chemist for more information)
- soil samples
- laptop computer with 9-pin port
- Lumex SOP
- Logbooks
- Decontamination material for spatula (alcohol wipes)
- Paper towels
- Quart-size plastic bags

VIII DOCUMENTATION OF RP-91C ACTIVITIES

1) INSTRUMENT LOGBOOKS

Each RP-91C and accompanying RA-915+ analyzer will have an instrument logbook assigned to it for documentation of instrument activity, problems, maintenance and repairs. The following field information must be documented in the instrument logbook for each day of its field use:

- Date of operation, site name, and operator name(s).
- Operations and QC check status summary.
- Summary and status of any RP-91C instrument problems or repairs encountered.
- Signatures of analysts.

The following problem, maintenance and repair information must also be documented in the instrument logbook:

- Date of monthly maintenance, repairs or problems.
- Summary of monthly maintenance, repairs or problems.
- Signatures of documenters.

2) FIELD LABORATORY NOTEBOOKS

Each project using the RP-91C will create a field analytical logbook for documentation of RP-91C field analytical activities. The field analytical logbook must be referenced in the site logbook. The following information must be documented in the field analytical logbook for each day of use:

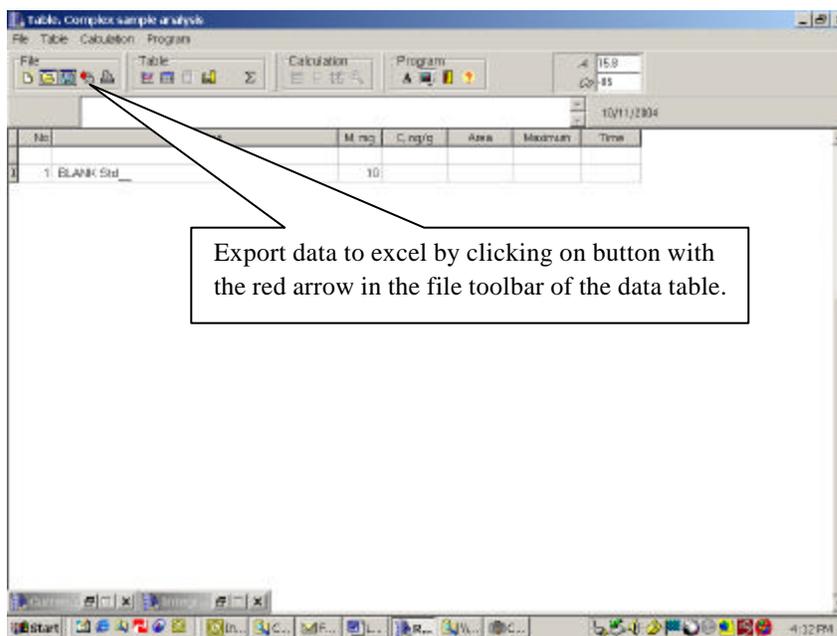
- Date of operation, site name, and operator name(s)
- All optional settings and QC information indicated in sections II through IV of this SOP
- The analysis result and standard deviation for all contaminants of concern for each sample and standard analyzed
- Sample Numbers (or Sample ID) of the samples analyzed on the instrument that day
- Summary and status of any RP-91C problems encountered
- Signature of documenters

IX ELECTRONIC DATA MANAGEMENT

1) EXPORTING RP-91C DATA TO EXCEL

RP-91C data can be exported to excel from the data table in the proprietary software to supplement the documentation of analytical results (see Figure IX - 1). The exported data must not be used in lieu of logbook or field data sheets.

Figure IX - 1 - Exporting Data to Excel



2) ANALYST TO CHECK EXPORTED DATA AT THE END OF EACH DAY

At the end of each day, the analyst should double-check the exported excel file for typos and errors. In addition, this is a good time to add a column for sample concentrations in mg/kg, if appropriate.

X DATA QUALITY/LABORATORY CONFIRMATION

1) FIELD SCREENING DATA

All data produced by the RP-91C is considered field-screening data and must be presented to any potential data user flagged with a “J”, for estimated quality. If better defined data is required the “field screening plus 10%” category may be achieved by sending at least 10% of results above the detection limit on the RP-91C to a laboratory for confirmation analysis by definitive methods. A regression may then be performed using the RP-91C and lab data to obtain information about the correlation between the two data sets. Note that although confirming 10% of the RP-91C samples results by definitive methods increases the *knowledge* of the quality of the data, it does not inherently increase the *quality* of the data itself. Also, all screening plus 10% category data must still be flagged with a “J.”

2) QUANTITY OF SAMPLES SENT TO LABORATORY

A minimum of one sample for every ten samples analyzed by the RP-91C should be sent for laboratory confirmation to verify the quality of RP-91C data. The ratio of RP-91C samples to confirmation samples may vary with the site-specific data quality objective. Confirmation samples should be selected from the lower, middle and upper range of concentrations including samples near the action level, if applicable. A minimum of seven samples (including a duplicate) should be sent in order to show a good correlation.

3) MASS OF SAMPLE REQUIRED FOR LABORATORY ANALYSIS

The laboratory will need approximately 1 gram of sample to analyze the soil sample by EPA Method 7471A (Mercury by Cold Vapor). Consider sending at least 2 grams for a normal sample and 3 to 5 grams of sample for a matrix spike or matrix spike duplicate sample. The analyst and project manager should consider sending an additional jar with the sample for percent moisture analysis. Contact the laboratory that will be performing the analysis to ensure that enough mass is being delivered.

4) DATA REVIEW

Data must always be reviewed prior to field reporting. The analyst and the project manager prior to field reporting should review one hundred percent of the raw and summary data. Final reporting of field RP-91C data should commence only after review and evaluation of RP-91C field data versus laboratory data (i.e. typically using a least squares linear regression approach). This evaluation will establish correlation with definitive data and with other QA/QC samples, help to estimate bias. Data points should range from non detect to several times the action levels. A correlation coefficient (R^2) of 0.7 or greater should be considered acceptable for screening level data without additional qualifications. However, an R^2 of 0.9 or greater would indicate definitive level data.

Additional data review, independent of project management, should be performed on all RP-91C data used or to be used as crucial information for any decision making process. Additional data review should include evaluation of bias, sampling uncertainty, analytical accuracy and precision and completeness of documentation.