NOTE ON ESTIMATING ASH CONTENT OF FLOUR MILL STREAMS OF HARD RED SPRING WHEAT BY PROXIMATE ANALYSIS OF THE MANGANESE CONTENT OF THE WATER EXTRACT BY USING ATOMIC ABSORPTION SPECTROPHOTOMETRY[1]

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Ash content of flour is used extensively in the flour milling industry as a quality control measure of the milling operation. The higher the flour extraction rate for any given wheat, the higher the ash content. The whiteness of flour decreases with increasing ash content. Generally, straight-grade flour milled to about 72% extraction in the U.S. would contain 0.42 to 0.46% ash. In spite of the extensive use of ash as a flour milling quality control measure, there is no acceptable rapid method for determining percentage of ash. The most rapid accepted methods are the acetate methods (1), which take about 1 hr. As a quality control measure for a pilot mill such as ours (2) or for large commercial mills, the methods are not acceptable because of the time involved. An oxygen method which takes about 15 min has not been officially accepted.

We have attempted to develop a rapid method for estimating the ash content

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of flour mill streams. Hopefully, the technique could be used to monitor the flour streams in place of ash. The amount of Mn in a simple water extract of flour was determined with an atomic absorption spectrophotometer.

MATERIALS AND METHODS

Samples of hard red spring wheat from the 1967–69 and 1974 crop years grown on 1-acre plots were milled on our pilot mill (2). Ash was determined on

TABLE I
Correlation Coefficients[a] between Percentage Ash and Manganese Content of the HCl and Water Extracts of 10 Flour Streams

Variety and Location	n	H_2O Extract[b]	0.096*N* HCl Extract
Chris			
Casselton, N. Dak.	10	0.931**	0.951**
Claremont, S. Dak.	10	0.927**	0.964**
Experimental A			
Casselton, N. Dak.	10	0.978**	0.955**
Claremont, S. Dak.	10	0.879**	0.959**

[a]** = highly significant at 1% level.
[b]Deionized distilled H_2O.

TABLE II
Correlation Coefficients[a] between Percentage Ash and Manganese Content of the Water Extract for Each Flour Product Stream of the Pilot Mill for 3 Years

	Correlation Coefficient			
Product[b]	1967 n=7	1968 n=45	1969 n=34	1967–69 n=86
1	−0.122	0.405**	0.262	0.246*
2	−0.038	0.542**	0.254	0.236*
3	0.541	0.746**	0.062	0.486**
4	0.747*	0.814**	0.745**	0.840**
5	0.335	0.867**	0.405*	0.703**
6	0.857*	0.662**	0.384*	0.661**
7	−0.219	0.331*	0.589**	0.687**
8	−0.186	0.132	0.157	0.170
9	0.316	0.284*	0.269	0.644**
10	−0.217	0.177	0.084	0.427**
1 through 7[c]	0.828**	...	...	...
1 through 10 + 12[d]	...	0.722**	0.812**	0.761**

[a]** = highly significant at 1% level; * = significant at 5% level.
[b]1 = 2nd + 3rd Midds; 2 = coarse + fine 1st midds + 4th midds + sizings; 3 = break flours + grader; 4 = 5th midds + tailings; 5 = low grade; 6 = low quality; 7 = red dog; 8 = tail shorts; 9 = head shorts; 10 = bran.
[c]n = 49.
[d]n = 451 for 1968; 341 for 1969; and 863 for 1967–69; 12 = Patent.

3-g samples according to AACC Method 08-01 (1).

In preliminary studies, 1-g samples were extracted with 25 cc of deionized water, 0.098*N* NaOH, or 0.096*N* HCl for 30 min by shaking on a Burrell shaker. The samples then were centrifuged at 7500 × *g* for 10 min and the supernatant decanted for analysis on an atomic absorption (AA) spectrophotometer. Absorbance of the supernatant was measured on a Beckman AA System 1301 in conjunction with a Beckman DB-G Spectrophotometer. The 1974 samples were run on a Perkin Elmer Model 305A with a slit width setting of 2, wavelength 279.5 nm, and a multiple element lamp at 30 mA. Standards for the Mn analyses were made from a certified AA standard, 1000 ppm, purchased from Fisher Scientific Co., Fair Lawn, N.J. The reference standard was diluted to the required concentration with deionized distilled water.

The preliminary studies indicated that extraction with 0.096*N* HCl gave the highest results. However, additional studies indicated an interference in the AA analysis because the HCl apparently caused some protein to be denatured, which clogged the atomizer of the AA. Therefore, in the procedure adopted, 1-g samples were extracted with 25 cc of deionized distilled water in a 125-ml Erlenmeyer flask for 30 min on a Burrell shaker and centrifuged for 10 min at 7500 × *g*. The supernatant was decanted for AA analysis. All analyses were made in duplicate. The standard deviation of duplicate determinations was 0.03 ppm Mn.

RESULTS AND DISCUSSION

Data in Table I show the relation between ash and Mn content when deionized distilled water and 0.096*N* HCl were used as the extractants. The Mn content as determined by the two methods is significantly and about equally related to ash content. The relation was about the same for the 10 mill streams of both varieties. Because no problems were experienced with the atomizer of the AA clogging when water was used as the extractant, all further extractions were with water.

Analyses were made on samples from the 1967–69 and 1974 crop years to

TABLE III
Correlation Coefficients[a] between Percentage Ash and Manganese Content of Water Extract for Six Flour Product Streams[b]

Samples	n	r-value	m Slope	b y-intercept
1967	54	0.88**	2.850	0.1211
1968	270	0.91**	2.585	0.1745
1969	202	0.91**	2.982	0.0608
1967–69	526	0.91**	2.766	0.1286
1974	207	0.87**	2.194	0.3085

[a]** = highly significant at 1% level.

[b]1967–69 product streams were: 2nd midds + 3rd midds; coarse + fine 1st midds + 4th midds + sizings; break flour + grader; 5th midds + tailings; low grade; low quality. 1974 product streams were: 1st midds; 2nd, 3rd, and 4th midds + 2nd sizing; 3rd break + break dust + 1st sizing + 5th midds; 1st, 4th, and 2nd break + 6th midds; low grade; low quality.

determine the influences of environment and variety on the results. Correlation coefficients between Mn content and percentage ash for several product streams for the 1967–69 crop years and the combined years are shown in Table II. Flour stream blends 1 through 4, when combined, make up the normal patent flour. The Mn content of blend No. 4 (5th midds + tailings) was highly correlated with ash content for all 3 years and for the combined years, indicating that Mn content of these fractions varies more directly with wheat ash content than any of the other product streams. The range in Mn and ash content of each flour stream blend is small, which is one reason for the low correlation coefficients. When all product streams were included in the statistical analysis, the relation between Mn and ash content was highly significant.

Correlation coefficients between Mn content and percentage ash for the six flour streams from the pilot mill by years are all highly significant (Table III). The slope and y-intercept remained relatively constant for the 4 years even though the flow of the mill was changed in 1974. The higher correlation coefficients shown in Table III *vs.* Table II are attributed to the widening of the range by combining the data for six streams.

The data indicate that AA analysis of the Mn content of the water extract of mill stream products could be used as a method of quality control. One analysis for Mn requires about 45 min. Simultaneous analyses of several samples would reduce time per sample. Only one weighing of the sample is necessary and the AA analysis can be completed in just a few seconds.

Literature Cited

1. AMERICAN ASSOCIATION OF CEREAL CHEMISTS. Approved methods of the AACC. Method 08-01, approved April 1961. The Association: St. Paul, Minn.
2. SHUEY, W. C., and GILLES, K. A. Laboratory scale commercial mill. Operative Millers Tech. Bull., p. 3100 (1969).

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