Analytical method for hydrogen peroxide

Determination of hydrogen peroxide decomposition (16 hours at 96°C)

GENERAL INFORMATION ABOUT THE METHOD

This method describes the determination of the hydrogen peroxide decomposition rate by 16 hours temperature treatment at 96°C. The permanent heating causes a gradually decrease of the hydrogen peroxide concentration.

The decomposition rate indicates the relative decrease of the hydrogen peroxide concentration after 16 hours at 96°C. The hydrogen peroxide content of the sample must therefore be exactly determined before and after the temperature treatment. The determination is carried out by titration with potassium permanganate in a sulfuric acid solution according to the following equation:

$$\mathbf{2\ KMnO_4 + 5\ H_2O_2 + 4\ H_2SO_4 \rightarrow 2\ KHSO_4 + 2\ MnSO_4 + 8H_2O + 5\ O_2}$$

The titration is usually carried out potentiometrically with a redox electrode, supported by electronic titration equipment. Alternatively, the titration can be performed manually. In this case, the end point detection is done visually (sample solution turns pink).

EQUIPMENT

- thermostat with water bath (96°C ± 1°C)
- analytical balance
- volumetric flasks, 100ml
- glass caps for covering the volumetric flasks
- pipettes, 50ml
- beakers (titration beakers), 250ml
- Erlenmeyer flask, 300ml (only for manual titration)
- single-use syringes, 1ml
- dispenser or measuring cylinder, 50ml

TITRATION DEVICES

- electronic titration equipment, prepared with an appropriate method for a potentiometric titration of hydrogen peroxide with potassium permanganate
- platinium redox electrode or Pt-Titrode
- dosing unit with a 50 ml dark glass cylinder including a supply bottle for the titration solution ($KMnO_4$)
- Dosimat or a 50ml dark glass burette (only for manual titration)
- thermostat with water bath (20°C)

REAGENTS

- hydrogen peroxide solution (testing material)
- sulphuric acid c(H_2SO_4) ~ 2.5 mol/l
- potassium permanganate solution c($KMnO_4$) = 0.05 mol/l
- high purity water (osmosis and ion exchange treated drinking water)

SPECIAL SAFETY INSTRUCTIONS

All reagents and chemicals must be handled according to the health and safety regulations. Refer to the safety data sheets.

SPECIAL PROCEDURE INSTRUCTIONS

Danger of decomposition by contact with incompatible materials, contaminants, metals, reducing agents, Alkalis.

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PROCEDURE

Take three volumetric flasks (100ml) for the determination of the decomposition rate and pipette 50ml of the hydrogen peroxide sample into each flask. Cover the flasks with glass caps to avoid loss by evaporation. Leave one of the flasks at room temperature (RT) for the next 16 hours and place the other two flasks in a water bath at 96°C for the next 16 hours. When 16 hours have passed, take the three flasks and temper them to 20°C in another water bath. Finally, fill them up to the 100 ml mark with high purified water and determine the hydrogen peroxide content of each flask (initial and final concentration).

Therefore, place 50 ml of sulfuric acid into a 250ml beaker. Fill a 1ml single-use syringe with the hydrogen peroxide sample material. Place the syringe on the analytical balance, then tare the balance to zero. Afterwards take the syringe and add some sample material into the beaker containing the sulfuric acid. Place the syringe back on the scale and note the sample weight (precision 0.0001g), or if possible, transfer it directly to the titration device. Finally add 50 ml of high purity water to the beaker. A double determination is required from each flask.

Choose an appropriate sample weight considering the 50ml volume glass cylinder containing the potassium permanganate solution. The consumption of potassium permanganate solution should not exceed the cylinders volume.

Sample weights for decomposition test:

H_2O_2 30% - 35%: 0.5g – 0.6g

H_2O_2 50%: 0.4g – 0.5g

H_2O_2 70% - 90%: 0.3g – 0.4g

The end point of the potentiometric titration is determined by a redox electrode (e.g. Platinium-Titrode).

Place the beaker with the prepared sample solution on the magnetic stirrer or the sample rack of the titration station. Immerse the electrode and the potassium permanganate dosing tip into the solution. Select the appropriate method and start the device to titrate the sample with the potassium permanganate solution $c(KMnO_4)$ = 0.05 mol/l under constant stirring.

If specified in the method, the result is calculated and documented by the titration device, otherwise carry out the calculation according to the formula below.

If the determination is carried out manually, titrate the prepared sample solution with potassium permanganate $c(KMnO_4)$ = 0.05 mol/l until a faint pink color persists for at least 30 seconds. The titration has to be carried out under constant stirring or shaking.

To maintain the temperature of the potassium permanganate solution at 20 ° C, the supply bottle should be stored in a thermostat.

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For calculation the titer of the potassium permanganate solution must be known exactly (observe manufacturer's certificates!). If the titer is unknown, a titer determination with di-sodium oxalate (primary standard) must be carried out before.

CALCULATION

Hydrogen peroxide concentration after 16h at RT and after 16h at 96°C (initial and final concentration):

$$\textbf{hydrogen peroxide}\ [\textbf{wt\%}] = \frac{\mathbf{V} * \mathbf{f} * \mathbf{0.425175}}{\mathbf{E}}$$

V = volume of potassium permanganate solution c($KMnO_4$) = 0.05 mol/l consumed for the titration [ml]

f = titer (factor) of the potassium permanganate solution

E = sample weight [g]

Determination of the decomposition rate:

$$\textbf{decomposition}\ [\%] = \frac{(\textbf{initial concentration} - \textbf{final concentration}) * \mathbf{100}}{\textbf{initial concentration}}$$

initial concentration = hydrogen peroxide concentration without temperature treatment (16h at RT) [%]

final concentration = hydrogen peroxide concentration after temperature treatment (16h at 96°C) [%]

ENVIRONMENT/DISPOSAL OF CHEMICALS

The disposal of laboratory quantities of hydrogen peroxide must be in accordance with local regulations.

LITERATURE

- Manufacturers equipment descriptions
- Product information "Hydrogen Peroxide"

REMARKS

The method is based on the internal analytical method WM21.

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