

Original paper

Some elements in domestic and imported fresh fruits marketed in the Netherlands

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Einige Mineralstoffe im einheimischen und importierten Frischobst vom niederländischen Markt

Zusammenfassung. Natrium, Kalium, Arsen, Selen und Zinn wurden in 38 verschiedenen Sorten von in- und ausländischem Frischobst bestimmt. Alle Proben ($n=242$) wurden auf Na und K untersucht; während nur eine beschränkte Zahl von Proben ($n=85$) für die Bestimmung von As, Se und Sn ausgewählt wurde. Die Medianwerte, in Massenanteilen der eßbaren Portionen, betrugen für frisches Obst: Na $<0,001\%$; K $0,17\%$; As $4\text{ }\mu\text{g/kg}$; Se $2\text{ }\mu\text{g/kg}$ und Sn $<0,05\text{ mg/kg}$. Die dazugehörigen Werte für den 90. Perzentil sind: Na $0,002\%$; K $0,34\%$; As $12\text{ }\mu\text{g/kg}$; Se $10\text{ }\mu\text{g/kg}$ und Sn $0,10\text{ mg/kg}$. Für einige Obstsorten wurden Selenwerte erhalten, die erheblich von denen der Deutschen Nährwert-Tabellen 1986/1987 abweichen. Frischobst, dessen Verzehr für Einwohner der Niederlande in der Altersklasse von 22–75 Jahre im Schnitt 129 g/Tag beträgt, liefert einen vernachlässigbaren Beitrag zur täglichen Einnahme von Natrium, Arsen, Selen und Zinn; für Kalium hingegen kann dieser Beitrag, mit bis zu 23% , erheblich sein.

Summary. Sodium, potassium, arsenic, selenium and tin have been determined in 38 different types of domestic and imported fresh fruits. All samples ($n=242$) were analysed for Na and K whereas a limited number of samples ($n=85$) of each fruit type was selected for the determination of As, Se and Sn. The median contents, in mass fractions of the edible portion, found for fresh fruits are: Na $<0.001\%$; K 0.17% ; As $4\text{ }\mu\text{g/kg}$; Se $2\text{ }\mu\text{g/kg}$ and Sn $<0.05\text{ mg/kg}$. The corresponding 90th percentile values are: Na 0.002% , K 0.34% ; As $12\text{ }\mu\text{g/kg}$; Se $10\text{ }\mu\text{g/kg}$ and Sn 0.10 mg/kg . Results for selenium reported here differ substantially from those given for a number of fresh fruits in the German Food Composition Tables 1986/1987. The average consumption of 129 g fresh fruit by Dutch citizens in the age category of 22–75 years con-

tributes, in general, marginally to the total daily dietary intake of sodium, arsenic, selenium and tin. Fresh fruit can contribute substantially, up to 23% , to the average oral daily intake of potassium.

Introduction

Global redistribution of elements by man causes increasing concern and interest in the elemental levels of foodstuffs and their safety. Initially these concerns predominantly focussed on heavy metal contaminants like lead, cadmium and mercury but later it was realized that the redistribution processes can have an impact on virtually all elements in foodstuffs and thereby possibly affect health. Consequently an increasing number of elements are being included in surveillance programmes for elemental levels of foods. In the late sixties several health authorities in The Netherlands initiated heavy metal surveillance programmes. By that time programmes for mapping radioactive (fall-out), pesticide and biological (parasites, bacteria) contamination of foods already existed for quite some time. In 1974 these programmes were pooled to one national and nationwide surveillance programme called "Man and Nutrition" of the State Supervisory Health Service. In the context of this programme our laboratory has analysed numerous types of individual foods and also duplicate 24 h diets for elemental composition and nutrients [1–5]. Human tissues, e. g. kidney, liver and vertebrae, have also been studied extensively and in particular for lead, cadmium, and mercury [6].

In 1983/1984, 242 samples of fresh domestic and imported fruit of 37 different types were analysed for cadmium, copper, lead, manganese, mercury, potassium, sodium and zinc. From these samples, 85 were selected for determination of arsenic, selenium and tin. Results for cadmium, copper, lead, mercury and zinc have already been published [7]. Because of the unique character of the information collected, results for potassium,

sodium, arsenic, selenium and tin are described and discussed in detail here.

Materials and methods

Sampling and sample pre-treatment. Selection of sample types and numbers was based on the most recent statistical per capita consumption figures for fresh fruits in The Netherlands at the time of sampling. However, at least three samples of each fruit type were collected to allow for a rough impression of the distribution of the elements in the fruits considered. All samples ($n=242$) were obtained in 1983 from retail outlets or local markets in the region Utrecht/Zeist. Domestic fruits were collected during the season when they are most abundant, i.e. between June and November. Fresh products were collected exclusively. Information on the various types of fruits sampled and the number of samples taken can be found in Table 1.

Sampling of fresh fruits expanded over almost half a year. Because of the number of elements under consideration, the limited re-

frigeration facilities and the risk of compositional changes during storage, especially for certain exotic fruits, it was decided to preserve all samples by lyophilization. After delivery to the laboratory, samples were stored at 4° C and processed further as soon as possible. Fruits were washed and stripped of inedible parts according to common household practices. Any adhering wash water was removed with a clean cotton cloth, whereupon the products were cut into small pieces and lyophilized. The dried materials were homogenized in a household blender fitted with stainless steel blades and the homogenate stored in tightly closed plastic containers at -20° C until analysis.

Analytical procedures

Sodium and potassium. Both these elements were measured by flame atomic absorption spectrometry (FAAS) of test solutions resulting from samples by wet pressurized digestion, dilution and pH adjustment of the digest. The method applied has been studied recently in a IUPAC collaborative trial. Full details of the method and results of the collaborative study have been published [8]. In brief, the procedure ran as follows. A 300-mg sample was digested under pressure overnight at 150° C with 5 ml conc. HNO_3 in a teflon crucible with lid, contained in a stainless steel vessel with screw-on cap. The digest was diluted with double-distilled water and adjusted to pH 4 with ammonia using bromocresol green as an indicator. To suppress ionization of sodium and potassium in the AAS flame, cesium chloride was added to bring the cesium content in the final (test) solution to 2000 mg/L. Additional dilutions to bring the concentration of the analyte within the sensitivity range of the method were done with solutions containing 2000 mg Cs/L. Sodium and potassium were measured with FAAS using an oxidant-rich (lean-blue) air/acetylene flame; wavelength settings 589.6 nm for Na and 766.5 nm for K. Non-specific absorption is compensated for by a hydrogen lamp background corrector. Results were quantified by relating the absorption signal to a calibration graph. The limit of determination for Na and K in the test portions, the lyophilized fruits, is 0.001%. In the fresh products the limit of determination is dependent on the dry matter content of the various products which can differ up to a factor of 5. For practical reasons, the limit in all fresh fruits was set here at 0.001%, i.e. worst-case situation.

Arsenic. Total arsenic was determined via a procedure of wet digestion, reduction of As in the trivalent state, isolation and complexation of arsine (AsH_3) and measuring with molecular absorption spectrometry (MAS). In brief, the procedure [9] ran as follows. 10 g lyophilized fresh fruit was mixed with 30 ml conc. HNO_3 and 10 ml conc. H_2SO_4 in a round-bottomed flask which was connected to a Bethge decomposition-distillation apparatus. After standing overnight in a fume hood, the mixture was boiled under reflux for 30 min, followed by decomposition until white acid fumes appeared. The flask was disconnected and the decomposition completed with 1 ml conc. HClO_4 and three repetitive treatments with 1 ml conc. H_2O_2 . Then the digest was transferred with water to the flask of an arsine generator and pentavalent As converted to the trivalent state with 2 ml 15% KI and 2 ml 40% SnCl_2 ; reduction time was 15 min at 55° C. After cooling to 0° C, 3 g Zn (10 mesh) was added and the flask connected to the absorption tube of the arsine generator which contained 3 ml 0.5% silver diethyldithiocarbamate (AgDDTC) in pyridine. AsH_3 generated from the test solution, was swept with excess hydrogen into the AgDDTC solution; the absorbance of the AsH_3 - AgDDTC complex at 526 nm was related to the As content of the sample by a calibration graph. Under the conditions described, the limit of determination is 15 $\mu\text{g/kg}$ lyophilized product. In fresh fruits these limits depend on the dry matter content which, for the products studied, varies up to a factor of five. Here this limit ranged therefore over 2–5 $\mu\text{g/kg}$ of fresh fruit.

Selenium. The method for selenium has been extensively validated, including a IUPAC-sponsored international collaborative trial [10, 11]. Selenium was determined by a procedure involving wet pressur-

Table 1. Sodium and potassium content of domestic and imported fruits. Total number of samples analysed (n) 242; number of fruit types 38. All data are on edible portions of the products

Fruit	n	Sodium (%)		Potassium (%)	
		Median	Range	Mean	Range
Apples	50	0.001	<0.001–0.002	0.13	0.07–0.22
Apricots	4	<0.001	^a	0.28	0.25–0.33
Avocados	3	0.002	0.002–0.004	0.38	0.33–0.45
Bananas	19	<0.001	<0.001–0.002	0.36	0.30–0.42
Barbary figs	3	<0.001	^a	0.14	0.10–0.18
Blackberries	4	0.002	<0.001–0.002	0.19	0.18–0.19
Blackcurrants	4	0.002	0.001–0.003	0.29	0.23–0.38
Blueberries	4	0.001	<0.001–0.001	0.09	0.07–0.10
Cherimoyas	3	0.008	0.007–0.015	0.25	0.22–0.30
Cherries (sweet)	5	<0.001	^a	0.24	0.20–0.29
Cranberries	3	0.001	0.001–0.001	0.09	0.08–0.09
Figs	3	0.001	<0.001–0.001	0.25	0.22–0.27
Gooseberries	5	0.002	0.001–0.007	0.20	0.10–0.35
Grapefruits	4	0.001	0.001–0.001	0.18	0.12–0.29
Grapes	4	<0.001	^a	0.19	0.19–0.20
Kiwi fruits	3	0.002	0.002–0.002	0.32	0.20–0.36
Lemons	4	0.001	0.001–0.002	0.19	0.15–0.22
Litchis	3	<0.001	^a	0.19	0.18–0.20
Mandarins	6	0.001	<0.001–0.003	0.13	0.10–0.16
Mangoes	3	<0.001	^a	0.15	0.14–0.16
Medlars	4	0.002	0.001–0.003	0.23	0.15–0.31
Melons	4	0.010	0.001–0.020	0.31	0.23–0.39
Nectarines	4	<0.001	<0.001–0.001	0.21	0.20–0.22
Oranges	35	<0.001	<0.001–0.001	0.16	0.09–0.22
Papayas	3	0.001	0.001–0.002	0.17	0.15–0.19
Passion fruits	3	<0.001	^a	0.24	0.22–0.25
Peaches	4	<0.001	<0.001–0.001	0.17	0.16–0.19
Pears	10	0.001	<0.001–0.001	0.11	0.09–0.13
Persimmons	3	<0.001	<0.001–0.001	0.18	0.16–0.21
Pineapples	3	<0.001	^a	0.18	0.14–0.23
Plantains	3	<0.001	^a	0.38	0.20–0.50
Plums	7	<0.001	^a	0.19	0.14–0.22
Pomegranates	3	0.001	0.001–0.001	0.22	0.21–0.23
Quinces	3	0.002	<0.001–0.002	0.16	0.14–0.17
Raspberries	4	<0.001	^a	0.23	0.19–0.25
Red currants	4	0.002	0.001–0.002	0.30	0.22–0.37
Strawberries	5	0.002	0.001–0.004	0.18	0.16–0.19
Uglis	3	<0.001	^a	0.16	0.15–0.17

^a All values below the limit of determination

ized digestion with HNO_3 , derivatization of Se to a fluorescent compound and measuring the fluorescence. In brief, this was done as follows. 300 mg lyophilized fresh fruit was digested under pressure with 5 ml HNO_3 overnight at 150°C in a teflon decomposition vessel. The digest was transferred to a 50-ml Kjeldahl flask and treated with conc. HClO_4 until copious white fumes were expelled. Next, Se was reduced to the tetravalent state with conc. HCl on a boiling water bath for 30 min, reacted with 2,3-diaminonaphthalene at 60°C for 40 min to give 4,5-benzopiazselenol, which was extracted into cyclohexane. The fluorescence of the extract is a measure of the Se content of the sample; excitation wavelength 375 nm, emission wavelength 520 nm. The fluorescence signals of the test solution were quantified by relating them to a calibration graph of selenium standard solutions taken through the entire procedure. The limit of determination for Se in the lyophilized fruits, i.e. the test portions, is $5\text{ }\mu\text{g/kg}$. Fresh fruits have various dry matter contents and thus different limits of determination. For the products studied, the highest value for the limit of determination was found to be $1\text{ }\mu\text{g/kg}$ in the fresh product; for practical reasons, this value was taken for all fresh fruits.

Tin. MAS was used to measure Sn in the test solutions. These were obtained by a procedure involving wet digestion, extractive isolation of Sn, back-extraction and reaction with a chromophore to a coloured Sn complex. The method has been tested successfully in an international collaborative trial by a IUPAC working group and is described in detail in the literature [12]. In brief, the procedure ran as follows. 10 g lyophilized fresh fruit was digested as described here for As. The resulting sulphuric acid solution was diluted to give an approximately 4.5 M acid solution. KI was added to give SnI_4 which was selectively extracted into cyclohexane. By shaking the organic layer with NaOH solution, the tin was back-extracted into aqueous solution which was then acidified. Free iodine was removed by reduction with ascorbic acid, the chromophore catechol violet added, the solution buffered at pH 3.8 and set aside for 45 min at room temperature. The absorbance of the resulting coloured complex was determined spectrometrically at 555 nm and the results quantified by relating the optical signal to that of a calibration graph for Sn standard solutions. Under the conditions described, the limit of determination for Sn is 0.25 mg/kg lyophilized fruit. Because of the differences in dry matter content of fresh fruits, this corresponds to $0.02\text{--}0.09\text{ mg/kg}$ in the fresh products considered here.

Analytical quality assurance. Over the years the analytical procedures described have been tested extensively for precision and accuracy. Besides numerous replicate and recovery experiments, these tests included analyses of as many food reference materials as available for the elements concerned [8–10, 12]. Part of these data were collected in this fresh fruit study. Moreover the procedures for sodium/potassium, selenium and tin have been successfully tested in IUPAC international collaborative studies, the results of which have been published [8, 11, 12]. Performance checks on the method also include participation in various national and international intercomparison studies and certification campaigns for candidate reference materials of the Community Bureau of Reference (BCR) of the EC. So far, good quality data have been obtained throughout these investigations.

Additional information on the precision of the results was collected by duplicate analyses for a different number of samples per element. The agreement between duplicates was defined by the term

$[(a-b)/\bar{x}] \cdot 100\%$, where a and b are the repeat determination results and \bar{x} the mean result. Precision estimates thus obtained per sample were averaged for each element and are summarized here.

On average, the estimated precision is quite good with highest average values of 13.2% for arsenic and 13.5% for selenium. For selenium the agreement within duplicates was sometimes disappointing. Here replicates can differ up to 46%, for instance for cherimoyas with a selenium content of $2\text{ }\mu\text{g/kg}$. It is obvious that this high difference within duplicates is inherent to the extremely low selenium levels of the fruits with 90% of the results below $10\text{ }\mu\text{g/kg}$ (Table 1). Nonetheless the precision has been studied in more detail for one of the samples, plantains, which have the highest selenium content (Table 3). Eleven replicates were done on this sample giving an average selenium content of $32\text{ }\mu\text{g/kg}$ with figures of $29\text{--}34\text{ }\mu\text{g/kg}$ for the range and a relative standard deviation of 4.6%. Clearly these figures stress the risk of highly imprecise measurements close to the determination limit of method. However, the overall conclusion is that, during the fresh fruits study, precision and accuracy of the procedures applied were well under control.

Results and discussion

Quite frequently it is difficult to assess whether foods have been contaminated, e.g. from the environment, by transport, processing or production, because of inadequate information about the background levels of the components of interest in the product. For 38 different species of fresh fruits collected in The Netherlands, such data are given here for sodium and potassium (Table 1) and for arsenic, selenium and tin (Table 2). Results for cadmium, lead, mercury, copper, manganese and zinc in the same batch of products have been published elsewhere [7].

In general, the results for sodium, potassium, arsenic, selenium and, to a lesser extent, for tin lie within a small concentration range. This is best illustrated by the median and 90th percentile values in Table 3 where for each set of results some statistical parameters are given. Also given there are the number of samples analysed and the percentage with a result below the limit of determination. The majority of results for sodium and tin, a contaminant, are below this limit whereas for arsenic, also a contaminant, and selenium this holds for 20% of the figures. For these four elements 90% of the results are quite close, a factor of ten or less, to the respective limits of determination. Clearly potassium is an exception to this with levels of 0.07–0.50% and a median value of 0.17% (Table 3).

Data on sodium and potassium in most fresh fruits are available from the German Food Composition Tables [13]. Sodium levels published there and found in this study are extremely low. However, it should be pointed out that the vast majority of our results are even below those in the food composition tables. Data on the potassium content of 31 of the 38 fruit species studied are here given in the food composition tables; levels in mandarins, passion fruits and pomegranates in our study are substantially lower, whereas those for raspberries and red currants are higher than those in the tables [13]. Varo et al. [14] studied the elemental composition of a number of fresh fruits and reported results for 14 fruit types very similar to those found here for these products. Information on selenium in a number of fresh fruits are also given in both references and again the results of Varo et al. are al-

Element	No. of replicate results	Estimate of precision (%)	
		Range	Mean
Sodium	6	0 –16.7	5.3
Potassium	6	0 – 5.3	2.3
Arsenic	9	2.1–28.6	13.2
Selenium	9	0 –46.2	13.5
Tin	5	0 – 1.5	0.4

Table 2. Arsenic, selenium and tin content of domestic and imported fresh fruits. Total number of samples analysed (*n*) 85; number of fruit types 38. All data are on edible portions of the products

Fruit	<i>n</i>	Individual results; range where <i>n</i> > 2 ^a					
		Arsenic (µg/kg)		Selenium (µg/kg)		Tin (mg/kg)	
Apples	5	<3–	7 (4)	<1–	1 (<1)	<0.03–	0.07 (<0.04)
Apricots	2	3–	5	1–	1	0.04–	0.07
Avokados	2	18–	37	5–	5	<0.05–	<0.06
Bananas	4	4–	10 (7)	<1–	22 (6)	<0.06–	0.07 (<0.06)
Barbary figs	2	2–	4	2–	3	<0.03–	<0.05
Blackberries	2	4–	6	1–	2	0.07–	0.18
Blackcurrants	2	6–	8	5–	5	<0.04–	<0.05
Blueberries	2	11–	19	<1–	<1	0.09–	0.13
Cherimoyas	2	6–	7	2–	3	<0.06–	<0.07
Cherries (sweet)	2	5–	5	3–	3	<0.06–	0.08
Cranberries	2	10–	11	2–	2	0.04–	0.05
Figs	2	<2–	<2	3–	3	^b	
Gooseberries	2	4–	16	<1–	2	0.09–	0.30
Grapefruits	2	2–	2	1–	2	<0.02–	<0.02
Grapes	2	<3–	4	1–	2	0.05–	0.12
Kiwi fruits	2	4–	5	1–	2	<0.04–	0.05
Lemons	2	3–	3	1–	1	<0.02–	<0.03
Litchis	2	<3–	<3	1–	2	<0.05–	<0.05
Mandarins	2	<2–	<2	<1–	<1	<0.03–	0.04
Mangoes	2	<2–	2	12–	18	<0.04–	0.04
Medlars	2	<4–	16	2–	3	<0.06–	0.07
Melons	2	2–	5	1–	4	<0.02–	<0.03
Nectarines	2	2–	2	1–	1	<0.03–	<0.03
Oranges	5	2–	19 (3)	<1–	<1 (<1)	<0.03–	<0.03 (<0.03)
Papayas	2	<2–	2	8–	28	<0.03–	0.03
Passion fruits	2	4–	8	12–	13	<0.07–	<0.07
Peaches	2	3–	4	<1–	1	<0.03–	0.10
Pears	3	2–	13 (3)	2–	3 (3)	<0.04–	0.08 (0.05)
Persimmons	2	3–	6	2–	3	<0.04–	<0.04
Pineapples	2	3–	8	1–	2	0.05–	0.05
Plantains	1	9		32		<0.09	
Plums	3	<2–	3 (<2)	<1–	1 (1)	0.07–	0.54 (0.21)
Pomegranates	2	4–	5	2–	3	<0.05–	0.05
Quinces	2	<2–	3	1–	4	<0.04–	<0.05
Raspberries	2	9–	13	1–	2	0.05–	0.08
Red currants	2	<2–	<4	2–	6	<0.04–	1.52
Strawberries	2	6–	21	1–	1	<0.03–	0.23 ^c (0.04)
Uglis	2	<2–	3	4–	4	<0.03–	<0.03

^a Median value in brackets where *n* > 2

^b Insufficient sample material left

^c Number of samples analysed: 4

Table 3. Summary results for sodium, potassium, arsenic, selenium and tin in fresh fruits

Parameter	Sodium (%)	Potassium (%)	Arsenic (µg/kg)	Selenium (µg/kg)	Tin (mg/kg)
Median	<0.001	0.17	4	2	<0.05
90th percentile	0.002	0.34	12	10	0.10
Lowest value	<0.001	0.07	<2	<1	<0.02
Highest value	0.020	0.50	37	32	1.52
No. of samples analysed, <i>n</i>	242	242	85	85	85
Data below limit of determination	51%	0%	20%	21%	59%

most identical to those reported here while for 7 out of 12 fruit species, selenium results reported in the food composition tables differ dramatically from those given by Varo et al. and in this study. In order to compare them these selenium figures [13] are reproduced on the next page (Table 5).

Presumably these differences, up to more than two decades when compared to Varo et al. and this study (Table 2), are due to difficulties with the analytical procedure for selenium.

Varo et al. also report arsenic results for 13 fruit types from the Finnish market. Although their limit of determi-

Table 4. Estimated contribution of fresh fruits to the daily dietary intake of some elements by Dutch adults aged 22–75 years

Element	Daily oral intake of some elements					
	Fresh fruit			Duplicate diet study		
	Median	Range		Median	Range	
Sodium (mg)	< 1.3	< 1.3	– 26 (2.6)	2355	900	–5590
Potassium (mg)	219	90	–645 (439)	2805	1000	–5500
Arsenic (μ g)	0.5	< 0.3	– 4.8 (1.5)	< 13	< 5	– 111
Selenium (μ g)	0.3	< 0.1	– 4.1 (1.3)	37	12	– 145
Tin (mg)	< 0.006	< 0.003	– 0.20 (0.013)	< 0.21	< 0.09	– 9.81

Calculation basis: average fresh fruit intake 129 g (13) and median and range levels of the products. Daily intake measured for 18–74 year old adults ($n=110$) in the 1984/1985 duplicate diet study [2–4] for comparison. Values in brackets are 90th percentile

Table 5. Selenium data from reference [13]

Fruit	Se content (μ g/kg)	
	Mean	Range
Apples	9	3– 60
Bananas	44	10–170
Grapes	28	0–200
Mandarins	170	130–210
Oranges	35	10–240
Pears	12	6– 80

nation for arsenic of 10 μ g/kg fresh product makes comparison with this study difficult, it can be concluded that results agree fairly well but are lower in this study. Data for tin levels in fresh fruits were not found in the literature.

Recently results were published [15] of a nationwide dietary record survey carried out in The Netherlands in 1987/1988, including 5898 Dutch individuals aged 1–75 years. From this database, the average daily fresh fruit consumption for those 22 years and over has been calculated as 129 g. Based on this figure and the elemental composition of fresh fruits (Table 3), a worst-case calculation has been made of the fresh fruit contribution to the total oral diet of the elements studied. The outcome is given in Table 4 where the oral intake data are also reproduced for the 1984/1985 24-h duplicate diet study involving 110 adults aged 18–74 years [2–4]. Results, median and 90th percentile, clearly indicate, that the contribution of fresh fruit consumption to the oral daily intake of the nutrients sodium and selenium and of the contaminants arsenic and tin in general is marginal.

Fresh fruit consumption, with 8% calculated for the median and 16% for the 90th percentile, contributes substantially to the average daily oral intake of potassium. According to the German Association for Nutrition [16], the recommended potassium intake for an adult is 3000–4000 mg/day. Keeping this in mind, and the fact that fresh fruits are very low in element contaminants (this study and [7]), individuals are well advised to improve their potassium status by eating more fresh fruit.

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