



Sodium Chloride - Analytical Standard



Determination of Potassium Sodium Tetrphenylborate Volumetric Method

EuSalt/AS 007-2005

Former numbering: ECSS/CN 183-1979 & ESPA/CN-E-103-1994

1. SCOPE AND FIELD OF APPLICATION

This EuSalt Analytical Standard describes a volumetric method using sodium tetraphenylborate for the determination of potassium in sodium chloride. The method is applicable to products of potassium content (K) equal to or greater than 100 mg per kilogram of salt.

2. REFERENCE

International Standard - ISO 2479

Sodium chloride for industrial use - Determination of matters insoluble in water or in acid, and preparation of principal solutions for other determinations.

3. PRINCIPLE

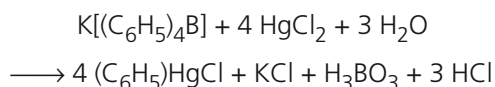
Dissolution of the sample in water or in acid and, if necessary, filtration of insoluble matters.

Precipitation of the potassium with sodium tetraphenylborate.

Filtration and re-dissolution of the potassium tetraphenylborate precipitate in acetone.

Reaction of the tetraphenylborate with mercury (II) chloride. Titration of the liberated hydrochloric acid.

Note: the potassium tetraphenylborate reacts with mercury (II) chloride according to the following equation:



4. REAGENTS

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

4.1. Acetone, neutral

Control the neutrality as follows:

Pour out 15.0 ml of acetone into 50 ml of water containing 3 drops of methyl red (4.9.). The addition of either 1 or 2 drops of hydrochloric acid (4.7.) or of sodium hydroxide solution (4.8.) should be sufficient to cause a colour change.

4.2. Mercury (II) chloride, saturated solution

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

Store this solution in a dark bottle.

4.4. Sodium carbonate solution, $\beta_{(\text{Na}_2\text{CO}_3)} \approx 160 \text{ g/l}$

4.5. Sodium tetraphenylborate solution,

$\beta_{(\text{C}_{24}\text{H}_{20}\text{BNa})} \approx 30 \text{ g/l}$

Dissolve 15.0 g of sodium tetraphenylborate in 500 ml of water. Check that the pH of this solution is between 6 and 7. If necessary, adjust by the addition of hydrochloric acid (4.7.) or sodium hydroxide solution (4.8.).

Add one drop of saturated aluminium chloride solution and 5 mg of lithium chloride to the generally cloudy solution. Filter the flocculated precipitate and store the clear solution in a polyethylene bottle.

4.6. Washing solution,

saturated potassium tetraphenylborate solution

Dissolve about 100 g of potassium chloride in 100 ml of water. Add 20 ml of sodium tetraphenylborate solution (4.5.). Filter the precipitate and wash it five times with water. Disperse the precipitate in 1 litre of water and leave for at least 1 hour, stirring occasionally. Filter and store the solution in a polyethylene bottle.

4.7. Hydrochloric acid,

$c_{(\text{HCl})} = 0.1 \text{ mol/l}$, standard volumetric solution

4.8. Sodium hydroxide,

$c_{(\text{NaOH})} = 0.1 \text{ mol/l}$, standard volumetric solution

4.9. Methyl red, 1 g/l solution

Dissolve 0.1 g of methyl red in 75 ml of 95% (v/v) ethanol and make up to 100 ml with water.

5. APPARATUS

Usual laboratory equipment and:

5.1. Filter crucible,

glass or porcelain, having pores of 5 to 15 μm diameter.

6. SAMPLING AND SAMPLES (see 9.1.)

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

7. PROCEDURE (see 9.1.)

7.1. Test portion

Weigh, to the nearest 0.1 g, about 100 g of the test sample.

7.2. Test solution

Use the test portion (7.1.) to prepare 1000 ml of a solution A (water solution) or of a solution B (acid solution), as described in ISO 2479.

7.3. Determination

Transfer 100 ml of the test solution (7.2.) and 5 ml of sodium carbonate solution (4.4.) to a 250 ml beaker. Heat to boiling for 1 minute. Allow to cool to about 70 °C, then add, drop by drop and with stirring, 10 ml of sodium tetraphenylborate solution (4.5.).

Cool quickly, putting the beaker into a water bath at ambient temperature. Filter under vacuum on the filter crucible (5.1.) fitted to a 250 ml Buchner flask. Perform three washings, each time using 5 ml of washing solution (4.6.), followed by three washings, each time with 2 ml of water.

Reject the filtrate and rinse the Buchner flask with water. Add 5 ml of mercury (II) chloride solution (4.2.) to the Buchner flask. Rinse carefully the inner part of the filter crucible and below the filter disc with water. Place the filter crucible again on the Buchner flask.

Wash the sides of the beaker, used for the precipitation, with 5 ml of acetone (4.1.) and pour this liquid into the filter crucible. Stir the precipitate with a glass rod to make the dissolution easier and suck dry. Rinse the beaker and the filter crucible with two more 5 ml portions of acetone, then three times with 10 ml portions of water.

Remove the filter crucible, add 5 drops of methyl red (4.9.) to the Buchner flask and titrate with sodium hydroxide solution (4.8.) until the colour changes from red to yellow and then add an excess of about 10 ml. Heat this solution to boiling point, add 10 ml of potassium iodide solution (4.3.), a further 5 drops of methyl red then back-titrate with hydrochloric acid (4.7.) to a sharp red end-point.

Add another 1 ml of potassium iodide solution and, if the solution returns to a yellow colour, continue the titration until an additional 1 ml of potassium iodide solution does not alter the red colour of the indicator.

8. EXPRESSION OF RESULTS

8.1. Evaluation

The potassium content of the sample, $\omega_{(K)}$, of salt is given by the formula:

$$\omega_{(K)} = (V_1 - V_2) \times \frac{39.0983}{3} \times \frac{1000}{100} \times \frac{1000}{m} = 1.303 \times 10^5 \times \frac{V_1 - V_2}{m}$$

where

- $\omega_{(K)}$ is the potassium content, in milligrams per kilogram of salt,
- m is the mass, in grams, of the test portion (7.1.),
- V_1 is the volume, in millilitres, of sodium hydroxide solution (4.8.) used during the determination (7.3.),
- V_2 is the volume, in millilitres, of hydrochloric acid (4.7.) used during the determination (7.3.).

8.2. Repeatability and reproducibility

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

| | Rock salt | Sea salt |
|-----------------------------|-----------|----------|
| Number of laboratories | 15 | 14 |
| Results, mg K/kg salt | | |
| Mean | 581 | 294 |
| Standard deviation for: | | |
| - repeatability (s_r) | 12 | 7 |
| - reproducibility (s_R) | 49 | 28 |

Reference: European Committee for the Study of Salt, ECSS/CN 172-1978, Statistical evaluation of the Interlaboratory Study of Br, K, Ca, Mg, F.

9. REMARKS

- 9.1. Ensure that no trace of potassium is introduced during the sampling operations and during the analysis.
- 9.2. As an addition to this standard, a procedure for demercurization of residual solutions of mercurimetric titrations is attached.

Appendix

Demercurization of residual solutions of mercurimetric titrations

1. SCOPE AND FIELD OF APPLICATION

This appendix describes a procedure for the demercurization of solutions from mercurimetric titrations to avoid pollution of waste waters.

2. PRINCIPLE

Collection of residual solutions in a container of sufficient capacity (50 l for example).

Precipitation of mercury in an alkaline medium with an excess of sodium sulphide.

Oxidation of this excess by hydrogen peroxide to avoid redissolution of mercury as polysulphides.

Decantation and filtration of the demercurized liquid prior to disposal to drain.

Transfer of the insolubles to a storage vessel for the subsequent recovery of mercury by a specialized company.

3. REFERENCE

Safe and Simple Method for the Disposal of Mercury-Containing Wastes from Kjeldahl Analysis, Margaret J. Mina - Analytical Chemistry, 1974, (46), 2250,1

4. APPARATUS

4.1. Polyethylene container of sufficient capacity (about 50 l).

4.2. Small laboratory pump or water pump connected to a fritted glass filter of porosity G4 (apertures of 10 to 20 µm) or a funnel fitted with cotton wool.

5. REAGENTS

5.1. Sodium sulphide, Na₂S·9H₂O, technical grade

5.2. Sodium hydroxide solution, β_(NaOH) ≈ 400 g/l

5.3. Hydrogen peroxide,

H₂O₂, approximately 30% (m/m) solution, technical grade.

6. PROCEDURE

Collect in the receiver (4.1.) the residual solutions with a mercury level high enough to pollute waste waters.

Stir with compressed air when the collected volume is about 40 litres and add successively 400 ml of sodium hydroxide solution (5.2.) and 100 g of sodium sulphide (5.1.). Wait 10 minutes and add slowly 400 ml of hydrogen peroxide (5.3.).

Allow to stand for 24 hours, then suck up the clear liquid using the pump (4.2.) for disposal to drain.

Rinse the receiver several times with a minimum of water to carry the insoluble matter to another receiver where it can be collected together with other insolubles obtained from subsequent operations.