pH Adapter & electrode

▶ pH Adapter

(Product No. 3125)

Range: 0 – 14 pH
Resolution: 0.01 pH

▶ pH Electrode

(Product No. 2251)

DATA HARVEST

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DS 026 No 4

Introduction

The *Smart Q* pH Sensor is a combination of a pH Adapter (Product No. 3125) and a general-purpose pH electrode (Product No. 2251).

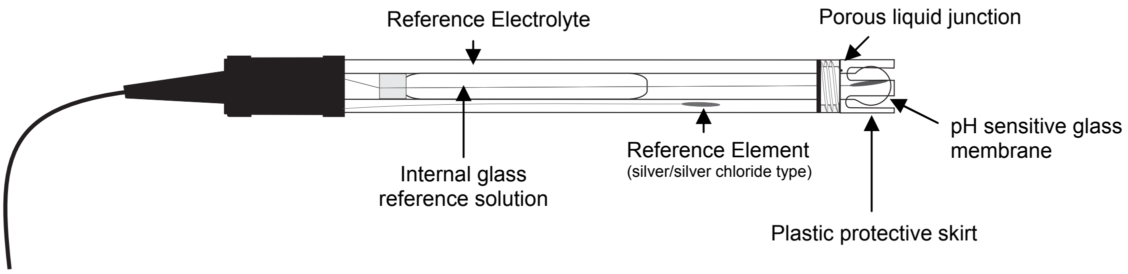
The *Smart Q* pH Adapter enables a pH electrode to be connected to an **EASY**SENSE unit. The **EASY**SENSE unit will detect that the pH Adapter is connected and automatically load the stored calibration. This calibration can either be the pre-set default calibration or a user calibration (see page 4).

IMPORTANT: Maintain the level of storage solution, the pH sensitive glass membrane must be kept **wet**.

The pH electrode

Product No. 2251 – RJ 0001
This is a general-purpose plastic bodied, single junction gel filled glass electrode, which is non-refillable.

Working Temperature range: 0 - 80°C.



Please Note: Store the electrode tip in a 1:1 solution of pH 4 buffer and 3.5 to 4 mol dm^{-3} KCl (see page 6).

Electrode preparation

1. Carefully remove the electrode from the bottle of storage solution.
2. Wash the glass membrane and liquid junction area (the lower section of the electrode) thoroughly with de-ionised or distilled water to remove any salt deposits from the exterior of the electrode.
3. Hold the electrode up to the light and check that the bulb at the tip of the electrode (glass membrane) is full of electrolyte. If air bubbles are present they can be removed by shaking the electrode firmly in a downward motion (like a clinical thermometer).
4. Screw on the clear plastic protective skirt if it's not already attached.

Connecting

Sensor cable

Line the pins up to the slots, push in and then rotate the BNC connector

- Push one end of the sensor cable (supplied with the **EASY**SENSE unit) into the hooded socket on the adapter with the locating arrow on the cable facing upwards.
- Connect the other end of the sensor cable to an input socket on the **EASY**SENSE unit.
- Connect the pH electrode to the pH Adapter (line the pins up to the slots, push in and rotate the electrodes BNC connector until it locks into place).

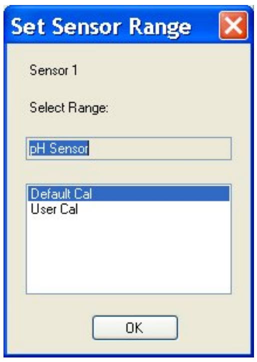
  Note: *To disconnect, twist the BNC connector in the opposite direction and pull.*
- The **EASY**SENSE unit will detect that the pH Sensor is connected and display values using the currently selected range.

Ranges

All *Smart Q* sensors are supplied calibrated. The stored calibration for the Sensor is the **Default Cal** range, which is suitable for most experiments. It is also possible for the user to adjust the calibration constants of an electrode for more accurate readings – this will be stored in the connected Adapter as the '**User Cal**' range. See user calibration on page 4.

To alter the currently selected range:

- Connect the pH sensor to the **EASY**SENSE unit.
- Start the **EASY**SENSE program and select one of the logging modes from the Home page e.g. EasyLog. Select **Sensor Config** from the **Settings** menu.
- Select the pH sensor from the list and click on the **Change Range** button.
- The current range will be highlighted. Select the required range and click on OK.
- Close Sensor Config. Click on New  and then Finish for the change in range to be detected.



The range setting will be retained until changed by the user. With some **EASY**SENSE units it is possible to change the range from the unit. Please refer to the **EASY**SENSE unit's user manual.

Measurement procedure

1. Connect the pH Sensor.
2. Rinse the electrode thoroughly in distilled water before use. (Salt deposits can get trapped between the skirt & tip).
3. Place the pH electrode in the sample to be tested, ensure the bulb is fully submerged.
4. Allow the electrode sufficient time to stabilise and then start taking readings.
5. Rinse the electrode between each measurement with either:
   - a portion of the next sample or
   - deionised or distilled water

User calibration

The stored calibration for a pH Sensor is the **Default** calibration, which is set for operation at a temperature of 25˚C.

If required the calibration constants of a pH electrode can be adjusted using Sensor Config in the **EASY**SENSE program. The settings for an electrode will be stored in the Adapter as the **User** calibration.

Note: *Mark the pH electrode and adapter combination so they are used as a pair.*

Standardised buffer solutions are used to adjust the sensor reading at either two or three points in its range. A slope adjustment is made using these points and will affect the whole range, between and beyond these points. The accuracy of the user calibration will depend upon the number of calibration points used and their spacing. Ideally the buffer solutions used should encompass the expected pH range and be as close as possible to the actual pH of the sample to be measured.

A User calibration is best used when the:

- experiment requires a very accurate calibration
- electrode has aged to the point where its glass membrane has changed resistance
- samples to be measured are at a lower or higher temperature than 25˚C. The buffer solutions used to set values must be at the same temperature as the samples in the experiment. Buffers values are temperature sensitive, enter a pH value for the buffer at that temperature.

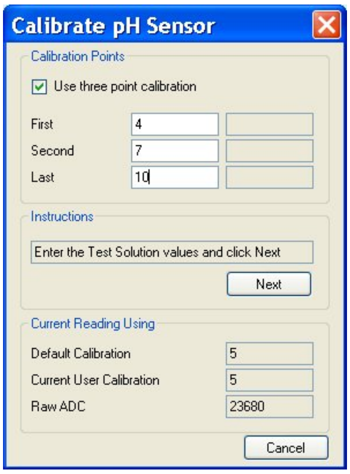
Values of pH buffers a various temperatures:

Temperature ˚C	pH 4.0 buffer	pH 7.0 buffer	pH 10.0 buffer
0˚	4.00	7.11	10.32
10˚	4.00	7.06	10.18
20˚	4.00	7.01	10.06
30˚	4.02	6.98	9.97
40˚	4.04	6.97	9.89
50˚	4.06	6.97	9.83

Please note that high pH buffers are less stable as they tend to absorb atmospheric CO_2 which lowers their pH. During calibration only open the bottle of buffer to pour it into a beaker. Never leave the bottle open.

How to calibrate

- Connect the pH Sensor to the **EASY**SENSE unit.
- Start the **EASY**SENSE program and select one of the logging modes from the Home page e.g. EasyLog. Select **Sensor Config** from the **Settings** menu.
- Select the pH Sensor from the list.
- Select **Change range** and alter the selected range to **User Cal**.
- Select **Calibrate Sensor**.
- If only two samples of buffers are available for setting the calibration points, click to deselect the tick in the Use Three point calibration box. Only the First and Last calibration points will then be available.



- **Type in the value of all the test solutions (buffers) that will be used** and click Next.
- Place the Sensor in the buffer with the lowest value e.g. pH 4 buffer. When the current reading stabilises, click on the **Next** button and the raw ADC value will automatically be entered into the First values box.
- Rinse the electrode in distilled water and place in the mid range sample e.g. pH 7 buffer. When the current reading stabilises, click on the **Next** button and the raw ADC value will automatically be entered into the Second values box.
- Rinse the electrode in distilled water and place in the highest value sample e.g. pH 10 buffer. When the current reading stabilises click on the **Next** button and the raw ADC value will automatically be entered into the Last values box.
- A message to say 'The User range has been reprogrammed ….' will be displayed. **Disconnect** the pH Sensor from the **EASY**SENSE unit and then reconnect for the calibration to be applied.

The User calibration values will be stored in the Adapter and will be retained until reconfigured by the user.

Note: *Mark the pH electrode and adapter combination so they are used as a pair.*

Practical information

- This general purpose Electrode (RJ001) is **non**-refillable
- Keep the pH sensitive membrane **wet** at all times. For the ion exchange process to occur properly, the glass needs to be hydrated. Check and maintain the level of storage solution. See page 6.
- If the electrode should inadvertently become dry, place in the storage solution for several hours in an attempt to recondition the glass.
- Care should be taken to avoid handling the pH sensitive glass membrane. Any damage to the surface, such as abrasion, may cause inaccuracies and result in a slow response time.

- Stirring of a sample will achieve a faster electrode response, but the glass membrane tip is very thin and requires care to prevent accidental damage. Broken glass bulbs are not covered by warranty.

- pH electrodes have a finite lifespan due to their inherent properties. How long a pH electrode will last will depend on how it is cared for and the solutions it is used to measure. Even if the electrode is not used, it will still age.

- Always use freshly prepared pH buffers. When not in use, pH buffers should be stored in sealed containers. High pH buffers are less stable as they tend to absorb atmospheric CO_2 which lowers their pH. During calibration only open the bottle of buffer to pour it into a beaker. Never leave the bottle open.

- Buffers and sample solution should be at the same temperature when measuring pH. The resistance of glass electrodes partially depends on temperature. The lower the temperature, the higher the resistance. It will take more time for the reading to stabilise if temperatures are cold.

- To allow the *Smart Q* pH Adapter to be used with any suitable pH electrodes with a BNC connector, automatic temperature compensation has not been built in.

- The electrical connections, terminations, etc, must be kept clean and dry. Failure to do so will result in a loss of insulation that will produce inaccurate results or total failure.

- If other electrochemical type Sensors (**Oxygen** and **Conductivity)** are put in the same solution at the same time and connected to the same **EASY**SENSE unit, they can interfere with each other's signals. Keep the Sensors as far apart as possible - the distance required will depend on the conductivity of the solution. If there is still a problem, try connecting the Sensors to different **EASY**SENSE units or take readings using one Sensor at a time.

Conditions to avoid:

- **Never** store the electrode in **deionised or distilled water,** as this will cause the migration of the electrode's fill solution.

- To maximise electrode life, avoid pH/ temperature extremes whenever possible. High temperature, strong acids or caustics (greater than 1.0 mol dm^{-3}) shorten electrode life. If used at high temperatures, the electrodes life is drastically reduced. The higher the range of temperature, the shorter the life of the electrode e.g. typical electrode life when used at ambient temperature is 1 – 3 years, if used at 80ºC this will be reduced to less than 4 months.

- Never expose to temperatures below -12˚C, freezing will damage the electrode.

- Coatings on the glass or junction surfaces e.g. proteins, will prevent proper operation (see maintenance on page 7). Avoid frequent or prolonged periods of use in these solutions.

Electrode storage

Maintain the level of pH electrode storage solution, the pH sensitive membrane must be kept **wet**.

Store the electrode in equal volumes of pH 4.0 buffer and 3.5 - 4 mol dm^{-3} Potassium Chloride (KCl) solutions (1:1 v/v).

Recipe: *Add 29 g of KCl to 100 cm^3 of distilled water. Add 100 cm^3 of a pH 4 buffer solution.*

Never store the electrode in **deionised** or **distilled water** - this will cause migration of the electrode's fill solution.

Electrode specifications

Range	0.00 to 14.00 pH
E°	0±25 mV
Stir Noise (pH 6.86)	±1000 µV
Slope (pH 4.00 – 6.86)	>96%
Drift (pH 4.00)	±1000 µV
Sodium Slope	>440 mV
Junction Resistance	300-32000 ohms

Maintenance

The glass bulb can become coated with any compound that has an affinity for glass.
After any cleaning procedure, soak the electrode in its storage solution for at least 30 minutes before use.

General cleaning procedure: - Soak the electrode in 0.1 mol dm^{-3} Hydrochloric acid (HCl) for 10 minutes. Rinse thoroughly with distilled water. Soak in its storage solution for at least 2 hours before use.

Inorganic coatings: - Soak in either 0.1 mol dm^{-3} Tetrasodium E.D.T.A acid solution or 1% Decon 90 solution for 1 – 2 hours.

Oil, Grease: - Carefully wash the electrode under warm tap water using a non-filming dish washing detergent. Do not use automatic or electric dish washing detergents. Rinse thoroughly with fresh tap water followed by a three rinses of distilled water. Soak the electrode in its storage solution for at least 30 minutes before use.

Protein & Fatty Materials: - Either gently wipe the bulb with a tissue soaked in propanol or soak in 1% pepsin in 0.1 mol dm^{-3} HCl for at least 10 minutes. Rinse thoroughly with distilled.

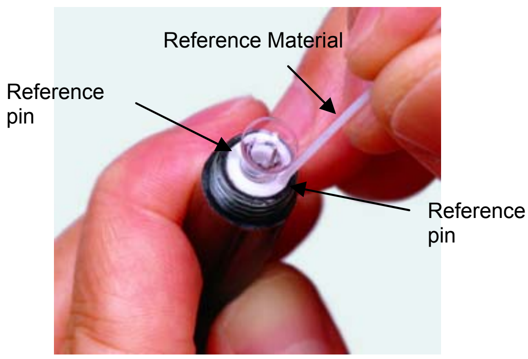
Highly resistant deposits: - Clean with H_2O_2 or sodium hyperchlorite.

Bacterial cultures: - Chemically sterilize with ethylene oxide, soak a cloth to wipe the entire body.

CAUTION - Do not use strong solvents such as halogenated hydrocarbons, petroleum ether, etc. for cleaning.

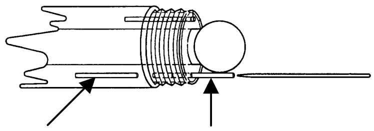
Reference pin replacement instructions

If the pH electrode fails to respond to cleaning, and the electrode response becomes slow or begins to drift, then the reference pins can be replaced.



- Use clean scissor or a craft knife to cut two 12 mm (½") lengths of reference material from the spare reference pin material provided with the electrode.
- Unscrew the threaded guard that protects the glass bulb.
- Hold the electrode and identify the two reference pins that are located on either side of the pH bulb.
- Use the toothpick provided (or a similar tool) and push each of the reference pins part way out of the reference assembly and into the reference reservoir.

- Using clean tweezers, insert the new reference pins into each of the holes in the reference assembly. The old reference pins will be forced out of the reference assembly and will remain in the reference reservoir. This is normal and will not affect electrode performance.



- Ensure the new reference pins stick out about 3 mm (⅛") from the surface of the reference. If the pins protrude to far from the reference assembly, the electrode may not operate properly.
- Reinstall the threaded guard onto the end of the electrode.
- Rinse the electrode with distilled water. It will now be ready for use.

Hints:

1. Prepare the new reference pins and have them available before you start.
2. Use clean tools, washed thoroughly with distilled water.
3. Flat style toothpicks work better than the round style.
4. Exercise caution when installing new reference pins. The pH bulb is thin glass and is easily scratched or damaged. Broken glass bulbs are not covered by warranty.
5. Do not allow KCl gel to run out of the electrode reservoir.
6. The pins will remain inside the reference reservoir. This is normal. Do not attempt to remove them; this does not affect electrode performance.

Theory of pH measurement

pH is a unit of measure which describes the degree of acidity or alkalinity of a solution and is usually written as:

$$pH = -\log [H^+]$$

Where 'p' is the mathematical symbol of the negative logarithm and $[H^+]$ is the concentration of Hydrogen ions.

pH levels generally range from 0 to 14. A pH value of 7 is described as neutral - the point at which the activities of hydrogen and hydroxide in solution are equal. When the pH value is less than 7, the activity of hydrogen ion is greater than that of the hydroxide ion and the solution is described as acidic. Conversely, as the hydroxide ion activity is increased the solution becomes alkaline (or basic) and the pH value is greater than 7.

The pH electrode is actually a combination of a two half-cells (electrodes) within a single body

- Internal pH Half Cell, the measuring electrode, whose voltage varies proportionately to the hydrogen activity of the solution, and a
- Reference Cell, the reference electrode, which provides a stable and constant reference voltage and completes the electrical circuit.

The pH Half Cell consists of a thin membrane of hydrogen ion sensitive glass blown on the end of a high resistance glass tube. Within this tube is an internal reference system, which remains constant.

The Reference cell uses a similar system, but without using a hydrogen sensitive glass. It is housed concentrically between the outer body of the electrode and the glass half-cell. It is comprised of a reference element (silver/silver chloride) and an electrolyte solution that seeps through a porous liquid junction (a small filter) to make the necessary electrical connection with the sample (the external liquid).

The pH Adapter measures the difference between the pH Half cell and the Reference cell in millivolts DC. This millivolt reading is displayed in pH units.

The electrode signal varies with the pH according to the Nernst Equation:

$$E = E^o + \frac{2.3\,RT}{nF} \cdot \log [H^+]$$

Where:
E = Measured electrode potential
Eº = Standard potential of the system (constant)
R = gas law constant (8.314 J/K Mol)
T = absolute temperature in ˚K (ºC +273)
F = Faraday constant (9.648×10^4)
n = valence factor (n = 1 in the case of hydrogen)

At 25˚C the theoretical slope is $\frac{2.3 \times 8.314 \times K}{96{,}486}$ = 59 mV/ per pH unit.

Temperature can affect the pH value in three ways:
1. The pH of the sample can change due to the hydrogen ion activity in the solution being temperature dependent. This factor is usually ignored because accurate pH measurement will be desired at that particular temperature.
2. Temperature will affect the glass membranes impedance.
3. Changes in temperature of a solution will vary the millivolt output of the electrode in accordance with the Nernst equation. Whether or not temperature compensation is needed will depend on the level of accuracy required.

pH error chart

ºC	2	3	4	5	6	7	8	9	10	11	12
15	-0.15	-0.12	-0.09	-0.06	-0.03	**0**	+0.03	+0.06	+0.09	+0.12	+0.15
25	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**
35	+0.15	+0.12	+0.09	+0.06	+0.03	**0**	-0.03	-0.06	-0.09	-0.12	-0.15
45	+0.30	+0.24	+0.18	+0.12	+0.06	**0**	-0.06	-0.12	-0.18	-0.24	-0.30
55	+0.45	+0.36	+0.27	+0.18	+0.09	**0**	-0.09	-0.18	-0.27	-0.36	-0.45

Trouble shooting

Wild readings, check for air bubbles in the electrode tip.

Response time and stability are affected by the condition of the electrodes glass membrane, reference junction and reference solution. Restoration to acceptable levels can often be accomplished by cleaning the electrode's glass surface.

Sluggish response, erratic readings, or a reading that will not change can indicate electrode demise.

Interference may occur between electrochemical sensors (**pH, Oxygen**, and **Conductivity)** if they are placed in the same solution at the same time and connected to the same **EASY**SENSE unit. This is because these sensors make an electrical connection to the solution; therefore an electrical path exists between the sensors through the solution. Maximise the distance between the Sensors to minimise the effect, the distance required will depend on the conductivity of the solution.

If the Sensors are being used in a solution that has a high conductance e.g. seawater, either

- Connect the Sensors to different **EASY**SENSE units or
- Take readings from the Sensors individually. (Place one Sensor in the solution, take a reading, and remove from the solution. Place the other Sensor in the solution, take a reading and remove).

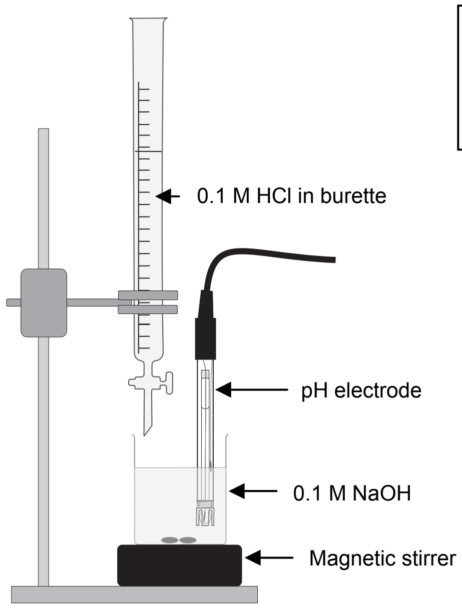
Investigations

- *Acid - base titration*
- *Monitoring photosynthesis*
- *Respiration*
- *Fermentation*
- *Activity of enzyme*
- *Studies of household acids & bases*
- *Monitoring pH change during chemical reaction*

Neutralisation of a strong base (sodium hydroxide) by a strong acid (hydrochloric acid)

This experiment uses the pH Sensor to monitor the pH as 0.1 mol dm^{-3} hydrochloric acid is added to a beaker containing 0.1 mol dm^{-3} sodium hydroxide.

Sodium hydroxide
0.5 to 2% (Solutions 0.05 to 0.5 mol dm^{-3})
Xi; R36/38
Wear pvc gloves and eye protection.
Sodium hydroxide solution is dangerous to eyes
Refer to Hazard Sheets for First Aid Measures

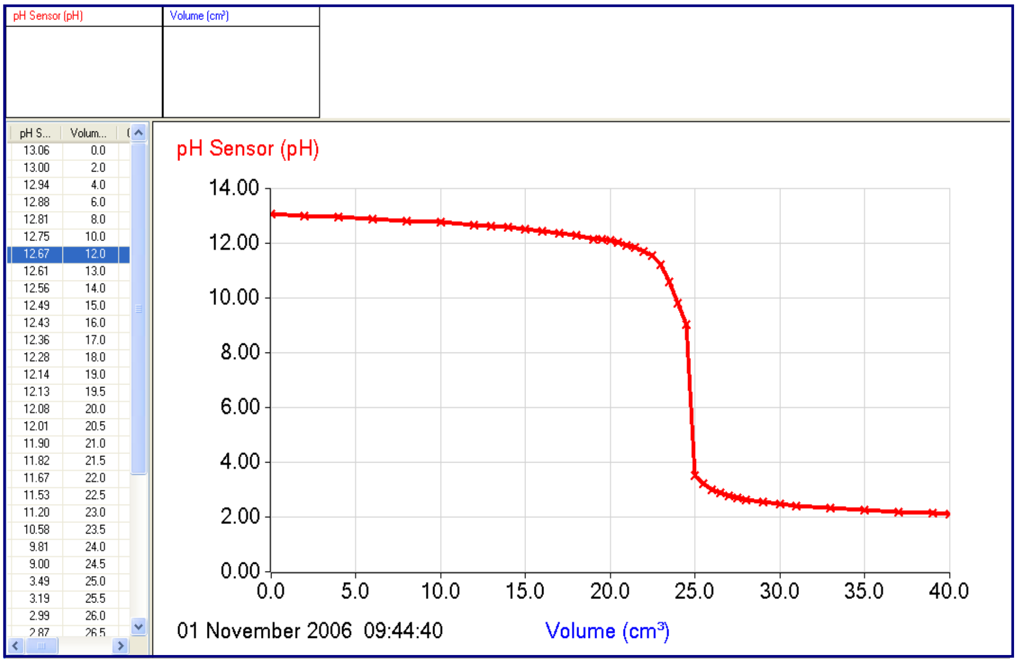


1. Assemble the apparatus as shown.
2. Carefully fill the burette with 0.1 mol dm^{-3} hydrochloric acid. Place a beaker under the reservoir and open the stopcock to allow a small amount of HCl to pass through. Pour the HCl from the beaker back into the burette.
3. Add 25 cm^{3*} of 0.1 mol dm^{-3} sodium hydroxide to the beaker and make sure the end of the pH electrode is covered.

   ***Note:** The volume of alkali may need to be increased, the solution should cover the bulb end of the pH electrode. Do **not** remove the electrodes protective skirt - the end is made from permeable glass, which is fragile and easily damaged*

4. Place a magnetic stir bar in the beaker and turn on the stirrer. Check the stir bar rotates freely and does not make contact with the electrode.
5. Open the **EASY**SENSE program and select **Snapshot** from the Home page.
6. Select **Pre-log Function** from the **Tools** menu.
7. Select a **Preset** function, with Titration from the first drop-down list and then **Asks for the Volume added** from the second list, Next and then Finish.
8. From the **Options** icon select **X-Axis** and select **Channel.** OK. If necessary, click below the X-axis so that 'Volume' is displayed.

9. Click on the **Start** icon to begin. Click in the graph area to record the first pH value with no acid added. Type 0 into the 'enter value box', OK.
10. Turn the tap on the burette to add a measured amount e.g. 2 cm^3. Let the solutions mix and then click in the graph area to record the pH value. Enter the volume of acid added so far.
11. Repeat the above until the pH value falls and levels out. The amount of acid added can be varied as required.
12. Click on the **Stop** icon to finish recording.



In this graph the quantity of acid added started at 2 cm^3 between each reading, after 12 cm^3 this was reduced to 1cm^3, and then down again to 0.5 cm^3 after 19 cm^3 had been added.

Buffers

Buffers are solutions that have constant pH values and the ability to resist changes in that pH value.

To make up your own solutions:

pH	Add	of	to	of
4.0	2 cm^3	0.1 mol dm^{-3} HCl	1000 cm^3	0.1 mol dm^{-3} potassium hydrogen phthalate
7.0	582 cm^3	0.1 mol dm^{-3} NaOH	1000 cm^3	0.1 mol dm^{-3} potassium dihydrogen phosphate
10.0	214 cm^3	0.1 mol dm^{-3} NaOH	1000 cm^3	0.05 mol dm^{-3} sodium bicarbonate

Warranty

All Data Harvest Sensors are warranted to be free from defects in materials and workmanship for a period of 12 months from the date of purchase provided they have been used in accordance with any instructions, under normal laboratory conditions. This warranty does not apply if the Sensor has been damaged by accident or misuse.

Damage to the pH sensitive glass bulb is **not** covered by warranty.

In the event of a fault developing within the 12 month period, the Sensor must be returned to Data Harvest for repair or replacement at no expense to the user other than postal charges.

Note: Data Harvest products are designed for **educational** use and are not intended for use in industrial, medical or commercial applications.

WEEE (**W**aste **E**lectrical and **E**lectronic **E**quipment) Legislation

Data Harvest Group Ltd is fully compliant with WEEE legislation and is pleased to provide a disposal service for any of our products when their life expires. Simply return them to us clearly identified as ‘life expired’ and we will dispose of them for you.