

FCH-PP-7-90-2020

PRODUCTS/CATEGORIES: ELECTROLYSIS PRODUCTS

Test method: Active chlorine content

(for Sodium hypochlorite technical and for biocidal use)

1. Sampling

The sampling is performed according to STN 65 0512. Only 500 ml of fractional sample is taken from each tank.

2. Testing

2.1 Determination of active chlorine content

2.1.1 Principle of determination

Active chlorine will release iodine from potassium iodide solution. The released iodine is titrated with a standard solution of sodium thiosulphate.

2.1.2 Chemicals and materials

- acetic acid 99 % ($\rho = 1,0525 \text{ g.cm}^{-3}$), diluted 1:3, or HCl p. a. diluted 1:1
- potassium iodide, 10 % solution, new prepared
- sodium thiosulfate, the volumetric solution $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$
- starch solution
- volumetric flasks capacity 500 ml
- titration flasks capacity 250 ml
- pipettes capacity 10 ml, 20 ml, 25 ml
- automatic burette capacity 50 ml, graduated at 0,1 ml intervals

2.1.3 Procedure for determination

25 ml of the sample is pipetted into 500 ml volumetric flask and filled up to the line with distilled water. After mixing, 20 ml from this solution is pipetted into 250 ml titration flask, 20 ml of potassium iodide and 10 ml of acetic acid are added. The released iodine is titrated with sodium thiosulfate to a bright-yellow colour. 2 ml of starch solution is added and titrated till the solution becomes colour less.

2.1.4 Calculation

The content of active chlorine (X) in g/l is given by the formula:

$$X = f \times b \times c_1 \times M \times \frac{Z}{z \times V} = f \times b \times 0,1 \times 35,453 \times \frac{500}{25 \times 20}$$

$$X = 3,545 \times f \times b$$

- where
- f is factor of $\text{Na}_2\text{S}_2\text{O}_3$ volumetric solution
 - b consumption of $\text{Na}_2\text{S}_2\text{O}_3$ volumetric solution in ml
 - c_1 concentration of $\text{Na}_2\text{S}_2\text{O}_3$ volumetric solution ($c_1 = 0,1 \text{ mol.l}^{-1}$)
 - M chlorine molecular weight ($M = 35,453 \text{ g.mol}^{-1}$)
 - Z stock solution volume in ml
 - z sample volume in ml
 - V stock solution volume pipetted for determination in ml

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FCH-PP-7-90-2020

PRODUCTS/CATEGORIES: ELECTROLYSIS PRODUCTS

Test method: Sodium hydroxide content

(for Sodium hypochlorite technical and for biocidal use)

1. Sampling

The sampling is performed according to STN 65 0512. Only 500 ml of fractional sample is taken from each tank.

2. Testing

2.1 Determination of sodium hydroxide content

2.1.1 Principle of determination

The sample is decomposed by hydrogen peroxide and sodium hydroxide is determined by titration with volumetric solution of hydrochloric acid.

2.1.2 Chemicals and materials

- hydrochloric acid, volumetric solution $c(\text{HCl}) = 0,1 \text{ mol/l}$,
- hydrogen peroxide, 30 % solution ($\rho = 1,11 \text{ g.cm}^{-3}$), neutral,
- barium chloride p. a.,
- phenolphthalein solution,
- distilled water,
- denatured alcohol,
- titration flasks capacity 250 ml,
- pipettes capacity 10 ml, 20 ml, 25 ml,
- automatic burette 50 ml
- analytical balance capable of weighing to 0,0001 g

2.1.3 Procedure for determination

10 ml of the sample is pipetted into 250 ml of titration vessel and diluted with 40 ml of water. During mixing hydrogen peroxide solution is added as long as the sample is decomposed during effervescence. 5 – 10 g of barium chloride and 5 drops of phenolphthalein are added and during constant mixing the solution is dropwise titrated with HCl volumetric solution till the solution becomes colorless.

2.1.4 Calculation

The content of sodium hydroxide (X) in g/l is given by the formula:

$$X = \frac{f \times a \times c_1 \times M}{V} = \frac{f \times a \times 0,1 \times 40}{10} = 0,4 \times a \times f$$

where f is factor of HCl volumetric solution

a consumption of HCl volumetric solution in ml

c_1 concentration of HCl volumetric solution ($c_1 = 0,1 \text{ mol.l}^{-1}$),

M NaOH molecular weight ($M = 40,0 \text{ g.mol}^{-1}$)

V sample volume in ml

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FCH-PP-7-90-2020

PRODUCTS/CATEGORIES: ELECTROLYSIS PRODUCTS

Test method: Sodium carbonate content

(for Sodium hypochlorite technical and for biocidal use)

1. Sampling

The sampling is performed according to STN 65 0512. Only 500 ml of fractional sample is taken from each tank.

2. Testing

2.1 Determination of sodium carbonate content

2.1.1 Principle of determination

Sodium carbonate content is determined by titration with HCl volumetric solution.

2.1.2 Chemicals and materials

- hydrochloric acid, volumetric solution $c(\text{HCl}) = 0,1 \text{ mol/l}$
- hydrogen peroxide, 30 % solution ($\rho = 1,11 \text{ g.cm}^{-3}$), neutral
- methyl orange solution
- distilled water
- denatured alcohol
- titration flasks capacity 250 ml
- pipettes capacity 10 ml, 20 ml, 25 ml
- automatic burette capacity 50 ml

2.1.3 Procedure for determination

10 ml of the sample is pipetted into 250 ml of titration vessel and diluted with 40 ml of water. During mixing hydrogen peroxide solution is added as long as the sample is decomposed during effervescence. 5 ml of methyl orange is added and titrated dropwise with HCl volumetric solution till the first orange-coloured.

2.1.4 Calculation

The content of sodium carbonate (X) in g/l is given by the formula:

$$X = \frac{f \times (b - a) \times c_1 \times M}{V} = \frac{f \times (b - a) \times 0,1 \times 53}{10} = 0,53 \times f \times (b - a)$$

where f is factor of HCl volumetric solution

b HCl volumetric solution consumption to methyl orange in ml

a HCl volumetric solution consumption to phenolphthalein in ml (in NaOH determination)

c_1 concentration of entry HCl volumetric solution ($c_1 = 0,1 \text{ mol.l}^{-1}$)

M Na_2CO_3 molecular weight ($M(\text{Na}_2\text{CO}_3) = 53 \text{ g.mol}^{-1}$),

V sample volume ($V = 10 \text{ ml}$)

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FCH-PP-7-90-2020
PRODUCTS/CATEGORIES: ELECTROLYSIS PRODUCTS
Test method: Iron content
 (for Sodium hypochlorite technical and for biocidal use)

1. Sampling

The sampling is performed according to STN 65 0512. Only 500 ml of fractional sample is taken from each tank.

2. Testing

2.1 Determination of iron content

2.1.1 Principle of determination

Fe³⁺ is formed by oxidation Fe²⁺ with hydrogen peroxide. Fe³⁺ with potassium thiocyanate formed red coloration and the colour intensity is measured spectrophotometrically.

2.1.2 Chemicals and materials

- hydrochloric acid, diluted (1:1)
- hydrogen peroxide, 30 % solution
- potassium thiocyanate, 20 % solution
- iron reference solution with concentration of 1 g.l⁻¹
- distilled water
- volumetric flask capacity 100 ml
- beaker capacity 250 ml
- spectrophotometer with accessories
- analytical balance capable of weighing to 0,0001 mg
- pipette capacity 10 ml
- measuring cylinder capacity 50 ml

2.1.3 Procedure for determination

10 ml of the sample is pipetted into a beaker of 250 ml. 40 ml of distilled water is added. During mixing, hydrogen peroxide solution (3 ml to 5 ml) is added dropwise as long as the sample decomposes with effervescence. 10 ml of HCl is added, boiled for about 5 minutes and after cooling, the solution is quantitatively transferred into a volumetric flask of 100 ml. Next, 10 ml of potassium thiocyanate solution is added, filled up to the line with water and mixed well. After 10 min. the colour intensity is measured by spectrophotometer at a wavelength of 470 nm against the blank (thiocyanate, peroxide, distilled water). The iron content in mg is read from the measured absorbance from the calibration graph.

NOTE: Spectrophotometer is regularly (at least 1 per year) calibrated to the required concentration range. At the same time, a calibration curve is constructed, where the Fe content is on the x-axis in 100 ml volumetric flasks and absorbance on the y-axis.

2.1.4 Calculation

The content of iron (X) in % is given by the formula:

$$X = \frac{A}{V} = 0,1 \times a$$

$$X = \frac{A}{k}$$

- where
- A is measured absorbance
 - V sample volume in ml
 - a the Fe³⁺ amount which is read from calibration curve in mg
 - k slope of a straight line (concentration of Fe³⁺ in dependence on absorbance)

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