STATION PROCEDURE COVER SHEET

A. IDENTIFICATION

Number: CP 801/2801/3801AJ Rev. 0

Title: INDUCTIVELY COUPLED ARGON PLASMA ANALYSIS

Prepared By: G. D'AURIA/J. GLAUB

B. REVIEW

I have reviewed the above procedure and have found it to be satisfactory.

TITLE	SIGNATURE/DATE	TITLE	SIGNATURE/DATE
DEPARTMENT HEAD	2-10-92		
Sr. Engineer	2-10-92		

C. SPECIFIC UNREVIEWED SAFETY QUESTION EVALUATION REQUIRED:

Modifies intent of procedure and changes operation of systems as described in design documents. YES [ ] NO [X]

(If yes, perform written USQ determination and Safety Evaluation, and contact Manager, Safety Analysis Branch to determine need for Integrated Safety Evaluation.)

ENVIRONMENTAL REVIEW REQUIRED

(Adverse environmental impact) YES [ ] NO [X]

D. SPECIFIC SAFETY EVALUATION REQUIRED

Affects response of safety systems, performance of systems which may have been credited in the safety analysis or non-credited systems which may indirectly affect safety system response. YES [ ] NO [X]

(If yes, perform written Safety Evaluation and contact Manager, Safety Analysis Branch to determine need for Integrated Safety Evaluation.)

E. INTEGRATED SAFETY EVALUATION REQUIRED YES [ ] NO [X]

F. BIENNIAL REVIEW

This revision satisfies biennial review requirements. YES [X] NO [ ]

G. PROCEDURE REQUIRES ~~PORC~~/SORC REVIEW

(In addition to review, items with a YES response must be documented in the ~~PORC~~/SORC meeting minutes.) YES [X] NO [ ]

H. ~~PORC~~/SORC APPROVAL ~~PORC~~/SORC Meeting Number 92-9

I. APPROVAL AND IMPLEMENTATION

The attached procedure is hereby approved, and effective on the date below:

STATION SERVICES UNIT DIRECTOR

3/2/92
EFFECTIVE DATE

INDUCTIVELY COUPLED ARGON PLASMA ANALYSIS

Page No.	Eff. Rev.
1-22	0

1. OBJECTIVE

The objective of this procedure is to provide instruction for the operation of the Thermo Jarrell-Ash (TJA) Inductively Coupled Argon Plasma (ICAP) Enviro-36 simultaneous plasma emission spectrophotometer.

2. REFERENCES

2.1 Operator's Manual For ICAP Spectrophotometer, 1991, Thermo Jarrell-Ash Corporation, Franklin, MA

2.2 Ultrasonic Nebulizer Instruction Manual, Cetac Technologies, Inc., Omaha, NE

2.3 Jarrell-Ash 2500 R.F. Generator User's Manual, 1986, Thermo Jarrell--Ash Corporation, Franklin, MA

2.4 Gilson Minipulse 2 Peristaltic Pump User's Guide,1990, Gilson Medical Electronics, Inc., Middleton, WI

3. PREREQUISITES

N/A

4. PRECAUTIONS

4.1 Do not operate ICAP if laboratory environmental conditions exceed 86° F and/or exceed 70% relative humidity.

4.2 Ensure that ventilation system is functioning properly before operating system.

4.3 Never open or uncover the power unit to the RF Generator. It contains high voltages that can cause injury or death.

4.4 Ensure that there are no persons with pace-maker implants in the vicinity of ICAP as generated radio frequencies may interfere with pace-maker operation.

4.5 Never directly view the plasma without using U.V. protective eye wear.

4.6 If reflected power from R.F. Generator exceeds 100 watts for more than a few seconds during operation, turn off R.F. power.

4.7 Do not operate Ultrasonic Transducer beyond 6 amps of Output power.

5. PROCEDURE

NOTE

This procedure deals only with the operational aspects of the TJA ICAP. As such, no specific operational methods are defined. Instruction is provided, however, so that analytical methods can be developed and documented. This provides the user with sufficient flexibility to create and optimize a particular method to meet specific sample conditions.

5.1 Initial Start up (Peristaltic pump with pneumatic nebulizer)

5.1.1 Check instrument exhaust ventilation velocity and drain bottle receiving capacity; Ensure both are adequate for instrument operation.

5.1.2 Open laboratory argon supply line and Verify that pressure is between 40 - 50 psi.

5.1.3 Flip up "Line" and "Control" circuit breakers.

5.1.4 Plug in power cord to circulating water pump.

5.1.5 Flip up "Torch" and "Sample" toggle switches and Adjust argon flow control knobs until the following flow rates are obtained.

5.1.5.1 Torch: 15 - 20 L/min

5.1.5.2 Sample: 0.6 - 1.0 L/min

5.1.6 Turn on the peristaltic pump and begin to Aspirate D.I. water into mixing chamber. Check mixing chamber for the formation of mist indicating proper sample delivery.

5.1.7 Continue to aspirate sample for a minimum of three (3) mins in order to purge air from Torch.

5.1.8 Terminate "Sample" argon flow by shutting flow control knob.

5.1.9 Observe mixing chamber and wait for mist to clear.

NOTE

The blue "RF Off" light should be illuminated indicating that all instrument interlocks have been satisfied and it is now safe to provide power to the RF generator.

5.1.10 Check to ensure that black "power control" knob is turned fully counterclockwise and that "Automatic Power Control" switch is in the manual position.

5.1.11 Press the red "RF On" button.

CAUTION

When the plasma forms it should appea. yellow-white when viewed through the tinted glass. If, instead, it appears bright orange-red, Depress the "RF Off" button immediately.

5.1.12 Slowly Turn "power contro!" knob clockwise until power reaches approximately 0.5 KW.

5.1.13 Press and release "ignitor" button periodically while continuing to increase pow:r.

5.1.14 Upon formation of white streamers around torch, cease Pressing "ignitor" button .

5.1.15 Gradually increase power until plasma is formed or until 1.7 KW is reached on the power meter.

NOTE

If plasma fails to form, or if other difficulties are noted, notify Chemistry Supervision.

5.1.16 Slowly restore sample argon flow to approximately .6 L/min.

5.1.17 Adjust the "power control" knob until a reading of 1.1 KW is obtained on the power meter.

5.1.18 Flip up the "automatic power control" switch to the automatic power position.

5.1.19 Turn the "power control" knob slightly clockwise to provide headroom for automatic power adjustment.

5.1.20 Allow 15 - 20 minutes for the instrument to warm up

5.1.21 Begin aspirating a 1000ppm solution of yttrium into the plasma. The point of interest will be a red "tongue" located within the plasma.

5.1.22 Adjust the sample gas flow until the tip of the red tongue is a couple of millimeters above the top of the torch. This indicates that the temperature within this region of the plasma is now set to provide optimum performance.

5.1.23 Open the access panel to the Optical Entrance Slit Assembly. The red illuminated area should fall squarely on the slit with equal portions of illumination on each side of the slit.

CAUTION

The following instruction can be followed to correct for optical system misalignment. However, it must be performed cautiously as there is danger of U.V. light exposure and potential for degradation of instrument performance.

5.1.24 If the illuminated area from the yttrium sample is too far to one side of the entrance slit, it can be adjusted by the following instruction.

5.1.24.1 Open the torch compartment access door located on the top right-hand side of the instrument. Do not look inside of compartment.

5.1.24.2 Reach inside compartment and turn lower adjustment knob while observing illuminated area on entrance slit.

5.1.24.3 Center illuminated area on entrance slit. Remove hand from compartment and Close access door.

5.2 Sample Analysis (Software Operation)

NOTE

When the computer is turned on the main menu will be displayed. Five pull-down menus will be available for selection. The left and right cursor keys are used to select the desired menu. If you are performing an analysis for which a method already exists, proceed to section 5.3, "Operation menu." If you need to develop a method, proceed to section 5.4., "Development menu."

5.3 Operation Menu (Sample Analysis)

5.3.1 Select "Analysis" from "Operation" pull-down menu and press <Enter>.

5.3.2 Three options for the selection of a method are now presented.

5.3.2.1 Default method name may be entered.

5.3.2.2 A new method name (existing) can be typed in, then entered.

5.3.2.3 A list of existing methods can be reviewed by Pressing <F6, List>.

5.3.3 Select desired method, then Press <Enter>.

5.3.4 If this is the first run of the day, or if the machine has not been profiled in the last 4 hours of operation, then Press <F5, Profile>.

NOTE

The RF generator must be warm to successfully profile the instrument. Therefore, allow at least 30 minutes to pass after instrument Start-up before beginning Profile operation.

5.3.5 Begin aspirating a sample containing between 1 to 10ppm copper.

5.3.6 Press <F3, Automatic>.

5.3.7 If the default values displayed in profile table are acceptable, Press <F1, Run>. If default values are not acceptable, type in desired values; Press <Enter>; then Press <F1, Run>.

5.3.8 Review profile scan. The center of copper peak should not have shifted more than 1 distance unit from central position designated as 0.

5.3.8.1 If center of copper peak is located more than 1 distance unit from central position (+/-), Notify Chemistry Supervision.

5.3.8.2 If center of copper peak is located within 1 distance unit from central position, Press <F9, Done/Keep>.

5.3.9 Press <Escape> to exit profile program.

NOTE

The instrument is now ready to be standardized. The standards may be run in any order, and not all standards have to be analyzed.

5.3.10 Press <F3, Stndrdize>. A table showing the standards to be run, as identified in the method, will be displayed on the screen.

5.3.11 Begin aspirating highlighted standard.

5.3.12 Press <F1, Run>.

5.3.13 Review results of standard run. Note relative standard deviation (RSD). It should not exceed 10%.

5.3.14 If standard results are acceptable, Press <F9, Done/Keep>.

5.3.15 Continue to run additional standards as required by method.

5.3.16 After <F9, Done/Keep> has been pressed for the last standard to be analyzed, Press <F9, Done/Keep> one additional time. A standardization report will be displayed.

5.3.17 Press <F6, Plot>. The standard curve for the first element will be shown. Review standard curve for expected slope, y axis intercept, and linearity.

5.3.18 If curve is acceptable, Press <Page Down> key to view curves for additional elements listed in method, if applicable.

5.3.19 Press <Esc> to exit Plotting program.

5.3.20 Press <F9, Done/Keep> to exit standardizing program.

5.3.21 If the method calls for blank subtraction, Press <F4, Blank>.

5.3.21.1 Enter appropriate information.

5.3.21.2 Begin aspirating Blank.

5.3.21.3 Press <F1, Run>.

NOTE

The instrument is now ready to analyze unknown samples

5.3.22 Press <F1, Analyze>

5.3.23 Begin aspirating sample.

5.3.24 Enter the following information.

5.3.24.1 Sample name

5.3.24.2 Comment (Optional)

5.3.24.3 Operator name.

5.3.25 Press <F1, Run>. Analysis begins.

5.4 Development Menu (Method Development)

5.4.1 Select "Methods" from the "Development" pull down menu and press <enter>.

5.4.2 Three options for Method selection are now available:

5.4.2.1 Press <enter> for default method name

5.4.2.2 Press <F6, List> to get a list of existing methods from which a method can be selected.

5.4.2.3 Type in a new name. The computer software will assume that a method is to be created and will display the method development screen.

NOTE

The following instruction is for creating a new method. Portions of the instruction may, however, be used to modify an existing method.

5.4.3 Press <F1, Elements> to bring up Periodic Table from which elements can be selected. The following color code defines availability.

Yellow — Elements that have lines installed in the instrument and can be selected for analysis.

White — Elements which have already been selected for analysis.

Black — Elements that do not have lines installed and are, therefore, cannot be selected.

5.4.4 To select an element to be analyzed, use the cursor keys to position the cursor on the yellow element to be analyzed and Press <F1, Select>.

NOTE

If there are two (or more) emission lines installed in polychromator for a given element, they are both included in the method when you select that element. The <F5, Lines> key may be used to deselect an individual line.

5.4.5 After all the desired elements have been selected, Press <F9, Done>. This returns you to the method development screen.

5.4.6 Press <F3, Method Info>.

NOTE

This section contains much default information. Use arrow keys to move cursor over desired table position and then make entry.

5.4.7 Enter information for the following method parameters. It is acceptable to use default settings that are appropriate for method being developed.

5.4.7.1 Sample Introduction device to be used. Normal indicates nebulizer.

5.4.7.2 Calibration mode. The two choices are "concentration," and "concentration ratio." The default mode of concentration is the normal choice.

5.4.7.3 Number of repeat integrations. The default value of 4 is usually sufficient.

5.4.7.4 Flush time. The default value of 30 seconds is fine for the pneumatic (normal) nebulizer, but a time of 1 minute should be used for the ultrasonic nebulizer.

5.4.7.5 Auto-Increment Sample names. Use default value.

5.4.7.6 Names of the analysis and calibration data files to be automatically addressed by the method. If the information is to be stored, it is advisable to create files specific to the method.

5.4.7.7 Names of tables to be automatically addressed by method. Again, it is advisable to have tables specific to the method.

5.4.7.8 Whether or not to automatically store analysis data to the database. For routine laboratory work, the default choice of"no" is preferred.

5.4.7.9 Whether or not to automatically store standardization data to the database. The default value of "no" is fine for routine laboratory work.

5.4.7.10 Whether or not to automatically store individual repeats to the database. Use default choice of "no."

5.4.7.11 Whether or not to automatically print the analysis data.

5.4.7.12 Autoprint standardization report. This function has four options:

a. None

b. Slopes

c. Slopes and readback

d. Slopes, readback and plots.

5.4.7.13 Condensed print format. This function determines whether or not a form feed should be issued at the beginning of each print, or if a dashed line should be printed instead.

5.4.8 After all the method information has been reviewed and entered, Press <F9. Done/Keep>. This returns you to the method development screen.

5.4.9 Press <F4. Output>.

NOTE

Almost all of the information in this section will be the default choice with the possible exception of blank subtraction.

5.4.10 Review the following parameters under the output section and make any changes as necessary.

5.4.10.1 Output mode, where concentration is the default mode and the alternatives are:

a. Intensity

b. Intensity ratio

c. Standardized intensity ratio.

5.4.10.2 Whether or not to override print limits. The default lower print limit is 0; therefore, if you wish to see concentration values less than 0, you must override the print limits.

5.4.10.3 Whether or not to override the number of significant figures defined in the method.

5.4.10.4 Whether or not to apply background correction.

5.4.10.5 Whether or not to apply blank subtraction. The default choice is "no," however, you may wish to use it if your method is approaching the detection limits of the instrument and your lowest standard is not a blank.

5.4.10.6 Whether or not to apply limit checking, and the default limit check table to use during analysis.

5.4.10.7 A default correction factor to be applied to the data. Check with Chemistry Supervision before selecting this option.

5.4.10.8 The choice of which information is to be provided to video screen, printer, or any other active remotes.

5.4.11 After all the Output information has been reviewed and accepted. Press <F9, Done/Keep>. This returns you to the method development screen.

5.4.12 Press <F5, Element Info>. This brings up the element information table, which consist of three parts:

5.4.12.1 Elements

5.4.12.2 Wavelengths

5.4.12.3 General Information

NOTE

Information that pertains to a specific element/wavelength combination is set up here. Use the arrow keys to move within a particular section, use Page up and Page down keys to move from section to section.

5.4.13 Review the element(s) listed at the top of the element information table for correctness (i.e., are they the elements that were selected for this method?)

5.4.14 Press the <Pgdn> key to move cursor to wavelength identified for element being reviewed.

5.4.15 Press the <Pgdn> key again to access the general information table.

5.4.16 The parameters to be entered in this table include:

5.4.16.1 The element name as it is to appear on the data printout. The default name should be acceptable for routine laboratory work.

5.4.16.2 The timing group number; This refers to number of desired internal standard entered in Internal Standards table.

5.4.16.3 Concentration at which line switching is applied if there are two emission lines installed for the same element.

5.4.16.4 Background correction method to be applied. The spectrum shifter is the only choice for our instrument. You may choose to perform background correction by subtracting the background intensity from the peak intensity (normal), or by dividing the peak intensity by the background intensity, by pressing the <BKG-OR> key. Most of our analytical methods should not require background corrections.

5.4.16.5 High and/or low spectrum offset positions at which to apply background correction. The default values of 0 indicate no background correction. You may either type in the values here (if known) or graphically determine the positions using the Method Development Scans.

5.4.16.6 Standardization method to be applied. The choices are:

a. Two point uncalibrated, (default value). This version simply draws a straight line between the high and low standards.

b. Two point calibrated. This version uses two standards to re-slope the calibration curve generated during the calibration and curvefit routines (See Section 5.4.27 for more information on calibration).

c. Multiple Standards. This version will use a least squares function to fit the best straight line to standardization data containing 2 to 5 standards.

5.4.16.7 The names and concentration of the standards to be used during the standardization.. The names of the two standards selected for the "two point calibrated" method should match the names of the standards identified in the curve fitting routines.

5.4.16.8 The high and low values that will be printed in the report. Generally, the defaults settings are satisfactory.

5.4.16.9 The number of significant figures to be reported. The default setting of 4 should be sufficient.

5.4.16.10 The units in which the results are to be reported. The default value is ppm and is appropriate for most laboratory work.

5.4.16.11 The spectrum shifter offset position at which the analyte is to be measured. A value of 0 indicates that the channel is being used for the element for which it was installed. If, however, you wish to analyze an element that falls within the channel, you do so here by entering the shift position where the center of the peak falls.

5.4.17 After all the parameters for the general information section have been reviewed and entered. Press <F9, Done/Keep>. This moves the cursor to the element section of the table.

5.4.18 Press <F9, Done/Keep> one more time to save Element Information Menu. This will return you to the Method Development screen.

5.4.19 Press <F7, Scans>. The Scans screen will be displayed.

NOTE

The <F7, Scans> key provides access to instrument functions which are important to method development. Scans can identify interfering elements, and can be used to set background correction points. Additionally, when method parameters are adjusted the Scans overlay feature can be utilized to evaluate the effectiveness of the change.

5.4.20 Press <F5, WL-Sim> to select simultaneous scans.

5.4.21 Press <F1, Instr> to acquire scans directly from the instrument.

NOTE

After selecting <F1, Instr> a table will be displayed. It is not necessary to fill out all requested information, however, such information may be useful for future reference. Additionally, the integration may be shorten for convenience but the resolution of the scan will suffer somewhat.

5.4.22 If satisfied with the information displayed on the data table, begin to Aspirate sample.

5.4.23 Press <F1, Run> to commence scan.

5.4.24 After the first scan is displayed, overlays can be done by aspirating different samples and then pressing the following keys:

5.4.24.1 <F5, Overlays>

5.4.24.2 <F5, Overlay>

5.4.24.3 <F1, Instr> (Alternatively, <File/V> can be selected)

5.4.24.4 <Enter> Retains current method.

5.4.24.5 <F1, Run>

5.4.25 Once the scans for standards, a sample and a blank are displayed. Press <F1, Expand> to display a more detailed view of the scan.

NOTE

There are two ways to calculate background correction factors. One is to identify a background range to the computer and then have the computer calculate the background. The other is to manually pick a point in the scan to be used as background value. The following instruction deals with the manual setting of background correction factor.

5.4.26 Figure 6.1 is an example of an expanded overlayed scan. Notice how there is a baseline shift in the background. Using this figure as an example, the background correction procedure proceeds as follows.

5.4.26.1 Using the arrow keys the cursor would be moved to a stable spot in the background. For this example the -12 point is a suitable position. It is not recommended to pick any points closer than ±12 units from analyte peak.

5.4.26.2 After the cursor is placed on the -12 point, <F1, SetLoBkg> is pressed.

5.4.26.3 Next, the <F9, Done> key is pressed. A white "L" appears on the screen indicating the position of the background setting.

5.4.26.4 Any subsequent elements requiring background correction can be done by pressing <F7, Next> and repeating the preceding three steps.

5.4.26.5 The expanded scan is exited by pressing the ESC key. This brings up the condensed scan menu.

5.4.26.6 <F6, Calculate BKG> is pressed even though a single point has been selected.

5.4.26.7 <F8, Save BKG> is pressed to save background.

5.4.26.8 The method is exited by pressing the following keys.

a. <ESC>

b. <ENTER>

c. <ESC>

d. <ESC>

e. <F9, Done/Keep>

NOTE

The following section deals with "calibrating" the TJA ICAP instrument. If it has been decided to "standardize" the instrument, this section does not have to be reviewed. The difference between "calibrate" and "standardize," and the advantages and disadvantages associated with calibrating the instrument, are described in the following.

One major difference between standardize and calibrate is that a standardization curve can only be linear fitted (i.e., only a straight line can be drawn through the data points). A calibration curve, comparatively, has various degrees of curve fitting routines available. Additionally, a maximum of 5 standards can be used to standardize the instrument, whereas there is no upper limit of standards that can be used to calibrate the instrument.

Most of our laboratory analyses should fall within a linear range; therefore, a standardization curve will be adequate to obtain the necessary accuracy. Confidence in the standardization curve can be strengthen by increasing the number of standards run - up to the maximum of 5.

For circumstances in which the desired analytical range does not have a linear relationship, it will be necessary to calibrate the instrument. Additionally, it may be necessary to experimentally determine the number of standards required to achieve the desired results.

Although the calibration process is analytically superior to the standardization process, it is much more time consuming and should only be used when necessary.

5.4.27 Using the arrow keys, Select "Calibration -Standards" under the "Development" pull-down menu and Press <Enter>. The computer will ask for the name of the standard table.

NOTE

Creation of a calibration curve is a three (3) step process:

1. First, a calibration table must be created. The name of which must exactly match the table name identified in the method for which the curve is being generated (Rf: 5.4.7., #7). This table contains information concerning: Standard Name(s), Element (analyte) identification, and standard concentration.

2. The Standards identified in the aforementioned table are then analyzed by the method for which the curve is being generated. Both the table name and data file name requested must match those identified in the method.

3. The data generated by the analysis of calibration standards is then curvefitted. Again, the names of the table and calibration file must match the names in the method. The type of curve fit will depend on the number of standards run (i.e., 2 standards linear fit; 3 standards - curvilinear fit; 4 or more standards - full curve fit).

5.4.28 Type in name of table to be created, then Press <enter>. The screen will display "The Standard File is Empty."

5.4.29 Press <F3, Add Std>. The screen displays the message "Standard 1" and asks for a name.

5.4.30 Enter name of standard and Press <F9, Done/Keep>. The computer will note that no elements have been selected.

5.4.31 Press <F1, Elements>. This will bring up the Periodic Table from which elements can be selected.

5.4.32 Use the arrow keys to move the cursor to the desired element and Press <F1, Select>. Repeat this process until all of the desired elements have been selected.

5.4.33 When all of the desired elements have been selected, Press <F9, Done>. The computer will ask for the concentration of the elements.

5.4.34 Enter concentration for each element then Press <F9, Done/keep>.

5.4.35 To add additional standards, Repeat steps 5.4.29 through 5.4.34.

5.4.36 When you have all of the standards entered, Press <F9, Done/Keep>. This will bring up the "Development" pull-down menu.

5.4.37 Move cursor to "Calibration-Analysis," Press <Enter>. The computer will ask for a method name.

5.4.38 Enter method name. The computer will display the names of the calibration table and data file

5.4.39 If the names of the standard table and data file are acceptable, Press <F9, Done>.

5.4.40 Aspirate standard, Press <F1, Run Standard>. The computer will ask for comments, and operator name.

5.4.41 Enter any desired comments, operator identification, the Press <F1, Run>. The instrument begins analysis.

5.4.42 After analysis of standard, review displayed data. If acceptable, Press <F9, Done/Keep>. The cursor moves down to the next standard to be analyzed and the word "done" appears next to standard that has just been run.

5.4.43 Repeat steps 5.4.40 through 5.4.42 for any additional standards that have to be analyzed.

5.4.44 After the last standard is run, Press <F9, Done>. This will bring up the "Development" pull-down menu.

5.4.45 Move the cursor to "Calibration Curvefit" then Press <Enter>. The computer will ask for a method.

5.4.46 Type in name of desired method and Press <Enter>. The computer will display the names of the calibration table and data file.

5.4.47 If the names of the calibration table and data file are acceptable, Press <F9, Done>. The computer will state that there is no fit for curve.

5.4.48 Press <F1, Fit Elem>, then Press <F1, Calculate>. A table will be displayed telling the type of fit plus additional data.

5.4.49 Press <F9, Done/Calc>.

5.4.50 Press either <F4, Lin Plot> or <F6, Log Plot> depending on the type of desired graph

5.4.51 Review curve against expected results.

5.4.52 Press <Escape> to exit curve plotting program.

5.4.53 Press <F9, Done/Keep>. The computer will provide a summary line for each element curvefit.

5.4.54 Press <F9, Done Fit>

NOTE

When you create a Calibration table for a method, the standardization section under Element Information will automatically default to "2 point calibrated."

5.5 Use of the CETAC U-5000 ULTRASONIC NEBULIZER.

NOTE

The Ultrasonic nebulizer is used to increase the sensitivity of the ICAP instrument. Figure 7.2 is an overlayed scan of a 50ppb chromium standard. The solid line is the scan of the standard using the ultrasonic nebulizer; the dotted line is the scan of the same standard using the pneumatic nebulizer. As shown by this figure, the ultrasonic nebulizer can greatly increase the signal to noise ratio.

5.5.1 Turn on the cooling unit to the ultrasonic nebulizer and check that temperature dial is set at 5° C. It will take approximately 90 minutes for the cooling system to reach this temperature and stabilize.

5.5.2 Press the red power button to the ultrasonic nebulizer; the red light will come on. The operating temperature is preset at the factor and should stabilize at 140° C in about 5 minutes.

5.5.3 Disconnect the sample tubing from the peristaltic pump and remove ICAP access panel to the spray chamber.

CAUTION

When removing pneumatic nebulizer from end of spray chamber do not use excessive force. The nebulizer sits firmly on the end of the spray chamber and can best be removed by gently twisting nebulizer while pulling straight backward.

5.5.4 Remove the pneumatic nebulizer as follows:

5.5.4.1 Place one hand on spray chamber to secure spray chamber position when removing nebulizer.

5.5.4.2 Grasp the nebulizer with free hand and Twist nebulizer from side to side while Pulling straight backwards.

5.5.4.3 After nebulizer slides off end of spray chamber. Detach argon sample tubing from instrument anchor nipple by Pulling tubing straight backwards from nipple.

5.5.4.4 Place nebulizer and attached tubing inside a clean plastic bag and Place on sample cart.

5.5.5 Using a bent piece of wire, Remove baffle device from spray chamber and Place in plastic bag.

5.5.6 Place peristaltic pump next to ultrasonic nebulizer and Attach drain line and sample lines. The drain line goes on the bottom.

5.5.6.1 Adjust setting on peristaltic pump to 550 to provide a flow of 2.5 mLs/min to ultrasonic nebulizer.

5.5.7 Attach argon sample line from ultrasonic nebulizer to instrument anchor nipple.

5.5.8 Attach the ultrasonic nebulizer spray chamber adapter as follows:

5.5.8.1 Loosen allen screws on the base of the adapter.

5.5.8.2 While holding the spray chamber with one hand, gently Push the adaptor into the spray chamber (notch side up) until it reaches the end.

5.5.8.3 Tighten, slowly and evenly, the two allen screws on the base of the adapter. As the screws tighten, periodically attempt to slightly rotate the adapter within the spray chamber, noting to always return the adapter to its upright position before resuming tightening.

5.5.8.4 Cease tightening allen screws when adapter fits snugly within spray chamber and will not move when moderate force is applied to rotate it.

5.5.9 Turn on nebulizer (sample) argon flow and Adjust flow rate to 0.7 L/min.

5.5.10 Press yellow operation button to ultrasonic nebulizer and let transducer run dry for approximately 1 minute (power meter must not exceed 6 amps).

5.5.11 Turn on peristaltic pump to deliver deionized water at a rate of 2.5 ml/min.

5.5.12 Note the density of the mist being formed in the aerosol chamber. The glass sample tube inside the aerosol chamber should be visible when viewed from the front, and should be slightly visible when viewed from the top.

NOTE

No mist will be visible in the spray chamber when using the ultrasonic nebulizer.

5.5.13 Initiate plasma as per section 5.1 instruction.

5.5.14 Perform analysis as per section 5.3 instruction or create/modify method as per section 5.4 instruction.

5.5.15 After performing desired analysis, Aspirate deionized water or dilute acid solution for at least 3 minutes before shutting down nebulizer.

5.5.16 Ultrsonic shut-down and System Restoration:

5.5.16.1 Turn off peristaltic pump and let nebulizer run dry for about a minute.

5.5.16.2 Turn off yellow operate and red power switches.

5.5.16.3 If the constant temperature bath is going to be used in the near future, leave power on - otherwise, turn power off.

5.5.16.4 Shutdown ICAP as per section 5.6 instruction.

5.5.16.5 Detach ultrasonic nebulizer argon sample line from instrument.

5.5.16.6 Loosen allen screws on base of spray chamber adapter and remove from spray chamber and place in plastic bag.

5.5.16.7 Insert baffle device into spray chamber.

5.5.16.8 Re-install pneumatic nebulizer and attach argon gas line.

5.5.16.9 Replace spray chamber access panel door.

5.5.16.10 Disconnect ultrasonic drain and sample lines from peristaltic pump and Place pump back on pneumatic

5.5.16.11 nebulizer sample cart

5.6 ICAP Shutdown

5.6.1 Aspirate deionized water for at least one minute before instrument shutdown.

5.6.2 If operating with pneumatic nebulizer, Shut-down peristaltic pump and Release tubing from rotor assembly.

5.6.3 Press blue "RF Off" button. The Plasma will extinguish.

5.6.4 Flip down black toggles switches for "Torch" and "Sample" argon gas flow.

5.6.5 Flip "Automatic Power Control" Switch to "Manual" position.

5.6.6 Rotate "Power Control Knob" fully counterclockwise until it stops moving.

5.6.7 Unplug power cord to circulating water pump.

5.6.8 Flip down "Line" and "Control" circuit breakers.

5.6.9 Switch off computer.

5.6.10 Shut off laboratory argon supply line.

6. CHEMISTRY FORMS

6.1 Chem. Form 801/2801/3801AJ-1, Millstone Chemistry Laboratory, Method Development For TJA ICAP.

7. FIGURES

7.1 Background Scan

7.2 Ultrasonic Nebulizer Scan Overlay

8. DISCUSSION

8.1 None

GD:tjp

BACKGROUND SCAN

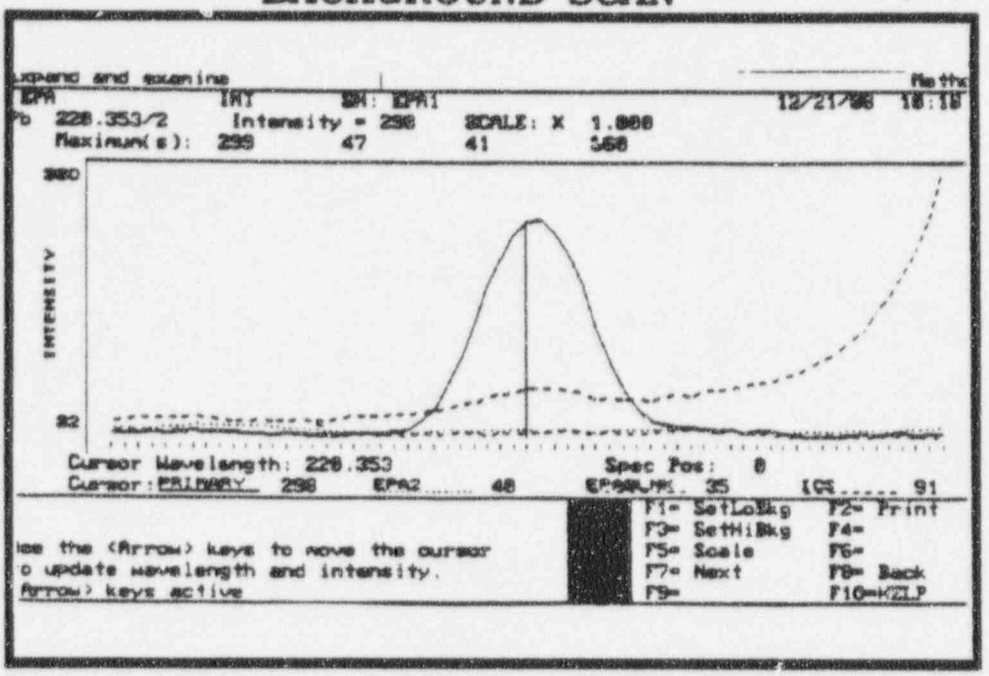

FIGURE 7.1

ULTRASONIC NEBULIZER SCAN OVERLAY

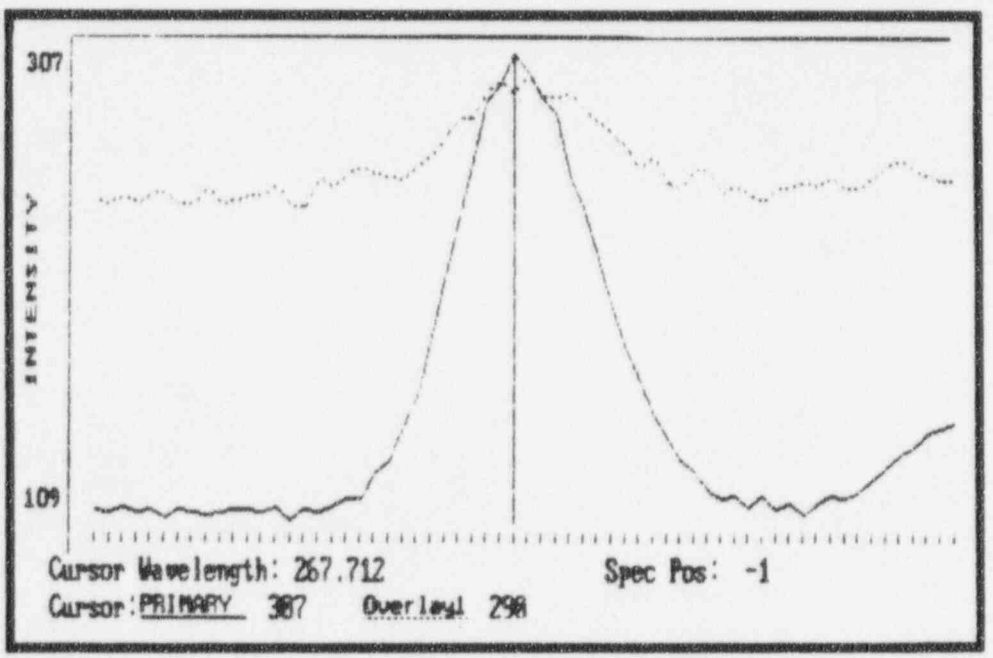

FIGURE 7.2