● C ISE-8780

Cyanide Ion Selective Electrodes

















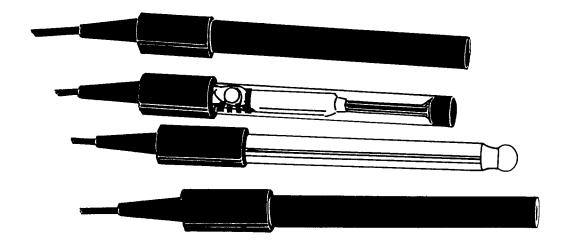














Operator's Manual M784/0892

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SECTION 1 GENERAL DESCRIPTION

The OMEGA® Cyanide Ion Selective Electrodes are used to quickly, simply, accurately, and economically measure cyanide ions in aqueous solutions.

CAUTION

Acidic cyanide solutions produce hydrogen cyanide (HCN) gas, highly toxic whether breathed or absorbed through the skin. Use of the proper and recommended ionic strength adjustor (ISA) will keep the solution pH above 10. If measurements in acidic solution are necessary, use the decomplexing procedure (as detailed in the Complexation section). THE PROCEDURE MUST BE DONE IN A HOOD.

Use a pipet bulb when pipeting cyanide solutions, as these solutions are highly toxic.

SECTION 2 PREPARATION FOR MEASUREMENT

2.1 REQUIRED EQUIPMENT

- 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. The OMEGA Cyanide Ion Selective Electrode, ISE-8780.
- 5. The OMEGA Double Junction Reference Electrode, PHE-3211, with 0.1M KNO3 outer fill solution.
- 6. Labware made of plastic, not glass.
- 7. Jeweler's rouge for polishing the electrode membrane, if necessary.

2.2 REQUIRED SOLUTIONS

1. Deionized or distilled water for solution and standard preparation.

- 2. OMEGA Cyanide Ionic Strength Adjuster (ISA), ISE-8710-R1. The ISA is used to adjust the pH of the solution to the proper operation range of the electrode and to provide a constant background ionic strength. To prepare the ISA (NaOH) from your own laboratory stock, fill a 100 mL volumetric flask about half full of distilled water. While gently swirling the flask, add 40 grams of reagent grade sodium hydroxide. Continue to swirl the flask until the NaOH is dissolved. Dilute to the mark with distilled water. Store in a plastic bottle.
- 3. Cyanide Standard (1 X 10⁻² M). Add 0.65 grams of dry, reagent grade NaCn to a one liter volumetric flask. Add 10 mL of ISA and about 500 mL of distilled water and swirl the flask gently to dissolve the solid. Dilute to the mark with distilled water and mix thoroughly.
- 4. Cyanide Standard (1000 ppm). Add 2.50 grams of dry, reagent grade KCN or 1.88 grams of dry, reagent grade NaCN to a one liter volumetric flask. Add 10 mL of ISA and about 500 mL of distilled water and swirl the flask gently to dissolve the solid. Dilute to the mark with distilled water and mix thoroughly.

CAUTION: HIGHLY TOXIC!

Store all standards in plastic bottles and prepare weekly.

2.3 ELECTRODE PREPARATION

Remove the rubber cap covering the electrode tip and the rubber band covering the filling hole of the cyanide combination ion selective electrode or the reference electrode.

Connect the electrode to the proper terminals as recommended by the meter manufacturer.

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.

2.3.1 Electrode Slope Check (with standard pH/mV meter)

Check the electrode on a daily basis. To a 150 mL beaker, add 100 mL of distilled water and 1 mL of 10M NaOH ISA. Place the beaker on a magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tip into the solution.

Using a pipet, add 1 mL 1.0 X 10^{-2} M or 1000 ppm standard to the beaker. Adjust the meter to 0 mV.

Again using a pipet, add 10 mL of the same standard to the beaker. After the reading has stabilized, record the mV reading. The electrode is operating correctly if the mV potential has changed by 57+3 mV, assuming the solution temperature is between 10° and 25°C. See the TROUBLESHOOTING section if the potential change is not within this range.

Slope is defined as the change in potential observed when the concentration changes by a factor of 10.

SECTION 3 MEASUREMENT PROCEDURE

3.1 MEASURING HINTS

All samples and standards should be at the same temperature for precise measurement. A difference of 1°C in temperature will result in about a 2% measurement error. Constant, but not violent, stirring is necessary for accurate measurement. Magnetic stirrers can generate sufficient heat to change the solution temperature. To counteract this effect, place a piece of insulating material, such as styrofoam sheet or asbestos sheet, between the stirrer and the beaker.

The electrode should be rinsed and blotted dry with a clean, lint-free, dry tissue between measurements to prevent solution carryover.

When making low level measurements (below 1 \times 10⁻⁵ M), use only plastic labware and cover the beaker with Parafilm to avoid loss of cyanide.

When making high cyanide measurements (above 1 \times 10⁻³ M), samples should be diluted before measurements.

3.2 SAMPLE REQUIREMENTS

Inorganic solutions will not affect the epoxy or the glass electrode body. Infrequent measurements in solutions containing methanol, benzene, or acetonitrile are permitted. Chloroform and acetone slowly attack the epoxy body electrode. Please check with OMEGA before using the electrode in other solvents.

Measurements above 1 \times 10⁻³ M should be done infrequently, as cyanide ion slowly erodes the membrane. It may be necessary to polish the membrane occasionally with jeweler's rouge as the electrode is used. Samples should be diluted below 1 \times 10⁻³ M if possible.

Proper pH is ensured if ISA is used. The pH should be above 10 so that cyanide is present as CN⁻¹ rather than as HCN in all standards and samples.

3.3 UNITS OF MEASUREMENT

Cyanide is measured in units of ppm, moles per liter, or any other convenient concentration unit. Table 3-1 indicates some concentration units and conversion factors.

TABLE 3-1
CONCENTRATION UNIT CONVERSION FACTORS

ppm CN ⁻¹	moles/liter
26.0	1.0×10^{-3}
10.0	3.8×10^{-4}
2.6	1.0×10^{-4}
1.0	3.8×10^{-5}
0.26	1.0×10^{-5}

3.4 DIRECT MEASUREMENT (using a standard pH/mV meter)

Direct measurement is a simple procedure for measuring a large number of samples. A single meter reading is all that is required for each sample. The ionic strength of samples and standards should be made the same by adjustment with ISA. The temperature of both sample solutions and standard solutions should be the same.

- 1. By serial dilution, prepare three standards solutions from the 1.0 X 10⁻² M or from the 1000 ppm stock solution. The resultant concentrations should be 10⁻³ M, 10⁻⁴ M, and 10⁻⁵ M or 10, 1, and 0.1 ppm. To each 100 mL of standard solution, add 1 mL of 10M NaOH (ISA). When calibrating, assume that the added ISA has no effect on the standard concentration.
- 2. Place 100 mL of the midrange solution, with added ISA, on the magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tips into the solution. Adjust the meter to zero mV.
- 3. Rinse the electrode with distilled water, blot dry and lower the electrode into the most dilute standard solution. After the reading has stabilized, record the mV value.
- 4. Rinse the electrode with distilled water, blot dry, and lower the electrode into the most concentrated standard solution. After the reading has stabilized, record the mV value.

- 5. Using the semi-logarithmic graph paper, plot the mV reading (linear axis) against concentration (log axis). A typical calibration curve can be found in Figure 3-1.
- 6. To a clean, dry 150 mL plastic beaker, add 100 mL of the cyanide sample and 1 mL of 10M NaOH.
- 7. Place the beaker on the magnetic stirrer and begin stirring. Rinse the electrode with distilled water, blot dry, and lower the electrode tip into the solution. After the reading has stabilized, record the mV reading. Using the calibration curve, determine the sample concentration.
- 8. The calibration should be checked every 2 hours. Assuming no change in ambient temperature, place the electrode tip in the midrange standard. After the reading has stabilized, compare it to the original reading recorded in Step 2. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of Steps 2-5.

3.5 DIRECT MEASUREMENT (using an ion meter)

- 1. By serial dilution of the 1.0 X 10⁻² M or of the 1000 ppm cyanide standards, prepare two standards whose concentration is near the expected sample concentration. Add 1 mL of 10M NaOH (ISA) to each 100 mL of standard. When calibrating, assume that the added ISA has no effect on the standard concentration.
- 2. Place the most dilute solution on the magnetic stirrer and begin stirring at a constant rate. Assure that the meter is in the concentration mode.
- 3. Lower the electrode tip into the solution.
- 4. Adjust the meter to the cyanide concentration of the standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.
- 5. Rinse the electrode with distilled water and blot dry.
- 6. Place the most concentrated cyanide standard on the magnetic stirrer and begin stirring at a constant rate.
- 7. Lower the electrode tip into the solution.
- 8. Adjust the meter to the concentration of the cyanide standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

- 9. For low level cyanide measurements (below 8 X 10⁻⁶ M or 0.2 ppm), place the rinsed, dried electrode into a solution containing 100 mL of distilled water and 1 mL ISA. After stabilization, fix the blank value in the meter according to the meter manufacturer's instruction.
- 10. After rinsing the electrode and blotting dry, place the electrode tips into 100 mL of the sample and 1 mL of ISA. After stabilization, read the concentration directly from the meter display.
- 11. The calibration should be checked every 2 hours. Assuming no change in ambient temperature, place the electrode tip in the first cyanide standard. After the reading has stabilized, compare it to the original reading in Step 4. A reading differing by more than 0.5 mV or a change in the ambient temperature will necessitate the repetition of Step 2-8 (2-9).

3.6 LOW-LEVEL DETERMINATION (using a standard pH/mV meter)

Use the following low-level cyanide measurement procedure in the non-linear portion of the calibration curve (below 8 X 10⁻⁶ M or 0.2 ppm). See Figure 3-1. A more accurate electrode indicator technique, such as titration, using a sulfide/silver ion selective electrode, may be preferred below these levels.

- By serial dilution, prepare 100 mL of 1.0 X 10⁻² M or 10 ppm cyanide standards. Add 1 mL of ISA to every 100 mL of standard.
- 2. To a 150 mL plastic beaker, add 100 mL of distilled water and 1 mL ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution. Assure that the meter is in the mV mode.
- 3. Increments of the standard should be added to the beaker according to the steps outlined in Table 3-2. After the reading stabilizes, record the mV reading for each addition. Reserve the final solution for checking the electrode each hour.
- 4. Plot the concentration (log axis) against the concentration (linear axis) as in Figure 3-1. A new low level calibration curve should be prepared daily using fresh standards.
- 5. To a 150 mL plastic beaker, add 100 mL of sample and 1 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. After rinsing the electrode, blot dry and lower the electrode tip into the solution. After stabilization of the reading, read the mV potential and determine the concentration from the calibration curve.

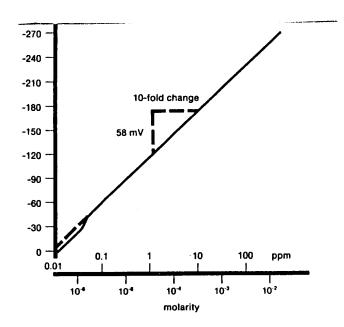


Figure 3-1 Typical Cyanide Electrode Calibration Curve

TABLE 3-2
LOW LEVEL MEASUREMENT CALIBRATION CURVE

		Added		Concer	tration
Step	Pipet	Volume	(mL)	ppm	M
1	A	0.1		0.01	1.0×10^{-6}
2	A	0.1		0.02	2.0×10^{-6}
3	A	0.2		0.04	4.0×10^{-6}
4	Α	0.2		0.06	6.0×10^{-6}
5	A	0.4		0.10	9.9 X 10 ⁻⁶
6	В	2		0.29	2.9×10^{-5}
7	В	2		0.48	4.8×10^{-5}

Pipet A = 1 mL graduated pipet

Pipet B = 2 mL pipet

Solutions: additions of standard/ISA

to 100 mL of distilled water and 1 mL ISA

SECTION 4 ELECTRODE CHARACTERISTICS

4.1 REPRODUCIBILITY

Direct electrode measurements reproducible to +/-2% can be obtained if the electrode is calibrated every hour. Factors such as temperature fluctuations, drift, and noise limit reproducibility. Reproducibility is independent of concentration within electrode's operating range.

4.2 INTERFERENCES

Solutions in which ions forming very insoluble silver salts are present at sufficiently high levels to form a layer of silver salt on the membrane surface will cause the electrodes to malfunction. Strong reducing solutions, such as photographic developer, should not be used with the cyanide electrode, since a layer of silver metal will be deposited on the electrode membrane. Electrode performance can be restored by polishing, if the surface of the sensing element becomes contaminated.

Solutions containing oxidizing agents, such as Fe⁺³, Cu⁺², and MnO4- will not affect electrode performance.

The maximum concentration of the more common interfering ions is given in Table 4-1. The maximum concentrations expressed as the ratio of the interfering ion concentration in moles per liter to the sample cyanide concentration in moles per liter. The electrode will malfunction if this ratio is exceeded. Neither the accuracy of the measurement nor the surface of the electrode membrane will be affected if the ratio is less than that listed in the table. Table 4-1 lists conversion factors from moles per liter to ppm.

4.3 LIMITS OF DETECTION

Cyanide levels from 8 X 10^{-6} M to 1 X 10^{-2} M cyanide can be measured with the cyanide electrodes. However, since cyanide ion attacks the electrode membrane, measurements above 1 X 10^{-3} M should be done only intermittently.

The electrodes respond to cyanide in the sample as well as to ions dissolved from the membrane at low levels. The electrode membrane shows a very slight water solubility. The detection limit of the electrode is determined by this factor. Figure 3-1 shows the theoretical linear response-the dashed line-in comparison with the actual response (solid line). The response to the dissolved membrane accounts for the discrepancy between the curves. The low-level procedure is recommended if measurements are to be made in the non-linear region below

Plastic labware must be used and the beakers must be covered with Parafilm for low-level cyanide determinations or cyanide will be lost. Allow a longer stabilization time before taking the meter reading for best results.

TABLE 4-1 CYANIDE INTERFERENCE RATIO

Interferences	Maximum Ratio (moles per liter)
S-2	must be absent
C1-	1×10^{-6}
Br-	5×10^{-3}
r-	1 X 10 ⁻¹

As an example: What is the maximum level of bromide allowable in a sample whose cyanide concentration is $1 \times 10^{-5} \text{ M}$:

Using Table 4-1, the maximum ratio is:

```
[Br-] = 5 \times 10^{-3} M
[CN-] = 5 \times 10^{-5} M
[Br-] = 5 \times 10^{-3} [CN-]
= (5 \times 10^{-3}) (1 \times 10^{-5})
= 5 \times 10^{-2} M = \text{maximum bromide concentration}
```

4.4 TEMPERATURE INFLUENCES

Samples and standards should be within +/-1°C of each other, since electrode potentials are influenced by changes in temperature. Because of the solubility equilibria on which the electrode depends, the absolute potential of the reference electrode (or reference portion of the combination electrode) changes slowly with temperature. The slope of the electrode, as indicated by the factor "S" in the Nernst equation, also varies with temperature. Table 4-2 gives values for the "S" factor in the Nernst equation for the cyanide ion.

TABLE 4-2
TEMPERATURE VS. VALUES FOR THE ELECTRODE SLOPE

T(°C)	S	T(°C)	S
0	54.20	30	60.15
10	56.18	40	62.13
20	58.16	50	64.11
25	59.16		

If changes in temperature occur, the electrodes should be recalibrated.

The temperature range for the OMEGA Cyanide Ion Selective Electrodes is 0°-100°C, provided that temperature equilibrium has occurred.

If the temperature varies substantially from room temperature, equilibrium times up to one hour are recommended.

Only intermittent use is recommended at temperatures above 80°C.

4.5 ELECTRODE RESPONSE

Plotting the electrode mV potential against the cyanide concentration on semi-logarithmic paper results in a straight line with a slope of about 58 mV per decade. Refer to Figure 3-1.

The time needed to reach 99% of the stable electrode potential reading, the electrode response time, varies from several seconds in highly concentrated solutions to several minutes near the detection limit. Refer to Figure 4-1.

The electrode membrane may need polishing if the electrode response drops off or if the readings drift.

To polish the membrane:

- 1. Place a cotton ball on the table top and flatten using the bottom of a beaker.
- Put 1-2 drops of distilled water in the center of the cotton pad.
- 3. Add a small amount of jeweler's rouge to the damp cotton.
- 4. With the membrane of the electrode facing downward, bring the membrane in contact with the cotton pad and gently rotate the electrode body for about 15-30 seconds using light pressure.
- 5. Rinse the electrode surface with distilled water and soak the electrode tip in 1 X 10⁻⁵ M or 1 ppm standard solution for about two minutes before use.

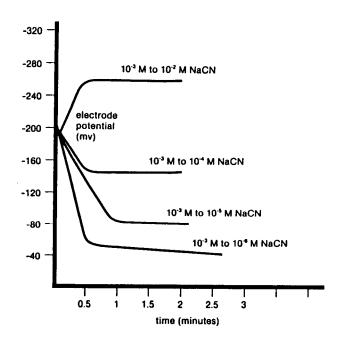


Figure 4-1 Typical Electrode Time Response

4.6 COMPLEXATION

Hydrogen ions and many metal ions form complexes with cyanide ions. The presence of any complexing agent lowers the measured concentration. Since the electrode measures only free cyanide ions, use of ISA is essential, since it eliminates complexation by hydrogen.

EDTA can be used to break up cyanide complexes formed with many metal ions, including cadmium, copper, nickel, and zinc. If a sample whose cyanide concentration is not more than 10 ppm, or about 1 X 10⁻³ M, add acetic acid to make the sample solution's pH 4. Add EDTA (tetrasodium) to about 0.02M (or about 0.76 grams Na4 EDTA per 100 mL sample.) In a hood, heat the mixture to about 50°C for about five minutes to speed up the decomplexation. After cooling the solution, add 10M NaOH(ISA) until the pH=~13. The cyanide remains free long enough for concentration measurements to be made, since EDTA complexes of the metals break up very slowly.

This method will not work for silver, mercury, gold, or cobalt, since cyanide will bind the metals too strongly.

4.7 ELECTRODE STORAGE

For storage, the cyanide electrode should be should be rinsed, blotted dry, and stored with the protective rubber cap in place.

The electrode lifetime will be adversely affected by exposure to high levels of CN-, since the membrane is dissolved by cyanide ion. Only intermittent measurements should be done above the 1×10^{-3} M level.

4.8 THEORY OF OPERATION

The OMEGA Cyanide Ion Electrodes consist of a solid membrane containing a mixture of inorganic silver compounds either bonded into the tip of an epoxy electrode body or fused into the tip of a glass electrode body. Silver ions dissolve from the membrane surface when the membrane is in contact with a cyanide solution. the movement of the silver ions within the membrane toward the membrane surface generates a potential difference that depends on the cyanide concentration in a solution. This potential is measured against a constant reference potential. The Nernst equation describes the level of cyanide ions in solution corresponding to the measured potential:

$$E = E_O - S \log X$$

where:

E= measured electrode potential E_O = reference potential (a constant) S = electrode slope (58 mV) X = level of cyanide in solution

The activity, X, represents the effective concentration of free cyanide ions in solution.

The activity is related to the free ion concentration, Cf, by the activity coefficient, fi, by:

X = Cf

Activity coefficients vary, depending on total ionic strength, I, defined as:

 $I = 1/2\Sigma CxZx2$

where:

Cx = concentration of ion X

Z = charge of ion X

 Σ = sum of all of the types of ions in the solution

In the case of high and constant ionic strength relative to the sensed ion concentration, the activity coefficient, fi, is constant and the activity, X, is directly proportional to the concentration.

All samples and standards containing cyanide ions have ionic strength adjuster (ISA) added so that the background ionic strength is high and constant relative to variable concentrations of cyanide. The recommended ISA for the cyanide electrode is sodium hydroxide (NaOH), though other solutions can be used as long as they do not contain ions that would interfere with the electrode's response to cyanide.

SECTION 5 TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
Out-of-range reading	Defective meter	Check meter with shorting strap
	Electrodes not plugged in properly	Reseat electrodes
	Reference electrode junction is dry	Hold cap and lift outer sleeve to expel a few drops of fill solution
	Reference electrode not filled	Be sure inner and outer chambers of double junction electrode are filled
	Calibration control not turned far enough	Continue turning calibration control
	Meter or stirrer not grounded	Ground meter or stirrer
"Incorrect answer" (but calibration curve is good)	Incorrect scaling of semi-log paper	Plot millivolts on the linear axis. On the log axis, be sure concentration numbers within each decade are increasing with increasing concentration.
	Incorrect sign	Be sure to note sign of millivolt number correctly
	"Bad samples"	Be sure ISA is added to sample
	Incorrect standards	Prepare fresh standards

SYMPTOM	POSSIBLE CAUSE	NEXT STEP
Drift (reading changing in one direction)	Samples and standards at different temperatures	Allow solutions to come to the same temperature before measurement
	Incorrect reference filling	Use recommended fill solution
Low slope or no slope	Standards contaminated or incorrectly made	Prepare fresh standards
	Standard used as ISA	Use ISA

SECTION 6 SPECIFICATIONS

CONCENTRATION RANGE: $1 \times 10^{-2} \text{ M}$ to $8 \times 10^{-6} \text{ M}$, 260 to 0.2 ppm

pH RANGE: 0-14 (10-14 recommended)

TEMPERATURE RANGE: 0°-80°C continuous use (80°-100°C

intermittent use)

RESISTANCE: 30 Mohms

REPRODUCIBILITY: +/-2%

SIZE: 110 mm (Length)

12 mm (Diameter)

1 m (Cable Length)

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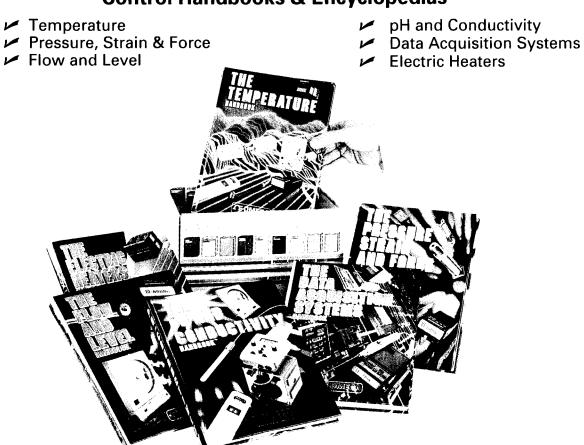
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