Nitrate and Nitrite Content of Market Potatoes

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Ninety-nine samples of retail market potatoes from New York City, Chicago, the Washington, D. C., area, Grand Forks, N. D., and Wenatchee, Wash., were analyzed for nitrate by the nitroxylenol distillation method and nitrite by diazotization. An overall average of 120 ppm of nitrate and 0.44 ppm of nitrite was found, with S = 64 and 0.36, respectively. Significant differences in nitrate content existed among markets (Wenatchee highest at 139, Grand Forks lowest at 82) and area of growth (Idaho highest at 151, north central lowest at 80). A significant positive correla-

Attention has recently been directed to the nitrate and nitrite content of our food and water supplies by an awareness of increasing levels of nitrate and (potentially) nitrite in the environment. The relation of these factors to infant methemoglobinemia and to the possibility of formation of carcinogenic nitrosamines has been reviewed (Phillips, 1971; National Research Council, 1972; Wolff and Wasserman, 1972) and will not be discussed here.

Nitrates are normal constituents of plant materials; there is a considerable amount of analytical data in the literature (Achtzehn and Hawat, 1969; Ashton, 1970; Fogden and Fogden, 1969; Jackson *et al.*, 1967; Rooma, 1971) on average values and ranges for many of our foodstuffs, including prepared infant foods. Of the common vegetables beets, celery, endive, kale, radishes, lettuce, spinach, turnips, and broccoli are usually reported to exceed 1000 ppm of nitrate (fresh basis), and infant foods containing these vegetables may be correspondingly high in nitrate.

A recent instance of relatively high levels of nitrate and nitrite in a lot of dried potatoes intended for further processing has prompted an inquiry into the nitrate and nitrite content of potatoes as available to the U. S. consumer.

The relatively sparse data available in the literature on nitrate levels in potatoes are summarized in Table I. Data of Gilbert *et al.* (1946) are not included because the potatoes used in their work were from soils known to produce vegetables of abnormally high nitrate content. No information concerning variety or origin of the potatoes is provided in any of the work listed in Table I.

There are likewise relatively few data available on the effects of nitrogen fertilization on the nitrate content of potato tubers. Possibly the most extensive paper is that of Hlavsová *et al.* (1970). Three Czech potato varieties were grown under eight fertilizer treatments, replicated six times. It was concluded that the nitrate content of the tuber was dependent both on the variety and on the amount of fertilization, with the highest nitrate values resulting from the heaviest fertilization. It was also noted that nitrate levels in tubers increased more rapidly than yield. The well-known decrease in solids content with fertilization was also evident in their data.

Selected data from the Czech study are shown in Table II. The original paper also includes data on yield, solids content, nitrogen removal, and several additional fertilization levels.

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tion (r = 0.25) was found for all samples between nitrate and nitrite content. Dehydrated potato flakes from eight varieties grown under four fertilization levels were analyzed. Nitrate levels (fresh weight basis) were strongly dependent upon variety; the average nitrate level for the eight varieties was significantly higher for the highest level of fertilization than other levels. No nitrite was found. The nitrate and nitrite contents of the potatoes are not considered excessive, but varietal and agronomic influences preclude direct comparison with the few literature values.

A limited sampling of fresh potatoes was made by USDA personnel from retail markets in five areas of the U. S. The nitrate and nitrite content of these samples has been determined and the results are presented and discussed in this paper. In addition, a series of samples of dehydrated potato flakes was made available to us which had been processed from potatoes (eight varieties) grown in the Red River Valley at four levels of fertilization. These samples have also been analyzed for nitrate and nitrite content.

MATERIALS AND METHODS

Nitrate Analysis. Four published procedures for nitrate determination were investigated. Two, requiring preliminary reduction to nitrite followed by diazotization to produce azo dyes (Middleton, 1957; Nelson *et al.*, 1957) were found to produce results variably lower than the two described below and were dropped from further consideration. The two procedures selected for final evaluation were the nitrate electrode and the nitroxylenol procedure.

For the electrode procedure the standard addition method was used, as described by Westcott (1971) using a Beckman nitrate electrode.

The nitroxylenol method of Lipp and Dölberg (1964) was employed in which 3,4-dimethylphenol is nitrated and the nitroxylenol is separated by distillation. The procedure as used is as follows. Pipet a 1.0-ml sample or standard into an iodine flask, add 0.1 g of 3,4-dimethylphenol and 10 ml of concentrated sulfuric acid, stopper the flask, and wait 2 min. Add 30 ml of distilled water, cool in cold tap water, hold 30 min, and transfer the entire reaction mixture with washings to a micro-Kjeldahl steam distillation apparatus. Collect the distillate in a 25-ml volumetric flask containing 3 ml of 5% sodium hydroxide. Distil to just below the mark and make to volume with distilled water. The absorbance at 430 nm is determined with a Beckman B spectrophotometer.

Nitrite Analysis. Two diazotization methods were investigated. One, using α -naphthol and sulfanilic acid, forms with nitrite a yellow dye [orange I (Middleton, 1957)]. The other, using 1-naphthylamine and sulfanilic acid, forms a red azo dye (Nelson *et al.*, 1954). Since both methods gave essentially the same results in preliminary work 1-naphthylamine was selected since the reagents are commercially available in premixed measured quantities. Further, the red dye differs more from the natural yellow color of the potato juice. The procedure is as follows. Four milliliters of potato juice or standard is pipetted into a 25-ml cylinder, 21 ml of distilled water is added, and the contents of a NitriVer (Hach Chemical Co., Ames, Iowa) powder pillow is added. A light red color develops and,

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Table I. Nitrate Content of Potatoes

No. of sam- ples	Avg, ppm NO ₃	Range	Reference
5	77	40106ª	Richardson (1907)
1	63		Wilson (1949)
5	104	34–143 ^b	Jackson et al. (1967)
?	40	3070 ^a	Achtzehn and Hawat (1969)
19	130	56-454	Hlavsová et al. (1969)
52	77.21		Subbotin ef al. (1970)
15	17.6		Rooma (1971)

^a In her review, Ashton (1970) erroneously quotes Richardson's range for potatoes at 39–119 ppm and Achtzehn and Hawat's (1969) range as 10–30 ppm, instead of the correct 30–70 ppm of nitrate. ^b In his review, Phillips (1971) erroneously quotes Jackson's range for potatoes at 8–142 ppm of nitrate nitrogen, instead of the correct 8–32 ppm of nitrate nitrogen.

Table II. Effect of Fertilization on Nitrate Content of Potatoes

Fertilization, kg/Ha					content in g/kg fresh	
Manure	N	P ₂ O ₃	K₂O	Saskia	Krasava	Blanik
0	0	0	0	122.7	123.7	67.2
250	0	0	0	133.2	124.8	80.0
250	40	40	80	134.5	116.7	94.7
250	80	40	80	128.7	152.0	84.5
250	120	40	80	196.8	190.5	106.8

^a From Hlavsová et al. (1970).

after 15 min, the absorbance is measured at 525 nm. A blank that contains the sample but not the reagents is employed.

After the research described in this paper was completed, it was learned that α -naphthylamine has been classified as a carcinogen under the "Emergency Temporary Standard on Certain Carcinogens" (Stender, 1973). We have discontinued use of this procedure and for subsequent research are using, for nitrite analysis, the method described by Schall and Hatcher (1968). This procedure uses sulfanilamide with N-(1-naphthyl)ethylenediamine for the coupling reagent, as earlier described for analysis of NO₂ in air by Jacobs and Hochheiser (1958). Attention of laboratories using α -naphthylamine is directed to the recommended precautions for laboratory workers handling carcinogenic aromatic amines published by the Chester Beatty Research Institute (1966).

Potato Samples. Potatoes in 5- or 10-lb bags as packed were purchased from supermarkets and other retail stores in five areas of the U. S. (New York City, Chicago, the Washington, D. C., area, Grand Forks, N. D., and Wenatchee, Wash.) in late November-early December, 1971, by ARS Market Quality personnel and immediately shipped to this laboratory, where they were stored in their original bags at 10° and 67% RH until analysis. A total of 99 samples was obtained.

The samples were prepared for analysis by selecting five potatoes at random from the 5- or 10-lb lot, thoroughly washing, scrubbing, drying, and grinding them, and separating the juice by means of a Juicerator (Acme Manufacturing Co., Lemoyne, Pa.). A sheet of filter paper was fitted on the centrifuge screen of the machine to retain fine particles. The juice was divided into two portions. One portion was frozen (whole juice) and held for future use; the other portion was deproteinized by heating to 95° , filtered, and then frozen. Experiments indicated deproteinization did not affect the nitrate content of the juice. The nitrite content before and after deproteinization was not compared because the turbid nature of whole juice prevented nitrite analysis without a clarification step such as deproteinization.

RESULTS AND DISCUSSION

Accuracy and Precision of Methods. The nitrate electrode and the nitroxylenol method were each applied to ten replicate potato juice samples with a standard deviation of 5.02 and 3.12 ppm of nitrate, respectively. Recovery of added nitrate for 76 samples was 91.5% for the nitrate electrode and 106.5% for the chemical method for 37 determinations. Standard deviation for nitrite in ten replications was 0.012 ppm, with 84.3% recovery of added nitrite.

To obtain a measure of the precision of the extraction and analysis, five tubers from one sample were quartered lengthwise and four samples were made containing one quarter from each. Results by the nitroxylenol method were 77.9, 83.0, 86.6, and 88.5 ppm, S = 4.66.

Variability within individual 10-lb lots of potatoes was examined. Five samples of five tubers were taken at random from each of three lots of potatoes. Single lengthwise quarters from each of the five potatoes were combined and analyzed for nitrate (nitroxylenol) and nitrite. Results appear in Table III. It is evident that individual variation in nitrate and nitrite content among tubers requires that a more elaborate sampling scheme be employed to obtain results truly representative of a large lot of potatoes.

All tuber samples were analyzed for nitrate by both the electrode method and the nitroxylenol method, and all results are expressed as parts of nitrate per million of potato juice. The average values found by each method, corrected for recovery of added nitrate as given above, are seen in Table IV. Analysis of variance was calculated for the uncorrected results and also for the corrected values. Table IV shows the F ratio values associated with methods and materials for the uncorrected data and also for the "corrected" data. In both cases, the difference due to methods

Table III. Variability of Nitrate and Nitrite Content among Five Samples of Three Lots of Tubers

	Ni	trate, pp	m	N	itrite, ppr	n
		Lot			Lot	
Sample ^a	A	В	С	A	В	С
1	237	56	84	0.84	0.32	0.35
2	269	82	52	1.11	0.44	0.08
3	181	69	74	0.81	0.27	0.27
4	172	72	46	1.10	0.26	0.29
5	177	81	132	0.90	0.19	0.57
Mean	207.2	72.0	77.6	0.95	0.30	0.31
S	43.4	10.6	34.2	0.14	0.09	0.18
CV (%)	21.0	14.7	44.0	15.0	31.4	56.4

 $^{\rm a}$ Each sample consisted of one lengthwise quarter from each of five tubers, which were selected at random from a 10-Ib lot of potatoes.

Table IV. Comparison of Nitrate Electrode and the Nitroxylenol Method

		Mean values		
		Uncorrected, ppm	Corrected, ppm	
Nitrate electrode		140.4	153.5	
Nitroxylenol		127.6	119.6	
		Analysis of varia		
Variance associated with	DF	Uncorrected, F	Corrected, F	
Methods	1	29.49	158.2	
Materials	97	35.95	29.63	
Error	97			
Whole Set	195			

" All F ratios significant at the 1% level.

Table V. Nitrate Content of Market Potatoes

	No. of samples	Range	Mean,∝ ppm	s
All samples	99	7–362	120	63.9
B	y location	of market		
Wenatchee, Wash.	14	52-238	139 a	69.4
Washington, D. C. ^b	24	28-362	137 ab	83.0
New York, N. Y.	28	31-247	127 abc	59.1
Chicago, III.	15	26188	105 d	48.6
Grand Forks, N. D.	18	7–181	82 e	54.9
	By va r	ietv⁰		
Russet Burbank	37	49-228	127	52.1
Red Bliss	4	60-186	118	60.2
Norland	4	76~98	85	9.3
Red Pontiac	8	7-159	61	58.8
Norgold Russet	2	148-156	152	5.7
Kennebec	2	47-64	55	12.0
В	y location	of origin ^o		
Maine	19	31-362	112	90.4
Long Island	12	44-222	114	51.9
Florida	4	60-186	118	60.1
Idaho	15	61-227	151	41.2
New Jersey	4	114-246	195	57.2
Pennsylvania	2	114-187	150	51.7
New York	2	94-111	102	12.0
Minnesota	11	12-98	62	31.8
Wisconsin	9	49–158	117	41.5
North Dakota	5	7-94	54	44.5

^a Means followed by the same letter are not significantly different. For probability levels see text. ^b Actually purchased in Beltsville, College Park, and Berwyn Heights, Md. ^c As stated on bag label.

Table VI. Nitrate Content of Potatoes from Different Areas

_	Area	Sample no.	Mean,ª ppm	S
	Idaho	15	151 a	41.2
	East Central	20	133 ab	58.4
	Maine	19	112 c	90.5
	North Central	25	80 d	46.0

 $^{\alpha}$ Means followed by the same letter are not significantly different at the 1% level, except for Maine and North Central, which differ at the 5% level.

is significant at the 1% probability level; however, "correcting" the values raises the variance due to methods to five times that due to sample differences and hence unresolved factors are present which influence the accuracy of one or both methods. Data from the chemical method were selected for reporting because it has been more widely used in the past and because the colored material that is measured is separated by distillation from the potato extract. The data reported in this paper are corrected for the 106.5% recovery.

Nitrate Content. Table V gives the mean corrected values for 99 potato samples, grouped by location of the market, by the variety insofar as stated on the package, and by geographic origin as shown on the package. Duncan's Multiple Range Test (Duncan, 1955) for significance of differences among means was applied to the data classified by location of market, since it alone included all samples. Results are shown in the table. All indicated differences were significant at the 1% probability level, except those between New York City and Chicago and Chicago and Grand Forks, which were significant at the 5% level.

Although classification of the data by location of market has no independent significance since variety preferences of consumers and proximity to growing areas control

Table VII. Extremes in Nitrate Content of Market Potatoes

Market	Origin	Variety	Nitrate, ppm
	Highe	st	
Washington	Maine	"Round White"	362
Washington	Maine	"Round White"	332
New York	New Jersey		247
New York			239
Wenatchee			238
Wenatchee			236
New York	ldaho	Russet Burbank	228
Wenatchee			228
Washington	Long Island	"Round White"	222
Washington	New Jersey	"Round White"	212
	Lowes	st	
Grand Forks	N. Dakota	Red Pontiac	7
Grand Forks	N. Dakota	Red Pontiac	8
Grand Forks	Minnesota	Red Pontiac	12
Grand Forks	Minnesota	Red Pontiac	14
Chicago	Minnesota		26
Washington	Maine	"Round White"	28
New York	Maine		31
New York	Long Island		44
Grand Forks	Minnesota	Kennebec	47
Chicago	Wisconsin	Russet Burbank	49

Table VIII. Nitrite Content of Market Potatoes

Market	No. of samples	Range, ppm	Mean,ª ppm	S , ppm
Washington, D. C.	24	0-1.71	0.56 a	0.54
Chicago, III.	15	0-1.12	0.54 ab	0.28
Grand Forks, N. D.	18	0-1.31	0.48 ab	0.37
Wenatchee, Wash.	14	0.08-0.61	0.35 c	0.22
New York, N. Y.	28	0-0.89	0.26 c	0.26
All samples	99	0-1.71	0.44	0.36

 a Means followed by the same letter are not significantly different at the 1% level, except for Grand Forks and Wenatchee, which differ at the 5% level.

these values, it is included here to permit its use by those interested in area differences in nitrate ingestion.

To permit an analysis of the data according to the origin of the potatoes, results were grouped into four categories: Maine, Idaho, East Central (New York, New Jersey, Pennsylvania), and North Central (Minnesota, North Dakota, Wisconsin). As shown in Table VI, means among all areas except Idaho and the East Central differed significantly. None of the potatoes from the Wenatchee markets were received with indication of origin; few of the potatoes obtained in New York carried any varietal identification. The term "round white" did appear on ten samples from the Washington area, but this is not a variety name. No statistical evaluation of varietal influence was done because of the limited sample size.

In Table VII are listed the ten samples with the highest nitrate content and the ten with the lowest. The wide range of the data, implied by the relatively large standard deviation values in Tables V and VI, is quite evident.

Nitrite Content. The nitrite content of all of the potato samples was relatively low. None contained over 2 ppm and only 6 of 99 fell between 1 and 2 ppm. For this reason the data are shown in Table VIII classified only by location of the market. Although differences between market means are significant by Duncan's Multiple Range Test, we have no explanation why this is so. It is noteworthy that the markets do not fall in the same order for nitrites as for nitrates. Certainly none of the nitrite values of the potatoes analyzed here are significant from the public health standpoint.

The data were tested for correlation between nitrate

Table IX. Correlation of Nitrate and Nitrite **Content of Market Potatoes**

Grouping	No. of samples	r	F^a
All samples	99	0.255	6.78*
	By market		
Washington	24	0.042	0.040
New York	28	0.293	2.45
Chicago	15	0.688	11.69**
Grand Forks	18	0.897	66.10**
Wenatchee	14	0.495	3.89
	By origin		
Maine	19	0.065	0.074
Idaho	15	0.284	1.14
North Central ^b	25	0.895	93.01**
East Central ^e	20	0.106	0.21

^a Significance: no asterisk, above 5% probability level; *, 5% level; **, 1% level. North Dakota, Minnesota, Wisconsin. ^c Pennsylvania, New York (including Long Island), New Jersey.

Table X. Effect of Variety and Fertilization on Nitrate Content of Dehydrated Potatoes

		Fertilizatio	on level a	dded, lb/A	4
Nutrient	A	В	С	D	
N	0	38	56	92	
Р	0	56	108	212	
К	0	86	112	164	
Variety	Nitrate	content, p	op m fresh	basis ^a	Avg
Norgold	81.9	82.8	98.0	85.6	87.1 a
Norland	67.8	67.6	58.8	70.8	66.3 b
Pontiac	51.5	64.9	68.5	74.3	64.8 bc
Norchip	55.3	47.1	69.2	87.0	64.6 bcd
La Chipper	75.5	43.8	60.7	56.5	59.1 bcd€
Kennebec	52.8	51.8	43.7	75.9	56.1 e
Norchief	43.0	49.2	56.7	61.1	52.5 e
ND 7196	37.2	49.5	52.3	67.9	51.7 e
Average	58.1 A	57.1 A	63.5 A	72.4 B	

^a Means followed by the same letter are not significantly different from each other at the 5% level.

Table XI. Analysis of Variance of Data from Table X

Variance associated with	DF	\$\$	MS	Fª
Variety	7	3585.632	512.23	5.31**
Treatment	3	1175.023	391.67	4.06*
Error	21	2026.649	96.51	
Whole set	31	6787.305		

Asterisks indicate significance: *, 5% level; **, 1% level.

content and nitrite content for several groupings of the samples. As summarized in Table IX, a significant correlation exists for all samples as a group. When considered in terms of the individual markets, the correlations were highly significant for Chicago and Grand Forks but not for the others. Grouping the samples by state (or area) or origin produced a highly significant correlation for the "north central" samples only. Examination of sample data showed that most of the potato samples obtained in the Chicago and Grand Forks markets originated in the "north central" area, Minnesota, North Dakota, and Wisconsin. This, of course, does not explain why potatoes from this area show this relationship. It must be noted that no data are available on the origin of the Wenatchee potatoes.

Relation of Nitrate Content to Variety and Fertilization. Thirty-two samples of dehydrated potato flakes were made available to us for nitrate and nitrite analysis. (The source was not the one referred to in the introductory remarks as processing material of high nitrate content.) These flakes were produced from eight varieties of potatoes grown with four levels of fertilization at the Red River Valley Potato Growers Association research farm in Grand Forks, N. D. Fertilizer levels and the results of analysis for nitrate content are shown in Table X, expressed on the fresh potato basis for comparison with other results given here. No information is available on the loss in processing, but it is noteworthy that the overall average value, 51.7 ppm of nitrate, is not far from the 59.4 average value for Minnesota-North Dakota (calculated from data in Table V), which are the lowest found in this study for any area. No nitrite was found in any sample.

An analysis of variance was carried out with the data in Table X, with the results seen in Table XI. Variance associated with variety was significant at the 1% probability level, and that for fertilization treatment at the 5% level. Duncan's Multiple Range Test further evaluates the differences among means.

In view of the strong influence of variety and location seen in the work reported here and of fertilization level (Hlavsová et al., 1970), there is little to be gained in comparing our data with published values (Table I) which include results from six studies from several countries over a period of 64 years.

ACKNOWLEDGMENT

The authors appreciate the cooperation of members of the Market Quality Division, Agricultural Research Service, in collecting samples of market potatoes, of Jack D. Westover, Pillsbury Co., Minneapolis, for the samples of potato flakes, and of the technical assistance of Robert Mink.

LITERATURE CITED

- Achtzehn, M. K., Hawat, H., Nahrung 13, 667 (1969).
 Ashton, M. R., "The Occurrence of Nitrates and Nitrites in Foods," British Food Mfg. Ind. Res. Ass., Lit. Survey No. 7, 1970.
- Chester Beatty Research Institute, London, "Precautions for workers who handle carcinogenic aromatic amines," Institute of Cancer Research, Royal Cancer Hospital, London, 1966.

- Cancer Research, Royal Cancer Hospital, London, 1966.
 Duncan, D. B., *Biometrics* 11, 1 (1955).
 Fogden, L. A., Fogden, M. W., *J. Ass. Pub. Anal.* 7, 133 (1969).
 Gilbert, C. S., Eppson, H. F., Bradley, W. B., Beath, O. A., *Wyo. Agr. Exp. Sta. Bull.* no. 277 (1946).
 Hlavsová, D., Tuček, J., Turek, B., *Cesk. Hyg.* 15, 203 (1970).
 Jackson, W. A., Steel, J. S., Boswell, V. R., *Proc. Amer. Soc. Hort. Sci.* 90, 349 (1967).

- Jacobs, M. B., Hochheiser, S., Anal. Chem. **30**, 426 (1958). Lipp, G., Dölberg, U., Beitr. Tabakforsch. **2**, 345 (1964). Middleton, K. R., Chem. Ind. (London) 1147 (1957). National Research Council, Committee on Nitrate Accumulation, "Accumulation of Nitrate," National Academy of Sciences, Washington, D. C., 1972.
- Nelson, J. L., Kurtz, L. T., Bray, R. H., Anal. Chem. 26, 1081 (1954). Phillips, W. E. J., Food Cosmet. Toxicol. 9, 219 (1971). Richardson, W. D., J. Amer. Chem. Soc. 29, 1757 (1907). Rooma, M., Gig. Sanit. 36(8), 46 (1971). Schall, E. D., Hatcher, D. W., J. Ass. Offic. Anal. Chem. 51, 763

- (1968).
- Stender, J. H., Fed. Regist. 38, 10929 (1973). Subbotin, F. N., Masol'nikova, T. K., Tomilina, K. A., Vop. Pitan.
- **29**(5), 65 (1970). Westcott, C. C., Food Technol. **25**, 709 (1971).
- Wilson, J. K., Agron. J. 41, 20 (1949).
- Wolff, I. A., Wasserman, A. E., Science 177, 15 (1972).

Received for review April 16, 1973. Accepted July 30, 1973. Mention of proprietary products is for identification only and does not imply endorsement by the U. S. Department of Agriculture over others of a similar nature not named.