IMPLICATIONS OF MOISTURE LOSS IN GRAINS INCURRED DURING SAMPLE PREPARATION

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ABSTRACT

Moisture loss incurred during the grinding process of sample preparation has been studied, using four different grinder models that are currently in operation in the Canadian Grain Commission's Grain Research Laboratory. The grains that were studied included three types of hard red spring wheat, soft red spring wheat, durum wheat, and two types of barley. Moisture levels ranged from 8 to 18%. Moisture content was determined with the official model 919 capacitance moisture meter and the AACC two-stage and single-stage air oven moisture tests. All grains were found to lose significant amounts of moisture at initial levels as low as 10%. Moisture loss during grinding has some implications on the subsequent computation of analytic results to a constant moisture basis. Such losses may constitute a continuing source of error in the second stage of two-stage air oven moisture testing in which grains have been ground at equilibrium moisture levels of 10% or more.

The moisture status of whole wheat may affect accuracy of protein results. This is caused in part by the dissipation of moisture from grains during the grinding process of sample preparation (1–4). The initial moisture status of the grain and the relative humidity of the atmosphere are among the factors that influence the extent of moisture loss.

The introduction of rapid electrical moisture-testing equipment to the grain industry (5–7) as a replacement for the Brown-Duvel distillation technique revolutionized the testing of grain for moisture. At the same time, it laid the foundation for the significant, perpetual source of error that prompted the investigations that are described here. The most common electronic methods for moisture testing use 50 to 250 g of intact grain kernels. Grain is usually pulverized in preparation for testing for protein and other constituents, however, and therein lies a significant source of error.

Reporting results of chemical analysis of grain on a constant moisture basis is a common practice, whether the chemical data is to be used in quality assessment for marketing or processing or for further scientific investigation. This procedure is probably used more widely in reporting protein content than is any other procedure. As a result, the moisture status of the test sample affects even the most painstaking protein tests, something that is frequently overlooked. Table I illustrates the influence of the inherent moisture content of wheat on correction of results of protein testing to a constant moisture basis. Table II shows the consequences of discrepancies in moisture content in terms of the magnitude of possible errors that are introduced at different protein levels. At an initial protein level of 14%, for example, a discrepancy of 3% in the moisture figure that is used to adjust the protein value to a constant moisture basis will lead to an error of nearly 0.5%.

When chemical constitution reflects the price of a commodity, the consistent loss or restitution of as little as 0.1% of moisture can have expensive

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consequences. Again, careful adherence to the accurate assessment of moisture status can minimize the consistent discrepancies of 0.1–0.3% in protein and other constituents that frequently perturb the practical appraisal of new analytic techniques. The chief objective of these investigations was to assess the manner in and extent to which this type of inaccuracy can arise in different grains.

MATERIALS AND METHODS

The study included seven types of grain, namely, hard red spring (HRS) wheat, grades No. 1 and 2 Canada Western (1 and 2 CW HRS) and No. 3 Canada Utility (3 CU HRS); barley, grades No. 2 CW 6-row (2 CW 6-row) and No. 1 Feed; amber durum (AD) wheat, grade No. 2 CW; and soft red spring (SRS) wheat, grade No. 1 CU Pitic 62 SRS. Oats were not included because of complications in grinding high-moisture oats on burr-type mills.

Samples (25-kg) of all grains were obtained from the Grain Inspection Division of the Canadian Grain Commission. The grains were subsamples of bulk supplies of cleaned grain originating from terminal elevators at Thunder Bay, Ont.

Four types of grinder were used, all of which are employed in certain aspects of the operation of the grain research laboratory. The Hobart 2040 coffee grinder was used for routinely processing wheat, durum wheat, barley, and rye for Kjeldahl protein testing. The Tecatur/Udy Cyclotec grinder, formerly the Udy Cyclone Sample Mill, was used for processing all grains for protein testing by

TABLE I
Influence of Inherent Moisture Content of Wheat on Correction of Protein Testing Results to Constant Moisture Basis of 13.5%

	Initial Moisture Content					
As-Is Protein Content	8%	10%	12%	14%	16%	18%
14.23% corrects to	13.4	13.7	14.0	14.3	14.6	15.0
11.16% corrects to	10.5	10.7	11.0	11.2	11.5	11.8

TABLE II
Influence of Discrepancies in Moisture Content of Wheat on Accurary of Reporting Subsequent Protein Tests

Moisture Discrepancy	Percent of Error at Various Protein Levels		
(%)	10%	14%	18%
±0.5	0.06	0.09	0.12
±1.0	0.11	0.16	0.21
±2.0	0.23	0.32	0.41
±3.0	0.34	0.48	0.61
±4.0	0.45	0.63	0.81

near infrared reflectance spectroscopy (NIRS). The Krups impeller-type mill, or an equivalent mill, was used to process high-fiber, high-moisture grains for Kjeldahl protein-testing. The Buhler Laboratory grinder was used for pulverizing grains in the second stage of two-stage air oven moisture (2SAO) testing.

Tempering the grains with appropriate additions of demineralized water produced high moisture levels. The water was added to each of four 250-gm replicate subsamples of each grain. The tempered grains were thereafter stored in sealed cans at room temperature (about 22° C). A pilot experiment showed that the grains attained a stabilized moisture level (919 meter) after 48 hours. The samples were allowed to stand for at least 20 days to ensure thorough equilibrium. After seven days at room temperature and during the final two weeks of tempering the samples were placed in cold storage at 5° C to inhibit development of molds. Allowing the grains to stand in open trays under warm (35° C), well-aerated conditions achieved low moisture levels. A total of 11 moisture levels were set for all grains.

Moisture was tested by three methods. The first was the model 919 (Motomco) meter. The AACC 2SAO test (8) was used for whole-grain studies; the Canadian Grain Commission regards this test as the standard moisture test against which all meters are calibrated. Finally, moisture status after grinding was determined by the AACC single-stage air oven (SSAO) test (8). The experimental design thus became a 4 × 3 × 11 factorial, with four replicates.

Samples (25-g) were ground on the respective grinders for SSAO testing, care being taken to ensure complete recovery. After grinding, all samples were stored in 2-oz salve tins and sealed with 3/4-in. masking tape. The sample size represents the amount that is normally ground for protein testing in the process that the

TABLE III
Rate of Moisture Loss From Ground HRS[a] Wheat Stored in Metal Cans[b] With and Without Taping

Days From Initial Moisture Test	Initial Moisture Content: 14.3% Moisture Loss (%)		13.4% Moisture Loss (%)		12.6% Moisture Loss (%)		11.3% Moisture Loss (%)		10.4% Moisture Loss (%)	
	Taped	Untaped	Taped	Untaped	Taped	Untaped	Taped	Untaped	Taped	Untaped
0	0	0	0	0	0	0	0	0	0	0
1	0	0.1	0	0	0	0.1	0	0	0	0
2	0	0.9	0	0.7	0	0.4	0	0.4	0	0.1
3	0	2.2	0	1.2	0	0.9	0	0.7	0	0.3
4	0	3.3	0	2.0	0	1.4	0	0.7	0	0.3
5	0	3.7	0	2.6	0	2.2	0	0.9	0	0.3
6	0	4.0	0	2.8	0	2.1	0	0.8	0	0.3
21	0.4	...	0.2	...	0.2	...	0.1	...	0.1	...
42	0.6	...	0.5	...	0.5	...	0.1	...	0	...

[a]HRS = hard red spring.
[b]2-oz (50-g) capacity.

commission uses. The cans were allowed to stand under normal laboratory conditions. Kjeldahl protein (N × 5.7 for wheat, N × 6.25 for barley) was determined on all samples; the results were corrected to a 13.5% moisture basis according to the various moisture values recorded to assess the influence of discrepancies in moisture values on protein results. The Winkler (boric acid) modification of the Kjeldahl procedure was used (9). A second pilot experiment was done to establish the validity of this storage method. Table III summarizes the results of storage with and without taping. The rapidity with which moisture disappeared from high-moisture–ground grain that was stored in untaped cans was remarkable.

RESULTS

Influence of Initial Moisture Level, Grinder, and Type of Grain on Moisture Status of Grain After Grinding

Tables IV–X summarize the moisture loss that the various grains sustained as a result of normal laboratory grinding. The moisture loss figures were attained by comparing the SSAO results of ground grains with the initial 2SAO results of intact grains. The 919 meter results are included for comparison and also to verify that a satisfactory tempering had been achieved in the case of the high

TABLE IV
Moisture Loss Incurred by 1 CW HRS[a] Wheat During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	7.4	7.9	0	0	0.4	0
	9.5	9.2	0.6	0.7	1.3	0.6
	10.0	10.0	0.3	0.6	0.9	0.2
	10.9	10.9	0.5	0.8	1.2	0.5
	11.8	11.6	0.5	1.2	1.4	0.4
	12.6	12.4	0.8	1.3	1.6	0.7
	13.5	13.3	0.7	1.7	1.7	0.5
	14.3	14.2	0.8	1.6	1.9	0.6
	15.4	15.5	1.0	2.3	2.4	0.8
	16.3	16.2	1.5	2.5	2.7	1.2
	17.3	17.0	1.3	3.4	2.9	1.2
SE/test[d]	0.149	0.080	0.070	0.101	0.145	0.082
b[e]	...	1.026	1.141	1.417	1.264	1.113
a[f]	...	−0.25	−0.93	−3.19	−1.30	−0.72
r[g]	...	0.997	0.993	0.991	0.989	0.996
SEE[h]	...	0.238	0.353	0.391	0.433	0.278

[a]1 CW HRS = grade No. 1 Canada Western hard red spring.
[b]2SAO = two-stage air oven test.
[c]2SAO result/SSAO result: mean of four replicates.
[d]SE/test = standard error per test.
[e]b = regression coefficient of individual X values on Y (2SAO).
[f]a = regression intercept.
[g]r = correlation coefficient.
[h]SEE = standard error of estimating Y from individual X values.

TABLE V
Moisture Loss Incurred by 2 CW HRS[a] Wheat During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	8.2	8.3	0.0	0.4	0.4	0.0
	9.8	9.8	0.9	0.0	0.2	0.1
	10.3	10.4	0.5	0.8	0.9	0.5
	11.2	11.2	0.5	1.0	1.6	0.6
	12.4	12.0	0.8	1.4	1.6	0.7
	13.6	12.8	1.0	1.8	2.0	1.0
	14.6	14.4	1.3	2.8	1.7	1.3
	15.7	15.5	1.3	2.9	2.0	1.4
	16.5	16.0	1.4	2.8	2.0	1.5
	18.0	17.7	2.1	4.0	2.6	1.8
	19.4	18.8	3.0	5.0	4.0	3.1
Mean Moisture (%)	13.61	13.4	12.5	11.4	11.9	12.5
SE/test[d]	0.15	0.15	0.25	0.17	0.20	0.19
b[e]	...	1.068	1.345	1.648	1.326	1.297
a[f]	...	−0.66	−2.96	−5.30	−2.21	−2.62
r[g]	...	0.996	0.955	0.982	0.982	0.991
SEE[h]	...	0.31	1.06	0.68	0.68	0.48

[a]2 CW HRS = grade No. 2 Canada Western hard red spring.
[b–h]See Table IV.

TABLE VI
Moisture Loss Incurred by 3 CU HRS[a] Wheat During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	8.9	8.2	0.3	0.3	0.1	0
	9.0	9.2	−0.4	−0.4	0.0	0.0
	10.1	10.2	−0.1	0.0	0.1	−0.2
	11.4	11.0	0.6	0.8	0.9	0.8
	12.6	12.0	1.2	1.6	1.4	1.2
	12.9	12.8	0.9	1.4	0.9	1.0
	14.0	14.0	0.9	1.6	1.4	0.8
	15.2	14.9	1.0	2.2	1.4	1.0
	16.2	15.9	1.2	2.4	1.6	1.2
	17.2	17.1	1.6	3.1	1.8	1.2
	18.1	18.0	1.8	3.5	2.1	1.2
SE/test[d]	0.242	0.049	0.120	0.077	0.144	0.098
b[e]	...	1.008	1.241	1.613	1.209	1.143
a[f]	...	0.041	−2.27	−5.77	−1.44	−0.65
r[g]	...	0.988	0.984	0.981	0.981	0.981
SEE[h]	...	0.499	0.57	0.625	0.628	0.632

[a]3 CU HRS = grade No. 3 Canada Utility hard red spring.
[b–h] See Table IV.

TABLE VII
Moisture Loss Incurred by 1 CU Pitic 62[a] Wheat During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	8.0	8.0	0	0.2	0.6	0.3
	8.7	8.9	0	0.3	0.4	0.1
	9.6	9.9	0.3	0.7	0.7	0.2
	10.3	10.9	0.1	0.2	0.3	0
	11.4	12.2	0.1	0.4	0.1	−0.2
	13.0	13.3	0.9	1.4	1.4	0.7
	13.5	14.2	0.8	1.1	0.9	0.3
	14.6	14.9	1.3	3.3	2.4	4.9
	15.3	15.8	1.7	3.8	2.1	4.5
	15.8	16.6	1.1	2.5	1.5	0.8
	17.2	17.8	1.4	3.2	1.7	1.0
SE/test[d]	0.151	0.100	0.123	0.133	0.181	0.152
b[e]	...	0.953	1.217	1.467	1.192	0.949
a[f]	...	0.14	−1.85	−3.558	−1.092	1.722
r[g]	...	0.996	0.993	0.942	0.979	0.808
SEE[h]	...	0.257	0.365	1.014	0.614	1.787

[a]1 CU Pitic 62 = grade No. 1 Canada Utility Pitic 62 soft red spring.
[b–h]See Table IV.

TABLE VIII
Moisture Loss Incurred by 2 CW 6-Row[a] Barley During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	8.0	9.5	0.3	0.6	0.6	0.1
	9.7	10.3	0.4	1.3	0.9	0.3
	11.4	11.6	0.8	2.0	1.0	0.5
	12.0	12.2	0.6	2.3	1.0	0.4
	13.0	12.9	1.1	2.4	1.3	0.6
	13.7	13.7	1.1	3.0	1.2	0.5
	14.9	14.5	1.4	3.9	1.9	0.9
	15.6	15.4	1.8	4.6	2.1	0.9
	17.6	17.5	2.6	4.9	2.2	1.2
SE/test[d]	0.176	0.086	0.110	0.123	0.130	0.084
b[e]	...	1.176	1.29	1.857	1.208	1.122
a[f]	...	−2.48	−2.26	−5.86	−1.03	−0.882
r[g]	...	0.991	0.992	0.980	0.992	0.996
SEE[h]	...	0.388	0.378	0.580	0.372	0.251

[a]2 CW 6-row = grade No. 2 Canada Western 6-row barley.
[b–h]See Table IV.

TABLE IX
Moisture Loss Incurred by No. 1 Feed Barley During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[a] (%)	919 Meter (%)	Hobart[b] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	6.3	7.0	0	0.1	0.1	0
	8.8	9.3	0	0.3	0.2	−0.1
	9.6	10.0	0.6	0.8	0.4	0.2
	10.4	10.8	0.4	1.2	0.3	0
	11.7	11.8	1.1	2.1	0.4	0.3
	12.7	12.8	0.5	2.7	0.3	0.1
	14.2	13.7	1.7	3.1	0.9	0.5
	15.5	14.7	0.7	3.6	1.7	0.8
	16.4	15.6	0.9	4.7	2.1	1.4
	17.6	16.8	1.2	3.6	1.8	1.0
	18.6	17.8	1.2	4.2	1.7	1.0
SE/test[c]	0.151	0.078	0.155	0.173	0.132	0.080
b[d]	...	1.123	1.089	1.559	1.189	1.114
a[e]	...	−1.33	−0.30	−3.45	−1.375	−0.933
r[f]	...	0.993	0.992	0.979	0.992	0.997
SEE[g]	...	0.462	0.466	0.788	0.479	0.306

[a-g]See Table IV, footnotes b–h.

TABLE X
Moisture Loss Incurred by 2 CW[a] Amber Durum Wheat During Grinding

	Initial Moisture		Moisture Loss			
	2SAO[b] (%)	919 Meter (%)	Hobart[c] (%)	Cyclotec (%)	Krups (%)	Buhler (%)
	7.4	7.9	−0.1	0.2	0.1	0.1
	8.7	8.9	0.3	0.5	0.3	0.1
	10.2	10.1	0.5	0.8	0.6	0.5
	11.2	11.0	0.8	1.1	1.0	0.3
	12.3	12.0	1.0	1.5	0.9	0.5
	13.5	13.1	0.8	1.5	1.1	0.7
	14.6	14.1	1.8	2.4	1.8	1.0
	15.3	14.8	1.8	2.6	1.9	0.9
	16.6	15.8	1.2	2.7	1.9	1.4
	17.7	16.6	1.7	3.3	1.8	1.2
	18.7	17.6	2.0	3.8	2.2	1.5
SE/test[d]	0.090	0.065	0.115	0.084	0.103	0.073
b[e]	...	1.148	1.191	1.447	1.225	1.142
a[f]	...	−1.53	−1.25	−3.265	−1.481	−1.016
r[g]	...	0.999	0.995	0.996	0.996	0.998
SEE[h]	...	0.127	0.369	0.337	0.309	0.216

[a]2 CW = grade No. 2 Canada Western.
[b-h]See Table IV.

moisture levels. (The Grain Inspection Division uses the 919 meter for official testing of all grains for moisture at terminal elevators and at all other government inspection laboratories.) The chief objective of the experiment, however, was to assess changes in moisture level compared with the standard, or 2SAO, method for moisture estimation. The regression data are included for predicting 2SAO figures from all other moisture data. The regression statistics are based on tests of all individual samples used in the experiment; each equation represents 44 sets of data except for the 2 CW 6-row barley, which incorporated 36 sets of data.

The generally close agreement between 919 meter and 2SAO results verified that a satisfactory degree of tempering had been achieved, and that on the average, the 919 meter procedure was capable of predicting the results of 2SAO testing to within about 0.33% of moisture, which represented a coefficient of variability of 2.5%. Table XI summarizes the standard error of duplicate testing by all methods. The standard error of testing with the 919 meter was significantly lower than that with the 2SAO method. The chief advantage of the latter is in accuracy rather than precision for the testing of all grain, particularly at moisture levels below 9% and above 17%. The 919 meter operates on the principle of capacitance and is less accurate at extremes of moisture content.

At moisture levels above 10%, the grinding of all grains was accompanied by losses in moisture that depended on the grain and the grinder as well as the moisture level. All grinding and therefore all SSAO testing was performed on subsamples of the same 250-gm sample that the 2SAO and 919 tests represent, so that whatever the final moisture status of the ground grain, all samples were of the same moisture status immediately before grinding.

TABLE XI
Standard Error of Duplicate Testing for Moisture[a]

	Test Procedure						
Grain	2SAO[b]	919 Meter	SSAO[c] Hobart	SSAO Cyclotec	SSAO Krups	SSAO Buhler	All Grinders
1 CW HRS[d] wheat	0.15	0.08	0.07	0.10	0.14	0.08	0.09[1]
2 CW HRS[e] wheat	0.15	0.15	0.25	0.17	0.20	0.19	0.21[3]
3 CU HRS[f] wheat	0.24	0.05	0.12	0.08	0.14	0.10	0.12[1]
1 CU Pitic 62[g] wheat	0.15	0.10	0.12	0.13	0.18	0.15	0.14[2]
2 CW 6-row[h] barley	0.18	0.09	0.11	0.12	0.13	0.08	0.11[1]
1 Feed[i] barley	0.15	0.08	0.16	0.17	0.13	0.08	0.14[2]
2 CW durum[j] wheat	0.09	0.06	0.12	0.08	0.10	0.07	0.09[1]
All grains	0.17[2]	0.10[1]	0.14[2]	0.11[1]	0.15[2]	0.11[1]	0.12

[a]Means with different superscripts were significantly different from other means ($P = 0.05$).
[b]2SAO = two-stage air oven test.
[c]SSAO = single-stage air oven test.
[d]1 CW HRS = grade No. 1 Canada Western hard red spring.
[e]2 CW HRS = grade No. 2 Canada Western hard red spring.
[f]3 CU HRS = grade No. 3 Canada Utility hard red spring.
[g]1 CU Pitic 62 = grade No. 1 Canada Utility Pitic 62 soft red spring.
[h]2 CW 6-row = grade No. 2 Canada Western 6-row.
[i]1 Feed = grade No. 1 Feed.
[j]2 CW = grade No. 2 amber durum.

Table XII indicates the average moisture lost during grinding with the different grains and grinders. The Cyclotec grinder sustained the greatest losses, followed by the Krups; the Buhler sustained the least losses. The 2 CW HRS wheat lost significantly more moisture than did all other grains, among which were no significant differences. The standard error of duplicate testing by SSAO after grinding revealed that the Cyclotec and Buhler grinders were significantly lower in standard error than were the Hobart and Krups grinders. Again, 2 CW HRS wheat was significantly higher in standard error of testing than were all other grains; the 1 CU Pitic 62 SRS wheat and 1 Feed barley were both significantly higher in testing error than were the remaining grains.

At high moisture levels the Hobart and Buhler grinders did not cause moisture loss to the same extent as did the other two grinders, although at low moisture levels less discrepancy appeared between the four sets of moisture results. The chief reason for this is that at high moisture levels, both burr-type mills (Hobart and Buhler) tended to gumup. The revolutions dropped markedly, and in some cases the samples could not be ground without increasing the clearance between the burrs. The Buhler grinder ceases to function if the torque ratio becomes excessive. Although sufficient material was ground to complete this study (*i.e.*, the necessary 25 g), the particle size of the ground high-moisture material was coarse. Residual material of each sample was removed from the grinder by scraping and brushing. This increases the standard error of testing for both moisture and protein, and would render both grinders completely unsuitable for the recently introduced NIRS technique. The pattern of moisture loss that the Buhler grinding caused was particularly erratic in the case of the SRS wheat. At 14–16% initial moisture, the moisture loss was greater even than with the Cyclotec mill; at higher moistures the loss dropped off to a low level. Again, this behavior was an illustration of the reaction of the grinder to the type of grain.

Table XIII summarizes the significant aspects of the analysis of variance for the moisture figures for 2 CW HRS wheat. Interactions between moisture level and moisture method, moisture level and grinder type, and moisture level, grinder, and moisture method were all significant, indicating that all three

TABLE XII
Mean Percent of Moisture Lost During Grinding on Different Grains and Grinders[a]

Grain	Hobart	Cyclotec	Krups	Buhler	All Grinders
1 CW HRS[b] wheat	0.73	1.46	1.67	0.61	1.12[2]
2 CW HRS[c] wheat	1.08	2.16	1.73	1.09	1.52[1]
3 CU HRS[d] wheat	0.86	1.54	1.06	0.78	1.06[2,3]
1 CU Pitic 62[e] wheat	0.70	1.55	1.10	1.18	1.13[2]
2 CW 6-row[f] barley	1.12	2.27	1.11	0.49	1.25[2,3]
1 Feed[g] barley	0.66	2.40	0.90	0.47	1.11[2]
2 CW durum[h] wheat	1.07	1.85	1.24	0.75	1.23[2]
All grains	0.89[3]	1.89[1]	1.26[2]	0.77[3]	1.20

[a]Means with different superscripts were significantly different from other means ($P = 0.05$).
[b-h]See Table XI, footnotes d–j.

variables affected moisture loss. Interaction between moisture method (*i.e.*, 919, SSAO, and 2SAO) and grinder type was not significant; this is logical, because two of the three tests are aimed at testing intact grains. This analysis of variance is typical of that for all other grains.

Influence of Time of Sustained Grinding on Moisture Loss by Cyclotec Grinder

The Cyclotec grinder is widely used for preparing samples for routine protein testing in the grain commission program for segregating HRS wheat on the basis

TABLE XIII
Summarized Analysis of Variance of Moisture Data for 2 CW HRS[a] Wheat

Source of Variance	F	P
Replication	0.6	NS[b]
ML[c]	2096.3	0.001
G[d]	29.3	0.01
MM[e]	32.2	0.01
ML × G	1.2	NS
ML × MM	11.2	0.01
G × MM	20.6	0.01
ML × G × MM	8.0	0.01
LSD[f]		
ML	0.13 ($P = 0.05$)	0.17 ($P = 0.01$)
G	0.22 ($P = 0.05$)	0.28 ($P = 0.01$)
MM	0.26 ($P = 0.05$)	0.35 ($P = 0.01$)

[a]2 CW HRS = grade No. 2 Canada Western hard red spring.
[b]NS = not significant.
[c]ML = moisture level.
[d]G = grinder.
[e]MM = moisture method.
[f]LSD = lowest significant difference.

TABLE XIV
Moisture Lost by Wheat After Different Intervals of Operation of Cyclotec Grinder[a]

Initial Moisture Level (919) (%)	Grinding Interval (min)					
	0	15	30	60	120	240
9.0	0.0	0.2	0.3	0.3	0.3	0.3
11.0	0.1	1.0	1.0	1.0	0.9	0.8
12.3	1.1	1.9	1.4	1.5	1.6	1.5
14.2	1.2	1.9	2.0	2.1	2.1	2.0
16.1	1.7	2.8	3.2	3.0	3.1	2.7
17.8	2.4	3.2	3.4	3.7	3.4	3.5
Mean	1.08[1]	1.83[2]	1.88[2]	1.93[2]	1.90[2]	1.80[2]

[a]Means with different superscripts were significantly different from other means ($P = 0.05$).

of protein content. More than 500 samples are commonly processed per day through a single grinder. To investigate the extent to which moisture was lost over prolonged periods of grinding, 12 samples of 1 or 2 CW HRS wheat of different moisture content were subdivided into six sublots of 50 g each. The high and low moisture levels were obtained as described above. Duplicate 25-g samples were ground on the same Cyclotec grinder after it had been operating continuously under routine grinding conditions for various intervals. The results are summarized in Table XIV.

The moisture loss increased markedly over the first 15 min that the grinder was in operation; increase in moisture loss was not significant thereafter with wheat at initial moisture levels of up to 14%. Higher-moisture wheat showed further slight increases up to 30 min, but no significant differences thereafter.

For this study, the initial reference moisture level was the 919 figure, since under all operational conditions it is the 919 meter that is used to assess the moisture status of all grains in the grain commission inspection laboratories and throughout the Canadian grain trade. This study indicated that differences do occur between the accuracy of the 919 and 2SAO test results, which may represent calibration effects.

The 919 results for 1 and 2 CW HRS wheat, 3 CU HRS wheat, and 2 CW 6-row barley all agreed closely with those for the 2SAO method, particularly at moisture levels above 10%. The 919 results for 1 Feed barley and 2 CW AD wheat deviated from those of the 2SAO, particularly at higher moisture levels. In both instances, the 919 results were higher than were the 2SAO results at lower moisture levels, which suggested that a change in the slope of the regression line could lead to improved accuracy.

On the other hand, the 919 results for the 1 CU Pitic 62 SRS wheat were all higher than those for the 2SAO test. Whenever the occasion arises to test a sample of 1 CU Pitic 62 SRS wheat for moisture, the practice is to use the hard red winter chart to convert the 919 readings to moisture. The above results indicate that a separate chart would be necessary for the testing of Pitic 62-type

TABLE XV
Reproducibility of Moisture Loss in Grinding of HRS[a] Wheat by Cyclotec Grinder[b]

Initial Moisture Level (919)[c] (%)	Average Moisture Loss (%)	Standard Deviation of Moisture Loss (%)	CV of Moisture Loss[d] (%)
9.5	0.6	0.31	3.3
11.0	1.0	0.42	3.8
12.5	1.7	0.31	2.4
14.2	2.2	0.39	2.8
15.8	2.8	0.43	2.7
17.4	3.4	0.48	2.8
19.0	4.0	0.55	2.9

[a]HRS = hard red spring.
[b]Covers period March 1975–December 1976.
[c]Mean of all initial 919 moisture figures.
[d]Coefficient of variability of standard deviation of moisture loss over initial 919 moisture figures.

wheats should they ever constitute a significant proportion of grain movements in Canada.

In the present study the use of 919 data throughout would have provided spurious data in that, compared with the 919, the Buhler-ground 2 CW AD wheat and 1 Feed barley did not differ significantly in moisture content from the results indicated by the 919 meter, even at nearly 18% moisture.

Slight variation in performance of 919 moisture meters is an accepted fact. The Canadian Grain Commission continuously monitors biweekly all 919 meters that the commission uses; a summary of these results is published in the grain research laboratory's annual report. The standard deviation of meter results from the overall mean is usually about 0.2%. The meter used in the above experiments was included in the biweekly checking and service; as a result, the above deviations for certain grains are not considered likely to have arisen as a result of abnormally variable meter readings.

Precision of Moisture Loss Results by Cyclotec Grinder

The regression coefficients for the SSAO moisture on the 2SAO moisture results after grinding on the Cyclotec grinder were significantly different from those of all other moisture test results, mainly because the discrepancies in the SSAO Cyclotec grinds from the 2SAO figures were larger than in all other systems. The grain commission widely uses the Cyclotec grinder for preparing samples of HRS wheat for protein testing, both by Kjeldahl and NIRS procedures. This is because the Cyclotec grinder has been the most satisfactory grinder for use in connection with NIRS.

Due to the large volume of Kjeldahl testing in surveys and monitoring services, the moisture tests on samples tested for Kjeldahl protein are usually done with the model 919 moisture meter, which works on intact kernels. As a result, the Canadian Grain Commission has reinvestigated the moisture loss/initial moisture status relationship on a quarterly basis since the first official use (June

TABLE XVI
Mean Annual Moisture Content of Principal Canadian Grains (1970–1976)[a]

Grain	Grade	Mean Moisture 919 Meter (%)	Probable Moisture Loss Cyclotec Grinder[b] (%)	Probable Moisture Loss Hobart Grinder[b] (%)
HRS[c] wheat	1 CW[d]	13.1	1.5	0.5
HRS wheat	2 CW	14.0	2.7	1.1
HRS wheat	3 CU[e]	14.2	1.7	1.0
Durum wheat	All grades	13.1	2.8	1.0
Feed barley	All grades	13.9	3.0	1.6
All grains	...	13.6	2.4	1.1

[a]All grains except durum wheat, weighted mean for carlots; durum wheat, weighted mean cargo shipments
[b]Calculated from regression equations based on 919 meter data.
[c]HRS = hard red spring.
[d]CW = Canada Western.
[e]CU = Canada Utility.

TABLE XVII
Discrepancies in Reporting 2 CW HRS[a] Wheat Protein on a Constant (13.5%) Moisture Basis Using Different Moisture-Testing Procedures

Initial Protein (%)	Percent of Moisture[b,c]															
	SSAO[d] (Cyclotec)				SSAO (Buhler)				919 Meter				2SAO[e]			
	10	12	14	16	10	12	14	16	10	12	14	16	10	12	14	16
10	9.7	10.1	10.5	11.0	9.5	9.9	10.2	10.7	9.5	9.8	10.1	10.4	9.5	9.8	10.1	10.3
13	12.7	13.2	13.7[f]	14.2	12.5	12.9	13.3	13.9	12.4	12.9	13.1[f]	13.5	12.4	12.9	13.1	13.4
16	15.6	16.2	16.8	17.5	15.4	15.9	16.4	17.0	15.4	15.8	16.2	16.6	15.4	15.7	16.1	16.5

[a] 2 CW HRS = grade No. 2 Canada Western hard red spring.
[b] All moisture figures converted to 2SAO equivalent, using regression equations derived from current experimental data.
[c] LSD ($P = 0.05$) = 0.12% protein.
[d] SSAO = single-stage air oven test.
[e] 2 SAO = two-stage air oven test.
[f] A sample of wheat at 13% protein and 14% moisture (model 919) would lose moisture when ground on the Cyclotec grinder so that the apparent Kjeldahl protein content of the ground sample would be raised to the extent that the result would be reported as 13.7% (instead of 13.1%) if the original 919 moisture figure were used to correct to 13.5% basis.

1975) of NIRS for protein testing of wheat in the segregation program. The moisture loss figures have been consistent over that period, as the regression data in Table XV indicate.

The work load increases significantly in May of each year and decreases in January due to freezing of the Thunder Bay port facility. Consequently, two different equations are used for correcting Kjeldahl protein to a constant moisture basis. The first is effective from January through April, and the second from May through December. At the average initial moisture level of about 13% for HRS wheat, the change in work load results in a decrease in moisture loss of about 0.5% during the "close season."

Implications of Moisture Loss on Protein Results

When protein results are to be reported on a moisture-free basis, or any other constant moisture basis, then losses of moisture incurred during grinding can constitute an important, continuous source of error. The moisture status of the sample becomes as important as the protein result itself, since when the protein content affects the price of the grain, even 0.1% of protein assumes a significance that depends on the incremental value of protein. In the northern part of the United States, for example, the price of red spring wheat not uncommonly increases 3–4 ¢ for each percentage point of protein. In the Canadian system, the segregation procedure has been honed to a fine degree of precision, and the binning guidelines for segregating designated levels of protein are based on mean protein, population standard deviation, area of origin of the wheat, and standard error of testing, among other parameters. When the parcels of wheat involved consist of multiples of 25-tonne (or 1000-bu) price increments of even 1¢ per 0.1% protein can result in serious financial consequences. The average moisture content of the top segregated grades of Canadian HRS wheat have averaged more than 13% moisture over the past six years since segregation commenced (Table XVI).

Table XVII summarizes the results of reporting protein figures on a constant moisture basis, using 919, SSAO, or 2SAO moisture figures. The moisture figures used in the protein segregation program for Canadian HRS wheat have always taken moisture loss into account. The current test procedure incorporates NIRS testing for both protein and moisture on the ground samples, so that moisture loss is not a factor. This was one of the attractive features of the NIRS process that prompted the grain commission's decision to employ the technique for all of its large-scale protein testing.

Table XVII clearly shows that consistent errors in protein testing can occur if moisture loss during grinding is ignored. Whether results are reported on a constant moisture basis or not, the discrepancies do occur, because the moisture status of grain almost invariably fluctuates between delivery, load out, and marketing; protein testing occurs in at least two of these three areas. These figures could be applicable to all of the grains tested in the above experiments, provided that the appropriate regression equations are used. Discrepancies between results of protein tests that were done on the same sample at different times or locations or both are frequently assigned to errors in the Kjeldahl (or other) test process, whereas in fact, many of the 0.1–0.3% deviations are caused by fluctuations in the moisture status of inadequately protected samples.

Influence of Moisture Loss on 2SAO Testing Procedure

The above experiments drew attention to a possible source of error in the standard AACC 2SAO procedure for moisture (8). According to the operating procedure, a weighed sample of whole grain at field, or "receival," moisture is allowed to reach a moisture level of less than 13% under test laboratory conditions, whereafter the grain is reweighed and the moisture loss recorded as A stage moisture. The sample is then ground to a meal and an SSAO test is carried out on the ground sample; this is the B stage moisture value. After making a correction for the loss in the initial weight of the sample during A stage, B stage and A stage figures are combined to compute the overall 2SAO test result.

A 13% moisture level at A stage may result in the introduction of an undetected error to the 2SAO test. Loss in moisture occurred at relatively low initial moisture levels for all grains studied in the above experiments with the Buhler grinder only, since this was the grinder that caused the lowest loss in moisture in the study. At the 10% initial moisture level, moisture loss ranged up to 0.5%; at the 13% initial level, loss mounted to 1.0% in wheat. This moisture loss would remain undetected in a 2SAO test, and would result in discrepancies that could be significant, for example, in the appraisal of new moisture-testing equipment or in the calibration of a new type of instrument or technique.

The AACC recommends use of the Wiley Intermediate grinder for the 2SAO test, since this grinder is reputed to cause no moisture loss up to 18% initial moisture. In our experience, however, significant amounts of moisture may be lost well below the 18% level with the Wiley grinder. Furthermore, certain laboratories that do not possess the specified grinder may find a 2SAO moisture test necessary at intervals that do not justify the purchase of the grinder for that sole purpose.

This potential source of error can be eliminated by ensuring that the grain during A stage of the 2SAO procedure is allowed to dry down to no higher than 9%, and preferably to 7–8%, moisture before proceeding to B stage. Allowing the grain to stand in a ventilated cabinet with the temperature about 35°C for 48 hours can achieve this. Without such a cabinet, lower moisture levels can be attained by allowing the grain during A stage to stand in a warm, well-aerated area of the laboratory, such as on top of an air oven. The samples should then be protected from dust accumulation by means of a layer of light tissue.

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