White Paper

Quantification of sodium and salt in foods: variations in analytical methods used in regulatory decision making

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Summary

The high level of sodium intake in our diets has been widely associated with increased blood pressure (a risk factor for certain non-communicable diseases such as cardio-vascular diseases) and has emerged as a growing public health concern.

The high level of sodium intake in our diets has been widely associated with increased blood pressure (*a risk factor for certain non-communicable diseases such as cardiovascular diseases*) and has emerged as a growing public health concern. Food manufacturers reduce the level of sodium in their products by reducing salt (*sodium chloride*) as well as other sodium-contributors such as monosodium glutamate (*MSG*) in their recipe to improve the quality of the nutritional profile of products in accordance with WHO recommendations. In order to achieve the target reduction and maintain consumer acceptance after reformulation of food products, one of the approaches that food manufacturers use is the partial replacement of sodium chloride (*NaCl*) with potassium chloride (*KCl*). Governments, on the other hand use different approaches to drive the reduction of sodium and salt in foods. Some provide guidelines and recommendations, while others implement standards or regulations stipulating maximum limits for sodium and/or sodium chloride in various food products.

Two fundamentally different analytical approaches are commonly followed for the quantification of sodium and salt in foods. The first involves elemental analytical techniques such as Inductively Coupled Plasma – Mass Spectrometry (*ICP-MS*), Inductively Coupled Plasma – Optical Emission Spectrometry (*ICP-OES*) and Atomic Absorption Spectrometry (*AAS*). The second involves potentiometric analytical techniques such as argentometric titration, sodium ion-selective electrode and more recently, the Multi-Standard Addition (*MSA*) method.

Elemental analytical techniques directly quantify the total sodium in the food matrix. The *argentometric titration* (*a potentiometric analytical technique*) quantifies the chloride then express it as the equivalent sodium or salt, while the sodium ion-selective electrode method and MSA (*other potentiometric techniques*) quantifies the sodium ions in the food matrix. The inherent difference in what is measured by the elemental analytical methods and the potentiometric methods in some instances, can lead to significant differences in the results obtained for both sodium and salt content in food matrices.

In this White Paper we discuss the results for sodium and salt (*NaCl*) content obtained from analysing five samples of culinary bouillon using elemental analytical techniques (*ICP-OES and ICP-MS*) and potentiometry (*argentometric titration*). Four of the five samples contained the same amount of sodium chloride, with varying levels of MSG and KCl. The fifth sample contained significantly lower amount of NaCl, higher amount of KCl and no MSG.

Only the elemental analytical techniques can be used to reliably assess the amount of sodium in foods.

Neither the elemental nor the potentiometric analytical techniques will quantify the exact amount of salt (NaCl).

Based on the results obtained, we have demonstrated that only the elemental analytical techniques can be used to reliably assess the amount of sodium in foods, especially if a salt replacer like KCl is used. We have also shown that neither the elemental nor the potentiometric analytical techniques will quantify the exact amount of salt (*NaCl*) as both methods will detect sodium and chloride in all ingredients in the product recipe. However, as sodium is the risk factor associated with non-communicable diseases and its reduction is the focus of all salt-reduction initiatives, we recommend that the focus should be on the determination of the total sodium content rather than the salt content in order to verify salt reduction in food products.

World Health Organization recommendation on sodium and salt reduction

> ***The WHO recommendations on daily intake of sodium proposed a target of 30% relative reduction in the intake of sodium/salt. This is equivalent to a target of less than 5g salt/day (equivalent to 2000 mg Na/day).***

Ever since the World Health Organisation declaration on the negative impact of high levels of sodium in our diet on human health (*1, 2*), many countries around the world have taken action to reduce, and in some cases, limit the amount of total sodium in several foods. The primary source of sodium in our diet is common salt (*NaCl*). The WHO recommendations on daily intake of sodium proposed a target of 30% relative reduction in the intake of sodium/salt. This is equivalent to a target of less than 5g salt/day (*equivalent to 2000 mg Na/day*) by 2025. The "Global Action Plan for the Prevention and Control of Non-communicable Diseases 2013-2020" (*3*) gives guidance and a menu of policy options for Member States, WHO and other UN agencies to achieve the targets.

Linked to WHO recommendations, many countries such as the USA (*4*) and the UK (*5*) provided guidelines for the reduction of sodium/ salt in processed foods while others – like the EU (*6*) - have implemented regulations for the declaration of salt (*total sodium x 2.5*) in packaged food. A few other countries such as South Africa and Portugal went further to put in place regulations that stipulate limits for total sodium in some food commodities as well as mandatory declaration of sodium and salt on food packaging labels (*7, 8*). Additionally, some other countries, like Nigeria (*9*), Ghana (*10*), Kenya (*11*) and Senegal (*12*), have well defined standards for some categories of food products for which a maximum level was prescribed for NaCl.

Sodium reduction in product formulation

> ***Salt is the major source of sodium in processed foods.***

> ***It was estimated that about 90% of the sodium consumed comes from salt.***

The overall taste of food is generally considered as the main consumer preference driver. Salt is the major source of sodium in processed foods and plays a primary role in its overall taste profile. Apart from salt, other sources of sodium are present (*naturally and added e.g. monosodium glutamate*) in foods. However, it was estimated that about 90% of the sodium consumed comes from salt (*2*). Therefore, in order to achieve sustained reduction of sodium in our diet, salt reduction must be conducted in a manner that maintains customer acceptance after reformulation, in order to limit discretionary addition of salt at the dining table. In this context, it must be noted that in addition to its contribution to the taste of food, salt in some cases plays an important role in the preservation and safety of some foods.

A common approach is the partial replacement of NaCl with KCl.

The food industry has adopted a number of strategies to reduce sodium in the formulation of manufactured foods. A common approach is the partial replacement of NaCl with KCl. The use of KCl has been the most promising replacer of NaCl and can help in achieving the WHO target of 5g salt/day, while simultaneously increasing our dietary intake of potassium (*13*). Like common salt, KCl contributes to the taste of the food and at the same time provides some of the technological functionalities of salt e.g. water activity reduction for preservation in specific foods.

Regulatory requirements for analyzing sodium and salt in foods

We must consider the specific analytical methods or techniques that are sometimes stipulated by the regulations and standards.

Before examining which of the analytical methods are most suitable for assessing compliance with regulations and food product standards, we must consider the specific analytical methods or techniques that are sometimes stipulated by the regulations and standards or recommended by the food control authorities.

The South African Sodium Reduction regulation stipulates that "*elemental methods or suitable sodium potentiometric methods*" should be used to analyze the total sodium in a list of food commodities. The South Africa regulation for mandatory nutritional labelling of packaged foods require the declaration of total sodium and whenever the salt content is required, it should be calculated as based on the total sodium content.

Other countries such as Nigeria, Ghana, Kenya and Senegal have Standards for food commodities that set out the permitted maximum level for sodium chloride. These standards also stipulate the analytical methods to be used to verify the sodium chloride content. This is illustrated in Table 1. These national standards for culinary bouillon, for example, appear to follow the Codex guidelines. In the examples above, the standards all refer to Codex STAN 117-1998, rev 2001 & 2015 (*Standards for Bouillons and Consommés*) (*14*). The analytical methods used are also in line with the recommendations of Codex Standard CXS 234-1999, Annex 1 (*15*), that provides guidelines on sampling and methods of analysis for commodities.

Some regulations require quantification of total sodium content and salt content based on total sodium while the national standards for some food commodities, require the quantification of sodium chloride by stipulated potentiometric techniques.

To summarise, some regulations require quantification of total sodium content and salt content based on total sodium while the national standards for some food commodities, culinary bouillon for example, require the quantification of sodium chloride by stipulated potentiometric techniques.

> *The capability of the analytical methods recommended is very important to ensure that the method can satisfy the regulatory requirements and yield results that are accurate and reliable.*

Consideration of the capability of the analytical methods recommended by the Regulations and Standards is very important to ensure that the method can satisfy the regulatory requirements and yield results that are accurate and reliable. The interpretation of the results produced by these methods is also critically important. As will be demonstrated later in this White Paper, the quantification of sodium and salt, based on total sodium or chloride, can sometimes yield significantly different results depending on the composition of the food matrix.

Table 1. List of analytical methods stipulation by the regulations and Standards with the associated techniques and target analyte (sodium and sodium chloride - NaCl).

Country	Regulations / Standards	Food Commodity	Analytical method stipulated	Technique	Quantify
South Africa	Regulation	Various	Elemental	Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) Atomic Absorption Spectrometry (AAS)	Sodium
			Potentiometry	Ion-Selective Electrode	Sodium
Nigeria	Standard	Bouillon	Potentiometry	Mohr Titration ($AgNO_3$ titration)	NaCl
Senegal	Standard	Bouillon	Potentiometry	AOAC 971.27 ($AgNO_3$ titration)	NaCl
Ghana	Standard	Bouillon	Potentiometry	AOAC 971.27 ($AgNO_3$ titration)	NaCl
Kenya	Standard	Bouillon	Potentiometry	AOAC 971.27 ($AgNO_3$ titration)	NaCl

Comparison of Analytical Techniques

When quantifying the sodium and salt content in food, two fundamentally different analytical approaches may be applied. These are elemental and potentiometric analytical techniques as listed in Table 1.

The elemental analytical techniques i.e. ICP-OES, ICP-MS and AAS (*shown in Table 1*) directly quantifies sodium from all sources. In cases where the salt content is required, it is expressed as total sodium multiplied by a factor of 2.5 (*e.g. salt = total sodium x 2.5*).

The other analytical approach is by potentiometry i.e. argentometric titration (*titration by* $AgNO_3$), ion-selective electrode and MSA methods. The ion selective electrode and MSA technique measures the sodium ions in the food matrix. The argentometric titration technique on the other hand quantifies the chloride, which is then expressed as sodium by multiplying by a factor of 0.65 (e*.g. sodium = total chloride x 0.65*). In cases where the salt content is required, it is expressed as the chloride multiplied by 1.65 (*e.g. salt = chloride x 1.65*).

> *The elemental analytical techniques measure the total sodium present in the food regardless of its source.*

To summarize, the elemental analytical techniques measure the total sodium present in the food regardless of its source, e.g. sodium chloride, sodium nitrate, sodium nitrite, sodium phosphate, mono-sodium glutamate, etc. The argentometric titration potentiometric technique, quantifies the total chloride regardless of its source e.g. sodium chloride, potassium chloride, calcium chloride, etc., as well as other halide ions like iodide, bromide, and fluoride. Conversely, the ion-selective electrode and MSA techniques quantifies the sodium ions in the food matrix.

> *Sodium and chloride in foods is based on the ingredients used in the recipe of the food product.*

It follows then that since the amount of sodium and chloride in foods is based on the ingredients used in the recipe of the food product, the quantification of total sodium and salt content can be over-estimated or under-estimated depending on the analytical method used. The results obtained from elemental analysis can overestimate the NaCl content if there are other sources of sodium present in the food. Likewise, the sodium and salt content obtained from argentometric titration can be over-estimated in the presence of other chlorides (*e.g. KCl*) and under-estimated in the presence of non-halide sodium salts like MSG in the food matrix. The issue therefore lies in the choice of the analytical method used to quantify total sodium or salt in the context of the regulations or standards.

In this White Paper, we use culinary bouillon as an example of a high sodium product to demonstrate that the choice of analytical methods used to assess compliance to regulations and standards, can produce significantly different results. This invariably influences the compliance or non-compliance of the food commodity with regards to the maximum limits for sodium and salt, as well as the mandatory minimum nutritional declaration for total sodium or salt content where required. Culinary bouillon was also used in this study because it is customarily added to many prepared foods and can sometimes be the main source of sodium and salt in some savory dishes. We also aim to show that the choice of analytical method can significantly determine the accuracy and reliability of the results obtained for total sodium and salt content in foods.

Determination of sodium and salt content of culinary bouillon by elemental and potentiometric analytical techniques

The total sodium and salt content reference samples of culinary bouillon powder, were analyzed using the elemental analytical methods (*AOAC 2011.14 – ICP OES and AOAC 2015.06 – ICP MS*) and potentiometry (*AOAC 971.27 – $AgNO_3$ titration*). The samples were analyzed in duplicates by three different laboratories on six different days. All the participating laboratories were ISO 17025 accredited for the use of these analytical methods. Ion-Selective Electrode (*ISE*) method (*AOAC 976.25*), though an official method, was not used in this study because the accuracy and reliability of the results may be affected by the presence of potassium ions (*K^+*) (*16*), which is present in significantly high concentrations in some of the samples used in this study. The MSA method (*17*) was also not considered because it is not an official method. In addition, this study was not meant to compare an exhaustive list of all available techniques but focused on two fundamentally different principles: i.e. elemental analysis by ICP versus argentometric titration.

For the formulation of the bouillon powder, we chose a model recipe that is representative of a regular bouillon powder in terms of salt content. We also varied the formulation in order to cover a wide range of concentrations for KCl and MSG. We also included a model recipe with a reduced NaCl content (*shown in Figure 1*).

The reference values for the total sodium and total chloride for each of the samples were based on the known composition of ingredients in the recipe as shown in Table 2. A reference value for the salt content, derived from the total sodium and the total chloride reference values for each sample, was also calculated. In addition to these reference values, there is also the NaCl reference value which is based on the real amount of NaCl in the recipe. The reference values for the total sodium and the NaCl as shown in Table 2, were used as the reference values in Figures 2 and 3, when comparing the analytical results obtained for total sodium and NaCl content using the elemental and potentiometric analytical methods.

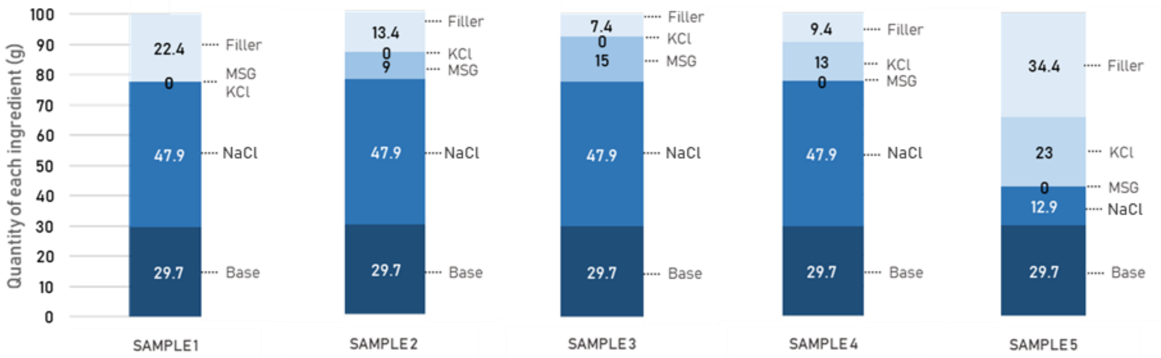

Figure 1. Composition of the reference samples used in the multi-lab analysis for total sodium and salt (NaCl) using elemental analytical methods (ICP-OES & ICP-MS) and potentiometry ($AgNO_3$ titration). The NaCl reference value (47.9 g/100g) was based on the known amount of NaCl in the Base Mix (2.9 g/100g) and the NaCl added (45 g/100g) in the recipe. The filler, a polysaccharide, was used to compensate for the variation in the dosages of NaCl, KCl and MSG.

Table 2. Comparison of the reference values for sodium and the reference value for salt content with the results obtained from elemental analytical methods (ICP-OES & ICP-MS) and potentiometry ($AgNO_3$ titration). The NaCl reference value (47.9 g/100g) was based on the known amount of NaCl in the Base Mix (2.9 g/100g) and the NaCl added (45 g/100g) in the recipe. The values for sodium by titration were obtained from the conversion of the analysed chloride content.

	REFERENCE SAMPLES				
	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5
Sodium Reference Value (mg/100g)	19011	20118	20856	19011	5256
Chloride Reference Value (mg/100g)	29075	29075	29075	35315	18870
Sodium by elemental analysis (ICP-OES & ICP-MS) (mg/100g) ($U_{95\%}$)	18892 (1677)	20063 (1769)	20698 (2174)	19222 (1330)	5353 (807)
Sodium by Titration (mg/100g) ($U_{95\%}$)	19027 (200)	18898 (329)	19151 (269)	23181 (237)	12382 (187)
NaCl Reference Value (g/100g)	47.9	47.9	47.9	47.9	12.9
NaCl based on Sodium Reference Value (g/100g)	48.3	51.1	53.0	48.3	13.4
NaCl based on Chloride Reference Value (g/100g)	48.0	48.0	48.0	58.3	31.1
NaCl by elemental analysis (ICP-OES & ICP-MS) (g/100g) ($U_{95\%}$)	48.0 (4.3)	51.0 (4.5)	52.6 (5.5)	48.8 (3.4)	13.6 (2.0)
NaCl by Titration (g/100g) ($U_{95\%}$)	48.3 (0.5)	48.0 (0.8)	48.6 (0.6)	58.9 (0.7)	31.5 (0.5)

$U_{95\%}$ = Expanded Uncertainty at 95% confidence level

As expected, the reference value for total sodium and total chloride in each sample varied depending on the quantity of MSG and KCl in the recipe of the bouillon. The variations in the total sodium and chloride reflects the real amount of sodium contributed by the MSG and chloride contributed by the KCl. This is evident from the increase in the total sodium with the addition of MSG as seen with Samples 2 and 3 (*Figure 1 and Table 2*), while it remained constant in Sample 4 in which KCl was added. Similarly, the total chloride remained the same in Samples 2 and 3 when MSG was added then increased in Samples 4 when KCl was added (*Figure 1 and Table 2*).

The NaCl content calculated from the reference total sodium (*NaCl = total sodium x 2.5*) and total chloride values (*NaCl = total chloride x 1.65*) as in Table 2, showed that the NaCl content based on total sodium was different from the NaCl content that was based on total chloride. These values varied depending on the amount of MSG, which increased the total sodium and KCl, which increased the total chloride. The calculated NaCl content differed most significantly in the samples in which either MSG or KCl was added. The calculated NaCl contents from total sodium were similar to the NaCl reference values for Samples 1, 4 and 5 (*no MSG*) while the NaCl content calculated from the total chloride were similar to the NaCl reference values of Samples 1, 2 and 3 (*no KCl*).

The experimental analytical results for total sodium obtained from the elemental analytical methods and argentometric titration compared to the reference value for total sodium are shown in Figure 2.

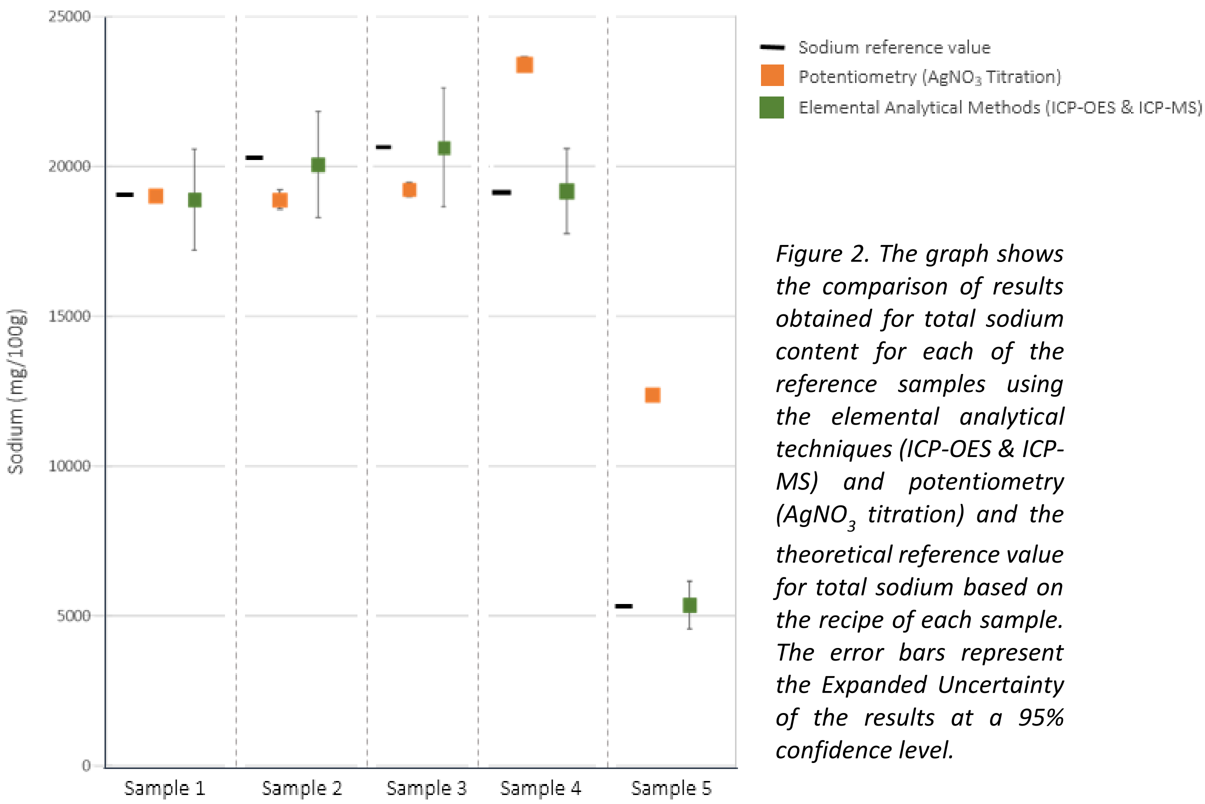

Figure 2. The graph shows the comparison of results obtained for total sodium content for each of the reference samples using the elemental analytical techniques (ICP-OES & ICP-MS) and potentiometry ($AgNO_3$ titration) and the theoretical reference value for total sodium based on the recipe of each sample. The error bars represent the Expanded Uncertainty of the results at a 95% confidence level.

Elemental analytical methods produced results similar to the reference value for total sodium content in all samples.

Analysis by the elemental analytical methods produced results similar to the reference value for total sodium content in all samples while the analysis by the argentometric titration produced results that were similar to the total sodium reference value in Sample 1 only. This was expected because the argentometric titration method measures chloride, which is then expressed as sodium based on the assumption that sodium chloride is the only source of sodium in the sample as in the case of

> *Argentometric titration showed a negative systematic bias when other sources of sodium were present, and a positive systematic bias when other sources of chloride were present.*

Sample 1. Overall argentometric titration showed a negative systematic bias when other sources of sodium were present, and a positive systematic bias when other sources of chloride were present.

The experimental results for the NaCl content obtained from the elemental analytical methods and potentiometry (*argentometric titration*) compared to the reference NaCl content is shown in Figure 3.

The results for NaCl content obtained by the elemental analytical methods were similar to the NaCl reference value for Samples 1, 4 and 5, where NaCl was the only source of sodium in the recipe. Conversely, the mean salt content for Samples 2 and 3 were higher than the NaCl reference value, showing a positive systematic bias. This was expected because the NaCl content in this case was expressed as based on total sodium, therefore any increase in the total sodium resulted in the exaggeration of the NaCl content. This is an example where expression of NaCl content based on total sodium results in the over-estimation of the NaCl content.

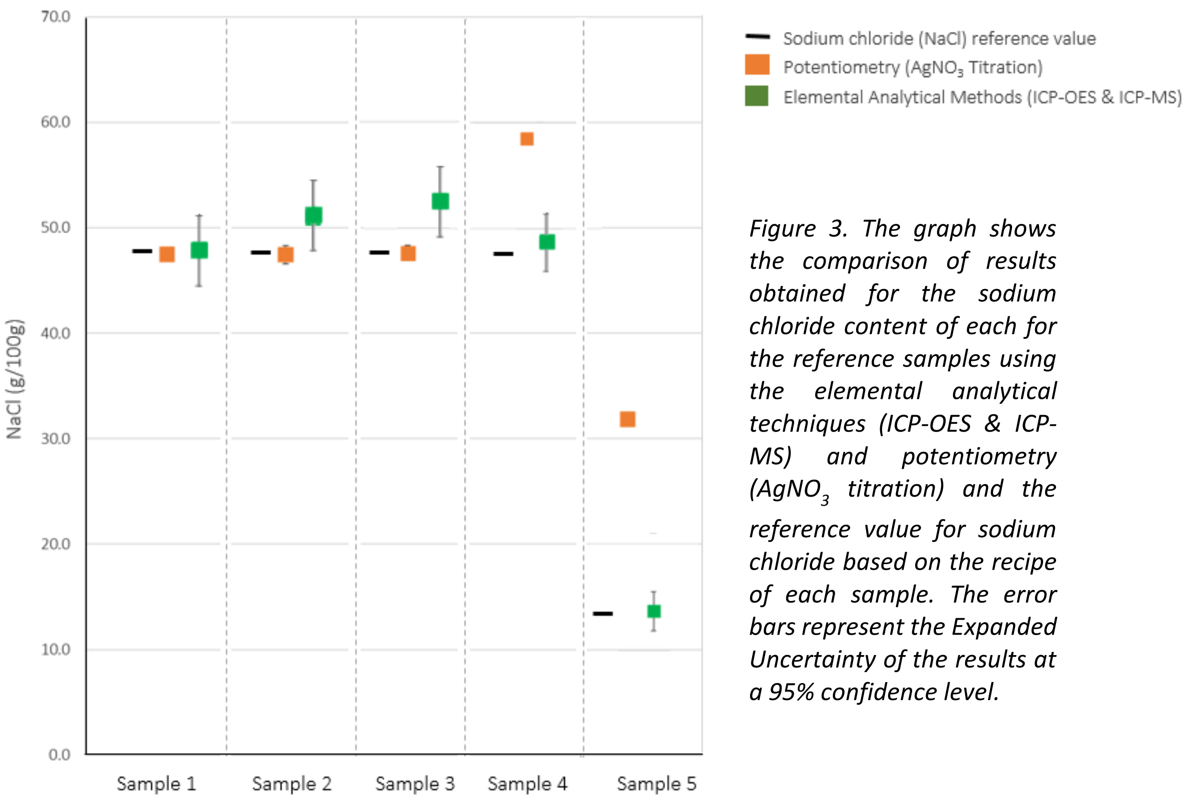

Figure 3. The graph shows the comparison of results obtained for the sodium chloride content of each for the reference samples using the elemental analytical techniques (ICP-OES & ICP-MS) and potentiometry ($AgNO_3$ titration) and the reference value for sodium chloride based on the recipe of each sample. The error bars represent the Expanded Uncertainty of the results at a 95% confidence level.

The experimental results for NaCl content obtained from the argentometric titration compared to the NaCl reference values were similar to the NaCl reference values for Samples 1, 2 and 3. Conversely, the results for the NaCl content was significantly higher than the NaCl reference value for Samples 4 and 5 which contained different levels of KCl. This was expected because this method quantifies chloride and expresses the NaCl content based on chloride, therefore any increase in the total chloride resulted in the apparent increase in the NaCl content of the samples.

Conclusion

> *Elemental analytical techniques such as ICP-OES and ICP-MS are most suitable for the quantification of total sodium in foods.*

In this White Paper, we have reviewed how the determination of sodium and salt could be significantly inaccurate depending on the analytical technique and method used as well as the composition of the product.

Based on the results from this multi-lab testing study, we can conclude that elemental analytical techniques such as ICP-OES and ICP-MS are most suitable for the quantification of total sodium in foods such as culinary bouillon. However, as always, care must be taken to ensure that validated methods are used, and the analysis must be performed correctly in a suitable laboratory environment to ensure reliability of the results.

We also conclude that the expression of salt content based on total chloride in the food matrix can result in significant under-estimation if the food contains other sources of sodium and over-estimation if the food contains other sources of chloride. Both elemental and potentiometric methods can be used to assess compliance with the food standards and nutritional labelling regulations for salt content verification. The choice of which method to apply, however, must be guided by the ingredients used in the recipe of the food product and the specifications in the relevant regulations.

Recommendation

> *It is important to take into consideration the performance and capability of analytical methods when assessing compliance with regulations.*

> *In order to verify salt reduction in food products, the focus should be on the determination of the total sodium content rather than the NaCl content.*

It is important to take into consideration the performance and capability of analytical methods when assessing compliance with regulations. It is also important to consider the evolution of our knowledge on the subject matter, the development of new ingredients and their applications, as well as the advancements in analytical science and technology. These factors may sometimes warrant review and updating of regulations and standards in order to make them more relevant to achieve their purpose.

The results obtained from our experiments confirm that the total sodium content of culinary bouillon with varied ingredient composition and non-NaCl sources of sodium and chloride can only be accurately and reliably quantified using elemental analytical methods. Neither the elemental analytical methods nor the potentiometric method will detect the exact amount of salt (*NaCl*) in foods as both methods will detect sodium and chloride respectively, from all sources. However, as sodium is one of the risk factors associated with non-communicable disease, we recommend that in order to verify salt reduction in food products, the focus should be on the determination of the total sodium content rather than the NaCl content.

The analytical methods that are recommended for the quantification of sodium and salt in foods must be fit-for-purpose. The regulations should also clearly define the expected performance criteria and the capability of the analytical methods to be used to quantify sodium and salt in foods in order to ensure regulatory compliance.

Official analytical methods that are recommended for the quantification of total sodium in various foods are shown in Table 3.

Table 3: International official methods for the determination of total sodium in various food matrices.

Official Analytical Methods for quantification of total sodium in foods	Technique
AOAC 2011.14 (*scope: fortified foods, infant formula, adult nutritionals and milk and milk products*) Method is equivalent to: ISO 15151 | IDF 229	ICP-OES
EN-16943	ICP-OES
EN-15505	FAAS
AOAC 2015.06 (*scope: infant formula and adult nutritionals*) Method is equivalent to: ISO 21424 | IDF 243	ICP-MS
ISO 8070 | IDF 119 (*scope: special foods, infant formula*)	FAAS
AOAC 990.23 (*scope: dried milk*)	FAAS

Provisions should be focused on sodium in line with the WHO recommendations, which focus on the reduction of sodium in food.

Based on our findings, we advise that Codex reconsider the recommendation to use potentiometric method for the determination of salt content (*as stipulated in Codex CXS-234 and CXS 117-1981*). We recommend that provisions should be focused on sodium in line with the WHO recommendations, which focus on the reduction of sodium in food. Consequently, elemental analytical methods should be considered to verify these provisions.

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