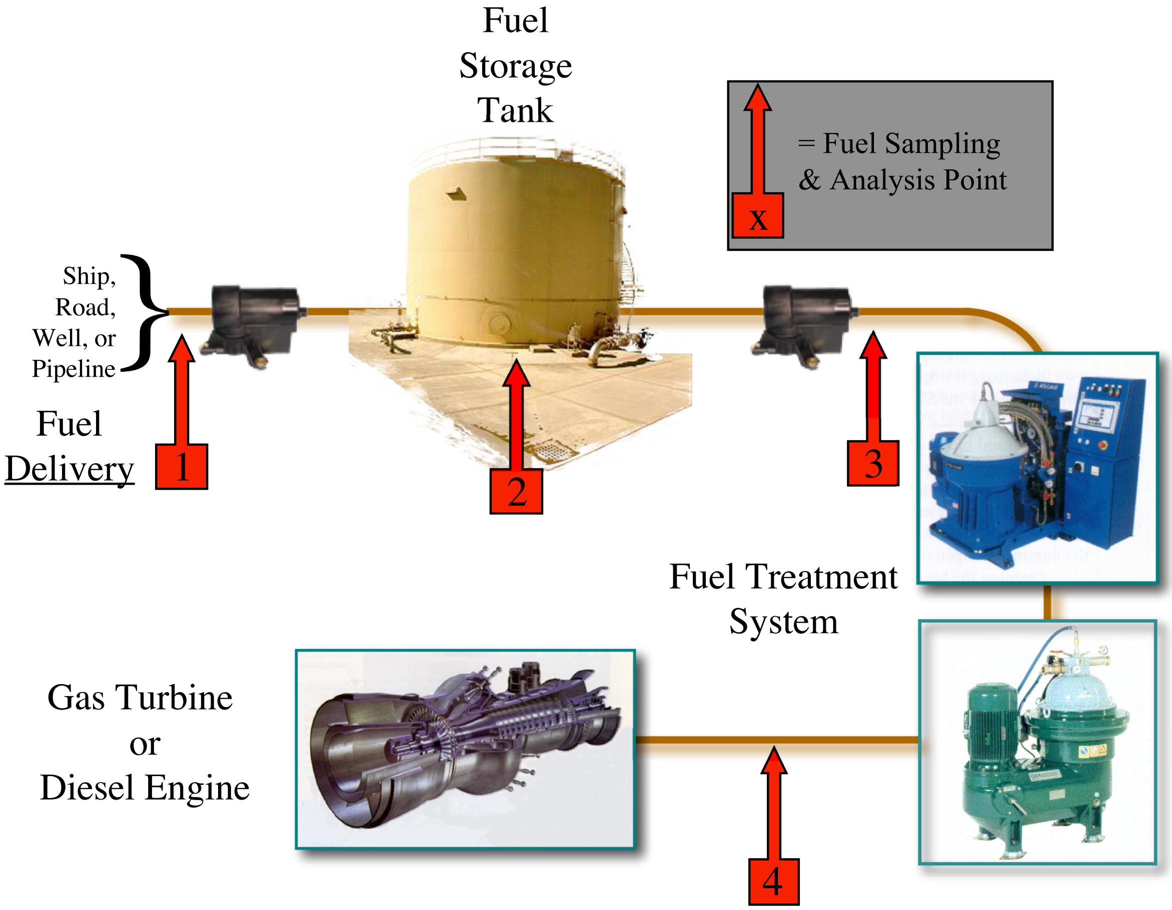
Operator's Manual

Spectroil M/F-LD

Fuel Analysis Spectrometers



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List of Effective Pages

Summary of Spectroil Modifications

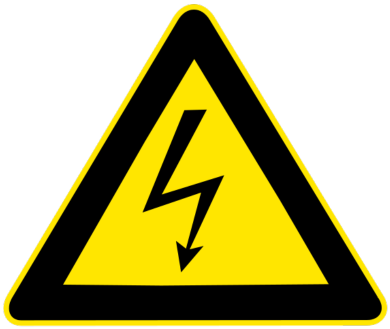
Modification	Description
Mod 0	Original CID version of Spectroil M
Mod 1	Upgrade with SFTM port and frequency adjustement potentiometer
Mod 2	Addition of solid state excitation ignition module and SFTM port
Mod 3	Upgrade to combined solid state source
OilMWindows	Upgrade to Windows hardware and software
Mod 4	Upgrade to panel PC hardware and Windows XP
Mod 5	CE version of the Spectroil M
Mod 6	CCD Optic and New Software v. 5, starting w/serial number 6001
Mod 7	C.E. version of Mod. 6

Summary of OilMWindows Modification 6 Hardware and Software Manual Versions

Change	Version	Date	Description
First Issue	3.0	7/1/07	Complete update of Mod. 5 version 2.5 manual to include CCD optic & updated software.
Change 1	3.1	10/24/07	Update for CE version, Mod. 7, changes. Addition of optic removal procedure. Grammatical corrections
Change 2	3.2	10/31/07	More updates to Chapter 2 for CE certification.
Change 3	3.3	2/18/08	Created separate M/F-LD Operator's Manual, updated accuracy table, included sample ID, operator's maintenance and sulfur analysis.
Change 4	3.4	4/15/08	Added NOx water analysis description
Change 5	3.4.1	9/23/09	Corrected figure 2-1 and table 2-1.

Total Number of pages in this manual is 46 consisting of the following:

Section	Page Numbers
Cover	2
Table of Contents	i - ii
List of Effective Pages	iii - vi
Chapters 1 - 5	1 - 38



WARNING!!!

High Voltages are Present During the Operation of the Spectroil M!

Observe all Safety Precautions!

Turn OFF the Main Power Switch and unplug the SPECTROIL M before any work is performed.

Definitions

The following definitions apply to specific instructions throughout this manual.

WARNING!!!
An operating procedure or practice that may cause injury if not carefully observed or followed.

CAUTION!!!
An operating procedure or practice that may cause damage to the LNF if not carefully observed or followed

NOTE!!!
An operating procedure or practice that is essential to emphasize

Software CAUTION!!!

The Spectroil M computer is capable of running multiple software applications and/or operating systems. However, as designed, the computer processor is dedicated to the operation and control functions of the Spectroil M. Do not attempt to add any software or alter the original factory installed software without checking first with the Spectro Inc. Service Department.

Note on Oil Standards

The Spectroil M series of spectrometers can be calibrated for military or commercial applications. As a rule, the Spectroil M/N-W is calibrated and standardized with D-19, D-12 and D-3 series of standards, and the Spectroil M/C-W with V-21 or S-21 series of calibration standards.

Although this manual frequently refers to the military “D” series of standards, the operator procedures are identical for all types of Spectroil M spectrometers. Commercial customers should substitute their equivalent “V” or “S” series of standards throughout this manual.

WARRANTY

The warranty period of the Spectroil M family of spectrometers is twelve (12) months from date of installation or fifteen (15) months from date of shipment, whichever occurs first. Spectro warrants the Spectroil under conditions of operation against defects of materials and workmanship. All defective material will be replaced providing damage was not caused by improper use. Warranty applies to parts and labor only.

Spectroil M/F-LD
Fuel Analysis Spectrometer

Operator's Manual

1.0 GENERAL OPERATING REQUIREMENTS

This manual provides the Spectroil M/F-LD operator with routine instructions on how to set-up the spectrometer, analyze fuel samples, and perform routine maintenance. The instructions apply to the Spectroil M/F-LD Fuel Analysis Spectrometer with Mod. 6 (CCD optic) and Mod. 7 (C.E. hardware).

1.1 Power Application and Systematic Power Removal

The Spectroil M/F-LD consists of the excitation, optics and readout subassemblies. Each subassembly requires specific voltages to perform as a system. These voltages are generated within each subassembly and originate from the main power distribution assembly. Main power for these subassemblies is fused with a 10 ampere circuit breaker CB1 which is mounted on the Power Connection Plate located on the right side near the back of the instrument. Power should not be applied to the instrument unless all specifications for input power requirements have been met.

To apply power to the instrument, place circuit breaker CB1 in the upright ON position, Figure 1-1.

Once power is applied to the instrument, two events occur. First, an internal controller will

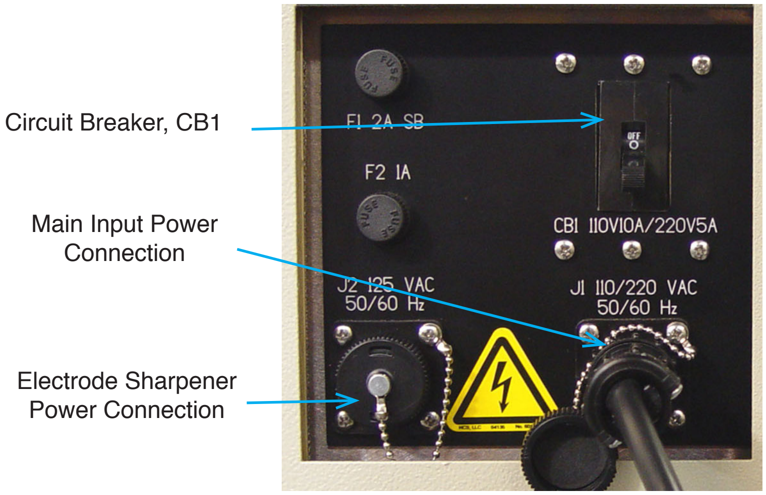


Figure 1-1, Right Side View Showing Circuit Breaker CB1 initialize and automatically load the application. Simultaneously, the readout system will load Windows XP® and start up the instrument's application program called OILMWindows®. The instrument will boot directly to the Analysis Screen, Figure 1-2.

If the system fails to establish communication, a screen similar to Figure 1-3 appears to select the Correct Configuration File Path. This will happen if the system cannot find a file in case it has been moved, updated or is corrupted. Refer to the Maintenance Manual Section 2.4.4.6 for assistance to diagnose and correct this condition.

Next, move the MODE switch to the OPERATE position and power will be applied to the excitation source and electrode sharpener. A noticeable increase in fan activity will be observed; this is normal.

At this point, the instrument is ready to begin operation. Some time will be needed before the instrument stabilizes after power is applied. It is recommended that the main power remain on when the instrument is not in use to maintain maximum instrument stability. When not in use, the MODE switch should be placed in the STANDBY position.

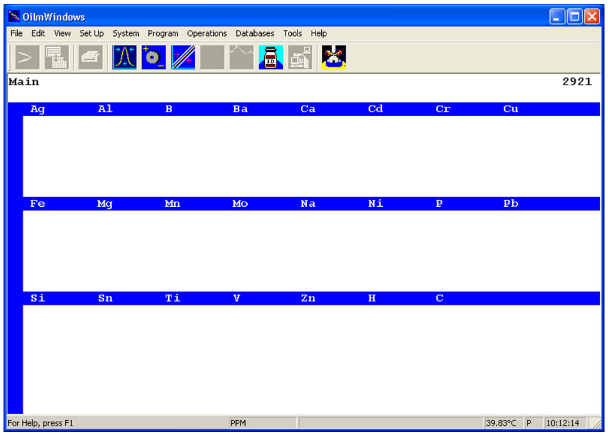
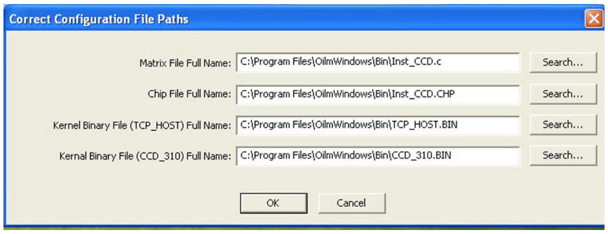


Figure 1-2, Analysis Screen



Matrix File Full Name: C:\Program Files\OilmWindows\Bin\Inst_CCD.c
Chip File Full Name: C:\Program Files\OilmWindows\Bin\Inst_CCD.CHP
Kernel Binary File (TCP_HOST) Full Name: C:\Program Files\OilmWindows\Bin\TCP_HOST.BIN
Kernal Binary File (CCD_310) Full Name: C:\Program Files\OilmWindows\Bin\CCD_310.BIN

Figure 1-3, Error Message if Communications Cannot be Established

To completely turn off the Spectroil M/F-LD, first shut down the OILMWindows software and then remove power from the instrument.

CAUTION: Utilizing the Windows XP® operating system dictates a specific series of steps to be performed in the process of shutting down the Spectroil M/F-LD. If power is accidentally removed from the Spectroil M/F-LD spectrometer or the circuit breaker CB1 is shut off while the readout system is running Windows XP® or the OILMWindows® application, an orderly shutdown would not be performed and as a result, the Windows XP® operating system must perform hardware and software diagnostics when power is reapplied.

To prevent this from happening, always follow the next steps to shut down the Spectroil M/F-LD.

1. Shut down the OILMWindows® application by left clicking the close box (box with an "X")in the upper right corner of the OILMWindows® header, or choose File/Exit from the pull down menu options. This will return the software to the Windows XP® desktop.

2. Left click the START menu and select Shut Down. A dialog will appear asking what do you what the computer to do. The options are, Standby, Restart, or Shutdown.

3. Highlight the Shutdown radio button and select OK.

4. The Windows XP® logo will appear with the instruction that it is shutting down.

5. After the logo disappears a message "It's now safe to turn off your computer " will appear.

6. It is now safe to place the main circuit break-

er CB1 in the down position to remove all power from the instrument.

If, by accident, power was removed from the instrument, a series of diagnostics will automatically be performed when power is reapplied. It is extremely important to allow these diagnostics to complete in their entirety before loading any application software. If problems are experienced during the process of running these diagnostics, contact the technical service department of Spectro Incorporated for instructions on how to recover and proceed with normal operation.

To restore power to the instrument, follow the instructions given above.

1.2 Rod Electrode Sharpening

The rod electrode, along with the disc electrode, form the analytical gap through which the oil or fuel sample are passed for analysis. An alternating current discharge will occur between the disc and rod electrode and vaporize the sample and the metallic components in it. This is the basis of the arc emission technique.

The preparation of the tip of the rod electrode plays a significant role in obtaining repeatable analytical data. The rod electrode must be cleaned prior to inserting it into the electrode sharpener. This is accomplished by taking a clean paper towel and removing the components of the burn residue from the previous analysis. Remove all residue from the tip and sides of the electrode by rotating the rod in the paper towel while applying pressure with the fingers of the opposite hand.

NOTE: The paper towel should be laboratory grade and free of silicon. Do not use household grade paper towels.

With the spectrometer on, turn the MODE switch to OPERATE. This applies power to J2, the electrode sharpener power connector. Momentarily press the power switch located on the base of the electrode sharpener. The sound of the electric motor should be heard and a slight vibration should be felt through the motor. The motor will continue to run on a self-timed cycle for approximately 3 to 5 minutes. Insert the rod into the rotating electrode guide hole until it comes in contact with the cutter blade. Apply inward pressure until approximately 1/8 to 3/16 inch (3 to 5 mm) is cut from the end of the rod. Slightly decrease the inward pressure on the rod electrode, but still maintain its contact with the cutter blade. This will polish the rod electrode tip.

Remove the rod electrode and visually inspect the tip. It should have a clean cut with no apparent chipping around the circumference of the rod. The surface should be very smooth and have a polished mirrored looking surface. If the quality of the surface is not as described, insert the rod into the sharpener and repeat the cutting and polishing procedure. Remove the rod, inspect the surface quality and if acceptable, place the rod electrode into the original box for storage until ready for use. To prevent contamination of a sharpened rod electrode, do not touch the tip or edge of the tip of the sharpened electrode with the fingers or metallic surfaces or anything but a fresh, clean laboratory grade paper towel. Do not use a rod electrode for analysis if the surface appears to have been damaged. Refer to Section 7.6.5.1 for the procedure to change/rotate the cutter blade.

NOTE: The electrode sharpener can be a potential source of contamination that can result in degraded accuracy. The electrode sharpener must be emptied and cleaned frequently.

The rod electrode sharpener has a graphite collector barrel assembly which is secured to the motor mount and face plate by a rubber o-ring. After approximately 250 sharpenings, this graphite collector will need to be emptied. To remove this barrel assembly, locate the sharpener over a waste basket with the collector barrel pointing downward. Grasp the barrel with the opposite hand and rotate it while pulling it away from the motor mount and face plate. Once the o-ring disengages from the collector barrel, it will be easy to separate and empty.

While the collector barrel assembly is removed, check the rod electrode cutter head and blade. There should not be an accumulation of oil or carbon in this area. If an oily residue is present, this is an indication that the rod electrodes are not being adequately cleaned before insertion into the electrode sharpener. To clean this assembly, remove the cutter blade and use general purpose spray and wipe detergent. Reassemble the cutter blade and the graphite collector barrel assembly.

The cutter blade, part number M90102, for the rod electrode sharpener is a three-sided tungsten cutter tool commonly used in machining operations. One side is capable of approximately 1000 sharpenings. The edge of a cutter is worn when the initial cut of the electrode requires an abnormal amount of inward pressure, and/or a smooth reflective surface cannot be achieved on the tip of the rod electrode. Refer to Section 3 for the procedure to replace the cutter blade.

The electrode sharpener power is on a timing circuit and will turn off after approximately 3 to 5 minutes. The electrode sharpener can also be turned off by placing the MODE switch in the STANDBY position.

1.3 Installing the Disc Electrode

The disc electrode is the most significant contributor to the accuracy and repeatability of the instrument. They are manufactured and then purified to strict specifications to ensure that they do not contain unacceptable levels of trace element contamination for the elements of interest. The care taken to properly install the disc on the shaft will help to ensure that excitation parameters will be kept as constant as possible, thus resulting in repeatable analytical data.

To install the disc electrode on the shaft, a laboratory grade disposable towel is recommended. See Section 1.7 for a description of the laboratory grade paper towel. Pour out a few disc electrodes onto a clean laboratory grade paper towel. Take a laboratory grade paper towel and double it to be sure that no contamination from the fingers will be absorbed into the disc. If large size paper towels are used, they should be cut with scissors into two inch squares to facilitate easy handling. Place the towel over the disc electrodes, and with the forefinger and thumb, grab one disc from the pile and place the disc on the shaft, Figure 1-4. With firm pressure, push the disc electrode onto the shaft until it comes to rest against the index shoulder of the shaft. If the disc electrode does not offer some resistance to the shaft as it is being inserted, remove and discard this electrode because the inner diameter has not been made to the tolerances specified.

CAUTION: The disc electrode shaft is designed to be replaced by the operator using a small jeweler's screwdriver. The shaft has right hand threads for tightening it into the commutator. When pushing the disc electrode on the shaft, do not apply counter-clockwise rotation on the disc electrode as this may cause the disc electrode shaft to loosen.

NOTE: Loose disc electrodes will produce erroneous results. If the disc electrode is too loose, arcing will occur between the inner diameter of the disc and the outer diameter of the shaft.

CAUTION: The disc electrode may be very hot to the touch.

Use a towel to remove a disc electrode from the shaft after an analysis, and to wipe away any oil which may have spilled over from the burn.

1.4 Installing the Rod Electrode and Setting the Gap

The rod electrode is installed after the disc electrode is already in place. To install the rod electrode, take the rod in the fingers of the right hand

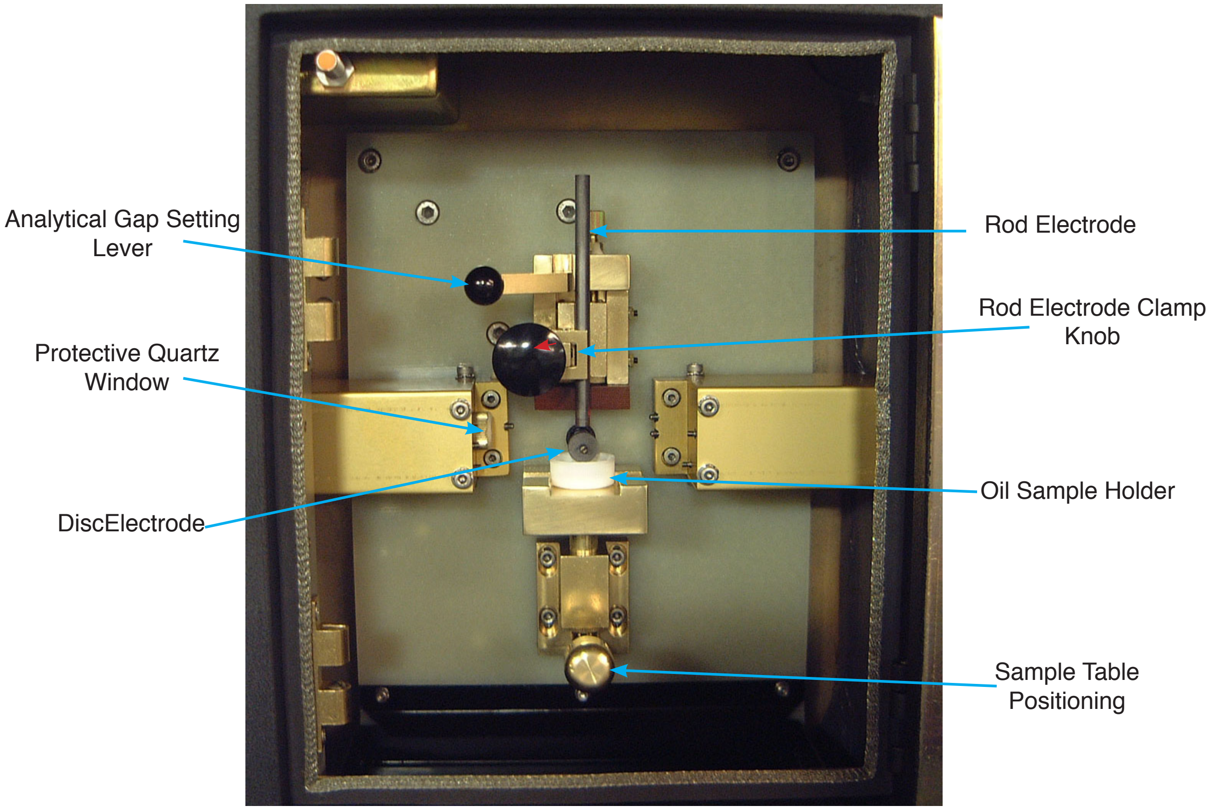


Figure 1-4, Sample Stand and Controls for Routine Operation

and with the left hand apply inward pressure to the round black rod electrode clamp knob, Figure 1-4. This will open the clamp door approximately 3/8 inch (9.5 mm).

Insert the rod electrode into the vertical "v" shaped channel until the sharpened tip can be seen protruding from the bottom of the rod holder and gap setting device. Release the rod clamp knob and the rod electrode will be pinched between the back of the rod clamp knob and the centering "v" channel. Press and then release the rod clamp knob again and the rod electrode will drop by gravity and come to rest on the disc electrode.

Raise the analytical gap setting lever. This action will drive the rod electrode holder and slide mechanism downward along the vertical axis. As the slide mechanism moves downward, the rod electrode remains in the installed position, because there is zero clearance between the disc and rod electrodes. The analytical gap setting lever will reach the end of its travel when it is raised to the full upward position. Return the analytical gap setting lever to the lowered position. As the lever begins to return to the lowered position, the rod electrode holder and slide mechanism begins to raise upward along the vertical axis until it is stopped by the analytical gap adjustment screw. This time the rod electrode, which is clamped in the rod electrode holder and slide mechanism, will travel upward with the slide mechanism. An analytical gap distance of 0.090 inches has now been precisely set.

NOTE: Care must be taken not to touch the brass block with the tip of the carbon rod in order to avoid false copper readings.

The analytical gap distance has been set during factory calibration and should not be readjusted during routine operation.

1.5 Installing and Positioning the Sample Holder and Cover

The Spectroil M/F-LD can accommodate several different types of sample holders and a sample holder cover. The following paragraphs describe the procedure to install disposable and reusable sample holders and the sample holder cover.

Disposable sample holder require the use of an adopter, Figure 1-5a. The adopter with the sample is placed in the slot at the top of the sample table.

The sample table also has a special cutout and groove to hold the reusable sample holder in place and to align it properly for the analysis, Figure 1-5b. The reusable sample holder should be cleaned with an ultrasonic bath and an environmentally acceptable cleaning solution.

When performing fluid analysis, an important consideration which has an effect on the reproducibility of the analysis is the quantity of the sample introduced into the analytical gap. This parameter is one for which the instrument cannot adjust. Proper level of oil in the sample holder is, therefore, part of any good operator technique. Standards and samples are analyzed either in disposable plastic sample holders or a reusable sample holder. In either case, it is recommended that the sample holder be filled level with the top.

Sample holder covers are required when flammable fuel samples are analyzed. Since these samples might ignite during the analysis, a cover is used to retard the flame and to minimize smoke which will attenuate signal fuel. The cover works with the disposable and reusable sample holders, Figure 1-6a and Figure 1-6b.

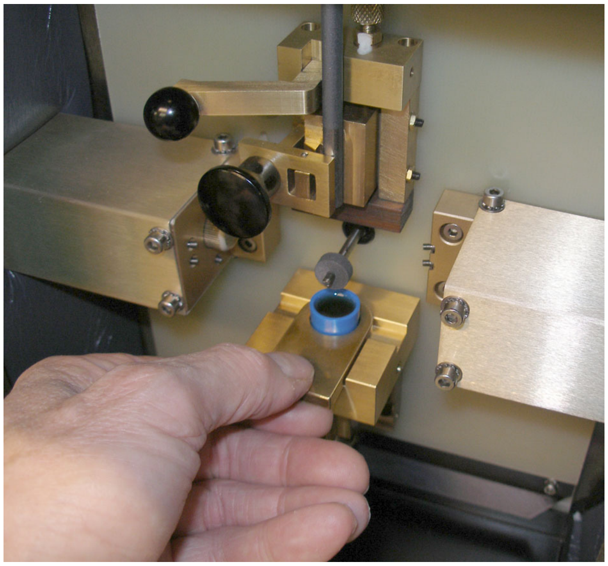


Figure 1-5a, Sample Stand with Disposable Sample Holder in Place

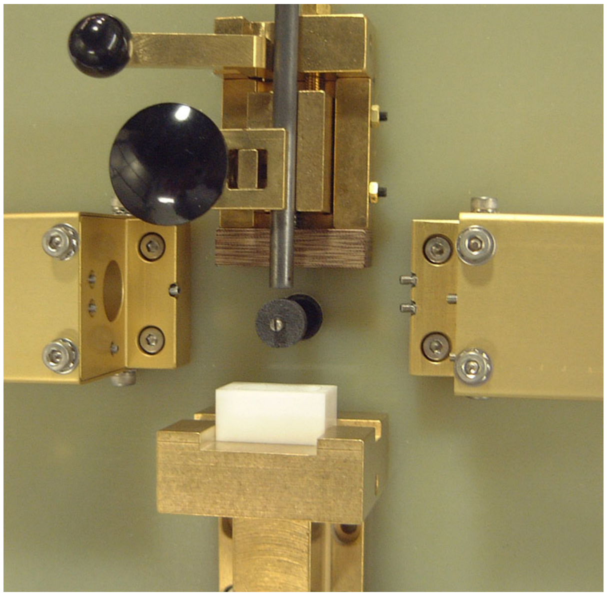


Figure 1-5b, Sample Stand with Reusable Sample Holder in Place

The following sample stand preparation sequence should be followed to analyze samples that require the sample holder cover:

1. Install the disc electrode, Section 1.3
2. Install and position the reusable sample holder, Figure 1-5b. Raise the sample holder in position and with a disposable pipette, fill

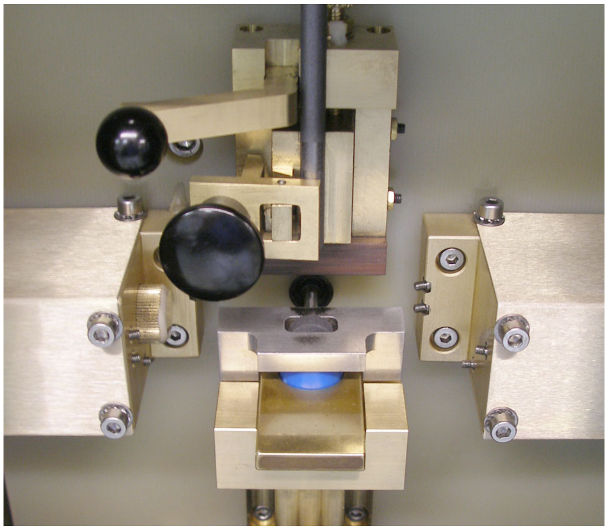


Figure 1-6a, Sample Stand with Disposable Sample Holder and Cover in Place

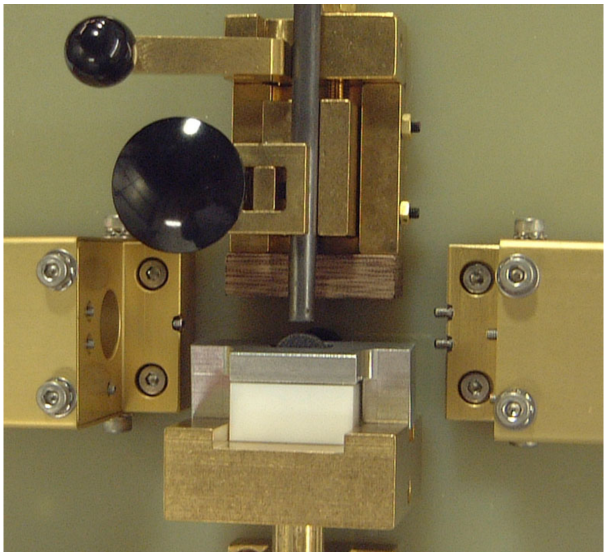


Figure 1-6b, Sample Stand with Reusable Sample Holder and Cover in Place

the sample holder with the fuel sample. Do not overfill the sample holder.

3. Place the cover over the reusable sample holder and disc electrode. Note that the cover only fits in one direction and has a cutout for the disc electrode shaft.

4. Install the rod electrode and set the gap, Section 1.4.

The sample is now ready for analysis.

When the analysis is complete, open the sample stand door, remove the rod electrode, remove the cover, lower the sample table, remove the sample, and remove the disc electrode. The sample stand is now ready for the next analysis.

1.6 Cleaning the Sample Stand

The Spectroil M/F-LD incorporates the rotating disc arc emission technique for excitation of the fluid sample. This technique produces a fine carbon residue which, when combined with oil droplets, produces an oil coating over the sample stand and door area. If allowed to accumulate, this coating will collect the carbon particles and eventually produce a lower resistance path than the analytical gap. If this occurs, the high voltage will not discharge across the analytical gap, but will discharge along the lower resistance path causing damage to the sample stand components.

To prevent arc-over, it is recommended that the operator perform the simple cleaning procedures outlined below.

WARNING
PROLONGED CONTACT WITH SOME SOLVENTS AND OILS MAY CAUSE CANCER!

WARNING
DO NOT USE ANY CHLORINATED SOLVENTS INTERNALLY OR EXTERNALLY ON THE INSTRUMENT!

CAUTION: All chemicals should be used in accordance with good laboratory practice. Proper ventilation is required when using any solvent. Skin contact and prolonged exposure to fumes produced by any solvent may be hazardous.

1.6.1 Cleaning After Each Burn Cycle

Take the paper towel used to remove the disc electrode from the shaft and clean the shaft, the sample table, and the sample plate area between the disc electrode shaft and the rod electrode clamp.

1.6.2 Cleaning After Each Operating Shift

After 8 hours of operation, the complete sample stand area must be wiped clean of the oil film buildup created by the burn cycles. If performed routinely, the sample stand can be cleaned simply with paper towels and moderate rubbing. However, if this procedure is performed sporadically or inadequately, an oil dispersant may be required to remove the buildup. A general purpose foam type spray detergent is recommended to dissolve the oil film buildup. A spray detergent is capable of contacting those areas which are hard to reach. Remove all detergent by wiping dry with paper towel.

1.6.3 Cleaning the Quartz Window

The quartz window that protects the lens and fiber optic must be cleaned frequently depending upon the type of fluid being analyzed. In general, this should be done at least every 5 burn cycles. To clean the protective quartz window, take a clean, soft, disposable laboratory tissue and wet one corner of the towel with isopropyl rubbing alcohol or ammonia based window cleaner. With the forefinger, rub the wetted portion of the paper towel along the surface of the window while applying moderate clockwise pressure on the window. This will disperse the oil film. Now take the dry portion of the paper towel and repeat this procedure until no oil can be seen on the tissue paper. A cotton swab can also be used for this purpose. A diluted solution of ammonia and water may be used.

CAUTION: The lens protected by the window does not require cleaning and should only be disassembled by a qualified engineer.

CAUTION: Do not use solvents to clean this window as they may selectively block or attenuate the passage of light necessary to determine the presence and concentration of the elements in the oil samples.

1.6.4 Cleaning Solutions

The Spectroil M/F-LD is designed to analyze petroleum and synthetic base products. In operation, the handling and actual analysis of these products create spillage and often leave an oily film on the instrument. In general, these spills can be adequately cleaned simply by wiping the surface with paper towels. There are occasions, however, where the petroleum/synthetic product may require a detergent to dissolve the petroleum base. For these occasions and for routine cleaning, a general purpose spray and wipe detergent is recommended for internal and external instrument components.

1.7 Paper Tissue for Operating and Paper Towels for Cleaning

Disposable paper tissues and towels are recommended for use in the daily operation of the Spectroil M/F-LD. The type of paper tissue used to handle the disc electrodes is very important. Most household tissue paper is treated with certain elements to make it softer or more absorbent. If used to handle the disc electrodes, these elements will contaminate the electrodes and produce erratic results, especially for silicon. Therefore, it is recommended that a laboratory grade paper tissue be used for this operation.

Paper towels are useful to clean the sample stand components and wipe spills which occur during routine operation. The type of paper towel used for this function is not critical. Typical household towels or C-fold janitorial towels work best for this function because of their absorbent characteristics.

1.8 Waste Oil Disposal Container

A waste oil container for oil analysis applications is required to properly dispose of the remaining oil sample after the analysis cycle. It is recommended that this waste oil container be in the form of a rectangular pan approximately 6 inches long, 4 inches wide, and 1 inch deep, with a screened cover to permit the remaining oil to drain through the screen. If a drain tube is installed on the bottom of the waste oil container, the waste oil container can continuously empty into a large capacity reservoir for proper disposal. Good laboratory procedures should be exercised in the disposition of all waste oils.

2.0 DAILY OPERATION

This section details those procedures that will be routinely used in the day-to-day operation of the Spectroil MF-LD. The operator must be familiar with the general operating requirements described in Section 1.0. A flow chart of the normal daily routines is shown on the next page in Figure 2-1. For convenience, the parentheses after each step in the chart refer to the corresponding sections in this chapter. The various procedures are explained in brief, easy to follow step-by-step instructions.

The spectroil M/F-LD may be equipped with the optional sulfur capability. The procedure to analyze samples for sulfur is described in Section 2.9 of this manual.

2.1 Daily Routine Prior to Use

1. Place the MODE switch on the operator's control panel to the OPERATE position, Figure 2-2. Power will be applied to the electrode sharpener and exhaust fans when MODE switch is in the OPERATE position.

2. Verify positive action from the sample stand exhaust system. With the sample stand door open, hold a piece of tissue paper up to the exhaust filter. It should be sucked up and held in place against the filter. Remove the tissue.

Figure 2-2, Control Panel

3. Turn printer ON and check to see that sufficient paper is available. If the printer has an ON LINE light, it should be illuminated.

4. Ensure that an ample supply of sample holders, sharpened electrodes and discs are on hand.

5. Select standards for daily use and shake vigorously for at least 30 seconds.

6. Have an oil waste container on hand (Section 1.8).

7. Have cotton swabs, contaminant free tissue paper and paper towels on hand (Section 1.7).

2.2 Warm-Up Procedure

If the Spectroil M/F-LD has been idle for several hours, it may be necessary to conduct a series of burns to introduce light into the optics and to allow the electronics to become warm. This warm-up exercise can be conducted with any oil sample or standard and can use electrodes which have been burned before. It is recommended that at least three warm-up burns be conducted.

1. Analyze or "burn" three or four samples (do not burn the same sample more than twice to prevent sample ignition) in accordance with the instructions given in Section 2.3 Routine Sample Analysis.

NOTE: For the warm-up cycle only, the same disc and rod electrodes can be utilized for up to four consecutive burns but the electrodes have to be regapped after each one.

2. The results produced by the warm-up burns are of no use. Press function key 6 (F6)AVERAGE . This prepares the screen for the next analysis at which time the three warm-up burns will be cleared.

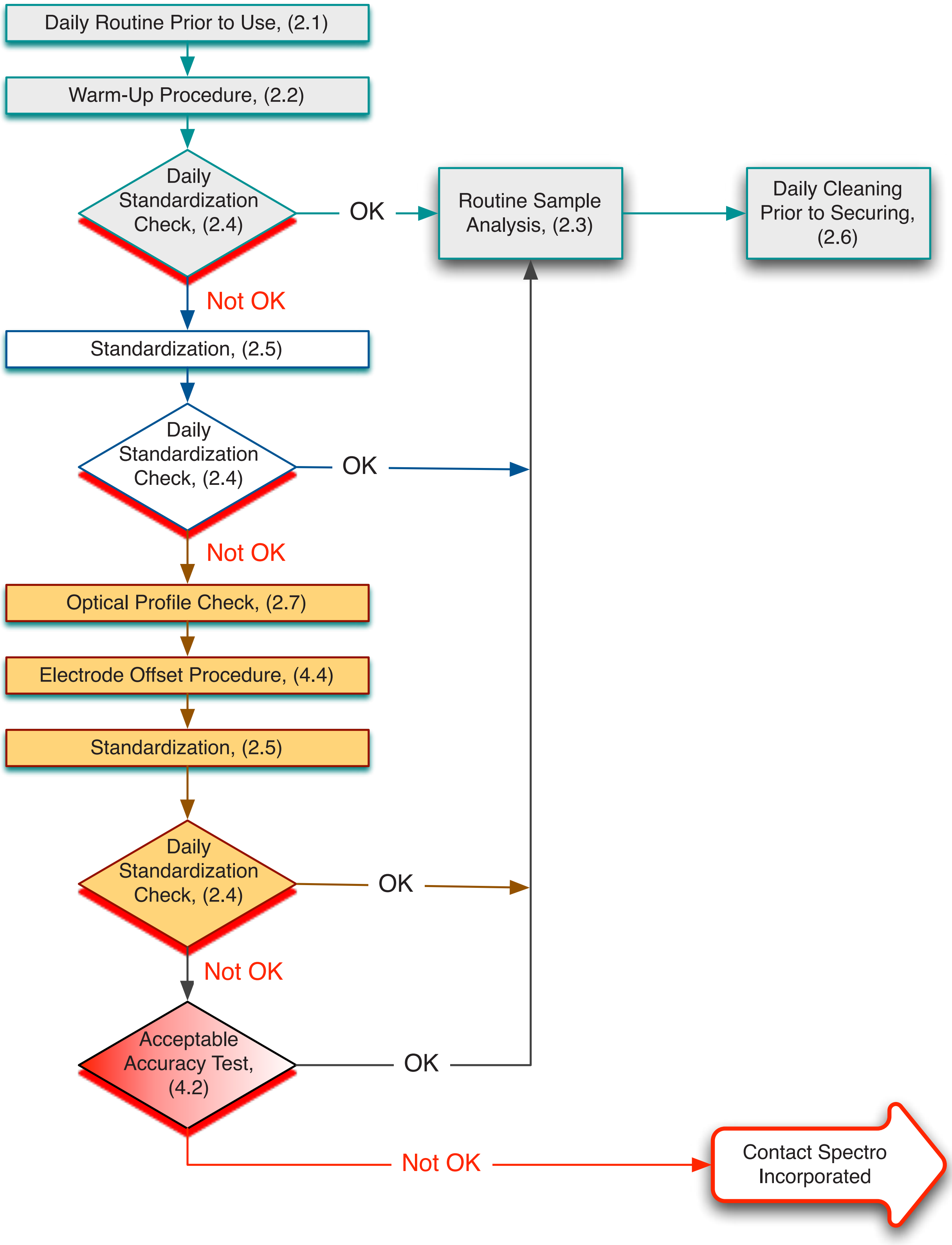


Figure 2-1, Daily Operating Procedure Flow Chart

2.3 Routine Fuel Sample Analysis

This paragraph gives the steps to follow to analyze or "burn" a fuel sample or a calibration standard using the disposable sample holders. Refer to the referenced sections for details. The various parts referred to are shown with labels in Figure 2-1. Refer to Section 2.9 if sulfur in fuel is to be analyzed.

NOTE: When a new lot of disc electrodes is started, either from a new manufacturer or a different lot from the same manufacturer, the disc electrode offset procedure of Section 4.4 must be performed.

Note On Sample Contamination

The data generated by an analytical instrument is only as good as the samples that are provided. It is imperative that the fuel sample be taken from the same sampling point and in an approved and repeatable manner such as ASTM D 4057 Manual Sampling of Petroleum and Petroleum Products. The sampling container must be clean and free of any contamination. In the laboratory, sample handling and analytical techniques are also important. Incorrect or careless procedures can influence analytical data that may lead to inaccurate data.

Contamination through careless operation can also be a source of error. The fuel sample and the graphite electrodes of the RDE spectrometer should never be touched by hand. The electrodes must always be installed with a laboratory disposable towel to avoid contamination from the fingers. Perspiration from the body will easily add several parts per million of sodium to the analysis. The the rod electrodes must be wiped off before sharpening and the electrode sharpener must be emptied and cleaned frequently. Resulting errors, when not detected can lead to erroneous conclusions and costly consequences about the status of the fuel or the fuel treatment system. Although the fuel analyzer is simple to operate, proper housekeeping procedures must always be followed.

NOTE: Rubber gloves or finger tip covers such as Anti-static Nitrie COTS should be worn when handling fuel samples, standards and consumables. This will protect the user and minimizes contamination.

1. The video monitor should display the Analysis Program screen, Figure 2-3. If a screen saver is in use, the Analysis Program screen will not be displayed. Press any key on the keyboard to terminate the screen saver and re-display the Analysis Program screen.

2. Install a carbon disc on the disc shaft using a clean laboratory grade tissue to avoid contact with fingers (Section 1.3).

3. Place a disposable sample holder on the sample holder adopter and fill the holder with sample to be analyzed, Figure 2-4. Be sure to always fill sample holders to the rim (Section 1.5).

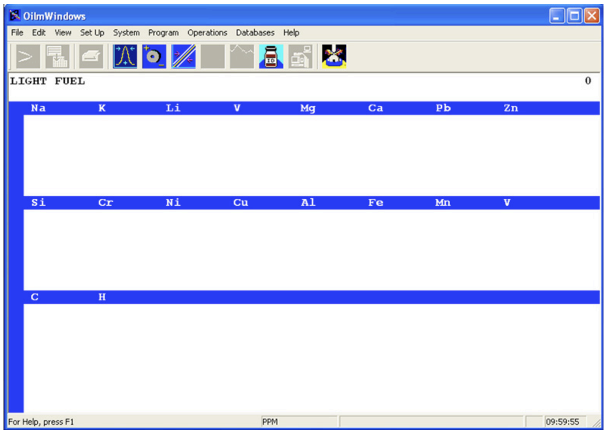


Figure 2-3, Sample Analysis Screen

Figure 2-4, Disposable Sample Holder on Adopter

4. Place the adopter with the filled sample holder on the table and slide it back to the end of the groove on the table (Section 1.5).

NOTE: When light fuel samples are analyzed, reusable sample holders and sample holder covers must be used to prevent sample ignition.

5. Raise the sample table using the sample table positioning lever (sample fluid will contact bottom of disc) (Section 1.5). See Section 2-8 if Sample ID's are to be entered.

6. Place the cover on the disposable sample holder and disc electrode, Figure 2-5. Note that the cover fits only in one direction and has a cutout for the disc electrode shaft.

7. To install the rod electrode, press inward on the black plastic knob of the spring loaded rod electrode clamp to open the jaws of the clamp. Insert a graphite rod electrode until the tip of the carbon rod is in contact with the disc electrode, then release the knob to secure the electrode in the clamp.

8. Set the analytical gap mechanism by raising and then lowering the analytical gap setting lever. This will set a gap distance of 0.090 inches between the disc and rod electrodes (Section 1.4).

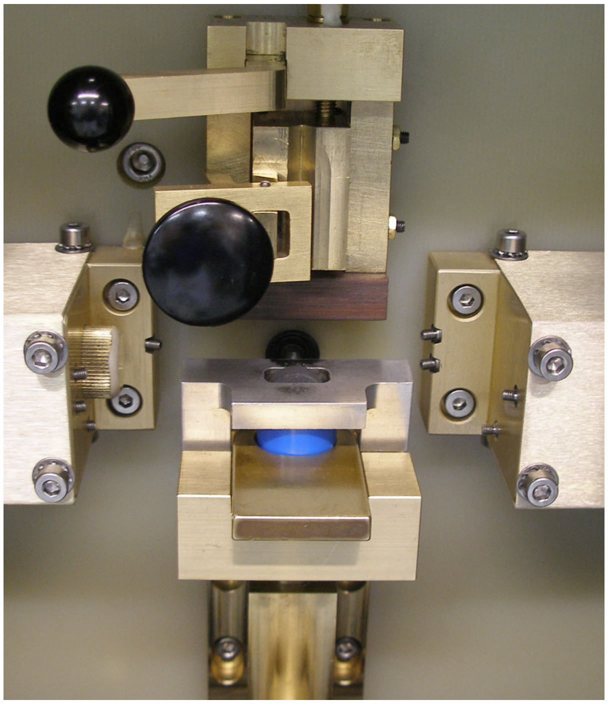


Figure 2-5, Cover Placement on Disposable Sample Holder

NOTE: The total analysis time on the Spectroil M/F-LD is set to 48 seconds (24 seconds pre burn and 24 seconds integration time.

9. Close the sample stand door and press the START button or function key 9 (F9) START. The analysis cycle for light fuels is 48 seconds consisting of 24 seconds of pre-burn followed by 24 seconds of the analytical cycle.

10. When the burn is complete, results will appear on the video screen.

11. After the burn is complete, open the sample stand door and remove the rod electrode. Set it aside for subsequent re-sharpening before it is used again.

12. Lower the sample table, remove and discard the sample holder and set aside the sample cover for cleaning.

CAUTION: The disc electrode will be hot to the touch.

13. Using a paper towel to protect fingers from the hot disc, remove and discard disc electrode.

14. Using a tissue or paper towel, wipe excess, spilled or splattered sample fluid from sample table and disc electrode shaft.

NOTE: The quartz protective window should be cleaned every burn.

15. The Spectroil M/F-LD is now ready to analyze another sample by again following the above 14 steps.

To improve precision and to comply withe ASTM D 6728, the sample should be analyzed at least three to five times and averaged. The spectroil M/F-LD will calculate an average for the analyses on the screen when function key 6 (F6) or the average icon are pressed.

NOTE: A copy of ASTM D 6728 Standard Test Method for Determination of Contaminants in Gas Turbine and Diesel Fuel by Rotating Disc Electrode Atomic Emission Spectrometry can be obtained from ASTM International at www.astm.org.

The 20% rule can be used to reject an analysis. View the analyses on the video screen and if one of three, or up to two of five are more than 20% different from the remainder, they should be rejected. To reject an analysis, click on the sample number to select the entire analysis and press the DELETE key. Pour another sample and analyze it to replace the rejected one.

16. When you are satisfied with the analyses, press function key (F6) or click on the Averages icon to complete the analysis procedure.

CAUTION: When all the fuel samples have been analyzed, any disc electrodes that have been dispensed from their holder should be set aside for warm-up burn use only, or they should be discarded to avoid the possibility of picking up contamination.

2.4 Daily Standardization Check

The standardization check is performed to verify that the instrument has remained in calibration. It is a quick method of verifying that the instrument can give accurate results without conducting a full standardization.

This procedure requires that the operator analyze three different levels of calibration standards and compare the results to Table 2-1. The first standard is a 0 ppm high-purity base oil (HP Base) standard, the next is a 1 ppm standard for Na, K and Li and finally a 10/30 ppm standard for the other elements.

The following steps should be conducted only after the warm-up procedure has been completed and the window has been cleaned in accordance with Section 1.6.3.

1. Prepare sample stand in accordance with Daily Routine Prior to Use, Section 2.1.

2. Make 3-4 warm-up burns per Section 2.2.

3. Clean the quartz window.

4. Make one burn of the HP Base standard.

5. Compare the results of this burn with Table 2-1. If all the elements are within the acceptable range, proceed to Step 7, 8, and 9. If the results are not within the range, proceed to Step 7.

6. Make a two more (total of three) burns of the HP Base standard and press function key (F6) to average the results. If all elements are within the range, proceed to step 7, 8, and 9. If not, perform complete Standardization Procedure , Section 2.5, in accordance with the Daily Operating Procedure Flow Chart.

7. Make one burn of the 1 ppm Na, K, Li stan-

Table 2-1, Acceptable Range Indices for Daily Standardization Check

Standard	Elements	Maximum	Minimum
HP Base (0 ppm)	Na, K, Li	0.05	-0.05
	All Others (excluding Na, K, Li)	1.0	-1.0
1 ppm	Na, K, Li	1.15	0.85
10/30 ppm	All Others (excluding Na, K, Li and Mg)	11.5	8.5
	Mg	33.0	27.0

dard and repeat steps 5 and 6.

8. Make one burn of the 10/30 ppm standard and repeat steps 5 and 6.

9. Daily Standardization Check is now complete.

2.5 Complete Standardization

Complete standardization is a procedure performed to place the calibration of the instrument as close to the standard values as the instrument originally produced during factory calibration. This procedure involves burning oil standards at predetermined points along the calibration curve. After these standards are analyzed, the computer software will determine mathematical factors to correct for any change in the calibration. Complete standardization is performed under the following conditions:

- When the instrument has been relocated to another site for operation. This is generally performed after the optical profile procedure has been completed.
- When results from the daily standardization check fall outside of acceptable limits for operation.
- After optical profiling procedure has been performed.

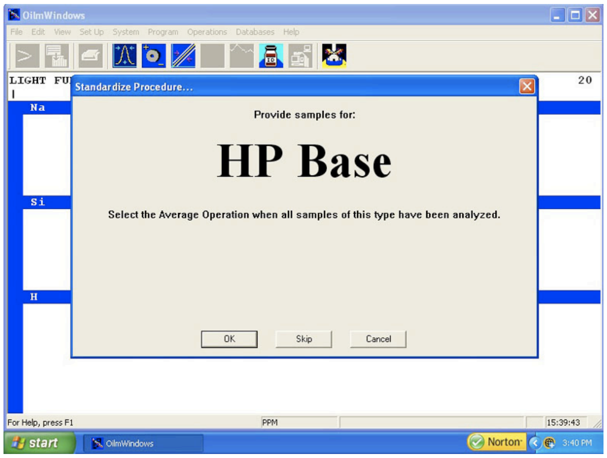
A complete standardization is performed by burning calibration standards that have been pre-selected during factory calibration of the instrument. The concentration levels for complete standardization have been selected based on the application and typical operating range for the elements of interest. For the light fuel analysis application, all elements are standardized with a High Purity Base (HP Base) standard to determine the background level. For a second calibration point, the alkali metals (Na, K and Li) are also standardized at 1 ppm and all other elements are standardized using the 10/30 ppm standard. The 10/30 ppm standard contains 10 ppm of all elements but 30 ppm of Mg. These concentration levels are programmed into the computer and are displayed at the appropriate time in the following procedure.

NOTE: Calibration standards are not flammable for normal analysis times and do not require a sample holder cover for analysis.

From the Analysis Program screen, press function key 7 (F7), choose the Standardization icon, or select the Operations/Standardize pull down menu. The software will automatically clear all previous measurements from the video display. A dialog with the name of the first calibration standard the instrument will expect to measure will appear. Refer to Figure, 2-6.

Three options exist when this dialog appears. The first is to select the OK button which confirms that the operator will begin to measure the 0 PPM (HP Base) standard. The second option is to select the SKIP button indicating the operator does not intend or need to measure the 0 PPM (HP Base) standard and wants to increment to the next standard in the standardization process. The last option is to press CANCEL and this action will terminate the standardization routine completely and return the software to the Analysis Program screen.

In most cases, the OK button will be selected and the dialog will disappear. Centered just below the tool bar will be the name of the standard, 0 PPM in a red banner. This banner will remain there



Provide samples for:

HP Base

Select the Average Operation when all samples of this type have been analyzed.

Figure 2-6, First Standardization Sample Dialog until an average is made and the next standard will appear. Those elements to be standardized at this concentration level will have their values appear and those elements not standardized will have no values appear.

NOTE: Reference channels are not standardized and therefore will not appear highlighted.

1. Select the HP Base standard and fill five sample holders. Take care to always fill the sample holder to the rim.

2. With an optical lens cleaning solution (not containing silicon), isopropyl alcohol, or a window cleaning solution with ammonia, clean the quartz window attached to the Fiber Optic Lens Holder in accordance with Section 1.6.3.

3. Following procedures set forth in Section 2.3, burn all five samples of the HP Base standard.

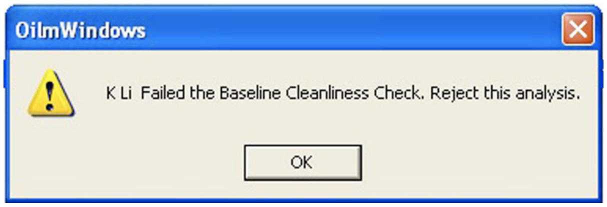
4. On completion of Step 3, look at the readings on the video screen. If one of the five burns does not appear to represent the other four, it may be rejected.

NOTE: The decision to accept or reject burns during this procedure is at the discretion of the operator. Quite often an operator will know the cause of a rejectable burn and therefore reject it almost automatically. Rejectable burns can be caused by inconsistencies in consumables such as excess variation in the specific density (hardness) of the disc electrode, a loose fitting disc electrode, a poorly sharpened rod electrode, an under- or over-filled sample holder or an analytical gapping error. For example, a rejectable burn is one which is obviously too high or too low when compared to the other four burns.

The Spectroil M/F-LD software also contains a ”Cleanliness Check” feature that monitors the analyses of the HP Base standard during standardization and the disc electrode offset procedure. When the set cleanliness value for sodium, potassium or lithium is overridden, an error message such as the one shown in Figure 2-7 appears. The error message may be due to sporadic contamination or a minor operator error and the element(s) that failed also appear in red on the analysis screen. Follow the instructions and reject the analysis. If 3 of the 5 analyses fail the cleanliness check, there may be a more significant problem. The screen shown in Figure 2-8 appears and Base Cleanliness Troubleshooting must be implemented, see Section 4.5.

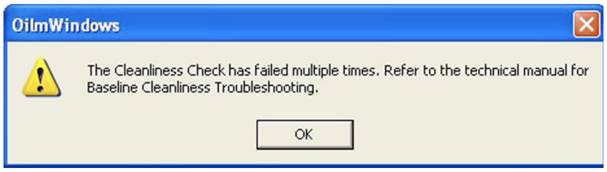
To reject one of the measurements from the video display, move the selection pointer over any portion of the measurement and left click the mouse one time. This will highlight the burn in a black background. Pressing the DELETE key on the keyboard one time will remove this measurement off of the screen. In the event that the wrong measurement has been highlighted, position the pointer over the measurement again and left click the mouse a second time. This will remove the highlight, then select the proper measurement to reject and press the DELETE key.

If a group of sequential measurements are to be rejected, for example measurements 3 and 4, place the pointer over the first measurement (#3) and left click the mouse one time to highlight the measurement. Then move the pointer over the next measurement (#4) and hold the SHIFT key down and left click the mouse one time. All measurements from number 3 through number 4 will be highlighted. Press the DELETE key to remove



K Li Failed the Baseline Cleanliness Check. Reject this analysis.

Figure 2-7, Error Message for Failed Cleanliness Check Baseline



The Cleanliness Check has failed multiple times. Refer to the technical manual for Baseline Cleanliness Troubleshooting.

Figure 2-8, Error Message for Multiple Analyses Failed Cleanliness Check Baseline the measurements.

5. After making as many measurements as necessary to obtain a good average, press function key 6 (F6), click the average icon, or select Operations/Average from the pull down menu. The average of each element will be calculated and displayed below each element's column of measurements. To make a printout of the measurements and their average, press the print icon. Automatically, the next dialog will appear providing instructions to make measurement of the 1 ppm standard as shown in Figure 2-9.

6. Select the 1 ppm standard and fill five sample holders. Take care to always fill the sample holder to the rim.

7. With an optical lens cleaning solution (not containing silicon), isopropyl alcohol, or a window cleaning solution with ammonia, clean the quartz window attached to the Fiber Optic Lens Holder in accordance with Section 1.6.3.

8. Following procedures set forth in Section 2.3, burn all five samples of the 1 ppm standard.

9. On completion of Step 8, look at the readings for Na, K and Li on the video screen. If one of the five burns does not appear to represent the other four, it may be rejected. Use the reject rule of ASTM 6728 to reject samples that differ more than 0.3 ppm at concentrations around 1 ppm, see also Section 2.3.

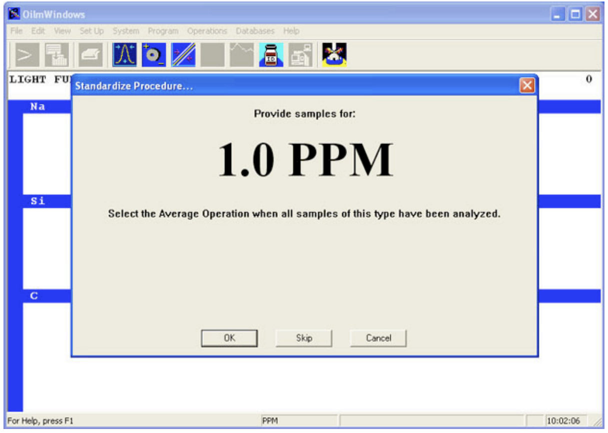
10. After making as many measurements as necessary to obtain a good average, press function key 6 (F6), click the average icon, or select Operations/Average from the pull down menu. The average of each element will be calculated and displayed below each element's column of measurements. To make a printout of the measurements and their average, press the print icon. Automatically, the next dialog will appear providing instructions to make measurement of the 10/30 ppm standard as shown in Figure 2-10.

11. Select the 10/30 standard and fill five sample holders. Take care to always fill the sample holder to the rim.

12. With an optical lens cleaning solution (not containing silicon), isopropyl alcohol, or a window cleaning solution with ammonia, clean the quartz window attached to the Fiber Optic Lens Holder in accordance with Section 1.6.3.

13. Following procedures set forth in Section 2.3, burn all five samples of the 10/30 ppm standard.

14. On completion of Step 13, look at the readings on the video screen. If one of the



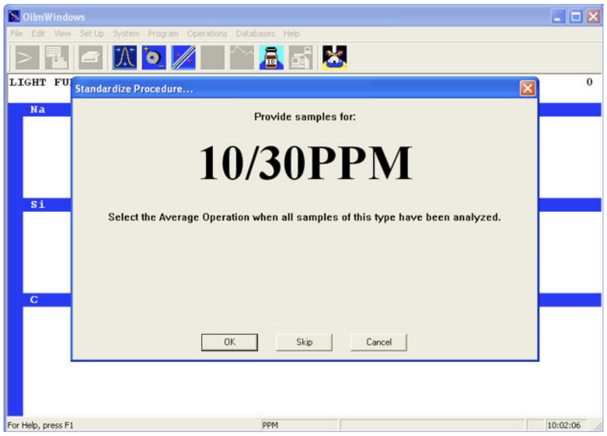
Standardize Procedure...

Provide samples for:

1.0 PPM

Select the Average Operation when all samples of this type have been analyzed.

Figure 2-9, Second Standardization Sample (Standard 2)



Standardize Procedure...

Provide samples for:

10/30PPM

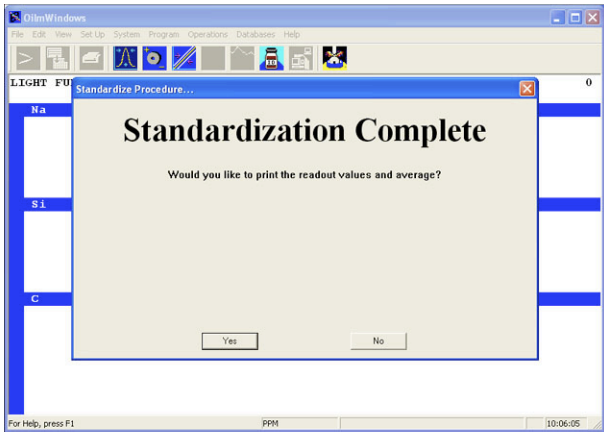
Select the Average Operation when all samples of this type have been analyzed.

Figure 2-10, Third Standardization Sample (Standard 3) five burns does not appear to represent the other four, it may be rejected. Refer to the note regarding the 80% rule to determine if a burn qualifies to be rejected.

15. After making as many measurements as necessary to obtain a good average, press function key 6 (F6), click the average icon, or select Operations/Average from the pull down menu. The average of each element will be calculated and displayed below each element's column of measurements. The next dialog to appear will indicate standardization is complete and inquire if the average and burns for the last standard measured should be printed. Refer to Figure 2-11.

16. Click on the Yes button if a printed copy of the analyses is desired. After clicking on the Yes or the No button, the Standardization Values Screen, Figure 2-12 is displayed. This table can also be displayed at any time by selecting Program/Standardization Samples/Standardization Values.

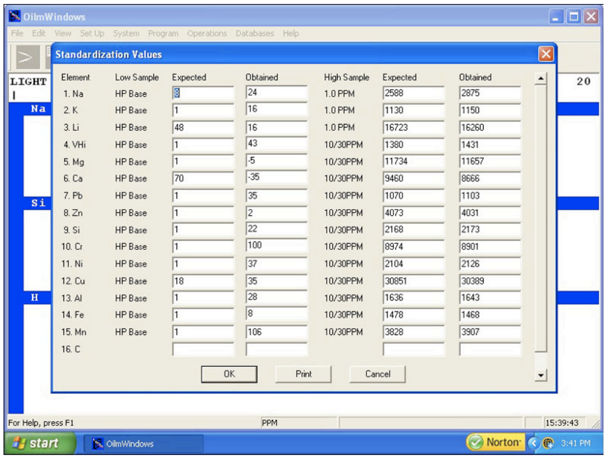
NOTE: It is strongly recommended that accurate records of the complete standardization data be kept for future reference. For this reason we highly recommend that printouts of all daily standardizations be printed and kept on file. This data reflects the current Spectroil M/F-LD standardization.



Standardization Complete

Would you like to print the readout values and average?

Figure 2-11, Standardization Complete, Dialog



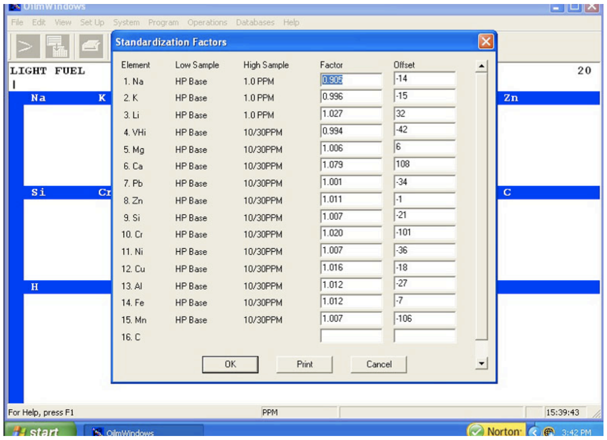
Element	Low Sample	Expected	Obtained	High Sample	Expected	Obtained
1. Na	HP Base	8	24	1.0 PPM	2588	2875
2. K	HP Base	1	16	1.0 PPM	1130	1150
3. Li	HP Base	48	16	1.0 PPM	16723	16260
4. VHi	HP Base	1	43	10/30PPM	1380	1431
5. Mg	HP Base	1	-5	10/30PPM	11734	11657
6. Ca	HP Base	70	-35	10/30PPM	9460	8666
7. Pb	HP Base	1	35	10/30PPM	1070	1103
8. Zn	HP Base	1	2	10/30PPM	4073	4031
9. Si	HP Base	1	22	10/30PPM	2168	2173
10. Cr	HP Base	1	100	10/30PPM	8974	8901
11. Ni	HP Base	1	37	10/30PPM	2104	2126
12. Cu	HP Base	18	35	10/30PPM	30851	30389
13. Al	HP Base	1	28	10/30PPM	1636	1643
14. Fe	HP Base	1	8	10/30PPM	1478	1468
15. Mn	HP Base	1	106	10/30PPM	3828	3907
16. C						

Figure 2-12, Standardization Values Table (Example)

The table in Figure 2-12 compares the expected intensities (the intensities generated during factory calibration) to the obtained intensities (the intensities obtained from the most recent complete standardization). From the relationship of the expected intensities compared to the obtained intensities of the low standard and the high standard, the standardization factor is derived.

Another table is displayed after you click OK or Cancel on the Standardization Values Screen. This second table is called the Standardization Factors Table. This table can also be displayed on the screen by selecting Program/Standardization Samples/Standardization Factors. Refer to Figure 2-13 .

This table displays a factor for each element. At best, the factor would be exactly 1.000. In practice, differences in electrode grades, standards, and instrument variables will cause the intensities achieved to result in factors which are either slightly above or below 1.000. These factors should remain somewhere between 0.5 and 5.0. If a factor exceeds these tolerances, it is not always an



Element	Low Sample	High Sample	Factor	Offset
1. Na	HP Base	1.0 PPM	0.905	-14
2. K	HP Base	1.0 PPM	0.996	-15
3. Li	HP Base	1.0 PPM	1.027	32
4. VHi	HP Base	10/30PPM	0.994	-42
5. Mg	HP Base	10/30PPM	1.006	6
6. Ca	HP Base	10/30PPM	1.079	108
7. Pb	HP Base	10/30PPM	1.001	-34
8. Zn	HP Base	10/30PPM	1.011	-1
9. Si	HP Base	10/30PPM	1.007	-21
10. Cr	HP Base	10/30PPM	1.020	-101
11. Ni	HP Base	10/30PPM	1.007	-36
12. Cu	HP Base	10/30PPM	1.016	-18
13. Al	HP Base	10/30PPM	1.012	-27
14. Fe	HP Base	10/30PPM	1.012	-7
15. Mn	HP Base	10/30PPM	1.007	-106
16. C				

Figure 2-13, Standardization Factors Table (Example) indication of an error or pending problem. If such a case should occur, consult Spectro Incorporated Field Service for analysis and explanation.

17. A daily standardization check in accordance with Section 2.4 should be carried out to verify calibration.

18. Standardization is now complete and it is possible to burn routine fuel samples.

2.6 Daily Routine Prior to Securing

1. Turn the MODE switch on the control panel to STANDBY.

2. Turn the printer power switch OFF.

3. Remove disc and rod electrodes.

4. Clean disc electrode shaft with a paper towel.

5. Clean and wipe the entire sample stand area.

6. Clean the quartz protective lens using a clean soft disposable laboratory tissue.

7. Wipe all oil standard bottles clean.

8. Check supply of standards (don't run out).

9. Clean and wipe used oil container.

10. Clean the working area.

11. Sharpen all rod electrodes and store them so they are protected from inadvertent contamination.

2.7 Optical Profiling

The Spectroil M/F-LD optical system is shock mounted in a light-sealed and environmentally protected temperature stabilized enclosure. Consequently, the optics do not need to be profiled frequently. However, detection limit and repeatability suffer when the optics are off profile. Unfortunately, there is no one rule which ensures that the optics are on profile. The following guidelines are presented to indicate when profiling should be done:

- At least once every month.
- After the instrument has been transported to a new location.
- Whenever the instrument has been subjected to temperature variations greater than 15° F (10° C).

If one of these apply, it is also reasonable to perform a standardization as detailed in Section 2.5.

Follow the next steps as detailed in this procedure to determine the optimum optical peak profile position for operation.

1. Prepare the sample stand with new electrodes in accordance with the appropriate paragraphs of the General Operating Requirements section of this manual.

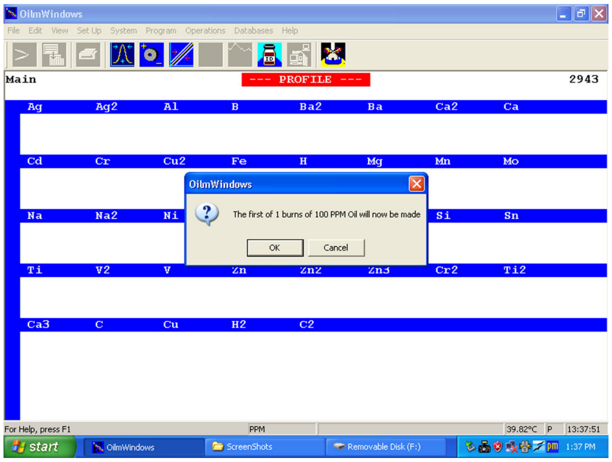
NOTE: it is important to only use the standard specified by the software profiling dialog for the profiling procedure.

1. To begin the optical profile procedure press function key 4 (F4), left click the profile icon, or select Operations/Profile from the pull down menu options.

2. The screen shown in Figure 2-14 appears and calls for the first analysis of the profiling standard..

3. Fill a sample holder with the profiling standard and following procedures set forth in Section 2.3, to burn an oil sample.

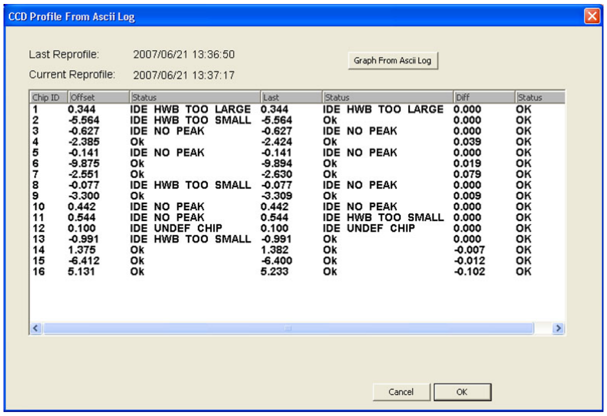
4. At the completion of the burn, a screen similar to Figure 2-15 will appear with the profile log. The profile log shows the current profile for each chip, the previous profile and



OilmWindows

The first of 1 burns of 100 PPM Oil will now be made

Figure 2-14, Optical Profile Screen



Last Reprofile: 2007/06/21 13:36:50

Current Reprofile: 2007/06/21 13:37:17

Chip ID	Offset	Status	Last	Status	Diff	Status
1	0.344	IDE HWB TOO LARGE	0.344	IDE HWB TOO LARGE	0.000	OK
2	-5.564	IDE HWB TOO SMALL	-5.564	Ok	0.000	OK
3	-0.627	IDE NO PEAK	-0.627	IDE NO PEAK	0.000	OK
4	-2.385	Ok	-2.424	Ok	0.039	OK
5	-0.141	IDE NO PEAK	-0.141	IDE NO PEAK	0.000	OK
6	-9.875	Ok	-9.894	Ok	0.019	OK
7	-2.551	Ok	-2.630	Ok	0.079	OK
8	-0.077	IDE HWB TOO SMALL	-0.077	IDE NO PEAK	0.000	OK
9	-3.300	Ok	-3.309	Ok	0.009	OK
10	0.442	IDE NO PEAK	0.442	IDE NO PEAK	0.000	OK
11	0.544	IDE NO PEAK	0.544	IDE HWB TOO SMALL	0.000	OK
12	0.100	IDE UNDEF CHIP	0.100	IDE UNDEF CHIP	0.000	OK
13	-0.991	IDE HWB TOO SMALL	-0.991	Ok	0.000	OK
14	1.375	Ok	1.382	Ok	-0.007	OK
15	-6.412	Ok	-6.400	Ok	-0.012	OK
16	5.131	Ok	5.233	Ok	-0.102	OK

Figure 2-15, Optical Profile Log

the difference between the two. The status for each chip should be OK, if not repeat the profile procedure one or two more times until the status is OK for all chips. If this condition cannot be achieved after three attempts, contact Spectro for assistance.

5. Click on OK to return to the main analysis screen. The profiling procedure is now complete.

2.8 Sample Identification (I.D.)

A sample identification (I.D.) can be added to each analysis for identification purposes. The ample I.D. can be configured by the user to include a variety of information about the analyzed sample. This section describes the setup process to format the sample I.D. and how to enter it in routine operation.

2.8.1 Setup of the Sample I.D.

To configure the format for the sample I.D., select "Sample I.D." from the System pull-down menu. This menu option produces a dialog, Figure 2-16, that can be configured by the user to meet any combination of alpha-numeric characters for global sample identification.

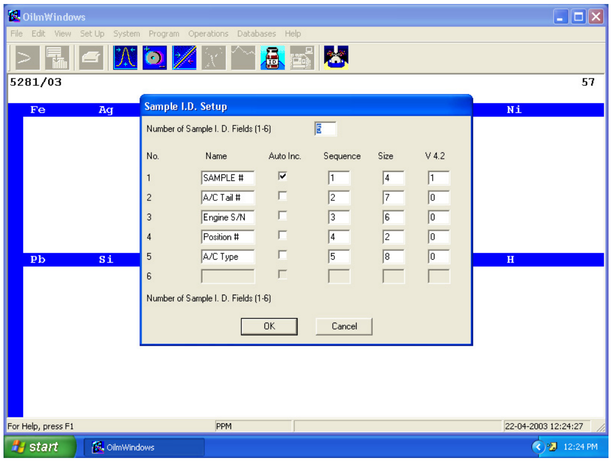
A sample can be identified by up to six field segments. This dialog permits the operator to choose how many field segments will be used for the sample identification and name each of these field segments.

Each field segment can also be auto-incremented, which means that after the first sample number is entered, and if all numbers that follow are in numeric order, they can be automatically filled in ascending order thus saving time.

The sequence of how the fields will appear can be determined or altered and the size of each field can be customized up to a maximum of 40 characters total. The last column of the sample I.D. dialog is for V4.2 protocol. This protocol is capable of storing up to two segments of the sample ID in the file. More than three segments are not permitted. That portion of the sample identification that has V4.2 assigned as segment 1 or segment 2 will be transmitted or stored under the V4.2 protocol.

2.8.2 Using Sample ID.'s

When the Spectroil M/F-LD is ready to analyze a



Sample I.D. Setup

Number of Sample I. D. Fields (1-6) 5

No.	Name	Auto Inc.	Sequence	Size	V 4.2
1	SAMPLE #	☑	1	4	1
2	A/C Tail #	☐	2	7	0
3	Engine S/N	☐	3	6	0
4	Position #	☐	4	2	0
5	A/C Type	☐	5	8	0
6		☐			

Number of Sample I. D. Fields (1-6)

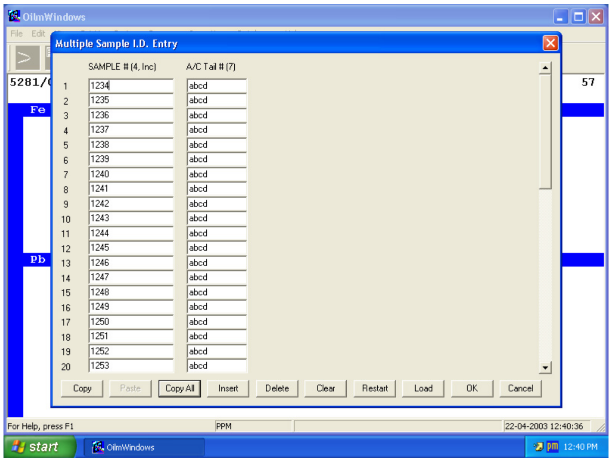
Figure 2-16, System/Sample ID Setup Menu sample, the Sample I.D. can be entered by pressing function key 3 (F3), or by clicking on the Sample I.D. icon. This menu option will produce a dialog to permit the entry of one single ID, Figure 2-17, or provide the capability to pre enter multiple sample ID's, Figure 2-18, using the MULTIPLE button. Both dialogs are configured at the system level by the System/Sample ID option.

The Multiple Sample ID Entry dialog allows up to 50 sample identifications to be pre loaded to facilitate rapid sample throughput. All sample ID fields are configured at the system level through the System/Sample ID menu option. Along the bottom of the Multiple ID dialog are buttons to expedite the entry of sample numbers. The Copy button will copy the contents of one field and permit it to be copied into another field of equal or greater field size using the Paste button. Copy All will copy one sample and insert it into all remaining empty fields in that column. If that column is set for auto increment, the sample number will increase one value per row. Insert, Delete, and Clear are self-explanatory.

Click load to proceed. The first time that this option is enabled, an input file layout screen, Figure 2-19 appears and must be filled in with the user layout preferences. Click OK when complete and the a screen enabling the user to select the sample ID files appears, Figure 2-20.

2.9 Sulfur Analysis

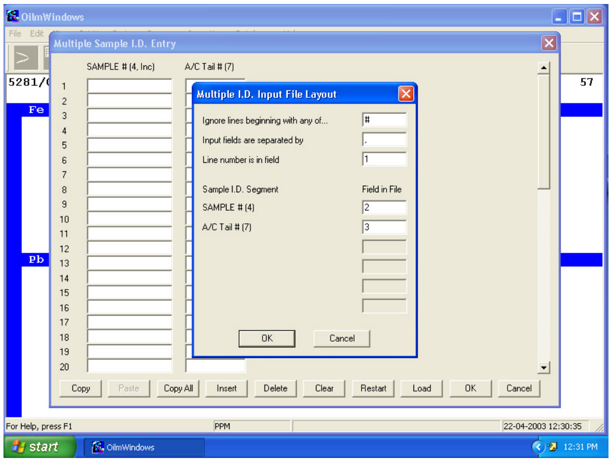
An optional sulfur analysis capability is available for the spectroil M/F-LD. The optional capability consists of a separate sulfur optic attached to the right hand side of the sample stand and some minor modifications to the sample stand. The sulfur spectral line is in a region of the spectrum



	SAMPLE # (4, Inc)	A/C Tail # (7)
1	1234	abcd
2	1235	abcd
3	1236	abcd
4	1237	abcd
5	1238	abcd
6	1239	abcd
7	1240	abcd
8	1241	abcd
9	1242	abcd
10	1243	abcd
11	1244	abcd
12	1245	abcd
13	1246	abcd
14	1247	abcd
15	1248	abcd
16	1249	abcd
17	1250	abcd
18	1251	abcd
19	1252	abcd
20	1253	abcd

For Help, press F1 | PPM | 22-04-2003 12:40:36

Figure 2-17, Single Sample ID Entry Menu

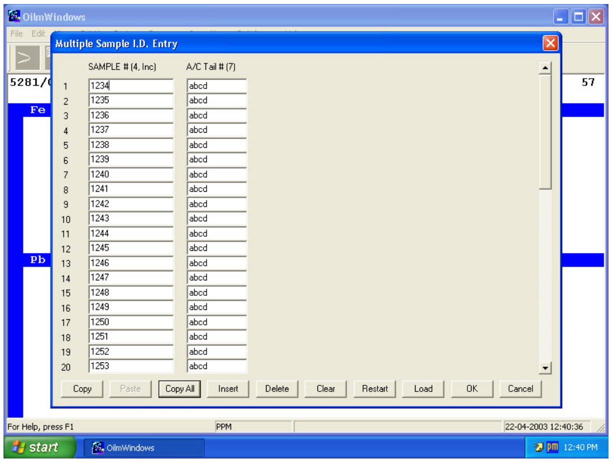


Ignore lines beginning with any of... #
Input fields are separated by ,
Line number is in field 1

Sample I.D. Segment	Field in File
SAMPLE # (4)	2
A/C Tail # (7)	3

For Help, press F1 | PPM | 22-04-2003 12:30:35

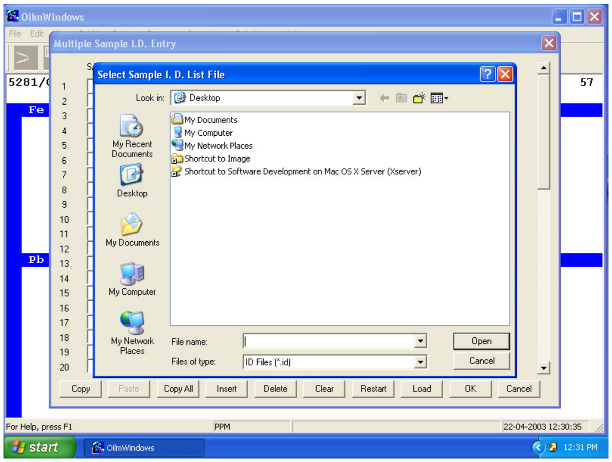
Figure 2-19, Sample ID File Layout Preference Screen



	SAMPLE # (4, Inc)	A/C Tail # (7)
1	1234	abcd
2	1235	abcd
3	1236	abcd
4	1237	abcd
5	1238	abcd
6	1239	abcd
7	1240	abcd
8	1241	abcd
9	1242	abcd
10	1243	abcd
11	1244	abcd
12	1245	abcd
13	1246	abcd
14	1247	abcd
15	1248	abcd
16	1249	abcd
17	1250	abcd
18	1251	abcd
19	1252	abcd
20	1253	abcd

For Help, press F1 | PPM | 22-04-2003 12:40:36

Figure 2-18, Multiple Sample ID Entry Menu



Look in: Desktop

- My Documents
- My Computer
- My Network Places
- Shortcut to Image
- Shortcut to Software Development on Mac OS X Server (Xserver)

File name:
Files of type: ID Files (*.id)

For Help, press F1 | PPM | 22-04-2003 12:30:35

Figure 2-20, Sample ID File Selection Screen where any signal resulting from the excitation process is absorbed by air. For this reason, when sulfur analysis is required, the sample stand area and the optic must be purged with an inert gas so that the CCD detector is able to see the sulfur signal generated at the analytical gap. To solve this problem, the Spectroil M/F-LD uses nitrogen as a gas and a separate optic just for the sulfur capability.

2.9.1 Set-up for Sulfur Analysis

Follow this procedure to prepare the sulfur optic for routine analysis.

1. Attach a low pressure regulator onto a Nitrogen bottle or other nitrogen source. The output of the regulator should have a 1/4 inch barbed quick disconnect, or ferrule hose fitting

2. Attach the 1/4 inch ID (inner diameter) hose that comes out of the back of the sulfur optic to the 1/4 inch barbed quick disconnect, or ferrule hose fitting on the regulator. Verify that all hose fittings are secure and no pressure leaks are present. Teflon tape should be used on all threaded hose fittings.

CAUTION: Pressure leaks will cause the introduction of fresh air to the purge path and will result in low readings for sulfur.

Sulfur is analyzed separately from the other elements, therefore the sample stand must be set-up so that the excitation process is viewed by the sulfur optic and not the routine optic. The excitation process can be selectively viewed to the left by way of the sulfur light pipe, or to the right for the routine analysis by way of the lens assembly.

3. To activate the view to the sulfur optic, the "L" bracket must be removed from the sulfur optic light pipe. Unscrew the round knurled nut that holds the "L" bracket in place and remove it from in front of the sulfur light pipe, Figure 2-21

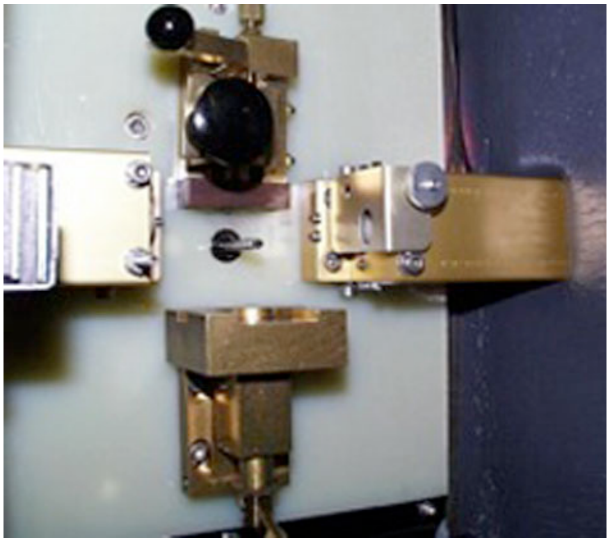


Figure 2-21, "L" Bracket Installed Covering Sulfur Optic Light Pipe

4. Relocate the "L" bracket over the lens assembly of the routine optic located to the left of the analytical gap, Figure 2-22. Use the round knurled nut to fasten it in the new location. This will help to keep the lens assembly clean during the sulfur analysis.

NOTE: Some instruments may be delivered with two "L" brackets installed. When that is the case, loosen the round knurled nut, flip up the "L" bracket for the sulfur light pipe and tighten the round knurled nut. Verify that the other "L" bracket over the lens assembly is in the "down" position.

5. Open the nitrogen bottle to allow the nitrogen to pass through the regulator, adjust the regulator to approximately 25 PSI.

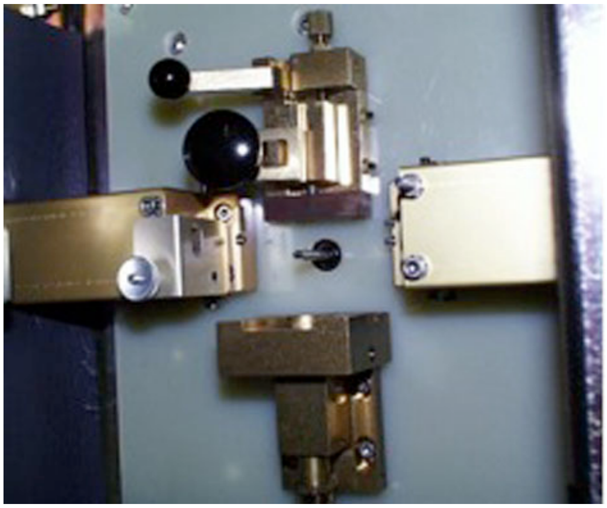


Figure 2-22, "L" Bracket Installed Covering Lens Assembly

6. Turn the main Nitrogen valve on the sulfur optic control panel to "ON", Figure 2-23.

7. If the Spectroil M/F-WLD is OFF, turn it ON by raising the circuit breaker (CB1) on the right side of the instrument. The spectrometer will power up and the OilM Windows software program will load.

8. Depress the "OPTIC PURGE" button, Figure 2-23, on the sulfur optic control panel for 5 seconds, and release for 5 seconds. Repeat this process 5 times to flush the optical system.

9. To get load the Sulfur program select **File** from the pull down menu options, then **Open**. A list of available programs will be displayed. Select the radio button for the Main Sulfur program and press **OK**. The sulfur optic is now ready for operation.

WARNING: Irritating and toxic hydrogen sulfide gas may be found in confined vapor spaces when samples with large percentages of sulfur are analyzed. Greater than 15 - 20 ppm continuous exposure (in air, not to be confused with the liquid calibration standard) can cause mucous membrane and respiratory tract irritation. 50 - 500 ppm can cause headache, nausea, and dizziness, loss of reasoning and balance, difficulty in breathing, fluid in the lungs, and possible loss of consciousness. Greater than 500 ppm can cause rapid or immediate unconsciousness due to respiratory paralysis and death by suffocation unless the victim is removed from exposure and successfully resuscitated.

Figure 2-23, Sulfur Optic Control Panel

The "rotten egg" odor of hydrogen sulfide is not a reliable indicator for warning of exposure, since olfactory fatigue (loss of smell) readily occurs, especially at concentrations above 50 ppm. At high concentrations, the victim may not even recognize the odor before becoming unconscious.

2.9.2 Profiling the sulfur Optic

The sulfur optic is profiled at the same time as the standard optic. Profiling should be performed if the instrument has been relocated and or shut off for an extended period of time.

The Nitrogen purge should be turned OFF at the sulfur optic control panel during profiling. To profile the optic, follow the instructions in Section 2. 7 in this manual.

Note: During the profiling procedure, both optics must see the analytical gap. Make sure the "L" brackets are removed from the sulfur light pipe and the lens assembly. If two "L" brackets are installed, they should be in the up position.

2.9.3 Standardization of the Sulfur Optic

The software must be in the sulfur program to perform this function. If not, see step 9 in Section 2.9.1.

1. From the main analysis screen, press function key 7 (F7), choose the Standardization icon or select the Operations/Standardize pull down menu. The software will automatically clear all previous measurements from the video display. A dialog box with the name of the first calibration standard the instrument will expect to measure will appear. A blank oil is used or this step and the

dialog box may call for "Base Oil", "Blank Oil", or 0 PPM Oil.

2. Following the procedure set forth in Section 2.3, burn three samples of Base/Blank oil and press function key 6 (F6), click the Average icon or select Operations/Average from the pull down menu to average them. To make a printout of the measurements and their average, press the print icon. Automatically, the next dialog box will appear providing instructions to make measurements of the next standard. The concentration of this standard is 1,000 PPM The name in the dialog box will vary depending on the calibration of the Spectroil M/F-LD which can be or light fuel, heavy fuel, crude oil, residual oil or # 2 diesel fuel.

3. Following the procedure set forth in Section 2.3, burn the three samples of the 1,000 PPM sulfur standard and press function key 6 (F6), click the Average icon or select Operations/Average from the pull down menu to average them.

4. The next dialog box that appears will indicate standardization is complete and inquire if the average and burns for the last standard measured should be printed, select YES.

5. After confirmation that the burn data and average are to be printed, the last dialog box to appear will inquire if the Factors and Offsets should be printed, again select YES. The Standardization procedure is now complete. Review the factor for sulfur; it should be close to one, +/- 20 %.

2.9.3 Verification

It is good practice to verify calibration after the Spectroil M/F-LD has been standardized.

1. In PPM mode, run five samples of each calibration standard in the set. Print all burns, averages and statistics. Remember the readings are displayed in percentages.

2. Depending on the type of standard analyzed, the averages should be better than +/- 15%. If not, repeat the standardization procedure, Section 2.9.3 and if it does, proceed with step 3.

3. Routine samples are analyzed for sulfur content per the procedure in Section 2.3, Routine Sample Analysis.

2.9.4 Clean Up and Final Checks

This section provides the steps that must be performed when the sulfur analysis procedure is complete nd the Spectroil M/F-LD will be used for the analysis of routine furl samples.

1. Turn the Main Nitrogen valve "OFF".

2. Remove the interchangeable "L" bracket from the lens assembly and place over the end of the sulfur light pipe in the sample stand. This will prevent any oil from traveling down the pipe and contaminating the sulfur optic lens assembly. If two "L" brackets are installed, flip down the sulfur light pipe bracket and flip up the lens assembly bracket.

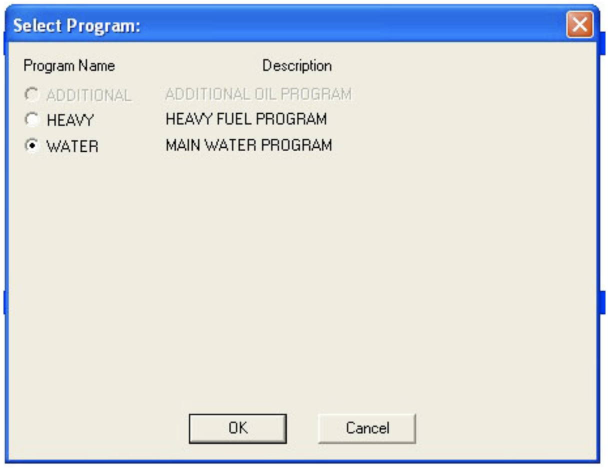
3. Place the Mode/Operate switch on the Spectroil M/F-LD control panel to STAND-BY.

4. Thoroughly clean the sample stand area, especially the quartz window and gap sensors. They will be extremely dirty since the nitrogen was blowing the sample flame towards the left side of the sample stand during the sulfur analysis.

2.10 Water Analysis

This section describes how to set up the Spectroil M/F-LD for the optional elemental analysis of turbine cleaning water and NOx suppression water.

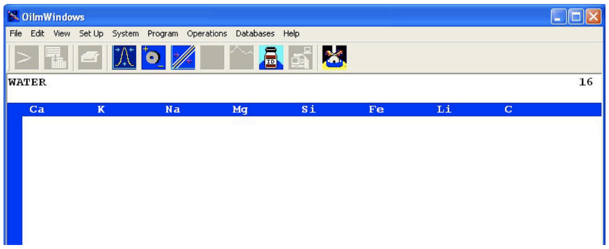
The optional water method is used for the analysis of corrosive elements in water in support of gas turbine operations. The method provides rapid analysis of sodium, lithium, potassium and other elements in water that is introduced into the engine combustor for the purpose of suppressing the oxides of nitrogen (NOx) in the exhaust gases and water used for on-line compressor cleaning and crank-soak compressor cleaning. All the elements are analyzed in less than one minute and without auxiliary gases or the need for sample preparation.



Program Name	Description
○ ADDITIONAL	ADDITIONAL OIL PROGRAM
○ HEAVY	HEAVY FUEL PROGRAM
● WATER	MAIN WATER PROGRAM

Figure 2-24, Select Program Screen

The RDE-AES technique of the Spectroil M/F-LD has been accepted by turbine and fuel treatment system manufactures as the ideal on-site analysis instrument for metallic contaminants in fuel. However, turbine manufacturers actually limit the total concentration of alkali metals ingested by the turbine from all sources. This includes not just fuel, but also contamination in compressor inlet air, steam/water injection for NOx control and cleaning water. A need thus exists for the on-site analysis of alkali metals in water.



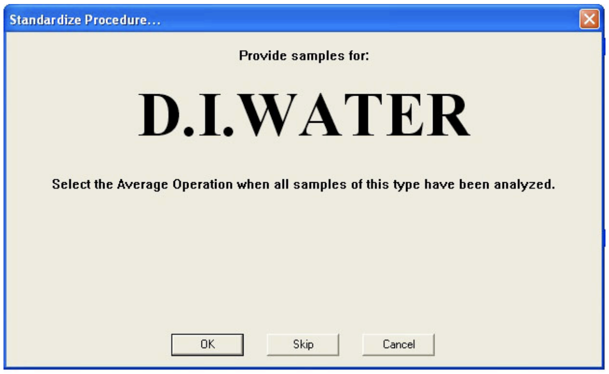
WATER 16

Ca	K	Na	Mg	Si	Fe	Li	C

Figure 2-25, Main Water Analysis Screen

2.10.1 Water Program Standardization

In order to prepare the Spectroil M/F-LD for the analysis of water samples, the software must be in the "WATER" program.



Provide samples for:

D.I.WATER

Select the Average Operation when all samples of this type have been analyzed.

Figure 2-26, First Water Program Standardization Point

1. To load the WATER program, select **File** from the pull down menu options, then **Open**. A list of available programs will be displayed, Figure 2-24. Select the radio button for the WATER (MAIN WATER PROGRAM) and press **OK**. The program is now loaded and ready for the standardization procedure.

2. From the main analysis screen, Figure 2-25, press function key 7 (F7), choose the Standardization icon or select the Operations/ Standardize pull down menu. The software will automatically clear all previous measurements from the video display. A dialog box with the name of the first calibration standard the instrument will expect to measure will appear. A blank water sample is used or this step and the dialog box will call for "D.I. Water, Figure 2-26.

3. Following the procedure set forth in Section 2.3, burn three samples of D.I. Water and press function key 6 (F6), click the Average icon or select Operations/Average from the pull down menu to average them. To make a printout of the measurements and their average, press the print icon. Automatically, the next dialog box will appear providing

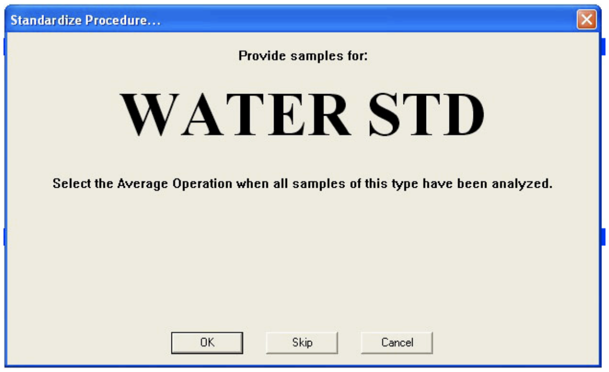
instructions to make measurements of the Water standard, Figure 2-27.

The Water standard is a multi level standard and the included elements and their concentration are stated on its label.

4. Following the procedure set forth in Section 2.3, burn the three samples of the Water standard and press function key 6 (F6), click the Average icon or select Operations/Average from the pull down menu to average them.

4. The next dialog box that appears will indicate standardization is complete and inquire if the average and burns for the last standard measured should be printed, select YES, Figure 2-28.

5. After confirmation that the burn data and average are to be printed, the last dialog box to appear will inquire if the Factors and Offsets should be printed, again select YES. The Standardization procedure is now complete. Review the factors, they should be close to 1, +/- 20 %.

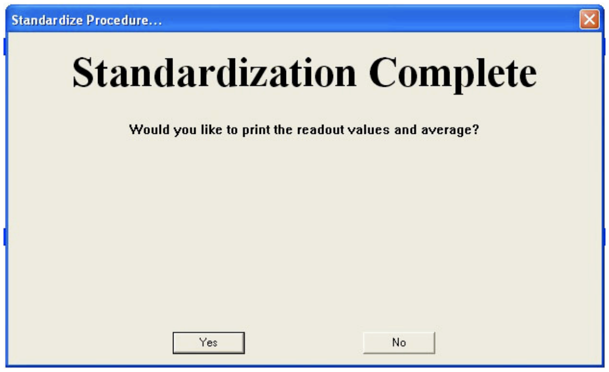


Provide samples for:

WATER STD

Select the Average Operation when all samples of this type have been analyzed.

Figure 2-27, Second Water Program Standardization Point



Standardization Complete

Would you like to print the readout values and average?

Figure 2-28, Standardization Couplet, Dialog

2.10.2 Verification

It is good practice to verify calibration after the Spectroil M/F-LD has been standardized.

1. In PPM mode, run five samples of the Water standard. Print all burns, averages and statistics.

2. Depending on the type of standard analyzed, the averages should be better than +/- 15% for the concentrations listed on the Water standard bottle. If not, repeat the standardization procedure, Section 2.10.1.

2.10.3 Routine Water Sample Analysis

Water samples are analyzed on the Spectroil M/F-LD as described in Section 2.3, Routine Sample Analysis.

3.0 OPERATOR MAINTENANCE

To maintain the Spectroil M/F-LD performance, periodic maintenance must be performed by the operator. This maintenance falls into two categories:

- Daily Maintenance
- Scheduled Maintenance

3.1 Daily Operator Maintenance

This section details the maintenance actions required of the operator on a daily basis. These maintenance actions pertain mainly to the operator accessible assemblies such as the sample stand, the readout and control panel, and the automatic printer. Any maintenance that is required to be performed in the excitation source, optics, or computer electronics is strictly limited to technically skilled personnel.

Table 3-1 lists each maintenance action that is authorized and required of the operator.

3.2 Scheduled Periodic Maintenance

This section of the manual details the maintenance action that is required to be performed on the Spectroil M/F-LD at regularly scheduled intervals. Periodic maintenance will keep the instrument in good working condition and help to identify sources of future trouble before they cause serious downtime. The following tables separate periodic maintenance inspections by subassembly and item. Each item has instructions on what maintenance action is required, the interval in which the maintenance should be performed and by which maintenance level.

The scheduled maintenance tables are as follows:

- Table 3-2, External Housing Inspections
- Table 3-3, Internal Housing Maintenance Inspections
- Table 3-4, Excitation Source and Power Distribution Maintenance Inspections
- Table 3-5, Microprocessor Maintenance Inspections

TABLE 3-1
DAILY OPERATOR MAINTENANCE

CAUTION: Do not use alcohol or Chlorinated Solvents to clean plastic or painted surfaces.

Component	Required Maintenance	Frequency	Maintenance Level
Plate, Mounting, Sample Stand Component	Clean to remove oil and carbon buildup especially between disc electrode shaft and rod electrode holder. refer to Section 1.6.	Every 5 burns	Operator
Window, Quartz, Protective	Clean to remove oil and carbon splashes with isopropyl alcohol or an ammonia based window cleaner. Refer to Section 1.6.3.	Every 5 burns	Operator
Sensors, Sample Stand	Using a Q-tip, clean to remove oil and carbon splashes with isopropyl alcohol or an ammonia based window cleaner. Refer to Figure 1-4.	Daily	Operator
Sample Stand Area	Clean complete sample chamber to remove oil splashes and carbon buildup. Refer to Section 1.6.2.	Twice Daily	Operator

Door, Sample Stand	Clean complete door to remove oil splashes and carbon buildup. Refer to Section 1.6.	Twice Daily	Operator
Electrode Sharpener	Rotate cutting blade to new edge. (Can be performed until all three edges have been used.) Refer to Section 3.3.	As Required	Operator
Panel, Readout and Control	Inspect for oil splashes and carbon residue. If present, remove with mild cleaning detergent.	Daily	Operator
Frame and Exterior Panels	Inspect for oil splashes and dust buildup. If present, remove with mild detergent. CAUTION: DO NOT USE ALCOHOL OR CHLORINATED SOLVENTS TO CLEAN PLASTIC OR PAINTED SURFACES.	Daily	Operator
Printer	Inspect for worn ribbon, loose cable connectors, and dirt and dust buildup. Replace worn ribbon, tighten loose connections and clean accordingly. Refer to printer operation and maintenance manual.	Daily	Operator

TABLE 3-2
EXTERNAL HOUSING MAINTENANCE INSPECTIONS

Component	Required Maintenance	Frequency	Maintenance Level
Filter on Heat Exchanger	Inspect for dust and dirt buildup. Clean in detergent and water bath by swishing vigorously.	Weekly or as required depending on operating environment.	Operator
Filter, Sample Stand Exhaust	Inspect for dust and dirt buildup. Clean or replace if holes in the filter are blocked.	Weekly	Operator
Frame and Exterior Panels	Inspect for oil, dust, dents, scratches and rust. Clean with mild detergent and if necessary, sand and repaint.	Monthly	Operator
Hardware	Inspect for loose or missing hardware. Tighten loose hardware and replace rusted hardware.	Monthly	Operator
External Cables	Inspect for loose connections. Inspect for damage.	Monthly	Operator
Shaft, Disc Electrode	Clean residue (varnish) from splined end with an ink eraser.	Monthly	Operator

TABLE 3-3
INTERNAL HOUSING MAINTENANCE INSPECTIONS

Component	Required Maintenance	Frequency	Maintenance Level
Fans	Inspect for smooth rotation. Check for dust and dirt buildup on blades. Replace if binding is evident. Clean blades if necessary. Frequency - Six months	Six Months	Operator
Wiring	Inspect for broken or bent wiring connections. Inspect for frayed or burned insulation.	Six Months	Operator
Fuses	Inspect for open or over rated fuse usage. Replace as required.	Six Months or as Required	Operator
Transformers	Inspect for good electrical connection and signs of overheating.	Six Months or as Required	Operator
Video Monitor	Inspect for dust and dirt buildup.With a soft cloth, wipe clean if necessary.	Six Months	Operator
Signal Connectors	Inspect all connectors for proper seating in sockets. *CAUTION: Do not remove or connect any signal cables with the power on.*	Six Months	Operator

TABLE 3-4
EXCITATION SOURCE AND POWER DISTRIBUTION MAINTENANCE INSPECTION

Component	Required Maintenance	Frequency	Maintenance Level
Component Mounting Boards	Inspect the board for proper connector seating. Inspect for burn marks or discolored components. Inspect lower cabinet for dust and dirt buildup. Vacuum if necessary.	Six Months or as Required	Operator/Technician
Capacitors	Check each capacitor for signs of bulging, discolored containers or signs of leaking. Replace if signs of overheating are evident.	Six Months or as Required	Operator/Technician
Resistors	Check each resistor for signs of bulging or discoloration. Replace if overheating is evident.	Six Months or as Required	Operator/Technician
Contactors	Inspect for good electrical connection. Observe relay operation. Replace if intermittent or sluggish.	Six Months or as Required	Operator/Technician
Transformers	Inspect for signs of arc-over and overheating. Replace if evident.	Six Months	Operator/Technician

Analytical Gap	Inspect the rod electrode holder and gap setting device for smooth sliding and release. If tight or binding, adjust or remove and replace gap setting device, refer to the Maintenance Manual Section 2.4.1.1. Check the analytical gap distance. It should be 0.090 inches.	Six Months or Every 2,000 Burns	Operator/Technician
Auxiliary Gap	Polish tips to remove corrosion.	Six Months or Every 2,000 Burns	Operator/Technician
Auxiliary Gap	Check electrode shape. If electrode points are flat, remove electrodes and replace them. At sea level, reset the auxiliary gap distance to approximately 0.135 inches. Verify distance by checking excitation source frequency with Source Frequency Test Meter (refer to the Maintenance Manual Section 2.2.)	Six Months or Every 2,000 Burns	Operator/Technician
Motor, Disc Electrode	Check motor rotation. If loose or binding, align and tighten.	Six Months	Operator/Technician
Cables	Check high voltage cables in the excitation source for signs of arc-over or damage. Check electrical connections. Replace cables if arc-over is evident. Tighten connections if loose.	Six Months	Operator/Technician
Shaft, Disc Electrode	Check to be sure that the disc electrode shaft is tight and properly aligned. See the Maintenance Manual Section 2.4.1.2 for replacement procedure and tracking check.	Six Months	Operator/Technician
Disc Electrode to Rod Electrode Alignment	Check the alignment. If the rod point is more than 25% away from disc center, adjust. See the Maintenance Manual Section 2.4.1.3	Six Months	Operator/Technician
Auxiliary Gap Fan	Check for smooth rotation and cleanliness. Clean if necessary.	Six Months or Every 2,000 Burns	Operator/Technician

TABLE 3-5
MICROPROCESSOR MAINTENANCE INSPECTIONS

Component	Required Maintenance	Frequency	Maintenance Level
Printed Circuit Assemblies	Inspect each card for signs of discoloration due to component overheating. If present, replace the appropriate card. Check each card for proper connector seating. Re-seat if necessary.	Six Months	Operator/Technician
Optical Fibers	Inspect each fiber optic in the M58000 and M59200 cards for a tight mounting. If loose, remove the fiber and reseat. If broken, replace. Frequency - Six months	Six Months	Operator/Technician
Cables, Interconnecting	Check both connectors of each interconnecting cable. If loose, Re-seat the connector in the appropriate plug. If broken or damaged, replace. CAUTION: Do not remove or connect any signal cables with the power on.	Six Months	Operator/Technician

3.3 Procedure to Replace Electrode Sharpener Cutter Blade, M90102

The cutter blade has three sharpened edges and can thus be used three times before it is replaced.

To replace or rotate the cutter blade to a new cutting edge, unplug the sharpener power connector J2 at the power connection plate. Next, remove the graphite collector barrel assembly to empty out any accumulated graphite and to expose the cutter blade, Figure 3-1.

To remove the barrel assembly, locate the sharpener over a waste basket with the collector barrel pointing downward. Grasp the barrel with the opposite hand and rotate it while pulling it away from the motor mount and face plate. Once the O-ring disengages the face plate, it will be easy to separate and empty. Use a flat blade screw driver to remove the #4-40 screw which mounts the cutter blade, see Figure 2-14. When replacing or rotating the cutter blade, be sure to place the rear edge of the cutter blade tight against the cutter head. This is the reference point to achieve the correct angle on the graphite rod electrode. Replace the barrel assembly and reconnect the sharpener to its power connector.

Figure 3-1, Electrode Sharpener Blade

4.0 PERFORMING CALIBRATION CURVE VERIFICATION

The purpose of performing a calibration curve verification is to determine if the instrument repeats the curve generated at the factory or by an authorized service representative.

To perform a calibration curve verification, the instrument must first be standardized. Refer to Section 2.5 in this manual for a detailed procedure on daily standardization. When the instrument has been standardized using the calibration standards for the new program, the calibration curve verification can be performed.

The calibration curve verification consists of performing an analysis of each synthesized standard as if it were an unknown sample. It is recommended that the operator conduct ten analyses of each standard and perform statistics on the measurements to obtain the average and standard deviation for each element. Instrument performance for alkali metal analysis should be within the limits listed in Table 4-1.

4.1 Repeatability Testing

Perhaps one of the most important technical characteristics of a spectrometer is its ability to perform the same measurement over and over again with the same result. This characteristic is referred to as repeatability, reproducibility, sigma, standard deviation or precision. Repeatability is determined by the standard deviation of a series of measurements made on the same sample. Mathematically, standard deviation is calculated as:

$$\text{Std. Dev.}=\sqrt{[N(\sum Xi2) - (\sum Xi)2] / [N(N-1)]}$$

where:
N = the number of analyses (normally 10)

∑Xi2 = is the sum of the 10 squared individual measurements

$(\sum X_i)^2$ = is the square of the sum of the 10 individual measurements.

For the purpose of conveniently comparing the standard deviation to the mean for a series of measurements, relative standard deviation, or R.S.D., is used. R.S.D. expresses the standard deviation as a percent of the mean and is calculated as:

$$R.S.D. = 100[\text{Std. Dev.}/X_{avg}]$$

The Spectroil M/F-LD automatically calculates mean, standard deviation and R.S.D. for a series of measurements by pressing function key 5 (F5), see Section 5.4.8.9 for a description of this function.

NOTE: At 0 ppm, R.S.D. is not considered a valid statistic. A minimum of three warm-up burns must be made prior to the performance of statistical analysis.

4.2 Accuracy

Accuracy is the ability of a spectrometer to give the correct concentration value of a standard. Accuracy requirements for fuel analysis are tighter than those for wear metals. Even extremely low concentrations of contaminants such as sodium, potassium or lithium in the fuel can have detrimental affects on a gas turbine. Typical performance requirements for the Spectroil M/F-LD for fuel analysis are shown in Table 4-1.

The average of ten burns should be used to determine the reading of a particular standard. Accuracy failures at low concentrations may be due to contamination and spot impurities in the disc electrodes. The effect of the contamination and impurities can be compensated for by performing the electrode offset procedure in Section 4.4. Spectro Incorporated Field Service should be consulted if the Spectroil M/F-LD is unable to meet the accuracy criteria presented in Tables 4-1.

Table 4-1, Acceptable Accuracy for Alkali Metals

Na, K and Li Accuracy Table	
0.0 to 1.0 ppm	+/- 10%
1.1 to 20 ppm	+/- 10%
>21 ppm	+/- 15%

4.3 Factors Affecting Repeatability and Accuracy

Many factors affect repeatability and accuracy on actual fuel sample. Among them are:

1. The sample must be homogenous. The accuracy test done at Spectro is always done with standards. Routine samples are never used for repeatability and accuracy testing because it cannot be assured they do not contain agglomerates, second phases and large particulates, all of which will affect repeatability and accuracy.

2. The Spectroil M/F-LD must be on profile. If analytical lines are off profile, the repeatability will be adversely affected. If the repeatability specifications cannot be met, one of the first diagnostic tests is to check profile (Section 2.7).

3. The quality and handling of the disc and rod electrodes will affect repeatability and accuracy. The density, and hence the porosity, residual contamination and dimensional accuracy of the disc and rod electrodes will affect repeatability. Care must be taken to properly sharpen the rod electrode (Section 1.2). Proper care must also be exercised when installing the electrodes (Sections 1.3 and 1.4).

4. The sample must be homogenized by shaking before filling the sample holders.

5. The sample holders must be filled to the same level (Section 1.5).

6. Line voltage to the Spectroil M/F-LD must

be within specification.

7. Electronic stability of the Spectroil M/F-LD will affect repeatability.

8. Sample stand geometry will affect repeatability. The rod electrode to disc electrode gap distance, the quartz lens assembly to arc distance, the position of the fiber optic within the lens assembly mounting block, and the angle of the quartz lens assembly with respect to the arc will affect not only the intensity of the light entering the entrance slit of the polychromator (optical assembly), but will also affect the repeatability . These adjustments to the Spectroil M/F-LD are optimized at the factory.

9. A variety of mechanical or electronic faults could degrade repeatability and accuracy. Among these are faulty CCD chips, damage to the entrance slit, or damage to the fiber optic cable.

The operator has control over the first five factors. If care is taken to properly operate the Spectroil M/F-LD and accuracy is still worse than specification and it is on profile, then it is recommended that Spectro Incorporated Field Service be consulted. It is strongly recommended that adjustments to the sample stand as described in item 8 above be made only by Spectro personnel or at the direction of Spectro personnel.

4.4 Disc Electrode Offset Procedure

The Spectroil M/F-LD is designed to incorporate a background measurement and correction system. The purpose of this system is to offset or null the output of all element CCD's when measuring a HP Base standard. This is also known as measuring background light because 0 ppm has no concentration of elements present in the sample. Therefore, the light produced when analyzing a 0 ppm standard must only be background emission. This is, however, only true in theory.

In practice, elemental contamination is present in everything used for the analysis process. The sample holders may pick up contamination from the environment, the HP Base standard may have sub-ppm trace levels of certain elements, and the graphite disc electrodes are known to have trace contamination of certain elements. Manufacturers of graphite electrodes commonly list and quantify the known trace or spot impurities on each box of disc electrodes. The purpose of this procedure is to offset these trace contaminates in the consumables and is absolutely necessary to be performed.

This procedure should be performed every time a new batch and/or lot number of disc electrodes are to be used. For maximum efficiency in a laboratory operation, all graphite disc electrodes should be grouped and stored by batch and lot number. Only one batch or lot should be used at a time until it is totally consumed. Once a new lot is opened and the instrument is standardized to the new lot, the low end of the calibration curve (5 ppm and/or 10 ppm) should always be checked. If accuracy at these levels fails to meet the specified criteria, it may be due to variance in trace contaminants levels and the following procedure should be performed to correct for the presence of this contamination.

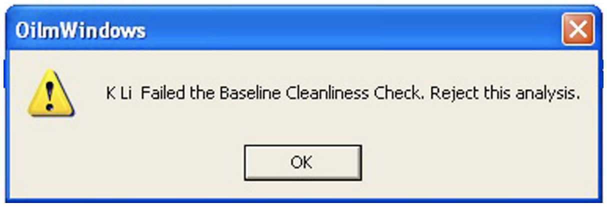
1. To perform the disc electrode offsets operation, the operator can select function key 10 (F10), left click the disc electrode offsets icon or choose Operations/Offsets/Perform Disc Offsets from the pull down menu options.

NOTE: The software has now been placed in a mode that is not to be used for normal operation. "Disc Electrode Offset" appears highlighted in red across the top of the analysis program screen to draw attention to the fact that this is not a normal condition for operation. Once this procedure is completed, the readout mode will have to be restored to the instrument's normal operating condition.

2. Prepare five sample holders of the HP Base oil standard. The screen will automatically clear any previous measurements when this mode is selected. Using the new batch or lot of disc electrodes, burn these five samples in accordance with the routine operating procedures outlined in Section 2.3.

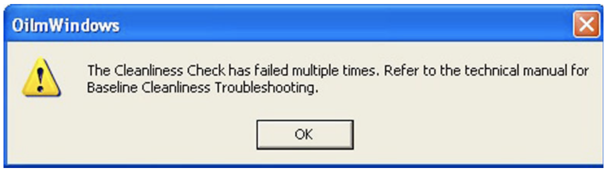
The Spectroil M/F-LD software also contains a ”Cleanliness Check” feature that monitors the analyses of the HP Base standard during standardization and the disc electrode offset procedure. When the set cleanliness value for sodium, potassium or lithium is overridden, an error message such as the one shown in Figure 4-1 appears. The error message may be due to sporadic contamination or a minor operator error and the element(s) that failed also appear in red on the analysis screen. Follow the instructions and reject the analysis. If 3 of the 5 analyses fail the cleanliness check, there may be a more significant problem. The screen shown in Figure 4-2 appears and Base Cleanliness Troubleshooting must be implemented, see Section 4.5.

To reject one of the measurements from the video display, move the selection pointer over any portion of the measurement and left click the mouse one time. This will highlight the burn in a black background. Pressing the DELETE key on the keyboard one time will remove this measurement off of the screen. In the event that the wrong measurement has been highlighted, position the pointer over the measurement again and left click the mouse a second time. This will remove the highlight, then select the proper measurement to reject and press the DELETE key.



K Li Failed the Baseline Cleanliness Check. Reject this analysis.

Figure 4-1, Error Message for Failed Cleanliness Check Baseline



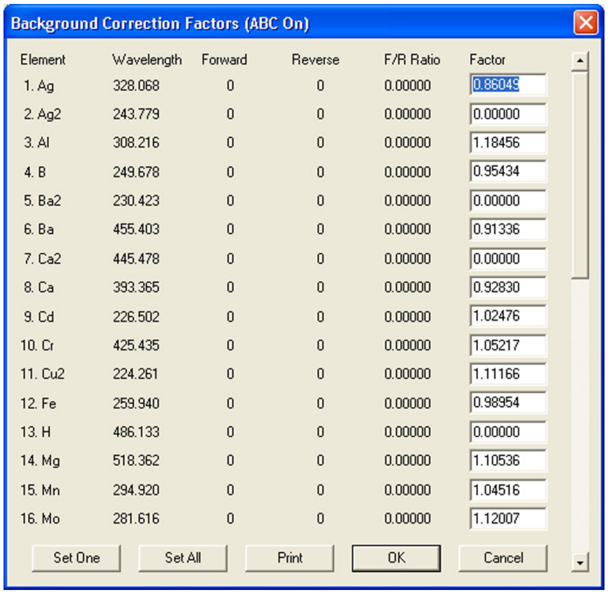
The Cleanliness Check has failed multiple times. Refer to the technical manual for Baseline Cleanliness Troubleshooting.

Figure 4-2, Error Message for Multiple Analysis Failing Cleanliness Check Baseline

3. After all five samples have been burned, press function key 6 (F6), the average icon, or Operations/Average to calculate the average of the five measurements. Once the average is calculated, a dialog with the title Background Correction Factors will appear on the screen. This table will vary from instrument depending on the analytical configuration of the spectrometer; however, the format is the same as shown in Figure 4-3.

4. This dialog will display (from the left) ELEMENT in the second column, WAVELENGTH in the third, FORWARD intensity in the fourth, REVERSE intensity in the fifth, F/R RATIO in the sixth and BKG FACTOR in the last column. The cursor will appear in the upper right corner of the screen under the column BKG FACTOR. The absolute value for this mode is 1.00000 which indicates that the intensity produced in the forward or peak measurement is identical to the intensity produced in the reverse or background measurement.

5. The purpose of this procedure is to set the



Element	Wavelength	Forward	Reverse	F/R Ratio	Factor
1. Ag	328.068	0	0	0.00000	0.86049
2. Ag2	243.779	0	0	0.00000	0.00000
3. Al	308.216	0	0	0.00000	1.18456
4. B	249.678	0	0	0.00000	0.95434
5. Ba2	230.423	0	0	0.00000	0.00000
6. Ba	455.403	0	0	0.00000	0.91336
7. Ca2	445.478	0	0	0.00000	0.00000
8. Ca	393.365	0	0	0.00000	0.92830
9. Cd	226.502	0	0	0.00000	1.02476
10. Cr	425.435	0	0	0.00000	1.05217
11. Cu2	224.261	0	0	0.00000	1.11166
12. Fe	259.940	0	0	0.00000	0.98954
13. H	486.133	0	0	0.00000	0.00000
14. Mg	518.362	0	0	0.00000	1.10536
15. Mn	294.920	0	0	0.00000	1.04516
16. Mo	281.616	0	0	0.00000	1.12007

Figure 4-3,Sample Background Correction Factors new ratio calculated and shown on the F/R RATIO column into the BKG FACTOR column for most elements.

NOTE: It is extremely important to pay attention to which elements this ratio is applied. Not all elements have a background factor as indicated by the value 0.00000. For the elements specified below, do not set the F/R RATIO value in the BKG FACTOR column. Failure to do so will adversely affect the analytical results of the instrument!*

*Spectroil M/F-LD - NaHi 568.861, P 510.656, Ca 445.478, and
H 486.133

*Spectroil M/F-LD/C - NaHi 568.861, P 510.656, Ca 445.478, MgHi 518.36, Ba 230.48, ZnHi 481.05 and H 486.133 and C 387.10.

*Spectroil M/F-LD/F - MgHi 518.36, C 387.10 and H 486.133.

6. The cursor will automatically be located in the first row of the background factor column. To set the new F/R RATIO for all elements, left click the Set All button. Automatically, the new factors calculated and displayed in the F/R RATIO column will appear in the background factor column.

7. Once all of the elements that have a background factor have been updated to the new F/R RATIO, a copy of this screen may be made for your records by left clicking the Print button. Keep this printout for future reference. Left click the OK button to exit this dialog.

8. To exit the disc electrode offset procedure, press function key 10 (F10), left click the icon, or select Operations/Offsets/Perform Disc Offsets from the pull down menu options. This will place the software back to the Analysis Program screen which is the normal mode for operation and the mode the instrument was in before initiating this procedure.

9. This concludes the disc electrode offset procedure. Standardize the instrument for normal operation in accordance with Section 2.5 of this manual. Perform a daily standardization check in accordance with Section 2.4 to confirm that all elements at the lower concentrations meet the accuracy criteria. If any element fails to meet this criteria, contact Spectro Incorporated Field Service for assistance.

4.5 BaseLine Cleanliness Troubleshooting

The base line cleanliness troubleshooting procedure must be implemented whenever there are three cleanliness errors either in the HP Base standardization procedure or the disc electrode offset procedure. The failures can be due to one or more influencing factors and this procedure will help to eliminate them.

An occasional operator error will result in one bad analysis which can be rejected. If, however, there are three bad analyses in a set of five, the problem is most likely due to contamination in the sample stand or the consumables.

The base line cleanliness troubleshooting procedure must be implemented when an error message as shown in Figures 2-7 and 2-8 or 4-1 and 4-2 appears during standardization or the disc electrode offset procedure. Follow the step below in order to eliminate the source of contamination.

1. Clean the sample stand, Section 1.6.

2. Empty and clean the electrode sharpener, Section 1.2.

3. Obtain new consumables that have not been handled and are still in their container. This includes:
   - Disc electrodes
   - Rod electrodes
   - Sample vessels

4. Perform a profile, Section 2.7.

5. Perform the disc electrode offset procedure, Section 4.4

6. Perform a complete standardization, Section2.5.

If steps 5 and 6 are performed successfully and without failing the cleanliness check, the problem(s) has been solved and the instrument is ready to analyze routine samples.

If, however, the cleanliness check fails again during the standardization or disc electrode offset procedure, service support may be required. Contact your local agent or Spectro Incorporated and please have your data available to expedite any necessary troubleshooting.

5.0 BACKUP OF COMPLETE OilM SOFTWARE

These steps will take the user through the process of backing up OilM for Windows on Microsoft® Windows XP.

1. While running OilMWindows open the File menu and select Exit. OilmWindows will shutdown.

2. Reopen OilMWindows by selecting from the Start menu or the shortcut on the desktop. After the communications are up select Operations/User Functions menu, Figure 5-1.

3. If user functions have been accessed, the window shown in Figure 5-4 open. Proceed with step 7. If no user functions have been previously accessed, the window as shown in Figure 5-2 will open. Proceed with step 4.

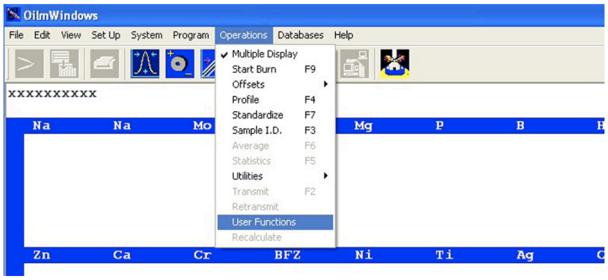
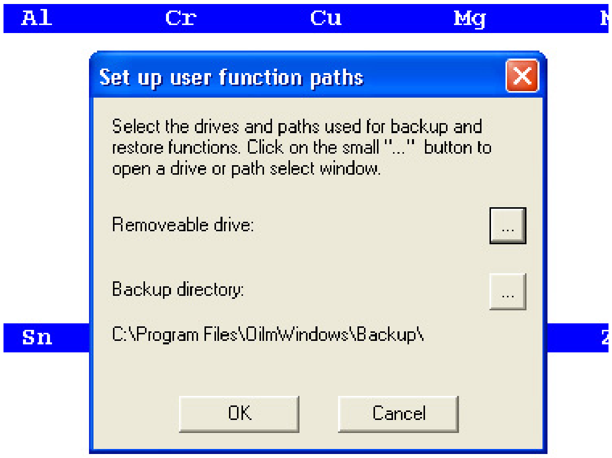


Figure 5-1, Operations/User Functions Menu



Select the drives and paths used for backup and restore functions. Click on the small "..." button to open a drive or path select window.

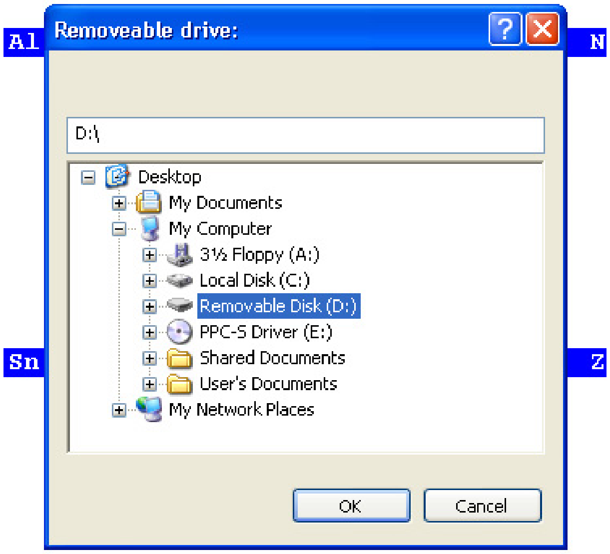
Backup directory: C:\Program Files\OilmWindows\Backup\

Figure 5-2, Selection of User Function Menu

4. In the "Set up user functions paths" window click on the square button to the right of Removable drive:.

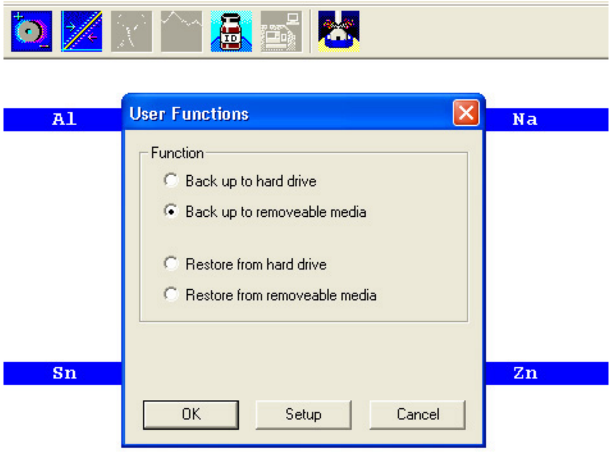
5. The "Removable Drive:" window will open, Figure 5-3. Select the drive that says "Removable Disc (D:)". With the disc drive highlighted click on the OK button.

6. The "Set up user functions paths" window, Figure 5-2, reopens. Click on the OK button to continue.



D:\

Figure 5-3, Set-up User Functions Paths Screen



User Functions

Function

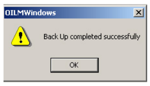
○ Back up to hard drive
● Back up to removeable media
○ Restore from hard drive
○ Restore from removeable media

Figure 5-4, Removable Drive Selection Screen

7. The “User Functions” window, Figure 5-4 opens. Select the “Backup to Removable Media” option. Click the OK button to continue.

8. OilMWindows will start a transfer of all OilM Windows and program files to the removable media.

9. When all the files are copied the “Backup completed successfully” message box will open. Click on the OK button to complete the back up procedure.



OILMWindows

Back Up completed successfully

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