Manganometric titration of iron(II) ions

Aims of the experiment

- To learn that a titration that does not depend on a neutralisation reaction.
- To learn potentiometry as an analytical measurement method.
- To track the potential change as a function of the addition of a reagent solution.
- To determine the iron(II) concentration through titration with a $KMnO_4$ solution.
- To understand manganometry as a sub-type of potentiometry.

Principles

Titration in general is a method of quantitative analysis. In titration, the concentration of a substance in a solution is determined through the addition of a titrant. The titrant has a known concentration and enters into a specific reaction with the substance to be investigated. Titrations are based on a variety of reactions. There is acid-base titration, complexometric titration, redox titration or precipitation titration. Potentiometric titration is also a titration method. Manganometric titration in this experiment is a sub-type of potentiometric titration.

Potentiometry is an electroanalytic method in quantitative analytical chemistry. In this method, the concentration dependence of the electrical potential is utilised. As described above, a substance is titrated using a titrant. During titration, an immediate reaction results in a measurable change in potential. The titration is tracked using an indicator electrode. For example, this can be a pH electrode or a redox electrode.

At the equivalence point of the reaction, the potential suddenly increases until it continues on in a plateau. This strong change in potential is the result of the fact that the voltage, prior to the equivalence point, is determined by the ions of the substance to be analysed. After the equivalence point, ions from the titrant are present in excess and determine the voltage. Then, the concentration of the substance to be determined can be calculated via the equivalence point.

Strictly speaking, pH titrations are also potentiometric titrations. Calibration converts the voltage to a pH in this case.

In this experiment, iron(II) is determined potentiometrically through titration with potassium permanganate. Manganometry is a redox titration that proceeds without the use of an additional indicator. In this case, the property being utilised is the fact that MnO_4^- ions are very red-violet in colour, and turn colourless when they are reduced to Mn^{2+}. In this experiment, the reaction is as follows:

$$MnO_4^- + 5\ Fe^{2+} + 8\ H^+ \rightleftarrows Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$$

In the reaction, a measurable change in voltage *U* occurs which can be tracked, and the equivalence point can be determined using it. The tracking of the voltage change is determined more precisely than the equivalence point simply because of the colour change.

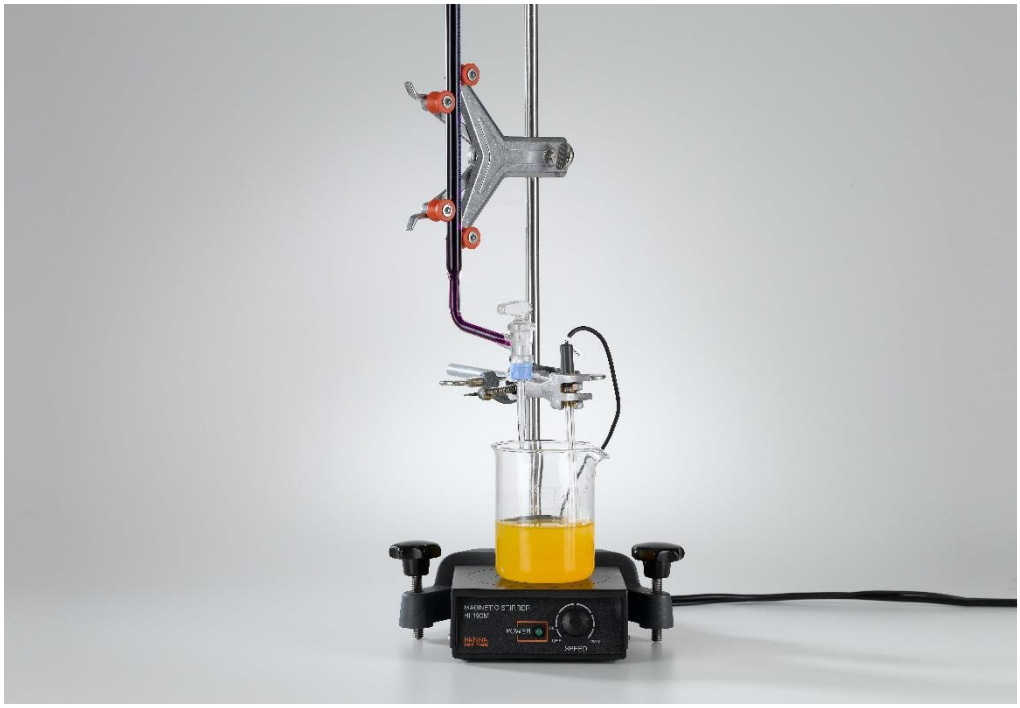

Fig. 1: Setup of the Experiment.

Risk assessment

When working with potassium permanganate, make sure that it does not come into contact with skin or surfaces since this will lead to unsightly brown spots. Using sulphuric acid requires personal protective equipment to be worn.

Potassium permanganate

Signal word:
Hazard

Hazard statements

H272 May intensify fire; oxidiser agent.

H302 Harmful if swallowed.

H410 Very toxic to aquatic life with long-lasting effects.

Safety statements

P210 Keep away from heat/sparks /open flames/hot surfaces. No smoking.

P273 Avoid release to the environment.

Sulphuric acid, dilute, approx. 2N

Signal word:
Caution

Hazard statements

H290 Can corrode metals.

H315 Causes skin irritation.

H319 Causes serious eye irritation.

Safety statements

P280 Wear protective gloves / eye protection.

P302+P352 IF ON SKIN: Wash with plenty of water and soap.

P305+P351+P338 IF IN EYES:

Rinse carefully with water for several minutes. Remove contact lenses if present and easy to do so. Continue rinsing.

P337+P313 If eye irritation persists: Get medical advice/attention.

Iron(II) sulphate · 7 H_2O

Signal word:
Caution

Hazard statements

H302 Harmful if swallowed.

H315 Causes skin irritation.

H319 Causes serious eye irritation.

Safety statements

P302+P352 IF ON SKIN:

Wash with plenty of water and soap.

P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and easy to do so. Continue rinsing.

Equipment and chemicals

1	Pocket-CASSY 2 Bluetooth	524 018
1	CASSY Lab 2	524 220
1	pH adapter S	524 0672
1	Single-rod Redox probe, BNC	667 416
1	Electronic Balance 440-3N, 200 g : 0.01 g	667 7977
1	Mini magnetic stirrer	607 105
1	Beaker, DURAN, 250 ml, squat	664 103
1	Volumetric flask Boro 3.3, 25 ml	665 791
1	Volumetric flask Boro 3.3, 50 ml	665 792
1	Stand rod 47 cm, 12 mm diam.	300 42
1	Stand base, V-shaped, small	300 02
1	Graduated pipette 10 mL	665 997
1	Pipetting ball	666 003
1	Burette, clear glass, 25 ml	665 845
1	Burette filling funnel, plastic, 25 mm diam.	665 816
1	Burette clamp, simple	666 559
1	Bosshead S	301 09
1	Universal clamp 0...80 mm	666 555
1	Iron(II) sulphate-7-hydrate, 100 g	671 9100
1	Potassium permanganate, 100 g	672 7000
1	Sulphuric acid, dilute, approx. 2 N, 500 ml	674 7920
1	Glucose, 100 g	672 1100
Also necessary for wireless measurement:		
1	Rechargeable battery for Pocket-CASSY 2 BT	524 019
1	Bluetooth dongle	524 0031

Set-up and preparation of the experiment

Set-up of the apparatus

1. The apparatus is set up as can be seen in Fig. 1.
2. Fix the stand rod in the stand base.
3. Clamp the single-rod redox probe to the stand rod using a bosshead S clamp and a universal clamp.
4. Connect the single-rod redox probe to the Pocket CASSY 2 using the pH adapter S.
5. Connect the Pocket CASSY 2 Bluetooth to a computer using a USB cable.

Note: For wireless measurement, connect the battery to the Pocket CASSY 2 and connect to the PC using the Bluetooth Dongle, which connects to one of the USB ports of a PC.

6. Also attach the burette clamp to the stand rod and place the burette into the holder.
7. Position an empty beaker with a stirring magnetic onto the magnetic stirrer.

Preparation of the experiment

1. Two solutions are needed for the experiment. First of all a 0.1 molar solution of iron(II) sulphate • 7 H_2O and secondly a 0.02 molar $KMnO_4$ solution.

Calculating the weight for Fe(II) sulphate • 7 H_2O

$c(FeSO_4 \cdot 7\,H_2O) = 0.1$ mol/l

$M(FeSO_4 \cdot 7\,H_2O) = 278$ g/mol

$V(FeSO_4 \cdot 7\,H_2O) = 25$ ml

$m(FeSO_4) = c(FeSO_4) \cdot M(FeSO_4) \cdot V(FeSO_4)$

$m(FeSO_4) = 0{,}1$ mol/l · 278 g/mol · 0.025 l

$m(FeSO_4) = 0.70$ g

Calculating the weight for $KMnO_4$

$c(KMnO_4) = 0.2$ mol/l

$M(KMnO_4) = 158.03$ g/mol

$V(KMnO_4) = 25$ ml

$m(KMNO_4) = c(KMnO_4) \cdot M(KMnO_4) \cdot V(KMnO_4)$

$m(KMnO_4) = 0.2$ mol/l · 158,03 g/mol · 0.025 l

$m(KMnO_4)$= 0.08 g

2. Weigh in both substances and dissolve in a volumetric flask of suitable size. Add enough water to the flask until the bottom of the meniscus of the water surface lines up with the calibration mark.

3. Place the potassium permanganate solution in the burette. Use a small funnel to help do this. As with the volumetric flask, fill the burette until the bottom of the meniscus of the liquid level lines up with the 25 ml mark.

Note: If too much solution is added to the burette, simply drain the excess back into the volumetric flask by opening the burette. Before filling the burette, test to make sure that the stopcock opens and closes freely. If not, apply a bit of stopcock grease.

4. Pipette 10 ml of the Fe_2SO_4 solution into the beaker using a graduated pipette and a pipetting ball and acidify with a small bit of diluted sulphuric acid.

Performing the experiment

1. Load the settings in CASSY Lab 2.

2. Dip the redox electrode into the beaker and fill it with water until the electrode dips just below the small lateral opening.

Note: Make sure that the stirring magnet cannot hit the redox electrode.

3. Turn on the magnetic stirrer to mix the liquid.

4. The measurements are recorded manually. This means that after a first stable measurement has established itself, the first measurement is recorded by clicking the button on the Pocket CASSY. Alternatively, the measurement can also be recorded by clicking the Measurement symbol in the CASSY Lab 2 software.

5. Now add 0.5 ml of potassium permanganate solution at a time to the Fe_2SO_4 solution and record a new measurement after each addition by clicking the Pocket CASSY.

6. Continue adding until a steep increase in the measurement curve occurs and a plateau forms thereafter.

Observation

At the beginning of the experiment, a clear Fe_2SO_4 solution is present in the beaker. When the potassium permanganate solution is added, a brief discolouration of the liquid will be observed initially at the drop point; this will disappear again almost instantaneously. The further the measurement proceeds, the more the solution assumes a red-violet colour. After reaching the equivalence point, the solution will be nearly as strong in colour as the potassium permanganate in the burette.

The titration diagram is shown in Figure 2.

Evaluation

Evaluation of the experiment is done using CASSY Lab 2. To determine the exact point of equivalence using CASSY Lab, click the right mouse button in the diagram and select the **Determine equivalence point** sub-item in the **Other evaluations** in the context menu. Mark the curve range and the equivalence point is provided by CASSy Lab. The value of the equivalence point can be added to the diagram by making another right click in the diagram and selecting **Text** under the **Set mark** point (see Fig. 2).

The concentration of the iron solution can be evaluated through calculation from the volume of potassium permanganate determined. In the process, be aware that a MnO_4^- ion can oxidise 5 Fe^{2+} ions.

Therefore, to determine the concentration of the Fe_2SO_4 solution, use the following formula:

$$c(Fe_2SO_4) = \frac{5 \cdot c(KMnO_4) \cdot V(KMnO_4)}{V(Fe_2SO_4)}$$

The following applies:

$c(KMnO_4)$ = 0.2 mol/l

$V(KMnO_4)$ = 10.2 ml

$V(Fe_2SO_4)$ = 10 ml

$$c(Fe_2SO_4) = \frac{5 \cdot 0.02\ \text{mol/l} \cdot 10.2\ \text{ml}}{10\ \text{ml}}$$

$$c(Fe_2SO_4) = 0.102\ \text{mol/l}$$

Since 10 ml were used, the deviation between the determined and the theoretical value is negligibly small.

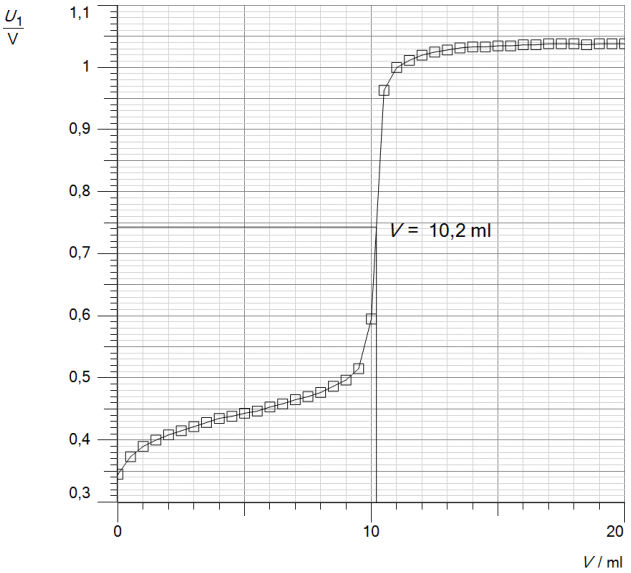

Fig. 2 Measurement curve for manganometric titration of iron(II) ions.

Results

In this experiment, a manganometric titration is carried out and the concentration of iron(II) ions in a solution is determined. An Fe_2SO_4 solution is titrated with a potassium permanganate solution of known concentration for this purpose. First, the colour of the permanganate solution always disappears since the added permanganate ions reduce to Mn^{2+} ions and Fe^{2+} reduces to Fe^{3+} ions. As soon as nearly all the iron(II) has reacted, the solution slowly changes colour until all iron(II) ions have been oxidised at the equivalence point, and the red-violet permanganate ions are in excess.

The slight deviation of about 2% shows that the titration is a very precise measurement method.

Cleaning and disposal

To dispose of the potassium permanganate, add glucose to the remaining solution. Potassium permanganate reduces to manganese dioxide through oxidation of the glucose and can then be disposed of as heavy metal waste. This should also be done to the solution in the beaker.

The remaining iron(II) sulphate solution can be disposed of as inorganic salt solution with heavy metals.