European Salt Producers' Association

Sodium Chloride - Analytical Standard

Determination of Potassium
Flame Atomic Absorption Spectrometric Method

EuSalt/AS 008-2005

Former numbering: ECSS/CN 184-1979 & ESPA/CN-E-104-1994

1. SCOPE AND FIELD OF APPLICATION

The present EuSalt Analytical Standard describes a flame atomic absorption spectrometric method for the determination of potassium in sodium chloride. The method is applicable to products of potassium content (K) equal to or greater than 2 mg per kilogram of salt.

2. REFERENCE

International Standard - ISO 2479

Sodium chloride for industrial use - Determination of matter 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.

Addition of cesium chloride as ionisation buffer, atomisation of the test solution in an acetylene-air flame and measurement of the absorbance at a wavelength of 766.5 nm or 769.9 nm (see 9.5.).

4. REAGENTS

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

4.1. Cesium chloride solution, $\beta_{(CsCl)} \approx 6.3$ g/l

4.2. Potassium, stock solution I, $\beta_{(K)} = 1000$ mg/l,

commercial standard solution or to be prepared as follows.

Weigh, to the nearest 0.1 mg, 1907 mg of potassium chloride previously dried for at least one hour at 400 °C and then cooled in a desiccator. Dissolve with water, make up to 1000 ml in a one-mark volumetric flask and mix.

4.3. Potassium, stock solution II, $\beta_{(K)} = 10$ mg/l

Transfer 10.0 ml of potassium stock solution I (4.2.) into a 1000 ml one-mark volumetric flask, make up to the mark and mix.

5. APPARATUS

Usual laboratory equipment (see 9.2.) and:

5.1. Atomic absorption spectrometer fitted with an acetylene-air burner

5.2. Potassium hollow cathode lamp

6. SAMPLING AND SAMPLES (see 9.1.)

A test sample of about 500 g should be taken for analysis, ensuring 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

Prepare 1000 ml of a solution A (water solution) or of a solution B (acid solution) with the test portion (7.1.) as described in ISO 2479.

Transfer 1.00 ml of solution A or B (see 9.4. and 9.5.) and 20 ml of cesium solution (4.1.) into a 100 ml volumetric flask. Make up to volume and mix.

7.3. Blank solution (see 9.3.)

Dilute to 1000 ml the same quantities of all the reagents used for the preparation of solution B (see 7.2.).

Transfer 1.00 ml of this solution and 20 ml of cesium solution (4.1.) into a 100 ml volumetric flask. Make up to volume and mix.

7.4. Calibration solutions

Transfer 20 ml of cesium solution (4.1.) and the volumes of potassium stock solution II (4.3.) as shown in the following table into a series of eight 100 ml volumetric flasks. Make up to volume and mix.

Calibration solution No.	Potassium, stock solution II, ml	Potassium concentration mg/l
1(*)	0	0
2	0.5	0.05
3	2.0	0.20
4	5.0	0.50
5	10.0	1.00
6	15.0	1.50
7	20.0	2.00
8(**)	50.0	5.00
(*) zero calibration solution			
(**) prepare this solution only if measurements are to be performed at 769.9 nm (see 9.5.)		

7.5. Determination

7.5.1. Apparatus setting

Equip the spectrometer (5.1.) with the potassium hollow cathode lamp (5.2.).

Set the lamp current, the slit and the pressure of acetylene and air according to the instruction manual of the instrument. Adjust the wavelength at the maximum of emission of the lamp at about 766.5 nm or 769.9 nm (see 9.5.).

7.5.2. Spectrometric measurements

Aspirate water after each measurement.

Aspirate the solutions into the acetylene-air flame and determine the absorbance of each one in the following order:

- the calibration solutions 1 to 7 (7.4.),
- the blank solution (7.3.),
- the test solution (7.2.),
- the calibration solutions.

7.6. Calibration curve

Subtract the absorbance of the zero calibration solution from that of each of the other calibration solutions and plot a graph showing the concentrations of potassium (K), in milligrams per litre, on the abscissa and the corresponding corrected absorbances on the ordinate.

8. EXPRESSION OF RESULTS

8.1. Evaluation

The potassium content of the sample, $\omega(K)$, is given by the formula:

$$\omega_{(K)} = \frac{10^5}{m} \times (\beta_1 - \beta_0)$$

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.),
- β_1 is the concentration, in milligrams of potassium per litre, of the test solution (7.2.),
- β_0 is the concentration, in milligrams of potassium per litre, of the blank solution (7.3.).

8.2. Repeatability and reproducibility

Analyses, carried out on three 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	Vacuum salt	Sea salt
Number of laboratories after elimination of outliers	15	13	14
Results, mg K/kg salt Mean Standard deviation for:	562	11	269
- repeatability (s_r)	8	1.1	7
- reproducibility (s_R)	53	5.8	24

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. All new glassware used for this determination should be washed as follows and carefully rinsed with water after each operation:

- with a brush and detergent if the walls are greasy,
- with diluted nitric acid, $c_{(HNO_3)} \approx 7$ mol/l.

9.3. When salt is dissolved in water and not in acid according to ISO 2479 (usual case), the zero calibration solution (see 7.4.) may replace the blank solution (7.3.).

9.4. If the estimated level of potassium is less than 50 mg per kilogram of salt, prepare the test solution (7.2.) with 5 ml of solution A or B. The formula for calculation (8.1.) must be modified accordingly.

9.5. Proceed as follows if the estimated level of potassium is higher than 2000 mg per kilogram of salt

- either dilute the sample of solution A or B (see 7.2.) and take an aliquot of this solution,
- or measure the solutions at the wavelength of 769.9 nm. In this case, use the solutions 1, 4, 5, 6, 7 and 8 (7.4.) as calibration solutions.

The formula for calculation (8.1.) must be modified accordingly.

In all cases, the report should contain an account of all details relevant to the analysis.

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