Workshop 1

Acetic Acid in Vinegar

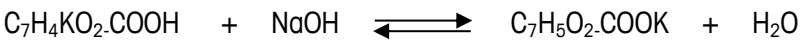
by Acid/Base Titration

Background

Vinegar is typically made from the fermentation of alcoholic liquids such as wine. Its main constituents are water and acetic acid, usually about 5 %. The acid in vinegar is produced by bacteria, which convert ethanol to acetic acid. An easy and accurate way to determine the acetic acid content is the direct titration with sodium hydroxide (NaOH).

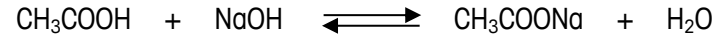
Reactions

1. Titer determination:



std. substance	titrant	reaction products
204.23 g/mol	40.00 g/mol	

2. Acetic acid content determination:



analyte	titrant	reaction products
60.05 g/mol	40.00 g/mol	

Safety

Sodium hydroxide is corrosive! Skin and especially eye contact must be avoided. Always wear a lab coat, gloves and protective goggles during this workshop.

Tasks

1. Titer determination:

Perform a titer determination (standardization) for the titrant NaOH, c = 0.1 mol/L, using pure potassium hydrogen phthalate (KHP, M = 204.23 g/mol, z = 1) as the titration standard. For this purpose accurately weight 0.08 – 0.12 g of potassium hydrogen phthalate into a titration beaker and add 50 mL of deionized water. If you use an indicator add a small amount of phenolphthalein solution (0.1 % in ethanol) to the solution. Start the titration by adding small amounts of titrant (manual titration) or by starting the automated titration, and stir during the whole titration. Titrate until the color changes from colorless to pink (manual titration) or until the EQP is recognized by the automatic titrator. Calculate the titer from the used volume. Rinse the electrode and dispensing tubes with deionized water between every measurement (only for automated titration).

Determine the titer at least three times and calculate the mean value, standard deviation (*s*) and relative standard deviation (*srel*).

2. Acetic acid content determination:

Determine the acetic acid content in a vinegar sample. Accurately weight about 1 g of vinegar in a titration beaker and add 50 mL of deionized water. To perform the titration, follow the same procedure as for the titer determination.

Determine the acetic acid content (in %) at least three times and calculate the mean value and standard deviation.

Waste disposal:

Neutralize (pH 7) the aqueous solutions before final disposal.

Acetic Acid in Vinegar

by Acid/Base Titration

Equipment

Manual titration:

- 1x Analytical balance
- 1x Manual burette (10 mL)
- 6x Titration beakers (e.g. 100 mL glass beakers)
- 1x Magnetic stirrer
- 3x Magnetic stirrer bars

Automated titration:

- 1x Analytical balance
- 1x Mettler-Toledo Easy pH or Easy Pro titrator with 10 mL burette and tubing
- 1x EG11-BNC pH Electrode
- 6x Titration beakers
- 3x Magnetic stirrer bars

Chemicals

The quantities below were roughly estimated for 5 titer determinations and 5 acetic acid determinations.

- 1 L deionized water
- 200 mL sodium hydroxide solution c = 0.1 mol/L (4 g solid sodium hydroxide in 1 L solution)
- 10 mL of phenolphthalein solution (0.1 % in ethanol, 0.01 g phenolphthalein in 10 g solution)
- 1 g potassium hydrogen phthalate of high purity (titration standard)
- 10 g vinegar as sample

Preparation

- Prepare the titrant and indicator solutions.
- Rinse the burette of the automatic titrator at least twice to dispel any air bubbles trapped in the burette and tubing.

Comments

- Prevent CO_2 uptake of the titrant. For this purpose place a drying tube filled with sodium hydroxide on a carrier on top of the titrant bottle.
- The method may be slightly adapted depending on the vinegar used and its acid content.

Solution

1. Titer determination:

Calculation:

$$t = \frac{m}{VEQ \cdot c \cdot C}$$

$$C = \frac{M}{10 \cdot p \cdot z}$$

t: Titer of the titrant (no unit)
m: Weight of standard substance used for the determination (in g, here: KHP)
VEQ: Titrant consumption at the equivalence point (in mL)
c: Nominal concentration of the titrant (in mol/L, here: NaOH, c = 0.1 mol/L)
C: Constant (in g/mmol, see equation above)
M: Molar mass of the standard substance (in g/mol, here: KHP, M = 204.23 g/mol)
10: Factor for mg to g and % conversion (in mg/(g·%))
p: purity of the standard substance (in %)
z: equivalent number (no unit, here: 1)

Expected result:

The titer should be around 1 for a fresh titrant solution.

2. Acetic acid content determination:

Calculation:

$$R = \frac{VEQ \cdot c \cdot t \cdot C}{m}$$

$$C = \frac{M}{10 \cdot z}$$

R: Result, content (in %, here: acetic acid content)
VEQ: Titrant consumption at the equivalence point (in mL)
c: Nominal concentration of the titrant (in mol/L, here: NaOH, c = 0.1 mol/L)
t: Titer of the titrant, as determined before (no unit)
C: Constant (in g·%/mmol, see equation above)
m: Sample weight (in g)
M: Molar mass of the analyte (in g/mol, here: Acetic acid, M = 60.05 g/mol)
10: Factor for mg to g and % conversion (in mg/(g·%))
z: equivalent number (no unit, here: 1)

Expected result:

The acetic acid content in vinegar is usually around 5 %.

Example

1. Titer determination:

Three titer determinations were performed using KHP with a purity of 99.8 %. In the following table the KHP amount (*m*), the titrant consumption (*VEQ*) and the calculated titer (*t*) for these three measurements are shown:

Nr.	*m*	*VEQ*	*t*
1	0.087 g	4.265 mL	0.9968
2	0.092 g	4.501 mL	0.9988
3	0.105 g	5.135 mL	0.9992

The titer of the first measurement was calculated as follows:

$$C = \frac{M}{10 \cdot p \cdot z} = \frac{204.23\ \frac{\text{g}}{\text{mol}}}{10\ \frac{\text{mg}}{\text{g}\cdot\%} \cdot 99.8\ \% \cdot 1} = 0.20464\ \frac{\text{g}}{\text{mmol}}$$

$$t = \frac{m}{VEQ \cdot c \cdot C} = \frac{0.087\ \text{g}}{4.265\ \text{mL} \cdot 0.1\ \frac{\text{mol}}{\text{L}} \cdot 0.20464\ \frac{\text{g}}{\text{mmol}}} = 0.9968$$

Based on these three determinations the mean, standard deviation (*s*) and the relative standard deviation (*srel*) were calculated:

Mean: 0.9983
s: 0.0013
srel: 0.13 %

2. Acetic acid content determination:

After the titer determination three measurements of a vinegar sample were performed. For these three measurements the following sample amounts were used and the following results were obtained:

Nr.	*m*	*VEQ*	*R*
1	0.982 g	7.165 mL	4.37 %
2	0.881 g	6.463 mL	4.40 %
3	0.912 g	6.686 mL	4.39 %

The calculation of the first measurement is shown here in detail:

$$C = \frac{M}{10 \cdot z} = \frac{60.04\ \frac{\text{g}}{\text{mol}}}{10\ \frac{\text{mg}}{\text{g}\cdot\%} \cdot 1} = 6.004\ \frac{\text{g}\cdot\%}{\text{mmol}}$$

$$R = \frac{VEQ \cdot c \cdot t \cdot C}{m} = \frac{7.165\ \text{mL} \cdot 0.1\ \frac{\text{mol}}{\text{L}} \cdot 0.9983 \cdot 6.004\ \frac{\text{g}\cdot\%}{\text{mmol}}}{0.982} = 4.37\ \%$$

As for the titer determination, the mean, *s* and *srel* were calculated:

Mean:	4.39 %
s:	0.02 %
srel:	0.35 %