



Original Research Article

Sodium chloride determination in meat products: Comparison of the official titration-based method with atomic absorption spectrometry

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ABSTRACT

The objective of this study was to compare the efficacy of the official chloride method (OM) and the atomic absorption spectrometry (AAS) technique to determine the salt/sodium content in different meat products with and without nutritional claims related to salt/sodium. Model meat systems with NaCl (2 %) and NaCl (1 %) + KCl (1 %) and commercial meat products, with and without nutritional claims, were analysed. Greater accuracy was found with AAS relative to OM in determining the percentage of salt in samples with added NaCl + KCl (1.66 % vs 0.91 % and 1.47 % vs 0.89 % for the model systems of burger meat and cooked sausage, respectively). However, there was poor correlation in the percentages of salt given by AAS, OM, and the nutritional composition or ingredient labeling, while the sodium and potassium concentrations determined by AAS agreed with the label information. Thus, the quantity of sodium, and even of potassium, should be shown on meat product labels, which should carefully specify the ingredients. The AAS method showed good performance (recovery 96.0 % and 99.2 %, precision 2.2 % and 1.3 %, linearity >0.997, limit of detection 0.004 and 0.002, limit of quantification 0.04 and 0.02, respectively for sodium and potassium).

1. Introduction

Sodium chloride is an essential ingredient in meat products since it positively influences their quality, especially sensory and safety aspects. It is responsible for the characteristic salty taste and improves the flavour (Tobin et al., 2012). The addition of sodium chloride also contributes to the texture of the meat products since it influences the activity of muscle enzymes and the solubilisation of the myofibrillar proteins (Bombrum et al., 2014). Also, due to its preservative effect, sodium chloride is essential from a safety point of view. It acts as a bacteriostatic by decreasing the water activity, thus slowing down microbial growth and inhibiting pathogenic microorganisms (Andrés and Ruiz, 2001). All these benefits have led the meat industry to use sodium chloride (common salt) as an indispensable ingredient in processing meat products.

However, sodium chloride is questioned from the nutritional point of view due to its over-consumption being linked to hypertension and cardiovascular disorders (Brown et al., 2009). It is worth mentioning that salt and sodium are normally used as if they were synonymous, but the adverse health effects are attributed to sodium. The World Health Organization recommends a daily intake of sodium chloride of less than 5 g for adults (i.e., < 2 g of sodium) (Kloss et al., 2015), but this amount is exceeded in some European countries by more than twice.

In meat products, sodium is principally incorporated through the addition of sodium chloride (NaCl), and to a lesser extent through some other additives such as sodium nitrate and nitrite (widely used as preservatives in meat products), sodium lactate (principally added as an emulsifier or flavour enhancer), or sodium ascorbate (used as an antioxidant). Thus, although the quantity of sodium in fresh meat is low (48–80 mg/100 g sample) (Wolmarans et al., 2010), meat products

Abbreviations: OM, official titration method; AAS, atomic absorption spectrometry; IC, ion chromatography; ICP-MS, inductively coupled plasma mass spectrometry; FES, flame emission spectrometry; BM, burger meat; CS, cooked sausage; S1, with added NaCl; S2, with added NaCl+KCl; T0 and T4, samples purchased four months apart.

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contribute 20–30 % of the total amount of salt ingested in the diet (Campagnol et al., 2017). Fermented and dry-cured meat products, such as sausages, hams, or loins, have the greatest amounts of salt, which is related to the dehydration that occurs during processing.

Consequently, there is a current trend to lower the content of sodium chloride that has been added to meat products (Vidal et al., 2021). To that end, the most commonly used strategy is based on partially replacing sodium chloride with potassium chloride, although magnesium and calcium salts and ascorbates have also been tested as sodium chloride substitutes (Lilic et al., 2019; Vargas-Ramella et al., 2021; Vidal et al., 2020). In this regard, there is European legislation (n°, 1924/2006; 1047/2012, and 432/2012) on nutrition claims (low sodium/salt; very low sodium/salt; sodium-free or salt-free; reduced sodium/salt; no added sodium/salt) and health claims (reducing consumption of sodium contributes to the maintenance of normal blood pressure) made for the salt/sodium content in foods (Table 1). The meat industry has been aware of these facts, and thus nowadays meat products labeled with some of these claims can be found on the market.

According to the European regulation EU (n°, 1169/2011), the food label must include the nutritional information of the energy value and the concentrations of lipids, saturated fatty acids, carbohydrates, proteins, sugars, and salt. This regulation also indicates that the term “salt” must be used instead of the corresponding nutrient “sodium”, while allowing the labeling to indicate, when applicable, that the salt content is due to the sodium naturally present in the food. The determination of the content of salt should also be specified: it must be calculated by multiplying the quantity of sodium by plus 2.5.

However, the official method recommended by the Association of Official Analytical Chemists (AOAC, 2005) to determine the salt content in meat and meat products is based on the determination of chlorides, assuming that sodium chloride is the major contributor to the sodium levels (Capuano et al., 2013). This method may also involve assuming that all the chlorides detected are from sodium chloride. Nevertheless, these assumptions may not hold for some of today’s meat products since other sodium salts, such as phosphates and nitrates, are usually added to meat products for technological purposes. Reflection is therefore merited on the appropriateness today of applying the official chloride-based method.

Despite the existence of the official method for salt determination in meat products, there have been some publications that determined the sodium content in meat products by means of other techniques, such as

Table 1
Nutritional and health claims for sodium-salt on foods according to Commission Regulations (EU) No 1924/2006, 1047/2012, and 432/2012.

NUTRITIONAL CLAIM	CONTENT	HEALTH CLAIM
Low sodium/salt	< 0.12 of sodium, or the equivalent value for salt, per 100 g of product	
Very low sodium/salt	< 0.04 of sodium, or the equivalent value for salt, per 100 g of product	
Sodium-free or salt-free	< 0.005 of sodium, or the equivalent value for salt, per 100 g of product	
Reduced sodium/salt	Where the reduction in content is at least 25 % compared to a similar product Where the product does not contain any added sodium/salt or any other ingredient containing added sodium/salt and the product contains no more than 0,12 g sodium, or the equivalent value for salt, per 100 g.	Reducing consumption of sodium contributes to the maintenance of normal blood pressure
No added sodium/salt		

ion chromatography (IC) (Aliño et al., 2010; Ripollés et al., 2011), inductively coupled plasma mass spectrometry (ICP-MS) (Lilic et al., 2019), flame emission spectroscopy (FES) (Capuano et al., 2013), and atomic absorption spectrometry (AAS) (Kameník et al., 2017; da Silva Araujo et al., 2021). Most of these studies merely focused on determining the content of sodium/salt. Only Capuano et al. (2013) and Kameník et al. (2017) compared the titration method of determining the chloride content with the assessment of the sodium content by FES and AAS, respectively. They analysed meat products with added sodium chloride, but no foods with other added salts. In the study by Kameník et al. (2017), the impact of the partial replacement of sodium chloride with potassium chloride or ammonium chloride in pork stew was investigated by AAS, but that work did not have as an objective any methodological comparison.

Given the assumption that the official titration method is not totally reliable for quantifying the sodium/salt content in meat products that have added salts other than NaCl, even though it is actually applied to such products, and the ability of AAS to determine minerals in food, the present study was aimed at taking a step forward in (i) comparing the suitability of the AOAC titration analysis based method and the AAS technique to determine the salt/sodium content in different meat products with or without nutritional claims related to salt/sodium, (ii) determining the potassium content by AAS in meat products with added potassium chloride, and (iii) getting comprehensive information about the labeling specifications of meat products with nutritional claims related to salt/sodium.

2. Material and methods

2.1. Reagents

Ethanol (Panreac, Barcelona, Spain), Carrez I and II solutions (potassium hexacyanoferrate (II) trihydrate and zinc sulfate heptahydrate) (Merck, Darmstadt, Germany), and distilled water (Milli-Q Integral; Merck, Darmstadt, Germany) were used for the preparation of the chloride extracts. For the titration, silver nitrate, nitric acid (Merck, Darmstadt, Germany), ammonium ferric sulfate, nitrobenzene (Sharlau, Barcelona, Spain), and distilled water were used.

2.2. Materials

Minced pork meat, minced pork belly, wheat flour, mashed potatoes, water, NaCl, and KCl, to elaborate the meat model systems, were acquired in supermarkets.

2.3. Experimental design

Firstly, two meat model systems – Burger Meat (BM) and Cooked Sausage (CS) – were elaborated by adding NaCl or NaCl + KCl salts to the rest of the ingredients. Two batches of each were prepared: one with NaCl (BM-S1, CS-S1) and the other with NaCl + KCl (50:50 w/w) (BM-S2, CS-S2). The BM model systems were prepared with minced pork meat (96.46 %), wheat flour (2.34 %), mashed potatoes (2.34 %), and salt (NaCl or NaCl + KCl) (1.87 %). The ingredients were mixed using a Thermomix model TM5 (Wuppertal, Germany). For the CS model system, minced pork (39.62 %), minced pork belly (39.62 %), water (18.87 %), and salt (NaCl or NaCl + KCl) (1.89 %) were kneaded by using a Thermomix model TM5 (Wuppertal, Germany). After being packed into 50 mL Falcon tubes, the mixes were heated at 80 °C for 15 min in a water bath and finally cooled in ice water. Five replicates (n = 5) were elaborated of each batch.

Secondly, different types of meat products (fresh: pork loin, pork-buff burger meat; heat-treated: oven-pickled loin, cooked turkey breast, cooked ham; not heat-treated: salami-type sausage, hard pork sausage, and dry-cured ham), with and without nutritional claims related to the salt/sodium content, were purchased in supermarkets at

two times four months apart (T0 and T4). Table 2 details the ingredients shown on the labels of the meat products analysed.

Salt-sodium contents were determined in the meat model systems and in the purchased meat products by means of the official method (AOAC, 2005) (OM) and the AAS technique. Also, in the meat model systems the quantity of potassium was determined by AAS.

2.4. Salt-sodium analysis

2.4.1. Official method (AOAC, 2005) (OM)

Salt content was determined in accordance with the AOAC method (2005 reference 969.23). Firstly, the extracts were prepared from 10 g of minced samples added to 40 % (150 mL) ethanol and heated (50 °C, 1 h) in order to extract the chloride ions. The mixture was filtered into a 250 mL volumetric flask, mixed with Carrez I and II (5 mL of each solution), topped up with 40 % ethanol, shaken, and left to stand in the dark for 10 min. Then, the content was centrifuged (3000 rpm, 10 min), filtered, heated to evaporate down to 100 mL, followed by the addition of distilled water until reaching 200 mL.

Table 2
Ingredients and salt content shown in the labels of the analysed meat products.

Commercial name	INGREDIENTS
Fresh loin	Not indicated
Oven-pickled loin	Pork loin (60 %), water, salt, soy protein, sugar, flavours, smoke flavour, stabilizers (E-451, E-407, E-420), flavour enhancer (E-621), conservative (E-250), antioxidants (E-316, E-331) and natural dye (E-120). Paprika and species. Traces of milk proteins.
Oven-pickled loin reduced salt	Pork loin (55 %), water, milk powder, potato starch, soy protein, sugar, potassium chloride, smoke flavour and species, stabilizers (E-451, E-407, E-420), flavour enhancer (E-621), antioxidants (E-316, E-331), conservative (E-250), paprika coating, conservative (E-200).
Pork-beef burger meat	Pork (45 %), beef (40 %), maize/pea (4 %), water, salt, sugar, onion, species, sodium lactate, sodium sulphite, sodium ascorbate, sodium citrates, cochineal.
Pork-beef burger meat reduced salt	Pork (52 %), beef (35 %), water, plant fibre (2.2 %), soy protein (1.8 %), salt, dextrose, sodium sulphite, sodium ascorbate, sodium citrates, cochineal.
Cooked turkey breast	Turkey breast (55 %), water, powder milk, soy protein, sugar, maize dextrose, flavours, smoke flavour, stabilizers (E-451, E-407), antioxidants (E-316, E-331), conservative (E-250). Traces of celery and egg.
Cooked turkey breast reduced salt	Turkey breast (67 %), water, soy protein, maize dextrose, flavours, potassium chloride, sugar, stabilizers (E-451, E-420, E-407), antioxidants (E-316, E-331), conservative (E-250). Traces of milk protein.
Cooked ham	Pork ham (70 %), water, salt, powder milk, sugar, maize maltodextrine, flavours, stabilizers (E-451, E-407), antioxidants (E-316, E-331), conservative (E-250). Traces of soy.
Cooked ham reduced salt	Pork ham (85 %), water, potassium chloride, maize dextrose, sugar, maize glucose syrup, flavours, stabilizer (E-407), antioxidants (E-316, E-331), conservative (E-250). Traces of milk protein, soy and its derivatives.
Salami-type sausage	Pork, salt, lactose, dextrin, soy protein, species, sugar, stabilizer (E-450), milk proteins, antioxidants (E-316), conservatives (E-250, E-252), dye (E-120).
Salami-type sausage 25 % reduced salt	Pork, sunflower oil, salt, stabilizers (E-508), soy protein, acidity corrector (E-262, E-327), dextrose, species, flavours, antioxidants (E-300), conservatives (E-250, E-252), dye (E-120).
Hard pork sausage	Pork, paprika, salt, garlic, conservative (E-250).
Hard pork sausage 40 % reduced salt	Pork, sunflower oil, stabilizer (E-508), specie, dextrin, soy protein, milk proteins, salt, dextrose, antioxidants (E-301), conservatives (E-252, E250), natural flavour, dye (E-120).
Dry-cured ham	Pork ham, salt, conservatives (E-250, E-252).
Dry-cured ham 25 % reduced salt	Pork ham, salt, stabilizer (E-508), conservatives (E-250, E-252).

Once the extract had been prepared, it was titrated. To do that, the extract (10 mL), silver nitrate 0.1 mol/L (10 mL), nitric acid (1 mL), ammonium ferric sulfate 4 % (1 mL), and distilled water (50 mL) were mixed, shaken, and left to stand in the dark for 10 min. In this way, the chlorides of the extract react with part of the silver nitrate, resulting in silver chloride and the 0.1 mol/L silver nitrate remaining. Then, nitrobenzene (1 mL) was added to coagulate the precipitate. Finally, the titration of the remaining 0.1 mol/L silver nitrate was done with 0.1 mol/L of potassium sulfocyanide. Simultaneously, a blank was run with 10 mL of water instead of the extract.

The results were expressed as g NaCl/100 g samples, based on the equivalence between NaCl and the remaining 0.1 mol/L of silver nitrate and considering the exact weight of the sample. Each sample was analysed fully (extract preparation and titration) in triplicate.

2.4.2. Atomic absorption spectrometry analysis

Sodium and potassium contents were determined in accordance with the AOAC (2005 reference 985.35); ISO 9964-1 (1993), and ISO 9964-2 (1993). The samples were calcined in a muffle furnace at a temperature of 600 °C until constant weight. Then the following process was carried out in triplicate: 1 g of calcined sample was digested by adding HCl-HNO₃ in a 3:1 ratio. This solution was then filtered into a 100 mL volumetric flask using Whatman n° 44 filter paper, and the volume was topped up to 100 mL using de-ionized water. Next, the necessary dilutions were made until reaching concentrations of sodium and potassium that were within the ranges of the standard curves.

For the digestion of the samples, hydrochloric acid (37 %, Sharlau, Barcelona, Spain) and nitric acid (69 %, Sharlau, Barcelona, Spain) were used. For the AAS measurements, the potassium chloride and sodium chloride standards were procured from Panreac (Barcelona, Spain). Standard solutions were prepared of sodium (1000 mg/L) and potassium (1000 mg/L). These standard solutions were diluted with de-ionized water to obtain a series of working standard solutions of sodium (0.1 mg/L, 0.2 mg/L, 0.4 mg/L, 0.6 mg/L, and 1 mg/L) and potassium (0.1 mg/L, 0.2 mg/L, 0.4 mg/L, 0.6 mg/L and 1 mg/L). Caesium chloride (Sharlau, Barcelona, Spain) was used to eliminate ionization interferences.

Sodium and potassium were determined by flame absorption spectrometry (Na: wavelength 589.0 nm, slit 0.2 nm, air-acetylene; and K: wavelength 766.5 nm, slit 0.2 nm, air-acetylene) on a PerkinElmer Analyst 700 atomic absorption spectrometer (PerkinElmer, CT). To guarantee the reliability of the results, the SPS-SW2 (Quality Control Material) reference material with established sodium and potassium contents was used.

2.5. Quality control in the atomic absorption spectrometry

To ensure the quality of the measurements, prior to taking the sample readings, de-ionized water was aspirated for 15 min to free the nebulizer inlet tube of any imperfections. A blank was always run to set the zero before sample analysis. After every 10 samples, the set-up was recalibrated by aspirating the blank solution and the standard solutions. All the samples were analysed in triplicate for precision. The inlet tubes were cleaned for 10 s between samples.

The limit of detection (LOD) and the limit of quantification (LOQ) were determined as $3 \times \text{standard deviation} / \text{slope of the calibration curve}$ and $10 \times \text{standard deviation} / \text{slope of the calibration curve}$, respectively. The blank was aspirated 20 times. To determine the accuracy, the percentage recovery was calculated in samples spiked with appropriate amounts of sodium and potassium (see Section 2.3). The precision was evaluated by calculating the relative standard deviation (RSD) for different parts of the sample. For this, different parts of the sample were digested and then assayed. The linearity was evaluated by means of the coefficient of determination (R^2) of sodium and potassium calibration curves.

The method was validated in accordance with the standard ISO

17,025:2017 guidelines against the certified reference material SPS-SW2 (surface water). All these results are specified in the following section.

2.6. Statistical analysis

Differences in (i) the percentage of salt between the official method and AAS in the meat model systems with added NaCl and NaCl + KCl and (ii) the quantity of potassium and sodium in commercial meat products with and without nutritional claims related to the salt/sodium content collected at two times four months apart were evaluated by one-way analysis of variance (ANOVA). Statistical calculations were done using the program IBM SPSS Statistics v.22 (IBM Co., New York, USA).

3. Results and discussion

3.1. Salt content in the meat model systems

Fig. 1 shows the percentage of salt determined by OM and AAS in the BM and CS meat model systems of this study. In the case of the BM batches with added NaCl, the percentage of salt determined by AAS was greater (2.01 %) than the value obtained by OM (1.74 %). Nevertheless, both percentages are within a narrow range and similar to the expected value (1.88 %). In the CS batches, no significant differences were found in the percentage of salt between AAS and OM. As for the batches with NaCl + KCl, in both model systems, the percentage of salt expressed as % NaCl was greater when determined by OM than by AAS (1.66 % vs 0.91 % and 1.47 % vs 0.89 % for BM and CS, respectively). The expected percentage of salt (NaCl) (0.95 %) was close to the AAS values. Thus, the percentage of Na expressed as salt (NaCl) determined by OM is overestimated when NaCl is partially or totally substituted by KCl. These results were to be expected because the OM determines chloride (AOAC, 2005), but they reveal that OM is not a reliable method to quantify the salt content in today's meat products. This is a back titration procedure used for determining silver salt soluble in nitric acid. In this method an excess of standard solution of silver nitrate is added to a chloride-containing sample solution. The excess silver is then back titrated using a standardized solution of potassium or ammonium thiocyanate with ferric ion as indicator. The simplicity of the procedure, which does not require complex instrumentation, led the AOAC to propose it as an official method to determine the salt in meat products. Haouet et al. (2006) performed a validation of the Volhard method for chloride determination in food. Overall, the validation parameters demonstrate the fitness for purpose of the Volhard method in its field of application, with limits that are adequate for the chloride contents of foods, including meat products. However, this method may solely be appropriate when sodium chloride is the only salt added to meat

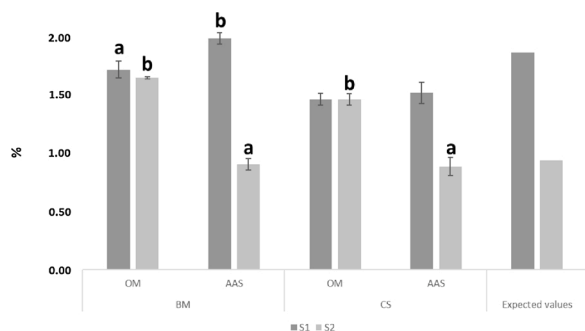


Fig. 1. Percentage of salt (average \pm standard deviation) determined by titration based analysis (AOAC) and atomic absorption spectrometry (AAS) in burger meat (BM) and cooked sausages (CS) model systems added with 2 % NaCl (S1) and 1 % NaCl + 1 % KCl (S2)*.

*Different letters (a,b) within the same product indicate significant differences between methods (AOAC vs. AAS).

products and is the major contributor to their sodium levels (Capuano et al., 2013). This is worth noting because the European regulation on food labeling (N° 1169/2011) indicates that the term “salt” must be used when referring to the sodium content.

3.2. Salt (NaCl) content in commercial meat products

The percentages of salt (NaCl) in the commercial meat products analysed by OM and AAS are listed in Table 3. In six of the seven meat products without nutritional claims related to salt/sodium, AAS showed greater percentages of salt than the official method (oven-pickled loin: 0.81 % vs 1.25 %; pork-beef burger meat: 0.65 % vs 1.90 %; cooked turkey breast: 1.00 % vs 2.79 %; cooked ham: 0.93 % vs 2.49 %; salami-type sausage: 2.54 % vs 4.69 %; dry-cured ham: 3.59 % vs 4.84 %; respectively). The same was found in the case of meat products with nutritional claims concerning salt/sodium, with AAS showing greater salt percentages in five of the seven samples (oven-pickled loin reduced salt: 0.50 % vs 1.11 %; cooked turkey breast reduced salt: 0.66 % vs 1.58 %; cooked ham reduced salt: 0.78 % vs 1.77 %; hard pork sausage 40 % reduced salt: 2.92 % vs 2.66 %; dry-cured ham 25 % reduced salt: 2.06 % vs 3.46 %; respectively). Similarly, Capuano et al. (2013) found significantly higher NaCl contents calculated from sodium by FES than from chloride in different meat products, which those authors related to the additional sources of sodium and/or chloride content of the ingredients. However, in the study by Kamenik et al. (2017) no differences were found in the salt levels determined by AAS and the Mohr titration method, this being ascribed to the high variability in the results. Also, in comparison with the percentage of salt indicated on the label of the commercial meat products analysed (Table 3), AAS showed values that were similar (pork-beef burger meat: 1.90 % and 2.2 %; pork-beef burger meat reduced salt: 0.81 % and 0.84 %; cooked turkey breast reduced salt: 1.58 % and 1.5 %; cooked ham: 2.49 % and 2.5 %; hard

Table 3

Percentage of salt (NaCl) in the label of commercial meat product with and without nutritional claims related to the salt/sodium and determined by titration based analysis (AOAC) and atomic absorption spectrometry (AAS)*.

Commercial meat product	Label	AOAC	AAS	p
Fresh loin	Not indicated	Not detected	0.25 \pm 0.05	–
Oven-pickled loin	2.6	0.81 \pm 0.01	1.25 \pm 0.05	<0.001
Oven-pickled loin reduced salt	2.2	0.50 \pm 0.07	1.11 \pm 0.10	<0.001
Pork-beef burger meat	2.2	0.65 \pm 0.29	1.90 \pm 0.05	<0.001
Pork-beef burger meat reduced salt	0.84	0.92 \pm 0.01	0.81 \pm 0.10	0.109
Cooked turkey breast	1.6	1.00 \pm 0.07	2.79 \pm 0.05	<0.001
Cooked turkey breast reduced salt	1.5	0.66 \pm 0.07	1.58 \pm 0.03	<0.001
Cooked ham	2.5	0.93 \pm 0.12	2.49 \pm 0.05	<0.001
Cooked ham reduced salt	1.3	0.78 \pm 0.07	1.77 \pm 0.59	0.034
Salami-type sausage	3.5	2.54 \pm 0.18	4.69 \pm 0.23	<0.001
Salami-type sausage 25 % reduced salt	2.8	2.87 \pm 0.18	3.42 \pm 0.55	0.119
Hard pork sausage	3.3	2.89 \pm 0.07	3.34 \pm 0.88	0.520
Hard pork sausage 40 % reduced salt	2	2.92 \pm 0.02	2.66 \pm 0.38	0.005
Dry-cured ham	5	3.59 \pm 0.12	4.84 \pm 0.07	<0.001
Dry-cured ham 25 % reduced salt	3.5	2.06 \pm 0.11	3.46 \pm 0.48	0.001

* p < 0.05 indicates significant differences between percentage determined by AOAC and AAS.

pork sausage: 3.34 % and 3.3 %; dry-cured ham: 4.84 % and 5 %; dry-cured ham 25 % reduced salt: 3.46 % and 3.5 %; for AAS and label respectively) or greater (cooked turkey breast: 2.79 % and 1.6 %; cooked ham reduced salt: 1.77 % and 1.33 %; salami-type sausage: 4.69 % and 3.5 %; salami-type sausage 25 % reduced salt: 3.42 % and 2.8 %; hard pork sausage 40 % reduced salt: 3.66 % and 2 %; for AAS and label respectively), except for oven-pickled loins with and without nutritional claims. The official method showed a lower percentage of salt than the values on the label of most samples (oven-pickled loin: 0.81 % and 2.6 %; oven-pickled loin reduced salt: 0.50 % and 2.2 %; pork-beef burger meat: 0.65 % and 2.2 %; cooked turkey breast: 0.00 % and 1.6 %; cooked turkey breast reduced salt: 0.66 % and 1.5 %; cooked ham: 0.93 % and 2.5 %; cooked ham reduced salt: 0.78 % and 1.3 %; hard pork sausage: 2.89 % and 3.3 %; dry-cured ham: 3.59 % and 5 %; dry-cured ham 25 % reduced salt: 2.06 % and 3.5 %; respectively), achieving similar values only in pork-burger meat reduced salt (0.92 % and 0.84 %, respectively) and salami-type sausage 25 % reduced salt (2.87 % and 2.8 %, respectively), and greater in hard-pork sausage 25 % reduced salt (2.92 % and 2 %, respectively). These results do not agree with those obtained in the meat model systems, which showed greater salt percentages with the official method when substituting NaCl by KCl. According to the label information about the ingredients of the meat products analysed (Table 2), the addition of potassium chloride (or E-508) had been applied in six of the seven meat products with nutritional claims concerning salt/sodium. However, the label of some of these meat products (oven-pickled loin reduced salt, pork-beef burger meat reduced salt, and cooked turkey breast reduced salt) did not include the term salt. This seems striking since the replacement of salt (NaCl) by sodium potassium must be limited, i.e., at most 40 % for dry-cured loin (Aliño et al., 2009), because total substitution leads to a bitter and metallic taste (Gelabert et al., 2003). Moreover, most of these commercial meat products have other additives containing sodium (E250: sodium nitrite; E252: sodium nitrate; E316: sodium erythorbate; E331: sodium citrate) or sodium and potassium (E451: triphosphate of pentasodium, pentapotassium, and sodium-potassium) (Table 2), which may explain the higher salt concentrations obtained by AAS in some meat products. In view of these findings, it is difficult to make any particular claim about good or poor agreement between the label information and the method for the percentage of salt. Consequently, the present study went one step further by using AAS to analyse the sodium and potassium concentrations in the commercial meat products.

3.3. Sodium and potassium contents of commercial meat products as determined by AAS

Fig. 2 shows the quantity of sodium and potassium in commercial

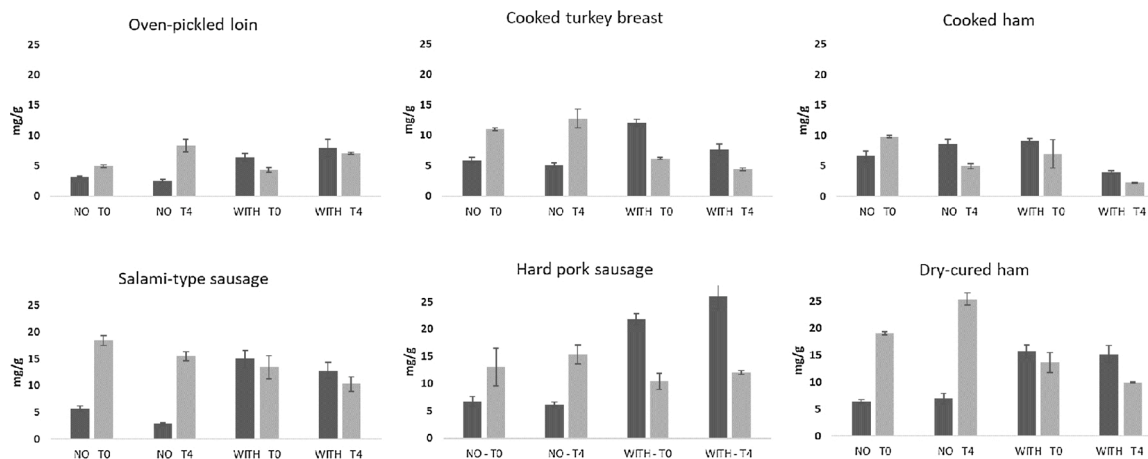


Fig. 2. Quantities of potassium (dark gray) and sodium (light gray) in meat products with (WITH) and without (NO) nutritional claims related to the salt/sodium at two different moments in time with four months apart (T0 and T4).

meat products with and without nutritional claims related to salt/sodium content collected at two different times four months apart. The statistical results (p -values) are given in Table 4. As can be seen, all the meat products analysed contained sodium (from 2.25 to 25.40 mg/g sample) and potassium (from 2.78 to 26.11 mg/g sample).

Statistical differences were found in the quantity of sodium and potassium between the products with and without nutritional claims related to salt/sodium. Meat products with nutritional claims had lower sodium and higher potassium concentrations than those not labeled with nutritional claims. This finding mostly agrees with the partial replacement of NaCl by KCl to decrease the quantity of sodium in meat products, which is recommended so as to not prejudice sensory quality (Aliño et al., 2009; Gelabert et al., 2003). However, it contrasts with nutritional claims related to salt/sodium in three meat products: oven-pickled loin reduced salt, cooked turkey breast reduced salt, and cooked ham reduced salt, whose labels indicate the addition of potassium chloride but not of salt (Table 2). Overall, no marked differences were obtained between the T0 and T4 samples, indicating the maintenance of the manufacturers' processing parameters. Thus, the determination of sodium and potassium by means of AAS seems to reveal to a great extent the added ingredients, both those added in major proportions, such as NaCl and KCl, and the lesser additives such as sodium nitrite and sodium phosphate. Consequently, the performance of AAS in determining sodium and potassium was evaluated to validate this method. In comparison with the results of the present study, higher Na levels have been reported by Capuano et al. (2013) in meat products (57–189 mg/g sample). This might be due to a general decrease in the content of NaCl added to meat products with time (from 2013 to 2020). In fact, Sparks et al. (2018) found 11 % reduction in the median sodium level of processed meats from 2010 to 2017. Moreover, the Food Safety Authority of Ireland, which has coordinated a salt reduction programme working in

Table 4

Statistical results (p -value) on the quantity of potassium and sodium in different meat products with (WITH) and without (NO) nutritional claims related to the salt/sodium at two different moments in time with four months apart (T0 and T4).

	WITH vs. NO		T0 vs. T4	
	Na	K	Na	K
Oven-pickled loin	0.379	0.005	<0.001	0.282
Cooked turkey breast	<0.001	0.002	0.992	0.115
Cooked ham	0.026	0.419	0.004	0.223
Salami-type sausage	0.002	<0.001	0.116	0.433
Hard pork sausage	<0.001	<0.001	0.751	0.677
Dry-cured ham	<0.001	<0.001	0.727	0.997

partnership with the food industry, monitored sodium and potassium concentrations in processed food from 2003 to 2018 by means of atomic emission spectrometry (FSAI, 2019). The results for the processed meat products of this study revealed an increase in potassium and a decrease in sodium over time – for example, 13 % and 35 % potassium increase in bacon rashers and meat pudding, respectively, and 11 %, 27 %, and 15 % sodium decrease in sausages, bacon rashers, and cooked ham, respectively.

These results on the percentages of NaCl and sodium determined by OM and AAS, the potassium concentrations, and the label information on the meat products may indicate that the direct determination of sodium by AAS is more useful than the indirect measurement of chlorides by titration to calculate the percentage of salt in meat products, especially when KCl is substituted for part of the added NaCl.

3.4. Performance of AAS

The quality parameters for sodium and potassium determined by the AAS method used in the present work are listed in Table 5. The linearity of the calibration curves was good ($R > 0.997$ for sodium and potassium). The LOD and LOQ were 0.004 and 0.04 mg/g for sodium, and 0.002 and 0.02 mg/g for potassium, respectively. These values are lower than the limits found in the Food Safety Authority of Ireland study (LOQ = 0.10 mg/g for sodium and potassium) (FSAI, 2019), and lower than those reported by Capuano et al. (2013) (LOD = 0.23 mg/g and LOQ = 0.46 mg/g for sodium). Accuracy and precision were high, with recoveries of 96 % and 99.2 % and RSD of 2.2 % and 1.3 % for sodium and potassium, respectively. According to Ribani et al. (2004), recovery can vary between 70 % and 120 %, and this is satisfied by the method proposed in the present study.

4. Conclusions

The AAS determination of the salt percentage in meat products with and without nutritional claims related to salt/sodium, which is centred on direct assay of the sodium content, is more accurate than the titration based method of analysis (AOAC, 2005). Nevertheless, there is no absolute agreement for the percentage of salt in meat products among the AAS and OM results and the nutritional composition or ingredients listed on the labels.

The titration based method of analysis (AOAC, 2005) is not totally reliable in quantifying the salt content in all of today's meat products, which may lead to a recommendation for this method to be revised.

Meat products with nutritional claims related to salt/sodium contained significant amounts of potassium due to the usual partial substitution of NaCl by KCl. This may suggest that sodium and potassium concentrations should be added to the label, besides the salt percentage, and that there is a need to review the labeling regulations for this kind of foodstuffs. Also, particular attention should be paid to specifying the ingredients of these products. Thus, consumers would have available to them the appropriate information about the sodium and potassium concentrations, whose influence on health has been well demonstrated.

The goodness of the quality parameters of the AAS method applied in this study makes it suitable for the quantification of sodium and potassium in meat products.

CRediT authorship contribution statement

Trinidad Perez-Palacios: Term; conceptualization; formal analysis; resources; data curation; writing - original draft; editing; visualization; project administration. **Alejandro Salas:** Validation; resources; review; supervision; project administration. **Adela Mejias:** investigation. **Eder-Renato Ojara:** Investigation. **Teresa Antequera:** Term; conceptualization; resources; review; supervision; project administration.

Table 5

Quality parameters for sodium and potassium determined by Atomic Absorption Spectrometry*.

	Accuracy (%) Recovery values)	Precision (%) RSD)	Linearity (R)	LOD (mg/g)	LOQ (mg/g)
Sodium	96.0	2.2	>0.997	0.004	0.04
Potassium	99.2	1.3	>0.997	0.002	0.02

Limit of detection (LOD) and quantification (LOQ); relative standard deviation (RSD); (R) correlation coefficient.

* Reference values for K and Na: 1.000 ± 0.005 and 10.000 ± 0.050 mg/L, respectively.

Declaration of Competing Interest

The authors report no declarations of interest.

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