ISO-H2S-2

Hydrogen sulfide sensor for use with
WPI Free Radical Analyzers

INSTRUCTION MANUAL

Serial No._____

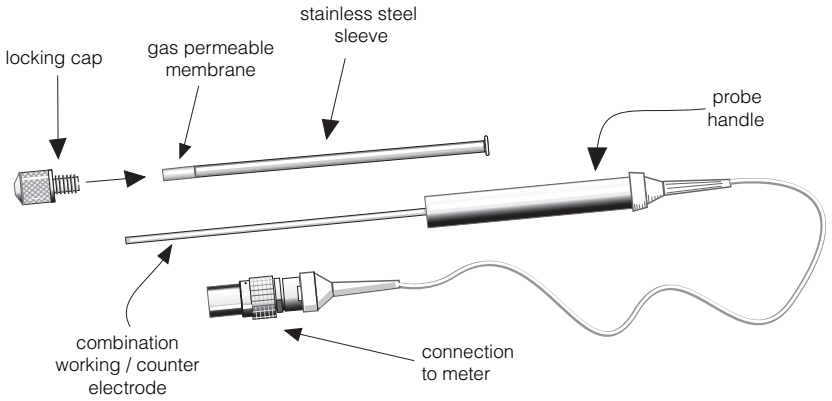
102711

World Precision Instruments

Introduction

When the **ISO-H2S-2** sensor is not being used it should be left connected to the free radical analyzer with the tip immersed in distilled water. The basic structure of the ISO-H2S-2 sensor is quite simple (see figure). It consists of an internal H_2S—sensing working/reference electrode combination. This electrode fits inside a disposable protective stainless steel sleeve (WPI **#600016**) which must contain fresh electrolyte (WPI **#100084**) and is separated from the external environment by a gas-permeable polymeric membrane covering the end of the stainless sleeve. The other end of the sleeve is flanged. The locking cap is used to attach the sleeve to the probe handle.

When the sensor is fully assembled (*i.e.*, with locking cap and sleeve in place) the internal electrode should be seen to press gently against the polymeric membrane, which will then be slightly stretched. This ensures that the electrolyte diffusion layer will be as thin as possible, which is necessary to minimize sensor response time. Once a membrane is stretched, it is permanently deformed and cannot usually be reused if the sleeve is removed from the electrode. Four additional membrane sleeves (WPI **#600016**) accompany the ISO-H_2S-2 in the start-up kit (WPI **#600015**), together with a **MicroFil™** electrolyte filling needle (WPI **#MF28G67-5**) and 1 mL syringe. With proper care and by following the instructions below a membrane sleeve should last more than one month.



Cleaning the Membrane

The membrane sleeve itself requires very little maintenance. The primary concern is to avoid damage to the membrane and to keep it as clean as possible. After each use, clean the membrane by immersing the tip in distilled water for 20-30 minutes to dissolve salts and remove particles which may have accumulated on the membrane. If the probe was used in a protein-rich solution, the membrane tip should first be soaked in a protease solution for several minutes to remove protein build-up, and then in distilled water. Enzymatic detergent (*e.g.*, Enzol, WPI **#7363**) can also be used. The membrane sleeves can also be sterilized chemically using an appropriate disinfectant (*e.g.*, Cidex, WPI **#7364**). Accumulated organic matter can be removed by briefly immersing the tip in an acid or base solution (at times both may be necessary) for 10 seconds. A good indication of a dirty membrane sleeve is a sluggish response or an unusually low sensitivity. If these problems are not rectified by the above cleaning procedures then the membrane sleeve should be replaced. The probe cannot be used in organic solvents.

Replacing a Membrane Sleeve

All membrane sleeves will eventually have to be replaced by the user. The procedure for doing so is simple and straightforward.

1. Unscrew the locking cap from the handle.

2. Hold the stainless steel sleeve and remove it with the locking cap from the internal electrode assembly, being careful not to bend the electrode assembly when doing so.

3. Rinse the internal electrode with distilled water (particularly the tip) and let it soak for at least 15 minutes. Be careful not to let water get into the handle.

4. Gently dry the electrode with a soft tissue (Kimwipes). Be sure to dry thoroughly the flat surface at the tip of the electrode. After drying, the current should stabilize fairly quickly to a low value (*e.g.*, 0 - 20 pA). If this occurs then the electrode is in good working order.

5. If the electrode is not clean, repeat steps 3 and 4. If necessary the ISO-H2S-2 can be rejuvenated using WPI **#JUV** to restore sensitivity of an old electrode (contact WPI for details).

6. Remove the locking cap from the old sleeve and gently slide it onto the new replacement sleeve.

7. Dip the internal electrode 1-2 cm into the electrolyte provided in the ISO-H2S-2 start-up kit; the current should go off scale during this. Using the **MicroFil™** nonmetallic syringe needle (WPI #**MF28G67-5**) and 1 mL plastic syringe, supplied with the replacement sleeve kit, inject approximately 100 microliters of electrolyte directly into the new sleeve. The MicroFil supplied should be less than the length of the sleeve, so that it will not puncture the delicate membrane at the tip of the sleeve during injection. If the MicroFil is longer than the sleeve it can be cut to the correct length.

8. Slowly and smoothly insert the electrode into the sleeve, and screw the locking cap into the handle. The electrode should be observed to press gently against the membrane.

9. The current displayed on the meter at this time will be high or off scale.

10. Suspend the tip of the new assembled probe into distilled water.

11. After 10-15 minutes the current should no longer be off scale and will gradually decrease with time. It may take several hours for the sensor current to reach a low stable value, at which time it will be ready for use.

12. The integrity of the new membrane can be determined by immersing the probe tip into a strong saline solution (1 M). If the current observed, after a few minutes in the saline solution, increases dramatically or is off scale then the membrane integrity is not good and a new membrane will have to be fitted.

13. When the ISO-H2S-2 is not being used it should be stored with the tip suspended in distilled water.

Additional membrane kits (WPI **#600016**) may be purchased separately.

Calibration of H_2S sensor (ISO-H2S-2)

1. **Preparation of Na_2S stock solution:** Dissolve 5 mg EDTA in 100 mL DI water (18 mΩ) in 100 mL volumetric flask. Purge the solution vigorously with argon gas for 15 minutes. Then weigh 48.0 mg sodium sulfide (Na_2S .9 H_2O) (reagent plus, 99.99+% from Sigma) and dissolve it in the solution under argon atmospheres. Then seal the flask with a rubber stopper. The solution is 2.0 mM Na_2S. Keep the solution in darkness and refrigerate at 2-8 °C.

2. Connect the H_2S sensor into a channel of an Apollo 4000™, Apollo 1000™, or TBR4100. Set the poise voltage to +150 mV. **For the Apollo 4000**, this is accomplished in the software. Use the custom option to manually set the voltage to 150mV (see page B8 in the manual). **For the Apollo 1000** select poise from the main menu and set it to "internal" (see page 10 in the manual). Then adjust the poise voltage using a small screwdriver to turn the "poise adjust" potentiometer on the front panel. **For the TBR4100**, simply set the "probe select' control on the front panel to H_2S. Let the sensor polarize for several hours. **NOTE:** Full polarization may take up to 12 hours.

3. Place 20 mL PBS buffer solution (pH 7.2, 0.05 M) in 20 mL vial (supplied in the calibration kit), drop a small stirring bar into the PBS solution and place the vial on a magnetic stirring plate. Immerse the H_2S sensor into this solution and allow background current to stabilize for few minutes. As soon as the background current becomes stable, start recording.

4. Using a pipette, sequentially inject four aliquots of Na_2S solution at 5 µL, 10 µL, 20 µL and 40 µL into the vial containing PBS buffer. The current will rapidly increase upon addition of the first aliquot and will reach a plateau within a few seconds. Inject the second aliquot of 10 µL as soon as the first signal reaches plateau. In the same way, inject the third and fourth aliquots. This will correspond to 0.5 µM, 1 µM, 2 µM, and 4 µM final concentrations, respectively.

5. Note — the volume of injected calibration aliquots may be adjusted accordingly to accommodate the anticipated concentration range for the experiment.

6. Use the recorded data to construct a linear calibration curve of concentration vs. current.

7. As H_2S sensor is sensitive to salinity and temperature and H_2S solubility to temperature, it is suggested that calibration and subsequent measurements should be performed in solution with the same temperature and salinity.

***The H_2S sensor measures the dissolved H_2S gas, which is only one component of the total sulfide equilibrium system. The total sulfide concentration $[S^2] = [H_2S] + [HS^-] + [S^{2-}]$, so the H_2S concentration can be calculated by: $[H_2S] = [Na_2S]/\{ 1+ K^1/[H^+] + K_1K_2/[H^+]\}$

For K_1 and K_2, (pK_1= 6.89, pK_2 =19) see

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Warranty

WPI (World Precision Instruments, Inc.) warrants to the original purchaser that this equipment, including its components and parts, shall be free from defects in material and workmanship for a period of 30 days from the date of receipt. WPI's obligation under this warranty shall be limited to repair or replacement, at WPI's option, of the equipment or defective components or parts upon receipt thereof f.o.b. WPI, Sarasota, Florida U.S.A. Return of a repaired instrument shall be f.o.b. Sarasota.

The above warranty is contingent upon normal usage and does not cover products which have been modified without WPI's approval or which have been subjected to unusual physical or electrical stress or on which the original identification marks have been removed or altered. The above warranty will not apply if adjustment, repair or parts replacement is required because of accident, neglect, misuse, failure of electric power, air conditioning, humidity control, or causes other than normal and ordinary usage.

To the extent that any of its equipment is furnished by a manufacturer other than WPI, the foregoing warranty shall be applicable only to the extent of the warranty furnished by such other manufacturer. This warranty will not apply to appearance terms, such as knobs, handles, dials or the like.

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• Inspect all shipments upon receipt. Missing cartons or obvious damage to cartons should be noted on the delivery receipt before signing. Concealed loss or damage should be reported at once to the carrier and an inspection requested. All claims for shortage or damage must be made within 10 days after receipt of shipment. Claims for lost shipments must be made within 30 days of invoice or other notification of shipment. Please save damaged or pilfered cartons until claim settles. In some instances, photographic documentation may be required. Some items are time sensitive; WPI assumes no extended warranty or any liability for use beyond the date specified on the container.

• WPI cannot be held responsible for items damaged in shipment en route to us. Please enclose merchandise in its original shipping container to avoid damage from handling. We recommend that you insure merchandise when shipping. The customer is responsible for paying shipping expenses including adequate insurance on all items returned.

• Do not return any goods to WPI without obtaining prior approval and instructions (RMA#) from our returns department. Goods returned unauthorized or by collect freight may be refused. The RMA# must be clearly displayed on the outside of the box, or the package will not be accepted. Please contact the RMA department for a request form.

• Goods returned for repair must be reasonably clean and free of hazardous materials.

• A handling fee is charged for goods returned for exchange or credit. This fee may add up to 25% of the sale price depending on the condition of the item. Goods ordered in error are also subject to the handling fee.

• Equipment which was built as a special order cannot be returned.

• Always refer to the RMA# when contacting WPI to obtain a status of your returned item.

• For any other issues regarding a claim or return, please contact the RMA department.

Warning: This equipment is not designed or intended for use on humans.

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