

MARTEK INSTRUMENTS, INC.



# **IN SITU CALIBRATOR**

# **OPERATION MANUAL**

Rev. NC(1) - 10/04

Unless otherwise stated, MARTEK INSTRUMENTS, INC. warrants this equipment to be free from defects in material and workmanship and to perform in accordance with applicable specifications for one year from date of shipment.

MARTEK will provide free service at the factory, including parts, labor and transportation back to the customer, for any malfunction of its products, which are returned transportation charges prepaid.

Customers desiring to return a product to MARTEK for repair should contact the Service Department by telephone (919) 790-2371 or fax (919) 790-2375 to obtain return authorization. The information required at this time will be the complete model number and serial number of the product and a brief description of the problem.

All shipments to MARTEK must be freight prepaid and addressed as follows:

MARTEK INSTRUMENTS, INC. 5201 Old Poole Rd. Raleigh, NC 27610 Attn.: Repair Department

A complete and detailed statement of the reason for return must accompany the unit. If possible, include a copy of sample reading or a printout.

Returned units must be packed as well as they were when first shipped. If possible, use the original packing. Do not return detachable cords or manuals with the unit.

MARTEK reserves the right to void this warranty if the product has been subjected to misuse, neglect, accident, improper installation or application, and for consumable items such as batteries, membranes, or solutions.

This warranty is expressly in lieu of all other obligations or liabilities on the part of MARTEK. MARTEK neither assumes nor authorizes any other person or organization to assume on behalf of MARTEK any other liability in connection with the sales of MARTEK instrumentation.

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## 1.0 WHAT IS A CALIBRATOR?

The Martek Mark 22 In-Situ Calibrator is a completely portable, battery-operated electronic instrument that can be used to validate the performance and accuracy of online, process water monitoring instruments. The In -Situ Calibrator is designed for use in applications in which the measurement of water properties such as temperature, conductivity, temperature-corrected conductivity, and pH are required.

The Mark 22 can be calibrated in the laboratory to ASTM (American Society for Testing of Materials) or NIST (National Institute of Standards and Testing) guidelines and procedures using any manufacturer's conductivity reference cell or pH electrode. The portability of the unit allows it to then be transferred to the sample stream for direct, on-site monitoring of the water properties used to determine water quality.

## 1.1 How Can The Mark 22 Benefit You?

By eliminating the need for physical grab samples, the Mark 22 provides a more reliable and efficient method for validating and/or calibrating on-line instrumentation. Its accuracy and resolution allow the operator to detect subtle changes in water chemistry that can affect the turbine steam quality , the performance of condensate polishers, resin beds, and other process systems.

In addition, the Mark 22 features battery

backed up NVM memory in which monitored data can be stored and retrieved later by a personal computer via the units serial port for storage, review and analysis. As an alternate recording method, a serial data input printer can be connected to the serial port of the Mark 22 in applications in which a hardcopy record of the data stored in the Mark 22 In-Situ Calibrator is required. This feature allows the operator to create and maintain a historical record of calibration and performance data for on-line instruments. The Mark 22 can also serve as a back-up monitoring system in the event of on-line equipment failure. A 0-1 Volt analog output signal for all parameters is also available to facilitate the interface of the Mark 22 directly to a paper strip-chart recorder.

## 1.2 How To Read This Manual

Carefully review the material in this manual before operating the Mark 22. The OVERVIEW in Chapter 2 provides brief descriptions of the features of the calibrator and an example of a quick calibration procedure for conductivity. Chapter 3 reviews the specifications for the instrument and gives a detailed description of how to set up the unit. The complete operational instructions are outlined in Chapter 4, and Chapter 5 details the calibration procedure. The MAINTE-NANCE & TROUBLESHOOTING chapter provides routine maintenance requirements for the Mark 22 and its probes.

Should you have any questions or comments regarding the Mark 22, its sensors, or this Manual, please contact the factory.

## 2.0 OPERATIONAL OVERVIEW

A short description of the different features of the Mark 22 is given below. Abbreviated instructions are provided for review of the connections of the conductivity, temperature, and pH sensors and the battery charger. A description of the operational features provided by the EIGHT keys on the front panel is also presented. For more detailed information on operation and calibration procedures, refer to the appropriate chapters within the Manual.

- Connect the Martek conductivity reference sensor to the connectors labeled "COND" and "TEMP" on the side of the Mark 22 readout module.
- Connect the pH sensor to the connector labeled "pH" on the side of the Mark 22 readout module. The temperature from the Martek conductivity reference sensor also provides the temperature for compensation of the pH measurement. If the Martek conductivity reference sensor is not used, you must use the separate temperature sensor located on the pH plug (P/N 180-51A) or the dip-style temperature sensor (P/N 182-24T).

NOTE: If the pH sensor is not being used, the shorting plug on the Mark 22 readout module must be connected to the pH input connector. This plug connects the input to the pH circuit to analog ground allowing ONLY conductivity and temperature measurements to be made.

To charge the internal batteries, (a) connect the circular plug from the AC battery charger to the Mark 22 and (b) connect the battery charger to an 120 V / 60 Hz AC power outlet.

CAUTION: Make sure the power switch is OFF when connecting or disconnecting the battery charger. The power switch can be on or off when charging the internal batteries.

- 4. After all sensor and power connections have been made, the Mark 22 can be operated. Flip the switch labeled POWER to the UP position ( power on ). Flip the switch labeled LAMP to the UP ( backlight on ) to illuminate the Mark 22's display. The Back Lighting Lamp in the Mark 22 display consumes considerable power, and as a result will limit the unit's battery powered operating time to approximately FOUR hours. With the Lamp OFF, the Mark 22 will provide reliable operation for approximately 12 hours or more.
- 5. The Mark 22 sequentially displays date, time and battery voltage; corrected conductivity, raw conductivity and temperature; and solution compensated pH, pH and temperature. Depress the SELECT key and observe that the letter "M" appears in the lower right corner of the Mark 22 display. The "M" indicates that Manual Mode is in operation. The Mark 22 will then continuously display the currently displayed parameter. Depress the SELECT key again, and the displayed data advances to the next set of parameters.

Depress the EXIT key to exit the Manual Mode. Observe that the letter "A" appears in the lower right corner of the units display. The letter "A" indicates that the Manual Mode has been exited and that the unit is now in the Auto Mode of operation. When in the Auto Mode the Mark 22 will continuously scan through all of its measured as well as manually entered parameters.

6. The FOUR MODE KEYS along the bottom row of the front panel (SETUP, CELL, LOG, and PRINT) refer to the different modes of operation featured by the Mark 22. The four tri-legened FUNCTION keys along the top row of the front panel are used differently during each mode. To facilitate operation of the Mark 22 the function legends and the associated mode have the same color legend and border.

In the Normal Display Monitoring Mode the top row of keys with the WHITE borders are function keys. The functions provided by these keys are identified by the WHITE key legends located just above the top row of function keys. The READ key will hold the display in Manual Mode. The RANGE keys will change the range for the conductivity display, and the AUTO key will restart the automatic display monitoring mode.

In the Setup Mode the top row of keys are used as function keys. The functions provided by these keys are as indicated by the YELLOW key legends located on each of the function keys. The SELECT key is used to select options from the dis-The UP ARROW key played menu. moves the blinking display down through the menus and increments the numeric digits when adjustment of the displayed numeric value is required. The DOWN ARROW key moves the blinking display up through the menus and decrements the numeric digits when adjustment of the displayed numeric value is required. The EXIT key allows the user to move backwards through the displayed menus.

In the Cell Mode, the top row of keys function as indicated by the BLUE key legend located on each of the top row of function keys. The K 0.01, K 0.1, K 1 and the K 10 keys are used to select the cell constant stored for Cell #1, Cell #2, Cell #3 and Cell #4 respectively.

In the Log Mode, the top row of keys function as indicated by the RED key legend located on each of the top row of keys. To log data, depress the LOG key once. The Mark 22 will display a file number. Depress the LOG key again, and observe that a black square will appear in the upper right corner of the display. This indicates that the Mark 22 is now in the Log Mode of operation. Depress the LOG key again, and observe that the black square is no longer present in the display. This indicates that the Mark 22 Log Mode has been exited. The FREE will display the amount of storage memory remaining in terms of storage time( hours ). When in the Log Mode, pressing the PAUSE key will cause a letter "P" to appear in the right corner of the display. The Mark 22 will stop logging but remain in the Log Mode. This procedure is used to conserve memory. Depress the RESUME function key to resume logging data. Finally, The CLEAR function key will clear all of the logged data from memory.

7. Depression of the yellow SETUP key enables the setup mode of operation and subsequently allows the operator access to the unit's setup functions. The Setup functions provided by the Mark 22 are as follows:

CAL (Calibration) option is used for establishment of the zero and span calibration points associated with temperature, conductivity and pH measurements as well as manual entry of the date and time of day parameters.

COMP (Compensation) option provides the selection of three temperature corrected conductivity equations: GE, Marsh & Stokes, and Truman Light in addition to the slope coefficients for conductivity and pH. This function also allows calibration of 0 - 1 Volt external recorder output.

CELL option allows the operator to manually input a specific cell constant for each of the four nominal cell constants. For ultrapure water, the nominal cell constant is 0.01.

The LOG option allows the operator to manually enter the start time to begin logging data, the logging interval (the time in minutes between scans), and the number of scans.

8. The PRINT Mode allows printing of the various data files by file number. To print or record data from the Mark 22's internal memory the unit must be connected via its serial port to computer or printer which has a serial input interface. This interface must be configured with the same baud rate, # of data bits, stop bits, and parity type configured inside the Mark 22 for serial communications. Refer to the section 4.2.4 of this Manual for more detailed information regarding the print function.

## 2.1 "QUICK-CAL" CALIBRATION PROCEDURE

This section provides an easy reference or "quick-cal" procedure for electronically calibrating the Mark 22 for conductivity measurements. However, before performing a calibration of the Mark 22 using this procedure, it is recommended that you review Chapter 5 ( CALIBRATION ) of this manual.

Calibration procedures for all parameters are located in the CALIBRATION section of this Manual.

- Select a cell number for calibration. Depress the CELL key and select a cell number 1 4. (Select a K factor of .01000 for ultrapure process water applications).
- 2. Place the Mark 22 in Manual Display Mode for the conductivity parameter.
- 3. Using the range keys (UP and DOWN ARROWS), set the decimal point to read the highest range (XXX.XX  $\mu$ S/cm).
- 4. Select the SETUP Mode of operation by depressing the SETUP key. From the setup menu select the CAL option. Select COND option from the CAL menu menu as the type of parameter to be calibrated. Select ZERO as the type of cali-

bration to be performed. Connect the zero calibration plug (P/N 400550) to the test leads. The voltage should be approximately -1.000 Volt. Enter the zero value for conductivity by pressing the SELECT key and entering 000.00  $\mu$ S/cm.

- Exit the set ZERO point function by depressing the EXIT key. Using the range keys (UP and DOWN ARROWS), set the displayed measured value to the lowest range (0.0000 μS/cm).
- 6. Select the SETUP Mode of operation by depressing the SETUP key. From the setup menu select the CAL option. Select COND from the displayed menu as the parameter to be calibrated. Select SPAN as the type of cali-bration to be performed. Remove the zero calibration plug and make sure the span calibration plug (P/N 400549) has been placed between the test leads. The voltage should be approximately +0.5000 Volts. Enter the span value for conductivity by pressing the SE-LECT key and entering 1.0000 µS/cm.
- 7. At this point, the Mark 22 is now calibrated for conductivity for any sensor utilizing a cell constant of 0.01. But before measurements can be taken, depress the CELL key and select a cell with the same cell constant as the specific conductivity reference sensor. Depress the CELL key again to return the Mark 22 to the manual display mode.

#### NOTES:

 If not using a Martek Conductivity Reference Cell, the temperature sensor must be connected.
Temperature must always be calibrated before conductivity and pH.

3. Use a 1K Ohm, .5% resistor for 10 mS/cm range. 4. Use a 100 Ohm, .5% resistor for 100 mS/cm range.

## 3.0 Specifications - Measurement

<u>Parameter</u>	<u>Range</u>	<u>Resolution</u>	<u>Accuracy</u>	
Temperature	0-100°C	±0.01° C	±0.1° C	
Conductivity*	0-1.0000 µS/cm	±0.0001 µS/cm	±0.001 µS/cm	
	0-10.000 μS/cm 0-100.00 μS/cm	±0.001 μS/cm ±0.01 μS/cm	±0.01 μS/cm ±0.1 μS/cm	
	0-1.0000 mS/cm	±0.0001 mS/cm	±0.001 mS/cm	
	0-10.000 mS/cm 0-100.00 mS/cm	±0.001 mS/cm ±0.01 mS/cm	±0.01 mS/cm ±0.1 mS/cm	
рН	2-12 pH	±0.01 pH	±0.1 pH	
* The Conductivity measurement accuracy is dependent on entry of the appro- priate cell constant for the indicated range of measurement and on the quality of the electrode used.				

## 3.1 Specifications - Physical

Case Dimensions:		Recorder Output:	
Front Panel -	7.0 x 6.5 x 4.0 inch (17.8 x 16.5 x 10.1 cm) L W H		RS 232C Serial ASCII or 0-1 volt DC analog output.
<u>Case Weight:</u>	6 lbs. (2.1 kg)		
<u>Power Requiremen</u>	<u>nt:</u>		
	6 Volt DC power		
	120 or 230 Volt AC when the factory supplied battery charger is used.		

### 3.1 Accessories and Sensors

The Mark 22 In Situ Calibrator comes complete from the factory with the following accessories:



# **SPECIFICATIONS AND SETUP - 3**

In addition, the following sensors and accessories may also be purchased from Martek for use with the Mark 22:







Conductivity Reference Cell (P/N 182-21, 0-50uS/ cm or P/N 182-22, 0-1000uS/cm)

pH Sensor (P/N 182-24S)

Temperature Sensor (P/N 182-24T)



Stainless Steel Flow Chamber (P/N 180-51)



pH Adapter Plug w/ Temp. Cable (P/N 180-51A)

# **3 - SPECIFICATIONS AND SETUP**

## 3.3 SETTING UP THE MARK 22

There are four circular connectors and a battery charger input jack located on the left side of the Mark 22 Readout Module. See Figure 3.1 below for connector location.





Each sensor input connector is clearly marked with the type of sensor that should be connected to it. The connector labeled REC is for the recorder cable. Below the REC connector is the battery charger input jack plug.

#### 3.3.1 Connecting the Conductivity Sensor

To connect the Martek conductivity reference cell, remove the dust caps from the connectors labeled TEMP and COND and connect the appropriately marked cables of the reference cell to the Mark 22. Make sure the correct cable is inserted into the correct connector ( the cable identification labels are located just below the reference cell plugs ).

To connect any other conductivity reference cell, connect the Conductivity Interconnect Cable to the Mark 22 connector marked COND. Then connect the leads from the reference cell to the red and black banana plugs according to the manufacturer's instructions.

When connecting any senors to the sample line, care should be taken to avoid any air from entering into the line. The intrusion of carbon dioxide into the sample can cause low pH values, erratic conductivity measurements, and fluctuating temperatures during measurement.

The Martek Conductivity Reference Cell comes with its own integral flow chamber. The 1/4 inch stainless steel tube fittings protruding from the cell can be used with heavy wall tygon tubing. It makes no difference which end is the inlet or the outlet. A minimum flow of 50 ml/minute is recommended to prevent bubbles from adhering to the inside of the cell. After the cell has been connected, a sharp rap to the sensor will dislodge any trapped air bubbles. For best results, the sensor should be hung vertically with the outlet at the top.

#### 3.3.2 Connecting the pH Sensor

To connect the Martek pH sensor ( p/n 182-245 ), remove the dust cap from the Mark 22 connector marked pH. Insert the plug of the BNC pH adaptor cable (P/N 603052) into the connector. Finally, connect the plug from the pH sensor to the socket of the adaptor cable.

To connect a pH sensor from another manufacturer, the sensor cable must be terminated with a BNC-style connector. Follow the same steps taken to connect a Martek pH sensor to the Mark 22 Calibrator.

The pH sensor should be immersed in a flowing water stream which has not been exposed to air. The assembly drawing in Figure 3.2 illustrates a complete layout of the pH Adapter Plug Assembly. If the Stainless Steel Flow Chamber (P/N 180-51) and the pH Plug Adapter (P/N 180-51A) have been purchased with the Mark 22, the following procedures should be followed:

- A) Connect the temperature cable to the BNC connector of the Adapter Plug.
- B) Remove the black plastic pH sensor retainer from the pH Plug along with its rubber O-ring. Remove the protective cap or soaker bottle from the pH electrode and carefully slide the retainer, then the O-ring, over the pH electrode and screw the threaded cap of the electrode into the retainer. Carefully screw the retainer back into the pH Plug until secure.
- C) Remove the plastic locking nut from the Stainless Steel Flow Chamber and carefully insert the pH Adapter Plug into the Flow Chamber until it rests flush with the top of the chamber. Slide the locking nut over the pH sensor and temperature cables and screw it firmly to the chamber.

# **3 - SPECIFICATIONS AND SETUP**

D) To connect the sample flow to the chamber, place the inlet flow on the left plastic fitting (with the mounting bracket facing away from you) and the outlet flow on the right plastic fitting.

The optimum flow for the Martek Ultrapure pH Sensor should remain between 45 - 150 ml/minute to avoid a streaming effect. The Martek temperature sensor or conductivity reference cell must be connected to the Mark 22 and subjected to the same sample stream if temperature - corrected pH data is to be obtained.



#### 3.3.3 Connecting The Temperature Sensor

Remove the dust cap from the Mark 22 connector marked TEMP, insert the plug of the temperature sensor, and secure. If the pH Adapter Plug (P/N 180-51A) is used, plug the temperature cable into the connector marked TEMP on the Mark 22.

#### 3.3.4 Connecting The Recorder Cable

To hook up the Computer Interface Cable, remove the dust cap from the Mark 22 connector marked REC, insert the plug and secure. Plug the DB25-style connector into the appropriate port on the computer or printer (See Section 4.2.4 for baud settings, etc.). To connect the Mark 22 to an external strip-chart or line recorder, remove the DB25 connector and connect the wires to the recorder. Refer to Section 4.2.5 for recorder output interface cable connection .

#### 3.3.5 Connecting The Battery Charger

The charging jack is located below the Mark 22 connector marked REC. When charging the Mark 22 internal batteries, it is important to follow these two steps:

- A) With the Mark 22 turned OFF, plug the charging plug into the charging jack.
- B) Plug the charger into a wall outlet that carries the same voltage as that listed on the battery charger. The Mark 22 can now be turned ON.

The Mark 22 can be operated either on AC or internal DC power. However, <u>the</u> <u>Mark 22 should be operated only on DC</u> <u>power when calibrating the instrument.</u> This will prevent AC electrical noise from being coupled through the battery charger and interefering with the parameter voltage signal being calibrated.

# 4 - OPERATION

## 4.0 THE MARK 22 CONTROL PANEL

Figure 4.1 depicts the Mark 22 Front Panel with the various keys identified.



Figure 4.1 - Mark 22 Front Panel

Two rocker switches labeled POWER and LAMP are located on the left side of the panel. To turn ON the Mark 22, flip the POWER switch up position. The display will activate and momentarily display the following message:



The number in the upper right corner refers to the version of software installed in the Mark 22 unit. After five seconds the initial screen disappears, and the display begins to cycle through the Display Mode. In the Display Mode the Mark 22 cycles through three screens: (1) corrected conductivity, raw conductivity, and temperature; (2) solutioncompensated pH, Nernstian-corrected pH, and temperature; and (3) date, military time, and battery voltage. If one of the sensors is not connected properly or the Mark 22 has not been calibrated, the screen will display a message indicating that the sensor is not connected or is not calibrated. The number in the upper right hand corner refers to the software version inside the Mark 22. If a sensor is connected and calibrated, the Mark 22 will commence monitoring.

For improved viewing angle and display contrast, flip the rocker switch marked LAMP to the UP position.

NOTE: The Mark 22 has an operating period of approximately 12 hours without the lamp on. If the lamp is activated, the Mark 22 will operate for approximately 8 hours before requiring recharging. Care should be taken to avoid allowing the internal batteries to fall below 5.9 Volts.

## 4.1 How To Use The Keys

Operation of some of the keys described below will require that the Mark 22 be calibrated. If the Mark 22 has not been calibrated, proceed to the chapter on calibration <u>after</u> reading this section.

In addition to the POWER and LAMP switches, there are two rows of buttons or keys outlined in various colors. The KEY LEGENDS indicate the operational mode or function associated with the each key. The bottom row of keys are Mode Keys that allow the user to select which operational mode you wish to place the Mark 22 in. The top row of keys are Function Keys that allow the user to select a particular value or function associated with the current mode of operation.

## READ RANGE AUTO



## 4.1.1 Normal Operating Mode

In the Normal Display Mode the top row of keys function as indicated by the white key lettering above the top row of FUNC-TION keys. The READ Key will hold the display in MANUAL MODE. The UP ARROW AND DOWN ARROW Keys will change the display range of the conductivity measurement, and the AUTO key restarts the automatic scan function in the display. Depressing of the READ will result in the continuous display of the data present in the display window at the time the key is depressed. This feature allows the user to select a particular sensor measurement for be continuous display and monitoring by the Mark 22. The letter "M" will appear in the lower right-hand corner of the display to indicate that the Mark 22 is in the manual mode for data presentation. If the READ key is pressed again, the display will advance to the next set of data. An example of the READ key being depressed three times is shown in following sequence of display screens:





READ Key - 3rd Depression

The first line of the screen indicates the date and military time. The second line contains the measured battery voltage. Unplug the battery charger to read the voltage stored by the battery. The potential should remain

# 4 - OPERATION

above 5.9 Volts to prevent degradation of the battery.

The first line of the conductivity screen displays the temperature-compensated conductivity (S/cm) measured by the reference cell. The second line of the display shows the raw conductivity (mS/cm) measured directly by the cell followed by the measured temperature (°C).

The first line of the pH screen shows a pH which has been compensated for temperature with both a correction for the expected voltage change (Nernstian correction) and a correction based on the ionization of the solution (not necessary in solutions with high ionic conductivity). The second line of the display shows a pH measurement which has been corrected only with the Nernstian correction and the measured temperature.

These RANGE keys allow the Mark 22 to select one of three conductivity ranges when the Mark 22 is monitoring conductivity in the MANUAL mode. The YELLOW ARROWS indicate which direction the conductivity range can be altered. If the UP AR-ROW key is depressed, the Mark 22 will move the decimal point shown in the display to the right. If the DOWN ARROW key is depressed, the Mark 22 will move the decimal point to the left. This feature allows you to select a low, medium, or high range of measurement. An example of the RANGE keys functions are shown below .

UP arrow

01.304mS C Cond 01.452mS 28.15° M

#### **UP** arrow

1.3042mS C Cond 1.4523mS 28.15° M

#### UP arrow

01.304mS C Cond 01.452mS 28.15° M

#### 4.1.2 Setup Mode

The SETUP mode allows the user to calibrate sensors, select temperature compensation formulas, change cell constants, set date and time, and to set up the start time and interval for the logging function. The SE-LECT Key is used to select options from a displayed menu. The UP ARROW Key moves the blinking display down through the menus or increments the displayed digits when adjustment of a numeric input entry is required. The DOWN ARROW Key moves the blinking display up through the menus or decrements the displayed digits when adjustment of a numeric input entry is required.

Depress the yellow SETUP key. The Mark22 will display the following screen:



The Mark 22 utilizes a Setup Option Menu format that provides the user with a list of the available Setup Options. The user is required to select from this list the desired Setup operation to be performed. After the SETUP key has been depressed, the top row of function keys are now in affect. All text and symbols in YELLOW are now applicable in regards to operation of the Mark 22.

To select a SETUP option, the user must depress the SELECT key. To move the flashing prompt up or down to another option, you would press either the UP or DOWN arrow keys. To exit from the Setup Option Menu the user must depress the EXIT key. which will return the Mark 22 to the normal display monitoring mode. The options presented in the SETUP menu are discussed in Section 4.2 and in the chapter on CALIBRA-TION. For now, simply depress the EXIT key. In the Setup Mode of operation the top row of keys function as indicated by the yellow text located on the top line of text on each of the function keys.

#### 4.1.3 Cell Mode

The CELL Key allows you to change the cell constant currently used by the Mark 22 to any of three different, preset cell constants simply by pressing the appropriate function key whose cell constant value is labeled in blue. Press the CELL key. The Mark 22 will respond with a display similar to the one pictured below:



To change this cell constant, simply press one of the function keys. Pressing a function key while in the Cell Mode will change the K factor used to calculate the raw and temperature-compensated conductivity measurements. While the nominal cell constant values listed on the function keys are 0.01, 0.1, 1, and 10, you can change any of these cell constants to whatever value is desired. Refer to Section 4.2 on how to change individual cell constants. To return to the monitoring mode, press the CELL key again. In the Cell Mode of operation the top row of keys functioon as indicated by the blue text located on the second line of text on each of the function keys. The K 0.01 Key selects the cell constant for Cell #1, the K 0.1 Key selects the cell constant for Cell #2, the K 1 key selects the cell constant for Cell #3 and the K 10 Key selects the Cell constant for Cell #4. The Cell Screen appears as follows:

> CURRENT CELL #1 IS K .01000

#### 4.1.4 Data Log Mode

In the Log Mode of operation the top row of keys function as indicated by the red text located on the third line of text on each of the function keys. The FREE Key will display the amount of storage memory remaining in terms of hours. The amount of data stored is directly related to the scan rate used during the logging operation. The PAUSE Key will suspend the logging function, and the RESUME Key will restart it. The CLEAR Key will clear all of the logged data from the unit's internal memory. The LOG key is one of two operational mode keys on the Mark 22 membrane panel outlined in RED. When the log Key is depressed, the function (labeled in red ) on each of the function keys becomes applicable. After the LOG key is depressed, the Mark 22 will display the following screen:

## CURRENT LOG FILE IS FILE # 01

Depress the LOG key to return to the to the normal display monitoring mode. However, a black square will be visible in the top right-hand corner of the display similar to the display shown below:



The Black Square indicates that the Mark 22 is in the data LOG mode of operation. Pressing the FREE key will display the amount of memory remaining for data storage in terms of logging hours remaining and then return to Log Mode. The LOG mode may be paused by depressing the PAUSE key. Logging will be suspended until the RE-SUME key is depressed. The upper right corner of the display will show a letter " p " instead of the black square when the Mark 22 is in the paused state of the Log Mode.

To stop logging, simply press the LOG key again. The next time you press the LOG key, the Mark 22 will increment the internal file counter and start logging data in the new file number. This feature allows you to assign different files to different monitoring points. Refer to Section 4.2 on how to set up the Mark 22 for logging at predetermined times and intervals.

#### 4.1.5 Print Mode

The PRINT key is identified by its RED key legend. When this key is depressed, the the top row of function keys operate as indicated by the RED legend located on each key. The Mark 22 will always send the displayed data information to the printer port under normal conditions. Pressing the PRINT key will allow the Mark 22 to print what has been stored in memory when it is hooked up to a computer or printer. Pressing the PRINT key will provide the following:

PRINTER READY ? FILE # 01

By pressing the RESUME key, the Mark 22 will start sending data to the printer. Pressing the PAUSE key will stop the printing. Pressing the RESUME key will resume printing. Pressing the FREE key will show how many scans or lines of data remain in that particular file. Finally, pressing the CLEAR key will increment the Mark 22 to the next file to be printed. To exit the printing mode, simply press the PRINT key again. Information on how to set up your computer or printer to communicating with the Mark 22 can be found in Section 4.2.

### 4.2 How to SET UP THE SYSTEM

This section describes the operation of the Mark 22 in the Setup Mode. However, the calibration procedures for temperature, conductivity, and pH are extensive, so Chapter 5 has been dedicated to the calibration of the Mark 22. The other three options on the initial Setup Menu are COMP, CELL, and Pressing the UP ARROW will move LOG. the flashing display from CAL to COMP, from COMP to CELL, from CELL to LOG, and from LOG to CAL. The DOWN ARROW moves the flashing display up through the menu. When the desired operation is flashing, press the SELECT key. To return to the Display Mode, press EXIT.

#### 4.2.1 Temperature Compensation Formulas

When utilizing conductivity and pH sensors in ultrapure water, compensation formulas for temperature and sample water impurities must be considered since sample sample temperatures are rarely maintained at precisely 25 degrees C. The Mark 22 is capable of providing raw parameter data, temperature, and temperature corrected data all in one display screen. The temperature elements for all Martek probes are rated to 100 deg. C and with the exception of pH, all sensors can be subjected to sample water with temperatures up to this limit.

For ultrapure conductivity measurement, the Mark 22 can utilize one of three different temperature compensation formulas. The three formulas are:

The slope coefficient a can also be adjusted within the Mark 22 software. The coefficient is typically 2.0%/°C for neutral salt Since cation conductivity measolutions. sures acidic streams, slope coefficients between 1.0 and 1.6%/°C are more appropriate for cation conductivity samples. If the Mark 22 is used to calibrate on-line instrumentation, match the temperature-compensation formula and slope coefficient to those applied by the on-line instrumentation. For more information about temperature compensation of conductivity, see "Temperature Compensated Conductivity—A Necessity for Measuring High-Purity Water" by Oscar Zabarsky, Ultrapure Water, Jan/Feb 1992, pp. 56-60.

#### Marsh and Stokes:

$$EC_{25^{\circ}C} = \frac{EC_{T_{c}} - \left[0.05498 + 2.9326 \times 10^{-3} (T_{c} - 25) + 6.0629 \times 10^{-5} (T_{c} - 25)^{2} + 4.2690 \times 10^{-7} (T_{c} - 25)^{3}\right]}{1 + a (T_{c} - 25)} + 0.05498$$

**Truman Light:** 

$$EC_{25^{\circ}C} = \frac{EC_{T_{c}} - \exp 10.858 - \frac{72158}{T_{c} + 273} + \frac{0.20004 \times 10^{7}}{(T_{c} + 273)^{2}} - \frac{0.3196 \times 10^{9}}{(T_{c} + 273)^{3}}}{1 + a (T_{c} - 25)} + 0.05479$$

#### GE:

$$EC_{25^{\circ}C} = \frac{EC_{Tc} - 0.0545[0.55\exp(0.0363T_{c}) - 0.36297]}{1 + a(T_{c} - 25)} + 0.0545$$

where

 $EC_{25^{\circ}C}$  = electrical conductivity at 25°C  $EC_{Tc}$  = electrical conductivity at T<sub>c</sub>  $T_c$  = sample temperature (°C) a = slope coefficient (1/°C) From the SETUP Menu, choose the compensation and press the select key. The display will indicate the following:

$\bigcap$	MARSH	LIGHT
	GE	SLOPE

The operator must use the UP and DOWN ARROW Keys, to navigate the display and highlight the desired compensation formula. The option is chosen by user when the SEECT Key is depressed while one of the formulas is highlighted.

The flashing prompt will automatically highlight the "SLOPE " option after the temperature compensation formula has been selected. The slope coefficient for conductivity and pH can be altered by selecting the SLOPE option and then choosing either conductivity or pH options.

To change a solution slope coefficient, use the UP or DOWN ARROW Keys to increment or decrement the displayed numeric value. Depress the SELECT Key to enter and save the displayed value and subsequently advance to the next the next digit. Use the EXIT Key to backspace and edit a previously saved value.

If conductivity is selected first and the slope coefficient has been entered, the Mark 22 will automatically default to the pH solution compensation factor. The solution compensation factor can be calculated for pH using the following formula:

pH Solution

Compensation= pH @ Low Temp - pH @ High TempCoefficientHigh Temp - Low Temp

For example, if we use 1 ppm ammonium hydroxide in ultrapure water, the above formula would be as follows: pH Solution Compensation = Coefficient

> <u>10.20 pH @ 10°C - 8.96 pH @ 50°C</u> 50°C - 10 °C

= 0.031 pH / °C

### 4.2.2 How to Select the Conductivity "K" Factor`

The Mark 22 allows you to adjust the value of the cell constant or K factor of four cell constants: K .01, K 0.1, K 1, and K 10. In this way, fine adjustments can quickly be made to the Mark 22 to match the cell constants of different conductivity sensors.

The K factors have been preset at the factory for the values listed above and for any Martek Conductivity Reference Cell ordered with the Mark 22.

Bbefore changing the cell constant for a particular conductivity sensor, you must make certain that the cell constant you are trying to input falls within the overall "K" factor range . The figure below presents the range of "K" factors that can be used with the four basic cell constants.

Basic "K" Factors	Conductivity Reference Cell "K" Factor Range
K = 0.01	"K" range: .005 - 0.05
K = 0.10	"K" range: 0.05 - 0.50
K = 1.00	"K" range: 0.50 - 5.00
K = 10.0	"K" range: 5.00 - 50.0

However, each cell constant can be changed to any value desired. To do this, simply press the SETUP key and select the option labeled CELL. The display will look like this:



If you press the SELECT key again, the display will show the cell number and the value of the cell constant which will be flashing.

# CELL #3 IS

SET TO K 1

with the "K 1" prompt flashing. Using the UP and DOWN ARROW keys, change the flashing "K" factor until it reads "K .01". Now press the SELECT key. The display will now indicate:

## CELL #3 IS

## = TO K .01000

and the "1" will be flashing. You can now alter the value of the flashing digit by pressing the UP or DOWN ARROW key. You can then move forward to the next digit by pressing the SELECT key and move back to a previous digit by pressing the EXIT key.

Another press of the SELECT key and the cell constant value can be adjusted by moving the digits up or down with the arrow keys then pressing the SELECT key to enter the value into the Mark 22's memory. Once the K factors have been set to the proper values, press the EXIT key to return to the set up menu. Figure 4.2 depicts a cell constant range chart to give you the low, medium, and high ranges for the four most commonly used K factors:

	LOW	MEDIUM	HIGH
K=0.01	1.0000 uS	10.000 uS	100.00 uS
K=0.10	10.000 uS	100.00 uS	1000.0 uS
K=1.00	100.00 uS	1.0000 mS	10.000 mS
K=10.0	1.0000 mS	10.000 mS	100.00 mS

#### Figure 4.2 - Cell Constant Range Chart

Once the correct cell constant has been selected for the conductivity sensor to be used, you are ready to start monitoring.

## 4 - OPERATION

#### 4.2.3 How to Log data

One of the most convenient features of the Mark 22 is its ability to log monitored data into memory and then down-load this data to either personal computers or printers. In addition, the Mark 22 can be programmed to start logging at a pre-determined time and interval for unattended monitoring of key process lines.

To set up the Mark 22 for logging, press the SETUP key and select the LOG option. The Mark 22 will display the following:



The STARTING TIME is used to set the logging function to begin at a predetermined time. Use the arrow keys to change the individual digits and use the select key to enter them. Upon completion, the Mark 22 will automatically prompt on PERIOD.

When PERIOD is selected, the Mark 22 will display the following:

## INPUT LOG PERIOD IN MINUTES 1-255

Entering any value between 1 and 255 minutes will set the amount of time between recorded measurements when logging.

When # SCANS is selected, the display will read as follows:



and the "1" will be flashing. Use the arrow keys to change the number of complete readings the Mark 22 will make during each scan period. Upon completion, the Mark 22 will return to the set up menu.

NOTE: The time remaining in memory is determined by the number of scans and the period between scans. The Mark 22 can hold a total of 371 scans.

For example, if the period between recordings is set for 30 minutes and the number of scans is set for 2, then there are 4 scans per hour. 371/4 = 92.75

The total logging time is 92.75 hours. The time remaining will be displayed as 92.8 hours when first starting to log.

## 4.2.4 Print Function Setup

Once data has been logged into the Mark 22, it can be down-loaded to any computer or printer that accepts serial ASCII RS 232 digital input. To down-load the stored data in the Mark 22 requires that you set your computer or printer with the same data flow configuration utilized by the Mark 22. The Mark 22 is preset at the factory for the following settings:

Baud Rate	1200
Data Bits	7
Stop Bits	1
Parity	Even

Consult your computer or printer manual for details on how to manually configure your system to the above settings. You can also use communication software programs such as PROCOMM or CROSSTALK that allow your computer to set the data flow configuration via the keyboard.

Once your computer is set up to communicate with the Mark 22, connect the interface cable to the Mark 22 and plug the DB25 style connector to the appropriate serial port on the computer or printer.

Pressing the **PRINT** key will cause the display to respond with:



At this point, review Section 4-1 again for instructions on how to use the print function keys.

To erase logged data, disconnect the Mark 22 from the computer or printer. Next, press the PRINT key, then the PAUSE key, then finally, the RESUME key. The Mark 22 will display the following messages:

INVALID DATA IN FILE (ABORTING)

CAUTION! ALL LOGGED DATA WILL

BE ERASED BY THE SELECT KEY

OR SAVED BY THE EXIT KEY

The display will then present you with the following:

# (SELECT) = ERASE (EXIT) = SAVE

If you wish to save the data stored in the Mark 22, press the EXIT key. If you wish to erase the data, press the SELECT key.

CAUTION! ERASING LOGGED DATA ERASES ALL DATA IN ALL FILES.

#### 4.2.5 How to use the Recorder Output

There are some instances when it is desirable to hook up the Mark 22 to a strip chart or line recorder in addition to or to substitute for the Mark 22's own internal logging capability. A likely example would be for monitoring on-line instrumentation that is already using analog recorders for data acquisition.

The Mark 22 comes with a simultaneous 0-1 volt DC recorder output for conductivity and temperature or conductivity and pH. If the pH sensor is not connected to the Mark 22, the pH recorder output signal will convert to a temperature recorder output signal. Refer to Figure 4.3 cable interface for wire lead identification.

In order to use the analog recorder output, the DB25-style connector on the serial cable (P/N 603050) must be removed and the wires tinned before connecting to the recorder. Once the recorder has been attached to the Mark 22, the user can calibrate each recorder output for the associated parameter.

The Mark 22 automatically defaults to the recorder output set up after the solution compensation for conductivity and pH have been entered into the Mark 22. The following message will be flashing in the display:

## SETUP RECORDER ZERO-FULL SCALE

Pressing the SELECT key will cause the Mark 22 to display the following:



and the COND=#1 will be flashing. Output #1 is for conductivity and Output #2 is for pH. Press the SELECT key again and the Mark 22 displays:



and the ZERO will be flashing. Now, if the SELECT key is depressed again with the Mark 22 connected to the recorder, the unit will send a 0 Volt signal to the recorder to simulate zero. The prompt will automatically shift to FULL. By pressing the SELECT key again, the Mark 22 will send a 1 Volt signal to the recorder to simulate full scale. To return to the previous menu, press the EXIT key. The pH=#2 prompt will now be flashing and you can repeat the same procedure for the pH analog signal. Upon completion, press the EXIT key to return to the Display Monitoring Mode.

# **OPERATION - 4**

				P1
	·	RED	REC - COND	1
		BLK	RETURN - COND	2
	1	WHT	NC	3
<u></u>		BLK	XMIT - SERIAL	Ĩ
				4
		] BLU	REC - pH	5
/		BLK	RETURN - pH	c
Ϋ́Υ.		GRN	REC - SERIAL	
TO EXTERNAL		BLK	COMM - SERIAL	
RECORDING DEVICE				8
			D/NL405016	

P/N 105316 CONN, PLUG



## **5 - CALIBRATION**

#### 5.0 Why Should You Calibrate the Mark 22?

Electronic instruments that measure physical or chemical reactions require periodic calibration in order to maintain their accuracy. This is done by comparing the measurements gathered by the instrument to a reference or standard that is well-known and widely accepted.

The most commonly used method for calibration involves two points: zero point and the span point. The purpose of these two points is to establish the slope of the line of measurement between the zero and span points. The accuracy of an instrument using a two-point calibration is therefore directly related to the ability of the sensor and the instrument to maintain linear output over the full scale of measurement.

#### 5.0.1 Materials Required for Calibration

The Mark 22 can be calibrated using solutions or electronic circuitry that simulates the electrical properties of solutions. In either case, the method chosen should be traceable to National Institute of Standards and Testing, NIST (Formerly National Bureau of Standards).

To calibrate the Mark 22 using solutions, the following items will be required:

- 1. Laboratory-grade thermometer
- 2. Temperature Bath (ice and warm water will also work)
- 3. Conductivity Reference Solution

4. pH Buffers 7 and either 4 or 10.

To maintain the specified measurement accuracy, always calibrate the Mark 22 in the range of the measurement to be performed. Pre-mixed conductivity solutions may be purchased from scientific equipment distribution warehouses or the solutions can be mixed in the laboratory by consulting a chemical handbook which compares potassium chloride concentrations to electrical conductivity values.

Items 1-3 of the above list are not necessary if the temperature and conductivity are calibrated electronically.

### 5.1 **TEMPERATURE CALIBRATION**

Temperature must always be calibrated before conductivity and pH since temperature is used in the Nernstian and solution compensation for pH, and for temperaturecorrected conductivity values and solution compensation for conductivity.

Temperature can be calibrated either electronically inside the Mark 22 (Auto Calibration) or physically using a temperature bath (Manual Calibration) to set the two point calibration of zero and span.

In an electronic calibration, a particular physical value can be determined by using its direct known resistance value and electronically substituting this value as the actual physical measurement. For calibrating temperature, the Mark 22 utilizes precision resistors in its circuitry to simulate 0°C and 50°C.

## 5.1.1 Auto Temperature Calibration

To set the instrument up for automatic temperature calibration, press the SETUP. The Mark 22 then displays the main Setup Menu. Select the CAL option from the menu. The display then appears as follows:



Select "TEMP". The display will prompt with:



Select "ZERO". The display will prompt with:



Select "AUTO". After a short delay, the display will appear as follows:



At this time, the Mark 22 acts like a voltmeter and shows a varying voltage. An internal relay has switched in the precision resistors simulating a temperature of 0°C (the voltage displayed will vary from monitor to monitor). Allow the displayed voltage to stabilize. When the voltage shown on the display is stable, depress the SELECT key. After the SECLECT key has been pressed, the display will prompt with "TEMP AUTO ZERO" and then automatically default to "SPAN". At this time, press the SELECT key. The display will prompt with:



Select "AUTO". Again, after a short delay, the display will prompt with a momentary message:



Again, the Mark 22 acts like a voltmeter and shows a varying voltage. An internal relay has switched in the precision resistors simulating a temperature of 50°C. Depress the SELECT key when the displayed voltage has stabilized. After the key has been pressed, the display will prompt with "TEMP AUTO SPAN" and then automatically default to "COND".

#### 5.1.2 Manual Temperature Calibration

The Mark 22 may also be calibrated using external reference points. A constant temperature bath would provide the most useful reference, however, ice and hot water can also be used if a temperature bath is not available. Also, make sure the temperature sensor is connected prior to operating the Mark 22 ( section 3.2.3 ).

#### Temperature Zero - Calibration

Depress the SETUP key and select "CAL" and then "TEMP" from the display menus. The display will prompt with:

# **5 - CALIBRATION**



Select "ZERO". The display will prompt with:



Select "MANUAL". After a short delay, the display will prompt with a momentary message:



Then will ask you to:

HIT KEY W/STABLE -0.0012 VOLT

Like the auto temperature calibration, the Mark 22 acts like a voltmeter and shows a varying voltage. At this time, place the temperature sensor in a temperature ice bath or a beaker filled with water and crushed ice. At the same time, measure the temperature of the ice slurry with an accurate thermometer.

NOTE: The freezing point of water does not actually have to be reached for the Mark 22 to determine the sensor's reading at 0°C. As long as the temperature low point is lower than that of the samples, a value above the freezing point of water can be used. For example, if your range of measurement is between 10° and 30°C, then you could calibrate the Mark 22 for temperature using 5° and 40° as your low (zero) and high (span) points. When the voltage reading on the display has stabilized, depress the SELECT key. The Mark 22 will prompt with the following display:



Input the temperature value as a 4-digit number (note the fixed decimal point). The value of the flashing digit may be altered by pressing the UP or DOWN ARROW keys. Move forward to the next digit by pushing the SELECT key. Move back to a previous digit by pushing the EXIT key.

Temperature Span - Calibration-

After the Mark 22 has been zeroed for temperature, it will automatically prompt with the following display:



Select "MANUAL". After a short delay, the display will prompt with a momentary message:



Then will ask you to:



At this time, place the sensor in a high (span) temperature bath where the temperature is approximately twice the sample temperature. To accurately measure the water temperature, place a thermometer into the solution. When the voltage displayed has stabilized, press the SELECT key. The Mark 22 will prompt with the following display:



Input the temperature value as a 4-digit number (note the fixed decimal point). You can edit the value of the flashing digit by pressing the UP or DOWN ARROW keys. Move forward to the next digit by pushing the SELECT key. Move back to a previous digit by pushing the EXIT key. After the last number has been entered, the Mark 22 will automatically respond with the following display:



and the "COND" prompt will be flashing. At this point, press the EXIT key twice to return to the Display Monitoring Mode.

## 5.2 CONDUCTIVITY CALIBRATION

## 5.2.1 Conductivity Zero Adjustment

After the temperature has been calibrated, connect a dry the Martek Conductivity Reference Cell or any other dry reference cell to the Mark 22 (See Section 3.2.1 for instructions on how to connect sensors to the Mark 22). [An alternate method is to use the conductivity interconnect cable (P/N 603051) to simulate a conductivity reference cell. To use this cable, connect it to the Mark 22 and insert the Martek Zero Calibration Plug (P/N 400550). [Select the cell constant you will be measuring with (i.e. 0.0100, 0.1000, 1.000, or 10.00), and proceed with the zero calibration.]

Next, press the CELL key to locate the correct cell constant or "K" factor that should be used with that particular conductivity sensor. The "K" factor is listed for reference on the Martek Reference Cell's cable. If the "K" factor listed on the conductivity sensor is not present in the Mark 22, review Section 4.2.2 on how to change cell constants. If using the calibration plug, select the "K" factor nearest the reference (i.e. 0.0100, 0.1000, 1.000, or 10.00) from the table in section 4.2.2. Once the "K" factor has been entered, depress the CELL key to return to the Display Monitoring Mode.

With the Mark 22 in the Display Monitorig Mode, depress the READ key to place the unit in Manual Monitoring Mode until the display indicates conductivity measurements. Next, place the monitor in the highest conductivity range by pressing the UP ARROW key until the decimal point is furthest to the right as possible. Calibration in this range minimizes the zero offset.

Return to the calibration mode by pressing the SETUP key then select the "CAL" option from the Setup Option Menu. The Mark 22 display will respond with the following:



Use the UP/DOWN ARROW Keys to get the COND prompt blinking. Depress the SE-LECT key to choose "COND". The display will then prompt:



Depress the SELECT key to choose "ZERO". The display will then prompt:

PLACE SENSOR IN LO COND STANDARD

Then will ask the user to:

## HIT KEY W/STABLE -0.9998 VOLT

Since there is no solution in the reference cell, the voltage displayed represents the zero voltage set point of the reference cell. As mentioned earlier, the voltage values shown in this manual are for example purposes only. The normal voltage displayed when zeroing the sensor should be approximately -1.00 Volt.). Once the voltage has stabilized, depress the SELECT key. The display will respond with the following:

## INP 5 DIGIT STD. LO C = 000.00 mS

Input the 5-digit low (zero) conductivity value by pressing the SELECT key. Once the zero point has been entered, the Mark 22 will automatically respond with the following display:



and the "SPAN" prompt will be flashing.

### 5.2.2 Conductivity Span -Calibration

There are two methods that can be used to set the high (span) point for conductivity:

- \* Electronic (dry) Method for calibration
- \* Dynamic (wet) Method for calibration.

The Electronic Method for Spanning Conductivity

What sets the Mark 22 apart from other calibrators is its ability to use alternative methods for calibration (ASTM D5391-95) as a substitute for standard reference solutions. To do this requires precise resistance values that can be traceable to NIST standards for accuracy and reliability. Martek Instruments provides a certified calibration plug with a precise resistance value equal to 1.000  $\mu$ S/cm with each Mark 22. Additional "solution" values (10.00 and 100.0  $\mu$ S/cm) are available from Martek. A precision resistance box with an accuracy of at least  $\pm$  0.05% may also be used instead of the calibration plug.

In addition to a known resistance value, the capacitance of the reference cell must be known. The capacitance of the Martek Conductivity Reference Cell is approximately 170 to 175 picofarads and this value is included within the calibration plugs to compensate for the Martek Reference Cell's impedance. [To determine the capacitance of other reference cells, consult the manufacturer or connect an impedance meter to the dry cell to determine its capacitance.]

# NOTE: the following procedure is for a 1.000 uS/ cm solution.

First, remove the conductivity reference cell that is hooked up to the Mark 22. If not already connected, hook up the conductivity interconnect cable (P/N 603051) with the span calibration plug (P/N 400549) to the Mark 22. Next, return to the monitoring mode by pressing the EXIT key. Place the Mark 22 in the manual monitoring mode for conductivity.

Now, press the CELL key. If not already

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selected, select a cell constant of K = 0.0100 then press the CELL key again to return to the monitoring mode.

Next, place the monitor in the lowest conductivity range by pressing the DOWN ARROW key until the decimal point is as far to the left as possible.

Enter the calibration mode and choose the "COND" option. Next, choose the "SPAN" option and press the SELECT key. The display will prompt with the following message:



Then will ask you to:



The voltage value for span should approximate +0.5000 volt.

Once the displayed voltage has stabilized, press the SELECT key. The display will prompt with the following message:



Enter the value of 1.0000 uS by pressing the SELECT key..

Once the last digit has been entered, the Mark 22 will automatically return to the calibration menu:



and the "pH" option will be flashing.

The Mark 22 is now calibrated for con-

ductivity. The conductivity reference cell can now be attached to the Mark 22 and measurements taken. However, before doing so, make certain that the specific cell constant listed on the reference cell being used is correctly entered into the Mark 22. A condensed version of the conductivity calibration procedure is located in Chapter 2.

**CALIBRATION - 5** 

The Dynamic Method of Spanning Conductivity

The Mark 22 conductivity calibration can also be determined by the traditional method of using reference solutions traceable to NIST standards for accuracy.

When calibrating conductivity with this method, special care must be taken to ensure that the correct "K" factor range of measurement is entered into the Mark 22 and that the raw conductivity value of the reference solution is used when inputting the conductivity span value.

Once you are certain that the correct "K" factor has been entered within its appropriate range for your particular reference cell, you can proceed to span the Mark 22 conductivity sensor.

The conductivity can be spanned using a conductivity reference solution of potassium chloride (KCL) equivalent to one of several specific conductivity values. You can purchase this reference solution or mix it yourself (Refer to a chemical handbook for potassium chloride concentrations versus conductivity values).

When using a reference solution, you must remember the following:

"The conductivity value of the certified reference solution is always measured at  $25^{\circ}$ C."

# **5 - CALIBRATION**

Therefore, unless solution temperature in your conductivity reference cell is exactly 25°C, you must measure the solution temperature and apply the following formula:

#### Solution Conductivity @ 25°C X [1 + .02(T - 25)] = Raw Conductivity

For example, let's say you are using a reference solution with a certified conductivity value of 30 microsiemens/cm @ 25°C. After measuring the solution with a thermometer, you determine the temperature of the solution to be 19°C. If you input these numbers into the formula stated above, the equation will look like this:

30 uS/cm X [1 + .02(19 - 25)] = 26.4 uS/cm

After processing the equation your answer should be 26.4 uS/cm. Again, this is the <u>raw conductivity</u> value of the reference solution and this is the value that you would enter into the Mark 22.

To span the conductivity value, place the reference solution in the conductivity reference cell. It is desirable that the solution be flowing through the cell, but if this is impractical, fill the cell completely with solution and seal the ends.

Now, enter the calibration mode and choose the "COND" option. Next, choose the "SPAN" option and press the SELECT key. The display will prompt with the following message:

## HIT KEY W/STABLE +0.5120 VOLT

Once the displayed voltage has stabilized, press the SELECT key. The display will prompt with the following message:

INP 5 DIGIT STD. HI C = 0.0000 uS

Enter the <u>raw conductivity</u> value of the reference solution by pressing the UP or DOWN ARROW key. You can then move forward to the next digit by pressing the SELECT key and move back to a previous digit by pressing the EXIT key. The position of the decimal point will vary when other cell constants are used.

NOTE: If the solution is not flowing through the cell, the voltage displayed may slowly start increasing due to polarization. Care should be taken to press the SELECT key as quickly as possible after the solution has been placed in the reference cell and as soon as the voltage appears to have stabilized.

Once the last digit has been entered, the Mark 22 will automatically return to the calibration menu:

TEMP	COND	
рН	TIME	

and the "pH" option will be flashing.

PLACE SENSOR IN HI COND STANDARD

## 5.3 pH Calibration

Measuring pH in ultrapure water requires specially designed electrodes. Unlike normal water applications, ultrapure water has very low ionic activity making it difficult to measure pH accurately. The Mark 22 can use a variety of pH electrodes from different manufacturers. However, the following calibration procedures apply to the ultrapure pH sensor offered by Martek Instruments.

The Martek pH sensor should be calibrated in buffer solutions that are traceable to NIST standards for accuracy and reliability.

Care should be taken to insure that fresh, uncontaminated buffer solutions are used. Buffer cups should always be rinsed with distilled water and dried before each use and used buffers should always be discarded.

Finally, the temperature of the buffer solution must be measured and the appropriate pH value versus temperature inputted into the Mark 22. You can use either the Martek Temperature Sensor or a laboratorygrade thermometer to determine buffer temperature. Most buffer solutions list the various temperature variations on the side of the buffer containers. If the values are not listed. consult the manufacturer or distributor for this information. Most importantly, care must be taken to ensure that the solution is properly grounded. If not, pH measurements can be erratic. Using the pH Adapter Plug (P/N 180-51A) is a good way to ensure accurate temperature measurement as well as proper grounding of the solution.

## 5.3.1 pH Zero Adjustment

Connect the pH sensor to the Mark 22 using the pH adaptor cable (See Section 3.2).

Place the pH sensor in pH buffer 7.00 solution.

Press the SETUP key and select the "CAL" option from the SETUP menu. Next, select the "pH" option. The display will prompt with the following:

![](_page_34_Picture_10.jpeg)

Press the SELECT key to choose "ZERO". The display will prompt with the momentary message:

![](_page_34_Picture_12.jpeg)

Then will ask you to:

HIT KEY W/STABLE -0.1412 VOLT

When the voltage reading on the display has stabilized, Press the SELECT key. The Mark 22 will prompt with the following display:

> INP 4 DIGIT STD. LO pH = 07.00

and the "7" will be flashing. Input the pH value as a 4-digit number (make sure you use the correct pH value corresponding to the temperature of the pH buffer and that you input a 0 before single-digit pH values). You can alter the value of the flashing digit by pressing the arrow keys up or down. Move forward to the next digit by pushing the SE-LECT key. Move back to a previous digit by pushing the EXIT key. Once the zero point has been entered, the Mark 22 will automatically respond with the following display:

# **5 - CALIBRATION**

![](_page_35_Picture_1.jpeg)

and the "SPAN" prompt will be flashing.

#### 5.3.2 pH Span Adjustment

After the pH sensor has been zeroed, the span adjustment can be set. Remove the pH sensor from the pH buffer 7.00 solution and rinse it with fresh water. Using a light tissue, gently pat the glass bulb of the pH electrode removing observable moisture.

Place the pH sensor in pH buffer 4.00 or 10.00 solution depending upon the pH range of the solution the sensor is to measure.

Choose the "SPAN" option by pressing the SELECT key. The display will prompt with the following message:

## PLACE SENSOR IN HI pH STANDARD

Then will ask you to:

![](_page_35_Picture_9.jpeg)

It may take several minutes for the voltage to stabilize. Once the displayed voltage has stabilized, press the SELECT key. The display will prompt with the following message:

![](_page_35_Picture_11.jpeg)

Input the pH value as a 4-digit number (make sure you use the correct pH value cor-

responding to the temperature of the pH buffer). You can alter the value of the flashing digit by pressing the arrow keys up or down. Move forward to the next digit by pushing the SELECT key. Move back to a previous digit by pushing the EXIT key. Once the span point has been entered, the Mark 22 will automatically respond with the following display:

![](_page_35_Picture_14.jpeg)

and the "TIME" option will be flashing.

## 5.4 How to set Date and Time

The Mark 22 uses an internal clock to maintain date and time for recording purposes. This feature allows you to record exactly when a measurement was made which can facilitate on-line system performance evaluations over a period of months or even years.

To set the date and time, press the SETUP key and select the "CAL" option. Next, select the "TIME" option. The Mark 22 will prompt with the following display:

![](_page_35_Picture_19.jpeg)

and the "DATE" option will be flashing. Press the SELECT key. The display will then indicate the following:

![](_page_35_Picture_21.jpeg)

The "mm" stands for month and the "dd" stands for day. Alter the value of the flashing digit by pressing the arrow keys up or

down. Move forward to the next digit by pushing the SELECT key. Move back to a previous digit by pushing the EXIT key. Once the last digit has been entered, the Mark 22 will automatically respond with the following display:

![](_page_36_Picture_2.jpeg)

and the "TIME" option will be flashing. Press the SELECT key. The display will then indicate the following:

![](_page_36_Picture_4.jpeg)

The "hh" stands for the hour and the "mm" stands for the minutes. The time must be inputted as a 24 hour clock, i.e. 1:00 PM would be inputted as 1300, 9:30 AM would be inputted as 0930, etc. Alter the value of the flashing digit by pressing the arrow keys up or down. Move forward to the next digit by pushing the SELECT key. Move back to a previous digit by pushing the EXIT key. Once the last digit has been entered, the Mark 22 will automatically respond with the following display:

![](_page_36_Figure_6.jpeg)

Press the EXIT key to return to the monitoring mode.

## 5.5 FREQUENCY OF CALIBRATION

Standard operating procedures for calibration vary with each organization. Martek Instruments recommends calibration of the Mark 22 for all parameters at least once a month. More frequent calibration may be required if the displayed values are unusually high or low, or if the application itself changes.

In general, pH sensors are unstable electrodes and will require more frequent calibration. Temperature and conductivity are much more stable and will require less frequent calibration.

# 6 - MAINTENANCE & TROUBLESHOOTING

#### 6.0 THE READOUT MODULE

The Mark 22 monitor is housed in an extruded aluminum case coated and painted to withstand exposure in a marine environment. However, <u>the Mark 22 is not waterproof</u>. Care should be taken to avoid moisture settling on the front panel. If water exposure is a problem, a large plastic zip-lock bag can be used to protect the Mark 22 from exposure.

While the Mark 22 is portable, it should be treated like a quality laboratory instrument. Excessive banging or dropping of the monitor should be avoided.

The lamp used in the Mark 22 display has a rated life-span of over 7,500 hours. The lamp should be used sparingly when surrounding light is poor. Replacement lamps are available from Martek Instruments.

### 6.1 SENSORS

The temperature sensor requires no maintenance.

The Martek Conductivity Reference Cell should be kept dry when not in use. After each use and before the sensor is stored, isopropyl alcohol should be flushed through the sensor to remove any dirt or salt build-up. If possible, use pressurized air to remove any remaining moisture.

The pH sensor should be kept in its soaker bottle or cap at all times when not in use. Potassium chloride (KCI) or a pH buffer solution should be used in the soaker bottle or cap. Do not use distilled water.

The connectors should be kept free of dirt and the cables carefully coiled when not in use.

#### 6.2 **T**ROUBLESHOOTING

There may be instances where the Mark 22 does not respond in the manner desired. This section deals with the errors most commonly encountered and offers suggestions to overcome potential problems.

1. The Mark 22 display indicates "No pH or Conductivity Sensor connected" even though sensors are hooked up to the monitor.

<u>ANSWER:</u> Check to see that the sensors are hooked up to the correct connectors on the monitor. If so, turn the Mark 22 off and then back on again to recycle the program.

2. The Mark 22 fails to respond to any key commands.

<u>ANSWER:</u> Make sure that the Mark 22 is not in the LOGGING mode. This is indicated by a black box or the letter "p" in the upper right hand corner of the display. If so, press the LOG key to disable the logging function.

3. The Mark 22 displays "MEM ERROR REDO CALIBRATION" after power is turned on.

<u>ANSWER:</u> The Nicad batteries used for internal memory power in the Mark 22 may not be charged. Charge the Mark 22 for at least 24 hours to allow the batteries to charge up.

 Conductivity readings are not close to the suspected value of the sample being measured.

<u>ANSWER:</u> Check to see that the correct "K" factor, range, and temperature compensation formula have been inputted

# **MAINTENANCE & TROUBLESHOOTING - 6**

into the Mark 22. Also make sure that the cell being used is clean and has no air bubbles in it when the sample passes through it.

5. pH readings are erratic and "noisy" when pH sensor is installed in adaptor plug.

<u>ANSWER:</u> Check to see that the flow to the chamber is very close to 50 ml/min. Higher flow rates could create a streaming affect that can cause pH values to fluctuate. Carbon dioxide may also affect pH readings. Make sure that the plug is properly grounded.