Elements in Major Raw Agricultural Crops in the United States. 1. Cadmium and Lead in Lettuce, Peanuts, Potatoes, Soybeans, Sweet Corn, and Wheat

Karen A. Wolnik, Fred L. Fricke, Stephen G. Capar,* George L. Braude, Milton W. Meyer, R. Duane Satzger, and Evelyn Bonnin

Six raw agricultural crops (lettuce, peanuts, potatoes, soybeans, sweet corn, and wheat) were collected from major U.S. growing areas uncontaminated by human activities other than normal agricultural practices and analyzed for Cd and Pb by using differential pulse anodic stripping voltammetry. Handling, preparation, and analysis of the 1645 samples were performed under carefully controlled conditions. Mean concentrations of Pb and Cd (μg/g wet weight) were, for lettuce, 0.013 and 0.026, for peanuts, 0.10 and 0.078, for potatoes, 0.009 and 0.031, for soybeans, 0.042 and 0.059, for sweet corn, 0.0033 and 0.0031, and, for wheat, 0.037 and 0.043, respectively. Most of these values, considered to be background, are much lower than those reported previously.

Over the last several years, increasing amounts of sewage sludge and other wastes have been applied to agricultural land to provide plant nutrients for crops and pastures. Concern has been expressed (Jelinek and Braude, 1978) about the potential contamination of such crops by the toxic trace elements, organic chemicals, and pathogens frequently present in waste. These contaminants may originate in the effluent from industrial operations and from street runoff. Cadmium and lead are the elements of most concern because of their potential for toxicity or accumulation in plants and animals. Some sludge contains high concentrations of these elements, which are retained in the treated topsoil when the sludge is applied to land. Soils and crops may also be exposed to contamination by Cd and Pb from such sources as aerial fallout near smelters or other industries and by automotive traffic near highways and in urban areas.

Cadmium in soil is translocated in varying amounts to edible portions of crops (Council for Agriculture, Science, and Technology, 1976, 1980). Lead is absorbed by plants, and its absorption may be increased under some conditions. Cattle that graze on pastures treated with contaminated sewage sludge can absorb and retain Cd and Pb in selected tissues (Kienholz et al., 1979).

To protect the public health, the U.S. government and several states have established regulations and guidelines that limit the levels of Pb and Cd (and other contaminants) in wastes applied to soil on which crops are grown and that specify proper application practices (*Fed. Regist.*, 1979a). However, reliable base-line data for elements in crops from nonpolluted areas are not available. These background values are needed for evaluating the toxicological significance of consumption of these elements and their possible increase due to food processing. The range of normal background concentration of these elements must be known to set action levels (if needed).

The Food and Drug Administration (FDA) conducts annual surveys to determine the trace element content of foods consumed by the average person or by selected groups. The surveys also measure other nutrient and toxic substances. In these "market basket studies", all foods are collected at the retail market level. These foods may have undergone many commercial food handling and processing steps, which could result in contamination. Before analysis, they were prepared (scrubbed, peeled, etc., as in a kitchen). Analytical results from these studies therefore do not reliably represent background levels of these elements. Until March 1982, food group composites rather than individual foods or crops were used, diluting the effect of contamination of samples (Compliance Program Evaluation, 1978). The surveys presently conducted by FDA (and not reported as yet) are based on new dietary intake data and involve the analysis of 234 individual food items.

For several years, including Fiscal Year 77, FDA analyzed a limited number of selected individual foods for toxic elements, such as Cd, Pb, and Zn (Compliance Program Evaluation, 1977). Foods, however, were always collected from warehouses and commercial distribution centers and not from fields.

The U.S. Geological Survey in Denver, CO, collected and analyzed a variety of crops for a large number of elements (Shacklette, 1980). However, no attempt was made to select only "clean" sites away from major sources of contamination such as roads or industries or where wastes (sludges, etc.) may have been applied. Sites were not selected to represent soils in major crop-producing areas. Kaferstein (1980) published a survey of Pb, Cd, and Hg levels in a variety of foods in Germany which had been analyzed by central and regional laboratories. A large number of samples were involved, but the accuracy and precision of the data are, according to Kaferstein, dependent on the practices of the individual laboratory and are quite variable. Additional studies have been reported in other countries [Sweden (Andersson and Pettersson, 1981); West Germany (Pfeilsticker and Maskard, 1975); England (Thomas et al., 1972); The Netherlands (Reith et al., 1974)], but a carefully controlled large-scale determination of background values with low detection limits has, to our knowledge, not been reported elsewhere.

The purpose of the present study, a joint effort between the U.S. Department of Agriculture (USDA), FDA, and Environmental Protection Agency (EPA), was to develop high-quality data on the background levels of Cd, Pb, and other elements in selected crops that provide the major portion of Cd in the human diet taken from major production areas (*Fed. Regist.*, 1979b). Corresponding soils were also collected from each site to develop soil–plant correlations for the elements studied that will be reported later (Holmgren et al., 1983).

Crops selected for sampling and analysis are those which are either major sources of Cd for the human diet and/or

Food and Drug Administration, Cincinnati, Ohio 45202 (K.A.W., F.L.F., R.D.S., and E.B.), and Washington, DC 20204 (S.G.C. and G.L.B.), and Soil Conservation Service, U.S. Department of Agriculture, Washington, DC 20013 (M.W.M.).

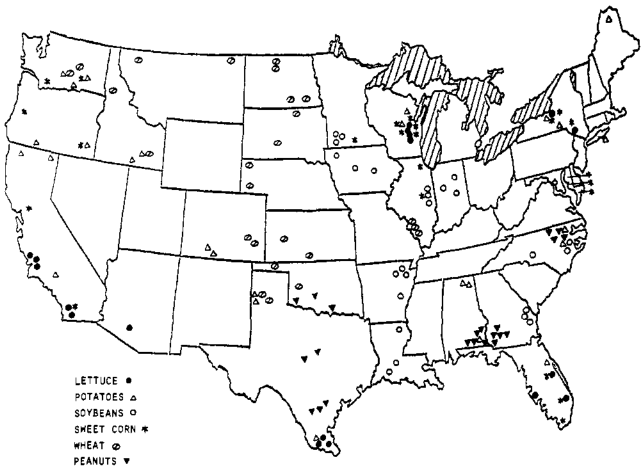

Figure 1. Sampling site locations: (*) corn; (△) potatoes; (⊘) wheat; (●) lettuce; (▼) peanuts; (○) soybeans.

are known to accumulate Cd, the element of primary toxicological concern. An attempt was also made to include different types of vegetables (leafy, root, legume), various grains, and the most important fruits, as this study continues. Special care was taken in the selection of sampling sites, in the handling, packaging, and shipping of the samples, and in the preparation and analysis of these samples in specially equipped laboratories. This paper presents the results of the first six crops analyzed.

MATERIALS AND METHODS

Site Selection. As shown in Figure 1, samples of the crops were collected in several of the main production areas of the United States. Strict limitations were placed on each individual site in an attempt to exclude the effect of more than minimum contamination by human activities, since they may affect the soil or crops. The sites were therefore more than 8 km downwind from power plants or smelters, at least 200 m from major roads, 100 m from current, abandoned, or other building sites, and 50 m from end rows and other areas where large quantities of fertilizers could have been deposited. In addition, information was obtained regarding the fertilization history of the site.

Crop Sampling. All crops were taken directly from the field and were sampled in sufficient quantities to provide a representative sample, such as 5 heads or bunches of lettuce, 10 potatoes, 200 heads of wheat, 80 pods of soybeans, 50 pods of peanuts, and 10 ears of sweet corn.

All samples were collected with plastic gloves and were packaged in plastic bags for shipment. Lettuce and sweet corn were shipped refrigerated with Blue Ice to avoid decomposition.

Sample Preparation. Distilled deionized water (DDW) with a measured resistance of 18 Mohm/cm (Milli-Q Water Purification System, Millipore Corp., Bedford, MA) was used exclusively. All equipment that contacted the samples during preparation was nonmetallic and was scrupulously cleaned (glassware with 30% HNO_3 and DDW and plastic ware with DDW). Laboratory ware was stored in a clean-air environment provided by laminar flow hoods equipped with 0.3-μm HEPA filters (Environmental Air Control, Inc., Hagerstown, MD). Bench tops were covered with sheets of poly(vinyl chloride). All sample handling subsequent to the washing procedure was done in a clean-air environment, and disposable polyethylene gloves were worn throughout the preparation procedure.

Stem ends were removed from lettuce samples and each leaf was thoroughly rinsed with DDW. Outer damaged leaves were discarded. Samples were finely chopped in a chopper (Hobart, Inc., Troy, OH) with the blade, bowl, and

Table I. Lead and Cadmium in Lettuce

			µg/g wet weight				
state	n	element	mean	median	minimum	maximum	% RSD[a]
AZ	14	Pb	0.020	0.016	0.003	0.051	77.8
		Cd	0.038	0.035	0.014	0.077	52.0
CA	42	Pb	0.009	0.008	0.001	0.026	59.6
		Cd	0.051	0.042	0.017	0.160	56.9
FL	23	Pb	0.035	0.029	0.009	0.078	62.8
		Cd	0.011	0.008	0.001	0.037	92.8
NY	24	Pb	0.005	0.004	0.002	0.011	53.9
		Cd	0.008	0.007	0.002	0.018	47.8
TX	23	Pb	0.008	0.007	0.003	0.018	56.9
		Cd	0.017	0.015	0.006	0.037	48.6
WI	24	Pb	0.008	0.006	0.003	0.023	58.4
		Cd	0.013	0.008	0.003	0.015	104.4
crop	150	Pb	0.013	0.008	0.001	0.078	112.7
		Cd	0.026	0.017	0.001	0.160	99.5
wwf[b]	150		0.0415	0.04	0.026	0.084	19.0

[a] Relative standard deviation. [b] Wet weight factor.

interior surface of the bowl cover coated with a vinylidene fluoride polymer (Kynar, Pennwalt Corp., Philadelphia, PA). Chopped samples were frozen in a shell freezer in glass freeze-dry flasks at −50 °C and freeze-dried (Labconco, Kansas City, MO). Freeze-dried samples were ground in a blender (Model 400-829303, Sears, Chicago, IL), equipped with a glass container, plastic blade holder, and hardened stainless blades, to pass a 0.42-mm polypropylene sieve. Composites of the individual samples were stored in acid-washed linear polyethylene bottles.

Peanut pods were cleaned with DDW, oven-dried at ≤50 °C, and shelled. The peanuts were covered with DDW, soaked overnight in small glass freeze-dry jars, freeze-dried, blender-ground, and stored in linear polyethylene bottles at −10 °C.

Potatoes were scrubbed with a plastic vegetable brush and DDW, peeled (as in home peeling) with a Kynar-coated stainless steel peeler, chopped, freeze-dried, and blender-ground.

Soybeans were removed from the pod, rinsed with DDW as quickly and thoroughly as possible, air-dried in a clean-air hood, and blender-ground.

Sweet corn samples were husked and rinsed with DDW. Kernels, including the germ, were removed from the cob with a plastic knife, freeze-dried, and blender-ground.

Wheat samples were dried in a forced air oven at <50 °C to facilitate removal of the grain from the head. The wheat was threshed and grain kernels were separated from the chaff with plastic drainers. Fine bits of chaff were removed with an air gun. Kernels were rinsed with DDW, soaked overnight in DDW, freeze-dried, and blender-ground.

Sample Analysis. *Moisture Determination.* For soybeans, the percent moisture was determined by oven-drying a portion of the ground composite to a constant weight. For other crops, percent moisture was determined by weighing the prepared sample before and after freeze-drying. A correction was made for residual moisture, which was determined on a portion of composite by Karl-Fischer titration in sweet corn and by oven-drying in the remaining crops.

Cadmium and Lead Determination. Sample composites were dry ashed with a sulfuric acid ashing aid, and Cd and Pb were determined by differential pulse anodic stripping voltammetry by using the procedure described and evaluated for these crops by Satzger et al. (1982). For every set of 15 samples analyzed, a duplicate sample, spiked sample recovery, and blank were carried through the procedure. Typical relative standard deviations for the

Table II. Lead and Cadmium in Peanuts

state	n	element	μg/g wet weight mean	median	minimum	maximum	% RSD[a]
AL	64	Pb	0.011	0.008	<0.0009	0.049	74.3
		Cd	0.050	0.040	0.019	0.144	59.7
GA	64	Pb	0.014	0.011	0.004	0.111	108.3
		Cd	0.042	0.037	0.010	0.099	52.3
NC	64	Pb	0.009	0.008	<0.0010	0.048	79.6
		Cd	0.091	0.076	0.019	0.247	58.7
OK	64	Pb	0.008	0.004	<0.0010	0.194	299.2
		Cd	0.118	0.095	0.018	0.480	71.0
TX	64	Pb	0.007	0.005	<0.0007	0.024	79.3
		Cd	0.091	0.072	0.010	0.588	91.0
crop	320	Pb[b]	0.010	0.007	<0.007	0.194	143.1
		Cd[b]	0.078	0.060	0.010	0.588	84.3
wwf[c]	320		0.8795	0.952	0.428	0.999	16.0

[a] Relative standard deviation. [b] Thirteen results below the minimum reported level. [c] Wet weight factor.

Table III. Lead and Cadmium in Potatoes

state	n	element	μg/g wet weight mean	median	minimum	maximum	% RSD[a]
AL	22	Pb	0.028	0.011	0.006	0.370	275.1
		Cd	0.027	0.027	0.011	0.042	33.4
CA	26	Pb	0.003	0.003	0.001	0.008	51.2
		Cd	0.038	0.016	0.002	0.182	115.5
CO	26	Pb	0.003	0.002	0.0002	0.008	65.0
		Cd	0.029	0.031	0.009	0.046	32.8
FL	15	Pb	0.003	0.003	0.001	0.005	44.6
		Cd	0.045	0.053	0.004	0.068	43.9
ID	26	Pb	0.010	0.009	0.002	0.031	61.6
		Cd	0.038	0.036	0.017	0.072	35.9
ME	26	Pb	0.009	0.009	0.002	0.019	47.4
		Cd	0.021	0.017	0.011	0.050	47.7
NC	26	Pb	0.005	0.004	0.002	0.019	72.9
		Cd	0.029	0.030	0.009	0.054	46.5
NY	26	Pb	0.010	0.009	0.002	0.023	64.0
		Cd	0.028	0.023	0.009	0.069	52.1
OR	28	Pb	0.003	0.003	0.001	0.007	50.0
		Cd	0.030	0.028	0.003	0.056	43.9
TX	24	Pb	0.028	0.012	0.008	0.280	203.3
		Cd	0.051	0.044	0.028	0.096	40.1
WA	26	Pb	0.009	0.008	0.003	0.020	49.6
		Cd	0.022	0.022	0.005	0.052	45.9
WI	26	Pb	0.004	0.003	0.0008	0.024	122.3
		Cd	0.025	0.020	0.008	0.049	43.9
crop	297	Pb	0.009	0.005	0.0002	0.370	292.6
		Cd	0.031	0.028	0.002	0.182	62.9
wwf[b]	297		0.193	0.193	0.126	0.259	12.7

[a] Relative standard deviation. [b] Wet weight factor.

crop analyses were 13% at 100 ng/g and 25% at 10 ng/g for Pb and 5% at 100 ng/g and 10% at 10 ng/g for Cd, on a dry weight basis.

Data Processing. Because of the large number of samples (1645 total) and the need to obtain extensive statistical analysis and (later) correlations with soil data, data processing was performed through the interactive computer language APL (Falkoff and Iverson, 1973) and the Statistical Analysis System (SAS, 1979, 1981). The computer system was an IBM 370/168 with APL.SV version 3.1 and was used in a time-sharing environment under the operating system MVS/SE2 Release 3.8. The SAS Release 79.5 was accessed by way of batch processing with job submission through APL. Interactive APL programs were developed for prompting, formating, storage on disk files, and submission of batch jobs for SAS processing of the analytical results. All elemental results were stored in terms of μg/g dry weight. Wet weight factors (wwf) were also stored for each sample. Wet weight values were obtained by multiplying the dry weight result by the wwf. The wwf reported can be used to obtain dry weights (divide values for elements by wwf) or moisture levels ($1 - \text{wwf} \times 100$). The SAS procedure UNIVARIATE was applied to each crop. UNIVARIATE produces a number of simple descriptive statistics including quantiles, vertical bar charts, normal probability plots, skewness, and median.

RESULTS AND DISCUSSION

Statistical data on Cd and Pb levels in the six crops examined are presented in Tables I–VI. More extensive

Table IV. Lead and Cadmium in Soybeans

state	n	element	μg/g wet weight mean	median	minimum	maximum	% RSD[a]
AR	33	Pb	0.046	0.035	<0.0016	0.323	117.0
		Cd	0.069	0.054	0.007	0.267	82.8
GA	42	Pb	0.011	0.010	0.003	0.040	61.9
		Cd	0.020	0.015	0.002	0.153	117.4
IA	42	Pb	0.045	0.037	0.015	0.223	71.7
		Cd	0.047	0.039	0.008	0.195	65.3
IL	38	Pb	0.043	0.039	0.010	0.113	59.5
		Cd	0.042	0.035	0.008	0.168	76.7
IN	41	Pb	0.044	0.042	0.009	0.088	45.2
		Cd	0.098	0.067	0.012	0.445	87.7
LA	39	Pb	0.070	0.052	0.014	0.271	70.7
		Cd	0.072	0.066	0.019	0.166	50.9
MN	43	Pb	0.049	0.037	0.010	0.204	75.7
		Cd	0.063	0.054	0.016	0.221	64.4
NC	44	Pb	0.027	0.021	0.008	0.124	78.4
		Cd	0.062	0.031	0.012	1.114	264.3
crop	322	Pb[b]	0.042	0.033	<0.0016	0.323	88.6
		Cd	0.059	0.041	0.002	1.11	131.8
wwf[c]	322		0.916	0.918	0.816	0.953	1.9

[a] Relative standard deviation. [b] One result below the minimum reported level. [c] Wet weight factor.

Table V. Lead and Cadmium in Sweet Corn

			µg/g wet weight				
state	*n*	element	mean	median	minimum	maximum	% RSD[a]
CA	21	Pb	0.006	0.002	<0.0002	0.034	156.3
		Cd	0.005	0.005	0.0004	0.011	52.7
FL	31	Pb	0.008	0.008	0.002	0.018	57.3
		Cd	0.005	0.0015	0.0002	0.039	192.2
IL	33	Pb	0.002	0.001	<0.0002	0.009	111.9
		Cd	0.002	0.0009	0.0002	0.006	93.2
MD	35	Pb	0.003	0.002	<0.0004	0.025	148.4
		Cd	0.003	0.001	<0.0003	0.020	166.6
MN	17	Pb	0.002	0.002	<0.0002	0.004	56.4
		Cd	0.001	0.001	<0.0003	0.005	103.0
NY	32	Pb	0.002	0.001	0.0001	0.014	121.5
		Cd	0.004	0.003	0.0005	0.015	82.7
OR	32	Pb	0.002	0.001	0.0005	0.005	68.7
		Cd	0.005	0.004	0.0007	0.013	59.2
WA	27	Pb	0.003	0.003	<0.0003	0.009	63.8
		Cd	0.002	0.001	<0.0001	0.016	137.5
WI	40	Pb	0.003	0.002	0.0009	0.009	67.8
		Cd	0.001	0.0009	<0.0002	0.007	106.0
crop	268	Pb[b]	0.0033	0.0019	<0.0002	0.034	126.1
		Cd[c]	0.0031	0.0017	<0.0001	0.0387	143.5
wwf[d]	268		0.214	0.215	0.083	0.468	28.4

[a] Relative standard deviation. [b] Eleven results below the minimum reported level. [c] Twenty-one results below the minimum reported level. [d] Wet weight factor.

Table VI. Lead and Cadmium in Wheat

			µg/g wet weight				
state	*n*	element	mean	median	minimum	maximum	% RSD[a]
CO	24	Pb	0.014	0.007	<0.0015	0.164	234.6
		Cd	0.030	0.024	0.006	0.071	51.5
ID	28	Pb	0.008	0.007	0.002	0.020	57.6
		Cd	0.022	0.021	0.005	0.043	44.6
IL	28	Pb	0.024	0.021	0.015	0.043	35.9
		Cd	0.043	0.037	0.012	0.092	47.5
KS	28	Pb	0.083	0.020	<0.002	0.716	173.7
		Cd	0.055	0.031	0.009	0.163	81.9
MT	26	Pb	0.065	0.045	<0.008	0.255	97.3
		Cd	0.064	0.053	0.015	0.146	61.4
ND	25	Pb	0.036	0.020	0.005	0.249	138.9
		Cd	0.074	0.053	<0.0017	0.207	83.0
NE	27	Pb	0.075	0.059	0.014	0.223	73.1
		Cd	0.056	0.047	0.014	0.148	60.2
OK	27	Pb	0.020	0.014	0.006	0.163	147.7
	28	Cd	0.029	0.025	0.007	0.063	53.3
SD	21	Pb	0.034	0.014	0.003	0.229	161.4
		Cd	0.029	0.019	<0.0018	0.101	89.9
TX	26	Pb	0.041	0.028	0.013	0.139	70.0
	27	Cd	0.043	0.028	0.012	0.111	64.7
WA	28	Pb	0.007	0.004	<0.0008	0.029	110.7
		Cd	0.024	0.023	0.011	0.055	39.0
crop	288	Pb[b]	0.037	0.017	<0.0008	0.716	168.5
		Cd[c]	0.043	0.030	<0.0017	0.207	82.3
wwf[d]	288		0.881	0.883	0.722	0.988	6.3

[a] Relative standard deviation. [b] Seventeen results below the minimum reported level. [c] Two results below the minimum reported level. [d] Wet weight factor.

listings are available from the supplementary material (see paragraph at end of paper regarding supplementary material). Results are given for each state where the samples were collected and overall values for the crop.

The minimum reported levels for Pb and Cd were 1 ng/g on a dry weight basis. Minimum levels listed in Tables I–VI on a wet weight basis vary depending on sample moisture content. In addition, matrix effects due to differences in ashing characteristics of some samples resulted in higher minimum reported levels for these samples. In instances in which the sample result was less than the minimum reported level, half of the minimum reported level was used in the statistical calculations.

Of the six examined, all but sweet corn are considered likely to accumulate Cd to levels greater than those of most other crops, especially if grown in Cd-contaminated soils. This is especially true for peanuts, soybeans, and lettuce and other green leafy vegetables. The mean Cd levels found, however, were relatively low.

Lead levels were also low in practically all samples, with only a few exceeding 0.5 µg/g on a wet weight basis.

Differences in Cd levels in crops from different states were occasionally evident. Relatively high levels of Pb were also found in individual samples, exceeding 1 µg/g of wet weight in a few instances. Overall statewide trends are not discernible. This is understandable because several cultivars were involved, often depending on the region in which the crop was grown. The cultivar frequently affects the uptake of Cd by the crop (Council for Agriculture, Science, and Technology, 1980), even if grown side by side

Table VII. Comparison of Mean Cadmium and Lead Levels[a] in Five Crops

reference	lettuce	potatoes	soybeans	sweet corn	wheat
		Cadmium			
this study	0.026	0.031	0.059	0.0031	0.043
FDA[b]	0.048	0.037	0.092	0.018	0.065
other studies	0.048[c]	0.0499[c]		0.0065[d]	0.066[e]
	0.0295[c]	0.0448[c]			0.096[e]
		Lead			
this study	0.013	0.009	0.042	0.0033	0.037
FDA[b]	0.075	0.038	0.095	0.018	0.115
other studies	0.029[d]	0.0666		0.22[d]	
	0.033, 0.62[c]	0.0749[c]			

[a] μg/g of wet weight. [b] Compliance Program Evaluation (1977). [c] Kaferstein (1980). [d] Shacklette (1980). [e] Andersson and Pettersson (1981).

in the same soils and geographical area. Other factors that could have contributed to variation in metal concentration were soil, climate, and fertilization practices.

Table VII compares the data from this study and other major surveys conducted by FDA or reported by other workers. Levels of Cd and Pb found in this study are generally much lower than those previously reported. A variety of factors may be responsible for this: selection of relatively uncontaminated fields, careful sampling and handling, and laboratory equipment and practices that reduce contamination of samples during analysis. An important consideration in evaluating the values for Pb found in other studies is whether the Pb in processed foods had been present when the crop was harvested from the field or had been added by canning, handling, packaging, or other food processing or distribution procedures.

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Supplementary Material Available: Complete tabulation of individual data (with part 2), additional statistics, histograms, and probability (SAS computer printouts) (12 pages). Ordering information is given on any current masthead page.

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Elements in Major Raw Agricultural Crops in the United States. 2. Other Elements in Lettuce, Peanuts, Potatoes, Soybeans, Sweet Corn, and Wheat

Karen A. Wolnik, Fred L. Fricke, Stephen G. Capar,* George L. Braude, Milton W. Meyer, R. Duane Satzger, and Roy W. Kuennen

Six raw agricultural crops (lettuce, peanuts, potatoes, soybeans, sweet corn, and wheat) were collected from fields in major U.S. growing areas and were analyzed for Ca, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Se, and Zn. Statistical frequency distributions of some of the major elements were normal.

In part 1 (Wolnik et al., 1983), the Pb and Cd content of six major agricultural crops was reported based on a large sampling and carefully controlled analytical program. While Cd and Pb are toxic and obviously undesirable in food crops, other major and minor elements are of interest for different reasons. Several elements, such as Ca, Fe, K, Mo, Na, Ni, and P, are essential for human and animal health, and knowledge about their levels in different raw foods will provide information on the nutritional adequacy of diets. Others, such as Cu, Se, and Zn, though essential,

Food and Drug Administration, Cincinnati, Ohio 45202 (K.A.W., F.L.F., R.D.S., and R.W.K.), and Washington, DC 20204 (S.G.C. and G.L.B.), and Soil Conservation Service, U.S. Department of Agriculture, Washington, DC 20013 (M.W.M.).