

USING A DENSITY GRADIENT COLUMN TO DETERMINE WHEAT DENSITY¹

WENDELL R. PETERS AND ROBERT KATZ

ABSTRACT

A wheat kernel placed in a density gradient column will sink until it reaches the level where its average density is equal to that of the liquid at that level. Knowing the density of the liquid as a function of height, one can measure accurately the density of the kernel to three decimal places in about 1 to 2 minutes per kernel. Kernel density ranged from a maximum of 1.416 at 7.5% moisture to a minimum of 1.255 g./cm.³ at 18.5% moisture, for the samples tested. Kernels of a homogeneously selected sample of hard spring wheat (Lee) varied in density from 1.29 to 1.41 g./cm.³ at 12% moisture. A clear separation of grain infested with rice weevil from noninfested grain was found for larvae 20 days old by using a solution of density 1.26 g./cm.³ Protein and ash content have been found to vary with density, and to pass through a maximum in some cases.

HRS

The aim of this work was to find a quick and accurate method to measure the density of individual wheat kernels and to exhibit some application of the method. Other workers (see references in Campbell and Jones, 1) have used varied procedures for measuring the density of grain. In one of the procedures previously used, a sample of many kernels of grain, along with liquid of known density, was put into a pycnometer bottle. Accurate measurement of average kernel density results from knowing the weight of the sample and the amount of liquid displaced from the bottle by the grain. This procedure allows no analysis of the density spectrum of individual kernels. Another form of density measurement previously used involves titrating a liquid of one density with a liquid of another density until the wheat kernel remains suspended in the liquid. The density of the kernel is then equal to the density of the suspending liquid. This procedure is quite time-consuming.

The present work represents a study of the application of the density gradient column to the study of wheat kernel density and infestation problems. With the use of the density gradient column, the densities of 10 to 20 kernels, introduced into the column at one time, can be measured with accuracy to the third decimal place at a rate of 1 to 2 minutes per kernel.

Materials and Methods

Constructing the Gradient Column. The density gradient column

¹Manuscript received February 19, 1962. Contribution No. 91, Department of Physics, Kansas Agricultural Experiment Station, Manhattan.

consists of a vertical tube containing a transparent liquid in which a decrease in height corresponds to an increase in density. A wheat kernel placed in this column will sink until it reaches the level where its average density is equal to that of the liquid at that level. When the column is calibrated with test objects of known density, the density of the kernel can be determined.

To maintain a stable gradient (5), it is necessary to have continuously operating sources and sinks for the diffusing components at the ends of the vertical column (Fig. 1). If the reservoirs, which

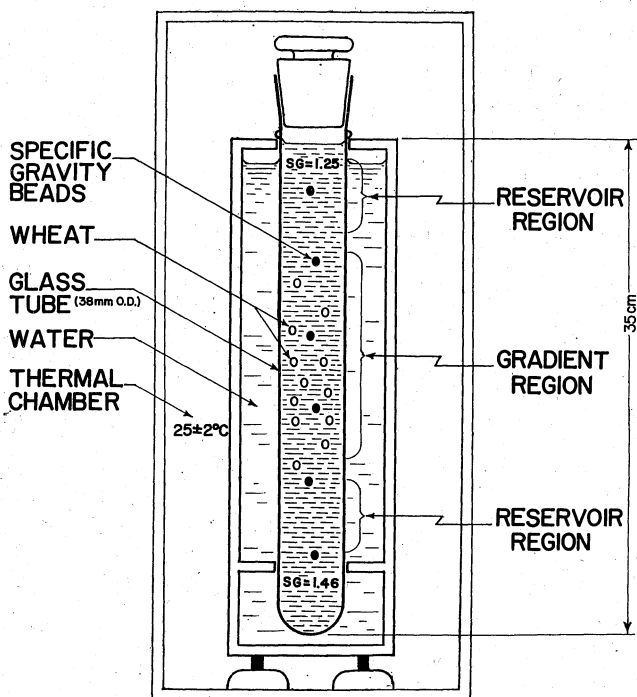


Fig. 1. The density gradient column is shown immersed in a water bath in a constant-temperature chamber. Mixtures of carbon tetrachloride and cyclohexane of different densities are placed in the bottom and top of the column, and establish a density gradient by diffusion. Glass beads of known density are used to calibrate the column.

contain solutions of different concentration, are sufficiently large, the column is useful for several weeks. In the columns used here the gradient gradually changed from approximately 0.1 to 0.005 g./cm.³ per cm. of height as the concentration of the reservoirs changed, but this did not interfere with practical operation of the column.

The occurrence of convection currents in the liquid is a serious threat to the stability of the gradient. To stabilize the temperature of the column, it was placed in a 10-liter water bath and the assembly was kept in a chamber whose temperature was thermostatically controlled at $25^{\circ} \pm 2^{\circ}\text{C}$. The column was supported by its neck in the bath. The chamber was kept isolated from drafts by screens, and was illuminated by a cool fluorescent lamp. To minimize vibrations, the chamber was mounted on small rubber shock absorbers, following the suggestion of Low and Richards (4).

The liquids used in the column were carbon tetrachloride (sp. gr. 1.585), and cyclohexane (sp. gr. 0.755). These liquids are nonsolvent for wheat and have surface tensions of about one-third that of water. The low surface tension helps prevent the formation of air bubbles around and in the crease of the kernel. A mixture of these two liquids giving a density of 1.25 g./cm.^3 was used for the lighter liquid and a mixture of density 1.46 g./cm.^3 , for the denser liquid.

To prepare the gradient column, the bottom half of the tube was filled with the denser solution. The lighter solution was very carefully added to the top half, minimizing turbulence at the interface of the liquids and preventing premature mixing. With care, a sharp separation of the two liquids was obtained. The two solutions were then partially mixed by a special stirrer consisting of a wire bent in the form of a helix of several turns which was lowered to the interface and moved up and down about 10 times with increasing amplitude. An experienced technician could avoid the necessity for stirring by careful pouring. After a day or so, diffusion eliminated any irregularities in the column and a smooth density gradient of about 0.01 g./cm.^3 per cm. of height was produced.

Glass beads², whose specific gravity was known to four decimal places, were dropped into the column along with the wheat kernels for each measurement. The plot of density against position of these beads was nearly linear. The vertical position of a kernel in the column was measured with a cathetometer and the corresponding density determined from the calibration curve. Since day-to-day changes cause small variations in the gradient, a new calibration curve was plotted each time the column was used. A small wire basket was used to remove the kernels from the column with little disturbance to the gradient after a series of measurements was concluded. In the present work, a column was allowed 24 hours to return to equilibrium, though measurements could have been made earlier.

²Available from Scientific Glass Apparatus Co., 100 Lakewood Terrace, Bloomfield, N.J.

Preparation of Wheat. The wheat samples measured in the tests are listed in Table I. The sample, Lee-2, was used only in the measurement of density variation with moisture content; its test weight, 1,000 kernel weight, and protein content were not determined.

TABLE I
LIST OF SAMPLES OF WHEAT AND PHYSICAL AND CHEMICAL PROPERTIES

WHEAT VARIETY	LOCATION OF TEST PLOT AND CROP YEAR	MOISTURE	TEST WEIGHT	1000-KERNEL WT.	PROTEIN ^a
		%	lb/bu	g	%
Hard spring					
Lee-1	North Dakota, 1960	11.5	60.6	34.7	15.2
Lee-2 ^b	Minot, N.D., 1956
Selkirk	North Dakota, 1960	11.3	57.8	35.3	14.3
Soft red					
Seneca-1	Wooster, Ohio, 1956	12.1	56.7	31.8	10.3
Seneca-2	Columbus, Ohio, 1960	11.5	62.2	40.6	10.00

^a14% moisture basis ($N \times 5.7$).

^bUsed only in moisture vs. density tests.

To obtain the desired moisture content, the wheat samples were placed in humidity chambers containing solutions of sulfuric acid and water as described by Katz. *et al.* (3). In a particular humidity chamber, the time required for the samples to reach moisture equilibrium was about 30 days.

According to Oxley (7), the precautions taken here to obtain moisture uniformity throughout each sample of wheat do not guarantee that each kernel has the same water content. However, his results show that the standard deviation in water content will not exceed 0.41% for the size of samples used here. This deviation is not large enough to affect our results significantly.

Results and Discussion

Density vs. Moisture Content. The density of the kernels from each wheat sample, measured at moisture contents from 7.5 to 18.5%, ranged from 1.29 to 1.38 g./cm.³ The relationship of moisture content to density for the wheat samples is shown in Fig. 2. Each plotted point represents an average of density measurements for 100 kernels. It is evident that wheat density has an inverse relation to the moisture content. To speak of density differences of wheat varieties without regard to the moisture content is meaningless. For example, if the density of Lee-1 was measured at high moisture content and that of Seneca-1 at a low moisture content, they would have the same density, though at 7.5% moisture they have a wide density separation.

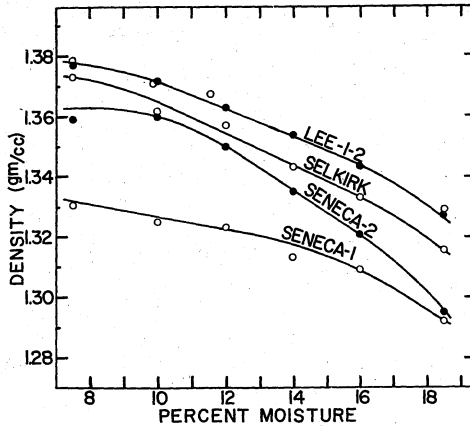


Fig. 2. Relation between moisture content and density for five samples of wheat. Each point on the curves represents the mean of density measurements of 100 kernels.

Density Spectrum at 12 Percent Moisture. The density spectrum of four wheat samples (Lee-1, Selkirk, Seneca-1 and -2) at 12% moisture is shown in Fig. 3. Each curve represents density measurements of 300 kernels.

The density spectrum of Lee-1 was highly peaked compared with that of Seneca-1. The two samples of Seneca showed different density spectra. Kernels of a single sample varied by as much as 0.1 g./cm.³,

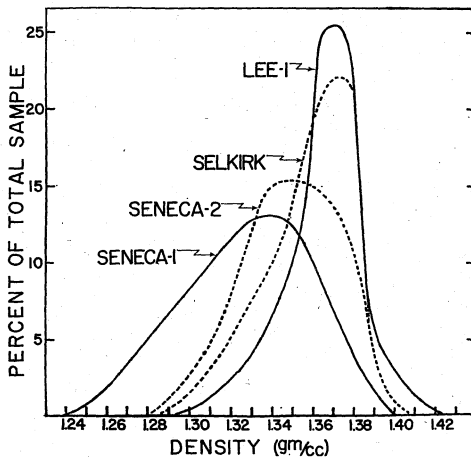


Fig. 3. Each curve represents the density spectrum for a 300-kernel sample of wheat. The curves were fitted to frequency histograms with cell widths of 0.01 g./cm.³ The cell boundaries are marked along the horizontal axis of the figure. The moisture content of all kernels was 12%.

at 12% moisture content.

Density spectrum curves also were constructed for other moisture contents. These density spectrum curves for wheat of moisture contents from 7.5 to 18.5% showed no significant variation in shape from those at 12% moisture, and are therefore not presented here.

Density vs. Protein and Ash. Each of the four wheat samples (Lee-1, Selkirk, Seneca-1 and -2) was divided into four subsamples according to their density at 12% moisture content. Protein and ash contents were determined on each subsample (Table II). In three of the four samples, protein content varied with density. Each of these three samples displayed an optimum subsample (or density interval) which gave a value of protein content that was higher than the average value for the sample before separation (Table I). Also, note that there is very little correlation between high protein content and high density or between high protein content and mean density among the subsamples tested here. The tests of Lee-1 showed that maximum protein content of 15.6% occurred at next to the lowest-density subsample, 1.33 to 1.36 g./cm.³, while maximum protein of about 11.3% in the two Seneca samples occurred in the highest-density subsample, 1.36 to 1.40 g./cm.³ In the Selkirk sample, all the subsamples showed a nearly uniform protein content, though the highest protein content of 14.6% occurred at the lowest-density subsample.

Milner, Farrell and Katz (6) reported a similar protein variation in tests run on the subsamples of wheat procured from the grain spectrometer. The grain spectrometer separates the wheat to a limited degree according to density.

Variations in ash percentage as a function of density were not so marked and regular as the variations in protein content, as shown in Table II. The subsamples of the Selkirk sample, in addition to showing a nearly uniform protein content, showed a uniform ash content. The maximum ash content for Seneca-2 occurred in the

TABLE II
CHEMICAL PROPERTIES OF FRACTIONS OF WHEAT^a

DENSITY INTERVAL	PERCENT PROTEIN (N X 5.7)				PERCENT ASH			
	Lee-1	Selkirk	Seneca-1	Seneca-2	Lee-1	Selkirk	Seneca-1	Seneca-2
<i>g/cm³</i>								
-1.30	...	14.6	9.8	9.4	1.80	1.81 ^b
1.30-1.33	14.6	14.3	10.5	9.2	1.73	1.89	1.78	1.79
1.33-1.36	15.6	14.4	10.7	10.1	1.81	1.89	1.81	1.84
1.36-1.40	15.4	14.3	11.5	11.0	1.81	1.89	1.78	1.88
1.40-	14.5	1.74

^a 14% moisture basis.

^b Only 1.3 g. used.

same density interval as did the maximum protein content for the sample.

Supplementary Data. Supplementary data on size distribution and yield, pearling, and average density for the wheat samples used here are shown in Table III. The method used for the sizing tests is out-

TABLE III
SUPPLEMENTARY DATA FOR WHEAT SAMPLES

SAMPLE	SIZING RESULTS AND YIELD ^a				PEARLING TEST	DENSITY ^b
	Over No. 7 Sieve	Over No. 9 Sieve	Over No. 12 Sieve	Flour Yield		
	%	%	%	%		
Lee-1	82.4	16.8	0.8	77.07	60.13	1.464
Selkirk	80.4	19.2	0.4	77.00	60.25	1.650
Seneca-1	84	16	..	77.2	35.15	1.441
Seneca-2	95.2	4.8	..	77.76	43.70	1.447

^a As outlined by Shuey (9).

^b On a moisture-free basis as done by Sharp (8).

lined by Shuey (9). The densities reported in Table III were determined on the conventional moisture-free basis and therefore do not agree with the densities reported above. In the pearling test, 20 g. of cleaned, unsized wheat was used in a barley pearler equipped with a No. 30 grit stone and a No. 20 wire sieve. These data are included to augment the description of the samples tested.

Density vs. Infestation. Other workers (see references in Cotton, 2) have utilized the change in density resulting from insect infestation to detect infested grain. In the present work the density of infested kernels was measured at various stages of the immature rice weevil. X-ray techniques were utilized to separate the infested kernels from the noninfested ones so that the density of the two could be compared. When the larvae were 20 days old, a nearly complete separation by density of infested from noninfested wheat could be obtained using a mixture of cyclohexane and carbon tetrachloride of density 1.26 g./cm.³ The insect cavities in the kernels at this immature stage were oval, being about 3.5 mm. in length and 1.5 mm. in breadth, and the diameters of the larvae were slightly less than 1.5 mm. The separation by density became more pronounced as the immature stage developed into the adult weevil.

Flotation separation of grain may be followed by visual inspection for exit holes (where X-ray inspection is not practicable), or by X-ray inspection of the lighter fraction in which the infestation will be concentrated; when used in connection with X-ray inspection a great saving in film and film-reading time will result.

Acknowledgments

We thank J. A. Shellenberger for providing samples for investigation and for helping us to plan this work. We are grateful to him and to A. B. Ward for their helpful comments and criticism of the completed work, and the test data of Table III.

Literature Cited

1. CAMPBELL, J. D., and JONES, C. R. The effect of temperature on the rate of penetration of moisture within damped wheat grains. *Cereal Chem.* **32**: 132-139 (1955).
2. COTTON, R. T. Pests of stored grain and grain products. Burgess Pub. Co., Minneapolis, Minn. (1956).
3. KATZ, R., COLLINS, N. D., and CARDWELL, A. B. Hardness and moisture content of wheat kernels. *Cereal Chem.* **38**: 364-368 (1961).
4. LOW, B. W., and RICHARDS, F. M. The use of the gradient tube for the determination of crystal densities. *J. Am. Chem. Soc.* **74**: 1660-1666 (1952).
5. LOWRIE, R. The density gradient column—Principles, techniques and applications. Bound Report, Job Order No. 840-91003-R. Metals Research Laboratories, Niagara Falls, N. Y. (April 7, 1959).
6. MILNER, M., FARRELL, E. P., and KATZ, R. The separation of grain by projection. II. Systematic differences in physical properties and composition of wheat fractions. *Cereal Chem.* **31**: 326-332 (1954).
7. OXLEY, T. A. Study of the water content of single kernels of wheat. *Cereal Chem.* **25**: 111-127 (1948).
8. SHARP, P. F. Wheat and flour studies. IX. Density of wheat as influenced by freezing, stage of development, and moisture content. *Cereal Chem.* **4**: 14-46 (1927).
9. SHUEY, W. C. A wheat sizing technique for predicting flour milling yield. *Cereal Science Today* **5**: 71-72, 75 (1960).

