EuSalt
European Salt Producers' Association

Sodium Chloride - Analytical Standard

Determination of Chloride
Potentiometric method

EuSalt/AS 016-2005

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1. SCOPE AND FIELD OF APPLICATION

The present EuSalt Analytical Standard describes a potentiometric method for the determination of chloride in sodium chloride. It is an application of the ISO 6227 Standard (1) to sodium chloride.

2. REFERENCES

(1) INTERNATIONAL STANDARD ISO 6227.
Chemical products for industrial use - General method for the determination of chloride ions - Potentiometric method.

(2) Akzo Nobel – RGA F98021 (June 1998): Final Research Report: The determination of chloride in NaCl.

3. PRINCIPLE

Chloride is determined by potentiometric titration with a standard silver nitrate solution using an automatic titrator.

Note: Polyvinyl alcohol is added to the solution to be analysed to avoid occlusions of chloride in the silver chloride precipitate.

4. REAGENTS

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

4.1. Nitric acid, $c_{(HNO_3)} \approx 1.4$ mol/l

Dilute 100 ml of concentrated nitric acid, $\rho \approx 1.40$ g/ml, 65 % (m/m), to 1000 ml with water.

4.2. Sodium chloride solution, $\beta_{(NaCl)} = 10.00$ g/l

Dissolve 10.00 g of sodium chloride (> 99.9 %), weighed to the nearest 1 mg, previously dried at 250 °C for 1 hour, in water. Make up to 1000 ml in a volumetric flask and mix.

This solution is usable for one month.

4.3. Polyvinyl alcohol (PVA) solution, $\beta_{(PVA)} \approx 2$ g/l

Dissolve 2 g of water soluble PVA in 1 l of water and heat to 60 – 70 °C for better solubility.

4.4. Silver nitrate solution, $c_{(AgNO_3)} = 0.100$ mol/l,

standard volumetric solution

This solution is standardised as described hereafter:

Transfer 10.00 ml of the sodium chloride solution (4.2.) and 2 ml of nitric acid (4.1.) and 5 ml of PVA solution (4.3.) into the titration vessel. Dilute to about 50 ml with water.

Titrate the chloride with the silver nitrate solution (4.4.) as described in section (7.3.).

The concentration of the silver nitrate solution, $c_{(AgNO_3)}$, expressed in mol/l is given by the formula:

$$\omega_{(AgNO_3)} = \frac{m}{58.443} \times \frac{10}{V}$$

where

- m is the exact mass of sodium chloride, in g, used for the preparation of the solution (4.2.),
- V is the volume of silver nitrate solution, in ml, used for the titration.

Carry out three determinations and calculate the mean value of $c_{(AgNO_3)}$.

5. APPARATUS

Usual laboratory equipment and:

5.1. Automatic titrator fitted with

- silver electrode and double junction reference electrode (Ag/AgCl/KCl/KNO_3). The KNO_3 solution in the outlet compartment has to be renewed each month.
- 20 ml burette.
- 150 ml titration vessel.

This instrument should be able to operate under the conditions shown in the next table:

Parameters	Values
Titration parameters	
Mode	Potentiometric detection with dynamic equivalence point titration
Increment near the equivalence point (EP)	25 µl
Signal drift after addition of titrant	15 mV/min
Maximum waiting time after addition of titrant	40 sec.
Stirring	Yes

5.2. Magnetic stirrer and stirring bars

6. SAMPLING AND SAMPLES

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

7. PROCEDURE

7.1. Test portion

Weigh, to the nearest 1 mg, about 10 g of the test sample.

7.2. Test solution

Transfer the test portion (7.1.) into a 1000 ml volumetric flask and dissolve in water. Dilute to the mark with water and mix.

7.3. Determination

7.3.1. Apparatus setting

Set all instrumental parameters of the titrator in accordance with the operating manual of the instrument's manufacturer including the parameters cited in (5.1.).

7.3.2. Measurement

With the pipette used for the calibration of the silver nitrate solution (4.4.), transfer 10.00 ml of the test solution (7.2.) into the titration vessel. Add 2 ml of nitric acid (4.1.), 5 ml of PVA solution (4.3.) and dilute to about 50 ml with water.

Titrate with the silver nitrate solution (4.4.).

8. EXPRESSION OF RESULTS

8.1. Method of calculation

The chloride content of the sample, $\omega_{(Cl)}$, is given by the formula and rounded to one decimal place:

$$\omega_{(Cl)} = V \times c_{(AgNO_3)} \times \frac{1000}{10xm} \times 35.453$$

where

- $\omega_{(Cl)}$ is the chloride content, in grams per kilogram of sample,
- m is the mass, in grams, of the test portion (7.1.),
- $c_{(AgNO_3)}$ is the concentration of the silver nitrate solution (4.4.) in mol/l,
- V is the volume of the silver nitrate solution used for the titration of the test solution (7.2.).

The sodium chloride content of the sample $\omega_{(NaCl)}$ is given by the formula and rounded to one decimal place:

$$\omega_{(NaCl)} = \omega_{(Cl)} \times \frac{58.443}{35.453}$$

where

- $\omega_{(NaCl)}$ is the sodium chloride content, in grams per kilogram of sample.

8.2. Repeatability and reproducibility

Analyses, carried out on four samples by 14 laboratories, have given the following statistical results, each laboratory having furnished results obtained by the same operator performing three analyses per sample :

	Vacuum salt 1	Vacuum salt 2	Sea salt	Rock salt
Number of laboratories retained after eliminating outliers	14	14	13	14
Results, g NaCl/kg salt				
Mean	999.9	998.5	999.2	992.1
Standard deviation for :				
- repeatability (s_r)	1.2	1.3	1.1	2.1
- reproducibility (s_R)	1.5	2.1	1.6	4.9
Repeatability limit (r)	3.4	3.8	3.1	6.0
Reproducibility limit (R)	4.3	6.0	4.6	14.0