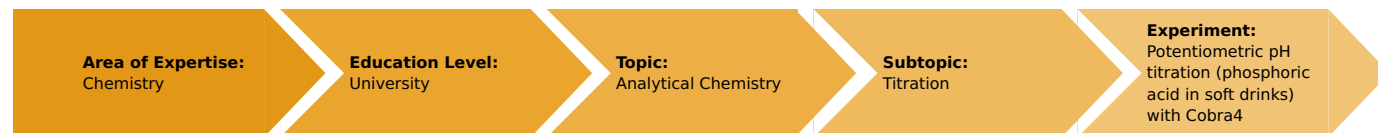


Potentiometric pH titration (phosphoric acid in soft drinks) with Cobra4 (Item No.: P3061760)

Curricular Relevance



Difficulty



Difficult

Preparation Time



1 Hour

Execution Time



2 Hours

Recommended Group Size



2 Students

Additional Requirements:

- PC with USB interface, Windows XP or higher
- Soft drink (Cola) containing phosphoric acid

Experiment Variations:

Keywords:

Galvanic cell, Galvani voltage, cell voltage, Nernst equation, potentiometry, volumetry, pH value

Overview

Short description

Principle

The cell voltage and the Galvani voltage of the electrodes of a galvanic cell are dependent upon the concentration of the ions involved in the potential forming process. Thus, conclusions can be made about the concentration of the ions to be investigated from the measured cell voltage at a constant potential of a suitable reference electrode (potentiometric titration).

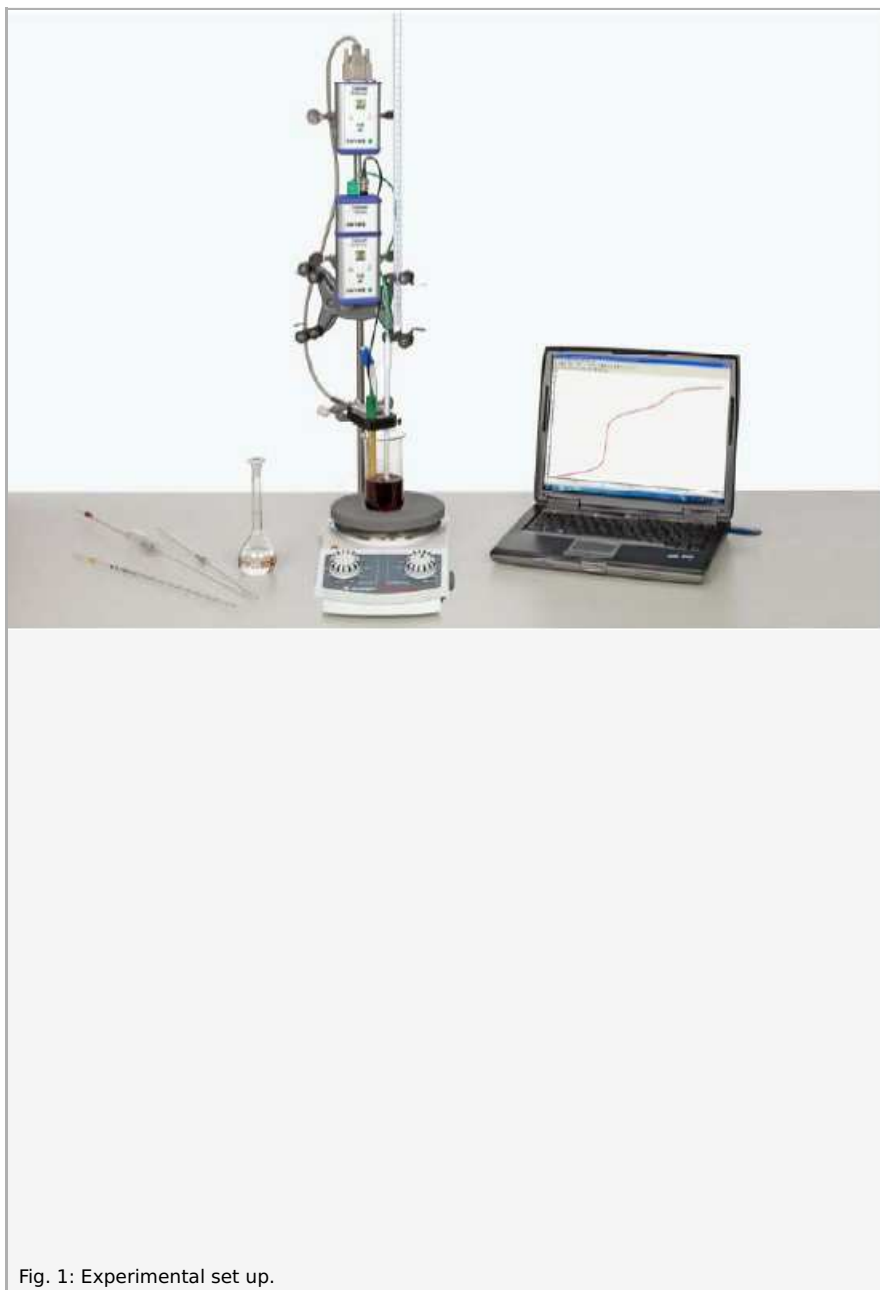
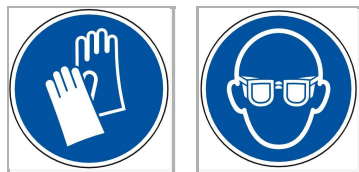


Fig. 1: Experimental set up.

Safety instructions



When handling chemicals, you should wear suitable protective gloves, safety goggles, and suitable clothing. Please refer to the appendix for detailed safety instructions.

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Caustic soda solution, 0,1 N

H290: May be corrosive to metals.

P234: Keep only in original container

P280: Wear protective gloves/protective clothing/eye protection/face protection.

Trisodium phosphate

H315: Causes skin irritation.

H319: Causes serious eye irritation.

H335: May cause respiratory irritation.

P280: Wear protective gloves/protective clothing/eye protection/face protection.

Equipment

Position No.	Material	Order No.	Quantity
1	Cobra4 Wireless/USB-Link incl. USB cable	12601-10	2
2	Cobra4 Sensor-Unit Chemistry	12630-00	1
3	Cobra4 Sensor-Unit Drop Counter	12636-00	1
4	Holder for Cobra4 with support rod	12680-00	2
5	USB charger for Cobra4 Mobile-Link 2 and Wireless/USB-Link	07932-99	2
6	pH-electrode, plastic body, gel, BNC	46265-15	1
7	Immersion probe NiCr-Ni, teflon, 300 °C	13615-05	1
8	Magnetic stirrer with heater MR Hei-Standard	35751-93	1
9	Magnetic stirring bar 15 mm, cylindrical	46299-01	1
10	Right angle boss-head clamp	37697-00	3
11	Retort stand, h = 750 mm	37694-00	1
12	Burette clamp, roller mount., 2 pl.	37720-00	1
13	Burette, lateral stopcock, Schellbach, 50 ml, graduations 0, 1 ml	36513-01	1
14	Funnel, d.40 mm, f.burettes	36888-00	1
15	Beaker, high, BORO 3.3, 50 ml	46025-00	2
16	Beaker, high, BORO 3.3, 150 ml	46032-00	1
17	Beaker, low, BORO 3.3, 150ml	46060-00	1
18	Beaker, low, BORO 3.3, 250 ml	46054-00	1
19	Volumetric pipette, 50 ml	36581-00	1
20	Pipettor	36592-00	1
21	Wash bottle, plastic, 500 ml	33931-00	1
22	Caustic soda sol., 0.1M 1000 ml	48328-70	1
23	Buffer solution, pH 4.62 1000 ml	30280-70	1
24	Buffer solution, pH 9 1000 ml	30289-70	1
25	Water, distilled 5 l	31246-81	1
26	curricuLAB measureLAB	14580-61	1

Task

Using the Cobra4-System, measure the change in pH in the titration of a sample of a carbonated beverage (Cola) containing phosphoric acid (E 338) with 0.1 molar sodium hydroxide solution and calculate the beverage's phosphoric acid content from the consumption of the standard solution.

Set-up and procedure



- Set up the experiment as shown in Fig. 1.
 - Combine the Cobra4 Sensor Unit Chemistry and the Cobra4 Drop Counter with the Cobra4 Wireless-Links.
 - Attach them to the retort stand with the holders for Cobra4 and right angle clamps.
 - Connect the pH electrode to the pH input of the Cobra4 Sensor Unit Chemistry and the temperature probe to temperature input T1.
 - Start the PC and connect the Cobra4 Wireless Manager with a USB socket of the computer.
 - After the Cobra4 Wireless-Links have been switched on, the sensors are automatically recognized. Some ID numbers (01 and 02) are allocated to the sensors, which are indicated in the displays of the Cobra4 Wireless-Links.
 - Call up the "measure" programme.
 - Click the "Unknown titration volume" button and confirm with "OK".
 - Boot the experiment "Titration of a soft drink with a strong acid with Cobra4" (experiment > open experiment). The measurement parameters for this experiment are loaded now.
-
- **For calibration:** Pour some buffer solution with pH 4.62 and pH 9.00 in two beakers.
 - Immerse the well-rinsed probe into one of the solutions.
 - In the Cobra4 Navigator under "Devices" double-click the "pH" symbol. Now you can change some measurement parameters.
 - Enter the pH value for the given solution under the menu point "Calibration" (Step 1, see Fig. 2).



Fig. 2: Settings for the calibration mode of the sensor.

- Click the “Apply” button.
- Repeat this procedure with the other buffer solution (Step 2).
- Finish the calibration with “OK”.

Procedure:

- Pour approximately 150 ml of the soft drink into a 250 ml beaker. Place the beaker on the magnetic stirrer and heat it carefully to remove dissolved carbon dioxide. Allow to cool to room temperature.
- Position the magnetic heating stirrer under the stand.
- Pipette 50 ml of the soft drink in the 150 ml beaker and slip a magnetic stirring rod in.
- Fill the burette up to the 50 ml mark with 0.1 molar sodium hydroxide solution.
- Position the beaker containing the soft drink on the magnetic stirrer so that the pH measuring electrode dips into the solution (*Note:* The pH electrode must dip at least so deep in the solution that the diaphragm is completely immersed in the solution. Add more water if necessary).
- Position the tap of the burette so that sodium hydroxide solution can drop into the beaker. Also ensure that individual drops will be recorded by the drop counter.
- Adjust the stirrer to a medium stirring speed (*Note:* Do not allow the magnetic stirring bar to hit against the measuring electrode.)
- Start measurement with a click on ● in the icon strip.
- Add sodium hydroxide solution drop-wise to the soft drink solution from the burette. (*Note:* Take care that the addition of the drops is not so rapid that the light barrier cannot register individual drops.)
- When about 30 ml of sodium hydroxide has been so added, close the tap of the burette and click on ■ the icon to terminate the measurement.
- Send all data to “measure” (see Fig. 3). Save the measurement (File > Save measurement as...).

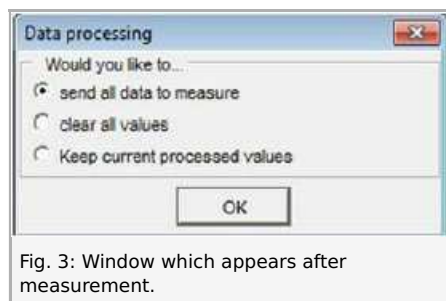
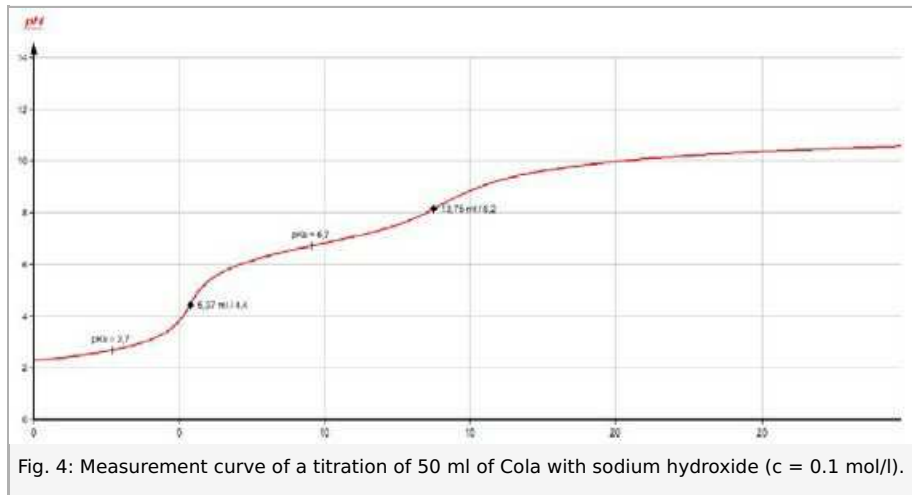


Fig. 3: Window which appears after measurement.

Fig. 4 shows the graph as presented by the programme.



To have the equivalence point and the pK_a value displayed use

Theory and evaluation

The titration curve recorded shows pH as a function of the volume of sodium hydroxide solution added.

The pH measurement is a potentiometric measurement. It can only be carried out, when the potential of the indicator electrode is measured against that of a reference electrode. Both electrodes are contained in single-rod pH measuring systems. A glass electrode is used as the indicator electrode. When this is immersed in an aqueous solution, a swelling layer (gel) is formed at the pH sensitive glass membrane. This also occurs at the inner side of the membrane, which is in contact with a defined buffer solution, commonly of pH 7. According to the pH of the test solution, hydronium ions diffuse either out of, or into, the outer swelling layer, whereby the potential of this layer changes. The pH, and so also the potential, of the inner side of the glass membrane remains constant while this occurs. With regard to the pick-up electrode, the same system is chosen as for the reference electrode (AgCl), so that the potential difference between the two electrodes is equal to zero. The voltage measured with the single-rod measuring system therefore results from the potential difference between the inside and outside swelling layers of the glass electrode.

When the system is dipped into a test solution of pH 7, then a potential of 0 mV should be given, as the inner potential is equal to the outer potential. Theoretically, when the pH changes by a single unit, the voltage should change by 59.16 mV. This can be calculated using the Nernst equation, which is as follows for hydrogen:

$$E = E_0 + 2.3 \cdot \frac{RT}{F} \cdot \log a(H)$$

where

$$E^0 = 0$$

$$\log a(H^+) \cong \log c(H) = -\text{pH}$$

$$2.3 \cdot \frac{RT}{F} = 59.16 \text{ mV (at } T = 298.15 \text{ K)}$$

From this it follows that: $E = -59.16 \text{ mV} \cdot \text{pH}$

T Absolute temperature

R Universal gas constant

F Faraday constant

This rise in the pH-characteristic line is called the slope of the single-rod pH measuring system. The slope of real pH measuring

systems deviates like the zero point from the theoretical value given by the Nernst equation.

Fig. 5 shows the titration curve for pure phosphoric acid (see experiment P3121260 – “Titration of a multiprotic acid with a strong base with Cobra4”).

Two equivalence points can be clearly seen here. They correspond to the following dissociation equilibria:

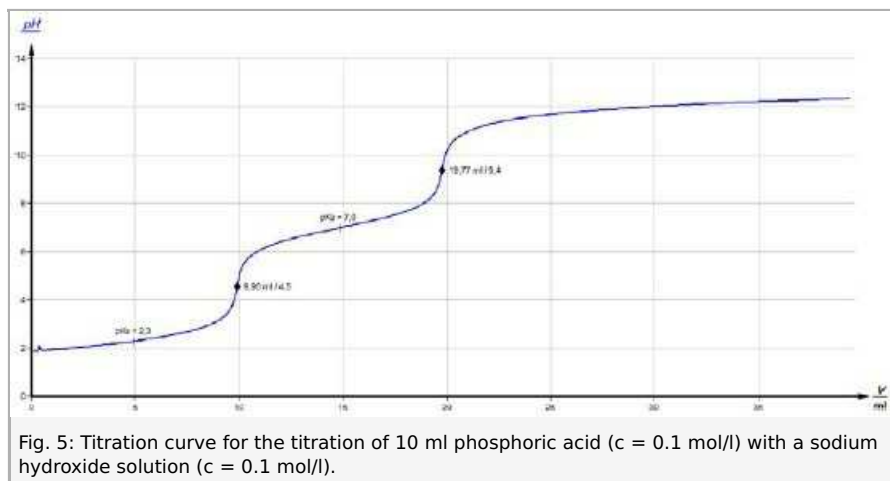
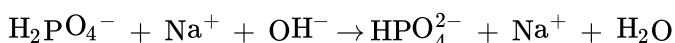
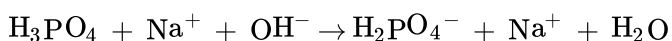


Fig. 5: Titration curve for the titration of 10 ml phosphoric acid ($c = 0.1 \text{ mol/l}$) with a sodium hydroxide solution ($c = 0.1 \text{ mol/l}$).



The second equivalence point is only weakly shown in the titration curve for the soft drink (see Fig. 4). This is because, in addition to phosphoric acid, further acids like citric acid are present here which also take part in the titration with sodium hydroxide solution and exert a buffering effect in the region of the second equivalence point. A knowledge of the first equivalence point suffices for the calculation of the quantity of phosphoric acid contained in the soft drink.

The concentration c_2 , and so the content m_2 of acid-forming substance in the sample of volume V_2 , can be calculated from the corresponding consumption V_1 of standard solution of known concentration c_1 .

$$c_1 V_1 = c_2 V_2 = m_2 / M_2$$

where

M Molar mass of phosphoric acid (= 98 g/mol)

Data and results

The first point of inflection of the titration curve gives $V_1 = 5.37 \text{ ml NaOH}$, from which a phosphoric acid concentration $c_2 = 1.07 \cdot 10^{-2} \text{ mol/l}$ can be calculated, and from this a content of $m = 52.63 \text{ mg H}_3\text{PO}_4$ in the volume of the sample tested ($V_2 = 50 \text{ ml}$).

A 1.5 l bottle of the soft drink tested therefore contains 1578.8 mg of phosphoric acid.

Disposal

The diluted and neutralised solutions of the used acid and base can be disposed by rinsing into the drain.

