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# Flame photometric determination of salinity in processed foods

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## Abstract

The sodium contents determined by flame photometry were used to estimate the salinities of processed foods. The interference effects of potassium and calcium ions on the determination of sodium ion were studied. The threshold interfering patterns of potassium ion and calcium ion for the determination of sodium ion were observed in the model systems. Both of the ions showed enhancement effects on the signals of sodium determination and the increased signal strength depended on the concentration of the ions added above a threshold level. While low level calcium ion hardly interferes with the measurement of sodium ion, the potassium ion exerts marked interfering effects. The salinities based on the chloride contents from Mohr's titration method were obtained for comparison purpose. The salinities of 48 processed food samples determined by the two methods were positively correlated with  $r^2 = 0.9995$  and did not show significant difference in the paired  $t$  tests ( $P = 0.2504$ ). Overall, the flame photometric determination of sodium ion provides a rapid, less tedious and more accurate alternative for the quantification of salinity in processed foods. © 2004 Published by Elsevier Ltd.

**Keywords:** Salinity; Sodium ion; Flame photometry; Interference effects

## 1. Introduction

Sodium chloride is the most important salt in human diet. In current theory, the dietary intake of sodium chloride is closely related to the development of hypertension and other cardiovascular diseases (Robinson, Lawler, Chenoweth, & Garwick, 1989). It has also been found that the sodium ion rather than chloride ion is responsible for such adverse effects (Herlitz, Dahlof, Jonsson, & Friberg, 1998). For the purpose of promoting flavor and increasing shelf life, sodium chloride is an essential ingredient in all processed foods. Thus, the salt contents present in foods are of great concerns for food processing and health care.

Mohr's titration method is commonly accepted as one of the methods for the determination of salinity in foods. In Mohr's method, the salinity of food is calculated based on the concentration of chloride ion titrated with silver nitrate solution. However, disadvantages of using Mohr's method include the de-coloration pretreatment required for foods with deep color, the adjustment to nearly neutral condition before titration (Skoog, West, Holler, & Crouch, 2000), and possible side reactions of silver ion with other anions such as carbonate or phosphate. Thus, the determination of salinity based on the direct determination of sodium ion might provide possible alternatives to overcome these disadvantages.

Atomic absorption spectrophotometry (Orzáez Villanneva, Díaz Marquina, Arribas de Diego, & Blázquez Abellán, 2000; Vaessen & van de Kamp, 1989), inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Fabec & Ruschak, 1985; Fingerová & Koplík, 1999), inductively coupled plasma-mass

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spectrometry (ICP-MS) (Fernández-Turiel et al., 2000; Fingerová & Koplík, 1999), ion selective electrode method (Balulescu, 1985; Fulton, Meloan, Wichman, & Fry, 1984) and flame photometry (Folarin, Bamiro, & Esuoso, 2001; Leenheer & Jans, 1986) are methods for direct detection of sodium ion concentration. Among them, flame photometry is a simple and relatively inexpensive method. Flame photometric technique was introduced around 1950s and many efforts on improving instrumentations or procedures were made to reduce interference and increase sensitivity (Dubbs, 1952; Porter & Wyld, 1955; Schrenk & Gendening, 1955; Spector, 1955). Applications of this method in determination of sodium ion concentrations in a wide variety of samples have been reported (Biffen, 1950; Havre, 1961; Inman, Rogers, & Fournier, 1951; Kingsley & Schaffert, 1953). Although the interference effects depend on the types of the samples (Havre, 1961), the method has been used for quantification of sodium ion in many food samples (Balulescu, 1985; Folarin et al., 2001; Fulton et al., 1984) without taking the interference of other food components into consideration.

For the sake of convenience or dietary habit, people around the world consume large quantity of processed foods in their daily life. The purpose of this investigation is to evaluate the feasibility of flame photometry in the determination of food salinity and the interference effects of potassium and calcium on the determination of salinity in processed foods.

## 2. Experimental section

### 2.1. Flame photometry

Flame photometry was conducted according to Helrich (1990). A simple flame photometer (Model 410, Corning, Halstead, UK) with filters for lithium, sodium, and potassium was used. Liquefied petroleum gas (China Petroleum Company, Taipei, Taiwan) and air were supplied as the source of flame. The flow rate of fuel was adjusted to get a maximum sensitivity. Standard curve with sodium concentration between 5 and 30 ppm was established daily and the signal of 30 ppm standard was checked occasionally during the analysis. The sodium content in appropriately diluted food sample was determined against the standard curve and the salinity (content of sodium chloride on weight basis) of the original food sample was calculated. Means with standard deviations of triplicate determinations were reported.

### 2.2. Mohr's titration

Mohr's titration was carried out according to Skoog et al. (2000) and James (1995). Silver nitrate, sodium

chloride, potassium chromate and sodium bicarbonate were purchased from Hayashi Chemical Ltd. (Osaka, Japan). Silver nitrate solution (0.1 M) was standardized with pure sodium chloride. Sodium bicarbonate powder was used to adjust pH of testing solution to nearly neutral if necessary. Potassium chromate (5%, 1 mL) was used as indicator during silver nitrate titration and the appearance of brick red precipitates was taken as the end point. Double distilled water served as the blanks was also included. The salinity of the food sample was calculated according to the amount of titrated chloride ion. Means with standard deviations of triplicate determinations were reported.

### 2.3. Sample preparation

Forty-eight food samples (Table 1) were chosen to analyze the salt content in this study. The selection criteria were based on the consumer preference, the representative for plant-origin and animal-origin foods, and different salt contents. The commercial package sizes for these food samples were 0.25–1.0 kg. All samples were purchased from local supermarkets and stored in a refrigerator (4 °C) until used. In the case when packing liquid and solid matter were packed together, the whole content was homogenized (Waring blender; Dynamics Co., New Hartford, CT, USA). A portion of slurry (10 g) was sampled and mixed with double distilled water (40 mL) and centrifuged (15,000g, 4 °C, 10 min; Centrifuge, Hitachi, Japan); and the supernatant was diluted and used in the analysis. For paste type foods, portion of sample (10 g) and double distilled water (40 mL) were homogenized, centrifuged and diluted as mentioned above. For Mohr's titration, deep-colored liquid samples (e.g. soy sauce) were de-colored by stirring with activated carbon (1 g activated carbon and 40 mL double distilled water per 10 g soy sauce) under boiling for 10 min, then filtered to remove active carbon and diluted. All the samples for metal ion determination were stored in polyethylene bottles that were cleaned, bathed in 5% HNO<sub>3</sub> overnight, and rinsed twice with double distilled water.

### 2.4. The influence of potassium and calcium ions on flame photometric determination

Potassium ion (0–5 ppm) and calcium ion (0–100 ppm) were separately added into standard sodium ion solutions (1, 10, and 20 ppm) to evaluate the interference effects. The relative error of the sodium contents due to the presence of potassium and calcium ions was determined. To investigate the interference effects on food samples, a predetermined amount of potassium or calcium ion was added into diluted food sample prior to flame photometric analysis.

Table 1  
The list of salty processed foods used in this study

Sample no.	Sample description
1	Pickled cabbage core (A)
2	Pickled cabbage core (B)
3	Pickled cabbage core (C)
4 <sup>a</sup>	Canned gluten (A)
5 <sup>a</sup>	Canned gluten (B)
6	Pickled cucumber (A)
7	Pickled cucumber (B)
8	Pickled cucumber (C)
9	Soft pickled cucumber (A)
10	Soft pickled cucumber (B)
11	Soft pickled cucumber (C)
12	Pickled black cucumber
13	Hou cucumber
14 <sup>a</sup>	Pickled bitter gourd (A)
15 <sup>a</sup>	Pickled bitter gourd (B)
16	Canned mushroom (A)
17	Canned mushroom (B)
18	Pickled onion
19 <sup>a</sup>	Canned peanut
20 <sup>a</sup>	Canned peas
21 <sup>a</sup>	Pickled pineapple (A)
22 <sup>a</sup>	Pickled pineapple (B)
23 <sup>a</sup>	Fermented black bean (A)
24 <sup>a</sup>	Fermented black bean (B)
25 <sup>a</sup>	Fermented bean curd
26	Soy sauce (A)
27	Soy sauce (B)
28 <sup>a</sup>	Spaghetti sauce
29	Vinegar
30 <sup>a</sup>	Chili sauce
31	Sweet osmanthus syrup (A)
32	Sweet osmanthus syrup (B)
33 <sup>a</sup>	Canned corn paste
34	Canned corn sprout (A)
35	Canned corn sprout (B)
36	Canned snail (A)
37	Canned snail (B)
38	Canned snail (C)
39	Pickled quail egg
40	Sausage (A)
41	Sausage (B)
42	Soup concentrate (A)
43	Soup concentrate (B)
44	Ox tail soup
46	Lobster soup
46 <sup>a</sup>	Dried scallop
47 <sup>a</sup>	Canned tuna
48 <sup>a</sup>	Shrimp paste

<sup>a</sup> Sample without packing liquid.

### 2.5. Statistic analysis

For each food sample, the salinities calculated from flame photometry and Mohr's titration method were statistically compared (Mason, Gunst, & Hess, 1989; Bartz, 1999). The interference effects in food sample were also studied statistically by comparing the calculated salinities with those added extra potassium or calcium ions. The linear regression data and the probability of paired *t* tests were calculated with SAS software (6th

edition, 1995; SAS Institute Inc., Cary, NC, USA) on a personal computer.

## 3. Results and discussion

### 3.1. Correlation of Mohr's titration and flame photometry in food salinity

The 48 food samples used in this study are listed in Table 1. The salinities of food samples determined by Mohr's titration and flame photometry are listed in Table 2; and the resulting statistics are listed in Table 3. Since the squared correlation coefficient of two methods was as high as 0.9995, the salinities determined by flame photometry and Mohr's were positively correlated. The probability of paired *t* test was 0.2504 (greater than 0.05), which indicated that the salinities determined by the two methods were not significantly different. However, no necessity of de-coloration, less tedious procedure and direct determination of sodium ion might make flame photometric analysis a better choice than Mohr's titration for the determination of salinity in processed foods.

### 3.2. The interference of potassium and calcium on sodium quantification in model system

The presence of the coexisting metal ions in the sample matrix might be the major interference in quantification of sodium content with flame photometry. Since potassium and calcium ions, in addition to sodium ion, are the other two major metal ions existing in food system in relatively large amounts (Reed, 1980), their effects on sodium ion quantification were studied. In preliminary test, a better linearity was obtained when the concentration of sodium ion is below 30 ppm. Therefore, the standard solutions containing 1, 10 and 20 ppm sodium ion and these solutions with extra potassium and calcium ions were tested. Their interference effects on sodium ion quantification are shown in Fig. 1. A threshold behavior of the interference resulting from extra potassium and calcium ions was observed. The sodium ion signal held constant for a certain range of extra metal ion concentration until exceeding a threshold. This phenomenon was similar to the results reported by Havre (1961). The enhancement of the signal increases with the final concentration of added potassium and calcium ions. For solutions containing 10 and 20 ppm sodium ion, extra potassium ion at the levels of 2 and 3 ppm raised the relative error over 5%. For solution containing 1 ppm sodium, 0.5 ppm extra potassium ion resulted in the quantification error as high as 20%. In contrast to potassium ion, for sodium ion at a level of 10 and 20 ppm, the addition of calcium ion up to 100 ppm

Table 2  
The food salinity (g NaCl/100 g sample) determined by Mohr's titration and flame photometry

Sample no.	A <sup>b</sup>	B <sup>b</sup>	C <sup>b</sup>	D <sup>b</sup>	E <sup>b</sup>	F <sup>b</sup>	Dilution factor (mL/g)
1	4.15(0.28) <sup>a</sup>	4.16(0.06)	4.16(0.03)	5.67(0.02)	4.23(0.02)	4.30(0.03)	995
2	3.54(0.05)	3.68(0.06)	3.69(0.02)	5.42(0.02)	3.60(0.02)	3.86(0.03)	1233
3	4.13(0.02)	4.23(0.00)	4.23(0.00)	5.62(0.04)	4.23(0.00)	4.33(0.00)	985
4	2.39(0.06)	2.47(0.01)	2.46(0.02)	3.22(0.00)	2.45(0.01)	2.45(0.03)	499
5	2.51(0.04)	2.56(0.01)	2.55(0.01)	3.34(0.00)	2.54(0.02)	2.54(0.02)	858
6	4.79(0.32)	4.77(0.02)	4.77(0.01)	6.38(0.04)	4.78(0.02)	4.78(0.00)	1208
7	4.34(0.26)	4.33(0.03)	4.32(0.02)	5.54(0.02)	4.29(0.02)	4.30(0.03)	981
8	4.43(0.02)	4.54(0.01)	4.54(0.00)	4.67(0.03)	4.62(0.03)	4.69(0.00)	2003
9	7.89(0.49)	7.70(0.05)	7.75(0.02)	10.21(0.00)	7.81(0.02)	7.95(0.03)	1574
10	9.12(0.27)	9.31(0.04)	9.21(0.03)	13.04(0.00)	9.18(0.04)	9.26(0.04)	3234
11	7.24(0.06)	7.44(0.06)	7.44(0.06)	7.85(0.06)	7.58(0.06)	7.82(0.00)	2083
12	2.57(0.02)	2.58(0.02)	2.59(0.00)	2.76(0.02)	2.69(0.02)	2.77(0.00)	1248
13	1.50(0.13)	1.50(0.00)	1.50(0.00)	2.12(0.02)	1.49(0.01)	1.51(0.01)	314
14	5.97(0.26)	5.78(0.02)	5.86(0.00)	7.09(0.02)	6.03(0.02)	6.43(0.01)	1779
15	3.13(0.02)	2.90(0.00)	2.90(0.02)	3.26(0.02)	3.02(0.00)	3.06(0.00)	990
16	1.25(0.01)	1.19(0.00)	1.19(0.00)	1.23(0.00)	1.20(0.00)	1.22(0.01)	416
17	0.86(0.00)	0.89(0.00)	0.90(0.01)	1.03(0.00)	0.90(0.00)	0.91(0.00)	249
18	1.52(0.00)	1.41(0.01)	1.41(0.00)	2.19(0.01)	1.42(0.02)	1.51(0.01)	495
19	9.57(0.09)	9.79(0.00)	9.79(0.02)	13.12(0.03)	9.92(0.03)	9.90(0.06)	1586
20	0.93(0.00)	0.87(0.00)	0.87(0.00)	0.91(0.01)	0.87(0.00)	0.89(0.02)	249
21	3.79(0.04)	3.83(0.02)	3.84(0.02)	5.77(0.02)	3.93(0.01)	4.17(0.02)	1248
22	5.59(0.02)	5.62(0.00)	5.78(0.04)	5.86(0.00)	5.74(0.00)	5.80(0.00)	2506
23	17.27(0.07)	16.74(0.07)	16.78(0.05)	23.61(0.08)	16.84(0.04)	17.21(0.07)	4950
24	12.29(0.03)	12.65(0.10)	12.69(0.07)	18.01(0.07)	12.32(0.05)	12.93(0.12)	4142
25	14.46(0.10)	14.00(0.06)	14.05(0.00)	17.28(0.02)	13.96(0.03)	14.22(0.04)	3966
26	13.12(0.19)	12.96(0.04)	13.00(0.03)	17.76(0.05)	13.06(0.02)	13.76(0.04)	2511
27	14.05(0.00)	13.34(0.00)	13.58(0.00)	13.82(0.00)	13.58(0.00)	14.15(0.13)	2489
28	1.77(0.08)	1.91(0.00)	1.90(0.00)	2.53(0.02)	1.91(0.01)	1.95(0.02)	478
29	5.73(0.05)	5.66(0.00)	5.66(0.00)	5.83(0.08)	5.71(0.04)	5.78(0.00)	2497
30	12.21(0.44)	12.04(0.04)	12.13(0.00)	13.34(0.02)	12.10(0.04)	12.20(0.03)	2341
31	3.87(0.10)	3.81(0.00)	3.84(0.00)	4.30(0.01)	3.82(0.01)	3.95(0.00)	893
32	4.03(0.04)	3.93(0.00)	3.93(0.00)	4.06(0.06)	3.95(0.00)	4.01(0.00)	1791
33	0.66(0.17)	0.70(0.00)	0.69(0.00)	0.75(0.00)	0.68(0.00)	0.70(0.02)	195
34	3.63(0.14)	3.47(0.01)	3.53(0.02)	3.88(0.00)	3.53(0.01)	3.64(0.03)	588
35	0.76(0.02)	0.72(0.00)	0.72(0.00)	0.76(0.00)	0.73(0.01)	0.73(0.00)	312
36	2.47(0.13)	2.42(0.01)	2.42(0.00)	3.21(0.01)	2.40(0.01)	2.46(0.02)	497
37	2.58(0.06)	2.79(0.01)	2.79(0.02)	3.76(0.04)	2.74(0.00)	2.93(0.01)	500
38	2.21(0.01)	2.14(0.00)	2.14(0.01)	2.29(0.01)	2.16(0.00)	2.24(0.00)	998
39	0.82(0.01)	0.78(0.00)	0.78(0.01)	1.14(0.00)	0.78(0.00)	0.82(0.01)	250
40	2.81(0.02)	2.86(0.00)	2.86(0.00)	2.95(0.00)	2.89(0.00)	2.95(0.00)	1250
41	2.53(0.09)	2.64(0.01)	2.66(0.00)	3.51(0.02)	2.64(0.01)	2.68(0.00)	497
42	1.04(0.01)	1.09(0.01)	1.09(0.00)	1.47(0.00)	1.10(0.01)	1.11(0.00)	250
43	0.85(0.01)	0.88(0.00)	0.88(0.00)	0.91(0.00)	0.88(0.00)	0.91(0.00)	500
44	2.71(0.02)	2.59(0.00)	2.59(0.00)	2.66(0.02)	2.59(0.00)	2.65(0.02)	714
45	0.80(0.02)	0.88(0.00)	0.86(0.00)	1.21(0.01)	0.88(0.00)	0.93(0.01)	239
46	1.37(0.09)	1.53(0.01)	1.54(0.00)	1.86(0.02)	1.47(0.01)	1.55(0.01)	311
47	0.74(0.02)	0.74(0.00)	0.74(0.00)	0.99(0.01)	0.74(0.00)	0.75(0.02)	250
48	26.64(0.50)	26.23(0.00)	26.05(0.09)	32.87(0.02)	26.05(0.09)	26.25(0.05)	4907

<sup>a</sup> Standard deviations of triplicate determination were included in parentheses.

<sup>b</sup> A, salinity without extra salt by Mohr's titration; B, salinity without extra salt by flame photometry; C, salinity with additional 0.1 ppm K by flame photometry; D, salinity with additional 1 ppm K by flame photometry; E, salinity with additional 20 ppm Ca by flame photometry; F, salinity with additional 100 ppm Ca by flame photometry.

caused only about 3% relative error in quantification. For the sodium ion levels tested, quantification errors raised by addition of 20 ppm calcium ion were all below 1%. It was much smaller than that reported by Spector (1955) and Havre (1961). Since the potassium has lower ionization energy than sodium (Foster & Hume, 1959), potassium atoms are more easily ionized

than sodium atoms and will provide electrons to repress the ionization of sodium atoms in flame. Higher unionized sodium will exert stronger signal of sodium atom in atomic emission analysis. Because calcium has higher ionization energy than sodium, calcium will not repress the ionization of sodium atoms and will not show pronounced effect on sodium quantification.

Table 3  
The statistics of salinities determined

Paired <i>t</i> test sample <sup>a</sup>	Linear regression data			Probability of <i>t</i> test
	Slope <sup>b</sup>	Intercept <sup>b</sup>	<i>r</i> <sup>2</sup>	
A vs. B	0.980(0.005)	0.07(0.03)	0.9995	0.2504
B vs. C	1.000(0.002)	0.01(0.01)	0.9999	0.1304
B vs. D	1.277(0.026)	−0.14(0.19)	0.9906	0.0001
B vs. E	0.999(0.003)	0.03(0.02)	0.9998	0.1406
B vs. F	1.018(0.005)	0.05(0.04)	0.9995	0.0001

<sup>a</sup> A, salinity without extra salt by Mohr's titration; B, salinity without extra salt by flame photometry; C, salinity with additional 0.1 ppm K by flame photometry; D, salinity with additional 1 ppm K by flame photometry; E, salinity with additional 20 ppm Ca by flame photometry; F, salinity with additional 100 ppm Ca by flame photometry.

<sup>b</sup> Standard deviations of slope and intercept were included in parentheses.

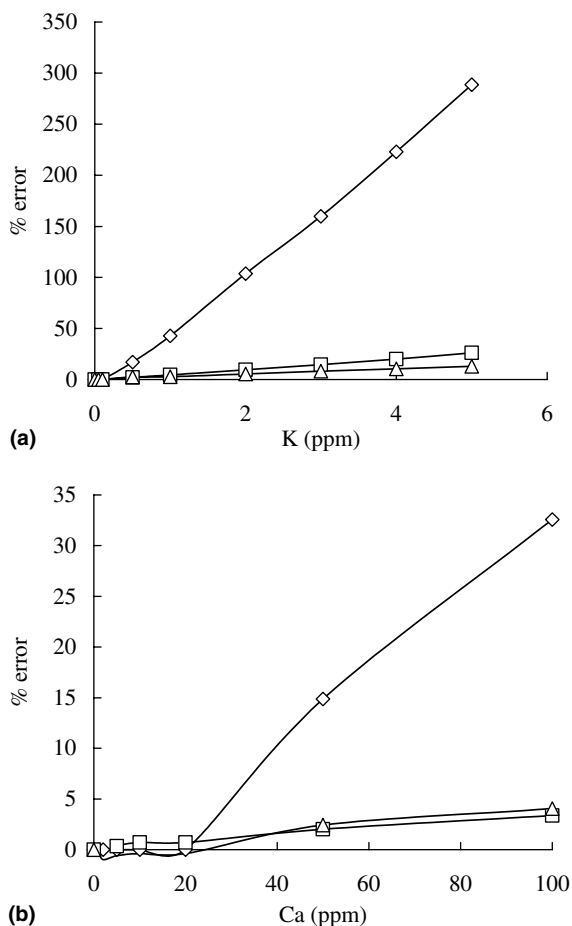


Fig. 1. The relative error (%) of the measured sodium ion concentration caused by the addition of potassium ion (a) and calcium ion (b). Mean of triplicate samples.  $\diamond$ , aqueous solution with 1 ppm sodium ion;  $\square$ , aqueous solution with 10 ppm sodium ion;  $\triangle$ , aqueous solution with 20 ppm sodium ion.

### 3.3. The interference of potassium and calcium on sodium quantification in food matrix

Based on the relationship between the interference effects and the sodium ion concentration studied above, the food sample was diluted to get a signal within the

range of 5–20 ppm sodium ion standard solutions detected with Corning 410 flame photometer. Furthermore, two levels of extra calcium and potassium were added into the food sample to test their interference effects in food matrix.

The flame-photometrically determined salinities of food samples added with extra potassium and calcium ions were listed in Table 2, and the statistics (squared correlation coefficient and probability) of comparing each type of sample to control (without extra potassium or calcium) were listed in Table 3. While 0.1 ppm extra potassium did not interfere the determination of food salinity, 1 ppm extra potassium ion resulted in higher salinity in testing sample. Since the extra potassium ion was added into the diluted sample prior to analyzing with flame photometer to obtain the exact amount of addition, this 0.1 ppm extra potassium ion represented as high as 50 mg of potassium per 100 g of the original food sample (calculated according to the dilution factor listed in Table 2). It is well known that metal ions are easily lost during food processing (such as washing, cutting, and blanching operations) but usually only sodium chloride is added back into foods as processing additive. Therefore, the high potassium contents in foods would show less interference effect in the determination of sodium in processed foods. For examples, major ingredient for samples 16–17 is mushroom, the potassium content in fresh mushroom is at the level of 520 mg/100 g (Krause, 1966). Determination of sodium is not affected by potassium contents in these food samples. This non-significant interference of preexisting potassium ions might be explained by the fact that the dilution of high salty food sample to meet the flame photometry operation requirement causes the final potassium concentration to fall below the interfering threshold. As for calcium ion, the calculated salinity of foods did not show any significant difference when as high as 20 ppm extra calcium ion (representing 10 g calcium per 100 g of original food samples) was added into the testing samples. The results indicate that flame photometry can be applied for salinity determination of high-calcium fortified foods.

The detection limit (DL) of sodium was calculated according to following equation:

$$DL = S_{\text{reag}} + t s_{\text{reag}},$$

where  $S_{\text{reag}}$  is signal of reagent blank,  $t$  is a factor accounting for the 95% confidence level, and  $s_{\text{reag}}$  is standard deviation of reagent blank. The calculated DL in this study was 0.14 ppm. The standard solution of 10 ppm sodium was used for the determination of repeatability; the mean value was 10.06 ppm with a standard deviation of 0.08 ppm ( $n = 20$ ), corresponding to a precision of 0.79%. In conclusion, the flame photometric determination of sodium provides a rapid, less tedious and more accurate alternative for the determination of salinity in processed foods except for foods with extremely high potassium contents (greater than 520 mg potassium per 100 g food) which are rarely found in commercial foods.

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