ELECTRODE START-UP & STORAGE PROCEDURE FOR GLASS BODY UNITS/LIQUID-FILLED

NOTE - **Possible crystallization build-up around edge of black storage cot is normal and can be easily removed by rinsing under warm running water.**

START-UP

1.) **Remove storage cot with caution from bottom of electrode. (NOTE: Glass bulb inside storage cot is fragile.)**

2.) **Rinse electrode glass bulb in distilled water.**

3.) **Check fill solution. See appropriate drawing diagram (Fig. 1 or Fig. 2) for correct level. If low, see chart below for correct fill solution and add through fill hole.**

SP135-500 FILL SOLUTION	
13-620-90	13-620-94
13-620-91	13-620-285
13-620-92	13-620-291
13-620-93	13-620-292

SP138-500 FILL SOLUTION	
13-620-270	13-620-286
13-620-271	13-620-293
13-620-280	13-620-296
13-620-281	13-620-297

4.) **Confirm fill solution flow at ref. junction. If no flow, pressurize by placing fill bottle tip on fill hole to make an air tight seal and squeeze until a bead of liquid forms at ref. junction. If bead fails to appear, see section entitled "Unblocking the Junction" in the main instruction sheet.**

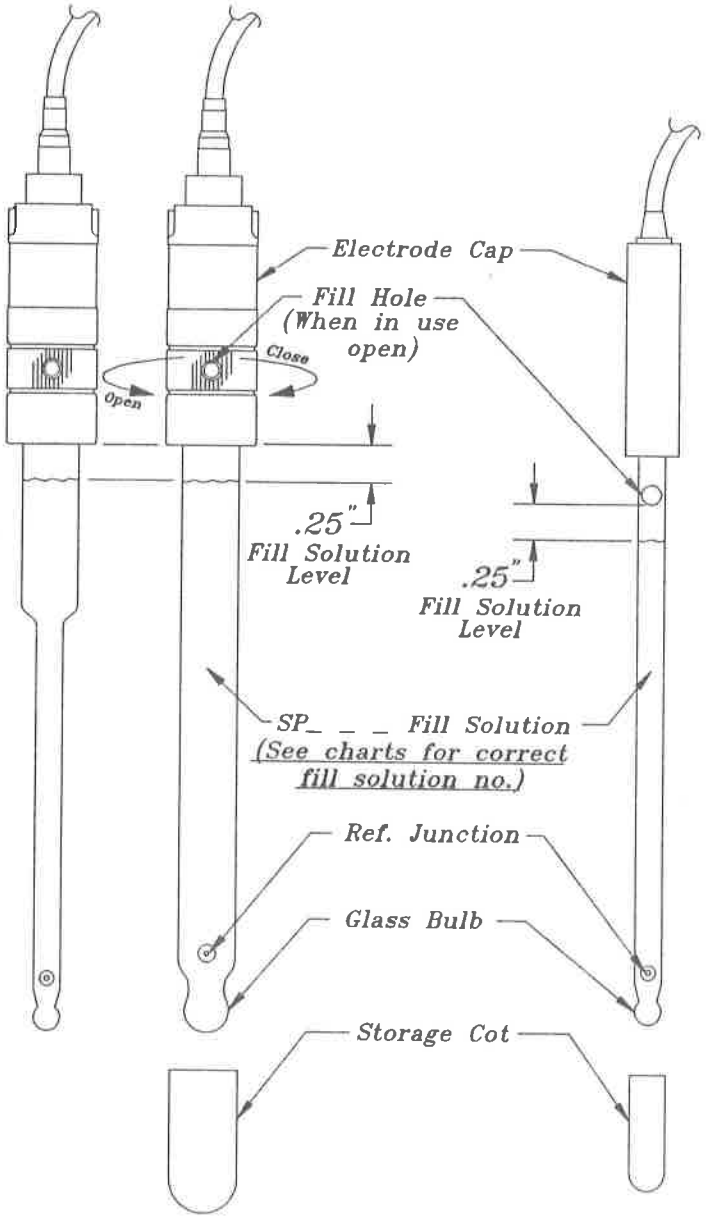
5.) **Connect electrode to meter.**

6.) **Allow the electrode to soak in buffer for 10 minutes prior to standardization.**

STORAGE

Store electrode in electrode storage solution (SE 40-1) or a KCL solution (SP138-500). Leave fill hole open. If storing in storage cot, make sure cotton inside is saturated with 4 or 7 buffer and close fill hole.

1/24/96



(FIG. 1)

CAT. NO.

13-620-90	13-620-285
13-620-91	13-620-286
13-620-270	13-620-293
13-620-271	13-620-296
13-620-280	13-620-297
13-620-281	

(FIG. 2)

CAT. NO.

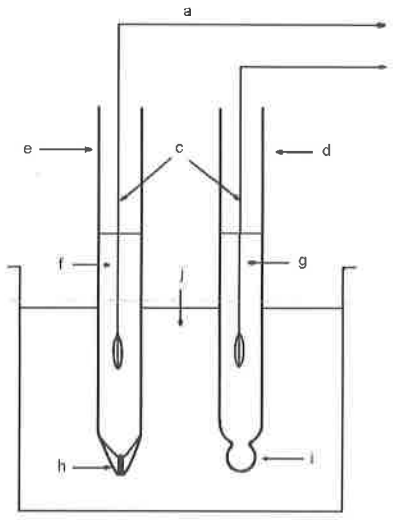
13-620-92
13-620-93
13-620-94
13-620-291
13-620-292

Accu-pHast™ Combination Electrodes

Catalog No.	Size	Body	Connector
13-620-113	Standard	Polymer	BNC
13-620-279	Standard	Polymer	US/PIN
13-620-280	Micro	Glass	US/PIN
13-620-281	Standard	Glass	US/PIN
13-620-296	Standard	Glass	BNC
13-620-297	Micro	Glass	BNC
13-620-298	Standard	Polymer	BNC

Accu-**pH**ast Combination Electrodes are designed to give both rapid and accurate results even when standardization and sample temperatures differ by as much as 70°C. The *micro* is for use in centrifuge tubes, viles, etc. The *standard glass* is for most laboratory applications. The *standard polymer body* is for use where breakage is a problem.

Figure 1
Conventional Electrode System



a) To reference on meter
b) To glass on meter
c) Reference elements
d) Glass half cell
e) Reference half cell
f) Reference fill solution
g) Glass fill solution
h) Junction
i) pH glass membrane

The Accu-**pH**ast design has several unique advantages over conventional pH measuring systems. Figure 1 is a simplified schematic of a conventional pH measuring system. A simplified equation of the measured potential is given in Equation 1.

Equation 1

$$E_{meas} = E_{in\ ref} + E_{ex\ ref} + E_{in\ glass} + E_{out\ glass} + E_{junc}$$

Here:

E_{meas} = The measured potential

$E_{in\ ref}$ = The potential of the internal reference

$E_{ex\ ref}$ = The potential of the external reference

$E_{in\ glass}$ = The potential of the inner glass surface

$E_{out\ glass}$ = The potential of the outer glass surface

E_{junc} = The junction potential

At a constant temperature, Equation 1 reduces to the following:

Equation 2

$$Emeas = k + \frac{RT}{nF} \ln \frac{AH^{+}_{ex}}{AH^{+}_{in}}$$

E_{meas} = The measured potential

k = Constant composed of $E_{in\ ref}$, $E_{ex\ ref} + E_{junc}$

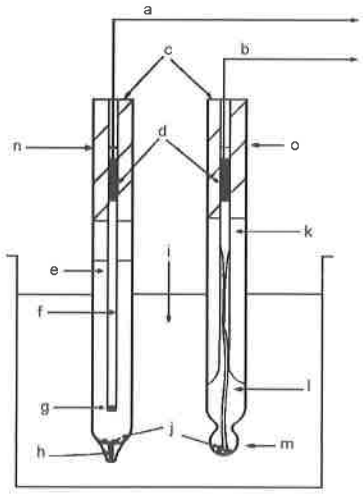
AH^{+}_{ex} = The activity of H^{+} external solution

AH^{+}_{in} = The activity of H^{+} in the internal fill solution

Theerfor, to make accurate measurements using temperature compensation with a conventional system, both "k" and AH^{+}_{in} must be held constant.

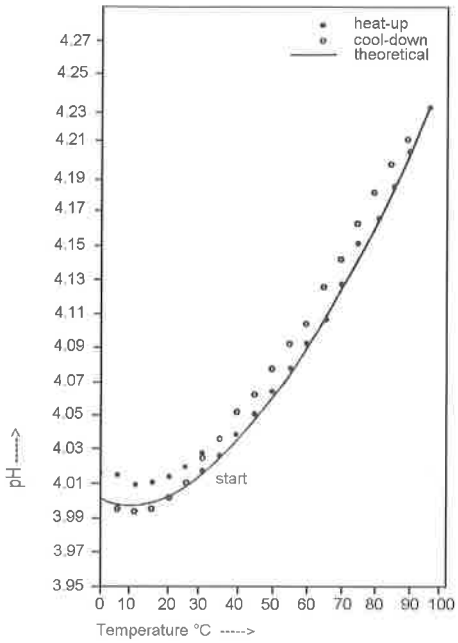
Figure 2. is a schematic of the Accu-**pH**ast system. This system differs from the conventional system in the location and design of the internal and external references. The references are designed to give symmetrical response to temperature change, yet they are isolated near the top of the electrode to minimize temperature effects. The internal buffer is formulated to give almost no change in pH with change in temperature. Therefor, the resulting system exhibits virtually no hysteresis on temperature cycling. The double junction feature represents an additional benefit which minimizes clogging problems associated with silver ions. Figure 3 shows typical experimental results on temperature cycling in 4.01 buffer. The solid line is the theoretical curve. The heat-up cycle is represented in solid circles. Results are all within ± 0.05 of theoretical.

Figure 2
Accu-pHast Electrode System



a) To reference on meter
b) To glass on meter
c) Incapsulating material
d) Silver/silver chloride reference packs
e) Reference fill solution
f) Gel layer
g) Inner junction
h) Outer junction
i) Sample
j) Excess KCl crystals
k) Wicking element
l) Glass fill solution
m) pH glass membrane
n) Reference half cell
o) Glass half cell

Figure 3
Accu-pHast pH Results on Heat Cycling in 4.01 (0.05 M KH Phthalate) Buffer



PERFORMANCE CHARACTERISTICS

Temperature Range	-5° to 100°C
pH Range	0 - 14
Drift	± .006pH per day
Slope	>95% of theoretical Nernstian
Isopotential point	pH 7.0 ± .1
Point of Accuracy of measuring a pH 4.01 buffer after standardization at 30°C	± 0.05pH from 0° to 95°C
Response time between 4.01 and 6.86 at 25°C	Stable to ± 0.005 in 15 seconds
Reproducibility in 4.01 at 30°C	± .003pH
Membrane Resistance	<100 megohms at 25°C
Sodium Ion Error	± 0.05pH at pH 13, 25°C

BUFFER VALUES AT VARIOUS TEMPERATURES

Temperature	0.05M	0.025 M Each	0.01M
°C	KH Phthalate	KH_2PO_4, Na_2HPO_4	$Na_2B_4O_7$
0	4.00	6.98	9.46
10	4.00	6.92	9.33
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.02	6.85	9.14
40	4.04	6.84	9.07
50	4.06	6.83	9.01
60	4.09	6.84	8.96
70	4.13	6.84	8.92
80	4.16	6.86	8.88
90	4.20	6.88	8.85
95	4.23	6.89	8.83

INSTALLATION

The electrode should be placed into service as follows:

CAUTION: Polymer-bodied electrodes should not be used in non-aqueous solvents, concentrated acetic acid, or concentrated oxidizing agents.

1. Carefully remove the plastic soaker bottle from the electrode. Save the bottle for future storage.
2. Rinse the electrode with distilled water to remove crystal residue that may have formed on the surface during storage. (The residue is caused by the natural leakage and gradual evaporation of electrolyte during storage).
3. If the fill hole is located on the cap rotate the cap ring hole from the closed to the open position. If the fill hole is located on the electrode body, lower the rubber sleeve and remove the white vinyl tape to expose the fill hole.
4. Check the electrolyte level in the reference cavity (outer annular space). If the fill hole is located on the cap the electrolyte level should be approximately 1/4 inch below the cap. If the fill hole is located on the body the electrolyte level should be approximately 1/4 inch below the fill hole. If the electrolyte level is too low, add electrolyte from the provided bottle of SP138 (saturated KCl).
   NOTE: Always use saturated KCl solution (SP138) as the electrolyte.
5. Proper electrode function requires electrolyte flow at the junction. To ensure adequate flow perform the following procedure:
   NOTE: The liquid junction is located on the side of glass bodied units (white ceramic dot) and in the silicon bung at the base of the electrode on polymer bodied units (white ceramic rod or dot).
   a. Hold the electrode upright at a 45° angle between the thumb and the forefinger of the left hand, so that the filling hole faces out and is directly opposite the base of the thumb.
   b. Insert the spout of the dispensing bottle into the fill hole.
   c. Making sure that the electrode is supported by the base of the thumb, firmly press the spout into the fill hole to make an airtight seal.
      NOTE: Normally, the spout tip will not touch the internal element. While applying pressure, care should be exercised to prevent contact with this element. If contact occurs, it will be necessary to cut off a small portion of the tip.
   d. While maintaining the seal, squeeze the filling bottle so that the electrode becomes pressurized. A bead of liquid should form at the liquid junction in about 30 seconds; in some cases it may be necessary to maintain pressure for several minutes.
6. Mount the electrode on a suitable holder and connect the jack/jacks to the meter. If the electrode was supplied with a removable plastic bulb protector, it can be removed at this time..
   NOTE: If a polymer bodied unit is being used, the length of the probe can be adjusted to match the length of the meter temperature compensator with the supplied rubber sleeve. If the sleeve covers the fill hole, align the hole in the sleeve with the fill hole.
7. The electrode can now be checked for span. A properly performing electrode should have an efficiency greater than 0.95 or a slope greater than 95%. Check the electrode's span by performing either of the following procedures:
   a. Standardize in a buffer of known value (one point standardization). Then measure the value of a second known buffer. Proper response is indicated if the error is less than ±0.04 pH per pH unit from the standardization point when using fresh buffers.
   b. Standardize using a two-point procedure. (Refer to meter manual). An efficiency greater than 0.95 or a slope greater than 95% indicates proper response.

8. If the electrode is to be used at elevated temperatures, an initial cycle between ambient and maximum temperature should be performed as follows:
   a. Place the electrode in buffer at ambient temperature; record the millivoltage.
   b. Transfer the electrode to buffer at elevated tremperature for 10 minutes; record millivoltage.
   c. Transfer the electrode to buffer at ambient temperature; record the millivoltage after 5 minutes.
   d. Repeat steps a-c until the mV readings at room temperature stay within ± 2.0 mV of one another.

OPERATION

For optimum performance, follow these procedures:

1. The electrode should be immersed approximately 1 inch into the solution being measured. It may be immersed further as long as the level of electrolyte in the reference is kept above the level of the solution being measured.
2. The electrolyte in the reference side should be maintained at a level approximately 1/4 inch below the fill hole. Add a solution of saturated KCl (SP138) as needed.
3. The level of electrolyte in the reference side must be kept above the end of the reference pack for the electrode to function. Add electrolyte (SP138) as needed.
4. Crystallized KCl in the reference compartment will not adversely affect measurements at ambient and elevated temperatures. If measurements are to be made at elevated temperatures for prolonged periods of time, KCl crystals should be added to the reference chamber to assure saturation at the elevated temperature. If measurements are to be made at reduced temperatures for prolonged periods of time, the crystals in the reference chamber should be removed by emptying the chamber, rinsing with deionized water to dissolve the crystals and refilling with fresh saturated KCl solution (SP138).
5. When transferring the electrode from one solution to another, always rinse the outer surface with distilled water. Alternately, the electrode can be conditioned with a small amount of the sample.
6. Prolonged immersion in strong alkali may harm the glass membrane. Therefor, when taking measurements in strong alkali solutions, keep the electrode immersed only as long as necessary to make a reading.
7. Because of the high impedance characteristics of glass electrodes, the connecting cable should not be moved or touched while measurements are being made; otherwise, unstable meter indications may occur.

STORAGE

1. Never store the electrode in distilled or deionized water. This may lead to slow sluggish response.
2. Between measurements immerse the electrode in a buffer solution (4 or 7 recommended). Keep the fill hole open to avoid contamination of the electrolyte from the buffer solution.
3. When the electrode is not in use, it is highly recommended that the soaker bottle be replaced over the probe tip. Make sure the bottle has been filled with pH 4 or 7 buffer. Then place it over the tip of the probe and slide it on. This procedure keeps the probe in a ready state. (Close the fill hole on liquid filled units.)
   NOTE: When returning a stored electrode to service, it may be necessary to repeat step 5 under INSTALLATION.

REJUVENATION

All pH electrodes will naturally age and undergo a reduction in their Nernstian response. Occasionally, this is accompanied by sluggish response. A sluggish response can also be caused by either contamination of the glass membrane or by clogging of the liquid junction on the reference electrode (In rare instances, poor response can be caused by a deterioration of the insulation between the shield and the internal element). Procedures are given below for cleaning the glass pH bulb.

Refer to separate instructions supplied with the reference electrode for junction rejuvenation.

CLEANING THE GLASS MEMBRANE

A dirty glass membrane is usually indicated by beads of water forming on the bulb when it is rinsed with distilled water. The bulb can be cleaned as follows:

1. For protein layers - soak in a freshly prepared solution of pepsin in 0.1N HCl (approximately 1/4 teaspoon/100 ml for 2 hours.
2. For inorganic deposits - wash with EDTA, ammonia, or acids.
3. For grease and similar films - wash with acetone, methanol, etc.
   NOTE: After performing the cleaning procedure, thoroughly rinse the electrode with water and repeat step 5 under INSTALLATION.

UNBLOCKING THE JUNCTION

If the liquid junction should become blocked or partially plugged, perform the following:

1. Inspect the reference cavity for excessive crystallization. If excessive crystallization is present, go to step 2; if not, proceed to step 3.
2. Remove crystals as follows:
   a. Remove the filling solution by shaking it out through the fill hole.
   b. Repeatedly rinse the reference cavity with distilled water until all the crystals are dissolved.
   c. Empty the reference cavity and refill it with SP138.
   d. Pressurize the electrode (see step 5 under INSTALLATION) and determine if flow is re-established. If no flow is found, proceed to step 3.
3. Perform the following procedures in sequence and as needed, depending on the severity of the blockage.
   a. Soak the electrode tip in warm water and apply pressure to the filling hole (see step 5 under INSTALLATION).
   b. Empty the filling solution; then, soak the electrode tip in concentrated ammonium hydroxide for 5 to 10 minutes. Rinse reference cavity; then, apply pressure to the filling hole (see step 5 under INSTALLATION).
   c. If the junction remains clogged, you may carefully sand or file the porous plug on glass bodied units, making certain not to contact the glass bulb.

Revision Date: June 2007

RECONDITIONING THE SENSING MEMBRANE

Prolonged use, excessive alkaline immersion, or high temperature operation will cause surface leaching of the membrane glass, resulting in extremely noisy and/or sluggish response which cannot be remedied by the steps outlined above. If this occurs, the following procedures will often provide renewed stability and pH sensitivity:

1. Immerse the electrode tip into 0.1M HCl for about 15 seconds; rinse with water, and immerse into 0.1M KOH for 15 seconds. Cycle the electrode through these solutions several times, always finishing with the HCl soak; then, recheck the electrode performance. If problem still persists, go to step 2.
2. Immerse the electrode tip into a 20% ammonium bifluoride solution for 30 seconds or a 10% hydrofluoric acid solution for 15 seconds.

   WARNING: The above fluoride solutions are extremely corrosive and hazardous, and proper safety procedures must be observed when handling and using them.

3. **a.** Thoroughly rinse the electrode with water, then immerse the electrode tip into concentrated hydrochloric acid for 30 seconds to remove any residual fluorides and rinse again with water.
   b. Soak the electrode in buffer (pH 4 recommended) for at least 1 hour. Then recheck electrode performance. If performance has not been restored, the electrode should be replaced.
   NOTE: Frequent fluoride treatment can shorten the electrode life and may eventually cause membrane cracking.