

# Elemental Composition of Soybean Meal and Interlaboratory Performance

Thomas W. Osborn

A large quantity of hexane-extracted soybean meal was prepared from Amsoy soybeans and analyzed for 87 elements in ten independent laboratories using a variety of techniques. A tabulation of the "best values" is included. This study has provided new data on all essential and toxic trace elements present in defatted soybean meal. The techniques used and the performance of the individual laboratories are evaluated. The performance of individual laboratories is highly variable. In general, single-element techniques, neutron activation analysis, and inductively coupled plasma emission compared favorably. Spark source mass spectrometry, the most comprehensive technique used, produced satisfactory results for some elements but was, generally, the least accurate of the quantitative techniques.

Current efforts to supplement or replace animal protein by soybean protein encounter questions about the levels of nutritional and toxic elements in soybean meal. Although data are available for some nutritionally important trace elements in soybeans (Liener, 1972) and for a few toxic trace metals in soy protein (Tanner et al., 1972; Furr et al., 1974) a comprehensive survey is not available. Therefore, a quantity of defatted soybeans was prepared for the purpose of obtaining a complete trace element profile of soybean meal. The fats and oils were extracted because their removal helped to prevent biological degradation during storage and because defatting the meal is the first step in preparing soybeans for human consumption. The soybean meal was used in this study to assess different analytical techniques and to evaluate the performance of individual laboratories.

## EXPERIMENTAL SECTION

**Preparation of the Soybean Meal.** Soybean meal was prepared from approximately 330 kg of Amsoy soybeans grown in Ross, Ohio, and harvested in 1973. The beans were carefully hand sorted to remove all rocks, dirt, and foreign organic material, e.g., weeds, discolored beans, and insects. The soybeans were then frozen at  $-196^{\circ}\text{C}$  and powdered with a carefully cleaned MIKRO-Pulverizer (type 1 SH). The grinding tips of the pulverizer were Haynes Stellite No. 1 composed of 55% Co, 30% Cr, 12.5% W, and 2.5% C. The powdered soybeans were collected in clean polyethylene bags.

The fats and oils were extracted from the ground meal with 4 kg of hot hexane for each kilogram of soybean meal. To reduce contamination from heavy particulate material, such as metal particles, some of the meal was allowed to settle from the hexane slurry and was discarded. The hexane extracted meal was uniformly spread on plastic trays, covered with cheesecloth, and placed in large forced-air drying ovens at  $66^{\circ}\text{C}$  for 16 h. The dried soybean meal was then transferred to large polyethylene sacks and stored at  $1^{\circ}\text{C}$ .

**Analytical Methods and Laboratories.** Samples of the soybean meal were dried at  $100^{\circ}\text{C}$  for 24 h, sealed in polyethylene vials, and sent to the laboratories. Laboratories 1-3 were independent Procter & Gamble laboratories; the others were commercial or contract laboratories. A variety of analytical techniques was used to determine the elemental composition of the soybean meal. The specific techniques used were: colorimetric, fluorescence, and wet chemical techniques (WC); flame and flameless atomic absorption (AA); spark emission spec-

Table I. Laboratories and Techniques Utilized

Laboratory	Technique used <sup>a</sup>
1	AA, WC
2	AA
3	NAA
4	ES
5	ICPS
6	SSMS
7	SSMS
8	AA, WC
9	AA, WC
10	NAA

<sup>a</sup> AA, atomic absorption and flameless atomic absorption; NAA, neutron activation analysis; ES, spark emission spectroscopy; ICPS, inductively coupled plasma atomic emission spectroscopy; SSMS, spark source mass spectrometry; WC, colorimetric, fluorescence, and wet chemical.

Table II. Results of Replicate Analyses of Defatted Soybean Meal

Element. anal.	Replicate	Mean concn. <sup>a</sup>
Ca, %	8	$0.223 \pm 0.008$
Mg, %	11	$0.325 \pm 0.010$
Fe, ppm	15	$138 \pm 4$

<sup>a</sup> Standard deviation reported.

troscopy (ES); neutron activation analysis (NAA); spark source mass spectrometry (SSMS); and inductively coupled plasma atomic emission spectroscopy (ICPS). The techniques used by the participating laboratories are listed in Table I.

## RESULTS AND DISCUSSION

**Homogeneity and Contamination.** The homogeneity of the meal was determined by analyzing a series of 100-mg samples for Ca, Mg, and Fe by atomic absorption in laboratory 1. The means and relative standard deviations for the analyses are reported in Table II. The small relative standard deviations (3-4%) for Ca, Mg, and Fe indicate a high degree of homogeneity at the 100-mg level.

The samples analyzed by spark source mass spectrometry and neutron activation analysis had very low levels of Cr (0.35 ppm), Co (0.24 ppm), and W (<0.1 ppm). The low W value indicates that contamination from the Haynes Stellite No. 1 during grinding was not detectable. A small quantity of soybean meal prepared by hand without the use of metal utensils was analyzed for Co, Cr, and Fe. The hand-prepared soybean meal contained 0.22 ppm of Co, 0.37 ppm of Cr, and 140 ppm of Fe. These values are the same within analytical error as those found in the large batch of soybean meal. The agreement of elemental abundances in samples prepared by different methods and the homogeneity of the meal indicate no detectable

The Procter & Gamble Company, Miami Valley Laboratories, Cincinnati, Ohio 45247.

Table III. Elemental Composition of Defatted Soybean Meal

Element	Best value, ppm <sup>a</sup>	No. of laboratories	Range	Techniques
Al	4 ± 1	5	3.6-6	ICPS, SSMS, NAA
Sb	0.03 ± 0.01	3	<0.01-<0.1	SSMS, NAA
As <sup>b</sup>	<0.04	5	<0.03-4.0	AA, ICPS, SSMS, NAA
Ba	4 ± 1	3	4	SSMS
Be	<0.01	2	<0.001-<0.01	SSMS
Bi	<0.02	2	<0.004-<0.02	SSMS
B	15	3	6-23	SSMS
Br	1.5	2	<0.4-1.5	SSMS, NAA
Cd <sup>b</sup>	<0.4	6	<0.06-<2.5	AA, ICPS, SSMS, NAA
Ca	2220 ± 80	7	0.218-0.5	AA, ICPS, SSMS, NAA
C (%)	43.8	1		WC
Ce	<0.1	3	<0.02-<2	SSMS, NAA
Cs <sup>b</sup>	<0.04	3	<0.02-<0.1	SSMS, NAA
Cl	105	5	6-1500	WC, SSMS, NAA
Cr	0.35	6	0.16-<0.5	AA, ICPS, SSMS, NAA
Co	0.24 ± 0.02	3	0.2-0.66	SSMS, NAA
Cu	20 ± 4	6	15-26	AA, ICPS, SSMS
Dy	<0.1	2	<0.04-<0.1	SSMS
Er <sup>b</sup>	<0.2	3	<0.04-<0.2	SSMS, NAA
Eu <sup>b</sup>	<0.04	3	<0.02-<0.1	SSMS, NAA
F	1.9	4	<0.04-23	WC, SSMS
Gd <sup>b</sup>	<0.07	3	<0.04-<0.1	SSMS, NAA
Ga <sup>b</sup>	<0.1	3	<0.01-<0.2	SSMS, NAA
Ge	<1	3	<0.01-<4	SSMS, NAA
Au <sup>b</sup>	<0.04	3	≤0.001-<0.04	SSMS, NAA
Hf <sup>b</sup>	<0.04	3	<0.02-<0.1	SSMS, NAA
Ho	<0.1	2	<0.01-<0.1	SSMS
H (%)	6.5	1		WC
In	0.03	4	0.03-<0.06	SSMS, NAA
I	0.84	3	≤0.01-100	WC, SSMS
Ir <sup>b</sup>	<0.02	3	<0.01-<0.1	SSMS, NAA
Fe	137 ± 10	7	129-280	AA, ICPS, SSMS, NAA
La <sup>b</sup>	<0.01	2	<0.005-<0.01	SSMS, NAA
Pb	1 ± 0.2	6	0.1-<5	AA, SSMS
Li	<0.1	2	<0.001-<0.1	SSMS
Lu <sup>b</sup>	<0.01	3	<0.01-<0.1	SSMS, NAA
Mg	3100 ± 200	6	25-0.62	AA, ICPS, SSMS
Mn	38 ± 3	5	20-41	AA, ICPS, SSMS, NAA
Hg	<0.06	5	<0.01-2.8	WC, AA, SSMS, NAA
Mo	2	4	0.47-2.8	AA, WC, SSMS, NAA
Nd	<0.1	2	<0.04-<0.1	SSMS
Ni	6	4	5-7.6	AA, ICPS, SSMS, NAA
Nb	<0.13	2	<0.1-0.13	SSMS
N (%)	7.72 ± 0.04	2	7.69-7.78	WC
Os	<0.1	3	<0.04-<0.5	SSMS, NAA
O (%)	26.9	1		WC
Pd <sup>b</sup>	<0.2	3	<0.1-<0.2	SSMS, NAA
P	7100 ± 400	5	1000-7800	WC, ICPS, SSMS
Pt	<0.1	3	<0.04-<2	SSMS, NAA
K (%)	2.52 ± 0.09	6	2.44-2.62	AA, ICPS, SSMS, NAA
Pr <sup>b</sup>	<0.1	2	<0.04-<0.1	SSMS, NAA
Re <sup>b</sup>	<0.02	3	<0.01-<0.02	SSMS, NAA
Rh	<0.2	2	<0.1-<0.2	SSMS
Rb	20	3	14-40	SSMS, NAA
Ru	<0.1	3	<0.04-<0.1	SSMS, NAA
Sm <sup>b</sup>	<0.04	3	<0.001-<0.04	SSMS, NAA
Sc <sup>b</sup>	<0.01	3	<0.001-<0.04	SSMS, NAA
Se <sup>b</sup>	<0.5	6	<0.01-2	WC, AA, SSMS, NAA
Si	140 ± 40	3	100-170	ICPS, SSMS
Ag <sup>b</sup>	<0.05	3	<0.01-<0.05	SSMS, NAA
Na	4 ± 1	6	0.7-6.8	AA, ICPS, SSMS, NAA
Sr	2.2	2	2-2.4	SSMS
S	5000 ± 400	3	0.06-0.55	WC, SSMS
Ta <sup>b</sup>	<0.2	3	<0.1-<0.2	SSMS, NAA
Te	<0.04	3	0.01-<3	SSMS, NAA
Tb <sup>b</sup>	<0.1	3	<0.01-<0.1	SSMS, NAA
Tl	<0.02	2	<0.01-<0.02	SSMS
Th	<0.03	3	<0.01-<4	SSMS, NAA
Tm	<0.1	3	<0.1-<0.04	SSMS, NAA
Sn	≤0.04	2	<0.04	SSMS
Ti	0.3	3	0.2-0.33	SSMS, NAA
W <sup>b</sup>	<0.04	3	<0.01-<0.1	SSMS, NAA
U <sup>b</sup>	<0.02	3	<0.01-<0.02	SSMS, NAA
V	<0.04	2	<0.01-<0.04	SSMS
Yb	<0.1	2	<0.04-<0.1	SSMS

Table III (Continued)

Element	Best value, ppm <sup>a</sup>	No. of laboratories	Range	Techniques
Y	<0.02	2	<0.02	SSMS
Zn	52 ± 6	7	4-61	AA, ICPS, SSMS, NAA
Zr <sup>b</sup>	<0.2	3	<0.01-<0.2	SSMS, NAA
% H <sub>2</sub> O	6.2	2	5.9-6.4	
Total	95.5%			
% Ash	6.0	2	5.96-6.10	

<sup>a</sup> See text for criteria used to determine the "best values". A standard deviation for the "best value" was reported when sufficient analytical data were available. <sup>b</sup> The upper limit reported is consistent with both SSMS and NAA.

Table IV. Performance of Individual Laboratories<sup>a</sup>

Laboratory	Total no. of elements determined	No. of elements compared	No. of elements within 10% of "best value"	Technique
1	18	10	9	AA, WC
2	8	6	3	AA
3	48	9	9	NAA
4	28	7	Semiquant. <sup>b</sup>	ES
5	15	8	5 <sup>c</sup>	ICPS
6	73	14	7	SSMS
7	75	12	9	SSMS
8	10	5	5	AA, WC
9	15	7	2	AA, WC
10	3	2	2	NAA

<sup>a</sup> The specific elements compared in this table are: Al, As, Ca, Cu, Cr, Fe, Hg, K, Mg, Mn, Ni, P, Se, and Zn. <sup>b</sup> This laboratory reported concentrations as a range covering a factor of 10 in concentration. See text for detailed evaluation of laboratory performance. <sup>c</sup> The analysts when reporting the data indicated uncertainty with two elements because the technique was still being developed. These two elements fell outside the satisfactory range.

metallic contamination. Soybean meal prepared without taking special precaution to exclude contamination by dust and particulate matter adhering to the beans contained from 14 to 60 ppm of Na. The Na content of the soybean meal prepared for this study was found to be 4 ± 1 ppm (nine analyses). The very low level of Na in the meal indicates no detectable contamination by dust and dirt during preparation and storage.

#### Evaluation of Data and Analytical Techniques.

The two general approaches to the determination of elemental composition of the soybean meal were single-element determination and multi-element surveys. The single-element techniques which optimize the analytical conditions for a particular element are expected, a priori, to be more accurate than multi-element techniques. The multi-element techniques included neutron activation analysis with high-resolution solid state detectors, spark and inductively coupled emission spectroscopy, and spark source mass spectrometry.

The analytical data for elements present in soybean meal can be evaluated by considering the single-element techniques and multi-element techniques separately. Evaluation of analytical data determined by single-element techniques in the concentration range from 0.1 to 50% revealed good agreement. Average abundances obtained from single-element techniques in different laboratories exhibited a relative standard deviation ranging from 1% for nitrogen to 7% for magnesium. Evaluation of analytical data determined by single-element techniques in the trace element concentration range showed considerable dispersion. For example, the range of concentrations reported by laboratories using single-element techniques varied by factors of 15 for chlorine, 100 for arsenic, 20 for mercury, and 2 for iron. Other trace elements, such as zinc, manganese, copper, and chromium, had average abundances which varied by ±10-20%.

Evaluation of the analytical data determined by multi-element techniques generally agreed within a factor of two. However, some elements, e.g., halogens and phos-

phorus, varied by factors of 3 to 100. The analytical data determined by spark source mass spectrometry accounted for most of the variation. Data determined by inductively coupled plasma atomic emission and neutron activation analysis exhibited a variation of ±10-20%.

A tabulation of the best concentration value for each element in the soybean meal reported in Table III was difficult. A rigorous statistical treatment was not possible because only a few elements were analyzed in four or more laboratories. As a result, it was necessary to examine the spread of values found for each element, bearing in mind the factors which may have contributed to the dispersion. The following criteria were used to establish the "best value" reported in Table III.

(1) The average of individual analyses for participating laboratories was reported. A standard deviation was reported when four or more analyses from two independent laboratories agreed. In some instances the concentration obtained by more "precise methods" was weighted more than a value obtained by SSMS. In several instances a value was totally excluded; for example, one laboratory analyzed duplicate samples for phosphorus and reported 0.04 and 0.75%. Seven other analyses by two different laboratories reported an average of 0.71%. Therefore, the low value was not used.

(2) When data were available only from SSMS, the values from the two independent laboratories were averaged. When an upper limit was reported from SSMS it was the highest value from the two laboratories. The elements indicated by footnote *b* (Table III) report the lowest upper limit that is consistent with both SSMS and NAA.

**Laboratory Performance.** The performance of the participating laboratories is summarized in Table IV. The criteria used for this study are similar to those used by Morrison (1971) for the evaluation of laboratories involved in the analyses of lunar samples. The criteria used were: only those elements for which data were available from four or more laboratories were used to compare per-

Table V. Comparison of "Best Values" with Previous Results

Element	Liener (1972)	"Best value"
Ca, %	0.24-0.31	0.222 ± 0.008
P, %	0.6	0.71 ± 0.01
Mg, %	0.24-0.30	0.31 ± 0.02
Zn, ppm	55-77	52 ± 6
Fe, ppm	140	137 ± 4
Mn, ppm	24-29	38 ± 3
Cu, ppm	14-24	20 ± 4

formances. Only analyses within  $\pm 10\%$  of the "best value" are considered satisfactory.

The analytical performance of laboratories using single-element techniques (1, 2, 8, 9, 10) is discussed in this paragraph. Ten elements analyzed by laboratory 1 were specifically compared in Table IV; nine of these elements agreed within  $\pm 10\%$  of the "best value". Arsenic, which is seldom analyzed by this laboratory, disagreed with the "best value". Six elements analyzed in laboratory 2 were specifically compared in Table IV; three elements agreed within 10% and the other three elements agreed within 20-30% of the "best value". Five of the eight elements analyzed by laboratory 8 were compared in Table IV and all agreed within  $\pm 10\%$  of the "best value". Seven elements analyzed by laboratory 9 were compared in Table IV; only two elements agreed within  $\pm 10\%$  of the "best value". Other trace elements analyzed by laboratory 9 but not compared in Table IV also showed poor agreement with the "best value", often differing by factors of 20 or more.

The analytical performance of laboratories using multi-element techniques (3, 4, 5, 6, 7) is discussed in this paragraph. The laboratories using multi-element techniques reported data for more elements than the laboratories using single-element techniques. Laboratory 3 which used neutron activation analysis reported data for 48 elements; nine elements were specifically compared in Table IV and all agreed within  $\pm 10\%$  of the "best value". Laboratory 4 used spark emission spectroscopy and reported results on 28 elements. The concentrations were reported as a range, covering a factor of ten in concentration. The semiquantitative data could not be analyzed according to the criteria used in Table IV. However, laboratory 4 reported concentration ranges for two elements that were not consistent with the "best value", and zinc at 52 ppm was not detected. Laboratory 5 using inductively coupled plasma atomic emission reported data on eight elements compared in Table IV; of these, five elements agreed within 10% of the "best value". This laboratory, when reporting the data, indicated uncertainty about two elements (Zn and Mn) because the technique was still being developed; these two elements fell outside the satisfactory range. Laboratory 6 using spark source mass spectrometry reported data for 73 elements. Data for 14 elements are specifically compared in Table IV; all values (except phosphorus) were within  $\pm 200\%$  of the "best value", and seven were within  $\pm 10\%$  of the "best value". Laboratory 7 which also used spark source mass

spectrometry reported data for 75 elements, and 14 elements were compared in Table IV. Zinc and phosphorus were low by more than a factor of 7, the 12 other elements compared were all within  $\pm 100\%$  of the "best value", and 9 were within  $\pm 10\%$ . Although the halogens were not included in Table IV, laboratory 7 reported values for the halogens which were factors of 10 or more lower than other laboratories.

#### CONCLUSION

The complete analysis of soybean meal is a difficult analytical problem because the concentrations range from percentages to a few parts per billion. The analyses performed account for 95.5% of the soybean meal. The ash content of the meal was 6%. No single technique is capable of obtaining precise data for all elements. The "best values" obtained in this study are compared in Table V to the abundances in soybean meal reported by Liener (1972). The "best values" of this study generally overlap or are within  $\pm 10\%$  of the range reported by Liener. The exception is Mn where Liener reported 24-29 ppm and this study reported  $38 \pm 3$  ppm.

The results reported in this study show there is considerable variability in the performance of different laboratories. In general, single-element techniques, neutron activation analysis, and inductively coupled plasma atomic emission compared favorably with one another. Spark source mass spectrometry, the most comprehensive technique, produced good results for some elements, but was overall the least accurate quantitative technique. Spark atomic emission produced only semi-quantitative results and was generally not as sensitive as the other techniques. Laboratories using multi-element techniques and achieving an acceptable degree of accuracy are simultaneously providing useful information on a larger number of elements than laboratories using single-element techniques.

In summary, the quality of the results is related to the techniques used, the skill of the investigator, and the time available for performing and reporting the results. To monitor the performance of a laboratory, a well-characterized sample, similar in composition to the material of interest, should be periodically submitted for analysis.

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