

Process Control

**Measurement
pH and Conductivity**

Courseware Sample

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By the staff of Festo Didactic

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




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Safety and Common Symbols

The following safety and common symbols may be used in this manual and on the equipment:

Symbol	Description
	DANGER indicates a hazard with a high level of risk which, if not avoided, will result in death or serious injury.
	WARNING indicates a hazard with a medium level of risk which, if not avoided, could result in death or serious injury.
	CAUTION indicates a hazard with a low level of risk which, if not avoided, could result in minor or moderate injury.
	CAUTION used without the <i>Caution, risk of danger</i> sign  , indicates a hazard with a potentially hazardous situation which, if not avoided, may result in property damage.
	Caution, risk of electric shock
	Caution, hot surface
	Caution, risk of danger
	Caution, lifting hazard
	Caution, hand entanglement hazard
	Notice, non-ionizing radiation
	Direct current
	Alternating current
	Both direct and alternating current
	Three-phase alternating current
	Earth (ground) terminal

Safety and Common Symbols









Symbol	Description
	Protective conductor terminal
	Frame or chassis terminal
	Equipotentiality
	On (supply)
	Off (supply)
	Equipment protected throughout by double insulation or reinforced insulation
	In position of a bi-stable push control
	Out position of a bi-stable push control

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Preface

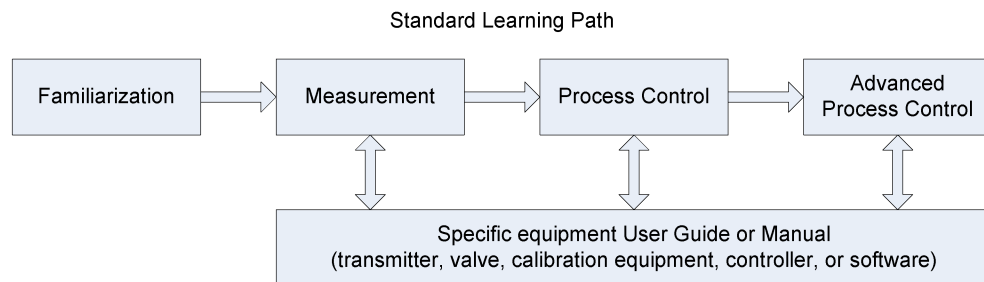
Automated process control offers so many advantages over manual control that the majority of today's industrial processes use it to some extent. Breweries, wastewater treatment plants, mining facilities, and the automotive industry are just a few industries that benefit from automated process control systems.

Maintaining process variables such as pressure, flow, level, temperature, and pH within a desired operating range is of the utmost importance when manufacturing products with a predictable composition and quality.

The Instrumentation and Process Control Training System, series 353X, is a state-of-the-art system that faithfully reproduces an industrial environment. Throughout this course, students develop skills in the installation and operation of equipment used in the process control field. The use of modern, industrial-grade equipment is instrumental in teaching theoretical and hands-on knowledge required to work in the process control industry.

The modularity of the system allows the instructor to select the equipment required to meet the objectives of a specific course. Two mobile workstations, on which all of the equipment is installed, form the basis of the system. Several optional components used in pressure, flow, level, temperature, and pH control loops are available, as well as various valves, calibration equipment, and software. These add-ons can replace basic components having the same functionality, depending on the context. During control exercises, a variety of controllers can be used interchangeably depending on the instructor's preference.

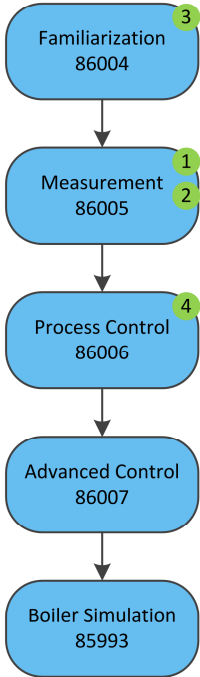
We hope that your learning experience with the Instrumentation and Process Control Training System will be the first step toward a successful career in the process control industry.



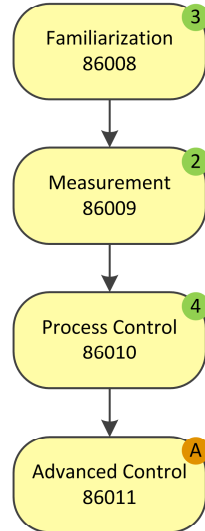
Preface

Manuals of the 353X Series

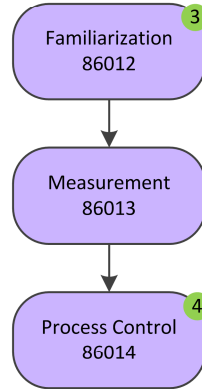
Pressure/Flow/Level



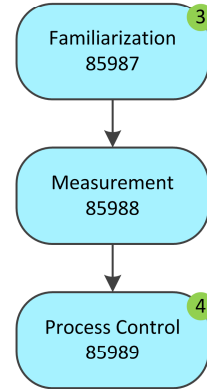
Temperature



pH and Conductivity



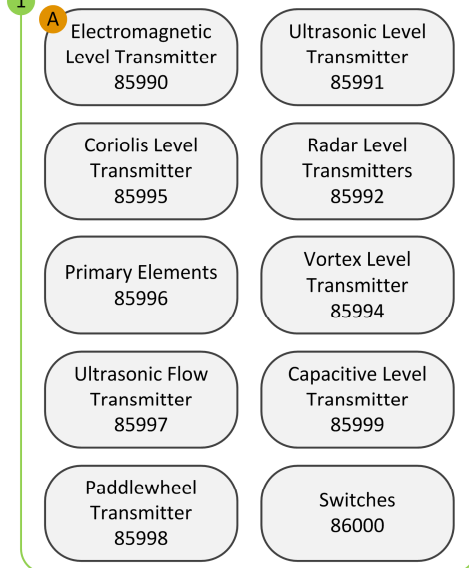
Pressure/Flow (Air)



How to read this chart

- Refer to optional manuals below, if required.
- This optional manual is required at this point.

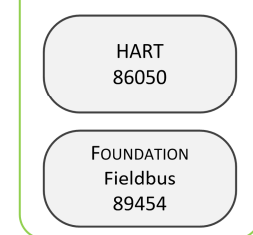
1 Pressure/Flow/Level Add-Ons



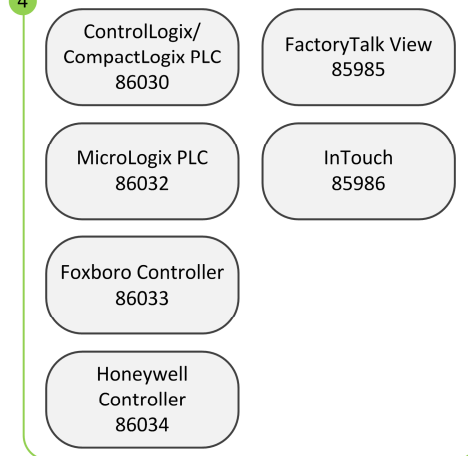
2 Final Elements



3 Communication Protocols



4 Controller/HMI Options



Preface

We invite readers of this manual to send us their tips, feedback and suggestions for improving the book.

Please send these to did@de.festo.com.

The authors and Festo Didactic look forward to your comments.

To the Instructor

You will find in this Instructor Guide all the elements included in the Student Manual together with the answers to all questions, results of measurements, graphs, explanations, suggestions, and, in some cases, instructions to help you guide the students through their learning process. All the information that applies to you is placed between markers and appears in red.

Accuracy of measurements

The numerical results of the hands-on exercises may differ from one student to another. For this reason, the results and answers given in this manual should be considered as a guide. Students who correctly performed the exercises should expect to demonstrate the principles involved and make observations and measurements similar to those given as answers.

Equipment installation

In order for students to be able to perform the exercises in the Student Manual, the Process Control Training Equipment – pH and Conductivity must have been properly installed, according to the instructions given in the user guide Familiarization with the Instrumentation and Process Control System – pH and Conductivity, part number 86012-E.

Sample Exercise
Extracted from
the Student Manual
and the Instructor Guide

pH Probes

EXERCISE OBJECTIVE

Familiarize yourself with pH probes, learn how to calibrate and use them for pH measurement. Also, learn how acid-base indicators work and use them to detect a pH change.

DISCUSSION OUTLINE

The Discussion of this exercise covers the following points:

- pH probes
- Acid-base indicators

DISCUSSION

pH probes

Probes used for pH measurement are usually made of glass because electric potential between its surfaces changes linearly with pH. Typical **pH probes** are made of two **electrodes**, one is the reference electrode providing a stable reference potential and the other is the glass measurement electrode.

The glass surface of the measurement electrode is coated with a thin layer of hydrated gel with a thickness that varies between 10 and 100 nanometers, depending on the type of electrode. The wire of the measurement electrode is immersed in a buffer solution containing Cl^- ions. This buffer solution usually has a pH of 7.

The reference electrode provides a reference potential to compare with the potential of the measurement electrode. The electrolyte solution of the reference electrode (usually potassium chloride) is in contact with the process through a junction (usually ceramic or Teflon). Ions migrate into the junction, establishing a diffusion potential. This configuration forms a galvanic half-cell.

Since the potassium chloride solution can flow slowly through the junction, care must be taken to avoid it running out. For this reason, the pH probe should always be stored in a storage solution containing potassium chloride. Figure 4-19 shows the principal components of a typical pH glass electrode.

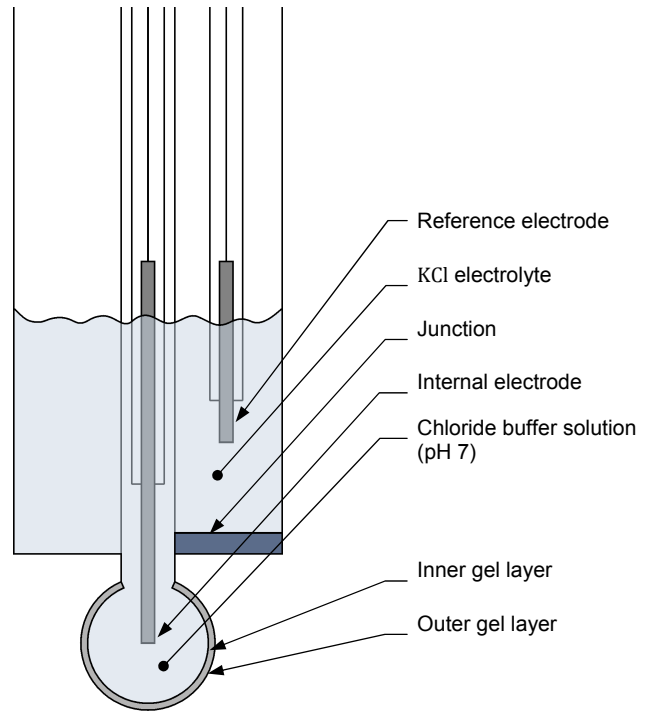


Figure 4-19. Typical pH glass electrode (single junction).

When the pH probe is immersed into the process solution, an electric potential, E_1 , is created between the outer surface of the glass electrode and the process solution. An electric potential, E_2 , appears between the inner surface of the glass electrode and the chloride buffer solution, as well as an electric potential, E_3 , between the electrolyte and the internal electrode. Hence, source voltage of the system is:

$$E_i = E_1 - E_2 - E_3 + E_4 + E_5 \quad (4-23)$$

- where
- E_i is the source voltage of the system
 - E_1 is the potential between the outer surface of the glass electrode and the process solution
 - E_2 is the potential between the inner surface of the glass electrode and the chloride buffer solution
 - E_3 is the potential between the chloride buffer solution and the internal electrode
 - E_4 is the potential between the electrolyte and the internal electrode of the reference electrode
 - E_5 is the diffusion potential of the reference electrode

To simplify, we will assume that E_3 is approximately equal to E_4 which is frequently the case. The potential E_5 is very small and can be neglected. Thus:

$$E_i = E_1 - E_2 \quad (4-24)$$

Figure 4-20 shows an equivalent electrical circuit representing some of the potentials and resistance values found in a pH probe.

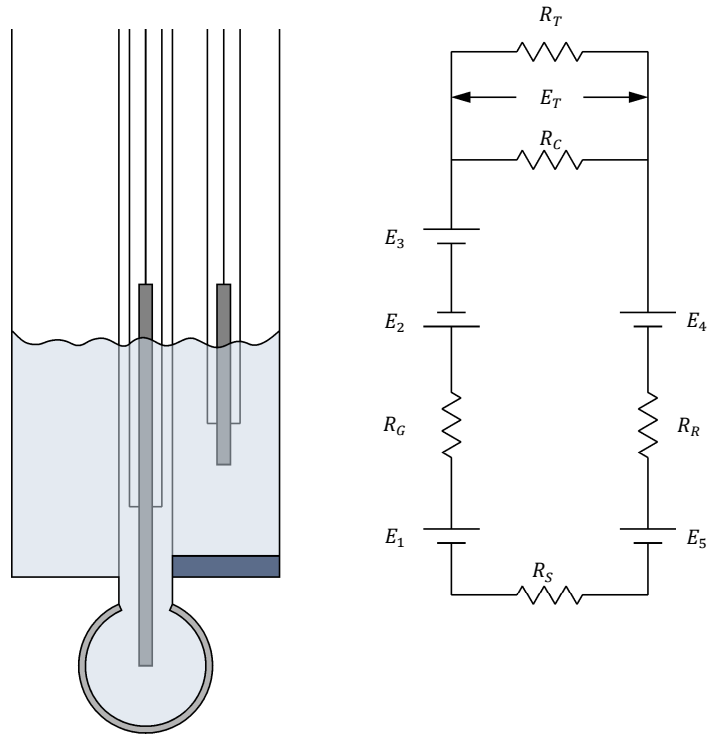


Figure 4-20. Equivalent electrical circuit representing a pH probe⁴.

where E_T is the potential at the poles of the transmitter or signal converter
 R_T is the input resistance of the transmitter or signal converter
 R_G is the resistance of the electrode glass
 R_R is the resistance of the reference electrode
 R_C is the insulation resistance between the connecting cables
 R_S is the resistance of the process solution

The potential of an electrode is given by the **Nernst equation**. When adapting this equation for potential E_1 and E_2 , the result is:

$$E_1 = E_0 + \frac{RT}{F} \ln[\text{H}_3\text{O}^+]_{\text{outer}} \quad (4-25)$$

$$E_2 = E_0 + \frac{RT}{F} \ln[\text{H}_3\text{O}^+]_{\text{inner}} \quad (4-26)$$

Combining the last three equations, and given the fact that

$$\ln[\text{H}_3\text{O}^+] = \ln 10 \times \log[\text{H}_3\text{O}^+] \quad (4-27)$$

⁴ For the sake of simplicity, some small electrical resistance values and other correction terms have been neglected.

we have

$$E_i = \frac{RT}{F} \ln 10 (\log[\text{H}_3\text{O}^+]_{\text{outer}} - \log[\text{H}_3\text{O}^+]_{\text{inner}}) \quad (4-28)$$

Since $\text{pH} = -\log[\text{H}_3\text{O}^+]$, Equation (4-28) can be rewritten as:

$$E_i = \ln 10 \frac{RT}{F} (\text{pH}_{\text{inner}} - \text{pH}_{\text{outer}}) \quad (4-29)$$

where

R	is the universal gas constant ($8.3145 \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$)
T	is the temperature in Kelvins
F	is the Faraday constant ($9.64853 \times 10^4 \text{ C}\cdot\text{mol}^{-1}$)
pH_{inner}	is the pH value of the glass electrode internal solution (usually equal to 7.0)
pH_{outer}	is the pH value of the process solution

From Equation (4-29) we can see that the slope of the curve of the electric potential generated by the electrode as a function of the pH of the process solution is about 59 mV at an ambient temperature of 298 K (Equation (4-30)). That is, 59 mV per pH unit.

$$E_i = 2.303 \frac{8.3145 \times 298}{96485.3} (7.0 - \text{pH}_{\text{outer}}) = 0.059(7.0 - \text{pH}_{\text{outer}}) \quad (4-30)$$

Figure 4-21 shows a graph of the electrode voltage output as a function of the pH. Four curves are shown on this graph; the black and blue curves are the standard output potential for pH probe. The black curve is the electrode output at a temperature of 25°C (77°F) and the blue curve is the uncompensated electrode output at a temperature of 75°C (167°F). The point where these two curves cross each other is called the **isopotential point**. At this point, the effect of temperature is null and the output is 0 mA. The green and turquoise curves are for measurement made at 25°C (77°F) and 75°C (167°F) respectively; but with a -100 mV bias. Such a bias usually indicates a change in the condition and path resistance of the reference electrode. It causes a vertical shift of the isopotential point on the graph. A horizontal shift of the isopotential point, such as shown on Figure 4-22, indicates a change in the activity and composition of the gel layer.

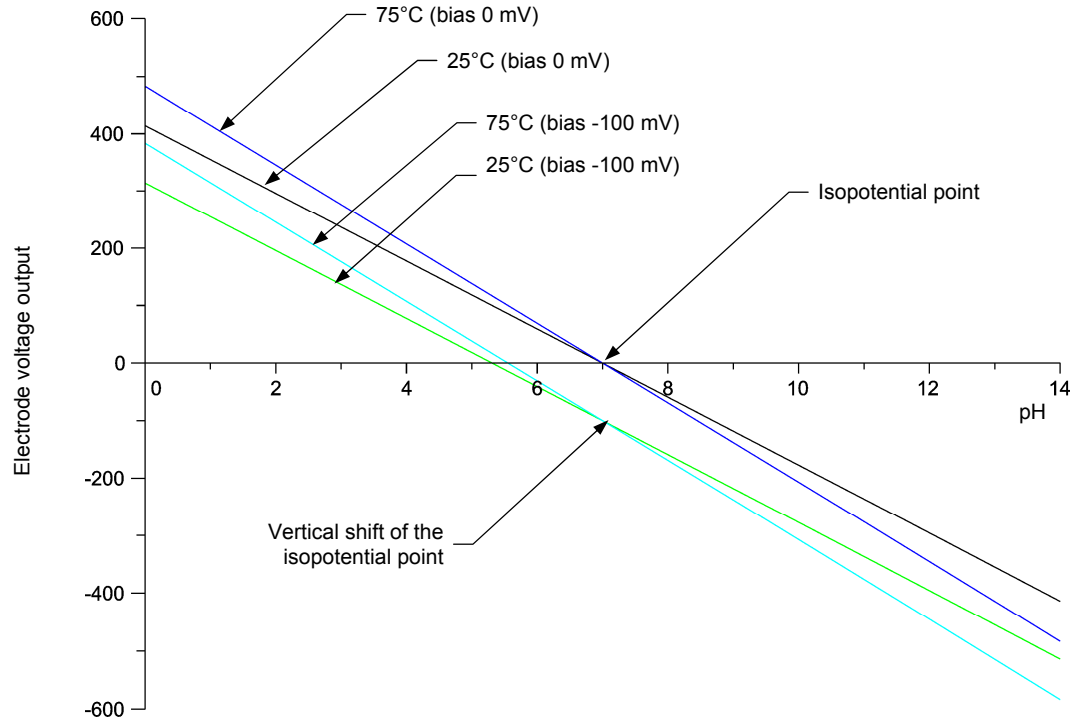


Figure 4-21. Vertical shift of the isopotential point.

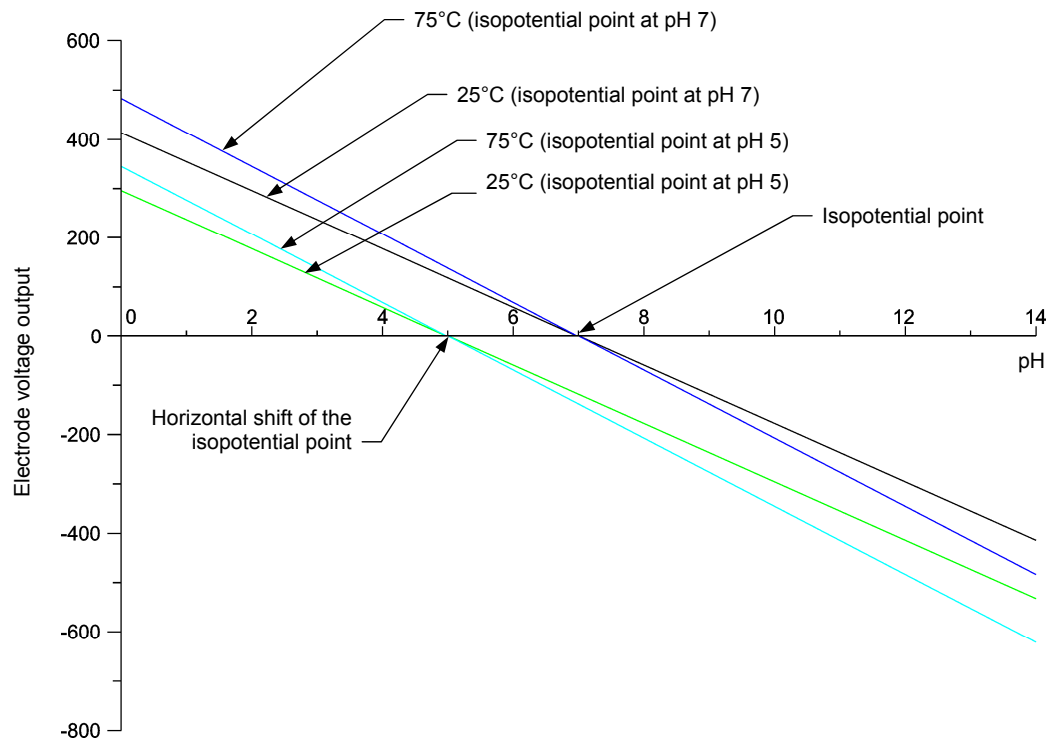


Figure 4-22. Horizontal shift of the isopotential point.

As shown in Equation (4-29), the pH measurement is also temperature dependant. Industrial pH probes are equipped with automatic temperature

compensators to automatically correct this kind of error. For pH probes without an automatic temperature compensator, a pH temperature error table such as Table 4-10 must be used for precise measurements.

Using E_i , the voltage at the poles of the transmitter, E_T , can be determined. The resistance values of R_R and R_S can be neglected for the calculation since they are very small compared to R_T and R_G (which is typically between 10 M Ω and 1000 M Ω). Thus, the voltage at the poles of the transmitter can be approximated as:

$$E_T = \frac{R_T}{R_G + R_T} E_i \quad (4-31)$$

Acid-base indicators

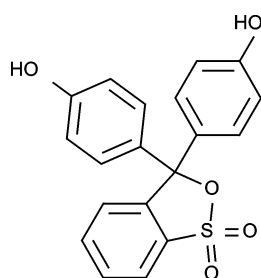


Figure 4-23. Phenol red (2D).

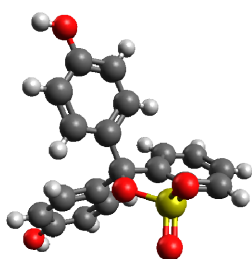


Figure 4-24. Phenol red (3D).

The use of a pH meter is not the only way to monitor or detect changes in the pH of a solution. Using substances that change color with pH is an easy and convenient way to detect if the pH of a solution is below, around, or above a given value. A substance that changes color with pH is called an **acid-base indicator**.

Acid-base indicators are commonly used by chemists for titration or to monitor pH dependant chemical reactions. Acid-base indicators are either vegetable pigments or synthetic dyes. A well known vegetable indicator is the litmus, which is extracted from a particular type of lichen. Other natural indicators are found in red cabbage, blueberries, and rhubarb for example. Synthetic acid-base indicators, such as **phenol red** presented below, are complex organic molecules whose properties change with the pH of the solution. Most acid-base indicators are weak acids that dissociate into water following the equilibrium reaction of Equation (4-32). Acid-base indicators are represented as HIn in chemical equations.



Since acid-base indicators are weak acids, they change the pH of a solution when added to it. However, since they are strongly colored, only a small quantity of indicator is required to tint a solution and its influence on the overall pH is minimal.

In its undissociated form (HIn) an acid-base indicator has a given color. When it dissociates, the conjugate base (In^-) has a different color. As for all weak acids, indicators have dissociation constants that obey to the following relation:

$$K_a = \frac{[\text{H}_3\text{O}^+][\text{In}^-]}{[\text{HIn}]} \quad (4-33)$$

When a solution is very acidic, the H_3O^+ are in excess and they react with the indicator conjugate-base to form undissociated indicator molecules, as shown in Equation (4-34). Hence, there are almost only undissociated indicator molecules and the color of the solution is the color of the undissociated indicator. In this case, the ratio $\frac{[\text{In}^-]}{[\text{HIn}]}$ is very small.



When a solution is very alkaline, the OH^- are in excess and they react almost completely with the undissociated indicator as shown in Equation (4-35). In this case, the concentration of the indicator conjugate base is much larger than the concentration of the undissociated indicator. Therefore, the solution takes the color of the conjugate base and the ratio $\frac{[\text{In}^-]}{[\text{HIn}]}$ is very large.



Not all acid-base indicators change color around the same pH value. The value around which the color transition takes place depends on the indicator dissociation constant. When the concentration in H_3O^+ is equal to the value of the dissociation constant, the concentrations of both the undissociated indicator (HIn) and its conjugate base (In^-) are equal. Therefore, they both contribute equally to the color of the solution and the ratio $\frac{[\text{In}^-]}{[\text{HIn}]}$ is equal to one. This occurs when Equation (4-37) is true. For phenol red, it is at a pH value of 7.9 (i.e., $\text{p}K_a = 7.9$).

$$\text{pH} = -\log k_a = \text{p}K_a \quad (4-36)$$

Figure 4-25 below illustrates the chemical changes that occur when a phenol red molecule dissociates. At the left is the undissociated phenol red molecule and at the right is its conjugate base. The removal of a hydrogen atom from the phenol red molecule changes its electronic structure and the frequency of the light the molecule can absorb; hence, it changes its color.

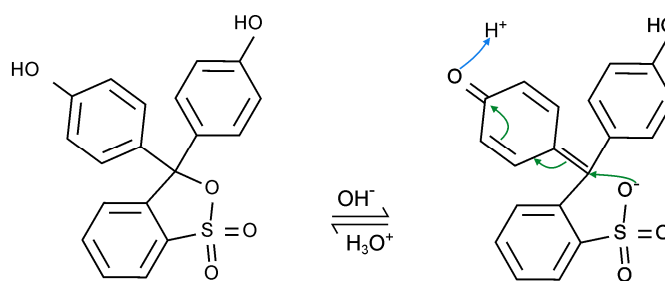


Figure 4-25. Chemical change in the phenol red (phenolsulfonphthalein) molecule.

Figure 4-26 shows the change in color of a phenol red tinted solution for different pH values. The color transition for phenol red takes place between pH values of 6.8 and 8.2.



Figure 4-26. Phenol red color for various pH values.

PROCEDURE OUTLINE

The Procedure is divided into the following sections:

- Set up and connections
- pH probe calibration
- pH measurement
- pH measurement with the process workstation
- Neutralizing the process water

PROCEDURE

Set up and connections

CAUTION



Before using ANY of the chemicals provided with the pH Process Control Training System, read the chemical MSDS and wear the appropriate personal protective equipment.

1. Connect the equipment as the piping and instrumentation diagram (P&ID) in Figure 4-27 shows. Use Figure 4-28 to position the equipment correctly on the frame of the training system from the 3532 series or use Figure 4-29 to position the equipment on the frame from the 3531 series. Use the basic setup presented in the *Familiarization with the Training System* manual. Table 4-11 lists the equipment you must add to the basic setup in order to set up your system for this exercise.

Table 4-11. Equipment required for this exercise.

Name	Part number	Identification
Volumetric flask	38406	
Scopulla	38442	
Sodium hydroxide solution 1.0 N	38444	

Hydrochloric acid solution 1.0 N	38445	
Phenol red	38446	
Graduated cylinder	38447	
Precision scale	38485	
Latex gloves	40331-54	
Pipette	40331-65	
Safety glasses	40348	
pH transmitter	46945	AIT
Metering pumps	46956	
Water analyzer	46989	
Chemical tanks	46994	
Buffer solutions	74238	
Acetic acid 5% (v/v)(vinegar)	76764	
Sodium bicarbonate (backing soda)	76765	

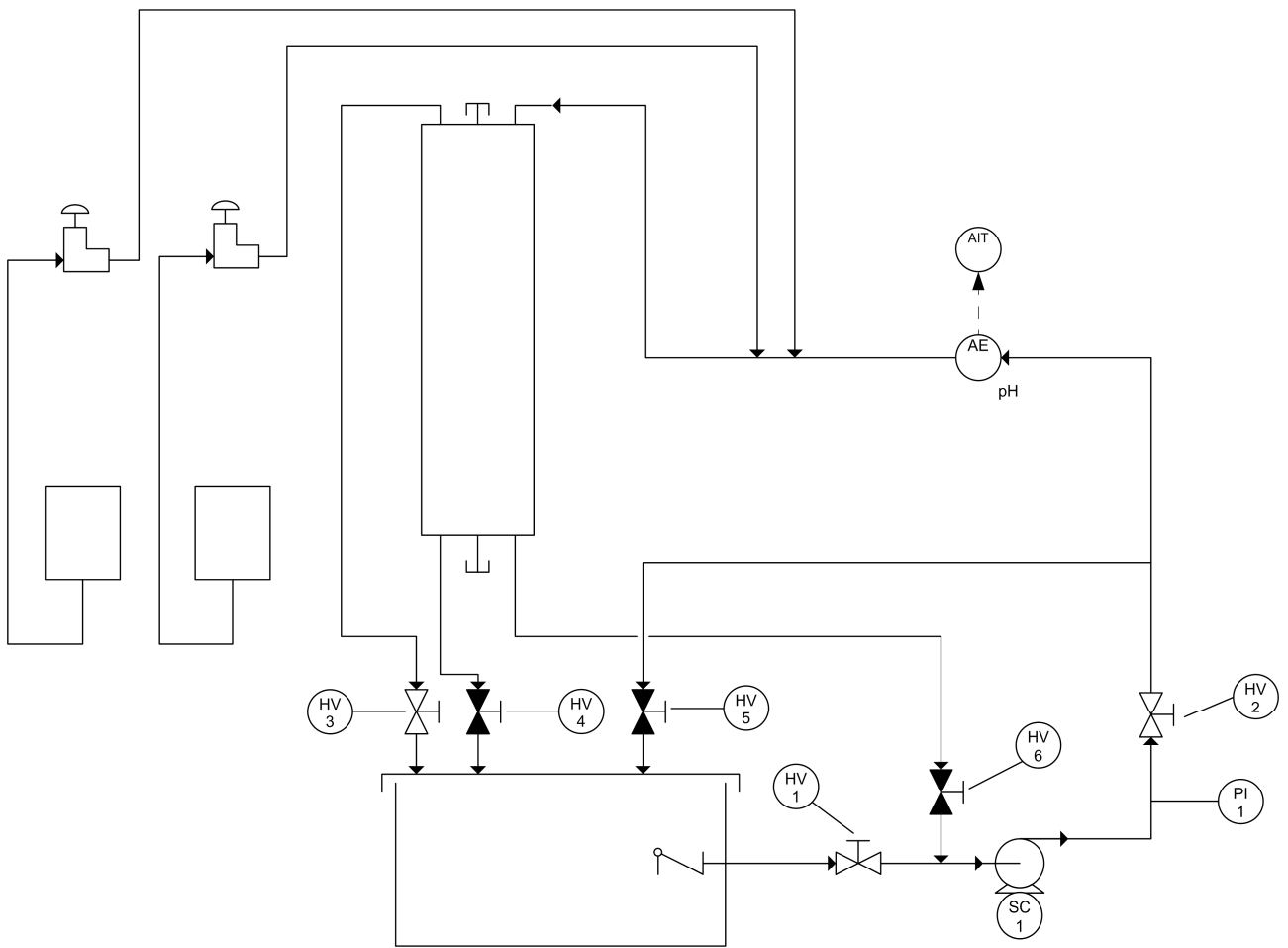


Figure 4-27. P&ID.

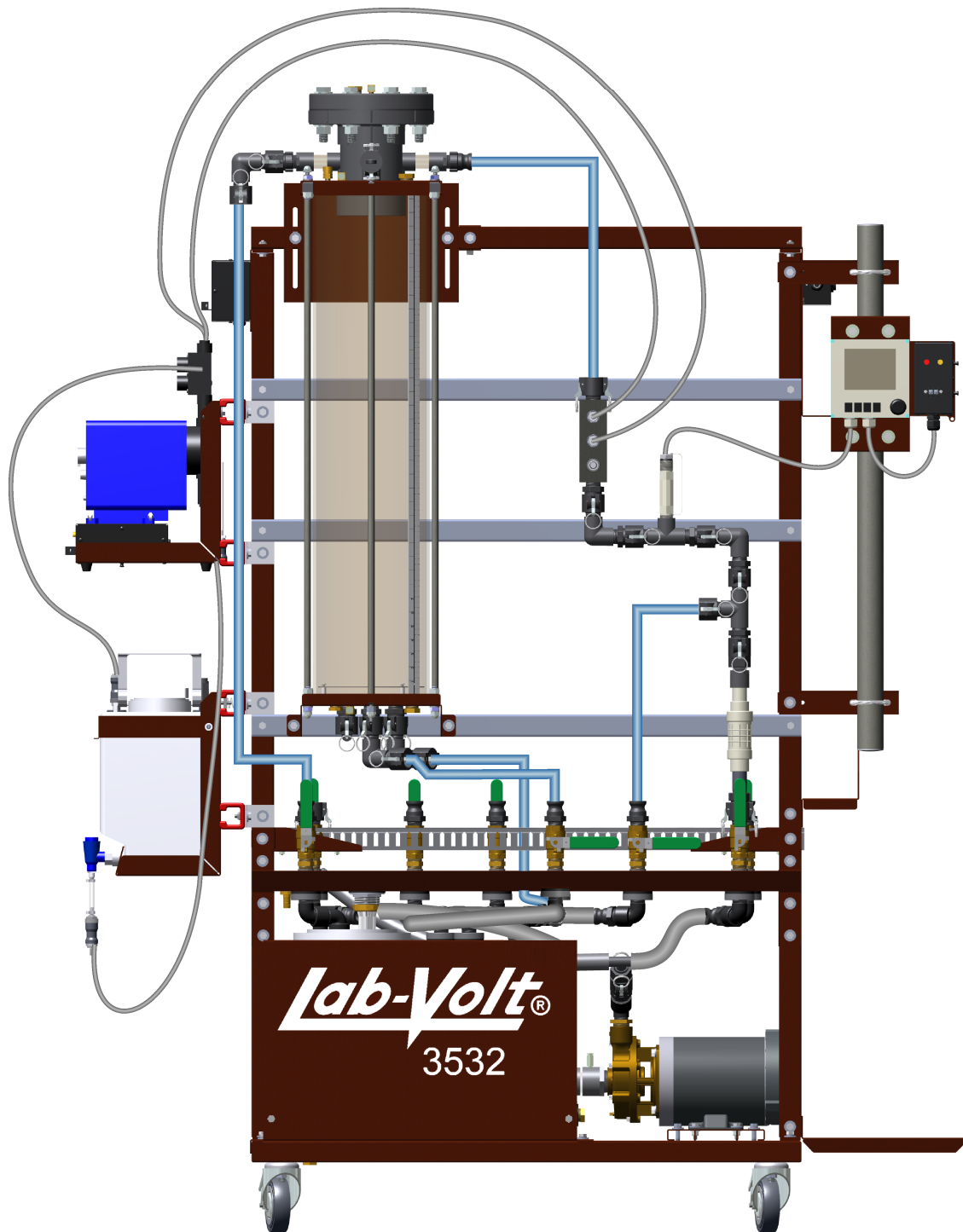


Figure 4-28. Setup (series 3532).

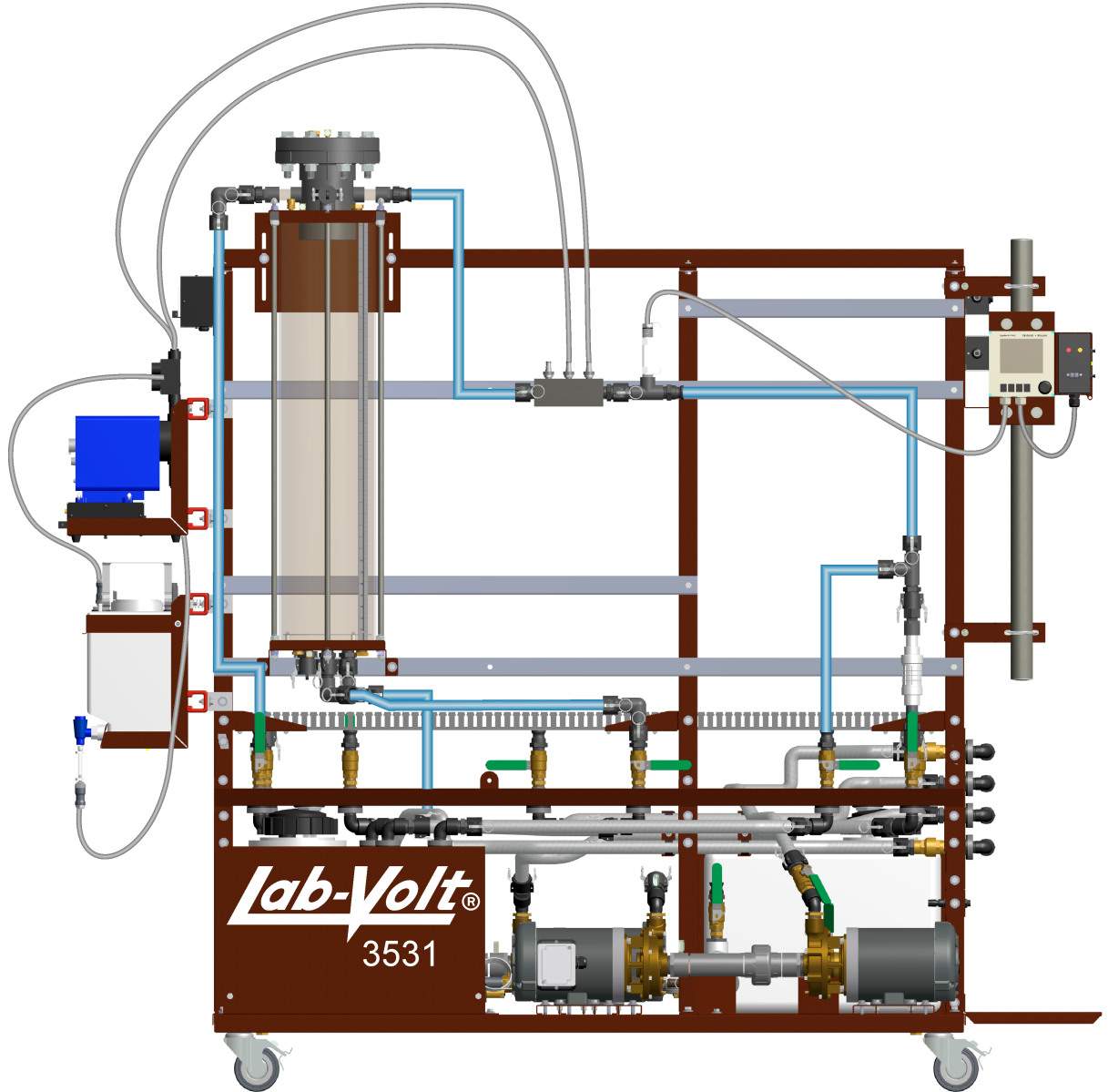


Figure 4-29. Setup (series 3531).

2. Wire the emergency push-button so that you can cut power in case of an emergency. The *Familiarization with the Training System* manual covers the security issues related to the use of electricity with the system, as well as the wiring of the emergency push-button.

3. Do not power up the instrumentation workstation yet. Do not turn the electrical panel on before your instructor has validated your setup—that is not before step 7.

⚠ CAUTION



Hydrochloric acid is corrosive and harmful if swallowed. Do not breathe vapor. Avoid eye and skin contact. When handling, wear chemical-resistant gloves, chemical safety goggles, and a lab coat. Refer to the MSDS for more details on this product.

⚠ CAUTION



Sodium hydroxide is corrosive and harmful if swallowed. Do not breathe vapor. Avoid eye and skin contact. Avoid prolonged or repeated exposure. When handling, wear chemical-resistant gloves, chemical safety goggles, and a lab coat. Refer to the MSDS for more details on this product.

4. Fill the first chemical tank with a solution of 0.08 mol/L of sodium hydroxide and the second chemical tank with a solution of 0.08 mol/L of hydrochloric acid. Carefully follow the procedure of Ex. 3-1 to prepare these solutions.
5. Before proceeding further, complete the following checklist to make sure you have set up the system properly. The points on this checklist are crucial elements for the proper completion of this exercise. This checklist is not exhaustive. Be sure to follow the instructions in the *Familiarization with the Training System* manual as well.



- All unused male adapters on the column are capped and the flange is properly tightened.
- The hand valves are in the positions shown in the P&ID.
- The chemical tanks are filled with the appropriate solutions and are carefully labeled.
- You are wearing the appropriate PPE.
- The vent tube is properly installed.

6. Ask your instructor to check and approve your setup.
7. Power up the electrical unit. This starts all electrical devices.

pH probe calibration

CAUTION

The pH probe of the Process Control Training System must be stored in a storage solution containing **KCl** to avoid damage. Refer to *Familiarization with the Training System* manual for more information on the storage of the pH probe.

8. The pH probe is a sensitive device and, by the nature of its construction, its properties are inclined to change with time and exposure to chemicals. Hence, it is of the utmost importance to check the calibration of the pH probe periodically. In an industrial environment where pH control is critical, it is a good practice to use redundant pH measurement devices for early detection of failure or drift of a pH probe.
9. Remove the pH probe from the connection port. Be careful, the tip of the probe is fragile.
10. Using the buffer solutions provided with the system, perform a two-point calibration as described in the *Familiarization with the Training System* manual. Make sure to rinse the tip of the probe between each measurement. Do not discard the buffer solutions. You will need them in the next steps.
11. Label three 1 oz. cups *Buffer 4.0*, *Buffer 7.0*, and *Buffer 10*, respectively, and pour the corresponding buffer solution into each cup.
12. Successively immerse the tip of the pH probe into each of the buffer solutions, being careful to rinse the tip of the probe between each measurement. Record the measured pH for each buffer solution in Table 4-12.

Table 4-12. pH measurement of buffer solutions.

Buffer solution	pH
Buffer 4.0	
Buffer 7.0	
Buffer 10.0	

13. The measured pH should be very close to the standard pH value of the buffer solutions. If it is not, calibrate the probe again using new buffer solutions.

pH measurement

14. Before making pH measurement with the process workstation, you need to familiarize yourself with the pH probe and transmitter by measuring the pH of various solutions.

15. First, fill a 100 mL beaker with tap water. Preferably, use water from the same source you have used to fill the system.

16. Immerse the tip of the probe into the tap water. Record the pH of the tap water below.

The pH of tap water can vary significantly.

17. Discard the tap water and fill the beaker with vinegar (i.e., a solution of 5% (v/v) of acetic acid).

18. Immerse the tip of the probe into the vinegar. Record the pH of the vinegar below.

The pH of the vinegar should be close to 2.4.

19. Discard the vinegar and rinse the beaker.

20. Using a precision scale, measure one gram of sodium bicarbonate.

21. Using a graduated cylinder, measure 50 mL of water.

22. Put the sodium bicarbonate into the beaker, add the 50 mL of water from the graduated cylinder, and stir.

23. Immerse the tip of the probe into the sodium bicarbonate solution. Record the pH of the sodium bicarbonate solution below.

The pH of the sodium bicarbonate solution should be close to 8. Refer to Ex. 4-4 of the student manual for details on how a sodium bicarbonate solution can act as a buffer.

pH measurement with the process workstation

24. Install the pH probe into the connection port on the process workstation.

25. Test your system for leaks. Use the drive to make the pump run at low speed in order to produce a small flow rate. Gradually increase the flow rate up to 50% of the maximum flow rate the pumping unit can deliver (i.e., set the drive speed to 30 Hz). Repair all leaks.

26. Start the pump and set the drive speed to 30 Hz.

CAUTION

This setup uses a telescopic pipe, which can extend if the pump runs at an excessive speed. Be sure to set the drive speed to a maximum of 30 Hz and secure the tubing with as much attach brackets, Model 85444, as possible.

27. Fill the column up to 25 cm of water. Then, close HV1 and open HV6 to put the process workstation into recirculation mode.
28. Remove one of the caps from the top of the column and, using a funnel, add about 20 mL of phenol red to the process water.
29. Once the process workstation is in recirculation mode, wait about 1 minute and read the pH value of the process water on the transmitter. Record the pH of the process water below.

The pH of the process water can vary significantly.

30. What tint did the phenol red give to the process water at this pH?

The tint given to the process water can vary significantly.

31. Using the metering pump connected to the chemical tank containing the solution of 0.08 mol/L of sodium hydroxide, start injecting alkaline solution into the process water.
32. On the pH transmitter, watch the pH of the process water increase up to 9.0 and then stop the metering pump.
33. What tint did the phenol red give to the process water at this pH?

Pink

34. Using the metering pump connected to the chemical tank containing the solution of 0.08 mol/L of hydrochloric acid, start injecting acid solution into the process water.
35. On the pH transmitter, watch the pH of the process water decrease down to 5.0 and then stop the metering pump.

36. What tint did the phenol red give to the process water at this pH?

Yellow

Neutralizing the process water

37. Before draining the system, you must neutralize the process water. That is, you must add acid or base until the pH of the process water is about 7.0. This can be tricky since the pH of the process water can change abruptly for a small addition of alkaline or acid solution.
38. At this step, the pH of the process water should be close to 5.0. Use the metering pump connected to the chemical tank containing the solution of 0.08 mol/L of sodium hydroxide to raise the pH of the process water. If necessary, reduce the delivery rate to avoid injecting too much alkaline solution.
39. When the pH gets close to 6.5, stop the metering pump and wait about one minute for the pH reading to stabilize.
40. If, after one minute, the pH is between 6.5 and 7.5, it is safe to drain the water in the column into the main tank of the process workstation. Open HV4 and HV5 to do so.
41. If the pH is not between 6.5 and 7.5, inject more acid and base until the pH is within the desired range. Then, drain the column.
42. Stop the system, turn off the power, and store the equipment. Do not forget to rinse the pH probe and store it in a storage solution as described in the *Familiarization with the Training System* manual.

CONCLUSION

In this exercise, you learned how to use and calibrate a pH probe for accurate pH measurement. You became familiar with the system, and used the metering pumps to inject acid and base into the process water. You also used an acid-base indicator to detect a pH change in the process water.

REVIEW QUESTIONS

1. How does a pH glass electrode work?

When the pH probe is immersed into the process solution, an electric potential proportional to hydronium concentration is created between the surfaces of the electrode bulb. By measuring this electric potential, the pH value of the solution can be deduced.

2. Which type of ion is usually present in the buffer solution inside the glass electrode?

Chloride ions

3. What is the pH of the buffer solution inside the glass electrode?

7.0

4. Why should you store the pH probe in a storage solution with KCl instead of distilled water?

To avoid the potassium chloride solution inside the reference electrode from flowing out into water through the junction.

5. Referring to the Nernst equation, which physical properties influence measurements taken with the type of pH probe described in this exercise?

Temperature

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