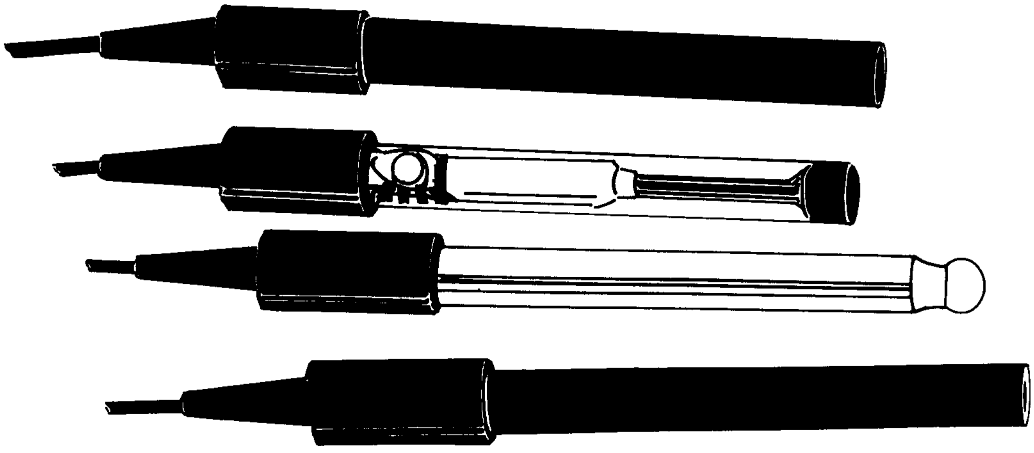
ISE-8750

Carbon Dioxide Gas-Sensing Electrodes



OMEGA®

An OMEGA Technologies Company

Operator's Manual
M783/0892

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SECTION 1 GENERAL DESCRIPTION

The OMEGA® Carbon Dioxide Gas-Sensing Electrode is used to quickly, simply, accurately, and economically measure carbon dioxide, carbonate, and bicarbonate in aqueous solutions. With a flow-thru cap, the electrode can be used in flow-thru applications.

SECTION 2 PREPARATION FOR MEASUREMENT

2.1 REQUIRED EQUIPMENT AND SOLUTIONS

1. A pH/mV meter or an ion meter, either line operated or portable.
2. Semi-logarithmic 4-cycle graph paper for preparing calibration curves when using the meter in the mV mode.
3. A magnetic stirrer.
4. Deionized or distilled water for solution preparation.
5. Carbon Dioxide Electrode, ISE-8750.
6. Sodium Bicarbonate Standard, 0.1M $NaHCO_3$, ISE-8750-S1. To prepare a 0.1M $NAHCO_3$ solution, dissolve 8.40 grams of reagent-grade $NAHCO_3$ in 500 mL of distilled water in a 1 liter volumetric flask and fill to the mark with distilled water.
7. Carbon Dioxide Standard, 1000ppm CO_2, ISE-8750-S2. To prepare a 1000 ppm CO_2 solution, dilute 227 mL of the 0.1M $NaHCO_3$ standard, ISE-8750-S1, to 1^2 in a volumetric flask.
8. Carbon Dioxide Buffer Solution, ISE-8750-R1. This buffer is used to adjust the pH of the solution to the operating range of the electrode. To each sample and standard solution, add 5 mL of carbon dioxide buffer. To prepare carbon dioxide buffer from your own stock, fill a 1 liter volumetric flask about full with distilled water, add 294 grams of $NaC_6H_5O_7.2H_2O$ (sodium citrate dihydrate), dissolve by swirling the flask, and fill to the mark with distilled water. Adjust the pH of the solution to 4.5 with concentrated HCl.
9. Sodium Chloride Solution, 0.1M NaCl, ISE-8770-S1. This solution is used to store the electrode. To prepare 0.1M NaCl, fill a 1 liter volumetric flask about half full of distilled water, add 5.8 grams reagent-grade sodium chloride, swirl the flask to dissolve NaCl, and dilute to the mark with distilled water.
10. Carbon Dioxide Electrode Filling Solution, PHFS-8750.

2.2 ELECTRODE ASSEMBLY

The electrode is shipped dry. Before using it, unscrew the big cap (at the top), remove the inner glass electrode from the outer body, and soak the inner glass electrode in internal filling solution for at least two hours. Fill the outer body half full with internal filling solution, place inner glass electrode into the outer body, and screw on the big cap till fingertight. After assembly, gently feel the membrane with fingertip to make sure that the inner glass electrode touches the membrane tightly. While using the electrode place the assembled electrode in an electrode holder with a 20° angle from the vertical to avoid trapping air bubbles at the bottom of the electrode.

NOTE

> Do not be alarmed if white crystals form at end of electrode. This is normal with pH electrodes. These crystals are potassium chloride. Remove the shipping cap and rinse with distilled water to dissolve the crystals.

2.2.1 Checking the Membrane

A small hole of any size on the membrane or the breakage of the membrane causes total failure of the measurements. It is recommended to check the membrane on every newly assembled electrode. A simple and effective way is available: connect a newly assembled electrode to a pH/mV meter and place the electrode into DI water. Record the reading after stirring the DI water for about 15 minutes. Add proper buffer solution (see Required Solutions) to that DI water. A drastic change on the reading indicates the damage of the membrane.

2.2.2 Changing the Membrane

Unscrew the small cap (at the bottom) from the outer body, and remove the membrane base out from the small cap with a screw-driver. Cut the O-ring which wraps the membrane onto the base; and discard the old membrane. Lay a piece of new membrane (20mm x 20mm) onto the base. Then, put a small O-ring (supplied) on the top of the cone-shaped membrane mounting tool, and slide the O-ring down to the bottom of the cone. Place the cone with the O-ring on it onto the membrane, gently slide the O-ring into the groove on the base to wrap the membrane. Do not push the cone too hard while sliding down the O-ring into the groove, or the membrane may be damaged. Trim the excessive membrane, screw the base into place, and re-assemble the electrode.

2.3 CHECKING THE ELECTRODE SLOPE

The electrode slope is the change in potential (mV) observed when the concentration changes by a factor of ten. The electrode slope should be checked on a daily basis.

To check the slope:

1. Measure out 45 mL of distilled water and 5 mL of carbon dioxide buffer. Add both to a 100 mL beaker, place on the magnetic stirrer, and begin stirring at a constant rate. Lower the electrode tip into the solution and make sure the meter is in the mV mode.

2. Pipet 0.5 mL of the 0.1M or 1000 ppm standard into the solution. Adjust the calibration control (offset adjustment) to zero.

3. Pipet 5.0 mL of the 0.1M or 1000 ppm standard into the solution. If the electrode is operating properly, a reading of 56^{O}2mV should appear on the meter, assuming the solution temperature is between 20° and 25°C. If the change in potential is not within this range, see Troubleshooting section.

2.4 ELECTRODE STORAGE

If storing the carbon dioxide electrode overnight or over the weekend, immerse the tip of the electrode in the 0.1M NaCl storage solution. For longer periods of time, completely disassemble the electrode, rinse the inner body, the outer body, and the cap with distilled water. After drying, reassemble the electrode without filling solution.

2.5 SAMPLE STORAGE

Samples should be measured immediately after preparation or collection, if possible. Wait only long enough for temperature equilibrium between sample and electrode. At 25°C, in an open 150 mL beaker, carbon dioxide diffuses out of an acidic solution at a rate of about 3%/minute with stirring and at a rate of about 0.5% without stirring. The loss of CO_2 increases with increasing temperature.

If samples cannot be measured immediately, add 10M NaOH to make them slightly alkaline (pH 8-9) and store them in tightly capped vessels to prevent infusion of CO_2 from the air. Prior to measurement, acidify these stored samples with carbon dioxide buffer.

The amount of 10M NaOH needed to adjust the pH to the alkaline range will depend on the sample's buffering capacity and the initial pH of the sample. If the samples contain less than 100 ppm CO_2 , collect in a stoppered glass bottle, filling completely and capping tightly to prevent CO_2 to escape. Do not add NaOH, since carbonate is usually present in the base as a contaminant. If the sample contains more than 100 ppm CO_2, is slightly acidic and unbuffered, adding 1 mL of 10M NaOH per 100 mL of sample will suffice.

SECTION 3 MEASUREMENT PROCEDURE

3.1 MEASURING HINTS

To minimize carbon dioxide loss from samples:

1. Samples should be measured immediately after collection.
2. Samples should be stored if immediate measurement is not possible.
3. The ratio of surface area to volume in the beaker should be minimized.
4. Beakers containing the samples or the standard should be kept covered between measurements.
5. Carbon dioxide buffer should be added just before measurement.
6. Samples, standards, and electrode must be at the same temperature.
7. Rinse the electrode with distilled water between measurements.
8. Stir samples and standards using magnetic stirrer.
9. Use an insulating material, such as gauze or styrofoam between the beaker and the magnetic stirrer to minimize heat transfer from the magnetic stirrer to the solution in the beaker.

3.2 SAMPLE REQUIREMENTS

Standards and samples must be at the same temperature, since a 1^o difference in temperature will result in a measurement error of approximately 2%. All samples must be aqueous.

Carbon dioxide buffer must be added to standards and samples before measurement. When the buffer solution is added, all standards and samples should be in the range of pH 4.8 to 5.2. In this range, all bicarbonate and carbonate species are converted to carbon dioxide and all interferences are minimized. Highly basic, highly acidic, or buffered samples must be adjusted to pH 4.8 to 5.2 before the carbon dioxide is added, since the buffering capacity of the acid buffer is limited.

Adding the buffer solution adjusts the total level of dissolved species in solution to 0.4M. The sample should be diluted before measurement if the total level of dissolved species is greater than 1M after the addition of the carbon dioxide buffer. For a further explanation, see Section 4.4.

3.3 UNITS OF MEASUREMENT

Measurement of carbon dioxide can be expressed in units of moles/liter, ppm carbon dioxide, ppm calcium carbonate, or other convenient concentration unit. Table 3-1 lists conversion units.

TABLE 3-1
Concentration Unit Conversion

Moles/liter	ppm CO_2	ppm $CaCO_3$
10^{-2}	440.0	1000:0
10^{-3}	44.0	100 0
10^{-4}	4.4	10.0

3.4 MEASUREMENT PROCEDURE

1. Using serial dilution of the 0.1M or of the 1000 ppm standards prepare 10^{-2} (1000 ppm), 10^{-3}M (100 ppm), and 10^{-4} (10 ppm standards).

2. Put 50 mL of the 10^{-3} M (100 ppm) standard into a 100 mL beaker, add 5 mL of carbon dioxide buffer, and stir continuously, using the magnetic stirrer. Immerse the tip of the electrode in the solution. Set the meter in the mV mode. Adjust the calibration control (offset adjustment) to zero.

3. Put 50 mL of the 10^{-2} M (1000 ppm) standard into a 100 mL beaker, add 5 mL of carbon dioxide buffer, and stir continuously. After rinsing the electrode, immerse the tip in the solution. When the reading has stabilized, record the value.

4. Put 50 mL of the 10^{-4} M (10 ppm) standard into a 100 mL beaker, add 5 mL of carbon dioxide buffer, and stir continuously. After rinsing the electrode, immerse the tip in the solution. Record the value when the reading has stabilized.

5. Using standard semi-logarithmic paper, plot the mV value (linear axis) vs. concentration (log axis). A typical calibration curve appears in Figure 3-1.

6. Measure 50 mL of sample, put it into a 100 mL beaker, and add 5 mL of carbon dioxide buffer. Place the beaker on the magnetic stirrer and begin stirring the solution. Rinse the electrode and immerse the tip in the solution. Wait until the reading stabilizes and record the value. Using the calibration curve, determine the sample concentration.

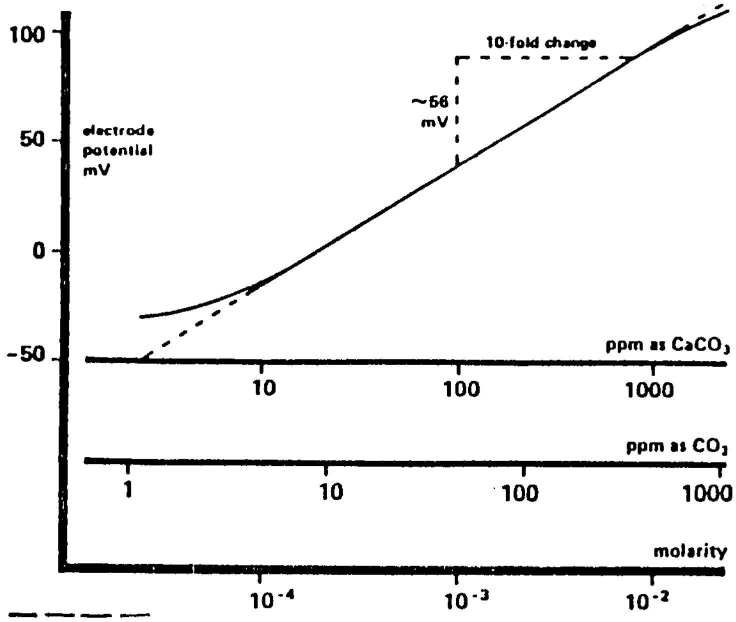


Figure 3-1. Typical Response of Carbon Dioxide Electrode

SECTION 4 ELECTRODE CHARACTERISTICS

4.1 ELECTRODE RESPONSE

Electrode potential response to carbon dioxide concentration is a straight line over two decades of concentration (5×10^{-4} M to 2×10^{-2} M) with a slope of about 56mV per decade, when plotted on semi-logarithmic paper. Refer to Figure 3-1.

For carbon dioxide concentration above 5×10^{-4}M, the electrode exhibits good time response (95% of total mV reading in one minute or less). Response times are longer below this value and carbon dioxide loss to air may become a source of error. The partial pressure of carbon dioxide in solution is greater than normal atmospheric partial pressure of carbon dioxide above 2×10^{-2} M, resulting in a loss of carbon dioxide to air. Dilute samples above 2×10^{-2} M in carbon dioxide concentration before measurement. Figure 4-1 indicates the time response of the Carbon Dioxide Gas-Sensing Electrode to changes in the carbon dioxide concentration.

4.2 INTERFERENCES

Certain volatile weak acids are potential electrode interferences. Concentrations of these interfering species that cause 10% error at 10^{-3} M (100 ppm $CaCO_3$ or 44 ppm CO_2) and at a pH of 4 and 5 are listed in Table 4-1.

TABLE 4-1
INTERFERENCE LEVELS - 10% ERROR AT 10^{-3}M CO_2

Interferences	pH 4	pH 5
HCOOH (formic acid)	7.5×10^{-3}M	2.0×10^{-2}M
CH_3COOH (acetic acid)	3.6×10^{-3}M (0.22g/100mL)	6.2×10^{-3}M (0.37g/100mL)
HSO_3^-(SO_2) (sulfur dioxide)	7.5×10^{-4}M (48 ppm)	5.0×10^{-3}M (320 ppm)
NO_2-(NO_2) (nitrogen dioxide)	5.3×10^{-4}M (24 ppm)	3.5×10^{-3}M (160 ppm)

4.3 THEORY OF OPERATION

A gas-permeable membrane is used to separate the sample solution from the electrode internal solution in the Carbon Dioxide Gas-Sensing Electrode. Dissolved carbon dioxide in the sample solution permeates the membrane until an equilibrium is reached between the partial pressure of the CO_2 in the internal filling solution and the partial pressure of the CO_2 in the sample solution. The partial pressure of carbon dioxide in any given sample will be proportional to the concentration of carbon dioxide.

Diffusion across the membrane affects the level of hydrogen ions in the internal filling solution:

$$(1) \quad CO_2 + H_2O \qquad H^+ + HCO_3^-$$

The relationship between the hydrogen ion, the bicarbonate ion, carbon dioxide, and water is given by the equation:

$$(2) \quad \frac{[H^+]\,[HCO_3^-]}{[CO_2]} = \text{constant}$$

The bicarbonate ion level can be considered constant since the internal filling solution contains a high level of sodium bicarbonate:

$$(3) \quad [H^+] = [CO_2] \times \text{constant}$$

The Nernst equation shows the relationship between the potential of the pH sensing element and the hydrogen ion concentration:

$$(4) \quad E = E_o + S \log [H+]$$

where
E = measured electrode potential
E_o= reference potential (a constant)
$[H+]$ = hydrogen ion concentration
S = electrode slope (see Section 2.3)

Because the hydrogen ion concentration is directly related to the carbon dioxide concentration (see (3) above), electrode response to carbon dioxide is also Nernstian:

$$(5) \quad E = E_1 + S \log [CO_2]$$

The reference potential, E_1, is partly determined by the internal reference element that responds to the fixed level of chloride in the internal filling solution.

A weak carbonic acid solution is formed when carbon dioxide reacts with water:

$$CO_2 + H_2O \text{ --> } H_2CO_3$$

Carbon dioxide exists as bicarbonate and carbonate in basic solutions:

$$CO_2 + OH^- \text{ --> } HCO_3^-$$

$$Co_2 + 2OH^- \text{ --> } CO_3^{-2} + H_2O$$

The solution's pH governs the amount of carbon dioxide present in the form of carbonate and bicarbonate ions.

Acetic acid, formic acid, nitrogen dioxide, and sulfur dioxide interfere significantly with the electrode's measurement of carbon dioxide in the CO_2 form below a pH of 5. At a pH of 5, essentially all the carbon dioxide in solution is in the C02 form.

The pH is held between 4.8 and 5.2 by the carbon dioxide buffer used in carbon dioxide determinations and converts the carbonate and bicarbonate to the CO_2 form:

$$2H^+ + CO_3^{-2} \dashrightarrow H_2O + CO_2$$

$$H^+ + HCO_3^- \dashrightarrow H_2O + CO_2$$

The total amount of carbon dioxide is then measurable in the solution.

The concentration of carbon dioxide in solution is directly proportional to the partial pressure of carbon dioxide over the solution. Henry's Law is used to describe this relationship:

$$K_h = \frac{[CO_2]aq}{PCO_2} = 1.26 \times 10^{-4} \text{ moles/liter at } 25°C$$

where $[CO_2]$ is the concentration of CO_2 in solution, PCO_2 is the partial pressure of CO_2, and K_h is Henry's constant, which varies with solution temperature.

4.4 EFFECT OF DISSOLVED SPECIES

One common substance that is a potential electrode interference is water vapor. The concentration of the internal filling solution under the membrane is changed when water, in the form of water vapor, moves across the electrode membrane. These changes will be seen as electrode drift. If 1) the total level of dissolved species in solution - the osmotic strength - is approximately equal to that of the internal filling solution and 2) the sample and electrode temperatures are the same, water vapor transport is not a problem.

Samples of low osmotic strength are automatically adjusted to the correct level through addition of carbon dioxide buffer. If samples have osmotic strengths greater than 1M, they should be diluted before measurement. However, this dilution should not reduce the carbon dioxide level below 10 M. If dilution is not possible, for the reason mentioned, the sample can be measured by adjusting the osmotic strength of the internal filling solution. The total level of dissolved species in the internal filling solution may be adjusted by adding 0.425 grams of reagent-grade sodium nitrate ($NaNO_3$) to 10 mL of internal filling solution.

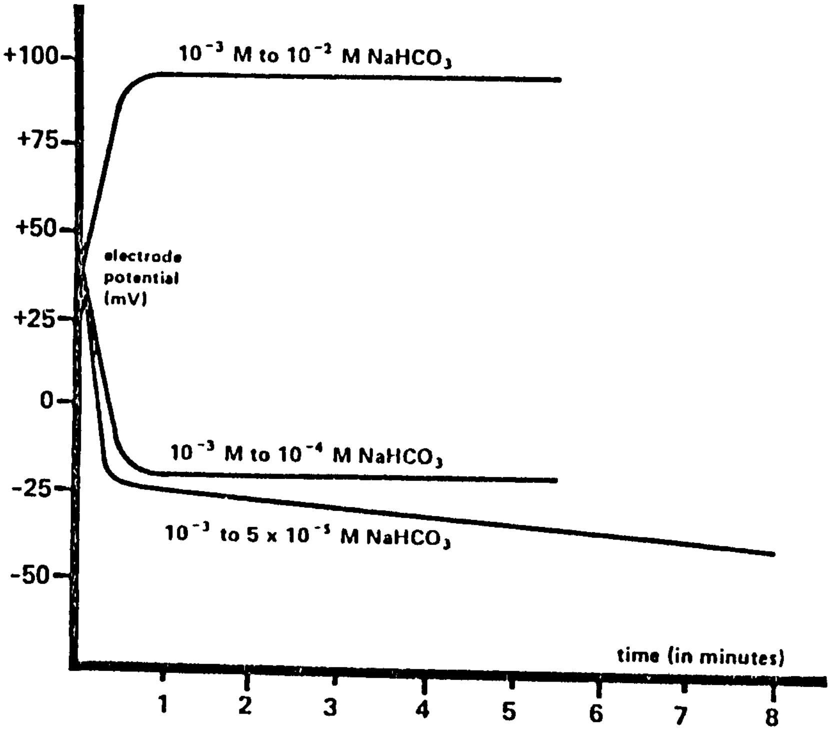


Figure 4-1. Typical Electrode Response to Step Changes in Carbon Dioxide

4.5 APPLICATIONS

4.5.1 Wines and Carbonated Beverages

Wines and carbonated beverages have a high (greater than 2 x 10^{-2} M or 880 ppm) carbon dioxide concentration. As a result, samples are measured after dilution. When samples are collected, they must be made basic immediately or CO_2 will escape into the atmosphere (see Sample Storage section). After diluting the sample, it must be acidified with carbon dioxide buffer. The carbon dioxide concentration is determined by direct measurement (see Measurement section). Finally, the original carbon dioxide concentration is calculated, taking the dilution factors into account.

The required equipment and solutions are found in Section 2. In addition, Sample Adjustment Solution (10M NaOH), freshly prepared to eliminate carbonate contamination, is required.

To measure a sample:

1. Calibrate the meter according to the directions in Section 3.

2. Add enough Sample Adjustment Solution to adjust the pH of the sample greater than pH 10. Add the 10M NaOH to the sample immediately when the sample container is opened.

3. To a 100 mL volumetric flask, quantitatively transfer 10 mL of the alkaline sample to the flask and dilute to the mark with distilled water. After agitating the flask to mix the contents, transfer the solution to a 150 mL beaker.

4. Place the beaker on the magnetic stirrer and begin stirring the sample. Add 10 mL carbon dioxide buffer. Place the tip of the electrode in the solution and record the mV reading.

5. Using the following formula, determine the carbon dioxide concentration:

$$Cs = \frac{10\ V_s + V_{Na}}{V_s}\ Cm$$

where Cs = sample concentration
Vs = sample volume before adding NaOH
V_{Na}= volume of NaOH
Cm = measured concentration

Example: Assume 12 mL of 10 NaOH was added to 360 mL of sample. The total volume would then be 372 mL. Add 10 mL of this mixture to a 100 mL volumetric flask. Dilute to the mark with distilled water. By taking into account the addition of base to the sample and the sample dilution, the measured concentration is related to the actual sample concentration:

$$Cs = \frac{10 \quad 360 + 12}{360} \quad Cm$$

$$Cs = 10.33 \ Cm$$

4.5.2 Groundwater

Free CO_2, bicarbonate and carbonate -the total amount of carbonate- in groundwater or seawater, will depend on location, temperature, depth, and pH. Using the measurement procedure in the Measurement section, the carbonate level in groundwater or seawater can be measured. Grab samples taken in the field or ocean can be preserved for laboratory testing by following the procedures given in the Sample Storage section.

4.5.3 Concentrated Ammonium Hydroxide

Since carbon dioxide is easily absorbed by highly basic solutions, concentrated ammonium hydroxide often contains carbonate as an impurity. By a variation of the direct measurement procedure, the amount of carbonate in concentrated ammonium hydroxide can be measured. By adding increments of a 10,000 ppm standard to an acid reagent, a calibration curve is constructed. By diluting the sample in acid reagent, the sample concentration is determined from the calibration curve, taking the dilution factor into account.

The required equipment is found in Section 2.1.
You will also need a 1 mL graduated pipet and a 10 mL pipet.

In addition to the solutions found in Section 2.1, prepare the following:

1. A standard solution of 10,000 ppm CO_3^{-2}. To prepare this solution, add 14.0 grams of reagent-grade sodium bicarbonate ($NaOH_3$) to a 1 liter flask half filled with distilled water, swirl the flask to dissolve the solid, and fill to the mark with additional distilled water.

2. 3M H_2SO_4 acid reagent. To prepare this solution, add approximately 500 mL of distilled water to one liter volumetric flask and, with extreme caution, add 167 mL of concentrated sulfuric acid (18M H_2SO_4), swirling the flask slowly, after small additions, to mix the acid and dissipate the heat. Finally, add remaining distilled water to the mark. Allow the solution to cool to room temperature. Allow the solution to cool to room temperature.

To measure a sample:

1. Add 100 mL of acid reagent to a 150 mL beaker. Place the beaker on the magnetic stirrer and begin stirring. Place the tip of the electrode in the solution. Add the standard solution (10,000 ppm), using the steps given in Table 4-1 below. Record the mV reading after each addition. Plot the concentration (log axis) against the mV potential (linear axis) on semi-logarithmic paper. The plotted concentration is eleven times the actual concentration due to sample solution.

2. Add 100 mL of the acid reagent to a 150 mL beaker and place the beaker on the magnetic stirrer. Begin stirring. Place the tip of the electrode in the solution. Add 10 mL of the sample to the beaker and record the mV reading. Determine the concentration from the calibration curve.

TABLE 4-1
PROCEDURAL STEPS

Step	**Pipet**	**Added Volume**	**Plotted concentration**
1	A	0.1 mL	109.9 ppm
2	A	0.9 mL	1089 ppm
3	B	10 mL	10900 ppm

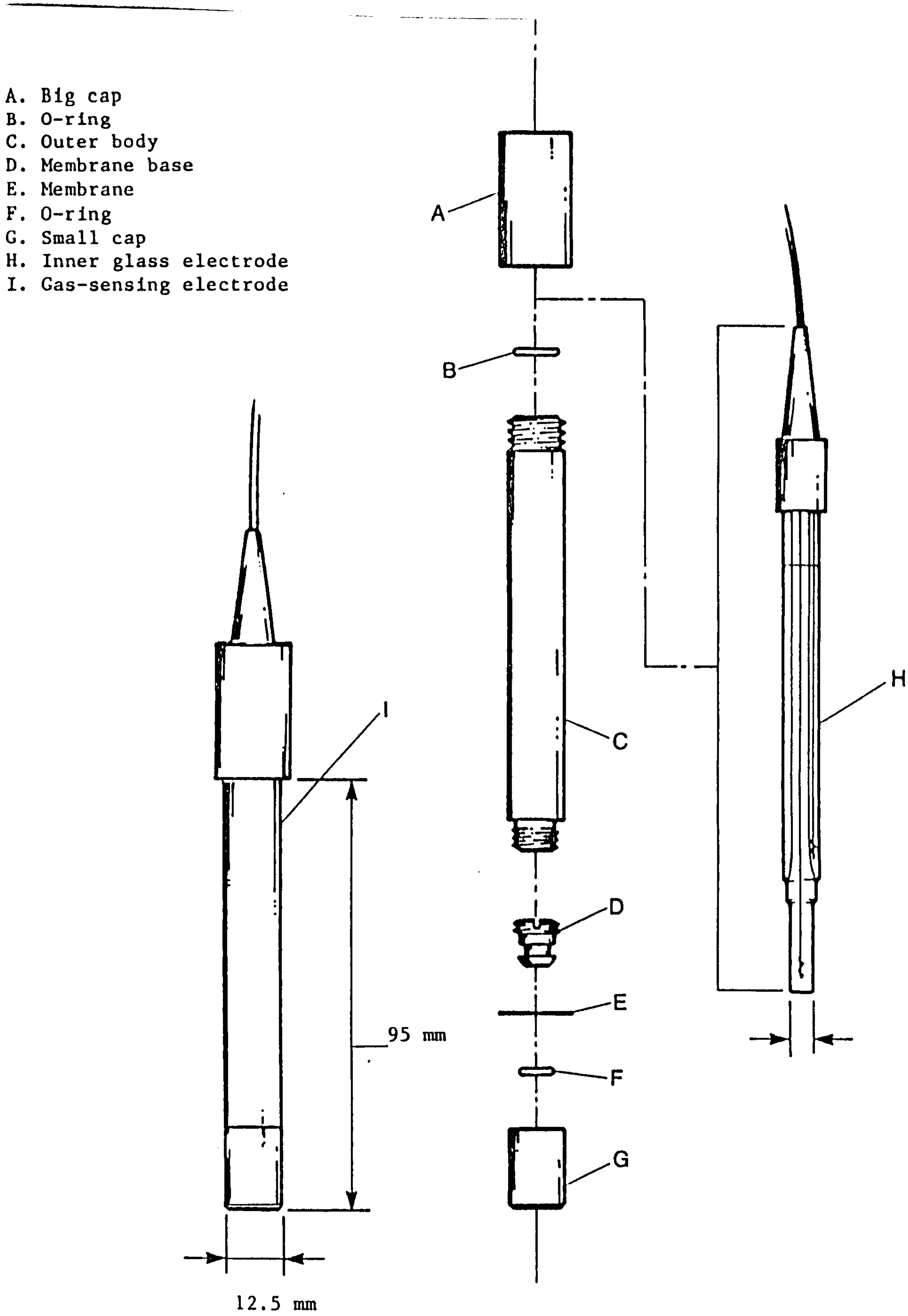
A = 1 mL graduated pipet
B = 10 mL pipet

SECTION 5 TROUBLESHOOTING

To check the operation of the Carbon Dioxide Gas-Sensing Electrode, use pH 4 and pH 7 buffers and the following procedure:

Disassemble the electrode. Soak the glass tip of the inner body in filling solution for at least two hours if the electrode is dry. Next, rinse the inner body with distilled water and immerse the tip in the pH 7 buffer, making sure that the reference element is covered. Use the magnetic stirrer to stir the solution during this procedure. Make sure the meter is in the mV mode. If the mV reading is not 0.0+20mV, adjust the calibration control (offset adjustment) to zero.

Remove the inner body from the buffer, rinse with distilled water, and immerse the tip in the pH 4 buffer, again making sure that the reference element is covered. Use the magnetic stirrer to stir the solution during this procedure. In less than 30 seconds after immersion in the pH 4 buffer, the reading should stabilize in the range of +150 to +190 mV. The millivolt difference between pH 7 and pH 4 should be greater than +150 mV if the inner body sensing elements are operating correctly.



A. Big cap
B. O-ring
C. Outer body
D. Membrane base
E. Membrane
F. O-ring
G. Small cap
H. Inner glass electrode
I. Gas-sensing electrode

Figure 4-2. Carbon Dioxide Electrode

NOTES

NOTES

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