EUROPEAN SALT PRODUCERS'ASSOCIATION

Sodium Chloride - Analytical Standard

Determination of Iron
1,10-phenanthroline Photometric Method

EUsalt/AS 003-2005

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1. SCOPE AND FIELD OF APPLICATION

The present EuSalt Analytical Standard describes a 1,10 phenanthroline photometric method for the determination of iron in sodium chloride. The method is applicable to products of iron content (Fe) equal to or greater than 1 mg per kilogram of salt.

2. PRINCIPLE

Dissolution of the sample in hydrochloric acid. Addition of bromine to convert complexed iron into free trivalent iron. Reduction of trivalent iron by hydroxylammonium chloride. Formation of the 1,10-phenanthroline - divalent iron complex in a buffered medium. Photometric measurement of the coloured complex at a wavelength around 510 nm.

3. REAGENTS

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

3.1. Hydrochloric acid, ρ ≈ 1.19 g/ml, 37% (m/m)

3.2. Hydroxylammonium chloride solution, $\beta(NH_2OH \cdot HCl) \approx 10$ g/l

3.3. 1,10-phenanthroline hydrochloride solution, $\beta(C_{12}H_8N_2HCl. H_2O) \approx 2.5$ g/l Note: This product may be replaced by 1,10-phenanthroline monohydrate ($C_{12}H_8N_2 \cdot H_2O$), β ≈ 2.1 g/l.

3.4. Bromine water, saturated at ambient temperature.

3.5. Buffer solution

Dissolve 305 g of sodium acetate trihydrate ($CH_3COONa. 3H_2O$) in about 500 ml of water. To this solution add 235 ml of glacial acetic acid, ρ ≈ 1.05 g/ml, 99 - 100% (m/m). Make up to 1 l and mix.

3.6. Iron, stock solution I, $\beta(_{Fe}) = 1000$ mg/l, commercial solution or to be prepared as follows : Dissolve 1.000 g of iron (metal 99.9%) in 20 ml of nitric acid, ρ ≈ 1.40 g/ml, 65% (m/m). Make up to 1000 ml in a one-mark volumetric flask and mix.

3.7. Iron stock solution II, $\beta(_{Fe}) = 10$ mg/l Transfer 10.0 ml of iron stock solution I (3.6.) into a 1000 ml one-mark volumetric flask, make up to the mark and mix. This solution has to be prepared immediately before use.

4. APPARATUS

Usual laboratory equipment and:

4.1. Spectrophotometer or

4.2. Photocolorimeter fitted with a filter ensuring maximum transmission between 500 and 520 nm.

Note: Specify the type of apparatus used (spectrophotometer or photocolorimeter), the path length and the wavelength (or type of filter).

5. SAMPLING AND SAMPLES (see 8.2.)

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

6. PROCEDURE (see 8.2.)

6.1. Test portion

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

6.2. Test solution

Transfer the test portion (6.1.), 350 ml of water, 50 ml of hydrochloric acid (3.1.) and 10 ml of bromine water (3.4.) into a 600 ml beaker. Heat to boiling until dissolution is complete and for at least 15 minutes. After cooling, transfer quantitatively into a 500 ml volumetric flask, make up to volume and mix.

Note: If the test solution is cloudy, it must be filtered prior to collection in the 500 ml volumetric flask.

6.3. Blank solution

Add 350 ml of water, 50 ml of hydrochloric acid (3.1.) and 10 ml of bromine water (3.4.) into a 600 ml beaker. Heat to boiling for at least 15 minutes. After cooling, transfer quantitatively into a 500 ml volumetric flask, make up to volume and mix.

6.4. Calibration

6.4.1. Calibration solutions

These preparations are used for photometric measurement in cuvettes of 4 or 5 cm optical path length. Transfer 0.5 ml of hydrochloric acid (3.1.) and the volumes of iron stock solution II (3.7.) indicated in the next table into a series of five 100 ml one-mark volumetric flasks.

Calibration solution No.	Iron, stock solution II, ml	Corresponding mass of iron µg
1(*)	0	0
2	2.5	25
3	5.0	50
4	10.0	100
5	15.0	150
(*) zero calibration solution		

6.4.2. Colour development

Add to each flask about 50 ml of water and:

- 5 ml of hydroxylammonium chloride solution (3.2.),
- 5 ml of 1,10-phenanthroline hydrochloride solution (3.3.),
- 25 ml of buffer solution (3.5.).

Make up to the mark, mix and allow to stand for at least 10 minutes.

6.4.3. Photometric measurements

Adjust the apparatus to zero absorbance against water.

Carry out the photometric measurements using a spectrophotometer (4.1.) set up at the maximum of absorption (wavelength around 510 nm) or a photocolorimeter (4.2.) fitted with the appropriate filter.

6.4.4. 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 quantities of iron (Fe), in micrograms, contained in 100 ml of calibration solution on the abscissa and the corresponding corrected absorbances on the ordinate.

6.5. Determination

6.5.1. Colour development

Transfer 25.0 ml (see 8.1.) of test solution (6.2.) or of blank solution (6.3.) into a 100 ml volumetric flask and continue as described in paragraph (6.4.2.).

6.5.2. Photometric measurements

Carry out the photometric measurements of the two solutions obtained in (6.5.1.) according to the instructions given in paragraph (6.4.3.).

7. EXPRESSION OF RESULTS

7.1. Evaluation

The iron content of the sample, ω(Fe), is given by the following formula:

$$m - m\ V$$

$$m \quad V\omega(Fe) = \underline{\hspace{2cm}}1\ \ 0 \times \underline{1}_2$$

where

- $\omega(Fe)$ is the iron content, in milligrams per kilogram of salt,
- m is the mass, in grams, of the test portion (6.1.),
- m_1 is the mass of iron, in micrograms, analysed in the final solution prepared in (6.5.1.) for the test solution (6.2.),
- m_0 is the mass of iron, in micrograms, analysed in the final solution prepared in (6.5.1.) for the blank solution (6.3.),
- V_1 is the volume, in millilitres, of test solution prepared in (6.2.),
- V_2 is the volume, in millilitres, of test solution used in (6.5.1.).

7.2. 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:

	Vacuum salt	Sea salt
Number of laboratories	14	13
Results, mg FE/kg salt	2.2	10.3
Mean		
Standard deviation for:		
- Repeatability (S_r)	0.40	0.45
- Reproducibility	1.92	2.43

8. REMARKS

8.1. For iron contents greater than 30 mg per kg of salt, the volume of test solution (6.2.) transferred into the 100 ml volumetric flask (see 6.5.1.) has to be reduced to such an extent that the amount of iron present in the 100 ml of final solution lies within the range of table (6.4.1.). The volume of blank solution (6.3.) to use has to be reduced in the same way. The formula for calculation (7.1.) must be modified accordingly.

8.2. Ensure that no trace of iron is introduced during the sampling operations and during the analysis.