

Same sample – different methods

On the following pages we have included examples of salt content analysis from same samples (one type of Italian cold cut and one salami) but with different analytical techniques. The sodium content is determined with atomic absorption spectroscopy and the chloride content is determined via titration.

In case of nutrition value determination, according to EU Regulation No. 1169/2011, the salt content has to be written on the product label (previously only the sodium was written on it). In this regulation, it is prescribed that the salt content can be calculated only from sodium (salt content=sodium x 2.5).

There are other regulations where the limits are given in sodium-chloride. It is not given what kind of measurement (chloride based or sodium based) has to be applied.

The advantage of a chloride based method is speed (much faster) and there is no need for big capital instrumental investments. Usually all the internal labs in the meat industry use the chloride based method. However, a titration method is less exact, with higher uncertainty.

In case of sodium determination, you need an instrument, but this provide you with a more precise method, and you have to use this procedure in case of nutrition value determination.

The experience from industry is, that especially in the meat industry, several food additives are used, which have sodium content beside the salt (e.g. sodium nitrite). In that case the salt content based on sodium are usually higher than the salt content based on chloride. The latter statement is verified in this example; where a difference of 0.22 and 0.14% in salt content was determined for the Italian cold cut and the salami sample, respectively.

Chloride content in meat and meat products

ISO 1841-1:1996

The method utilise the extraction of a test portion with hot water and precipitation of proteins. After filtration and acidification, excess of silver nitrate solution is added to the extract, and titration is carried out of the excess with potassium thiocyanate solution.

Reagents (*only use recognized analytical grades, unless otherwise specified*)

Water, distilled and halogen free (for chromatography; LiChrosolv® (1.15333)
Water should comply with at least grade 3 in accordance with ISO 3696)

Nitrobenzene, $\geq 99.0\%$ (8.06770)

Nitric acid; Suprapur® c(HNO₃) = 4 mol/L (1.00441)

Carrez Clarification Kit reagent kit for sample prep in food analysis, 5-fold concentrate(1.10537)
(Ready-to-use Carrez clarification kit can accelerate the analyses)

Silver nitrate solution; Reag. Ph Eur, Reag. USP; c(AgNO₃) = 0.1 mol/L (0.1 N) Titripur® (1.09081)

Ammonium iron(III) sulfate , EMSURE® ACS, ISO, Reag. Ph Eur (1.03776)

Potassium thiocyanate, std volumetric solution E EMPLURA®, c(KSCN)=0.1 mol/L; (1.05124)

Dissolve in water about 9.7 g of potassium thiocyanate.

Transfer quantitatively to a 1000 mL one-mark volumetric flask and dilute to the mark with water. Standardize the solution to the nearest 0.0001 mol/L against the silver nitrate solution using the ammonium iron(III) sulfate solution as indicator.

Apparatus:

Analytical balance

Homogenizing equipment

One-mark volumetric flask, of capacity of 200mL

Conical flask of capacity 250 mL

Burette of capacity 50 mL

Boiling water bath

Chloride content in meat and meat products

ISO 1841-1:1996

Procedure:

Weigh about 10 g of the sample (to the nearest 0.001 g) and transfer it quantitatively to a conical flask (referred to as test portion)

Deproteination (elimination of protein from the sample)

1. Add 100 ml of hot water to the test portion.
2. Heat the flask and its contents for 15 minutes in the boiling water bath.
3. Every 3-5 minutes shake the contents of the flask.
4. Allow the flask and its contents to cool to room temperature and add 2 ml of Carrez I and 2 ml of Carrez II solution. Mix thoroughly after each addition.

Allow the flask to stand for 30 minutes at room temperature.

Transfer the contents quantitatively to a 200 ml volumetric flask and dilute to the mark with water. Mix contents and filter through a fluted filter paper.

Determination:

Transfer 20 ml of the filtrate to a conical flask and add 5 ml of the diluted nitric acid and 1 ml of ammonium iron(III) sulfate solution as indicator.

Transfer 20 ml of the silver nitrate solution to the conical flask, then add 3 ml of the nitrobenzene and mix thoroughly. Shake vigorously to coagulate the precipitate.

Titrate the content of the conical flask with potassium thiocyanate until the appearance of a persistent pink coloration. Record the volume of the potassium thiocyanate solution required, to the nearest 0.05 ml.

Blank test: Carry out a blank test using the same volume of silver nitrate solution.

Calculation:

Chloride content = $58,44 \times (V_2 - V_1) / m \times C$

V₁: is the volume, in milliliters of the potassium thiocyanate solution used in the determination

V₂: is the volume, in milliliters of the potassium thiocyanate solution used in the blank test

m: is the mass, in grams of the test portion

C: is the concentration of the potassium thiocyanate solution in moles per liter (L).

Chloride content in meat and meat products

ISO 1841-1:1996

Samples:

sample ID 2015/P/20298 (Italian cold cut)

Sample ID 2015/36814 (salami)

Results:

Chloride content = $58,44 \times (V2 - V1) / m \times C$

Salt content = Chloride content $\times 1.65$

	sample 2015/P/20298 (cold cut)	sample 2015/36814 (salami)
V1	18.4	15.7
V2	19.9	19.9
m	10.0514	10.0597
C	0.1	0.1
Chloride content (%)	0.87	2.43
Salt content (%)	1.43	4.01

Determination of Sodium content in meat

EN 15505:2008

The sample is digested in closed vessels in a microwave oven in nitric acid. The resulting solution is diluted with water, and the sodium content is determined by flame-AAS using matrix modifier.

Reagents (*only use recognized analytical grades, unless otherwise specified.*)

Thus use only reagents/water, with an element level low enough not to affect results.

Water, LiChrosolv® (p/n 1.15333)

Water should comply with at least grade 3 in accordance with ISO 3696)

Nitric acid 65% Suprapur® (1.00441)

Sodium (1000 mg/l Na in HNO₃ 0.5 mol/l) traceable to SRM from NIST; CertiPUR® (1.70238)

Cesium chloride 99.995 Suprapur® (1.02039)

Apparatus:

(All glassware and plastic ware should be carefully cleaned and rinsed to Avoid cross contamination. The exact method of cleaning is described in the EN 13804 standard.)

Laboratory mill (e.g. knife mill)

Laboratory microwave oven

Atomic absorption spectrometer

Element specific lamps:

For sodium element specific lamps with wavelength of 589,0 nm is applied.

(Tips: Sodium can be measured with AAS in emission mode.)

Acetylene with appropriate quality.

Air

Determination of Sodium content in meat

EN 15505:2008

Procedure:

1. Homogenize the sample with a laboratory mill.

Suggestion: in some cases, the drying of the sample is needed in a way that does not affect the element contents, e.g. by freeze drying.

2. Sample preparation

- Weigh 1.0-2.5 g of sample in a vessel.
- Add 5.0 ml of nitric acid. After 30 minutes add 5.0 ml of distilled water and mix it gently.
- Allow samples to predigest by standing open for a minimum of 15 minutes before sealing vessels and proceeding to heating program.

Tips: In case of samples with high fat content, reduce the test portion 0.5-1.0 g.

In case of samples with high water content, the test portion can be increased up to 2.0-3.0 g.

Samples with high carbon content (e.g. sugar, fat) may cause sudden pressure peaks during the process. Allow these samples to predigest by standing an overnight.

3. Microwave heating program

Step	Time (min)	Temperature (°C)
1	0-15	Up to 190 °C
2	15-35	200 °C
3	From 35 and onwards	Cooling down

4. Dilution

Pipette a suitable volume of the sample solution, add 1 ml of Cs-solution and dilute (practically to 500 ml) this volume with 2,7 % nitric acid so that the final concentration of Na is within the range of measurement of the element.

5. Atomic absorption spectrometry

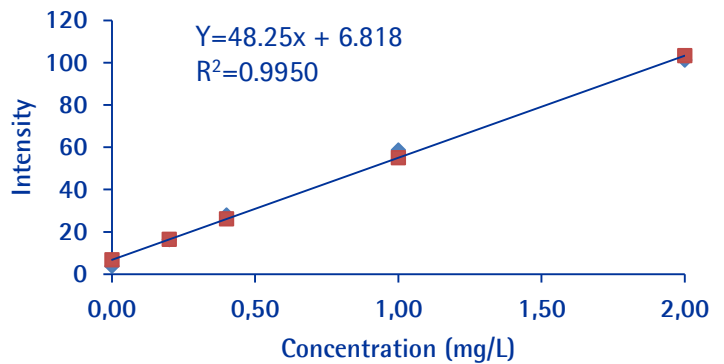
Before every determination, adjust the instrument as specified in the manufacturer's operating manual. The exact settings of our instrument is attached in a separate file.

Determination of Sodium content in meat

EN 15505:2008

6. Calibration - Sodium

Sample ID	"Intensity" (Height)"	"Conc (mg/L)"
Blank	3.7	0.0000
Std1	16.3	0.2000
Std2	28	0.4000
Std3	58.6	1.0000
Std4	101.2	2.0000
P-20298	50.4	0.8246
36814	55.1	0.9231



7. Calculation

$$c = (a \times V \times F) / m \quad \text{where}$$

c: is the mass fraction of sodium in milligram per kilogram

a: is the content of the element, in mg/l

V: is the volume of the digestion solution, in ml

F: is the dilution factor of the test solution

m: is the initial sample, in grams

	sample 2015/P/20298 (cold cut)	sample 2015/36814 (salami)
a (mg/L)	0.8246	0.9231
V (mL)	500	500
F (dilution)	25	50
m (g)	1.5713	1.3890
Sodium content (%)	0.66	1.66
Salt content (%)	1.65	4.15