Workshop 2

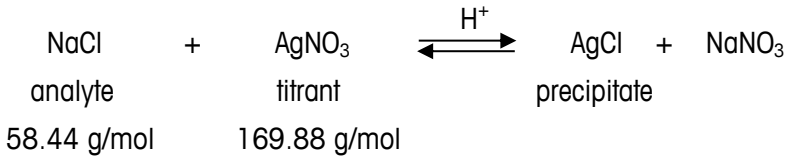
Sodium Chloride Content in Ketchup

by Precipitation Titration

Background

Sodium chloride is one of the most common substances found in nature. Knowing the salt content in food products is important not only for taste but also for health and compliance of recognized standards. One of the methods most commonly used to determine the NaCl content is the precipitation reaction with silver nitrate in an acidic medium. The result is usually calculated as percent of sodium chloride.

Reaction



Safety

Always take the necessary safety precautions when working in the laboratory: work carefully; wear a lab coat, protective gloves and safety goggles. Silver nitrate causes serious skin and eye irritation. It stains skin and other materials black. Potassium dichromate is very toxic and corrosive. Please read the material safety data sheet (MSDS) carefully before working with it. Potassium dichromate must only be used for manual titration. Titration solutions are dangerous to the environment as they contain contaminants such as heavy metals. Please dispose of solutions according to the instructions below.

Tasks

1. Titer determination:

Perform a titer determination (standardization) of the titrant $AgNO_3$ c = 0.1 mol/L by titrating a standard sodium chloride solution (c = 0.1 mol/L). Add exactly 5 mL of the standard solution into a titration beaker by using a pipette.

For a manual titration with visual equivalence point determination add 40 mL of deionized water and a few drops of saturated potassium dichromate indicator solution. Place the beaker under the burette and start the titrating by slowly adding titrant to the standard sodium chloride titration solution under permanent stirring. Titrate until the equivalence point is reached as is evident by a color change from yellow to orange-red. Note the volume consumed. Calculate the titer by using the correct formula.

If you use an automatic titrator with a potentiometric electrode (silver ring electrode) add 40 mL of H_2SO_4 solution, c = 0.02 mol/L, to the titration beaker. This dilutes and acidifies the standard solution. Immerse the titration tube and the electrode in the titration solution and start the titration. The titration will stop automatically when the endpoint is reached or when the maximum volume of titrant is added. The result, consumed titrant and titer, will be displayed.

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

2. Sodium chloride content determination:

To determine the sodium chloride content in ketchup first weight about 1.5 g of ketchup accurately into the titration beaker. Dissolve the sample in deionised water. Then follow the same sample preparation and titration procedure as for the titer determination above.

Determine the sodium chloride content (in %) at least three times and calculate the mean value, standard deviation (*s*) and relative standard deviation (*srel*).

Waste disposal

Filter the titration solutions and dispose of the precipitate as special waste in the bins labelled heavy metal waste. The liquid phase must be neutralized (pH 7) before it can be disposed of as waste water.

Note

Be careful not to add too much acid to titration solution. The optimal value is pH 4-5. If the solution is too acidic it will begin to dissolve the silver in the electrode thus releasing silver ions. These silver ions bind the chloride in solution and give a falsely low chloride concentration measurement.

Teacher Information

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Equipment

Manual titration:

- 1x Analytical balance
- 1x Accurate pipette (5 mL or 1 – 10 mL)
- 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 Accurate pipette (5 mL or 1 – 10 mL)
- 1x Mettler-Toledo Easy Cl or Easy Pro titrator with 10 mL burette and tubing
- 1x EM45-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 (mainly for manual titration and rinsing)
- 500 mL sulphuric acid solution, $c = 0.02$ mol/L (2.06 g H_2SO_4 95 % in 1 L solution, for automated titration)
- 200 mL silver nitrate solution, $c = 0.1$ mol/L (16.99 g solid silver nitrate in 1 L solution)
- 10 mL of saturated potassium dichromate solution (about 1.2 g potassium dichromate in 10 mL solution, only for manual titration)
- 30 mL sodium chloride standard solution, $c = 0.1$ mol/L (titration standard)
- 10 g ketchup as sample

Preparation

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

Comments

- This method may be slightly adapted depending on the sample used and its chloride content.

Solution

1. Titer determination:

Calculation:

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

$$C = \frac{1}{cst \cdot z}$$

t: Titer of the titrant (no unit)
m: Volume used (in mL, here: NaCl standard solution)
VEQ: Titrant consumption at the equivalence point (in mL)
c: Nominal concentration of the titrant (in mol/L, here: $AgNO_3$, $c = 0.1$ mol/L)
C: Constant (in L/mol, see equation above)
cst: Concentration of the standard solution (in mol/L, here: NaCl, $cst = 0.1$ mol/L)
z: equivalence number (here: 1)

Expected result:

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

2. Sodium chloride content determination:

Calculation:

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

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

R: Result, content (in %, here: sodium chloride content)
VEQ: Titrant consumption at the equivalence point (in mL)
c: Nominal concentration of the titrant (in mol/L, here: $AgNO_3$, $c = 0.1$ 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: NaCl, $M = 58.44$ g/mol)
10: Factor for mg to g and % conversion (in mg/(g·%))
z: equivalence number (no unit, here: 1)

Expected result:

The sodium chloride content in ketchup is usually around 3%. The expected concentration is declared on the label of the bottle.

Example

1. Titer determination:

Three titer determinations were performed by titrating 5 mL of sodium chloride standard solution (c = 0.1 mol/L). The sample volume and results are shown in the following table.

Nr.	*m*	*VEQ*	*t*
1	5 mL	5.050 mL	0.9901
2	5 mL	5.062 mL	0.9878
3	5 mL	5.041 mL	0.9919

The titer of the first measurement was calculated as follows:

$$C = \frac{1}{cst \cdot z} = \frac{1}{0.1 \frac{\text{mol}}{\text{L}} \cdot 1} = 10 \frac{\text{L}}{\text{mol}}$$

$$t = \frac{m}{VEQ \cdot c \cdot C} = \frac{5 \text{ mL}}{5.050 \text{ mL} \cdot 0.1 \frac{\text{mol}}{\text{L}} \cdot 10 \frac{\text{L}}{\text{mol}}} = 0.9901$$

The mean, standard deviation (s) and the relative standard deviation ($srel$) were calculated for the three measurements:

Mean: 0.9899
s: 0.0021
$srel$: 0.21 %

2. Sodium chloride content determination:

Three measurements of a ketchup sample were performed to determine the sodium chloride content. The following table shows the sample weight and results.

Nr.	*m*	*VEQ*	*R*
1	1.367 g	7.553 mL	3.196 %
2	1.268 g	7.058 mL	3.180 %
3	1.166 g	6.422 mL	3.186 %

The detailed calculation for the first measurement is:

$$C = \frac{M}{10 \cdot z} = \frac{58.44 \ \frac{g}{mol}}{10 \ \frac{mg}{g \cdot \%} \cdot 1} = 5.844 \ \frac{g \cdot \%}{mmol}$$

$$R = \frac{VEQ \cdot c \cdot f \cdot C}{m} = \frac{7.553 \text{ mL} \cdot 0.1 \ \frac{mol}{L} \cdot 0.9899 \cdot 5.844 \ \frac{g \cdot \%}{mmol}}{1.367 \text{ g}} = 3.196 \ \%$$

For these three measurements the mean, *s* and *srel* are as follows:

Mean: 3.187%
s: 0.008 %
srel: 0.26 %