Health Canada Santé Canada

Laboratory Procedure

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A Rapid Method for the Determination of Sodium and Potassium

Definition: This method is applicable to the determination of sodium and potassium in processed foods.

Scope: The rapid method has been evaluated by comparison with a dry ashing sample preparation. The sodium content of seven foodstuffs and the NBS-1577 Liver powder standard was measured in two laboratories. A comparison of sodium results (rapid method ÷ dry ash method x 100) showed good agreement in both laboratories with an average, based on the analyses of eight items, of 100.5 and 100.7 (Table 2). The potassium content of the same eight items was measured in one laboratory. A comparison of results for potassium (rapid method ÷ dry ash method x 100) is 97.9. The sodium and potassium content of the NBS-1577 Liver powder standard was measured in the two laboratories using both the rapid and dry ash methods which generated results which were within the certified limits for both sodium and potassium.

Principle: The method consists of preparing a bulk homogeneous sample, from which a sub-sample is taken. The sub-sample is mechanically homogenized and dispersed in an appropriate volume of deionized water to achieve solution of sodium and potassium. After filtration, a particle-free filtrate is diluted appropriately and sodium and potassium are determined by flame atomic emission spectro-photometry.

Apparatus:

1. Flame atomic absorption/emission spectrophotometer.
2. Homogenizing mill with stainless steel shaft and cutters, capable of shearing, cutting, impact, cavitation and a speed of 45,000 rpm.
3. Centrifuge with head to hold 250 mL centrifuge battles and be capable of centrifugation at 1000 x g.
4. Food blender with rheostat to control speed.
5. Top loading balance of 1000 g capacity, capable of ± 0.01 g accuracy.
6. Polypropylene (PP) ware:
   i) straight side wide mouth jars (950 mL) with screw caps.
   ii) wide mouth centrifuge bottles (250 mL) with screw caps.
   iii) bottles with screw caps, 60, 120 and 250 ML.
   iv) funnels (for 11.0 cm filter paper).
   v) stirring rod.

7. Filter paper, Whatman No. 41 (11.0 cm, ashless) or equivalent.
8. Plastic gloves.
9. Pyrex beaker.
10. Plastic wash bottles (one for deionized water, the other for acetone).

Reagents:
1. Deionized water (resistivity of 1 megohm or better), referred to as H_2O.
2. Sodium atomic absorption standard solution, 1000 ppm (commercially available).
3. Potassium atomic absorption standard solution, 1000 ppm (commercially available).
4. Reagent grade concentrated hydrochloric acid (HCl), 37%.
5. Dilute HCl (approximately 0.37% HCl) prepared from reagent grade HCl by diluting concentrated HCl (37%) 1.2 g to 100.00 g with H_2O.
6. Reagent grade acetone.

Standards: Standards are prepared on a weight for weight basis using the appropriate size of PP bottle.

1. a) Prepare a 100 ppm Na solution by diluting 10.00 g of 1000 ppm Na to 100.00 g using H_2O.
   b) Prepare a 100 ppm K solution as in 1.a).
2. Prepare an intermediate solution of 25 ppm Na/K solution by diluting 50.00 g of the 100 ppm Na (1.a)) plus 50.00 g of the 100 ppm K (1.b)) plus 2.40 g concentrated HCl to 200.00 g, using H_2O.
3. Prepare working standards of 0.5, 1.0, 2.0, 3.0 and 4.0 ppm Na/K by diluting 2.00, 4.00, 8.00, 12.00 and 16.00 g respectively to 100.00 g, using dilute HCl.

Preparation: Wearing plastic gloves is mandatory throughout the sample preparation procedure (Na/K from skin can be a source of contamination). The glass and plastic ware must be rinsed in H_2O, dilute HCl and then H_2O, then dried in an oven at 100°C.

Procedure: A. Preparation of the sample.

1. Prepare a homogeneous, finely divided sample of 500 g or more using the blender with rheostatic control. Use the PP stirring rod to facilitate mixing.
2. Transfer the blended sample to a straight side, wide mouth jar and refrigerate until analysis.

B. Extraction

1. Allow the refrigerated samples to come to room temperature (about 30 minutes).
2. Into a previously tared centrifuge bottle (250 mL), weigh about 10 g of the blended sample, with the use of a PP stirring rod. Record sample weight.
3. Add sufficient H_2O (usually 100 mL to cover the cutters of the homogenizing mill and homogenize at maximum speed (about 90 seconds for meat and tune, and about 60 seconds for vegetables).
4. Rinse the shaft and cutters of the mill with H_2O, allowing the rinse water to flow

into the centrifuge bottle. Make up to about 200 g. Record the solution weight.

5. Cap the centrifuge bottle and shake vigorously to disperse the homogenate.
6. After every sample.
   a) rinse the shaft and cutters with acetone.
   b) Immerse the cutters in a beaker of acetone and run at half speed.
   c) repeat 6.a) and b) with H_2O.
7. Prepare a blank with each series of samples in the same manner as the samples, starting at step 8.3.
8. Centrifuge the bottles for 20 minutes at 1000 x g to settle fibrous material.
9. Filter a portion of the supernate (about 50 mL) into a PP bottle, discarding the first 5 to 10 mL. Dilute and determine Na/K in the filtrate within 3 days.

C. Dilution

Dilute the sample (to obtain a concentration of about 1.5 ppm for Na/K) and the blank weight for weight, using dilute HCl and PP bottles of the appropriate size.

D. Determination

1. Set up the spectrophatometer for flame emission according to the manufacturer's instructions.
2. Determine Na at a wavelength of 589.6 nm, with a spectral band width of 0.3 nm and K at a wavelength of 769.9 nm, with a spectral band width of 1.0 nm.
3. Use the highest standard (4 ppm) to adjust the photo multiplier voltage to give a reading of about 1 abosrbance unit.
4. Run standards and samples.

Reference: Spitzer, M.E., Ritchey, C., Glennon, J.M., Villarreal, Y, and Mason, Jr., A.D. A rapid method of preparing food for sodium and potassium analyses. J Am Dietetic Assoc 62:44-46, 1973

METHOD EVALUATION

The rapid method has been evaluated by comparison with a dry ashing sample preparation. Homogenates of weiner, bologna, canned tuna, canned peas, canned corn, canned carrots and canned green beans were prepared in one laboratory and an aliquot of each shipped to the second laboratory. A sample of NBS-1577 bovine liver powder standard was also analyzed in both laboratories.

The results obtained by the two laboratories are given In Tables 1, 2 and 3. The results of the sodium analyses in Table I show that the coefficient of variation for both methods in two laboratories were generally low and did not exceed 6.3%. As indicated in Tables 2 and 3, comparable results for the sodium content of eight items were achieved using both methods in two laboratories.

Table 4 shows the results of the potassium analyses using both the rapid method and the dry ash method in one laboratory.

The certified 95% limit of sodium and potassium content of the MS-1577 liver powder standard was 230-256 mg/100 g and 910-1030 mg/100 g respectively. Analyses of this standard for both sodium and potassium, using both methods in two laboratories generated results which fell within the certified limits for sodium and potassium (Table 1, Table 4).

TABLE 1

Sodium Content of Some Foods Determined by Two Laboratories

	Dry Ash Method		Rapid Method	
	Lab A	Lab B	Lab A	Lab B
Weiner	970±18 (1.9)	925±12 (1.6)	965±12 (1.2)	935±5 (0.5)
	1080±8 (0.7)	1022±22 (2.2)	1001±22 (2.2)	984±10 (1.0)
Tuna	484±11 (2.3)	460±17 (3.7)	449±2 (0.4)	455±8 (1.8)
Peas	229±10 (4.4)	244±3 (1.2)	256±7 (2.7)	246±6 (2.4)
Corn	209±10 (4.8)	198±3 (1.5)	209±7 (1.4)	203±3 (1.5)
Carrots	341±10 (4.8)	350±3 (0.9)	352±10 (2.8)	356±2 (0.6)
Green Beans	314±14 (4.4)	344±7 (2.0)	336±10 (3.0)	337±2 (0.6)
NBS-1577 Liver Powder Std	238±15 (6.3)	233±3 (1.3)	231 -	247±8 (3.2)

Results expressed in mg/100g, mean±S.D., n=6

( ) = coefficient of variation, %

TABLE 2

Comparison of Sodium Results from Two Methods

	(rapid method ÷ dry ash method) x 100	
	Lab A	Lab B
Weiners	99.5	101.0
Bologna	92.7	96.3
Tuna	92.8	98.9
Peas	111.8	100.8
Corn	100.00	102.5
Carrots	103.2	101.7
Green Beans	107.0	98.0
NBS-1577 Liver Powder Std.	97.1	106.0
Average	100.5	100.7

TABLE 3

Comparison of Sodium Results from Two Methods

	(Lab A ÷ Lab B) x 100	
	Dry Ash Method	Rapid Method
Weiners	104.9	103.2
Bologna	105.7	101.7
Tuna	105.2	98.7
Peas	93.9	104.1
Corn	105.6	103.0
Carrots	97.4	98.9
Green Beans	91.3	99.7
NBS-1577 Liver Powder Std.	102.1	93.5
Average	100.8	100.4

TABLE 4

Potassium Content of Some Foods Determined in One Laboratory

	Dry Ash Method	Rapid Method
Weiner	163±5 (3.1)	158±2 (1.3)
Bologna	169±3 (1.8)	164±3 (1.8)
Tuna	171±3 (1.8)	168±3 (1.8)
Peas	75±1 (1.3)	73±2 (2.7)
Corn	196±2 (1.0)	191±2 (1.0)
Carrots	106±1 (0.9)	104±1 (1.0)
Green Beans	105±1 (1.0)	102±2 (2.0)
NBS-1577 Liver Powder Std*	989±2 (0.2)	1004±19 (1.9)

Results expresse in mg/100g, mean±S.D., n=6
( ) = coefficient of variation, %
* The second laboratory also analysed
this standard 970 943