Acid - base titration with drop counter

Technician and teacher sheet

Apparatus

Drop counter (set 27 drops per cm^3 range)

pH sensor

Drop counter reagent reservoir with two stopcocks.

2 x 200 ml beakers

Magnetic stirrer and follower (flea).

20 cm^3 of 0.1 mol dm^{-3} Sodium Hydroxide (NaOH)

40 cm^3 of 0.1 mol dm^{-3} Hydrochloric acid (HCl)

Measuring cylinders (50 ml capacity)

Accurate small volume device e.g. 2.5 or 1.0 ml syringe barrel (syringe connector blocked)

Data recording setup.

Sample period 2 seconds

Select stop to finish

(Expect a titration to take about 8 - 10 minutes)

Introduction

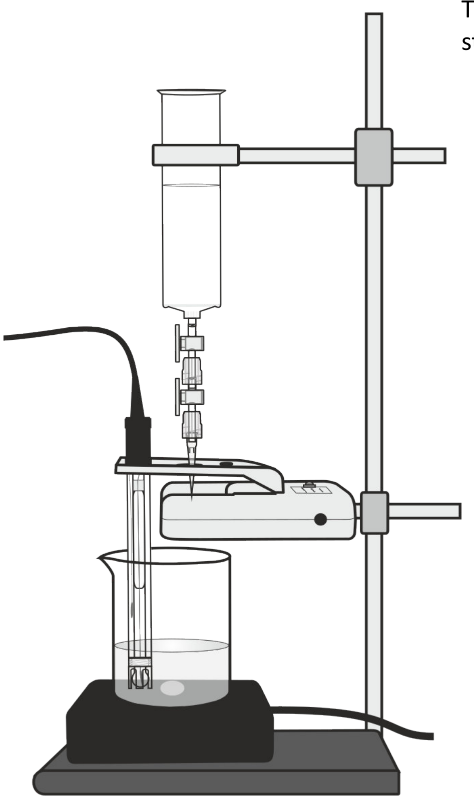
In this investigation the Drop counter is user to electronically measure the volume of acid titrated into a known volume of alkali. The drop counter gives a very accurate titre (one drop at a time).The experiment will use acid (Hydrochloric) and alkali (Sodium hydroxide) of the same molar strength (0.1 mol dm^{-3}).

The equation of the reaction is,

$$\mathbf{NaOH_{(aq)} + HCl_{(aq)} \quad NaCl_{(aq)} + H_2O_{(l)}}$$

As it is a 1:1 reaction (that is 1 particle of NaOH reacts with 1 particle of HCl) the end point of the reaction (when using solutions of the same molar strength) should be when the same of amount of acid is run into the same amount of alkali. In the investigation double the volume of acid to alkali is used to make sure the end point is reached and the titration curve is extended beyond.

The method described will work for the standard titrations of strong / strong, strong / weak and weak / weak.



This represents a generic setup for the use of the drop counter.

A beaker is used to give space for the apparatus.

The wireless pH sensor electrode connects direct to the sensor body, but extension BNC leads are availableto move the sensor body away from the titration apparatus. You will need a male - female extension

Calibrating the Drop counter

This investigation uses the Drop counter with one of its preset calibrated ranges e.g. 27 drops/cm^3 so the drops counted are automatically converted and displayed as volume in cm^3.

If accuracy is not critical and you are using the reagent reservoir and tip supplied with a low viscosity liquid (like water) and the flow rate set to: -

- Fast e.g. 10 plus drops per second, use the **24 drops**/cm^3 range.
- Medium e.g. between 5 – 10 drops per second, use the **25 drops**/cm^3 range.
- Slow e.g. between 1.5 – 5 drops per second, use the **26 drops**/cm^3 range.
- Very Slow e.g. less than 1.5 drops per second, use the **27 drops**/cm^3 range

When used with a pH Sensor, the flow rate is best set very slow (less than 1.5 drops per second) to allow the pH Sensor time to settle to a new reading after addition of the titrant.

To calculate the number of drops in a cm3

- Set up the reagent reservoir in the alignment adapter of the Drop / Bubble counter. Close both stopcocks and fill the reservoir with the type of solution being used.
- The first step is to adjust the flow rate. Place a beaker under the stopcock to catch the drops. Fully open the lower stopcock. Slowly turn the top stopcock round until it begins to produce drops and then finely adjust the drop rate. When the correct flow rate of drops is achieved close the lower stopcock to stop the flow. Now the 'flow rate' is set, do not adjust the top stopcock – leave in this position. Use the lower stopcock to turn the drops on and off.
- Top up the reservoir. Place an accurate measuring container e.g. volumetric flask (10 cm^3 or less) under the dropping tip. Open the lower stopcock fully and count the number of drops required to fill up to the volume mark on the measuring container. You can use the Drop / Bubble counter to count the total number of drops (set to the Drop / bubble count range). Make sure you zero the Sensor before each run. Close the lower stopcock to stop the drops.
- Divide the number of drops by the volume (in cm^3) to get the drops per cm^3 value e.g. 272 drops fill a capacity of 10 mls = 27.2 drops/cm^3. Top up the reservoir and repeat three times to get an average value.

Practical notes

Using the Drop counter does allow the student to move away from the practical, this may free up some time to discuss calculations used in titration e.g. molar calculations or pKa values.

The high granularity of the data does mean that students have enough data to produce and equivalence point estimation by first and second derivative, either through spreadsheet calculation or functions within the data logging software.

A typical drop counter titration will take between 6 and 30 minutes, depending upon the flow rate selected.

Analysis

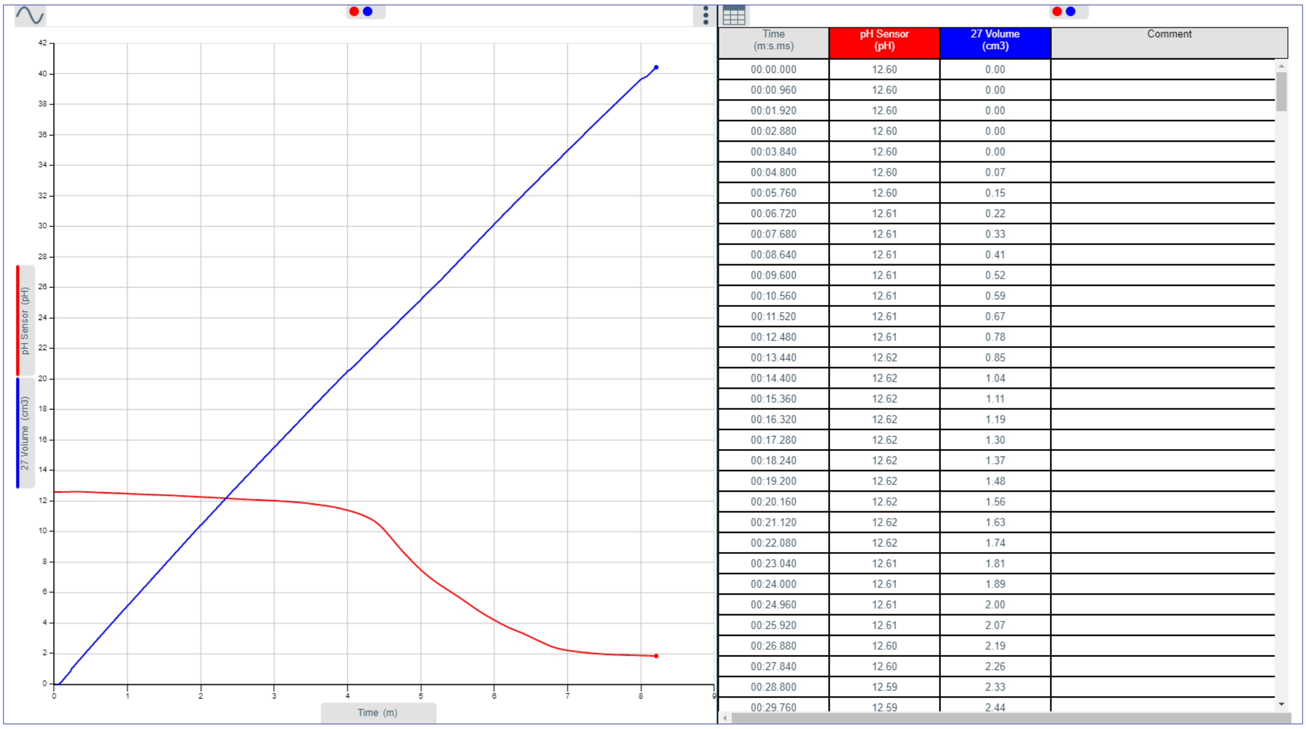
If the purpose of the data is to find the volume (and therefore concentration, molar quantities) the values tool will give pH and Volume data without any further treatment of the data.

If the data is to be presented as a pH vs. Volume graph, this can be done within ES2 by changing the axis label of the axis and selecting the sensor to be displayed.

If you wish to consider the "first derivative" approach where the peak of the rate of change of pH is used to pin point the equivalence point and volume, this can also be conducted in ES2. Use the calculation dx/dy, use pH as the data series for the calculation. This will give a sharp peak at the "point", the values tool can then be used to find the volume.

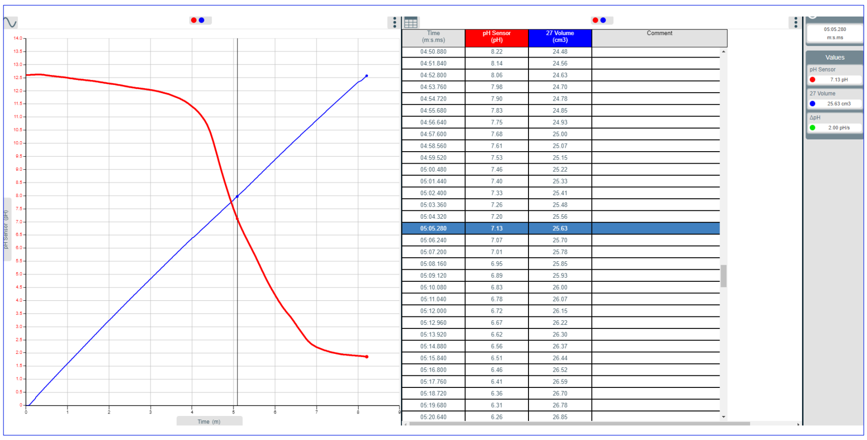
A further application of the dx/dy calculation on the 1st derivative data will give a more accurate (sic) point, the 2nd derivative plot will create a line that crosses the x axis at equivalence.

Example data



Time (m:s.ms)	pH Sensor (pH)	27 Volume (cm3)	Comment
00:00.000	12.60	0.00	
00:00.960	12.60	0.00	
00:01.920	12.60	0.00	
00:02.880	12.60	0.00	
00:03.840	12.60	0.00	
00:04.800	12.60	0.07	
00:05.760	12.60	0.15	
00:06.720	12.61	0.22	
00:07.680	12.61	0.33	
00:08.640	12.61	0.41	
00:09.600	12.61	0.52	
00:10.560	12.61	0.59	
00:11.520	12.61	0.67	
00:12.480	12.61	0.78	
00:13.440	12.62	0.85	
00:14.400	12.62	1.04	
00:15.360	12.62	1.11	
00:16.320	12.62	1.19	
00:17.280	12.62	1.30	
00:18.240	12.62	1.37	
00:19.200	12.62	1.48	
00:20.160	12.62	1.56	
00:21.120	12.62	1.63	
00:22.080	12.62	1.74	
00:23.040	12.61	1.81	
00:24.000	12.61	1.89	
00:24.960	12.61	2.00	
00:25.920	12.61	2.07	
00:26.880	12.60	2.19	
00:27.840	12.60	2.26	
00:28.800	12.59	2.33	
00:29.760	12.59	2.44	

The example data is shown as collected. A data table has been shown to show the detail of the data collected.

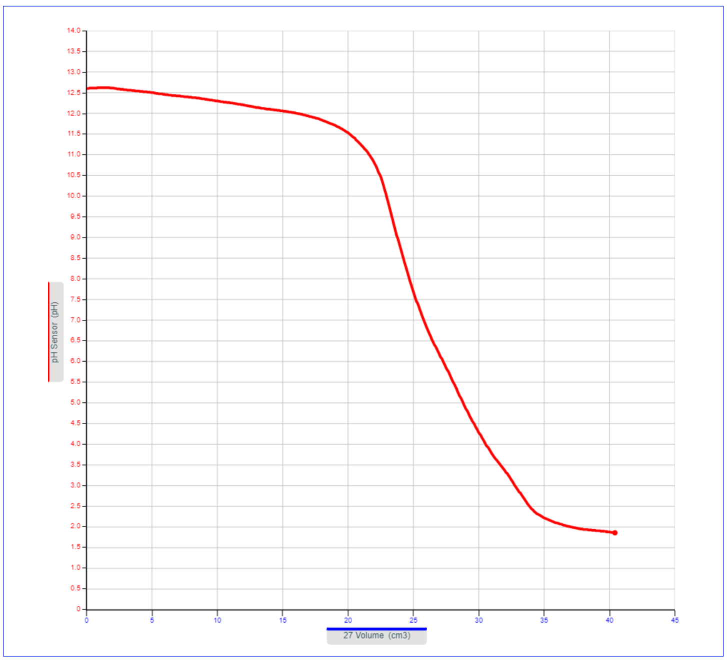


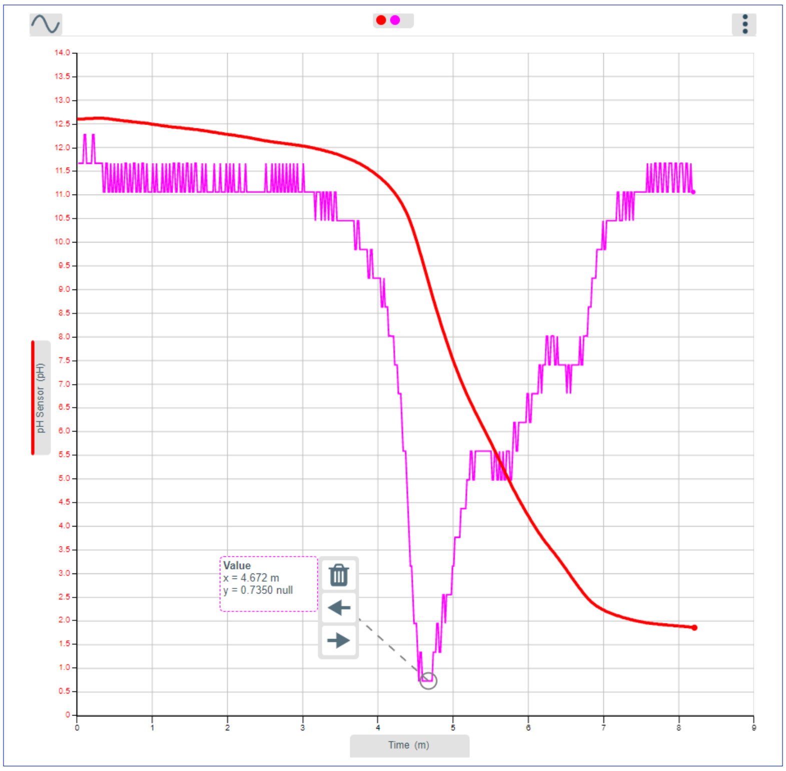
Time (m:s.ms)	pH Sensor (pH)	27 Volume (cm3)	Comment
04:50.880	8.22	24.48	
04:51.840	8.14	24.56	
04:52.800	8.06	24.63	
04:53.760	7.98	24.70	
04:54.720	7.90	24.78	
04:55.680	7.83	24.85	
04:56.640	7.75	24.93	
04:57.600	7.68	25.00	
04:58.560	7.61	25.07	
04:59.520	7.53	25.15	
05:00.480	7.46	25.22	
05:01.440	7.40	25.33	
05:02.400	7.33	25.41	
05:03.360	7.26	25.48	
05:04.320	7.20	25.56	
05:05.280	7.13	25.63	
05:06.240	7.07	25.70	
05:07.200	7.01	25.78	
05:08.160	6.95	25.85	
05:09.120	6.89	25.93	
05:10.080	6.83	26.00	
05:11.040	6.78	26.07	
05:12.000	6.72	26.15	
05:12.960	6.67	26.22	
05:13.920	6.62	26.30	
05:14.880	6.56	26.37	
05:15.840	6.51	26.44	
05:16.800	6.46	26.52	
05:17.760	6.41	26.59	
05:18.720	6.36	26.70	
05:19.680	6.31	26.78	
05:20.640	6.26	26.85	

Values tool being used to find pH and volume data.

Axes changed to give pH vs Volume plot more familiar to text book illustrations.

When using electronic graphing with data selection this is no a necessary graph to produce, data can be found electronically by use of tools in the software.





First derivative graph, the mathematical application amplifies positions where data does not change and shows many erratics. But it is obvious where the peak of greatest change of pH is taking place and the local point tool can be used to find the volume.

In this example the x axis has been changed to time to allow use of the values global tool.

Software knowledge required.

1. Connecting the sensor(s) to the software.
2. Change the range of the sensors to correct drops range
3. Use set up to change the intersample period (1 count per 2 seconds)
4. Change axis limits.
5. Use values, difference and interval to find times between areas of interest
6. Use the runs manager to show individual or multiple runs on the same chart.
7. Use calculate to create 1st and 2nd derivatives