



# DISSOLVED OXYGEN ANALYZER

OPERATION MANUAL

# WARRANTY POLICY

Unless otherwise stated, MARTEK INSTRUMENTS, INC., warrants this equipment to be free from detects 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 at (919) 790-2371 or by fax at (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 sent freight prepaid and addressed as follows:

MARTEK INSTRUMENTS, INC 2609 DISCOVERY DR. RALEIGH, NC. 27616

Atm: 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 power 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 to assume for them any other liability in connection with the sales of MARTEK instrumentation.

# TABLE OF CONTENTS

		PAGE
CHAPTER	1 Introduction	
	1.0 - What is the Mark 21?	1 1 2
CHAPTER	2 Specifications & Setup	
Section	2.0 - System Specifications	3 4 6 7 8 8
CHAPTER	3 Operation	
Section	3.0 - How to turn on the Mark 21	9 10 11 12 13 13 15 15 17 17 19 21
CHAPTER	4 Calibration	
Section	4.0 - Why should you calibrate the Mark 21? 4.0.1 - Materials Required for Calibration	22 22 23 23 25 28 28 29 31 33

# TABLE OF CONTENTS (cont.)

	4.3 4.4	- How to set Date and Time	
CHAPTER	5 <b>M</b> a	mintenance & Trouble-shooting	
Section	5.1	- The Readout Module	
APPENDI	( A -	Solubility of Oxygen in Water Exposed to Water-Saturated Air. 39	

# 1.0 What is the Mark 21?

The Martek Mark 21 Portable Dissolved Oxygen Monitor is a completely portable, battery-operated electronic instrument that can be used to determine trace amounts of dissolved oxygen in process streams and/or verify the performance and accuracy of online instrumentation used to measure dissolved oxygen.

The Mark 21 can be calibrated in a laboratory to ASTM (American Standard of Tables and Measurements) or NIST (National Institute of Standards and Technology) qualifications, then carried to the sample stream for direct, on-site monitoring of dissolved oxygen and temperature.

Four ranges of oxygen concentration are provided by the Mark 21 (0-20 ppb, 0-200 ppb, 0-2 ppm, 0-20 ppm), all featuring temperature-compensated dissolved oxygen measurement as well as temperature as a separate parameter.

### 1.1 How can the Mark 21 benefit you?

By eliminating the need for physical grab samples, the Mark 21 provides a more reliable and efficient method for verifying and/or calibrating on-line instrumentation. Its accuracy and resolution allow the operator to detect subtle changes in dissolved oxygen levels that can affect the performance of boilers, condensate polishers, and other deaerated process systems.

In addition, the Mark 21 has the capability of storing monitored data in memory for direct input into any personal computer or printer. This valuable feature allows the operator to maintain a historical record of calibration and performance data for on-line instruments and, with appropriate software, can provide differential analysis of numerous monitoring points. The Mark 21 can even serve as a back-up monitoring system in the event of on-line equipment failure. A 0-1 volt analog output signal indicating dissolved oxygen and the range of measurement is also available from the Mark 21 for hooking up directly to a strip-chart or line recorder.

### 1.2 How to read this Manual

Carefully review all of the material in this Manual before operating the Mark 21. Special attention should be given to the sections on SETTING UP, OPERATION, and CALIBRATION of the instrument and sensor. A MAINTENANCE & TROUBLE-SHOOTING section has been provided to assist you in obtaining the maximum use and value from the Mark 21. While not a prerequisite, a basic knowledge of water chemistry or process control systems is desirable before attempting to operate the Mark 21.

Should you have any questions or comments regarding the Mark 21, its application, or this Manual, please contact us.

### 2.0 Specifications - Measurement

Parameter	<u>Range</u>	<u>Accuracy</u>	Resolution
Temperature	0-50°C	±0.10°C	<u>+</u> 0.01°C
Dissolved Oxygen	0-20.00 ppb 0-200.0 ppb 0-2.000 ppm 0-20.00 ppm	±0.50 ppb * ±2.00 ppb ±0.01 ppm ±0.10 ppm	±0.01 ppb ±0.10 ppb ±0.001 ppm ±0.01 ppm
*At temperatu	re of calibration	on.	

# Specifications - Physical

Case Dimensions:  $7.0 \times 6.5 \times 4.0$  inch

(17.8 x 16.5 x 10.1 cm) LWH

Case Weight: 6 lbs. (2.1 kg)

Power Requirement: 6 volt DC power

120 or 230 volt AC when factory supplied battery charger is used.

supplied bactery charger is used

Recorder Output: RS 232C Serial ASCII or

0-1 volt DC analog output

Sensor Type: Galvanic (platinum/lead)

Sensor Materials: 316 Stainless Steel, Delrin, CPVC,

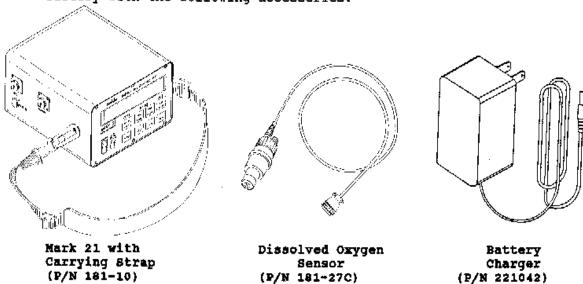
Pure Platinum, Pure Lead

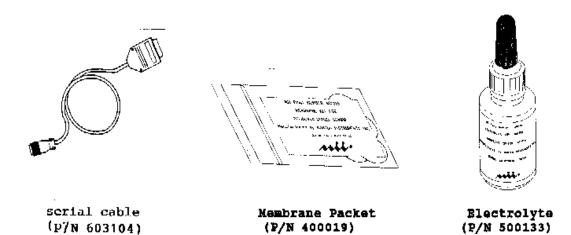
Flow Rate: 100 - 250 ml/min

Operating Pressure: 1 Atmosphere (0 psig)

# 2.1 Accessories and Sensors

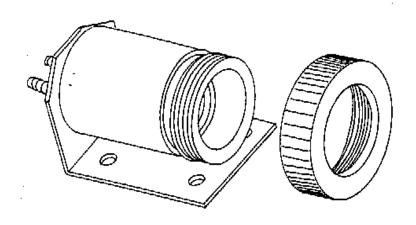
The Mark 21 Dissolved Oxygen Monitor comes complete from the factory with the following accessories:



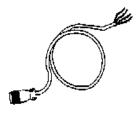


If any of these items are missing, contact the factory immediately.

In addition, a stainless steel flow chamber may also be purchased from Martek for use with the Mark 21 Dissolved Oxygen Sensor:



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



RECORDER CABLE (P/N 603050)

# 2.2 Setting Up The Mark 21

There are two connectors located on the left side of the Mark 21 Readout Module. See Figure 2-1 below for connector location.

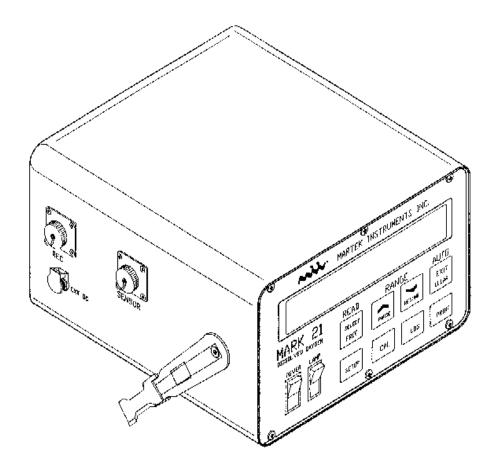


Fig. 2-1 Mark 21 Connector Locations

Each connector is clearly marked with the names SENSOR and REC. The connector labeled REC is for the recorder cable. Below the REC connector is the charging jack for the battery charger plug.

### 2.2.1 Hooking up the Dissolved Oxygen Sensor

To hook up the dissolved oxygen probe, remove the dust cap from the Mark 21 connector labeled SENSOR, insert the plug from the dissolved oxygen probe and secure.

If the optional stainless steel Flow Chamber (P/N 180-51) is to be used, follow the steps outlined below:

- A) Gently remove the vinyl wetting cap from the end of the dissolved oxygen probe.
- B) Remove the plastic locking nut from the Stainless Steel Flow Chamber and carefully insert the dissolved oxygen probe into the flow chamber until it rests flush with the top of the chamber. Try to avoid twisting the probe. Any twisting of the probe should always be clockwise. Place the locking nut over the DO sensor cable and secure firmly onto the chamber. You can now secure the plug from the DO probe to the Mark 21.
- C) To hook up 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.

Refer to the assembly drawing in Figure 2-2 below for a complete layout of the dissolved oxygen sensor and flow chamber.

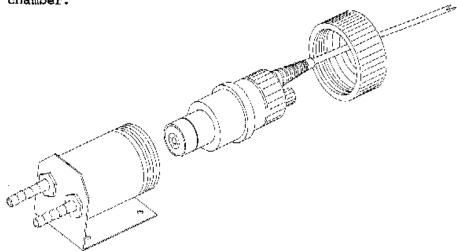


Figure 2-2 Dissolved Oxygen Probe with Flow Chamber

### 2.2.2 Hooking up the Serial Cable

To hook up the Serial Cable, remove the dust cap from the Mark 21 connector marked REC, insert the plug and secure. Next, plug the DB25-style connector into the appropriate port on the computer or printer (See Section 3.2 for baud settings, etc.).

If you desire to hook up the Mark 21 to an external strip-chart or line recorder, Refer to Section 3.2 for recorder output hook-up.

# 2.2.3 Hooking up the Battery Charger

The charging jack is located below the Mark 21 connector marked REC. When charging the Mark 21 internal rechargeable batteries, it is important to follow these two steps:

- A) With the Mark 21 turned off, plug the charging plug into the charging jack them,
- B) Plug the charger into a wall cutlet that carries the same voltage as that listed on the battery charger. The Mark 21 can now be turned on.

The Mark 21 can be operated either on AC or DC power. However, the Mark 21 should be operated only on DC power when calibrating the instrument. This will prevent AC voltage "spikes" from distorting the calibration data stored in memory during calibration.

# 3.0 How to turn on the Mark 21

Figure 3-1 below depicts the Mark 21 front panel with the various keys identified.

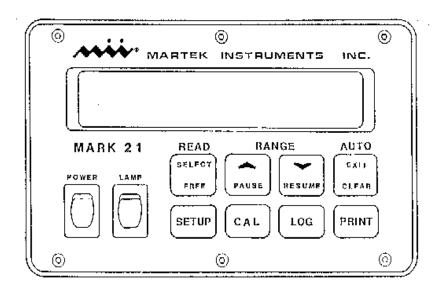


Figure 3-1 Mark 21 Front Panel

Two rocker switches labeled POWER and LAMP are located on the left side of the panel. To turn the Mark 21 on, flip the POWER switch up. The display will activate and momentarily display the following message:



The number in the upper right hand corner refers to the software version inside the Mark 21. If no sensor is hooked up to the Mark 21, the display will indicate 0 ppm. If a sensor is hooked up but not calibrated, the Mark 21 will indicate such. If a sensor is hooked up and calibrated, the Mark 21 will commence monitoring.

For better viewing, flip the rocker switch marked LAMP in the up position. The backlight behind the liquid crystal display will provide a bright display for clearer viewing.

MOTE: The Mark 21 has an operating pariod of approximately & hours <u>without</u> the lamp on. If the lamp is activated, the Mark 21 will operate for approximately 4 hours before requiring recharging,

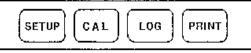
# 3.1 How to use the keys

Several of the keys described below will require that the Mark 21 be calibrated. If the Mark 21 is not 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 and listing the particular function of that key.

An important clue to understanding the operation of the keys is to remember the following:





are **Subject Keys** that allow you to choose which operation you want the Mark 21 to perform.

The Top Row of Keys



are Function Keys that allow you to select a particular value or function in the subject you are in.

# 3.1.1 How to use the SETUP key

The first key you should become familiar with is the **SETUP** key. This key allows you to set the start time and interval for the logging function.

Now, press the yellow **SETUP** key. The display will respond with the following:



The Mark 21 utilizes a menu format that provides you with a set of options from which you can select from. Since the SETUP key has been pressed, the top row of function keys are now in affect. All words and symbols colored yellow are now activated.

To select a particular option, you would press the **SELECT** key. To move the flashing prompt up or down to another option, you would press either the **UP** or **DOWN ARROW** keys. Finally, to exit out of the menu you would press the **EXIT** key which will return the Mark 21 to the monitoring mode. The options presented in the **SETUP** menu are discussed in Section 3.2. For now, simply press the **EXIT** key.

# 3.1.2 How to use the CAL key

The next key you should become familiar with is the CAL key. This key allows you to calibrate temperature, dissolved oxygen, recorder output, and to change date and time. Press the CAL key. The Mark 21 will respond with the following display:



Like the SETUP key, all the letters and symbols colored yellow in the top row of the Function Keys are now in affect. To move the flashing prompt up or down to another option, you would press either the UP or DOWN ARROW keys. To exit out of the menu you would press the EXIT key which will return the Mark 21 to the monitoring mode. Since the options presented in the CAL menu are discussed in Section 3.2, simply press the EXIT key for now.

# 3.1.3 How to use the Log key

The LOG key is one of two keys on the Mark 21 membrane panel outlined in red. When this key is pressed, the words labeled in red on the function keys are in affect. When the LOG key is pressed, the Mark 21 will display the following:



Pressing the LOG key again will return the Mark 21 to the 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 21 is in the logging mode. By pressing any of the function keys, you will be able to pause the logging of data (indicated by the letter "p" in the right-hand corner of the display), resume the logging of data, show the amount of free memory remaining, and clear or erase the data already logged. To stop logging, simply press the LOG key again. The next time you press the LOG key, the Mark 22 will increment to file #02 and start logging data in that particular file. This feature allows you to assign different files for different monitoring points. Refer to Section 3.2 on how to set up the Mark 22 for logging at predetermined times and intervals.

# 3.1.4 How to use the PRINT key

The last key is the **PRINT** key. The **PRINT** key is also outlined in red. When this key is pressed, the words labeled in red on the function keys are in affect. The Mark 21 will always send out the display information to the printer port under normal conditions. Pressing the **PRINT** key will allow the Mark 21 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 ?

By pressing the RESUME key, the Mark 21 will start sending data. 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 all files. Finally, pressing the CLEAR key will increment the Mark 21 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 communicate with the Mark 21 can be found in Section 3.2.

### 3.1.5 How to use the DISPLAY CONTROL KEYS

The last thing you need to know about using the keys on the Mark 21 are the DISPLAY CONTROL KEYS. The display control keys are the same keys used as function keys when the Mark 21 is not in the monitoring mode. The display control keys become activated only when the Mark 21 is monitoring data. The words READ, RANGE, and AUTO are labeled in white above the keys. If the READ key is pressed, the Mark 21 will display either dissolved oxygen and temperature or date, time, and battery voltage. In this way, you can select a particular measurement to be displayed continuously in the Mark 21. The letter "M" will be present in the lower right-hand corner of the display to indicate that the Mark 21 is in the manual mode of presenting data. An example of the READ key being pressed twice is shown in sequence on the next page.

READ

D GL/D3 T G9:56

BATTERY 6.54 V

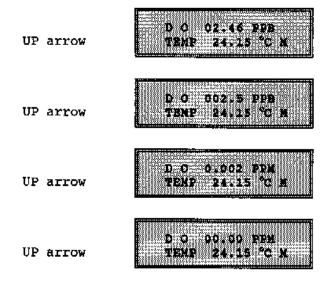
D C 06.75 PPB

READ

TEMP 19:69 C N

The AUTO key is an abbreviation of "AUTORANGE". If the AUTO key is pressed, the Mark 21 will continuously display dissolved oxygen and temperature measurements. However, the auto mode allows the Mark 21 to switch from one particular dissolved oxygen range to another automatically as DO levels rise or fall. The letter "A" will be present in the right-hand corner of the display to indicate that the Mark 21 is in the "autoranging" mode of displaying data.

The two remaining display control keys are located beneath the RANGE inscription. These keys allow you to manually select one of four dissolved oxygen ranges when the Mark 21 is monitoring dissolved oxygen in the "manual" mode. The arrows indicate which direction the dissolved oxygen range can be altered. If the UP ARROW is pressed, the Mark 21 will move the decimal point shown in the display to the right. If the DOWN ARROW is pressed, the Mark 21 will move the decimal point to the left. This feature allows you to select a low and high ppb or ppm range of measurement. An example of the RANGE keys functions are shown below:



Notice that the last display shown indicates a zero amount of dissolved oxygen. This will occur if the parts per million range is selected and the actual dissolved oxygen present in the stream is only a few parts per billion. If the two lower ranges are selected and the DO content exceeds the range displayed, the Mark 21 will simply display "2001" with the decimal point positioned in whatever range the Mark 21 is monitoring in.

Finally, please note that the RANGE keys do not function when the Mark 21 is in the "auto" mode of monitoring data.

# 3.2 Preparation and Operating Conditions

### 3.2.1 When and How to Replace the Membrane

The dissolved oxygen probe is normally shipped from the factory with a membrane in place and electrolyte inside the probe. A vinyl wetting cap is filled with water to keep the Teflon membrane moist when the probe is not being used. Extra caps, membranes, and a bottle of electrolyte (potassium iodide), are also provided.

The dissolved oxygen probe consists of five principle parts each identified in Figure 3-2 below:

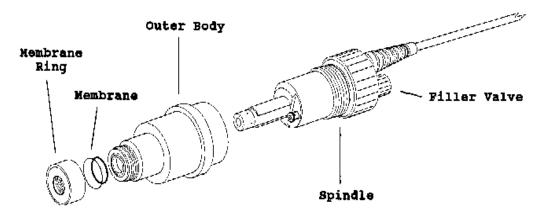


Figure 3-2 Dissolved Oxygen Probe Assembly

The membrane should be changed under the following conditions:

- A) The membrane becomes torn or coated
- B) Air bubbles are visible beneath the membrane
- C) A leak is detected evidenced by salt deposits, sudden changes or drifting readings
- D) Whenever electrolyte is replaced

The following procedure should be used when changing the membrane or replacing the electrolyte.

- 1. Turn the Filler Valve clockwise until it stops.
- Unscrew the black Outer Body counter-clockwise and remove it from the grey Spindle. Empty out any electrolyte still in the Outer Body.
- Unscrew the stainless steel Membrane Ring from the Outer Body and remove the old membrane.
- Place a new membrane in the bottom of the Membrane Ring and place several drops of electrolyte on the membrane.
- Screw the Membrane Ring onto the Outer Body and gently tighten.
- Using the dropper from the electrolyte bottle, carefully fill the four small flow-thru holes located around the platinum tip of the Spindle.
- 7. Fill the Outer Body with electrolyte just above the interior ledge of the Outer Body.
- 8. With the Filler Valve facing up at a 45° angle, screw the Outer Body back onto the Spindle and gently tighten (some electrolyte should flow out of the Filler Valve).
- 9. Gently screw out Filler Valve until it stops.
- Calibrate monitor (See Chapter 4 CALIBRATION)

If the dissolved oxygen probe is not going to be immediately used, fill the vinyl wetting cap with water and place over the Membrane Ring squeezing as much air as possible from the cap.

If the probe is not be used within two weeks, empty the electrolyte out of the probe, dry all of the parts and store the probe in a cool place.

If the probe is to be used with the stainless steel Flow Chamber, review Section 2.2.1 "Hooking Up the Dissolved Oxygen Sensor".

### 3.2.2 Hooking up the Mark 21 to a Sample Stream

To afford maximum accuracy, the Mark 21 Dissolved Oxygen Sensor should be used with the stainless steel Flow Chamber supplied by Martek Instruments. To hook up 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. Heavy-wall Tygon tubing should be used. If the flow chamber is to be permanently mounted on-line, stainless steel swaged fittings with either 1/8 or 1/4 inch stainless steel tubing is recommended.

The flow rate should be maintained between 100 - 250 ml/minute for optimum results. Pressure should not exceed 1 Atmosphere (0 psig). When moving the dissolved oxygen probe from different monitoring points, care should be taken to avoid undue exposure to the atmosphere. Keeping the probe in an anaerobic environment quickens its response time.

### 3.2.3 How to Log data

One of the most convenient features of the Mark 21 is its ability to log monitored data into memory and then down-load this data to either personal computers or printers. This saves the operator the additional chore of writing the data down and then keying the information into a computer for graphic analysis or record storage. In addition, the Mark 21 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 21 for logging, press the **SETUP** key. The Mark 21 will display the following:

STARTING TIME PERIOD # SCAMS

and STARTING TIME will be flashing. Now, press the SELECT key. The Mark 21 will prompt with the following:

START TIME hhmm 24 Kr. Time 1200

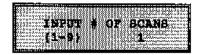
The STARTING TIME is used to set the logging function to begin at a predetermined time. The "hh" stands for the hour and the "mm" stands for the minutes. The time must be inputted as a 24 hour clock, i.e. 1:00 PM would be inputted as 1300, 9:30 AM would be inputted as 0930, etc. Alter the value of the 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 21 will automatically prompt on PERIOD.

When PERIOD is selected, the Mark 21 will display the following:

INPUT LOG PERIOD
IN KINUTES 1-255

Entering any value between 1 and 255 minutes will set the amount of time between recorded readings when logging. Upon completion, the Mark 21 will prompt next to # SCANS.

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



Use the **ARROW** keys to change the number of complete readings the Mark 21 will make during each scan period. Upon completion, the Mark 21 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 21 can hold a total of 371 scans.

For example, if the period between recordings is set for 30 minutes and the number of scans 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.

### 3.2.4 How to Print data

Once data has been logged into the Mark 21, 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 21 requires that you set your computer or printer with the same data flow configuration utilized by the Mark 21. The Mark 21 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 21, connect the recorder cable to the Mark 21 and plug the DB-25 style connector to the appropriate serial port on the computer or printer.

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

PRINTER READY? FILE # 61

At this point, review Section 3.1 again for instructions on how to use the print function keys.

To erase logged data, disconnect the Mark 21 from the computer or printer. Next, press the **PRINT** key, then the **PRUSE** key, and finally, the **RESUME** key. The Mark 21 will display the following messages:

INVALID DATA IN FILE (ABORTING)

CAUTION: ALL LOGGED DATA WILL

of Crased by The Select key

OR SAVED BY THE SXIT 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 21, press the EXIT key. If you wish to erase the data, press the SELECT key.

CAUTION! BRASING LOGGED DATA BRASES ALL DATA IN ALL PILES.

### 3.2.5 How to use the Recorder Output

There are some instances when it is desirable to hook up the Mark 21 to a strip chart or line recorder in addition to or, to substitute for, the Mark 21'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 21 provides a simultaneous 0-1 volt DC recorder output for dissolved oxygen as well as the range the sensor is monitoring in. The analog recorder output for each ranges can be set to a particular voltage scale. Refer to Chapter 4 CALIBRATION, Section 4.2.4, for details.

In order to use the analog recorder output, an optional analog recorder cable (603050) can be purchased and installed in place of the serial cable.

Refer to Figure 3-3 below for wire lead identification.

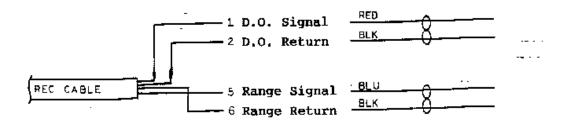


Figure 3-3 Analog Recorder Output Hook-Up

### 4.0 Why should you calibrate the Mark 21?

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 of calibration involves two points: A zero point and a span point. The purpose of these two points is to establish a base value and a value proportionately greater than the base value. The accuracy of an instrument using a two-point calibration is directly related to its ability to maintain linearity over the full scale of measurement.

### 4.0.1 Materials Required for Calibration

The Mark 21 can be calibrated using solutions and gases or electronic circuitry that simulates the properties of gases. In either case, the method chosen should be traceable to ASTM or NIST (National Institute of Standards and Technology - formerly National Bureau of Standards).

If you choose to use solutions and gases for calibration, the following items will be required:

Laboratory-grade thermometer

Temperature Bath (ice and warm water will also work)

Inert Gas (Nitrogen, Helium, or Argon)

Certified Mixed Gases

Electrolyte (Potassium Iodide, 2:1 dilution)

1 mil Teflon membrane (0.75 inch dia.)

The last two items are supplied with the Mark 21, but if you run out you can supply your own electrolyte and membranes or purchase these items from Martek.

If you choose to calibrate temperature and dissolved oxygen electronically, you can eliminate the first four items on the list.

### 4.1 Temperature Calibration

Temperature must always be calibrated before dissolved oxygen since temperature is used to compensate for the porosity of the dissolved oxygen membrane.

Temperature can be calibrated either electronically inside the Mark 21 (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 21 utilizes precision resistors in its circuitry to simulate 0°C and 50°C.

# 4.1.1 Auto Temperature Calibration

To set the instrument up for automatic temperature calibration, press the CAL key until the window display responds with:



Select "TEMP". The display will prompt with:



Select "ZERO". The display will prompt with:



Select "AUTO". After a short delay, the display will prompt with the following message:

HIT KEY W/STABLE +0.0500 VOLT

At this time, the Mark 21 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). The Mark 21 will now wait for you to press the **SELECT** key when the voltage shown on the display has stabilized. After the key has been pressed, the display will prompt with "TEMP AUTO ZERO" then automatically default to "SPAN". At this time, press the **SELECT** key. The display will prompt with:



Select "AUTO". After a short delay, the display will prompt with the following message:



Again, the Mark 21 acts like a voltmeter and shows a varying voltage. An internal relay has switched in the precision resistors simulating a temperature of 50°C. The Mark 21 will now wait for you to press the **SELECT** key when the voltage shown on the display has stabilized. After the key has been pressed, the display will prompt with "TEMP AUTO SPAN" and then automatically default to the calibration menu with the "D.O." option flashing.

### 4.1.2 Manual Temperature Calibration

If standard operating procedure or your own conviction requires that you perform a dynamic calibration, the Mark 21 is capable of being calibrated using an external reference. A temperature bath would be the most useful reference, however, ice and hot water can also be used if a temp bath is not available.

### Temperature Zero Adjustment

Hook up the dissolved oxygen sensor, then press the CAL key and select "TEMP". The display will prompt with the following:



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:



Like the auto temperature calibration, the Mark 21 acts like a voltmeter and shows a varying voltage. The Mark 21 will now wait for you to press the **SELECT** key when the voltage shown on the display has stabilized. At this time, place the dissolved oxygen sensor in a temperature ice bath or a beaker filled with water and crushed ice. At the same time, measure the ice slurry with an accurate temperature thermometer.

NOTE: The freezing point of water does not actually have to be reached for the Mark 21 to determine the sensor's reading at 0°C. As long as the temperature low point is lower than the entire range of measurement, a value above the freezing point of water can be used. For example, if your range of measurement is between 10° and 10°C, then you could callbrate the Mark 21 for temperature using 5° and 40° as your low (zero) and high (span) points.

When the voltage reading on the display has stabilized, normally after several minutes, press the **SELECT** key. The Mark 21 will prompt with the following display:

INP A DIGIT STD. LG TEMP = 00.00

Input the temperature value as a 4-digit number (note the fixed decimal point). 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.

### Temperature Span Adjustments

After the Mark 21 has been zeroed for temperature, it will automatically prompt with the following display:



Select the "SPAN" option. The display will then prompt with:



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

PLACE SENSOR IN HI TEMP STANDARD

Then will ask you to:

HIT KEY W/STABLE +0.7883 VOLT

At this time, place the sensor in a high (span) temperature bath where the temperature is approximately twice the nominal temperature measurement. Again, to accurately measure the water temperature, place a thermometer into the solution. When the voltage displayed has stabilized, press the SELECT key. The Mark 21 will prompt with the following display:

INP 4 DIGIT STD. HI TEMP = 50.00

Input the temperature value as a 4-digit number (note the fixed decimal point). 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. After the last number has been entered, the Mark 21 will automatically respond with the following display:

TEMP D.G. TIME RECORD

and the "D.O." option will be flashing.

### 4.2 Dissolved Oxygen Calibration

The Mark 21 utilizes a galvanic-type dissolved oxygen sensor. Unlike polarographic DO sensors which require a reference voltage to maintain equilibrium, the galvanic sensor utilizes a basic electro-chemical premise: If there is no oxygen, there will be no current flow. Likewise, if oxygen exists, the current produced will be directly proportional to the amount of oxygen present.

In light of this, the Mark 21 dissolved oxygen sensor's zero calibration can be accomplished via the electronics located inside the Mark 21 monitor. This "auto-calibration" feature makes calibration of the sensor easy and consistent.

### Dissolved Oxygen Zero Calibration

### 4.2.1 Auto Zero Calibration Adjustment

After temperature has been calibrated, the dissolved oxygen must be zeroed. Like temperature, two methods, auto zero and manual zero calibration, are available. With the calibration menu in the display and the "D.O." option flashing, press the SELECT key. The display will prompt with the following:



Select the "ZERO" option. The Mark 21 will next display:



Select the "AUTO" option. After a short delay, the Mark 21 will display the following:



Just like the auto temperature calibration sequence, the Mark 21 acts like a voltmeter and shows a varying voltage. An internal relay has switched in the electronics simulating a zero current. The Mark 21 will now wait for you to press the SELECT key when the voltage shown on the display has stabilized. After the key has been pressed, the Mark 21 will briefly display "AUTO = 00.00 ppM", then automatically default to the "SPAN" option.

### 4.2.2 Manual Zero Calibration Adjustment

The dissolved oxygen sensor can be zeroed manually as well. To do so requires an anaerobic environment where the precise amount of oxygen is known. Inert gases are one method of zeroing the dissolved oxygen sensor that is in accordance with ASTM and NIST standards. However, this method is not as convenient as the auto zero calibration procedure previously discussed.

Another method for manually zeroing the dissolved oxygen probe involves using sodium sulfite dissolved in a small beaker of distilled water. While not as accurate as inert gases, it does provide a fairly reliable alternative.

When using sodium sulfite, place a small amount in the vinyl wetting cap, fill with distilled water, and gently place the wetting cap onto the probe taking care to squeeze any excess air out of the cap. Next, place the sensor in a 500 ml beaker of distilled water (containing approximately 1 tablespoon of sodium sulfite dissolved into the water) and proceed with the zero calibration. This method can also be used to test the response of the sensor after all calibrations are completed.

To manually zero the dissolved oxygen probe, make certain the Mark 21 is displaying the calibration menu with the "D.O." option flashing, then press the **SELECT** key. The display will indicate the following:



Select the "ZERO" option. The Mark 21 will next display:



Select the "MANUAL" option. After a short delay, the Mark 21 will display the following messages:

PLACE BENSON IN LO D.O. STANDARD

GELECT RANGE TO ZERO

20 ppM 2 ppM 200ppB 20 ppB

and the "20 ppM" option will be flashing. Unless you absolutely know the exact amount of oxygen present, select the 20 ppm range. Choosing the highest range allows for the least amount of error since any trace amount of oxygen will be shown as zero in the ppm range. Once you press the SELECT key, the Mark 21 will display the following:

HIT KEY W/STABLE -0.0006 VOLT

Again, the Mark 21 acts like a voltmeter and shows a varying voltage. The Mark 21 will now wait for you to press the **SELECT** key when the voltage shown on the display has stabilized. After the key has been pressed, the Mark 21 will briefly display the following:

DAP 4 DIGIT STD. D.D.= 00.00 PPM

Input the dissolved oxygen value as a 4-digit number (note the fixed decimal point). 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. After the last number has been entered, the Mark 21 will automatically respond with the following display:



and the "SPAN" option will be flashing.

### 4.2.3 Manual Dissolved Oxygen Span Calibration

Span calibration of the dissolved oxygen sensor to a known value cannot be done electronically. Therefore, only manual calibration is available. However, there are two methods of span calibration that can be utilized.

One method of span calibration involves the use of mixed gases that have been certified for their oxygen content. These gases are available from certain commercial gas outlets and certification laboratories in small cylinders. The disadvantage of using these gases is their relatively high cost.

Another method of span calibration that is widely accepted is the use of water-saturated air. This method is based on the physical assumption that dry air contains 20.90% oxygen and water-saturated air contains specific levels of oxygen based on temperature and pressure.

If the dissolved oxygen sensor is to be spanned with mixed gases, the sensor will have to be placed in an air-tight container, preferably the stainless steel Flow Chamber provided by Martek.

The following procedures can be used with either method of calibration.

With the Mark 21 displaying the calibration menu, choose the "D.O." and press the **SELECT** key. The display will prompt with the following:



Select the "SPAN" option. The Mark 21 will display the following messages;

PLACE SENSOR IN HI D.O. STANDARD

SELECT RANGE TO CALIBRATE

20 ррм 2 ррм 200ррв 20 ррв

and the "20 ppM" option will be flashing. At this point, if you are using mixed gases, select the range that is appropriate for the value of dissolved oxygen represented by the gas. If you are using the water-saturated air method, press the **SELECT** key to choose the "20 ppM" option. The display will respond with the following:

HIT KEY W/STABLE +1.7534 VOLT

Again, the Mark 21 acts like a voltmeter and shows a varying voltage. The Mark 21 will now wait for you to press the **SELECT** key when the voltage shown on the display has stabilized. After the key has been pressed, the Mark 21 will briefly display the following:

INP 4 DIGIT BTD. D.G.W GB.RG PPM

You will notice that a dissolved oxygen value will already be present in the display. One of the convenient features of the Mark 21 is that it automatically calculates the dissolved oxygen value of water-saturated air for the surrounding temperature and displays

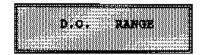
this calculated value in the display. This calculated value is based on measurements made in fresh water at sea-level (760 mm of mercury). For other pressures and varying levels of chloride, consult APPENDIX A, "Solubility of Oxygen in Water Exposed to Water-Saturated Air" at the end of the Manual.

Input the dissolved oxygen value as a 4-digit number (note the fixed decimal point). 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. After the last number has been entered, the Mark 21 will return to the calibration menu and the "TIME" option will be flashing.

### 4.2.4 Analog Recorder Output Calibration

In addition to the RS232 Serial ASCII digital output, the Mark 21 also provides a 0-1 DC analog output (Refer to Section 2.2.2, "Recorder Output Hookup", for details on how to hookup up the recorder cable to a recorder).

There are two recorder output channels available from the Mark 21: Dissolved Oxygen and Range. By selecting a particular channel, the Mark 21 can force its recorder output to zero and full scale so that the external recording instrument can be directly calibrated to the Mark 21's output. With the Mark 21 displaying the calibration menu, select the "RECORD" option. The Mark 21 will display the following:



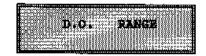
Select the "D.O." option. The display will prompt with:



If you select the "ZERO" option, the Mark 21 will force the analog output for dissolved oxygen to zero volts. At this time, you would adjust the recording instrument to read zero.

If you select the "FULL" option, the Mark 21 will force the analog output for dissolved oxygen to full scale or 1 volt. At this time, you would adjust the recording instrument to read full scale.

To exit from the dissolved oxygen recorder option, press the EXIT key. The Mark 21 will provide the following display:



and the "RANGE" option will be flashing. Pressing the SELECT key provides the following display:



After the zero and full scale analog recorder adjustments for dissolved oxygen have been completed, the range adjustments can be made. The purpose of the range channel is to indicate to the recording instrument the particular range the Mark 21 is monitoring in. The adjustments for the range channel utilize the same procedure as that used for the dissolved oxygen channel. Refer below for the voltage scale and its corresponding range.

Zero Volts for the 20 ppb range 1/3 Scale for the 200 ppb range 2/3 Scale for the 2.0 ppm range Full Scale for the 20 ppm range

# 4.3 How to set Date and Time

The Mark 21 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 CAL key and select the "TIME" option. The Mark 21 will prompt with the following display:

DATE TIME

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

IMPUT mmdd DATE 0101

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 21 will automatically respond with the following display:



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



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 21 will automatically respond with

the following display:

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<b></b>	**************
<b>#</b> Y^Y^Y^Y^? <b>???</b> \$?\$?\$?\$?\$	mioroxoxoxoxo
	***********
B(0) (1) (1)	
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	والمتبالسلاماتين

and the "RECORD" option will be flashing.

### 4.4 Frequency of Calibration

Standard operating procedures for calibration vary with each organization. Martek Instruments recommends calibration of the Mark 21 at least once a month. More frequent calibration may be required if displayed values are unusually high or low given the application, or the application itself changes.

The best results occur when the same method of calibration is used, conducted by the same operator. You can use any of the methods described in this Manual to determine which is the most acceptable and convenient method to employ.

### 5.0 The Readout Module

The Mark 21 monitor is housed in an extruded aluminum case coated and painted to withstand exposure in a marine environment. However, the Mark 21 is not waterproof. 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 21 from exposure.

While the Mark 21 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 21 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.

### 5.1 The Sensor

THe dissolved oxygen sensor consists of a galvanic-style probe (platinum/lead) with an electrolyte consisting of potassium iodide and six feet of shielded sensor cable.

Normally, very little maintenance is required and the sensor can operate for weeks, even months without the need to change the electrolyte or membrane, especially if the sample stream is running in the low ppb range.

However, over time or in a sample stream with a high dissolved oxygen content, lead oxide will form on the lead portion of the Spindle. A possible indication of lead oxide build-up is lower than normal oxygen readings due to less surface area on the lead. The lead strip will appear dull in appearance and possibly have yellow deposits near the edges.

To clean the lead oxide off, simply take a toothbrush or plastic scouring pad (such as Scotch-Brite) and gently scrub the lead with distilled water until it shines brightly. You should also check the platinum tip and the four flow-thru holes (located around the platinum) for dirt or debris. If any exists, simply scrub it away with the toothbrush.

Also make sure that the O-rings used in the Outer Body, Filler Valve, and Flow Chamber are intact and in good condition.

After cleaning the lead, replace the membrane and electrolyte and calibrate the sensor as you normally would.

### 5.2 Trouble-shooting

If maintenance procedures are followed regularly, very little can go wrong with the Mark 21. However, there may be instances where the Mark 21 does not respond in the manner desired. The following section deals with the errors most commonly encountered and offers suggestions to overcome potential problems.

### The Mark 21 fails to respond to any key commands.

ANSWER: Make sure that the Mark 21 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 present, press the RESUME key then the LOG key to disable the logging function.

# 2. The Mark 21 displays "MEM ERROR REDO CALIBRATION" after power is turned on.

ANSWER: The Nicad batteries used for internal memory power in the Mark 21 may not be charged. Charge the Mark 21 for at least 24 hours to allow the batteries to charge up. Make sure the Mark 21 is turned on during charging.

### 3. Dissolved Oxygen readings are unusually high and erratic.

ANSWER: The Teflon membrane may be torn or have a pinhole leak. Re-membrane and fill with fresh electrolyte.

APPENDIX A

Solubility of Oxygen in Water Exposed to Water-Saturated Air\*

ppm CL % NaCL	0	5,000 0.8%	10,000 1.6%	15,000 2.5%	20,000 3.3%
°c		Dissolve	d Oxygen (p)	ρm)	
11	11.1	10.5	9.9	9.4	8.8
12	10.8	10.3	9.7	9.2	8.6
13	10.6	10.1	9.5	9.0	8.5
14	10.4	9.9	9.3	8.8	8.3
15	10.2	9.7	9.1	8.6	8.1
16	10.0	9.5	9.0	8.5	8.0
17	9.7	9.3	8.8	8.3	7.8
18	9.5	9.1	8.6	8.2	7.7
19	9.4	8.9	8.5	8.0	7.6
20	9.2	8.7	8.3	7.9	7.4
21	9.0	8.6	8.1	7.7	7.3
22	8.8	8.4	8.0	7.6	7.1
23	8.7	8.3	7.9	7.4	7.0
24	8.5	8.1	7.7	7.3	6.9
25	8.4	8.0	7.6	7.2	6.7
26	8.2	7.8	7.4	7.0	6.6
27	8.1	7.7	7.3	6.9	6.5
28	7.9	7.5	7.1	6.8	6.4
29	7.8	7.4	7.0	6.6	6.3
30	7.6	7.3	6.9	6.5	6.1

\*At a total pressure of 760 mm mercury. Under any other barometric pressure, the solubility, 8' (ppm), can be obtained from the corresponding value in the Table by the following equation:

$$S' = S[(P - p) / (760 - p)]$$

in which 8 is the solubility at 760 mm (29.92 inches) and p is the pressure (mm) of saturated water vapor at the temperature of the water. For elevations less than 1,000 meters (3,000 ft.) and temperatures below 25°C, p can be ignored. The equation then becomes:

$$8' = 8(P / 760) = 8(P / 29.92)$$

Dry air is assumed to contain 20.92% oxygen. (Calculations made by Whipple & Whipple, 1911.)

# Oxygen Solubility in water saturated with air (mg/l)

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_	5, 12 5, 12 5, 13	11 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	00 - Nu	00000 00000	5 7.105 5 7.105 6 7.105	7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	33053	10,19 26 9,95 15 9,51	4641	26 12.45 10.45 10.45 10.45	0 225
	5.24 5.24 5.07	2000 2000 2000 2000 2000 2000 2000 200	5.05	6 6 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	7.754	7 6.28 5 7.92 7.92 7.93	000000	9 10.26 5 10.00 5 10.00 1 9.59 1 9.37	3 .1	10.00 10.00	344
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	36434 244 244	5.07 5.07 5.07 5.07 5.07	5.5.7.7. 5.5.7.7.4. 5.5.7.7.4.4.	7.47 7.35 7.41 7.41	8.45 8.00 7.26 7.73	8 6 7 7 4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	4.00 4.00 4.00 4.00 4.00	50.55 1955 1955 1955	######################################	7.00 7.00 7.00 7.00 7.00 7.00 7.00 7.00	245
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	30931 30931	0 0 0 0 0 10 0 4 0 0 10 0 0 0 4	7,18 9,06 6,85 6,74	49499 <b>44</b> 569	8.48 8.19 8.05 7.91	9.30 9.12 6.95 6.79	10.25 10.25 9.87 9.67 9.67	11,51	13,00 12,67 12,35 12,07	15.00 10.00	795