

A Rapid, Objective Method to Measure the Degree of Milling of Rice¹

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ABSTRACT

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A rapid, objective method was developed to rate the degree of milling of rice. It involves 5-min extraction of 10 g of milled rice with 40 ml of isopropyl alcohol-water (1:1, v/v) and measurement of the electrical conductivity ($\mu\text{mhos/cm}$) of the extract. The objective and subjective ratings for the degree of rice milling were highly correlated and the

correlations of both types of ratings with ash, surface lipid, and Agtron color were higher for experimentally than for commercially milled samples. The objective test was sensitive to the percentage of broken kernels in the sample.

The whiter the milled rice, the more it is desired by consumers. Therefore, a characteristic of well-milled rice is lack of bran layers (pericarp, tegmen, and aleurone) and embryo.

Methods proposed for measuring the degree of milling of rice can be classified in two groups: (a) those that estimate objectively or subjectively the amount of outer layer remaining on the starchy

endosperm and (b) those that express the chemical or optical character of the milled product. The proposed methods have been discussed by Hogan and Deobald (1965), Barber (1972-74, 1975-76), Pomeranz et al (1975) and Stermer et al (1977).

The two methods used most involve extracting surface lipids (Hogan and Deobald 1961) and measuring the colored Bran Index (FAO 1972). In the first method, fat is extracted from whole rice by a refluxing flammable solvent, the solvent is evaporated from the extract, and the lipid residue is weighed. Because the bran (mainly aleurone) and germ portions contain more lipid than the endosperm, extractable fat decreases progressively with milling. In the second method, the bran is stained with the May-Grünwald reagent (1% methylene blue and 1% eosine in methyl alcohol), and the stained area is measured by planimetry. The arbitrary colored Bran Index ranges from 100 for brown rice to 5 for commercial samples and 0 for well-milled rice. This method measures the degree of milling of rice and also permits evaluation of the homogeneity of milling. The method is tedious, however, and analysis of one sample requires 1.5 hr. Neither method is entirely suitable for grading rice.

The method reported here involves extracting minerals from

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whole milled rice and measuring the electrical resistance or conductivity of the extract. The analysis takes about 10 min, and we obtained objective data that correlated with the degree of milling. The method could be used for routine testing and is based on the premise that percent ash content and degree of milling correlate well because concentrations of ash in the aleurone and in the central starchy endosperm are about 20:1. Because determining ash is time-consuming, since it requires incineration, we determined soluble electrolyte content by measuring conductivity of an aqueous extract of the rice. Pomeranz and Lindner (1956) and Clements (1977a,b) described the principle in determining degree of wheat flour milling.

MATERIALS

Pearled Samples

A sample of long-grain (Labelle) rice was milled in a McGill sheller to remove the hulls and in a Strong-Scott barley pearler to remove the bran. By pearling different batches for 30, 45, 60, and 90 sec we obtained material that differed in degree of milling to determine the effects of extraction factors—alcohol concentration, type of alcohol, time, and temperature.

Experimentally Milled Samples

Four sets of rice samples representing short (Caloro), medium (Nato and Brazos), and long (Labelle) grain varieties were obtained from B. D. Webb, Beaumont, TX. Samples from each variety were milled by the procedures shown in Table I.

Interpretive Line Samples

Nine interpretive line-reference samples that had been milled on a commercial rice mill and stored at 4.4°C were used as standards in the subjective grading. They were obtained from the Federal Grain Inspection Service (FGIS), U.S. Department of Agriculture (USDA), Washington, DC.

Commercially Milled Samples

Twenty-one commercial samples of short, medium, and long grain rice were obtained through the Board of Appeals and Review, Standardization Division, FGIS, Washington, DC. They were shipped in plastic bags.

METHODS

Degree of Milling

Degree of milling was evaluated subjectively on a scale of 4 to 0, respectively, as: well milled, reasonably well milled, lightly milled, under milled, or brown rice. The graders were two inspectors at the Board of Appeals and Review, FGIS, USDA.

Analytical Methods

Rice samples were analyzed for nitrogen, ash, and moisture by AACC methods. Protein content was calculated as the product of Kjeldahl nitrogen and 6.25; results are expressed on a 14% moisture, wet basis.

Unstained rice was measured on a Hunterlab Model D-25 color

difference meter with a blue Hunterlab standard 025-932 as a reference color and on an Agtron Model M-500-A reflectance spectrophotometer with a blue filter. Differentiation among samples was greatest when these instruments were used with the respective blue reference color and filter. The total color difference recorded for the Hunterlab color difference meter was

$$\sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$$

where Δ is difference and L, a, and b are components of color. The Agtron meter was calibrated with No. 12 and No. 63 standard discs to read 0 and 100% reflectance, respectively, for the blue spectral line (436 nm). Relative reflectances of milled rice samples were read directly.

Surface lipids in 10 g of whole grain rice were determined according to Hogan and Deobald's method (1961). The rice was then ground and analyzed for free lipids by AACC Method 30-25. A Soxhlet extractor, Whatman No. 5 filter paper, Whatman thimble (33-mm diameter \times 80 mm high), and petroleum ether solvent (B.P. 35–60°C) were used for the 6-hr extraction at a condensation rate of 4–5 drops per second.

Kernel weight was determined for 1,000 whole kernels picked by hand from each sample.

Rice Extraction and Conductivity Measurements

Ten-gram samples of rice were extracted for 5 min at 25°C using 40 ml of 1:1, v/v isopropyl alcohol-water. The electrical conductivity or resistance, or both, of the unfiltered extracts was measured at $25 \pm 0.1^\circ\text{C}$ with a Beckman Model RC16B2 conductivity bridge. In preliminary work to test the effects of various factors on the conductivity/resistance of extracts, extraction and electrical measurements were made at 30°C. Measurements were corrected for the cell constant, and all data refer to either specific resistance or specific conductivity. A 0.0005N KCl solution (75 $\mu\text{mhos/cm}$ at 25°C) was used to establish the cell constant.

An apparatus was built to submerge five rice samples automatically and simultaneously in solvent contained in 100-ml centrifuge tubes and to drain the solvent from the samples (Fig. 1). The baskets, made entirely of 16-mesh stainless-steel screen and containing 10 g of rice, were lowered into and raised from 40 ml of solvent as a group every 6 sec for 5 min. Extraction temperature was 25°C, except as noted.

RESULTS AND DISCUSSION

To study the effects of the various factors, it was more practical to plot specific resistance than to plot specific conductivity (the reciprocal of resistance).

Effects of Different Alcohols

Alcohols tend to toughen the surface of biological material; hence, alcoholic extracts of milled rice should contain a minimum of materials from within the rice kernel. Effects of different alcohol-water (1:1, v/v) solutions as extractants of electrolyte are shown in

TABLE I
Milling Procedure for Experimentally Milled Samples

Procedure No.	Satake TM-05 Mill (at 1,500 rpm)	Satake One-Pass Test Pearler	Time on Mill (sec)	Pressure Plate	Added Weight	
					(g)	Position on Pressure Plate
0	— ^a	—	—	—	—	—
1	+ ^b	—	5	—	—	—
2	—	+	—	+	—	—
3	—	+	—	+	37	3
4	—	+	—	+	37	8
5	—	+	—	+	69	8
6	+	—	60	—	—	—

^a—no treatment.

^b+ treatment.

Fig. 2. Extraction was for 5 min at 30°C. The aqueous isopropyl alcohol solution differentiated best between undermilled and well-milled rice samples and was therefore used in all subsequent work.

Effect of Isopropyl Alcohol Concentration

The effects of isopropyl alcohol concentrations on the specific resistance of rice extracts are shown in Fig. 3. Extraction was for 5 min at 30°C. A 1:1 (v/v) solution of isopropyl alcohol-water was chosen for all future work because it provided an extract with a reasonably high (not excessive) specific resistance reading for well-milled rice. The variations in conductivities of extracts using different alcohols (Fig. 2) and different alcohol-water ratios (Fig. 3) are probably mainly due to the effect of the alcohols on conductivity rather than to differences in electrolyte concentrations.

Effect of Extraction Time

Effects of extraction times with 1:1 isopropyl alcohol-water at 30°C on specific resistance of extracts of rice milled to different degrees are shown in Fig. 4. The spread among the five samples, which represented three different degrees of milling, was greatest at 2.5 min; however, we chose an extraction time of 5 min for subsequent work because it permitted more time for equilibration and was more convenient.

Effect of Temperature

Effects of extraction temperatures on the specific resistance of extracts of rice milled to different degrees are shown in Fig. 5. Three degrees of milling were represented, the extraction medium was (1:1, v/v) isopropyl alcohol-water, and extraction time was 5 min. The spread among the samples was greatest at about 21°C; however, we used 25°C for subsequent work because it is easier to maintain in most laboratories. The standard temperature for conductivity measurements also is 25°C.

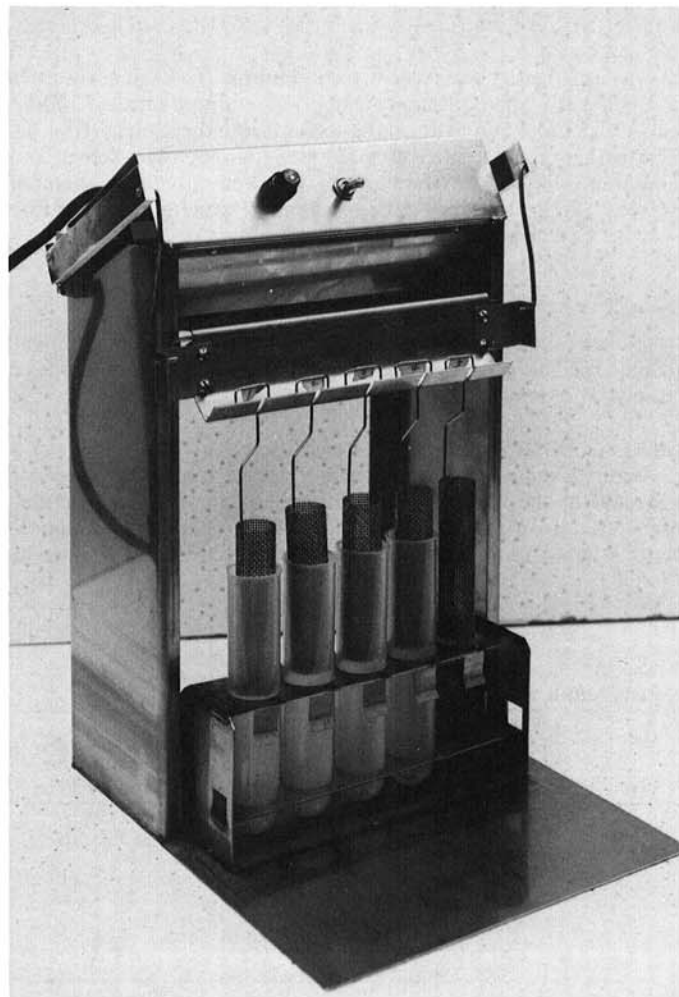


Fig. 1. Extraction apparatus.

Evaluation of Laboratory Milled Rice Samples

Table II shows subjective evaluations, objective measurements, and evaluations of several sets (varieties) of laboratory-milled samples. The two inspectors graded five of the 28 samples differently. Correlations between the subjective ratings and all analytical factors are shown in Table III. Although there are only four subjective ratings and the correlation coefficients between them and the objective analytical data are therefore not accurate, the data nonetheless serve to compare the ability of the various analytical factors to measure the degree of milling. The brown rice samples (milling procedure 0) were not included in the calculations because brown rice is not graded for degree of milling. Furthermore, until the surface of brown rice is abraded, the surface fat is not extractable.

The poor correlations between kernel weight and other variables were probably due to differences in kernel weight among varieties. For the experimental samples (Table II), percent weight loss by milling can be calculated from the kernel weights of the milled rice and brown rice. All milled samples started with the same weight. Figure 6 shows the expected sigmoid relation between percent ash and the percent weight loss by milling.

The relation between percent weight loss by milling and subjective rating is shown in Fig. 7. The subjective rating was not as sensitive as desired to extent of bran removal; the samples were rated from undermilled to well milled over a difference in weight loss by milling of only 1.75%. All samples with greater than 7% of weight loss by milling were rated as well milled.

Figure 8 shows the relation between percent surface lipid and

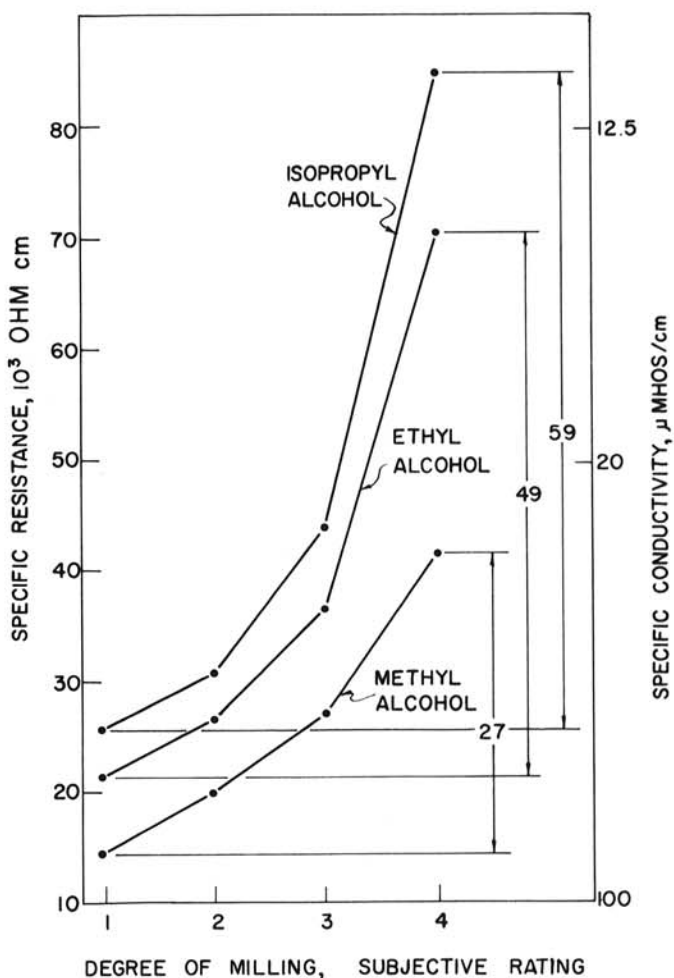


Fig. 2. Effects of different alcohol-water (1:1, v/v) solutions as extractants of electrolyte from rice with different degrees of milling. 1) Undermilled, 2) lightly milled, 3) reasonably well milled, 4) well milled.

percent weight loss by milling. The relation was as expected. The figure also shows the relation between percent surface lipid and the subjective rating. All experimentally milled samples containing less than 0.39% surface lipids were rated as well milled. This is in

reasonable agreement with the results of Watson et al (1975), who found that samples with 0.36% surface lipid content and less than 0.36% ash content graded well milled.

With the 28 experimentally pearled or milled samples (Table II),

TABLE II
Evaluation of Rice Samples Experimentally Milled to Different Degrees

Rice Grain Type	Variety	Milling Procedure ^a	Moisture (%)	Ash 14% mb (%)	Protein 14% mb (%)	Surface Lipid 14% mb (%)	Free Lipid 14% mb (%)	Color Reading (Blue Filter)		Kernel Weight ^b (mg)	Specific Conductance ^b (μmhos/cm)	Degree of Milling		
								Agtron	Hunter			Based on Specific Conductance	Subjective Rating	
													I	II
Long	LaBelle	Pearling	15.8	0.75	7.8	0.67	0.14	29.5	34.6	15.9	24.4	2	1	2
			15.8	0.53	7.7	0.35	0.07	45.0	29.0	15.1	17.7	3	3	3
			15.4	0.39	7.3	0.29	0.05	51.0	31.6	14.7	14.7	4	4	4
			15.3	0.15	7.1	0.06	0.04	60.0	30.2	14.3	9.2	4	4	4
Short	Caloro	0	13.0	1.10	6.5	0.12	2.18	4.0	39.3	24.2	17.4
		1	12.8	0.87	6.2	0.73	0.87	18.0	36.4	23.0	28.7	1	1	1
		2	13.0	0.67	6.2	0.69	0.46	25.0	35.0	22.9	24.1	2	2	2
		3	13.1	0.47	5.9	0.40	0.20	42.0	32.3	22.6	18.6	3	4	3
		4	13.0	0.39	5.8	0.34	0.14	45.0	31.8	22.2	17.4	3	4	4
		5	13.1	0.34	5.8	0.25	0.10	53.0	31.3	21.8	14.9	4	4	4
Medium	Nato	0	13.1	1.41	8.5	0.15	2.18	11.5	38.2	19.1	9.3
		1	13.0	1.01	8.0	0.71	0.66	31.0	34.5	18.1	30.8	1	2	2
		2	13.3	0.59	7.7	0.47	0.15	34.0	33.5	17.3	20.3	2	3	4
		3	13.1	0.40	7.6	0.27	0.08	40.5	32.6	17.3	14.8	4	4	4
		4	12.9	0.35	7.4	0.20	0.06	42.0	32.3	17.0	12.4	4	4	4
		5	12.8	0.31	7.4	0.15	0.05	43.5	31.8	17.0	10.2	4	4	4
Medium	Brazos	0	11.4	1.18	8.5	0.09	1.98	10.5	38.1	24.2	12.9
		1	11.8	0.95	8.2	0.62	0.72	29.0	34.7	22.9	32.2	1	1	2
		2	11.9	0.64	7.9	0.50	0.20	33.0	33.4	22.8	26.5	2	2	3
		3	11.6	0.44	7.8	0.27	0.07	42.5	32.2	22.4	18.3	3	4	4
		4	11.8	0.41	7.7	0.24	0.06	46.5	32.3	22.3	15.7	4	4	4
		5	11.6	0.38	7.6	0.13	0.05	51.0	32.2	21.8	11.2	4	4	4
Long	LaBelle	0	12.9	1.34	8.6	0.20	2.41	13.0	37.8	18.4	12.5
		1	13.0	0.73	8.0	0.80	0.26	38.0	32.8	16.6	25.8	2	2	2
		2	13.0	0.39	7.7	0.37	0.07	47.0	31.6	16.6	14.3	4	4	4
		3	12.9	0.34	7.6	0.27	0.05	50.0	31.8	16.5	9.7	4	4	4
		4	12.9	0.31	7.6	0.19	0.04	53.0	30.5	16.2	9.2	4	4	4
		5	12.8	0.19	7.6	0.14	0.04	60.0	29.7	15.9	7.1	4	4	4
6	13.0	0.28	7.3	0.18	0.06	61.5	29.3	15.9	9.3	4	4	4		

^aSee Table I for all procedures except pearling.

^bAs-is moisture basis.

TABLE III
Correlation Coefficients Between Variables for Rice Milled to Different Degrees^a

Variables	21 Commercially Milled Samples ^{b,c}									
	SR	SPC	SPCR	SL	FL	AC	HC	KW	P	A
SR	...	-0.883*	0.916*	-0.897*	-0.841*	0.818*	-0.765*	-0.263	-0.152	-0.911*
SPC	-0.498	...	-0.946*	0.927*	0.817*	-0.887*	0.830*	0.422	0.186	0.963*
SPCR	0.672*	-0.842*	...	-0.900*	-0.857*	0.852*	-0.790*	-0.365	-0.164	-0.946*
SL				...	0.759*	-0.881*	0.814*	0.245	0.191	0.925*
FL	-0.497	0.401	-0.384		...	-0.748*	0.767*	0.446	-0.029	0.849*
AC	0.672*	-0.394	0.430		-0.729*	...	-0.929*	-0.376	-0.192	-0.880*
HC	-0.636*	0.391	-0.402		0.723*	-0.987*	...	0.456	0.118	0.830*
KW	-0.237	0.090	-0.266		0.184	0.143	-0.209	...	-0.401	0.316
P	-0.193	0.146	0.016		0.115	-0.456	0.515	-0.727*	...	0.281
A	-0.587*	0.507	-0.441		0.559*	-0.777*	0.793*	-0.389	0.779*	...

^aSR = Subjective rating (average of two), SPC = specific conductance, SPCR = specific conductance rating, SL = surface lipid, FL = free lipid, AC = Agtron color (blue filter), HC = Hunterlab color (blue filter), KW = kernel weight, P = protein, A = ash.

^bCorrelation coefficients above the diagonal refer to the experimentally milled samples. Asterisk indicates values that exceed 0.478 and differ significantly from zero ($P < 0.01$).

^cCorrelation coefficients below the diagonal refer to the commercially milled samples. Asterisk indicates values that exceed 0.549 and differ significantly from zero ($P < 0.01$).

ranges of specific conductances (Table IV) were selected by trial and error and assigned values for degree of milling to obtain the best correlation with the subjective degree of milling ratings supplied by two federal grain inspectors (Table II). These subjective ratings differed for five samples. The objective degree of milling ratings based on the specific conductance data in Table IV also differed from the subjective ratings by both inspectors for four samples. In all cases, except one about which the inspectors disagreed, the objective specific conductance rating (based on Table IV) was either lower than both subjective ratings or agreed with the lower of the two subjective ratings. We do not know whether subjective evaluations are affected by factors other than degree of milling, such as discoloration and chalkiness.

TABLE IV
Assignment of a Range of Specific Conductance Values to Samples Differing in Degree of Milling

Degree of Milling	Specific Conductance ($\mu\text{MHOS/cm}$)	Assigned Rating
Undermilled	28.1-40.0	1
Lightly milled	19.1-28.0	2
Reasonably well milled	17.1-19.0	3
Well milled	6.0-17.0	4

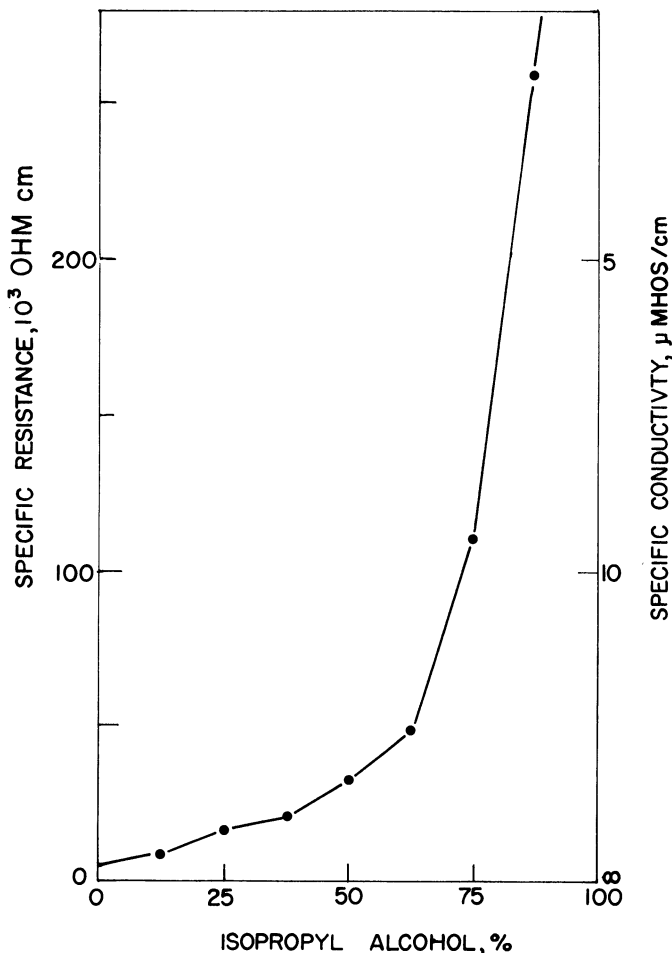


Fig. 3. Effects of isopropyl alcohol concentrations on the specific resistance of an extract of well-milled Labelle rice.

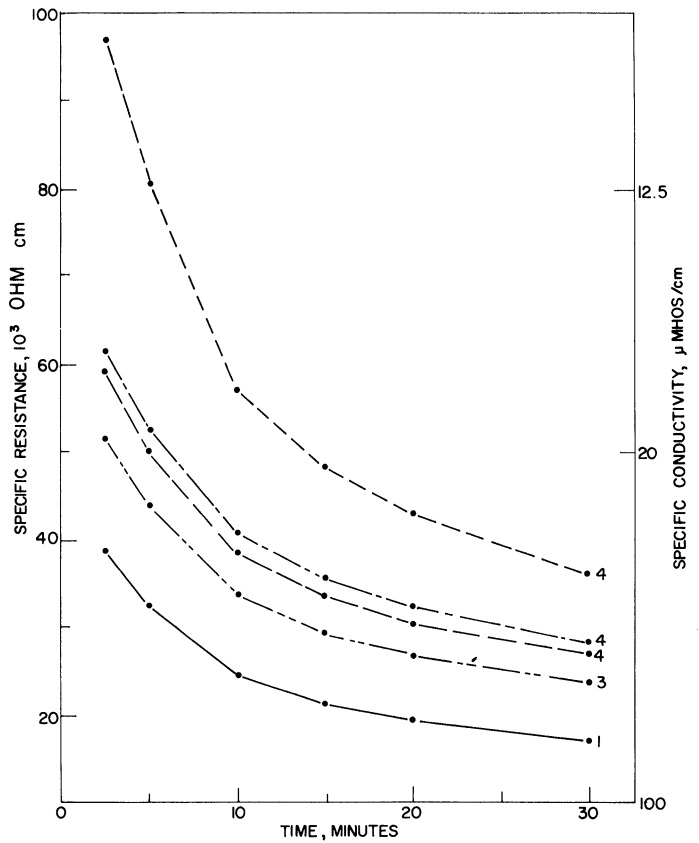


Fig. 4. Effects of extraction times with isopropyl alcohol-water (1:1, v/v) on specific resistance of extracts of rice milled to different degrees. 1) Undermilled, 3) reasonably well milled, 4) well milled.

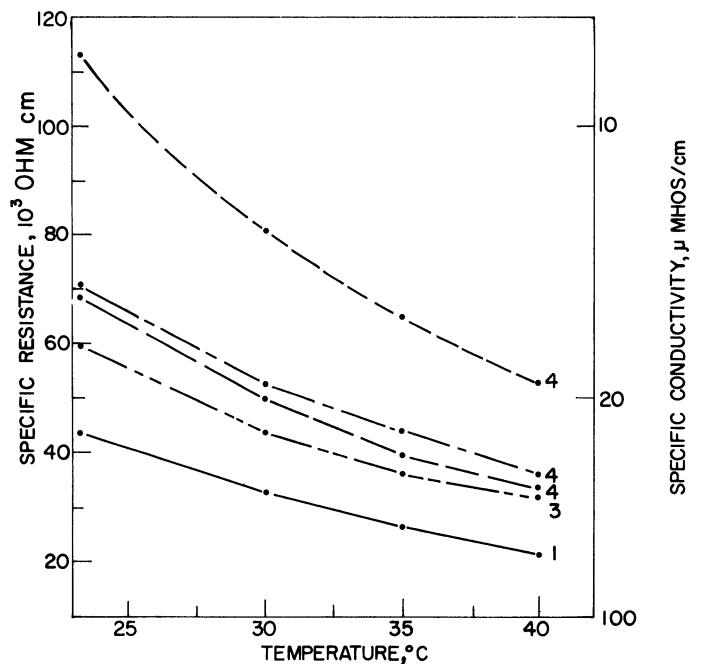


Fig. 5. Effects of extraction temperatures on the specific resistance of extracts of rice milled to different degrees. 1) Undermilled, 3) reasonably well milled, 4) well milled.

Evaluating Commercially Milled Rice Samples

Table V shows the subjective and conductance-based evaluations of 21 commercial rice samples. Two graders graded 7 of the 21 samples differently. When the ratings based on specific conductance differed from one or both of the corresponding subjective ratings, usually only one grade unit of difference was

involved. Correlations between the average subjective ratings and all analytical factors are shown in Table III. The correlations between surface lipid and other factors are not included because the assays for surface lipid content were not reasonable, i.e., all samples high in moisture content ($> 15.9\%$) were extremely low in surface lipid content. In studying grain deterioration, Baker et al (1957)

TABLE V
Evaluation of Commercially Milled Rice Samples

Rice Grain Type	Moisture	Ash 14% mb (%)	Protein 14% mb (%)	Surface Lipid 14% mb (%)	Free Lipid 14% mb (%)	Color Reading (Blue Filter)		Kernel Weight ^a (mg)	Specific Conductance ^a 14% mb (μ mhos/cm)	Degree of Milling			
						Agron	Hunter			Based on Specific Conductance	Subjective Rating		
											I	II	
Short	16.4	0.53	6.7	0.06	0.20	42.0	32.4	23.3	19.4	2	3	1	
	13.3	0.58	6.7	0.34	0.19	45.0	32.1	21.5	20.1	2	3	3	
	16.4	0.47	6.8	0.04	0.10	47.5	31.6	22.2	14.5	4	3	2	
Medium	15.9	0.92	7.6	0.12	0.68	12.0	37.1	19.3	17.7	3	2	2	
	12.8	0.53	6.4	0.44	0.15	37.0	33.4	20.9	23.5	2	3	3	
	16.4	0.52	6.1	0.07	0.17	31.0	33.4	20.4	19.7	2	3	1	
	11.1	0.69	7.4	0.57	0.38	30.0	34.4	18.1	26.8	2	3	3	
	16.0	0.56	7.4	0.04	0.09	33.0	33.8	17.6	13.3	4	4	2	
	14.9	0.50	7.6	0.16	0.06	39.5	33.1	17.7	16.0	4	4	4	
	12.8	0.69	7.4	0.51	0.16	32.5	34.1	18.7	24.0	2	3	3	
	12.6	0.32	6.8	0.22	0.08	45.0	32.4	18.0	10.7	4	4	4	
	Long	11.9	0.92	9.0	0.74	0.27	25.5	35.3	15.8	24.9	2	1	1
		16.3	0.98	9.0	0.23	0.25	10.0	37.4	16.7	19.4	2	1	1
11.2		0.75	7.7	0.61	0.17	33.5	34.0	15.6	22.0	2	2	2	
12.0		0.79	7.8	0.54	0.16	33.0	34.1	15.9	20.6	2	2	2	
12.0		0.80	8.4	0.44	0.09	40.0	32.8	15.9	17.5	3	3	4	
11.8		0.74	8.2	0.43	0.09	39.0	32.9	15.9	16.9	4	3	4	
12.4		0.64	8.0	0.51	0.12	40.0	32.8	14.9	16.4	4	4	4	
13.0		0.69	8.3	0.50	0.13	39.0	33.1	15.1	18.2	3	4	4	
12.0		0.58	7.6	0.48	0.14	40.0	33.2	16.6	16.9	4	3	4	
12.8		0.44	7.0	0.37	0.06	45.0	32.3	15.5	12.7	4	4	4	

^aAs-is moisture basis.

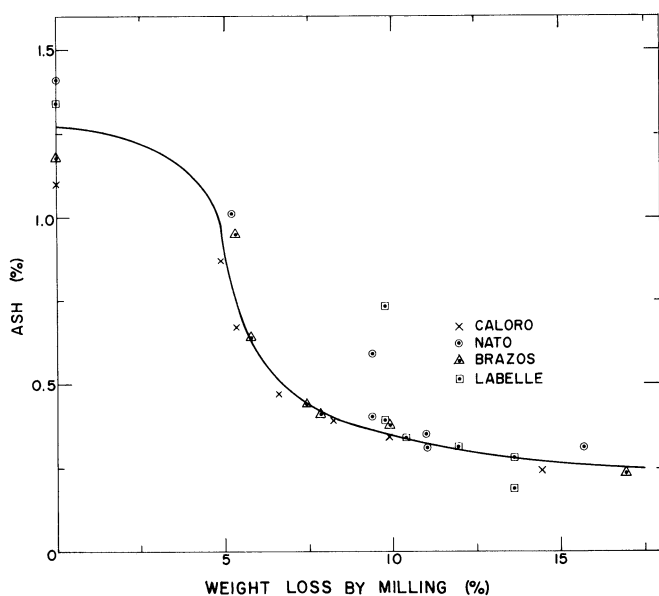


Fig. 6. Relation between percent weight loss by milling and ash content of experimentally milled rice samples.

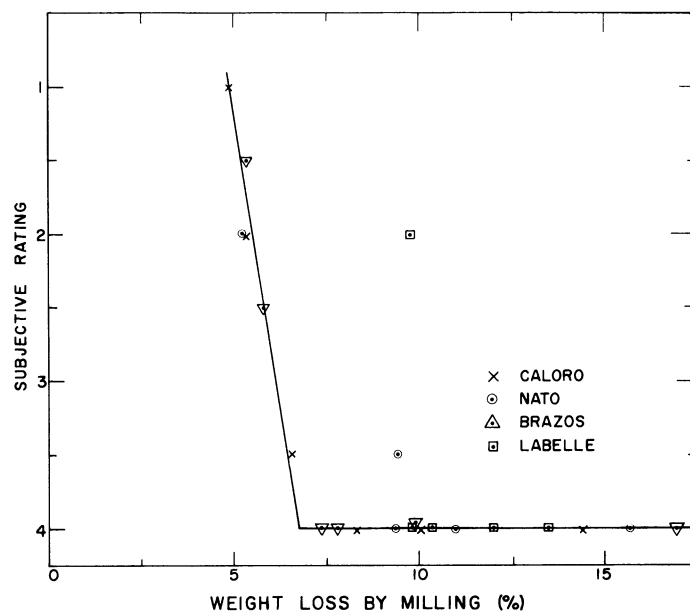


Fig. 7. Relation between percent weight loss by milling and subjective rating for experimentally milled rice samples.

found that only the fat acidity values were comparable in samples with up to 10% moisture.

The significant correlation coefficients for various objective data with the degree of milling determined subjectively are summarized in Table VI. The correlations were somewhat improved when calculated according to grain type (Table VII). For the commercially milled samples shown in Table VII, subjective rating correlated better with specific conductance, specific conductance

TABLE VI
Summary of Significant ($P < 0.01$) Correlation Coefficients Between Indicated Variables for Rice Milled to Different Degrees

Variables ^a		r Values	
		Experimental Samples	Commercial Samples
SR	SPC	-0.883	
SR	SPCR	0.916	0.672
SR	SL	-0.897	
SR	FL	-0.841	
SR	AC	0.818	0.672
SR	HC	-0.765	
SR	A	-0.911	
SPC	SPCR	-0.946	-0.842
SPC	SL	0.927	
SPC	FL	0.817	
SPC	AC	-0.887	
SPC	HC	0.830	
SPC	A	0.963	
SPCR	SL	-0.900	
SPCR	FL	-0.857	
SPCR	AC	0.852	
SPCR	HC	-0.790	
SPCR	A	-0.946	
SL	FL	0.759	
SL	AC	-0.881	
SL	HC	0.814	
SL	A	0.925	
SL	KW		
FL	AC	-0.748	-0.729
FL	HC	0.767	0.680
FL	A	0.849	
HC	A	0.830	
AC	A	-0.880	-0.777
AC	HC	-0.929	-0.926
A	P		0.779

^aSR = subjective rating (average of two), SPC = specific conductance, SPCR = specific conductance rating, SL = surface lipid, FL = free lipid, AC = Agtron color (blue filter), HC = Hunterlab color (blue filter), KW = kernel weight, P = protein, A = ash.

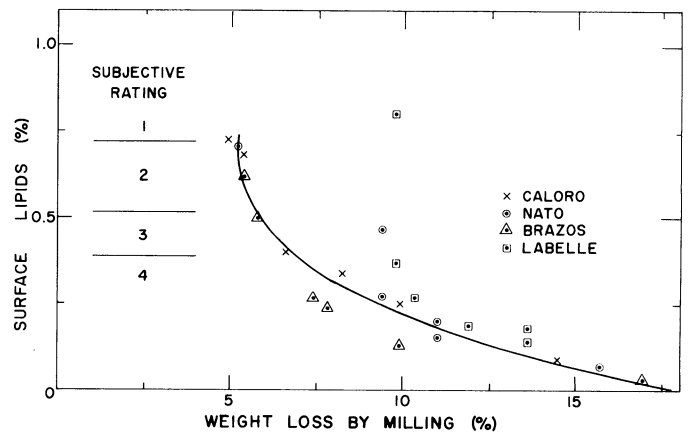


Fig. 8. Relation between percent weight loss by milling and surface lipid content of experimentally milled rice samples.

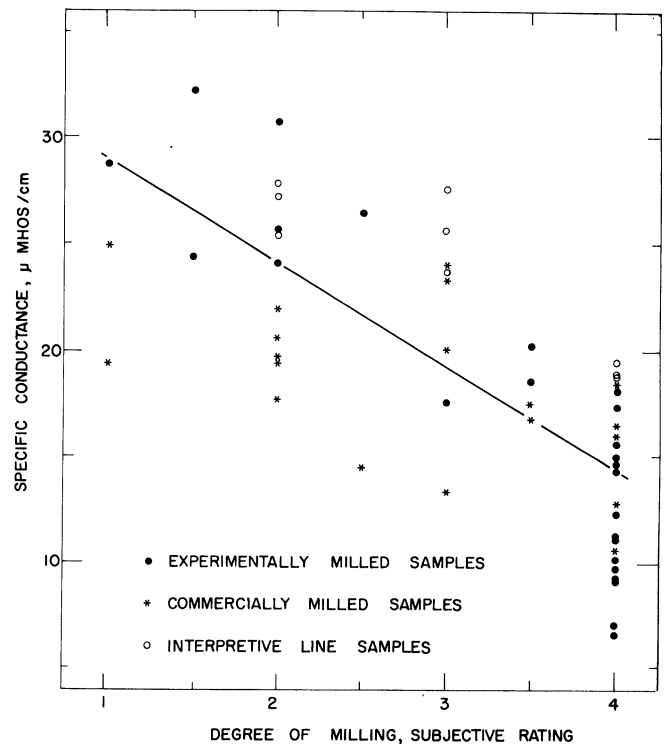


Fig. 9 Relation between specific conductance values of an isopropyl alcohol-water (1:1, v/v) extract of rice and the degree of milling determined subjectively.

TABLE VII
Correlation Coefficients^a for Selected Variables Calculated According to Grain Type

Variables ^a	Number of Commercially Milled Samples			Number of Experimentally Milled Samples			
	Medium Grain	Long Grain	Total Grain	Short Grain	Medium Grain	Long Grain	Total Grain
	8	10	18	6	12	10	28
SR-SPC	-0.362	-0.816* ^b	-0.498	-0.945*	-0.922*	-0.921*	-0.883*
SR-SPCR	0.572	0.865*	0.672*	0.946*	0.924*	0.992*	0.916*
SR-AC	0.811	0.873*	0.672*	0.936*	0.720*	0.882*	0.818*
SR-SL	0.143	-0.118	0.052	-0.921*	-0.890*	-0.892*	-0.897*

^aSR = subjective rating, SPC = specific conductance, SPCR = specific conductance rating, AC = Agtron color, SL = surface lipid.

^bAsterisk indicates values differing significantly from zero ($P < 0.01$).

rating, and Agtron color reading than with surface lipid, but the correlation coefficients were much lower than for experimentally milled samples. Short grain, commercially milled samples (three) were too few for meaningful statistical analyses.

Effect of Broken Kernels on Specific Conductance

We believed that the content of broken kernels was higher and more varied in the commercial than in the laboratory samples and contributed to the wide differences among the former samples. To evaluate the validity of that belief, we compared the analyses of three samples with those of their respective broken-kernel and whole-kernel components. Table VIII shows that specific conductance was greater for broken than for whole kernels. While admittedly fortuitous, the high conductance of broken kernels would lower the rating for degree of milling.

Extraction of Samples by Shaking

For laboratories that may not wish to build the extraction device shown in Fig. 1, the data in Table IX show that a commercially available laboratory shaker bath, such as the Thermoshake Bath (Forma Scientific Co.) operating at speed 5, could produce results

TABLE VIII
Specific Conductance ($\mu\text{mhos/cm}$) Values for Three Rice Samples and for Their Intact-Kernel and Broken-Kernel Components (Extraction at 25°C with 1:1, v/v Isopropyl Alcohol-Water)

Rice Grain Type	Original Sample	Whole Kernels	Broken Kernels
Long	12.7	12.9	14.0
Medium	23.4	21.8	27.8
Short	20.2	20.2	25.5

TABLE IX
Specific Conductance ($\mu\text{mhos/cm}$) for Rice Samples Extracted at 25°C by Different Methods

Rice Grain	Automatic ^a	Shaker ^b (Speed 5)	Shaker ^b (Full Speed)
Short	20.1	22.5	25.6
Medium	23.5	25.6	28.2
Medium	24.0	24.0	28.8
Long	12.7	13.6	16.0
Long	17.7	18.2	23.1

^a Device shown in Fig. 1.

^b Commercially available Thermoshake Bath (Forma Scientific Co.).

comparable to those we obtained. Although the correlation coefficient between the data obtained by the first two methods was 0.991 ($P < 0.01$), the shaker bath is empirical and the operator must observe specific conditions for extraction time and shaker movement.

Evaluating Interpretive Line Samples

Table X summarizes the subjective and objective evaluations of nine commercially milled, interpretive line samples. The high percentage of broken kernels in the short grain samples was of concern. When specific conductances of the whole-kernel short-grain samples were measured, the values were 17.8, 23.9, and 23.9. Similar measurements for whole-kernel medium and long grain samples differed only slightly from the values in Table X.

The correlation coefficients for the subjective ratings with ash, surface lipid, free lipid, Agtron color, Hunter color, specific conductance, and specific conductance rating were -0.872, -0.771, -0.839, 0.532, -0.592, -0.891, and -0.866, respectively; the correlations for ash, free lipid, specific conductance, and specific conductance rating were all significant ($P < 0.01$).

The objective degree of milling ratings based on specific conductance differed from the subjective ratings for six samples. In all such cases the objective rating was lower (indicating that the sample was less well milled) than the subjective rating.

Reproducibility of the Results

Portions of undermilled and well-milled rice were analyzed in duplicate on 10 consecutive working days. The data (Table XI) show that the reproducibility of the specific conductivity method is very good. The standard deviation for the results for the undermilled sample was 0.57 (0.40 for the means of the two duplicate values) and 0.29 (0.21 for the means of the two duplicate values) for the well-milled sample. The coefficients of variation (relative standard deviations) for the two samples were 2.0 and 2.8%, respectively, indicating that the variation, as measured by the standard deviation, is a small percentage of the mean.

Comparison of the Subjective and Objective Specific Conductance Methods

The data in Fig. 9 indicate that an association between the subjective method for rating the degree of milling and the values obtained by measuring the specific conductance of an isopropyl alcohol-water (1:1, v/v) extract of rice. The standard correlation coefficients are not applicable for these data because the underlying assumptions for a valid correlation are violated, ie, correlation coefficients are not designed to correlate the four values that the subjective rating employs. Therefore, we used the method of Olkin and Tate (1961) to compare the subjective and objective methods. (Table XII). The small probabilities (Table XII) indicate that the correlation coefficients are detectably different from zero. This

TABLE X
Evaluation of Commercially Milled, Interpretive Line Samples

Rice Grain Type	Moisture (%)	Ash 14% mb (%)	Protein 14% mb (%)	Surface Lipid 14% mb (%)	Free Lipid 14% mb (%)	Color Reading (Blue Filter)		Kernel Weight ^a (mg)	Specific Conductance ^a ($\mu\text{mhos/cm}$)	Degree of Milling	
						Agtron	Hunter			Based on Specific Conductance	Subjective Rating
Short	13.4	0.49	6.6	0.40	0.14	54.5	30.0	21.5	19.5	3	4
	12.8	0.68	6.7	0.56	0.29	45.0	31.2	21.6	25.6	2	3
	13.7	0.74	6.8	0.62	0.29	37.5	32.0	22.2	27.3	2	2
Medium	11.4	0.57	8.1	0.31	0.07	35.0	32.1	16.9	18.8	3	4
	11.7	0.75	8.3	0.60	0.34	31.5	32.8	17.9	27.6	2	3
	12.1	0.76	7.0	0.79	0.44	26.5	34.1	18.7	27.9	2	2
Long	13.2	0.66	8.2	0.59	0.15	34.0	32.6	15.5	18.6	3	4
	12.5	0.74	7.9	0.79	0.14	30.5	33.4	15.5	23.9	2	3
	12.5	0.85	8.0	0.87	0.36	25.5	34.1	15.7	25.7	2	2

^a As-is basis.

TABLE XI
Specific Conductance ($\mu\text{mhos/cm}$) Values for Two Rice Samples
Measured in Duplicate on 10 Different Days^a

Undermilled		Well Milled	
28.6	27.9	10.4	10.6
27.6	27.6	10.0	10.2
29.1	29.6	10.7	10.6
28.8	28.8	10.4	10.5
29.0	28.6	10.4	10.5
29.0	28.6	10.5	10.6
28.3	29.2	11.0	10.6
27.7	28.6	10.9	11.4
28.8	27.9	10.4	10.5
29.0	28.2	10.4	10.4
Mean	28.5		10.6

^aFive-minute extraction of 10-g sample at 25°C using 40 ml of isopropyl alcohol-water (1:1, v/v).

TABLE XII
Statistical Evaluation^a of Data in Table II

Subjective Rating (from Table II)	Correlation Coefficient	Probability
	Between Specific Conductance and Subjective Rating	
I	-0.892	2.2×10^{-6}
II	-0.866	6.3×10^{-6}

^aAs described by Olkin and Tate (1961).

evidence and the relationship shown in Fig. 9 indicate that the association between the two methods is not likely to be due to chance.

There are scientific reasons why the objective specific conductance method is better than the subjective rating for measuring the degree of milling of rice. The specific conductance method is rapid and reproducible, can be performed by relatively unskilled personnel, and removes the error in judgment (Tables II and V) in the subjective method. In general, a method that allows continuous measurement, such as the specific conductance method, is preferable to a discrete rating method such as the subjective rating now employed to measure the degree of milling of rice.

It is questionable whether a new method, such as the specific conductivity method, can adequately predict what the rating would be by subjective rating. The objective rating could be superior to the subjective rating system and should not be restricted to predicting the results of a possibly inferior method. Large buyers of milled rice probably will not continue to be satisfied with results from subjective visual evaluation of rice if testing in rice mills indicates

that the objective method is better. If the objective method is adopted for measuring the degree of milling of rice, ranges for specific conductance (Table IV) should be allocated based on whole numbers, perhaps with inclusion of additional ranges.

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LITERATURE CITED

- AMERICAN ASSOCIATION OF CEREAL CHEMISTS. Approved methods of the AACC. Method 08-01, approved April 1961; Method 44-15A, approved April 1967; and Method 46-11, approved April 1961. The Association: St. Paul, MN.
- BAKER, D., NEUSTADT, M. H., and ZELENY, L. 1957. Application of the fat acidity test as an index of grain deterioration. *Cereal Chem.* 34:226.
- BARBER, S. 1972-74. Test methods for rice. ICC Study Group Report No. 21.
- BARBER, S. 1975-76. Test methods for rice. ICC Study Group Report No. 40.
- CLEMENTS, R. L. 1977a. Distribution of ash among flour extracts and fractions and its relation to electrical conductivity. *Cereal Chem.* 54:840.
- CLEMENTS, R. L. 1977b. Electrical conductivity of flour suspensions and extracts in relation to flour ash. *Cereal Chem.* 54:847.
- FAO. 1972. Degree of milling of rice: The standard method adopted by the Food Agency, Government of Japan. Sub-Group on Rice Grading and Standardization, Intergovernmental Group on Rice. FAO Document CCP:RI/GS C.R.S.1, Rome.
- HOGAN, J. T., and DEOBALD, H. J. 1961. Note on a method of determining the degree of milling of whole milled rice. *Cereal Chem.* 38:291.
- HOGAN, J. T., and DEOBALD, H. J. 1965. A review. Measurement of the degree of milling of rice. *Rice J.* 68(10):10.
- OLKIN, I., and TATE, R. F. 1961. Multivariate correlation models with mixed discrete and continuous variables. *Ann. Math. Stat.* 32:448.
- POMERANZ, Y., and LINDNER, C. 1956. The use of ion exchangers for determining the grade of flour. *Anal. Chim. Acta* 15:330.
- POMERANZ, Y., STERMER, R. A., and DIKEMAN, E. 1975. NMR-oil content as an index of degree of rice milling. *Cereal Chem.* 52:849.
- STERMER, R. A., WATSON, C. A., and DIKEMAN, E. 1977. Infrared spectra of milled rice. *Trans. ASAE* 20:547.
- WATSON, C. A., DIKEMAN, E., and STERMER, R. A. 1975. A note on surface lipid content and scanning electron microscopy of milled rice as related to degree of milling. *Cereal Chem.* 52:742.

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