

INTERNATIONAL UNION OF PURE
AND APPLIED CHEMISTRY

APPLIED CHEMISTRY DIVISION
COMMISSION ON FOOD CHEMISTRY*

A Collaborative Study and Description of the Method

**SODIUM AND POTASSIUM ASSAY OF
FOODS AND BIOLOGICAL SUBSTRATES
BY ATOMIC ABSORPTION
SPECTROSCOPY (AAS)**

Prepared for publication by

H. A. M. G. VAESSEN and C. G. van de KAMP

Laboratory for Residue Analysis, National Institute of Public Health and
Environmental Protection, POB 1, 3720 BA Bilthoven, The Netherlands

*Membership of the Commission during the preparation of the report (1985–87) was as follows:

Chairman: A. E. Pohland (USA); *Vice-Chairman:* P. S. Steyn (Republic of South Africa);
Secretary: D. L. Park (USA); *Titular Members:* R. Battaglia (Switzerland); P. Krogh (Denmark);
H. A. M. G. Vaessen (Netherlands); *Associate Members:* M. J.-J. Castegnaro (France); H. B. S.
Conacher (Canada); R. L. Ellis (USA); J. Fremy (France); J. Jacob (FRG); M. Jemmali
(France); S. J. Kubacki (Poland); R. Livingston (USA); P. G. Thiel (Republic of South Africa);
Y. Ueno (Japan); T. Yasumoto (Japan); *National Representatives:* M. M. Abdel Kader (Arab
Republic of Egypt); A. Calvelo (Argentina); P. B. Czedik-Eysenberg (Austria); J. Davidek
(Czechoslovakia); E. Lück (FRG); R. Lásztity (Hungary); Z. Berk (Israel); S. S. A. Marina
Miraglia (Italy); T. Kato (Japan); C. L. Lim (Malaysia); A. Rutkowski (Poland); L. E. Coles (UK).

Republication of this report is permitted without the need for formal IUPAC permission on condition that an acknowledgement, with full reference together with IUPAC copyright symbol (© 1989 IUPAC), is printed. Publication of a translation into another language is subject to the additional condition of prior approval from the relevant IUPAC National Adhering Organization.

Sodium and potassium assay of foods and biological substrates by atomic absorption spectroscopy (AAS)

Abstract - An atomic absorption spectrometric (AAS) method is described for the single run determination of sodium and potassium in a wide variety of foods and biological substrates. Accuracy and precision of the method were established by repetitive analyses of eight standard reference materials. Additional performance characteristics i.e. repeatability and reproducibility, were established in a collaborative trial involving 10 laboratories and 5 samples. For both analytes the method is adequately precise and accurate. Repeatability value r and reproducibility value R for sodium and potassium are a linear function of their respective concentration in the samples. The suitability of the procedure is demonstrated for a wide range of biological substrates covering levels of 0.01 to 0.5 percent by mass for sodium and of 0.1 to 2 percent by mass for potassium.

Many analytical procedures are described in the literature for the determination of sodium and potassium in foods and biological materials. This also holds for the handbooks of food analysis including the authoritative "Official Methods of Analysis of the Association of Official Analytical Chemists (1984)" in which for both elements gravimetric, volumetric, ion selective, and various spectrometric procedures are described. As well as the measuring techniques, a variety of sample preparation procedures are recommended for bringing the analytes into an appropriate test solution. In the case of certain types of liquids this is achieved by simple dilution but, more generally, wet digestion or dry ashing is required.

Keeping a multitude of techniques operational at the required Good Laboratory Practice (GLP) level not only requires a considerable effort of control laboratories but also blocks valuable analytical capacity. It thus is much more practical to have a single method covering both a wide range of products and of sodium and potassium levels. However, there is another important reason to advocate the development of such a method. Recently Parr (1980) published details on analytical problems revealed by results for biological reference materials assayed in a number of cooperative trials. This data provide insight on the status of elemental analysis as practised by typical analytical laboratories. Results reported were judged significantly in error whenever the reported mean values differed from the relevant certified values by more than + 20%. According to this measure of reliability 7% of this laboratories reported inaccurate data for potassium in Bowen's Kale and IAEA*H-4 Animal Muscle. For sodium these figures were 19% for Bowen's Kale and 21% for Animal Muscle.

Recently we developed an atomic absorption spectrometric (AAS) method for sodium and potassium which is simple, fast and precise and overcomes the problems outlined. The performance characteristics and the results of the collaborative trial are given here. The method is described in detail in the Annex.

METHOD STATUS

The method described was applied for a number of reference materials covering roughly the levels of 0.01 to 0.5 percent by mass for sodium and 0.1 to 2 percent by mass for potassium. The results of these analyses are summarized in Table 1 and illustrate the adequate accuracy and precision of the procedure and its suitability for a broad variety of biological materials.

COLLABORATIVE TRIAL

To establish additional performance characteristics of the method a collaborative trial was organized the details and outcome of which are given here.

Design and samples

Early in 1987 thirteen laboratories were sent the following packet of materials and information :

- five plastic vials containing 1 gramme each of the samples A,B,C,D and E,
- approximately 500 mg of bromocresol green,
- a reporting sheet including sample information and handling instructions,
- a detailed description of the analytical procedure in ISO lay-out.

The deadline for submitting results was set at May 15th.

Duplicate analysis were requested for each analyte and sample; in addition blank results were requested.

* Refer to footnote in Table 1

Table 1: Summary of results for reference materials, content on dry weight basis and in percent by mass*

Sample code**	Reference Material	SODIUM		POTASSIUM	
		Certified ²⁾	Found	Certified ²⁾	Found
NBS-SRM-1567	Wheat Flour	$(8.0 \pm 1.5)10^{-4}$	-	0.136 ± 0.004	$0.132 \pm 0.005(3)$
NBS-SRM-1568	Rice Flour	$(6.0 \pm 1.5)10^{-4}$	-	0.112 ± 0.002	$0.113 \pm 0.006(3)$
NBS-SRM-1571	Orchard Leaves	$(82 \pm 6)10^{-4}$	$(80 \pm 5)10^{-4}(10)$	1.47 ± 0.03	$1.45 \pm 0.03(10)$
NIES-CRM 1	Pepperbush	$(106 \pm 13)10^{-4}$	$(94 \pm 4)10^{-4}(10)$	1.51 ± 0.06	$1.51 \pm 0.04(10)$
NBS-SRM-1572	Citrus Leaves	$(160 \pm 20)10^{-4}$	$(170 \pm 6)10^{-4}(10)$	1.82 ± 0.06	$1.75 \pm 0.02(10)$
IAEA-RM-H-4	Animal Muscle	0.206 ± 0.13	$0.224 \pm 0.011(10)$	1.58 ± 0.06	$1.67 \pm 0.03(10)$
NBS-SRM-1577	Bovine Liver ¹⁾	0.243 ± 0.13	$0.240 \pm 0.013(20)$	0.97 ± 0.06	$0.98 \pm 0.03(20)$
IAEA-RM-A-11	Milk Powder	0.442 ± 0.33	$0.454 \pm 0.014(10)$	1.72 ± 0.10	$1.80 \pm 0.03(10)$

1) Values are on freeze-dried product as issued by NBS. 2) Certified or "best" values claimed by issuing organization.

* Mean value and standard deviation; number of replicates in. ** NBS; National Bureau of Standards, Washington DC/USA.

- Not determined; below limit of determination of method.

Samples A and B were made by freeze-drying subsamples of the same total diet lot. However, in the case of sample B the subsample was enriched, prior to freeze-drying, with sodium chloride and colorant to give a coloured and 0.040% sodium enriched duplicate of sample A. Sample C was a commercial coffee creamer; Kale Powder was sample D and the NBS Bovine Liver (Table 1) was included as sample E.

For reasons of objectivity participants were not informed about these details.

Homogeneity of each sample was checked prior to shipment by repetitive analysis (N=5) of its sodium and potassium content. Coefficients of variation for sodium and potassium were better than 5% relative for each sample. Thus, homogeneity is estimated adequate in the context of the collaborative trial.

Mean sodium levels of samples A and B differed 0.0455% by mass which is 114% of the spike.

Results and discussion

Initially 13 laboratories accepted the invitation to participate in this trial. Results were received from 10 participants and 3 laboratories withdrew from the study due to lack of capacity or for technical reasons. All original data submitted are compiled in Table 2 for sodium and in Table 3 for potassium.

The statistical parameters of these data were calculated according to ISO 5725 and Pocklington (1986) and are given in Tables 4 and 5.

Mean sodium and potassium values for Bovine Liver (sample E), NBS reference material No. 1577, agree well with the certified values given in Table 1. Samples A and B are blind duplicates with respect to their potassium content, whereas their sodium content differs theoretically 0.040% by mass. From the set of accepted data for potassium (Table 3) it is obvious that none of the laboratories had major difficulties with these samples. However, for sodium (Table 2) most laboratories failed to identify the small difference in sodium level of samples A and B. Most probably this is due to a combination of lack of experience with the method and the small relative difference of 6.2% in sodium level between the samples. Obviously most measuring systems require scrupulous attention to detail in tuning and operation if small differences in signal at the sodium levels involved are to be determined. Blank figures of both analytes vary considerably between laboratories; Tables 2 and 3. Laboratory 5 seems to have a major problem in this respect for both potassium and to a lesser extent, for sodium. The other blank values are acceptable in the context of the limit of determination of 0.005% of the method. Nonetheless, it should be stressed that a number of blank results clearly demonstrate that it is feasible to substantially reduce the contribution of the blank to the analytical result. In doing so reliability of results at the lower end of the concentration scale will improve.

The repeatability and reproducibility values i.e. r - and R -values in Tables 4 and 5, were plotted against the levels of the analytes in the samples. From these plots it was evident that, for the concentration levels involved, a linear relation exists between the sodium (m_{Na}) and potassium (m_K) levels and the corresponding R - and r -values. The equations which reflect best this linear relation are:

$$\begin{aligned} \text{Sodium} \quad r &= 0.005 + 0.042 m_{Na}; \text{ correlation coefficient } 0.921 \\ R &= 0.024 + 0.078 m_{Na}; \text{ correlation coefficient } 0.912 \end{aligned}$$

$$\begin{aligned} \text{Potassium} \quad r &= -0.027 + 0.092 m_K; \text{ correlation coefficient } 0.995 \\ R &= -0.058 + 0.279 m_K; \text{ correlation coefficient } 0.990 \end{aligned}$$

where m_{Na} and m_K are in percent by mass.

Table 2: Original data for SODIUM; results are from duplicate analyses and in percent by mass of sample as received. Collaborative method of analysis.

Laboratory	SAMPLE TYPE AND CODE					
	Total Diet		Coffee Creamer	Kale Powder	Bovine Liver ⁵⁾	Blank ⁴⁾
	A	B	C	D	E	
1	0.642-0.622	0.657-0.661	0.233-0.231	0.0397-0.0376	0.233-0.226	0.0008-0.0008
2	0.682-0.683	0.683-0.650	0.255-0.253	0.0369-0.0347	0.252-0.251	0.0002-0.0003
3	0.624-0.632	0.659-0.659	0.222-0.245	0.036-0.036	0.208-0.220	<0.001-<0.001
4	0.643-0.647	0.654-0.686	0.259-0.251	0.053-0.056	0.243-0.248	<0.001-<0.001
5	0.654 ¹⁾ -0.338 ¹⁾	0.712-0.710	0.225-0.227	0.034-0.035	0.222-0.218	0.008-0.005
6	0.607-0.594	0.630-0.636	0.233-0.235	0.0498-0.0493	0.225-0.224	0.0047-0.0063 ³⁾
7	0.664-0.693	0.681-0.706	0.251-0.261	0.0381-0.0402	0.241-0.248	0.0005-0.0005
8	0.812 ²⁾ -0.830 ²⁾	0.857 ²⁾ -0.819 ²⁾	0.282-0.305	0.034-0.032	0.260-0.252	0.003-0.002
9	0.616-0.620	0.651-0.646	0.235-0.238	0.0418-0.0436	0.230-0.224	0.0029-0.0029
10	0.655-0.650	0.669-0.674	0.246-0.241	0.0374-0.0410	0.232-0.230	0.001-0.00367 ³⁾

1) Outliers according to Cochran's outlier test 2) Ibid according Dixon's test 3) Range of set of data (N>2)
4) Test portion: 0.3 ml of double distilled water 5) NBS reference material 1577

Table 3: Original data for POTASSIUM results are from duplicate analyses and in percent by mass of sample as received. Collaborative method of analysis.

Laboratory	SAMPLE TYPE AND CODE					
	Total Diet		Coffee Creamer	Kale Powder	Bovine Liver ⁴⁾	Blank ³⁾
	A	B	C	D	E	
1	0.717 ¹⁾ -0.640 ¹⁾	0.656-0.648	1.011-0.981	2.256-2.204	0.920-0.937	0.0032-0.0016
2	0.732-0.738	0.705-0.708	1.08-1.07	2.46-2.55	0.983-0.937	0.003-0.003
3	0.615-0.640	0.620-0.616	0.945-0.995	2.433-2.256	0.874-0.916	<0.001-<0.001
4	0.555-0.573	0.520-0.558	0.851-0.898	2.45-2.47	0.884-0.937	<0.001-<0.001
5	0.756 ¹⁾ -0.378 ¹⁾	0.664-0.650	1.028-1.007	2.044-1.877	0.967-0.949	0.121 ¹⁾ -0.037 ¹⁾
6	0.619-0.612	0.618-0.641	0.978-0.986	2.406-2.377	0.908-0.929	0.0003-0.0013 ²⁾
7	0.689-0.693	0.681-0.706	1.10-1.06	2.50-2.57	1.06-1.05	0.0005-0.0005
8	0.605-0.591	0.594-0.592	0.874-0.951	2.052-1.975	0.868-0.824	0.000-0.000
9	0.674-0.671	0.666-0.660	1.021-1.023	2.537-2.561	0.995-0.999	0.0017-0.0017
10	0.644-0.649	0.634-0.637	1.01-0.999	2.45-2.38	0.956-0.948	0.00167-0.00167

1) Outliers according to Cochran's outlier test 2) Range of set of data (N>2)
3) Test portion: 0.3 ml of double distilled water 4) NBS reference material 1577

Table 4: Statistical data for SODIUM calculated from the original figures in Table 2 by applying ISO 5725 and Pocklington (1986); outliers excluded. Results in % (m/m) unless otherwise indicated.

Code	Sample	Number of accepted results	Mean result	Repeatability			Reproducibility		
				sd _r ¹⁾	CV _r ²⁾	r-value ³⁾	sd _R ²⁾	CV _R ²⁾	R-value
A		16	0.642	0.010	1.55%	0.028	0.029	4.44%	0.081
B		18	0.668	0.013	1.88%	0.036	0.023	3.48%	0.066
C		20	0.246	0.008	4.24%	0.023	0.020	8.00%	0.056
D		20	0.040	0.001	3.69%	0.004	0.007	17.0%	0.019
E		20	0.234	0.004	1.79%	0.012	0.014	6.13%	0.041

1) sd: standard deviation 2) CV: coefficient of variation % relative 3) Confidence level: 95%

Table 5: Statistical data for POTASSIUM calculated from the original figures in Table 3 by applying ISO 5725 and Pocklington (1986); outliers excluded. Results in % (m/m) unless otherwise indicated.

Code	Sample		Repeatability			Reproducibility		
	Number of accepted results	Mean result	sd _r ¹⁾	CV _r ²⁾	r-value ³⁾	sd _R ²⁾	CV _R ²⁾	R-value
A	16	0.643	0.009	1.38%	0.025	0.053	8.32%	0.151
B	20	0.639	0.012	1.90%	0.034	0.049	7.73%	0.140
C	20	0.993	0.026	2.65%	0.075	0.067	6.72%	0.189
D	20	2.34	0.066	2.83%	0.187	0.215	9.16%	0.607
E	20	0.947	0.020	2.12%	0.057	0.062	6.51%	0.175

1) 2) 3) Refer to Table 4 for footnotes

The repeatability is the value below which the absolute difference between two single test results, obtained within short interval by the same operator using the standardized method on identical material, may be expected to lie with a probability of 95%. In the definition of reproducibility the "same operator" is replaced by "different operators in different laboratories".

If, for example, a laboratory wishes to test the method for the NBS Reference Material "Bovine Liver" in Table 1, certified or "true" value for sodium 0.243% by mass and for potassium 0.97% by mass, the chance is 95% that the duplicate results for sodium are within $0.243 \pm 0.012\%$ and for potassium $0.97 \pm 0.057\%$.

These results fit very well with the certified range; Table 1. However, in the case of different operators in different laboratories the results would be expected to lie in the range of 0.243 ± 0.041 for sodium and 0.97 ± 0.0175 for potassium; confidence 95%.

In general, the overall precision is acceptable given the fact that, except one, all laboratories were not familiar with the method and were self-selected. Therefore, it is realistic to expect a considerable improvement in precision when laboratories get more familiar with the procedure.

CONCLUSION

The atomic absorption spectrometric method described here for the single run determination of sodium and potassium was successfully tested for a variety of foods and biological substrates. Accuracy of results was found very satisfactory as illustrated by the results for a number of reference material covering a wide range of substrates and analyte levels. Additional method performance characteristics established in a collaborative trial in which 10 laboratories were involved, demonstrate that, in general and for the analyte levels considered, repeatability and reproducibility is satisfactory. None of the results accepted were more than 20% from the mean.

The method can be recommended where a single procedure for sodium and potassium is required for a wide range of biological materials and concentration levels.

Acknowledgements The authors express their appreciation to the following individuals who participated in this collaborative trial:

- H. Beernaert; Instituut voor Hygiëne en Epidemiologie, Brussel/Belgium.
- H.J.M. Bowen; University of Reading, Reading/United Kingdom.
- A. Broekman; Rijkskeuringsdienst van Waren, Leeuwarden/The Netherlands.
- A. van Ooik; Rijksinstituut voor Volksgezondheid en Milieuhygiëne, Bilthoven/The Netherlands.
- I. Lumley; Laboratory of the Government Chemist, London/United Kingdom.
- P.J. van Niekerk; National Food Research Institute, Pretoria/South Africa.
- M. Ozaki; Ajinomoto Co. Inc., Central Research Laboratories, Kawasaki/Japan.
- K. Rieder; Kantonales Laboratorium, Bern/Switzerland.
- W. de Schrijver; Laboratorium voor Toegepast Wetenschappelijk Onderzoek, Geel/Belgium.
- F.J. Slikkerveer; Unilever Research Laboratorium, Vlaardingen/The Netherlands.

REFERENCES

1. Parr, R.M.; Trace Element Analytical Chemistry in Medicine and Biology (1980), 631-655. Walter de Gruyter & Co.; Berlin, New York.
2. ISO Standard 5725; Precision of Test Methods - Determination of Repeatability and Reproducibility by Inter-Laboratory Tests. First edition - 1981.04.01. Ref. No. ISO 5725-1981 (E).
3. Official Methods of Analysis of the AOAC; Fourteenth Edition 1984. Association of Official Analytical Chemists Inc., Arlington, Virginia/USA. ISBN 0-935584-24-2.
4. Pocklington, W.D.; Guidelines for the Development of Standard Methods by Collaborative Study. Laboratory of the Government Chemist publication No. 1986 OP 001, London, 1986. ISBN 0-948926-00-7.

DETERMINATION OF SODIUM AND POTASSIUM CONTENT IN FOODSTUFFS AND BIOLOGICAL MATERIAL BY ATOMIC ABSORPTION SPECTROMETRY

1. SCOPE AND FIELD OF APPLICATION

This standard specifies an atomic absorption spectrometric (AAS) method for the determination of sodium and potassium in foodstuffs and all other types of biological material. Under the conditions specified in this standard sodium and potassium down to 5.10^{-3} percent by mass can be determined.

2. DEFINITION

Sodium and potassium content of foodstuffs and biological material: the sodium and potassium content determined according to the procedure specified in this standard and expressed in percent by mass.

3. PRINCIPLE

Organic matter present in the test portion taken for analysis is wet digested at 150°C in a pressure decomposition vessel and the digest is diluted and adjusted to pH 4 with ammonia. To suppress ionization of sodium and potassium in the flame of the AAS-instrument, cesium chloride solution is added to give a content of 2000 mg cesium per litre in the final solution. Additional dilutions are made as appropriate and the sodium and potassium content of the final solution is measured by atomic absorption using an oxidizing (lean-blue) air-acetylene flame; wavelength setting for sodium 589.6 nm and for potassium 766.5 nm.

4. REAGENTS AND MATERIALS

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

Select reagents lowest in sodium and potassium impurity. Avoid, wherever possible, the use of glass labware and store solutions, including twice distilled water, in polyethylene or polypropylene containers and labware to minimize alkali metal contamination from glass.

If the use of glass labware is unavoidable transfer all reagents and solutions as soon as possible to suitable polyethylene or polypropylene ware.

- 4.1 Nitric acid $\rho_{20} = 1.40$ g/ml. "Suprapur" grade Merck* (Darmstadt/GFR) or equivalent.
- 4.2 Hydrochloric acid, $\rho_{20} = 1.15$ g/ml. "Suprapur" grade Merck* (Darmstadt/GFR) or equivalent.
- 4.3 Ammonia, $\rho_{20} = 0.91$ g/ml. "Suprapur" grade Merck* (Darmstadt/GFR) or equivalent.
- 4.4 Nitric acid, $c(\text{HNO}_3) = 4$ mol/l.
- 4.5 Hydrochloric acid, $c(\text{HCl}) = 4$ mol/l.
- 4.6 Potassium chloride, "Ultrex" grade Baker* (Phillipsburg/USA) or equivalent.
- 4.7 Sodium chloride.
- 4.8 Cesium chloride, "Suprapur" grade Merck* (Darmstadt: GFR) or equivalent.
- 4.9 Bromocresol green, "Analysed" grade Baker* (Phillipsburg/USA) or equivalent.
- 4.10 Bromocresol green solution. Dissolve 0.1 g of bromocresol green in 14.3 ml of 0.01 mol/l ammonia solution, dilute to 250 ml with water and mix.
- 4.11 Cesium solution, 50.0 g/l. Dissolve 15.84 g of cesium chloride (4.8) in water, add 1 ml of nitric acid (4.1), dilute to 250 ml with water and mix.
- 4.12 Cesium solution, 2000 mg/l. Place 4.0 ml of the cesium solution in a 100 ml volumetric flask, add 50 ml of water and 5.0 ml of nitric acid (4.1), swirl, and add, by means of a pipette, 0.02 ml of the bromocresol green solution (4.10). Add sufficient ammonia solution (4.3) to change the colour of the indicator from yellow, via green, to just blue. Adjust the solution to pH 4 with hydrochloric acid (4.5), the indicator should just turn yellow, dilute to volume with water and mix.
- 4.13 Sodium/potassium stock solutions:
 - 4.13.1 Sodium/potassium solution, 1000 mg Na/l and 1000 mg K/l. Dissolve 1.907 g of potassium chloride and 2.542 of sodium chloride in water, add 67 ml of nitric acid (4.1) dilute to 1000 ml with water and mix.
 - 4.13.2 Sodium/potassium solution, 100 mg Na/l and 100 mg K/l. Place 10.0 ml of the sodium/potassium solution (4.13.1) in a 100 ml volumetric flask, dilute to volume with water and mix.
 - 4.13.3 Sodium/potassium solution, 10 mg Na/l and 10 mg K/l. Place 10.0 ml of the sodium/potassium solution (4.13.2) in a 100 ml volumetric flask, dilute to volume with water and mix.
- 4.14 Standard sodium/potassium solutions.
For each of the concentration ranges 0 to 1, 0 to 10 and 0 to 50 mg of sodium and potassium per litre, prepare five sodium/potassium standard solutions covering the levels specified as adequate as possible. For this purpose transfer by pipette a suitable aliquot of the stock solutions (4.13) into a 100 ml volumetric flask. Add

* Reference to company and/or branch names is for identity purposes exclusively and does not imply a guarantee or preference of the product specified to the exclusion of others which may be equally suitable.

4.0 ml of the cesium solution (4.11), 50 ml of water and 5.0 ml of nitric acid (4.1) and swirl to mix. Add 0.2 ml of the bromocresol green solution (4.10) and sufficient ammonia solution (4.3) to change the colour of the solution from yellow to just blue. Adjust the solution to pH 4 with hydrochloric acid (4.5), the indicator colour should just turn yellow, dilute to volume with water and mix.

5. APPARATUS

Standard laboratory apparatus and labware, and in particular the items specified hereafter. Avoid, wherever possible, use of glass labware because of the potential problem of alkali metal contamination associated with this material. Reusable polypropylene or polyethylene ware is recommended.

- Decontamination

Soak all the clean labware in nitric acid (4.4). Thoroughly rinse three times before use with water and store, if not immediately used, at a dust free place.

- 5.1 Bottles, screw-capped, of 50 ml. Wide mouth types of polyethylene or polypropylene are most suitable.
- 5.2 Graduated cylinders, polypropylene, of capacity 50 ml.
- 5.3 Volumetric flasks, polypropylene, of capacity 100, 250 and 1000 ml.
- 5.4 Graduated pipette, with glass piston, of capacity 5 ml.
- 5.5 Adjustable pipettes, with disposable plastic tips, and of continuous adjustable volumes of 50 to 250 μ l, 200 to 2000 μ l and 1000 to 5000 μ l. Finnipipettes^{**}, Labsystems. Oy (Helsinki/Finland) or equivalent.
- 5.6 Pressure decomposition vessel. Teflon crucible and lid contained by compact stainless steel body and screw on cap, crucible of capacity 25 ml. Uni-Seal^{**} Decomposition Vessel (Haifa/Israel) or equivalent.
- Decontamination
Teflon crucibles and lids are boiled 1 hour in hydrochloric acid (4.5), rinsed with water and boiled 1 hour in water before drying at 120°C in an electrical heated oven. Cleaned crucibles and lids are stored at a dust free place.
- 5.7 Electrically heated oven, capable of being maintained at 150°C and equipped with a thermometer. The oven is located in a fume hood.
- 5.8 Atomic absorption spectrometer, fitted with a burner for an air-acetylene mixture and suitable for measurements at a wavelength of 589.6 nm for sodium and 766.5 nm for potassium.
- 5.9 Hollow cathode lamps, single element type, for sodium and for potassium.

6. SAMPLING AND SAMPLES

- 6.1 Laboratory samples
Avoid contamination by sodium and or potassium. Store samples in plastic containers in such a way that deterioration and change in composition are prevented.
- 6.2 Test sample
Proceed, if available, from a portion of at least 200 g of the laboratory sample. In the case of food, remove inedible parts and adhering dust and dirt according to common household practice and blend or chop to a homogeneous consistency. Whenever washing is required (eg. vegetables) use distilled water to do this and remove adhering washwater. If the product is lyophilized or dried, re-homogenization after this process is necessary. Store the homogenized test sample in a completely filled and closed container in such a way that deterioration and change in composition are prevented. Take a test portion for analysis (7) as soon as possible after homogenization. Allow frozen or deep-frozen products to thaw and, if required, rehomogenize before the test portion is taken.

7. PROCEDURE

- 7.1 Test portion
Weigh, to the nearest 0.1 mg, a test sample portion (6.2) of 200 to 300 mg into the teflon crucible of a pressure decomposition vessel.
NOTE
For safety reasons the decomposition vessel should never be charged with more than 350 mg of sample.
- 7.2 Decomposition
 - 7.2.1 Add 5.0 ml of nitric acid (4.1) to the test portion, place the teflon crucible in the stainless steel body, close with the teflon lid and screw on the stainless cap. Close handtight or according to manufacturers instructions.
 - 7.2.2 Heat the vessels during 10 hours at 150°C in an oven maintained at this temperature. It is adequate and safe to do this overnight in which case heating is timeswitch controlled and the oven and decomposition vessels are cooled down next morning. Remove the vessels from the oven, unscrew the cap and transfer the contents of the teflon crucible, including drops adhering at the inner side of the lid, to a 100 ml volumetric flask. Wash crucible and lid with water and combine these washings with the contents of the volumetric flask. Use approximately 50 ml of water to transfer the digest and to wash the teflon crucible and lid.

^{**}Refer to footnote on page 2.

7.2.3 Add 4.0 ml of the cesium solution (4.11) and 0.2 ml of the bromocresol green solution to the diluted digest (7.2.2), use adjustable pipettes to do this, and mix. Add ammonia solution (4.3) to change the colour of the indicator from yellow, via green, to just blue and swirl. Dropwise add hydrochloric acid (4.5) until the colour of the solution just remains yellow, pH 4, dilute with water to volume and mix.

7.3 Blank test

Carry out a blank test using the same conditions as in 7.1 and 7.2, but replacing the test portion by 0.3 ml of water.

7.4 Determination

7.4.1 Sodium measurement

7.4.1.1 Select the appropriate settings and conditions for the flame atomic absorption measurement of sodium, lean-blue air-acetylene flame, and adjust the spectrometer to obtain optimal measuring conditions for sodium at 589.6 nm wavelength. Zero the instrument with water and aspirate the blank test solution obtained (7.3) and the test solution (7.2.3).

Adapt, if required, the optical pathlength in the flame and record the instrument readings.

7.4.1.2 Select a set of standard sodium/potassium solutions (4.13) covering the sodium content of the test solutions as adequately as possible and aspirate as indicated.

Dilute, if required, the test solutions (7.2.3) with cesium solution (4.12) in a screw-cap bottle using adjustable pipettes (5.5) and carry out the sodium measurement as specified above.

7.4.1.3 Draw the calibration graph for the standard solutions selected and correlate the readings of the test solutions with the corresponding sodium content of the solution. Under optimal conditions the readings of the blank test solution and the zero standard solution (7.4.1.2) should be identical.

7.4.2 Potassium measurement

Carry out the potassium measurement as described under 7.4.1 substituting sodium for potassium. Set the instrument at 766.5 nm wavelength for this element.

8. RESULTS

8.1 Methods of calculation and formulas

- The sodium content, expressed in percent by mass, of the product is given by the formula:

$$\frac{c_1 \times f_1 \times 10}{m}$$

where

c_1 is the sodium content of the test solution in milligrams per litre, read from the calibration graph (7.4.1.3)

f_1 is the dilution factor of the test solution (7.4.1.2)

m is the mass, in milligrams, of the test portion (7.1)

Report the results in three significant decimal places.

- The potassium content, expressed in percent by mass, of the product is given by the formula:

$$\frac{c_2 \times f_2 \times 10}{m}$$

where

c_2 is the potassium content of the test solution, in milligrams per litre, read from the calibration graph (7.4.2)

f_2 is the dilution factor of the test solution (7.4.2)

m is the mass, in milligrams, of the test portion (7.1)

Report the results in three significant decimal places.

8.2 Repeatability

The difference between the results of duplicate determinations, obtained almost simultaneously or in rapid succession by the same analyst, shall not exceed 5 percent of the arithmetic mean for sodium and 3 percent of the mean for potassium.