

# RESEARCH NOTE ON PRESSURE DIGESTION OF CEREAL GRAINS AND FLOURS FOR MINERAL ANALYSIS BY ATOMIC ABSORPTION

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The mineral concentrations of cereal grains and flours can be determined by atomic absorption without appreciable spectral interferences and with only minor chemical interferences (1). The precision and accuracy of the technique are equal to or, in some instances, better than those obtainable with other methods involving various separation steps before measurements by colorimetric, flame-photometric, and titration techniques.

Decomposing the organic material without losing mineral elements is most important. Dry-ashing in a muffle furnace at high temperatures is a common means of organic matter destruction. However, losses due to volatilization and incorporation in some solid material, either in the ash residue or in the ashing vessel, can be quite serious with dry-ashing (2).

Wet-ashing is a more reliable preparation technique before measurements by atomic absorption. However, the technique also has a few shortcomings. It requires time because of the need for frequent technician attention. There is still a possibility of spray losses and losses at time of transfer of the digest to volumetric flasks for dilution.

Adrian (3) suggested a wet-digestion technique for biological materials which uses pressure; this requires considerably less attention by a technician, and eliminates spray losses and losses due to volatilization and sample transfer after digestion, since the entire digestion and initial dilution is done in capped plastic tubes. Overall, the method also requires less time.

The applicability of this method to cereal grains and flours, however, has not been demonstrated. If whole grains can be digested without having to be ground, this method could prove to be very advantageous. Metal contamination due to grinding has occasionally been a problem.

It was the purpose of this study to compare Adrian's (3) pressure technique with an accepted wet-ashing procedure for digestion of wheat flours and whole grains for mineral analyses by atomic absorption.

## MATERIALS AND METHODS

### Sample Description

The samples of wheat and flour used in this study were collected from mills in different parts of the U.S. after the 1975 harvest. The wheat samples represent various classes of wheat grown under different agronomic and climatic conditions. The flour samples, although of approximately the same extractions, are of different patent percentages. The description and proximate analyses of the samples are given in Table I. Moisture, protein, and ash were determined using AACC approved methods (4).

**Atomic Absorption Spectroscopy**

*Wet-Ashing Procedure.* Approximately 4-g samples of each of the ground grains and flours were digested in 250 ml Erlenmeyer flasks with 10 ml HNO<sub>3</sub> and 0.5 ml of a concentrated HClO<sub>4</sub>-H<sub>2</sub>SO<sub>4</sub> (1:9) mixture. The samples were heated gently on a hot plate to avoid foaming. Heating was continued until solutions were clear. Solutions which did not become clear were allowed to cool. An additional 10 ml of HNO<sub>3</sub> was added and heating was continued until the contents of the flasks were clear. The solutions were allowed to cool, and 2 ml of concentrated HNO<sub>3</sub> was added to the flasks which were then heated gently with a cover glass in place to get a reflux action. The walls of the flasks were rinsed with distilled water and the mixture evaporated to 2 ml. Approximately 10 ml of distilled water was added and the total contents transferred to 25-ml volumetric flasks, which were made up to volume.

*Pressure Digestion Procedure.* A modification of the method by Adrian (3) was used to prepare the samples for analyses. Approximately 0.5 g of the whole grains and of the flours was weighed into 16 × 125-mm plastic screw cap test

**TABLE I**  
Description and Proximate Analyses of Samples

Flours— Class and Source	Extraction %	Patent %	Moisture %	Protein <sup>a</sup> %	Ash <sup>a</sup> %
1) Hard winter wheat—Kans.	74	85	13.8	9.8	0.39
2) Soft red wheat—Mo.	74	100	12.2	8.7	0.51
3) Soft red wheat—Ill.	76	65	12.7	7.8	0.32
4) Hard spring wheat—N. Dak.	73	98	13.8	12.6	0.51
5) Hard spring wheat—Minn.	74.5	100	14.0	13.9	0.58
Wheats					
1) Hard spring wheat—Minn.	...	...	11.4	10.6	1.69
2) Hard winter wheat—Kans.	...	...	12.2	12.0	1.75
3) Soft red wheat—Ohio	...	...	14.6	9.5	1.77
4) Hard spring wheat—Mont.	...	...	11.7	12.7	1.57
5) Hard spring wheat—Idaho	...	...	14.3	12.9	1.94

<sup>a</sup>On 14% moisture basis.

**TABLE II**  
Atomic Absorption Operating Conditions and Instrumental Parameters

Element	Cathode Lamp mA	Wavelength nm	Slit Setting	Linear Working Range
Na	15	295—VIS	4	up to 1 μg/ml
Mg	15	285—uv	4	up to 0.5 μg/ml
K	12	383—VIS	4	up to 2 μg/ml
Ca	15	211—VIS	4	up to 7 μg/ml
Mn	15	279—uv	3	up to 3 μg/ml
Fe	30	249—uv	3	up to 5 μg/ml
Cu	20	325—uv	4	up to 5 μg/ml

TABLE III  
Mineral Composition of Flours<sup>a</sup> (Wet-Ashing vs. Pressure Digestion)

Element	Digestion Procedure	HWW Kans.	SRW Mo.	SRW Ill.	HSW N. Dak.	HSW Minn.
K, %	Wet ash	0.093 ± 0.014	0.149 ± 0.020	0.088 ± 0.008	0.157 ± 0.002	0.114 ± 0.001
	Pressure	0.088 ± 0.006	0.150 ± 0.006	0.113 ± 0.010	0.160 ± 0.007	0.147 ± 0.008
Mg, %	Wet ash	0.027 ± 0.001	0.023 ± 0.004	0.011 ± 0.001	0.031 ± 0.008	0.035 ± 0.010
	Pressure	0.034 ± 0.006	0.035 ± 0.003	0.018 ± 0.001	0.038 ± 0.004	0.044 ± 0.003
Ca, %	Wet ash	0.015 ± 0.001	0.016 ± 0.001	0.011 ± 0.001	0.017 ± 0.003	0.013 ± 0.001
	Pressure	0.019 ± 0.001	0.018 ± 0.000	0.016 ± 0.001	0.017 ± 0.002	0.016 ± 0.001
Na, ppm	Wet ash	16.2 ± 1.0	17.2 ± 0.6	12.6 ± 0.2	18.4 ± 4.2	16.3 ± 0.4
	Pressure	20.1 ± 3.3	19.2 ± 0.4	18.9 ± 2.1	22.2 ± 2.5	18.6 ± 2.2
Mn, ppm	Wet ash	10.7 ± 0.5	12.1 ± 1.5	8.8 ± 1.6	9.2 ± 1.5	9.2 ± 1.0
	Pressure	10.8 ± 0.3	13.6 ± 2.8	10.0 ± 1.3	8.4 ± 1.4	11.0 ± 0.9
Fe, ppm	Wet ash	16.2 ± 1.4	15.7 ± 2.5	11.8 ± 0.0	24.7 ± 3.6	22.8 ± 2.6
	Pressure	13.9 ± 1.9	16.7 ± 1.3	12.1 ± 0.6	20.4 ± 2.0	23.3 ± 1.7

<sup>a</sup>Expressed on a moisture-free basis.

TABLE IV  
Mineral Composition of Wheats<sup>a</sup> (Wet-Ashing vs. Pressure Digestion)

Element	Digestion Procedure	HSW Minn.	HWW Kans.	SRW Ohio	HSW Mont.	HSW Idaho
K, %	Wet ash	0.339 ± 0.033	0.503 ± 0.025	0.484 ± 0.040	0.314 ± 0.006	0.476 ± 0.013
	Pressure	0.401 ± 0.027	0.458 ± 0.013	0.513 ± 0.015	0.324 ± 0.023	0.494 ± 0.021
Mg, %	Wet ash	0.154 ± 0.027	0.189 ± 0.027	0.159 ± 0.030	0.116 ± 0.010	0.212 ± 0.000
	Pressure	0.167 ± 0.002	0.170 ± 0.008	0.164 ± 0.005	0.129 ± 0.012	0.214 ± 0.002
Ca, %	Wet ash	0.030 ± 0.002	0.031 ± 0.001	0.029 ± 0.000	0.026 ± 0.001	0.030 ± 0.001
	Pressure	0.033 ± 0.001	0.033 ± 0.001	0.032 ± 0.001	0.029 ± 0.002	0.036 ± 0.007
Na, ppm	Wet ash	27.2 ± 0.8	24.5 ± 1.5	25.1 ± 3.2	27.5 ± 2.4	32.8 ± 2.0
	Pressure	25.6 ± 0.0	31.4 ± 3.6	26.3 ± 2.2	27.4 ± 3.0	36.2 ± 6.3
Mn, ppm	Wet ash	44.9 ± 5.9	52.3 ± 2.6	50.8 ± 3.0	49.2 ± 0.7	55.2 ± 3.0
	Pressure	46.2 ± 3.4	47.5 ± 2.3	51.9 ± 2.4	50.4 ± 4.5	56.2 ± 4.6
Fe, ppm	Wet ash	39.2 ± 1.7	33.1 ± 2.6	37.8 ± 2.6	47.5 ± 1.8	37.2 ± 4.8
	Pressure	45.9 ± 2.3	36.4 ± 1.8	42.9 ± 0.6	52.2 ± 3.0	43.4 ± 3.9

<sup>a</sup>Expressed on a moisture-free basis.

tubes (#2025 Falcon Plastics). Two milliliters of concentrated  $\text{HNO}_3$  was added and the test tubes were loosely capped. The samples were predigested for 12 hr before 2 ml of concentrated  $\text{HClO}_4$  was added. The tubes were tightly capped and immersed in  $70^\circ\text{C}$  water for 3 hr. Digestion was done under a closed hood because the samples develop pressure sufficiently high to cause a test tube to explode occasionally. After digestion, the liquid is diluted to volume with glass distilled water. Further dilutions were prepared for individual determinations, when needed, to stay within the linear working range for the element.

*Instrumentation.* A Perkin-Elmer Model 303 atomic absorption spectrophotometer was used and operated for absorption measurements as shown in Table II. Hollow cathode lamps were used for the determinations at the recommended current rating. A set of standards was run at the beginning and end of each series of samples. Primary stock solutions were prepared from chemicals of high purity. Secondary working standard solutions were prepared from the primary stock solutions covering the desired concentration range for each element. The results represent the averages of three separate determinations.

*Statistical Evaluation.* Significant differences between methods were evaluated by statistical evaluation for interaction using analysis of variance. Each element was evaluated separately using pooled compositional data obtained from three separate determinations for each of the five flours or grains.

## RESULTS AND DISCUSSION

The use of the pressure technique resulted in solutions which were clear. Even the whole grain kernels disintegrated completely.

Mineral compositions of the flours and grains, expressed on a moisture-free basis, are given in Tables III and IV. Compositions differed as the result of

TABLE V  
Statistical Comparison of Digestion Methods

Mineral	Sample	AOV F-Ratio	Calculated $\alpha$	Significance $\alpha = 0.05$
K	Flour	28.67	0.000	S
K	Grain	29.39	0.000	S
Mg	Flour	16.69	0.000	S
Mg	Grain	8.22	0.000	S
Ca	Flour	5.87	0.002	S
Ca	Grain	3.79	0.014	S
Na	Flour	4.69	0.005	S
Na	Grain	2.59	0.062	NS
Mn	Flour	4.50	0.007	S
Mn	Grain	2.47	0.067	NS
Fe	Flour	6.48	0.002	S
Fe	Grain	11.30	0.000	S

variety and location of growth, as would be expected.

Both methods produced values for K, Mg, Ca, Na, Mn, and Fe which are expected by comparison with literature values. With few exceptions, however, the pressure technique resulted in slightly higher values, which is due to the avoidance of losses that normally occur with other ashing procedures.

When the concentrations of minerals obtained by the two methods were compared using analysis of variance, the data summarized in Table V resulted. Among the 12 comparisons listed, only two were found not to be different statistically. The sodium and manganese levels in grains did not differ as influenced by digestion method. For all other comparisons, the pressure digestion method gave statistically significant higher values.

The data presented in this study indicate that the pressure digestion method can be used on whole wheat grain, thus eliminating the need for grinding and subsequent losses and possible contamination. The proposed method also results in equal or significantly higher values than the wet-ashing method of digestion for flours for most minerals evaluated.

#### **Acknowledgment**

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