

Sodium Chloride - Analytical Standard



Determination of Nitrite Permanganate / Thiosulphate Titrimetric Method

EUsalt/AS 001-2005

Former numbering: ECSS/CN 90-1973 & ESPA/CN-E-112-199

1. SCOPE AND FIELD OF APPLICATION

The present EUsalt Analytical Standard describes a titrimetric method for the determination of nitrites in nitrated sodium chloride. The method is applicable to products of nitrite content (expressed as sodium nitrite) equal to or greater than 0.15 g per kilogram of salt.

2. PRINCIPLE

Dissolution of the sample in water.

Oxidation of nitrite to nitrate with potassium permanganate.

Addition of potassium iodide and titration of the liberated free iodine corresponding to the excess of potassium permanganate with sodium thiosulphate using starch as indicator.

3. REAGENTS

Unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

3.1. Sodium chloride, nitrite free

3.2. Sulphuric acid, $C(H_2SO_4) \approx 1.5 \text{ mol/l}$

Add 18 ml of sulphuric acid, $\rho \approx 1.84 \text{ g/ml}$, 96 % (m/m), to 200 ml of water and mix well.

3.3. Potassium iodide solution, $\beta_{(KI)} \approx 100 \text{ g/l}$

Store this solution in a dark bottle.

3.4. Potassium permanganate solution,

$C(1/5 KMnO_4) = 0.1 \text{ mol/l}$

3.5. Sodium thiosulphate, $C(Na_2S_2O_3) = 0.1 \text{ mol/l}$, standard volumetric solution

3.6. Starch solution, 2 g/l

Prepare this solution at the time of use from soluble starch.

4. APPARATUS

Usual laboratory equipment.

5. SAMPLING AND SAMPLES

A test sample of 500 g should be taken for analysis, ensuring it is representative of the whole batch.

6. PROCEDURE

6.1. Test portion

Weigh, to the nearest 0.1 g, about 50 g of the test sample.

6.2. Test solution

Transfer the test portion (6.1.) and 300 ml of water into a 500 ml volumetric flask, stir to dissolve, dilute to the mark and mix.

Filter this through a filter crucible or a filter paper (see 8.2).

6.3. Blank solution

Transfer 50 g of sodium chloride (3.1.) and 300 ml of water into a 500 ml volumetric flask, stir to dissolve, dilute to the mark and mix.

6.4. Determination

Proceed with the solutions prepared in (6.2.) and (6.3.) in the following way.

Transfer 100.0 ml of the solution into a 500 ml conical flask. Add 25.00 ml of potassium permanganate standard volumetric solution (3.4.) and 5.0 ml of diluted sulphuric acid (3.2.).

Allow to stand for exactly 3 minutes and add 5.0 ml of potassium iodide solution (3.3.).

Titrate with the sodium thiosulphate standard volumetric solution 0.1 mol/l (3.5.) using a burette. When the solution is nearly discoloured, add 1 ml of starch solution (3.6.) and continue the titration until the blue colour disappears for at least 3 seconds.

7. EXPRESSION OF RESULTS

7.1. Evaluation

The nitrite content of the sample, $\omega_{(\text{NaNO}_2)}$, is given by the formula:

$$\omega_{(\text{NaNO}_2)} = 172.5 \times C_{(\text{Na}_2\text{S}_2\text{O}_3)} \frac{(V_0 - V_1)}{m}$$

where

$\omega_{(\text{NaNO}_2)}$ is the nitrite content, expressed as grams of sodium nitrite per kilogram of salt,

m is the mass, in grams, of the test portion (6.1.),

V_1 is the volume, in millilitres, of sodium thiosulphate (3.5.) used for the titration of the test solution (6.2.),

V_0 is the volume, in millilitres, of sodium thiosulphate (3.5.) used for the titration of the blank solution (6.3.),

$C_{(\text{Na}_2\text{S}_2\text{O}_3)}$ is the molar concentration of the sodium thiosulphate standard volumetric solution (3.5.).

The result is expressed to two decimal places.

7.2. Repeatability and reproducibility

Analyses, carried out on one sample by 11 laboratories, have given the following statistical results, each laboratory having furnished results obtained by the same operator performing two analyses per sample:

$\omega_{(\text{NaNO}_2)}$	k	p	n	S_r	S_R
5.93	11	11	2	0.04	0.065

where

$\omega_{(\text{NaNO}_2)}$ is the content, in g NaNO_2/kg ,

k is the number of analysts,

p is the number of laboratories,

n is the number of results per series

S_r is the repeatability standard deviation, in g NaNO_2/kg ,

S_R is the reproducibility standard deviation, in g NaNO_2/kg .

8. REMARKS

8.1. The method is only valid in the absence of other reducing agents capable of reacting with potassium permanganate or when their concentration is negligible.

8.2. The filtration of the test solution (6.2.) is only necessary when the nitrated salt contains insoluble reducing agents which would influence the used quantity of potassium permanganate.

8.3. An automatic titrator provided with a platinum electrode and an Ag/AgCl reference electrode may be used. In this case, do not add starch solution (3.6.) during the determination (6.4.).