# MICROHARDNESS OF WHEAT

by

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A THESIS

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TABLE OF CONTENTS

# INTRODUCTION

A number of attempts have been made to find a quantitative measure of the hardness of individual kernels or of the average hardness of a collection of kernels.

The Smetar hardness tester utilizes the penetration of a pyramid shaped stylus into a section of a wheat kernel as a measure of hardness (7). This tester, produced by Miag, is similar in concept to the Vickers hardness tester used in metallurgy, except that the stylus of the Smetar tester is larger. Because of the large stylus only one or two measurements can be made on a kernel section.

The laboratory-scale barley pearler is used to measure hardness of bulk samples of grain (4). With this apparatus the fraction of a small sample of wheat which is pearled off in a short time interval by an abrasive stone is used as an index of hardness.

Another index of hardness is the energy required to mill one gram of grain. The Brabender hardness tester, which consists essentially of a small burr mill fitted to the dynamometer coupling of the Farinograph, has been used by Paukner (6). This device measures the torque required to drive the burr mill as a small sample of grain is milled. The data are then reduced to terms of the energy required per milled gram of grain.

In the present work, the penetration of a small spring-loaded stylus into a kernel section is used as a measure of hardness (3). A commercial portable hardness tester called the Barcol Impressor, made by the Barber-Colman Co., has been adapted for work with grain by suitable choice of stylus and spring loading. A model of the wheat tester has been developed which is applicable to all wheat varieties under a wide range of ambient humidity. Approximately six independent hardness measurements may be made on a single transverse kernel section with this tester, which is called Model II (to distinguish it from an earlier tester which had been called Model I) (3).

## APPARATUS

The Barcol Impressor is a portable hardness tester designed for testing soft metals. The Impressor consists essentially of a spring-loaded stylus, a spindle and a dial micrometer. Hardness is measured by the distance the stylus is displaced into the spindle when the tester is pressed against a test object. The greater the displacement of the stylus, the harder the object. Readings of the micrometer are referred to as hardness numbers. The initial compression of the spring and the reading of the dial micrometer at its greatest deflection may be adjusted.

It was necessary to modify the Impressor for work with wheat. In order that the sections of wheat to be tested could be mounted and positioned the Impressor chasis and a two-way microscope stage were mounted on an oak platform. This arrangement is shown in Plate III of the appendix. This allowed kernel sections, cemented to microscope slides, to be set on the platform and located to 0.1 mm. by the vernier scales of the microscope stage.

In developing the wheat hardness tester a stylus of hardened steel drill rod was provided with a conical tip of $13^{\circ}$ included angle, terminated in a spherical surface of radius 0.11 in. The original spring of the Impressor was replaced with a music wire spring having free length

1.25 in., wire diameter 1/32 in., outer diameter 21/64 in., eleven free coils and plain ground ends. Because certain soft wheats crumbled under test conditions suitable for hard wheats it was necessary to use two different initial compressions of spring. For the hard wheat compression a force of 3.6 lb. was necessary to achieve maximum deflection of the stylus. At the soft wheat compression a force of 2.2 lb. was necessary. The dial was adjusted to read 100 for maximum deflection of the stylus.

To test the accuracy and reproducibility of the readings of the modified Barcol Impressor, a series of lead-antimony alloy blocks was prepared, with antimony content ranging from 0 to 6%, in steps of 1%. A series of nine hardness measurements on each block was made with the wheat tester and with the Vickers hardness tester. The results are shown in Plate I of the appendix, where each point is plotted as a cross giving the mean hardness and the standard deviation of each block. From the diagram it is apparent that the consistency of the wheat hardness tester is within 1/2 hardness number for a single specimen. Some variations may be due to nonuniformity of the specimen, as may be inferred from the range of DPH numbers for each point. Measurements of the hardness of wheat resulting in greater variation than 1/2 hardness number must be attributed to hardness differences in the wheat kernel itself.

To relate the two scales of the wheat hardness tester, a series of measurements of durum (Mindum) wheat kernels was made. Durum wheat had shown the greatest uniformity of all wheat tested in preliminary hardness measurements. Groups of 16 durum wheat sections were exposed to varying conditions of humidity. After the sections had come to equilibrium, one measurement was made on each cheek of each section with the soft wheat range, and one measurement on each cheek with the hard wheat range. The 32 hardness values thus obtained for each humidity were then averaged. The results of these measurements are shown in Plate II of the appendix. Measurements with the two ranges were also made on blocks of pure lead, indium solder, and polyethylene. Points obtained from these measurements are also indicated in Plate II. A photograph of the wheat hardness tester is shown in Plate III of the appendix.

## PROCEDURE

Sections of wheat kernels were prepared with a freezing microtome following the technique of Grosh and Milner (2). Kernels were placed on the microtome stage and embedded in warm aqueous gelatin solution. Freezing was quickly accomplished with intermittent blasts of carbon dioxide. The kernel was pared down to the desired position and removed from the stage by heating the stage with a soldering iron. The solid slab of gelatin containing the remaining part of the kernel was then inverted and refrozen to the stage. The section was then cut to a thickness of 1.0 mm. by cutting the slab to the thickness of a piece of balsa wood frozen to the stage. For good hardness readings, the kernels must have parallel faces and be free from cracks. Sections showing such defects on visual examination were culled from the test sample. Transverse sections were used to avoid difficulty with the crease of the kernel in subsequent hardness measurements.

Immediately after the kernel sections were prepared, they were cemented to glass microscope slides with Duco cement. After the cement had dried the kernel sections were exposed to appropriate experimental conditions (for example, in a humidity chamber). After the sections had reached equilibrium with the experimental environment, their hardness was tested.

To ascertain whether freezing and thawing of the wheat kernels associated with use of the freezing microtome had any effect on kernel hardness, forty kernel sections were prepared, twenty with the freezing microtome and twenty from the same lot of durum wheat by using wax to secure them to the microtome stage during sectioning. Both groups were kept at 81 percent relative humidity. After one week one section from each group was tested every day for twenty days. No difference in hardness of the two groups was detected. It was therefore concluded that freezing and thawing on the microtome stage did not affect hardness measurements significantly.

The hardness of kernel sections was measured after exposure to eleven different values of relative humidity, from 10 to 95 percent, at a laboratory temperature of $25^{\circ}$ C. These humidities were obtained within humidity chambers containing solutions of sulfuric acid in water, of a concentration appropriate to the desired humidity (5). To insure that sections had reached equilibrium with the chamber atmosphere, a twenty-gram sample of the variety being tested was placed in the humidity chamber, and weighed on successive days until constant weight was reached. The time to reach moisture equilibrium varied from 10 to 18 days, with the extremes of humidity requiring the longest time. When equilibrium had been reached, the twenty gram samples were analyzed for moisture content. The kernel sections were removed from the humidity chamber and examined microscopically for evidence of mold growth. The sections exposed to the 95 percent relative humidity atmosphere were the only ones showing evidence of mold growth. This growth was prevented in subsequent work by placing an open dish of toluene in the 95 percent humidity chamber.

In making a hardness test, the microscope slide was mounted on the microscope stage of the hardness tester. Readings from the vernier scales on the microscope stage allowed the position of the measurement to be plotted on an enlarged diagram of the kernel to within $\pm$ 0.05 mm. The actual hardness measurement was made by pressing down on the framework of the tester with the hand until the flat part of the spindle was in contact with the surface of the section. The dial reading reached a maximum and remained there as long as the force was applied. This reading was recorded as the hardness number of a particular point on the wheat kernel section. The results were not altered by reasonable variations in hand pressure. All kernels sufficiently hard for use of the high range of the tester were measured at five different points on the kernel section; at high humidities, and for soft wheat, where the low range of the tester was used, three or four measurements were made on a kernel section. In all cases the average of the readings for a kernel was taken as representative of the kernel section. For each batch of grain the hardness of nine kernel sections was measured at each humidity. For convenience in presenting the data, hardness numbers obtained on the low range of the tester with hard wheats at high humidity were converted to high range values by use of Plate II.

Plate IV of the appendix shows a photograph of a durum section after hardness measurements were made.

## EXPERIMENTAL RESULTS

Tables 1, 2, 3 and 4 in the appendix show the hardness and/or moisture content data for varieties of hard red winter, durum and soft white winter wheat at equilibrium with various controlled humidity conditions.

The hard red winter wheat varieties were Ponca, Kiowa and Wichita. These samples were harvested at test plots in Manhattan, Hays, and Belleville, Kansas in 1957. The durum varieties, Mindum, Vernon and Langdon, were harvested at Minot, North Dakota in 1956. The soft white winter wheat varieties, Brevor and Elmar, were harvested at Manhattan, Kansas in 1956.

The available samples of hard red winter wheat varieties were too small to allow determinations of the moisture content of each variety at each humidity. Mixtures of hard red winter wheat varieties, grown at the selected test plots, were exposed to the appropriate experimental conditions and their moisture content analyzed. These data are shown in Table 2.

Data listed under hardness in the tables are given as the mean hardness and standard deviation of each variety at each humidity. Hardness measurements on the hard wheats at high humidities were made with the low range of the tester. Hardness values thus obtained were converted to high range values by use of Plate II. These converted values are preceded by an asterik in the Tables. The low range of the tester was used for all measurements on soft wheats. These measurements appear as low range values in the soft wheat table.

Plate V of the appendix shows the hardness of Vernon and Langdon (durum) kernels as a function of relative humidity. Curve A concerns Vernon and curve B concerns Langdon. Hardness values of Ponca wheat grown at Manhattan, Kansas are plotted on this graph to show that hard red winter wheat is, in general, as hard as durum. Plate VI of the appendix shows the hardness of the soft white varieties, Elmar and Brevor, as a function of relative humidity.

Data listed under relative humidity in the tables are referred to the wet weight of the samples. The relationship between ambient humidity and moisture content at equilibrium at 25° C., for samples of Elmar (soft white), Mindum (durum) and hard red winter wheat, is shown in Plate VII of the appendix. This relationship is typical of all wheat tested.

## DISCUSSION OF RESULTS

Hardness of the hard wheat varieties diminished regularly with increasing humidity (or increasing moisture content). The decrease in hardness was gradual up to a humidity of 80 percent, approximately 15 percent moisture, above which there was a rapid decrease. Soft winter wheats showed no significant change in hardness up to a humidity of 70 percent. Above this humidity their hardness showed a rapid decrease. In all cases the kernel to kernel variation in hardness was much greater at high moisture content than at low moisture content. Durum kernels were the most uniform; soft winter kernels were the least uniform.

While all varieties of durum wheat were more uniform in hardness than other types of wheat, Mindum and Vernon kernels were significantly harder than Langdon kernels in the range of 40 to 80 percent humidity. Mindum and Vernon kernels were of essentially the same hardness at corresponding humidities.

Similar varietal differences were displayed by the soft white winter varieties, Elmar and Brevor. Brevor was consistently softer than Elmar at corresponding humidities, the difference being greatest at higher moisture content.

No differences in hardness due to variety or test plot location were found in the hard red varieties Ponca or Kiowa grown in Belleville, Hays, and Manhattan, Kansas, in 1957. At higher moisture contents some differences were found in the hardness of Wichita wheat, with samples raised at Manhattan being somewhat softer than samples raised at Hays or Belleville.

The hardness of hard red wheat was nearly equal to durum wheat at relative humidities below 70 percent, but at higher humidities durum wheat was generally harder. The hardness of soft white winter wheat was considerably less than that of either durum or hard red winter wheat. This was true under all humidity conditions.

There was no significant differences, due to type or variety, in the relationship between ambient humidity and moisture content. This is in agreement with Coleman and Fellows (1).

## CONCLUSIONS

The development of the new wheat hardness tester has provided a reliable and consistent means for measuring the hardness of wheat kernel sections. Following the described testing technique measurements may be made on any type of wheat over a wide range of moisture content. The testing proceeds rapidly, approximately 30 sections may be tested in one hour, but some time is necessary for the specimen to reach equilibrium with the ambient humidity condition.

It can be concluded that significant differences in hardness exist between wheat varieties of the same wheat type, as in the case of durum and soft white winter varieties. The durum varieties, Mindum and Vernon, are significantly harder than Langdon in the range of humidities from 40 to 80 percent. It is not clearly evident that differences in agronomic conditions cause differences in hardness. The hardness of Wichita ( hard red ), grown at Manhattan, Kansas, varied differently with humidity condition than that grown at Hays, or Belleville, Kansas. However, Kiowa and Ponca (hard red) showed no such differences.

# ACKNOWLEDGMENTS

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# APPENDIX

Table 1. Hardness of hard red winter wheat varieties at various relative humidities.

| Variety | Percent Relative Humidity | High Range Hardness: Sample Grown at Manhattan | Sample Grown at Hays | Sample Grown at Belleville |
|---|---|---|---|---|
| Ponca | 11 | 92.1 ± 1.5 | 93.0 ± 2.2 | 92.8 ± 1.8 |
| | 21 | 91.3 ± 1.4 | 89.9 ± 2.3 | 89.9 ± 2.4 |
| | 31 | 90.8 ± 3.0 | 91.0 ± 2.3 | 91.5 ± 3.1 |
| | 40 | 92.1 ± 2.7 | 93.4 ± 3.2 | 95.2 ± 1.6 |
| | 50 | 93.6 ± 2.1 | 93.6 ± 3.0 | 92.0 ± 2.9 |
| | 60 | 87.0 ± 2.8 | 89.2 ± 3.1 | 89.5 ± 2.0 |
| | 69 | 89.1 ± 1.8 | 87.3 ± 2.1 | 87.5 ± 3.7 |
| | 79 | 79.7 ± 3.8 | 83.7 ± 3.0 | 85.9 ± 2.1 |
| | 85 | *80.5 ± 4.5 | *72.5 ± 6.2 | *68.1 ± 7.0 |
| | 90 | *57.7 ± 3.7 | *63.3 ± 5.5 | *71.5 ± 2.0 |
| | 95 | *67.0 ± 5.0 | *76.8 ± 3.3 | *72.0 ± 4.3 |
| Kiowa | 11 | 91.5 ± 3.0 | 92.9 ± 1.6 | 92.7 ± 1.8 |
| | 21 | 90.3 ± 1.9 | 90.6 ± 1.7 | 91.5 ± 1.8 |
| | 29 | 89.6 ± 1.3 | 93.1 ± 1.7 | 92.1 ± 1.0 |
| | 40 | 90.9 ± 2.7 | 93.1 ± 1.0 | 92.6 ± 1.8 |
| | 50 | 90.1 ± 2.4 | 88.1 ± 2.1 | 88.2 ± 1.9 |
| | 60 | 90.5 ± 2.1 | 90.9 ± 1.6 | 91.5 ± 1.6 |
| | 70 | *86.5 ± 3.5 | *82.5 ± 3.8 | *83.2 ± 4.0 |
| | 80 | *79.5 ± 2.8 | *80.5 ± 2.5 | *78.8 ± 3.2 |
| | 85 | *76.8 ± 5.3 | *81.3 ± 3.8 | *82.7 ± 5.3 |
| | 90 | *53.5 ± 5.0 | *69.5 ± 4.1 | *73.0 ± 5.7 |
| | 95 | *66.0 ± 4.0 | *44.5 ± 8.8 | *65.0 ± 12.8 |
| Wichita | 9 | 90.4 ± 4.6 | 90.3 ± 3.2 | 88.9 ± 1.9 |
| | 21 | 91.4 ± 2.4 | 92.5 ± 4.0 | 90.2 ± 3.6 |
| | 31 | 88.1 ± 3.8 | 90.8 ± 2.7 | 89.0 ± 4.4 |
| | 40 | 88.3 ± 4.4 | 90.2 ± 2.6 | 89.0 ± 3.1 |
| | 50 | 83.9 ± 4.3 | 87.3 ± 3.0 | 86.8 ± 2.6 |
| | 59 | *80.9 ± 4.5 | *90.0 ± 3.8 | *83.7 ± 4.2 |
| | 69 | *78.5 ± 4.2 | *86.9 ± 3.2 | *85.3 ± 2.0 |
| | 80 | *76.0 ± 4.2 | *83.3 ± 4.5 | *83.3 ± 3.0 |
| | 85 | *76.0 ± 6.5 | *78.9 ± 5.0 | *67.5 ± 7.5 |
| | 90 | *38.5 ± 10.8 | *60.3 ± 9.5 | *61.7 ± 14.5 |
| | 95 | (59.1 ± 8.3)** | *59.5 ± 9.5 | *62.5 ± 7.5 |

**This is a low range hardness number which was too low to convert.

Table 2. Moisture content of a mixture of hard red winter wheat varieties at various relative humidities.

| | | | | | | |
|---|---|---|---|---|---|---|
| Percent Humidity : | 12 | 21 | 29 | 40 | 50 | 60 |
| Percent Moisture Content: | 5.78 | 7.33 | 8.65 | 9.47 | 10.23 | 10.88 |
| Percent Humidity : | 69 | 80 | 85 | 90 | 95 | |
| Percent Moisture Content: | 12.27 | 14.40 | 16.26 | 18.05 | 21.70 | |

Table 3. Hardness of durum wheat varieties at various relative humidities.

| | Mindum | | Vernon | | Langdon | |
|---|---|---|---|---|---|---|
| Percent Relative Humidity | Percent Moisture Content | High Range Hardness | Percent Moisture Content | High Range Hardness | Percent Moisture Content | High Range Hardness |
| 12 | 6.72 | 93.3 ± 0.6 | 6.74 | 93.2 ± 1.0 | 5.64 | 92.7 ± 1.8 |
| 22 | 7.33 | 92.5 ± 0.7 | 7.47 | 92.8 ± 0.4 | 7.41 | 93.8 ± 1.8 |
| 31 | 8.52 | 93.1 ± 1.1 | 8.56 | 92.4 ± 0.7 | 8.43 | 91.8 ± 0.9 |
| 42 | 10.18 | 91.6 ± 2.0 | 10.19 | 92.9 ± 0.9 | 9.14 | 89.6 ± 1.7 |
| 50 | 10.66 | 91.1 ± 1.4 | 10.99 | 91.0 ± 1.1 | 10.58 | 86.9 ± 2.3 |
| 59 | 11.17 | 90.0 ± 1.6 | 11.94 | 90.8 ± 0.8 | 11.42 | 87.6 ± 1.7 |
| 70 | 12.76 | 89.2 ± 0.9 | 13.38 | 88.7 ± 1.4 | 13.00 | 85.3 ± 2.6 |
| 80 | 14.70 | *86.0 ± 1.2 | 15.15 | *86.8 ± 2.0 | 14.86 | *83.0 ± 3.5 |
| 86 | 16.27 | *81.0 ± 1.2 | 16.51 | *83.3 ± 1.5 | 16.13 | *80.0 ± 3.0 |
| 90 | 17.64 | *76.0 ± 2.7 | 17.97 | *71.2 ± 5.3 | 17.36 | *71.5 ± 3.5 |
| 95 | 20.83 | *75.5 ± 2.0 | 21.20 | *66.1 ± 5.0 | 20.37 | *70.5 ± 4.5 |

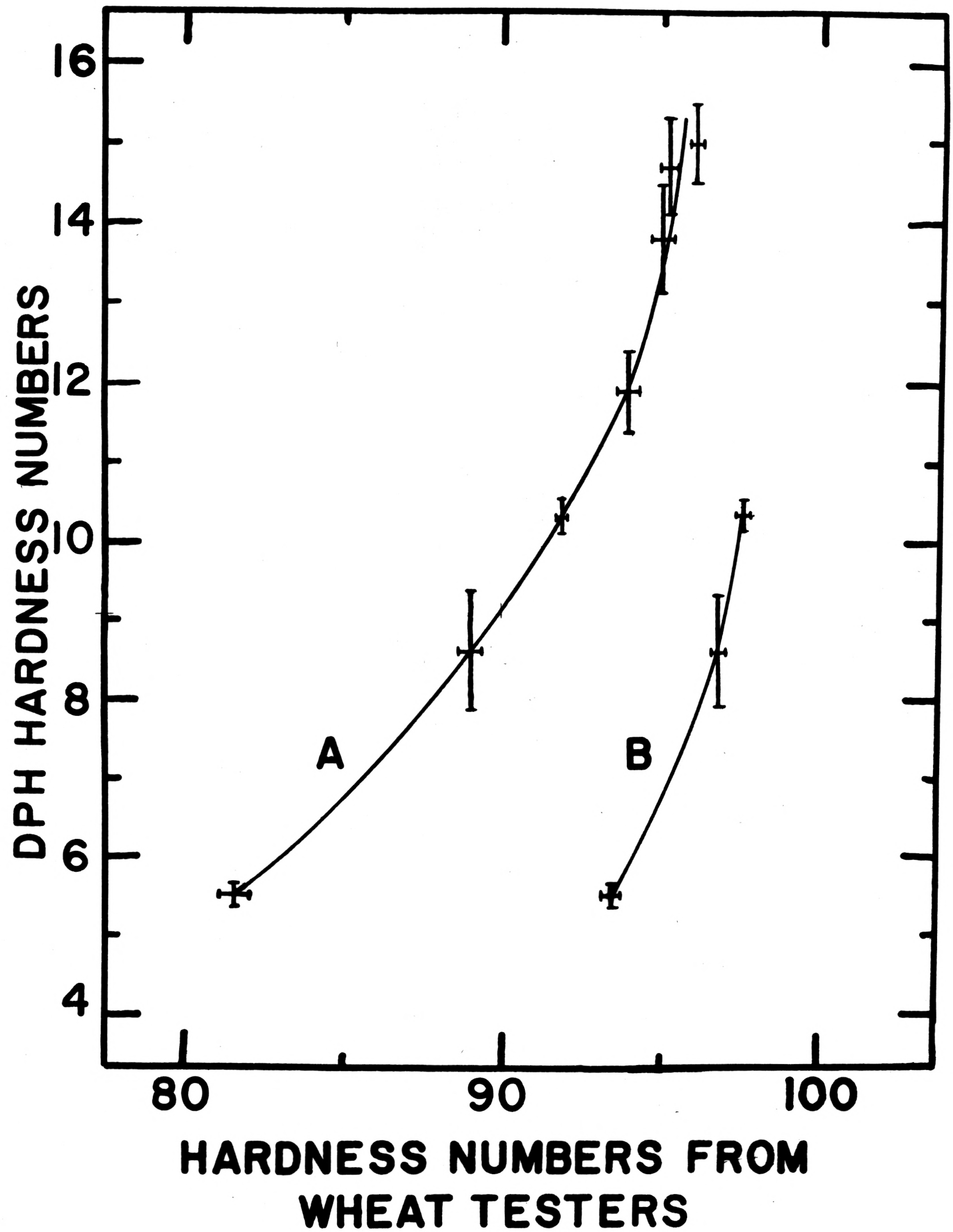
Table 4. Hardness of soft white winter varieties at various relative humidities.

| | Brevor | | Elmar | |
|---|---|---|---|---|
| Percent Relative Humidity | Percent Moisture Content | Low Range Hardness | Percent Moisture Content | Low Range Hardness |
| 12 | 5.48 | 86.0 ± 2.8 | 5.36 | 88.0 ± 3.1 |
| 22 | 7.93 | 87.7 ± 4.8 | 7.81 | 88.6 ± 3.2 |
| 30 | 8.94 | 87.6 ± 4.4 | 8.85 | 90.4 ± 1.5 |
| 41 | 9.19 | 85.8 ± 3.9 | 9.38 | 87.7 ± 3.0 |
| 50 | 10.22 | 86.7 ± 3.9 | 10.64 | 89.8 ± 1.6 |
| 59 | 10.97 | 86.7 ± 2.7 | 11.26 | 89.5 ± 3.6 |
| 70 | 13.03 | 86.2 ± 3.8 | 13.21 | 89.8 ± 1.6 |
| 80 | 15.01 | 82.2 ± 5.7 | 14.87 | 85.8 ± 2.6 |
| 85 | 16.42 | 68.9 ± 5.2 | 16.20 | 73.3 ± 4.3 |
| 90 | 18.03 | 53.0 ± 9.7 | 18.59 | 76.1 ± 6.2 |
| 95 | 20.61 | 43.7 ± 6.8 | 20.45 | 63.6 ± 11.1 |

## EXPLANATION OF PLATE I

Diamond Pyramid Hardness, as found with a Vickers hardness tester on samples of lead-antimony alloy, vs. hardness values from the two ranges of the wheat hardness tester. The samples ranged from 0 to 6 percent antimony. Curve A concerns the hard wheat range of the tester and curve B concerns the soft wheat range.

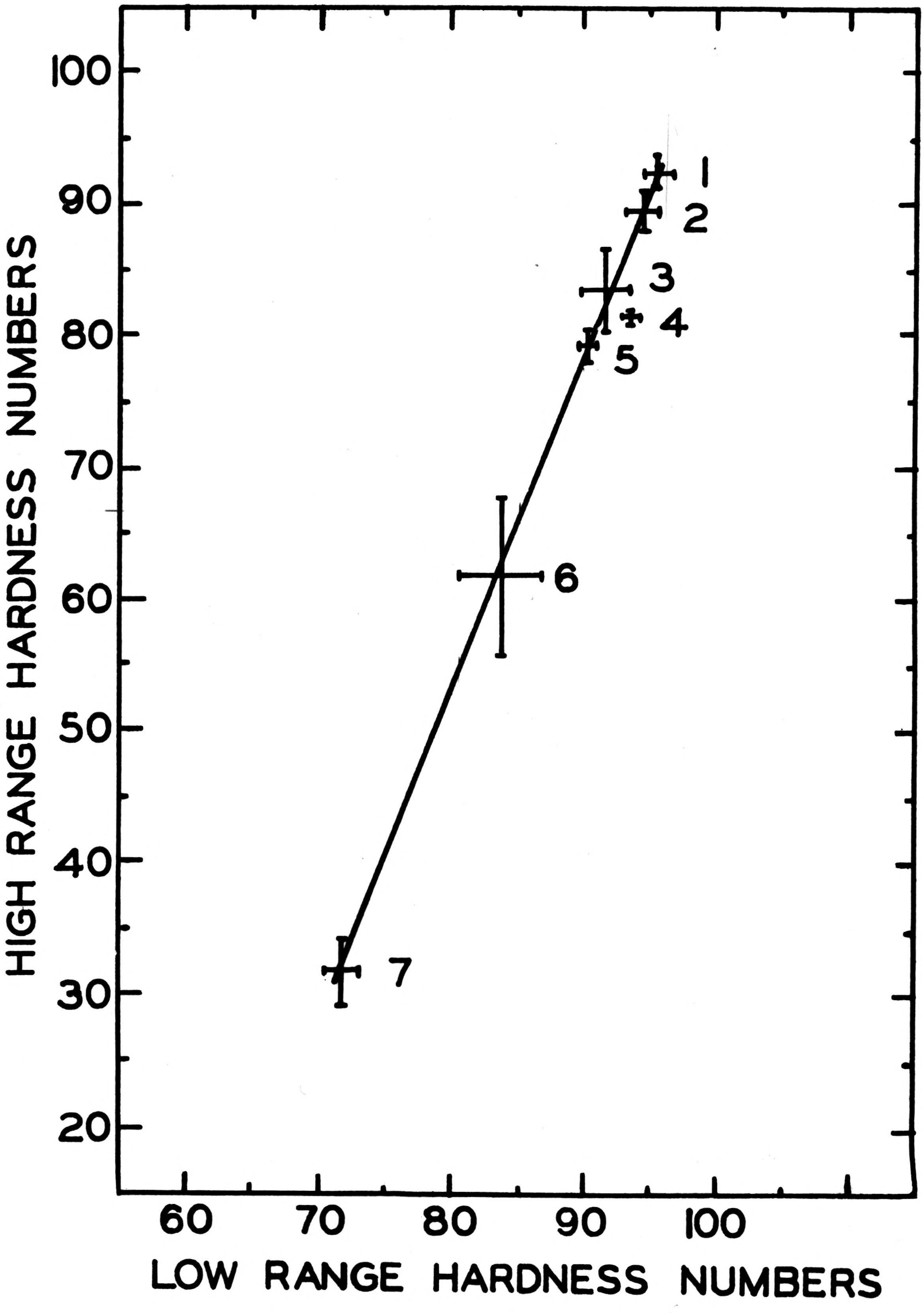
PLATE I


16
14
12
10
8
6
4
DPH HARDNESS NUMBERS
A
B
80
90
100
HARDNESS NUMBERS FROM
WHEAT TESTERS

EXPLANATION OF PLATE II

Graph relating hardness values from the hard wheat (high) range of the hardness tester to values from the soft wheat (low) range. The samples used were: 1, durum sections exposed until an equilibrium condition was reached at 33 percent relative humidity (r. h.); 2, durum sections from wheat at 75 percent r. h.; 3, durum sections from wheat at 81 percent r. h.; 4, pure lead; 5, indium solder; 6, durum sections from a 93 percent r. h.; 7, polyethylene.

PLATE II

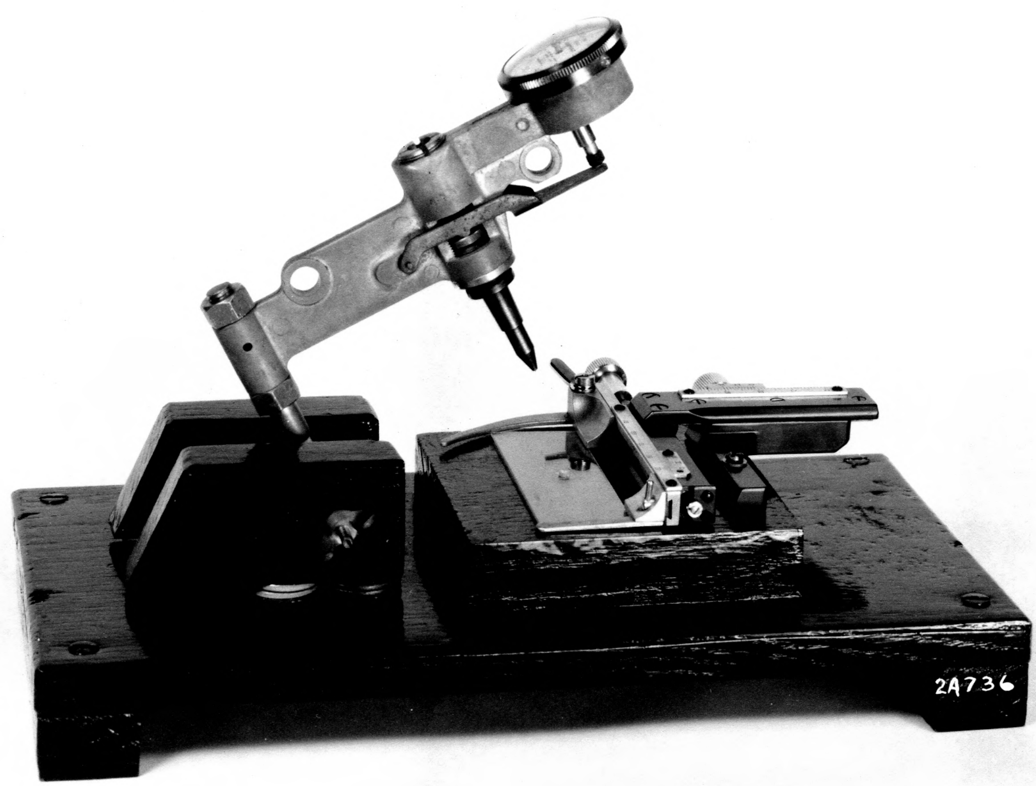


HIGH RANGE HARDNESS NUMBERS
100
90
80
70
60
50
40
30
20
1
2
3
4
5
6
7
60
70
80
90
100
LOW RANGE HARDNESS NUMBERS

## EXPLANATION OF PLATE III

The converted Barcol Impressor.

PLATE III



## EXPLANATION OF PLATE IV

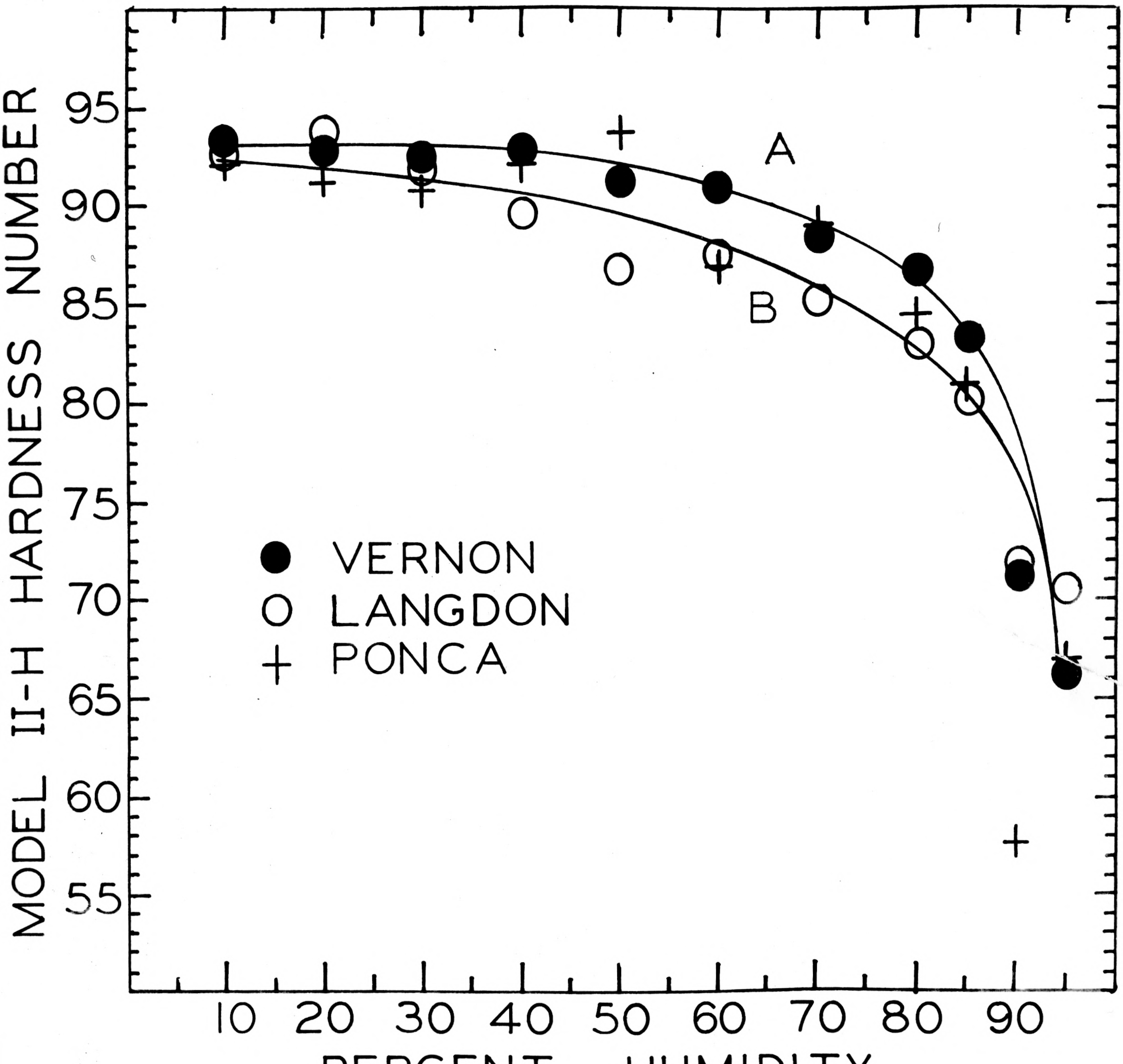
Photo of a durum wheat section after testing.

PLATE IV

## EXPLANATION OF PLATE V

Hardness of Vernon and Langdon (durum) and Ponca (hard red) as a function of relative humidity. Model II-H hardness refers to values from the hard wheat range of the tester.

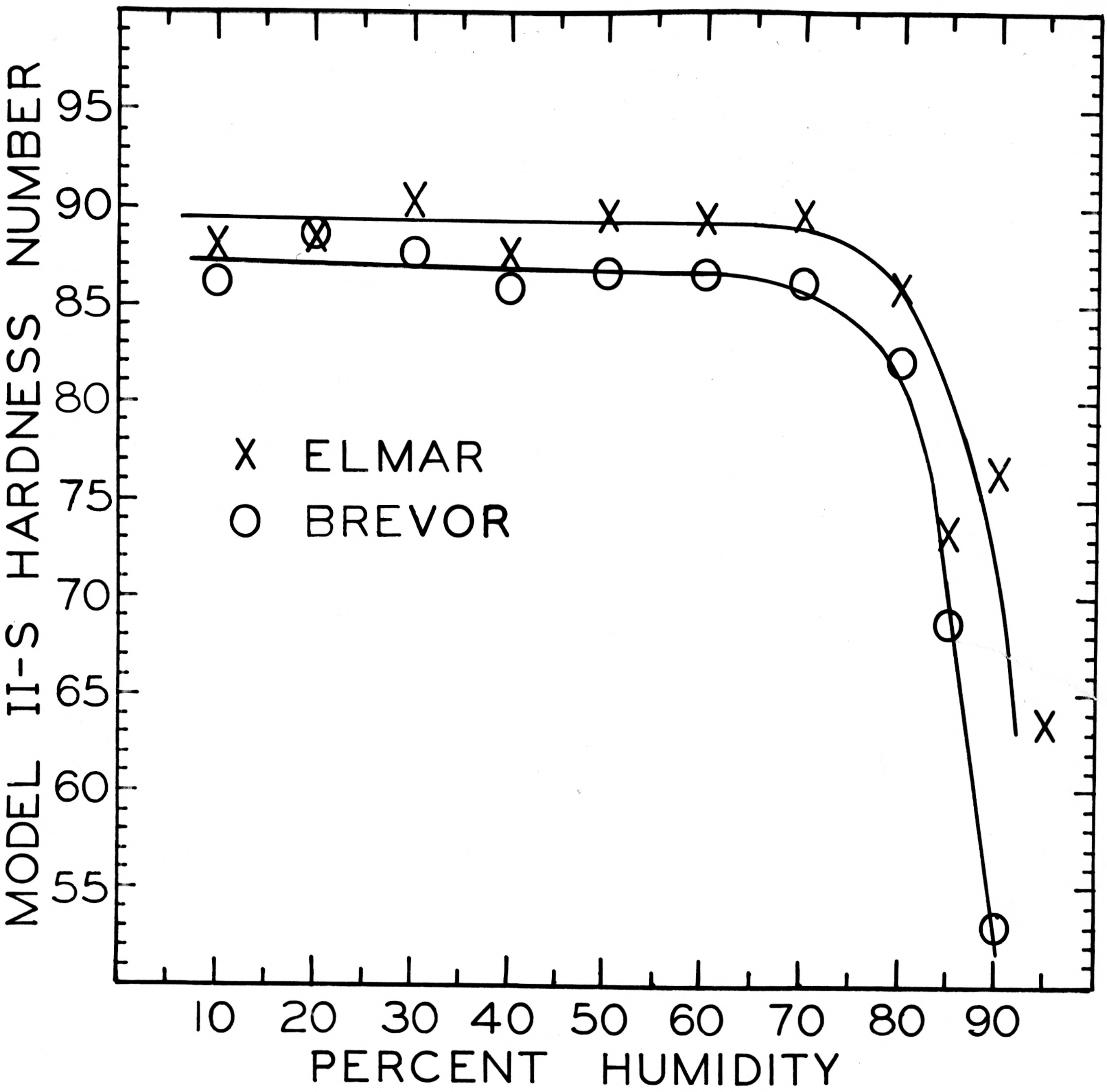
PLATE V


MODEL II-H HARDNESS NUMBER
95
90
85
80
75
70
65
60
55
10 20 30 40 50 60 70 80 90
PERCENT HUMIDITY
A
B
VERNON
LANGDON
PONCA

## EXPLANATION OF PLATE VI

Hardness of Elmar and Brevor (soft white) as a function of relative humidity. Model II-S hardness refers to values from the soft wheat range of the tester.

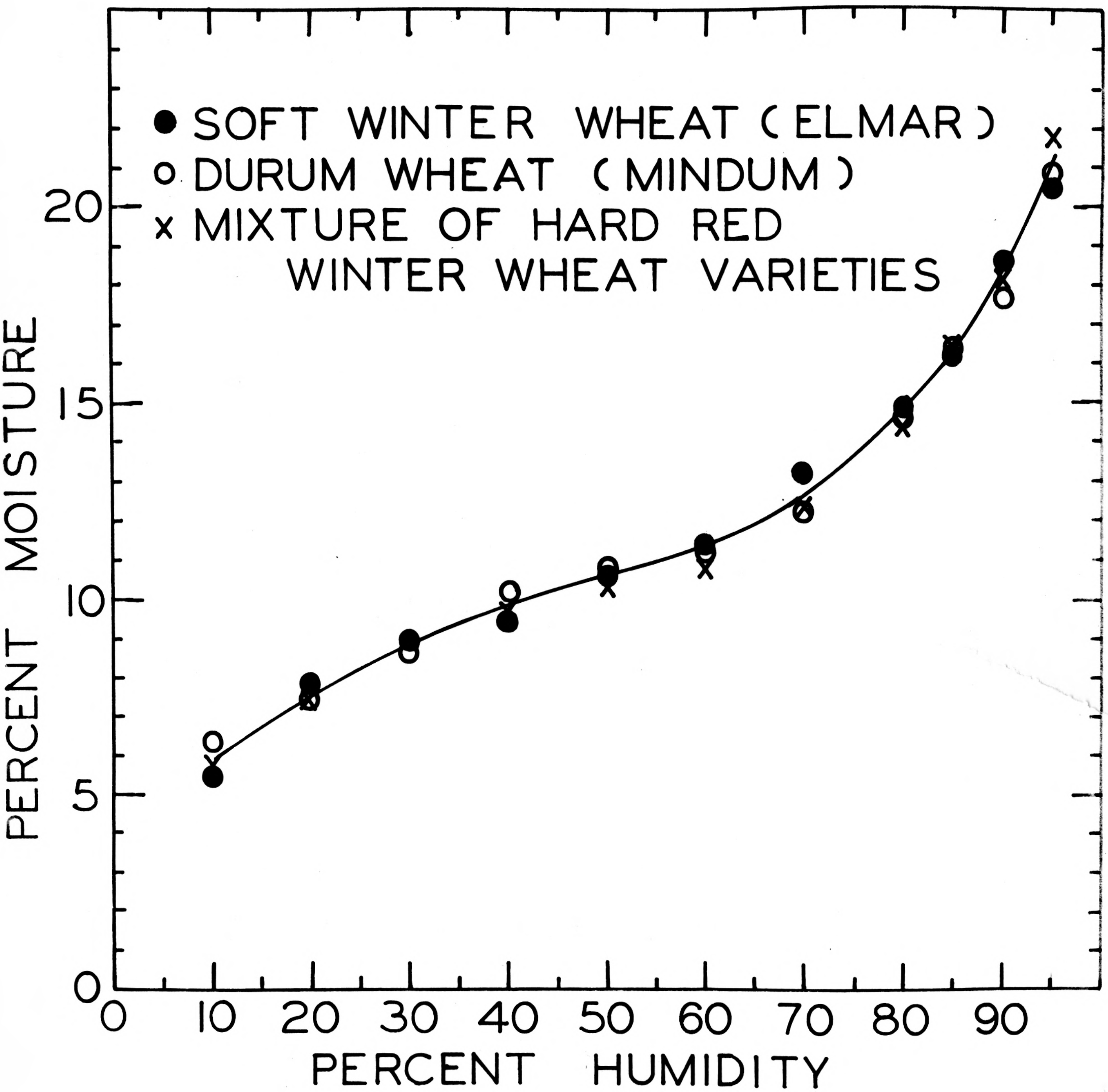
PLATE VI


MODEL II-S HARDNESS NUMBER
95
90
85
80
75
70
65
60
55
X ELMAR
O BREVOR
10 20 30 40 50 60 70 80 90
PERCENT HUMIDITY

## EXPLANATION OF PLATE VII

Moisture content of wheat kernels in equilibrium with humid air at 25°C.

PLATE VII


● SOFT WINTER WHEAT (ELMAR)
○ DURUM WHEAT (MINDUM)
x MIXTURE OF HARD RED WINTER WHEAT VARIETIES
PERCENT MOISTURE
0
5
10
15
20
0
10
20
30
40
50
60
70
80
90
PERCENT HUMIDITY

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AN ABSTRACT OF A THESIS

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To make a hardness measurement, a specimen was placed in position on the platform of the hardness tester. The frame work of the tester was pushed down with the hand until the flat part of the spindle was in contact with the surface of the specimen. The micrometer dial reading reached a maximum and remained there as long as the force was applied. This reading was considered the hardness of that particular point on the specimen. Tests on selected specimen with the wheat tester and the Vickers tester (a standard metallurgical hardness tester) showed the former to be both consistent and reliable.

The hardness and moisture content of hard red winter, soft white winter and durum wheat was measured after exposure to eleven different values of relative humidity, from 10 to 25 percent, at a laboratory temperature of 25$^{\circ}$ C. Hardness of the hard wheat varieties diminished regularly with increasing ambient humidity (or increasing moisture content). The decrease in hardness was gradual up to a humidity of 80 percent (approximately 15 percent moisture) above which there was a rapid decrease. Soft white winter wheats showed no significant change in hardness up to an ambient humidity of 70 percent. Above this humidity their hardness showed a rapid decrease.

Durum kernels were the most uniform in hardness; soft white winter kernels were the least uniform. Durum and hard red winter varieties were nearly equal in hardness at relative humidities below 70 percent, but at higher humidities durum wheats were generally harder. The hardness of soft white winter wheats was considerably less than that of the hard wheats at all ambient humidity conditions. Varietal differences were displayed by durum and soft white winter wheats. The durum varieties Mindum and Vernon were significantly harder than Langdon kernels in the range of 40 to 80 percent humidity. The soft white winter variety, Elmar, was consistently harder than Brevor at all humidities tested.

All wheat tested, regardless of type or variety, showed the same relationship between ambient humidity and moisture content.