Grab Sample Calibration

1. Grab sample calibration of a pH sensor is more valid when the sensor has been previously “characterized” to its pH meter via the two point pH buffer calibration procedure. The grab sample technique will evaluate the pH sensor’s performance under actual operating conditions which differ from the pH buffer calibration conditions previously seen by the sensor.
2. For a proper grab sample technique, a known good laboratory pH sensor and pH meter that are in calibration with each other are required equipment. The laboratory pH sensor should be exposed to the grab sample at the identical temperature that the on-stream pH sensor sees in service to prevent the introduction of an “error” caused by the solution-temperature-effects on accurate pH measurements.
3. No two pH sensors are identical, therefore, exact pH readings are rarely achievable. The on-stream pH sensor has been conditioned to the process environment and may be more correctly reporting the process pH than a laboratory pH sensor which has not yet totally acclimated to the process conditions.
4. The grab sample should be taken as physically close to the on-stream pH sensor as possible to ensure that a “representative” sample is being taken. The pH readings should be compared immediately. If required, adjust the on-stream pH meter to match the reading of the grab sample pH meter. Avoid any time lag from the grab sample pH reading and the calibration adjustment to the onstream pH meter.

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Broadley® James

Manufacturers of Sensors for Science and Industry

MicroFlow®

pH Sensor

Designed with a Sealed Ag/AgCl and Dual Salt Bridge Reference Half-Cell

Instruction Sheet

For use in biopharmaceutical and proteinaceous media pH measurement applications

Sensor Specifications

pH Range with least Na+ Error	0–14 pH 1–10 pH
Glass Membrane	HT-3, 350 MΩ @ 25°C
Temperature Range	0–60° C
Flow Rate	0 to 25 mL/min
Sample Chamber Volume	50 µL
Cable	Low Noise Coax Dual shielded

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Preparation for Use

1. Remove “storage tubing” containing the storage solution (3.8 Molar KCl) by unscrewing the chromatography fittings at both ends of the sensor.
   Note: Drain out all storage solution and thoroughly flush sensor flow chamber with deionized (DI) water.
2. Save the sensor’s “storage tube” for future use as a storage container for the MicroFlow pH Sensor when it is not in service. Be certain to use 3.8M KCl (BJC P/N: AS-3120-C20-0500) as the storage solution.
3. **For first-time use after removing the MicroFlow pH Sensor from its storage solution:** inspect the sensor for any signs of breakage or shipping damage and commence with its use in your application..
4. **For reuse of the MicroFlow pH Sensor, or after long term storage in a solution other than the recommended 3.8M KCl solution:** fill the flow chamber of the MicroFlow pH Sensor with a 3.8M KCl solution for 10 to 30 minutes. This treatment conditions the pH sensitive bulb and prepares the ceramic liquid junction for contact with solutions to be tested..

Testing Procedure

1. Flush the MicroFlow pH Sensor thoroughly with DI water to remove all traces of storage solution, process medium, or previous test solution and to prevent “carry over” contamination of pH buffer test solutions. Thoroughly flush the MicroFlow pH Sensor with DI water after each buffer test.
1. Fill the MicroFlow pH Sensor with 7.0 pH buffer solution. Allow a minimum of 30 seconds for sensor to thermally equilibrate with the buffer solution before taking a pH reading. The pH reading should be 7.0 pH ± 0.33 pH (±20 mV) @ 25°C. Make necessary adjustment to the pH meter with the “standardize” or “zero” control for a pH indication = 7.0 pH.
2. Flush the MicroFlow pH Sensor with DI water and fill with a 4.01 pH buffer solution. Allow a minimum of 30 seconds for proper sensor/solution equilibration before taking a pH reading. Make any necessary adjustments to the pH meter with the “slope” or “span” control for a reading = 4.01 pH units.

Notes:

- Always use “fresh” pH buffer solutions for best results.
- pH buffer solutions above 7.0 pH are less stable and have a very limited life. These high pH buffers will more readily absorb CO_2 from the atmosphere and will typically change to a lower pH value when left open.
- Keep in mind the “older” a sensor becomes, it will exhibit slower response times and will become less efficient in terms of its ability to span several pH units with the same repeatability.
- pH sensors are imperfect devices and require “calibration” from time to time in order to be properly characterized to its host pH meter.

Cleaning a MicroFlow pH Sensor with Impaired Response

Used pH sensors which are physically intact can sometimes be restored to an improved level of performance. All pH sensors have a given useful life span depending upon the conditions of use. One of the following cleaning procedures may prove helpful in restoring a used pH sensor.

1. **Initial Cleaning:** Wash with a solution of liquid detergent or enzyme detergent and warm water by flushing the sensor flow chamber. Follow with thorough rinse with DI or clean tap water.
2. **Inorganic Scale Deposits:** Attempts to dissolve the deposit inside the sensor's flow chamber with dilute hydrochloric acid is not recommended.
3. **Organic Oil or Grese Films:** Perform initial cleaning procedure. If film is known to be soluble in a particular organic solvent which is not harmful to glass, flush flow chamber with this solvent. Repeat step #1 above. Depending on the extent of the oil or grease contamination, it's possible that the ceramic liquid junction may be damaged beyond recovery. Soak in 3.8M KCl solution for a minimum of 30 minutes before recalibration and returning sensor to service.
4. **Plugged or Dry Ceramic Liquid Junction:** Remove any observed contaminant with one of the above procedures, then soak in 3.8M KCl solution for a minimum of 30 minutes.

Notes:

- Never permit the pH electrode to dehydrate or dry out. Always keep it in a wetted environment, especially when not in service.
- Cracked or broken electrodes are not repairable.
- Inspect cable and connector to ensure that the insulation integrity is intact and that there are no signs of corrosion or contaminants on the metal components.

Storage

1. **Short Term:** Fill sensor flow chamber with 3.8M KCl. If this solution is not available, use 4.01 pH buffer, clean tap water, or lastly, a sample of the process being measured to keep the sensor hydrated.
2. **Long Term:** Fill the sensor flow chamber with a freshly prepared 3.8M KCl solution and insert storage tube. Be certain that the sensor's sealing chromatography caps are securely in place. Tighten caps hand tight only. The sensor should be stored in an upright (vertical) position.

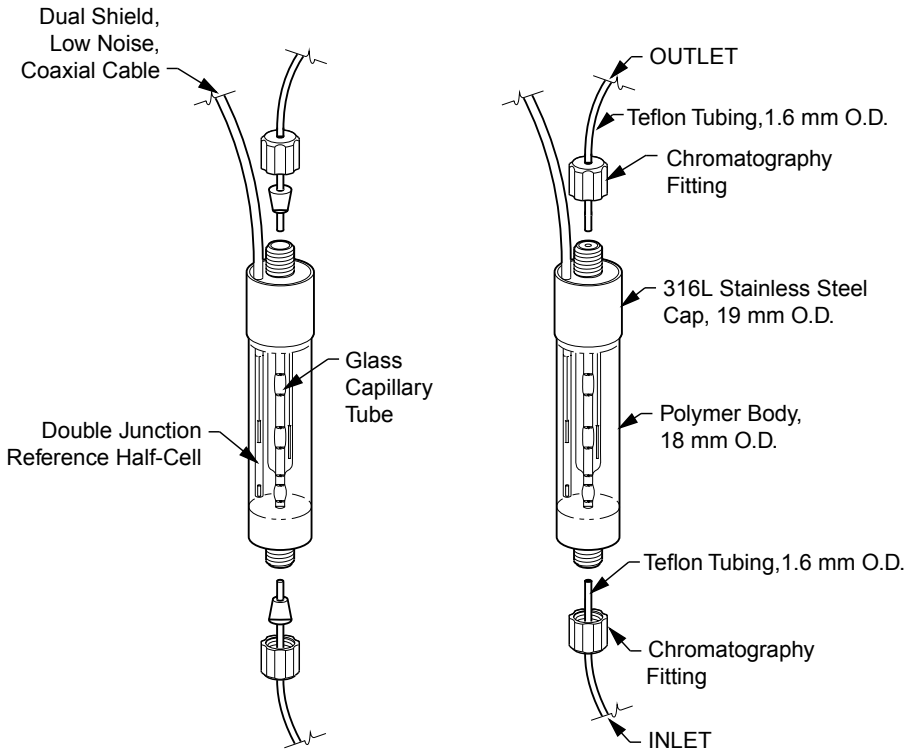

Figure 1. MicroFlow pH Sensor

Suggestions for Use

It is recommended that the sensor be held rigidly by a clamp, electrode holder, or similar device. For best measurement results the sensor should be kept in a vertical position with the stainless steel cap at the top. Do not use the sensor in the horizontal position. At most, tilt the sensor 45° from vertical during measurements.

The sample stream should enter the bottom of the sensor flow chamber and exit from the top. To fill the sample chamber with buffer or sample solution, it is best to draw the solution up into the sensor flow chamber by placing the inlet tube into the solution, attaching a syringe to the outlet tube, and then apply a gentle suction until the syringe begins to fill.

The entire sensor assembly may be sub-merged in a temperature bath to avoid possible measurement errors due to solution temperature coefficients.