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THE STARCHES  
OF THE  
GRAIN SORGHUMS  
A TECHNICAL BULLETIN

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DEPARTMENT OF CHEMISTRY

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## THE STARCHES OF THE GRAIN SORGHUMS

BY C. K. FRANCIS AND O. C. SMITH\*

### INTRODUCTION

The experiments described in this bulletin were planned as part of an Adams Project, for the purpose of determining the specific nature of the starches obtainable from the different grain sorghums. It is important that the characteristics of a starch be known in order to determine its adaptability for use in the various trades.

A previous bulletin\*\* of this Station described kafir and discussed its value as a food when compared with corn, and reported the determinations of the physical and chemical constants of the fat which had been extracted from the grains of kafir. It is known that there are many uses for corn, and that the corn products industry is growing more valuable and of more importance to the human family each year. The chemical composition of feterita, kafir and milo closely resembles that of corn, and, it is both possible and reasonable that these crops should, to a great extent, replace corn in sections where annual crops of corn are not produced. It is the purpose of this bulletin to report in detail the work that has been done on the one constituent, starch, of several of the sorghums.

It is a well recognized fact that a specific genus, and very often a variety, will yield a starch having well defined characteristics, and certain properties are so prominent in some that it is a very simple matter to identify them. Wheat, bean and potato starches may be quickly identified by means of a microscope, because of the peculiar shape or markings. However, it is no easy matter to differentiate the wheat starches, because the general properties of the plant and the composition of the grain appear to be but little altered during the process of developing new types such as are found in the different wheats.

The botanical characteristics of the grain sorghums are, superficially at least, the same. The young plants resemble common corn, but the mature plants are quite unlike it. The fruiting members and seeds are but indefinitely similar to ordinary corn. While it appears that the grain sorghums are apparently closely related to corn, there has been but little chemical work done to show the differences existing in the compounds produced by each variety.

In arranging the data of this bulletin an effort has been made to place the tests on the starches of the grain sorghums on a comparative basis with corn.

\*Acknowledgement is made of the assistance which was given, when this work was started, by Mr. A. A. Jones, formerly of this department.

\*\*Oklahoma Agricultural Experiment Station Bulletin No. 89.

## OUTLINE OF THE METHOD USED FOR MANUFACTURING STARCH

In the manufacture of starch the grain is steeped in tanks, in the presence of sulfurous acid, for the purpose of softening it, and is then passed to the grinding mills where it is crushed and the starch material set free. The thick, semi-liquid pulp is run onto a series of shakers, which remove the outer coatings of the grain. The liquid flows from the shakers through a tank fitted with a series of paddles which revolve and keep the liquid in motion, but do not agitate it. Water is flowing into this tank, and as the gluten comes to the surface it is carried away by means of a tailing pipe set in one end of the tank.

The starch liquor, freed from most of the gluten, is then run into tanks with a great excess of water. The depth of the tank is greater at one end than at the other, the fall being about one inch in each twenty feet, the liquid entering at the shallow end where any heavy foreign substance is deposited. The starch covers about three-fourths of the length of the tank, while the gluten, which was not removed by the previous treatment, settles at the lower end. After the water is drawn from the tank the starch is shoveled into drying cars which are run into drying rooms where the starch is dried for about twelve hours.

When the starch comes from the first drying it is slightly moist, not wet, similar to moist soil. Next it is ground and pressed into iron tanks by means of hydraulic pressure and then it is heated for the final drying. From these tanks the starch is obtained in the form of large cakes which are broken into lumps for ordinary laundry starch.

When a table starch is to be made the process is the same, but more care is observed to see that everything is clean and all acid is neutralized with marble dust or some alkaline substance.

### SPECIFIC GRAVITY OF THE GRAIN

The quantity of grain to the plant, ear or head is of very material importance. A knowledge of the weight or specific gravity of any grain is of particular value when considering it as a possible source of raw material for the manufacture of starch. A number of tests have been made in this laboratory for the purpose of determining these facts in so far as they concern the grain sorghums, but it has been possible to obtain good specimens of only a small number of the varieties grown in Oklahoma, so the results shown in Table I may be considered but approximately correct:

TABLE I  
Specific Gravity and Percent of Grain on the Head

SAMPLE	NUMBER HEADS EXAMINED	SPECIFIC GRAVITY	WEIGHT IN GRAMS OF THE HEAD COMPLETE			AVERAGE PERCENT GRAIN ON THE HEAD
			MAXIMUM	MINIMUM	MEAN	
White kafir .....	10	1.3151	250.00	180.00	207.00	67.50
Pink kafir .....	1	1.3305	.....	.....	110.00	77.27
Red kafir .....	1	1.3070	.....	.....	98.00	77.55
White milo .....	5	1.2937	180.00	135.00	153.00	77.14
Yellow milo .....	1	1.2891	.....	.....	120.00	80.83
Feterita .....	7	1.2825	125.00	58.00	78.00	84.69
White kaoliang .....	3	1.3266	43.00	33.00	38.00	78.10
Brown kaoliang .....	1	1.2950	.....	.....	88.00	82.95

**EXTRACTION OF STARCH**

The starch was extracted from the grains by the process briefly described below. The grains were steeped in water for several days until slight putrefaction was noticed, then crushed in a mortar. The material was next washed with water on a brass 100-mesh sieve until the washings became clear. After standing about thirty minutes the supernatant liquid was decanted and the residue washed twice by decantation. The impure starch was suspended in water and stirred continuously while enough 5% sodium hydroxide solution was added to produce a yellow color. This was set aside for six hours, the liquid decanted and the starch washed four times with water, once with 95% alcohol and finally with ether. Prepared in this way the starch dried well when placed over sulfuric acid in a desiccator.

**DETERMINATION OF STARCH\***

**Reagents**—Copper Sulphate Solution.—Dissolve 34.639 grams copper sulphate in water and dilute to 500 cc.

Alkaline Tartrate Solution.—Dissolve 173 grams of Rochelle salts and 125 grams of potassium hydroxide in water and dilute to 500 cc.

Hydrochloric Acid Solution.—Prepare a solution of HCl having a specific gravity 1.125 at 15° C.

**Removal of the Sugars.**—Stir 2.5 grams of the ground seed in 50 cc. of water for one hour, then wash onto a filter with 250 cc. of cold water.

**Inversion of the Starch.**—Transfer the residue to an 800 cc. lip-less beaker, add 200 cc. of water and 20 cc. of HCl sp. gr. 1.125; boil 2.5 hours. During the boiling the beakers are covered with round-bottom flasks filled with cold water to serve as condensers, which should be changed every thirty minutes.

Cool the liquid and nearly neutralize with NaOH solution. Dilute to 250 cc. and filter.

**Determination.**—Place 30 cc. of the copper sulphate solution, 30 cc. of the alkaline tartrate solution and 60 cc. of water in a beaker and heat to boiling. Add 10 cc. of the starch solution, boil for exactly two minutes, filter at once onto a fairly thick pad of asbestos in a Gooch crucible. Wash with water and alcohol, dry and weigh. Calculate the weight of dextrose from the weight of cuprous oxide obtained and multiply the result by 0.90, which will give the weight of starch.

TABLE II  
Quantity of Starch in the Grain Sorghums

SAMPLE	STARCH PERCENT
Kafir .....	63.6
White milo .....	64.8
Yellow milo .....	63.1
Feterita .....	63.8
Darso .....	63.7

The quantity of starch found in the different grain sorghums is fairly uniform, as indicated by the several determinations reported in Table II.

\*Official Methods of Analysis. U. S. Bu. Chem. Bul. 107 (Rev.), p. 53.

**APPARATUS AND METHODS USED FOR MAKING THE  
PHOTOMICROGRAPHS**

**Camera.**—The instrument used was a vertical photomicrographic camera, size 4x5, without lens, consisting of a bellows, ground glass focusing screen, plate holder and shutter with speed regulator. The camera was clamped to the stand, Figure 1, with the lower edge of the adjusting screw a at 22.5 and the lower edge of screw b at 36.0.

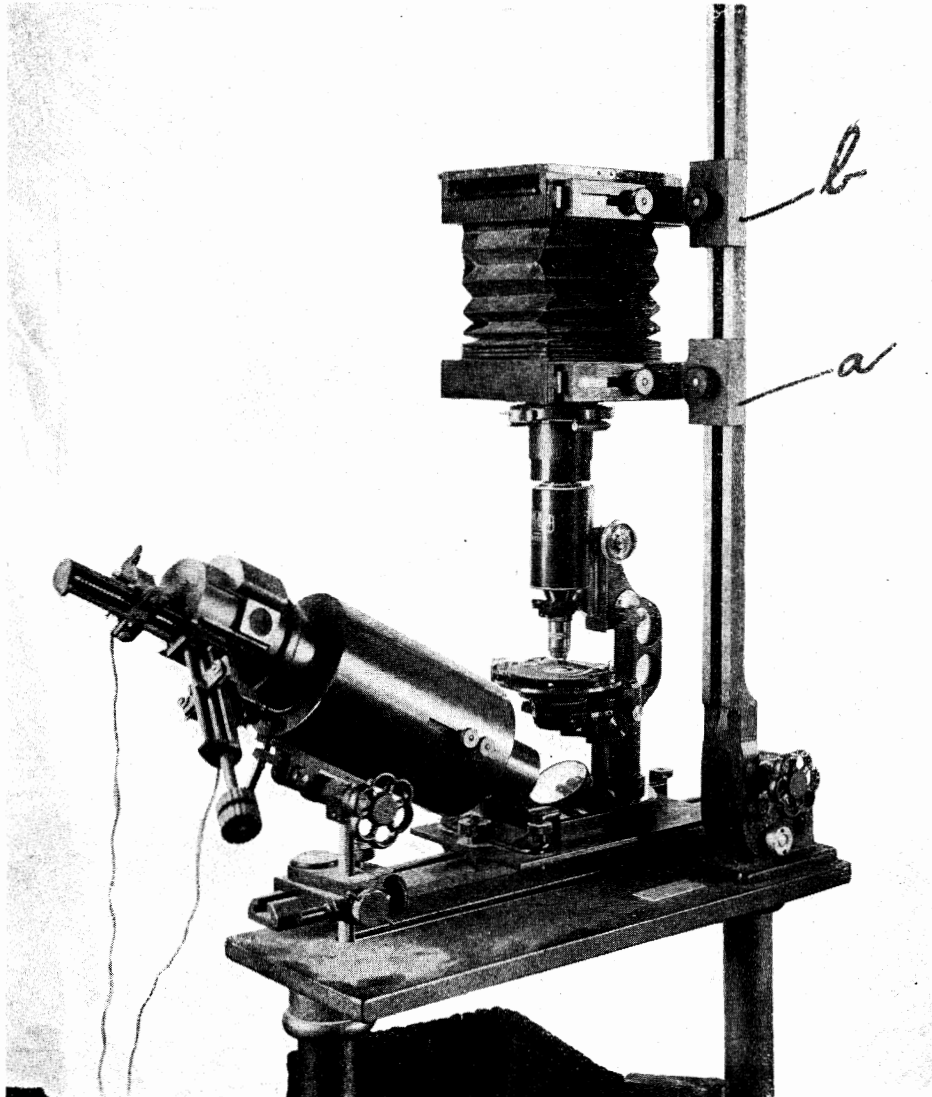


FIGURE ONE

**Microscope.**—All of the photographs, except those with polarized light, were made with a Zeiss microscope, using a 4 mm. aplanatic, aprochromatic objective with cover glass correction, a No. 8 compensating ocular and aplanatic condenser. It was necessary to use the ocular in order to get the desired magnification.

**Light.**—An electric arc lamp was used as a source of light so ar-

ranged as to diffuse the light rays and not to concentrate them. A piece of paper was placed over the mirror and the light focused on it until a dark spot appeared in the center. The iris diaphragm was closed and the condenser lowered until the detail became good, the least light giving the best detail being found to give the best results.

**Staining.**—The solution for staining was made by dissolving 25 mg. of iodine and 1 gram of potassium iodide in 5 to 10 cc. of water, and after the iodine was completely dissolved, diluting with water to 500 cc.

**Method.**—A counting chamber with lines 1-20 mm. apart was used for adjusting the magnification and for focusing. The light was centered on the ground glass focusing screen and the bellows of the camera adjusted so that the lines of the counting chamber were 15 mm. apart on the screen; this gave a magnification of 300 diameters. Then some starch was examined and the light and microscope arranged so that the hilum was in perfect focus.

It was necessary to make a correction for the plane occupied by the focusing screen and the photographic plate. Coincidence was accomplished by making one-tenth of a turn upward with the fine adjustment screw on the microscope after each specimen had been focused on the ground glass plate.

A small amount of starch, just enough to cover the end of a glass stirring rod, was placed in a 2-inch watch glass and covered with 2 cc. of the iodine solution. This was stirred with the rod for a few seconds, or until all of the starch was moistened, and allowed to stand for one minute, then the excess liquid decanted. By means of the glass rod 1 drop of the starch mixture was placed on a slide, the cover glass put in place, and, after focusing the plate was exposed for 1-25th of a second.

An effort was made to photograph the starch grains without the aid of a stain, but the pictures were not clear and lacked detail. Several stains were tried, but a very weak solution of iodine gave the best results.

**Developing Plates and Papers.**—The plates were developed slowly, usually for ten minutes. Exposure for various lengths of time were made, which necessarily influenced the time required for developing, but the most satisfactory results were obtained by a 1-25th-second exposure and ten minutes for developing.

#### Developing Solutions

##### Solution 1

Water, distilled .....	16.00 oz.	330.00 cc.
Sodium sulfite, anhydrous .....	2.5 oz.	50.00 gm.

##### Solution 2

Water, distilled .....	16.00 oz.	330.00 cc.
Sodium carbonate, anhydrous .....	1.25 oz.	25.00 gm.

##### Solution 3

Water, distilled .....	24.00 oz.	500.00 cc.
Oxalic acid .....	15.00 gr.	0.75 gm.
Pyrogallie acid .....	1.00 oz.	20.00 gm.

To develop use—

Water (winter) .....	6.00 oz.	175.00 cc.
Water (summer) .....	8.00 oz.	230.00 cc.
No. 1 .....	.50 oz.	15.00 cc.
No. 2 .....	.50 oz.	15.00 cc.
No. 3 .....	.50 oz.	15.00 cc.

## Acid Fixing Bath Solutions

## A

Water, distilled .....	48.00 oz.	1,075.00 cc.
Sodium thiosulfate, anhydrous .....	16.00 oz.	320.00 gm.
Sodium sulfite, anhydrous .....	1.00 oz.	20 gm.

## B

Water, distilled .....	16.00 oz.	330.00 cc.
Chrome alum .....	.10 oz.	20.00 gm.
Sulfuric acid, conc .....	.12 oz.	1.50 cc.

When dissolved pour B into A slowly with constant stirring. The Metol hydrochinone developer was tried but it did not seem to give any better results than the pyrogallic acid.

In using the developer not more than three plates were developed in the same solution. After developing, the plates were rinsed in distilled water and fixed fifteen minutes in the acid fixing bath, washed in running water one-half hour, then rinsed in distilled water and dried.

A number of different plates were tried. Hammer Extra Fast and Seeds 30 both gave good pictures. The plates used for all of the pictures shown here were "The Standard Polychrome Emulsion No. 9723, Extremely Rapid", made by the Eastman Kodak Company, Rochester, New York.

The papers were developed to the required depth in the following solution:

## Paper Developer

Dissolve in order named.

Water .....	300.00 cc.
Elon .....	.50 gm.
Hydrochinone .....	2.00 gm.
Sodium sulfite, anhydrous .....	7.00 gm.
Sodium carbonate, anhydrous .....	13.00 gm.
Potassium bromide, 10 percent solution .....	5.00 cc.

Dilute with equal volume of water for use.

The papers were fixed in plain "hypo" solution, made by dissolving  $\frac{1}{4}$  ounces of thiosulfate in 16 ounces of water.

The papers were printed fifteen seconds and developed to the required depth. This took about two or three minutes.

It was found that a photographic paper having a high gloss was the best for this work. The grade selected was "F Hard X, Single Weight, Glossy White Azo", manufactured by the Eastman Kodak Company. These papers were printed by an exposure of fifteen seconds at a distance of eight inches from a 40-Mazda electric light.

**Photomicrographs With Polarized Light.**—The Nicol prisms available would not fit our photomicroscope so we were compelled to use an ordinary microscope for this work. The process of getting the stands adjusted and the correct magnification was the same as with the plain pictures. The microscope and arc stands were in the same place as they were with the other set of pictures. In this work all the light possible was admitted. The arc was focussed to a small pencil of light on a piece of paper held on the concave mirror. This was necessary as there was no condenser used and therefore nothing to collect the rays and throw them into the objective.

The condenser was removed and the lower Nicol prism inserted in its place. The upper prism holder was in two parts. These were separated and the ocular placed in the tube and the upper part containing the prism put in place. This placed a prism below the objective and one above the ocular.



A No. 1 ocular with a 4 mm. objective was used. The bottom edge of the lower part of the camera was at 24.5 and the bottom edge of upper part of camera was at 33.5. This gave a magnification of 300 diameters.

No stain was used on the starch for this work; it was simply stirred up with water and mounted on the counting chamber. The upper prism was then turned until all of the light possible came through. After the grains were in focus the upper prism was turned to the darkest point. At this point very little light comes through, and scarcely anything may be seen, however, with the aid of a black head covering it will be seen that each starch grain has a cross of some sort on it. A sensitive plate was now put in place and exposed ten seconds.

The developing solutions were the same as those used in the other picture. These plates were developed five minutes and fixed fifteen minutes. The papers were the same as were used with the other pictures. They were printed five seconds and developed to the desired density.

#### THE DETERMINATION OF THE GELATINIZING TEMPERATURE OF THE STARCHES BY MEANS OF A THERMO SLIDE

The usual method for determining the gelatinizing temperature of starches requires that small quantities of the material be placed in test tubes which are immersed in a water bath, the temperature of which is gradually raised, and portions of the starch removed at intervals for microscopical examination. This has been found to be a tedious method and the results not so accurate as desired.

The apparatus designed for our work on the starches obtained from the various grain sorghums, permitted direct readings so that a large number of tests were made in a comparatively short time; triplicate determinations checked well within one degree. The thermo slide, Figure 2, was made in this laboratory and may easily be constructed by any one having a little ability to handle tools. The material consists of: A, three pieces of steel 5 inches long,  $1\frac{3}{8}$  inches wide and  $\frac{1}{8}$ -inch thick, with a central opening  $7\frac{1}{8} \times 2\frac{3}{4}$  inches cut in each piece, approximately one inch from each end and one-fourth inch from each side. Two  $\frac{1}{4}$ -inch nipples are fitted into the steel piece, No. 1, the holes for these being drilled three-quarters of an inch from each end. Two openings  $\frac{1}{4}$ -inch wide are cut in steel piece No. 2, as indicated in top view, Figure 3.

B. Two glass slides 1x3 inches.

C. Six rubber gaskets 1.5x5 cut from 1-16-inch. rubber sheets. Two of these gaskets are cut to match steel plate No. 2.

The pieces of steel and rubber have two  $\frac{1}{4}$ -inch holes drilled in each end through which bolts pass to clamp the entire system together.

The  $\frac{1}{4}$ -inch piping is connected by means of nipples fitted into the top plate. The thermometer is small enough to fit inside the T opposite the exit pipe and the opening made watertight by slipping a piece of tubing over the thermometer and the nipple. The general arrangement of the micro-polariscope, the thermo-slide and connections for the warm water are illustrated in Figure 2, and further details of the thermo slide are shown in the drawing, Figure 3, page 11.

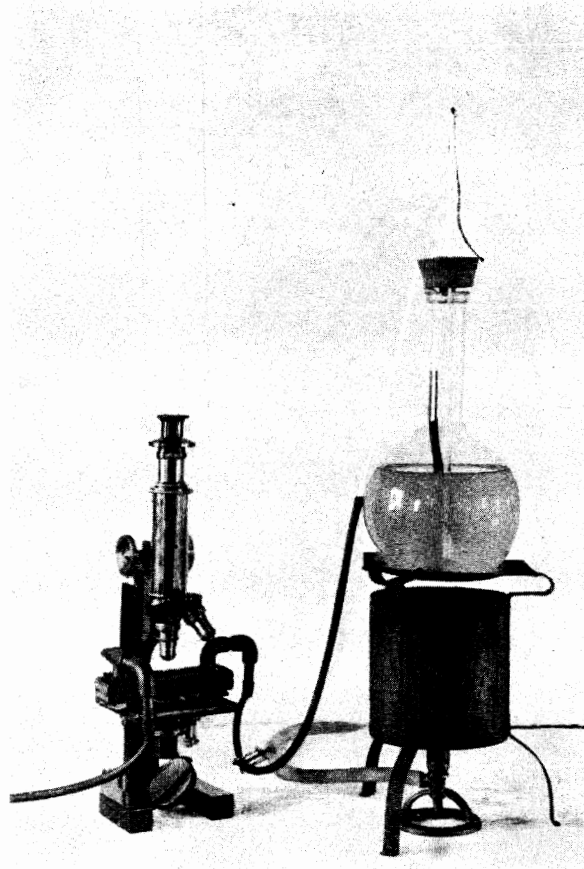


FIGURE TWO

**Procedure.**—The apparatus is arranged as shown in Figure 2, and the syphon started, then the flow adjusted by means of the pinch-cock so that the water will run at the rate of not over 100 cc. a minute. A second pinch-cock may be used as a total cutoff. The temperature in the flask is brought to 60° C., then the heat controlled so that the thermometer in the flask will indicate a rise of 1° every minute. The water bath (flask) and the microscope are placed not over eighteen inches apart so that there will be but little difference in the temperatures recorded in the flask and in the slide. This difference should not be over three degrees.

A small quantity of dry starch is placed on the thermo-slide, moistened with an excess of water, and a cover glass placed over it. The low-power, 16 mm. objective is adjusted and focused, then the analyzer put in place and turned to the dark field. Now turn on the water and note the temperature when all the starch granules have lost their polarizing properties. This temperature should not be considered to be the correct one, but is to be used as a guide.

The slide is now cleaned and a second portion prepared in the same way as has been described. Heat the water in the flask to the same degree which was recorded for the preliminary test and this will give a temperature in the thermo-slide about two to three degrees below that first found, and consequently below the gelatinization

point. The temperature of the water is slowly raised and a record made of the temperature indicated by the thermometer in the thermoslide, when the observer can no longer see any signs of anisotropy. This may be considered to be the temperature of gelatinization. By repeating the determination and with a little experience it will be found that the gelatinizing point is quite sharp, and may be usually read within a few tenths of a degree.

It is important that the starch be well covered with water during the test, because if the water is permitted to evaporate, gelatinization may not occur, or it may be delayed for several degrees beyond the true temperature. This action of water has been observed on potato

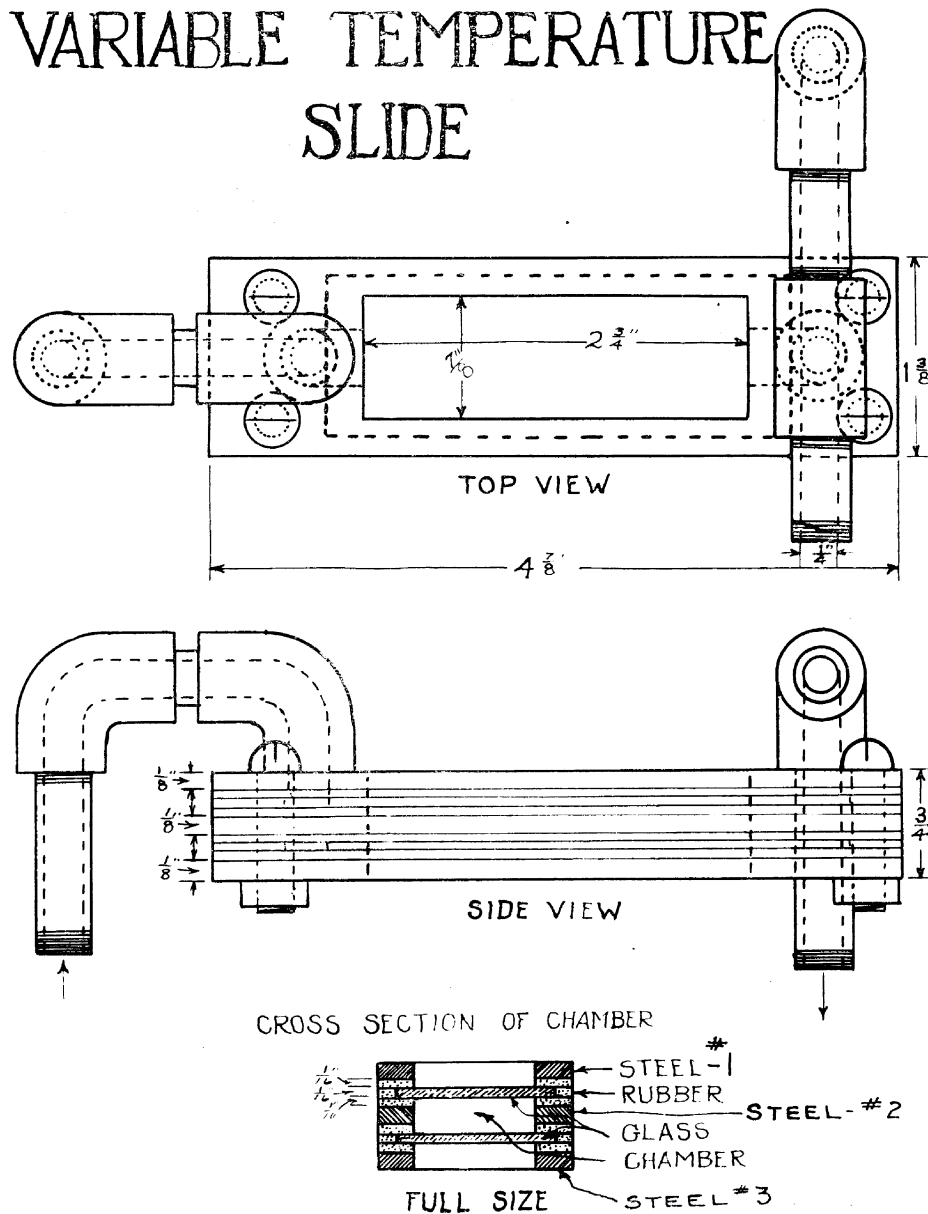


FIGURE THREE

starch, which gelatinizes from 67.4° to 67.8° C. in the presence of water, in an experiment during which the samples were covered with glycerine and heated to 100° without any apparent action on the granules.

The test must not be prolonged because high results may be obtained, due to the fact that some granules may partly gelatinize and the resulting paste protect the remainder from the action of the water. When using the water-bath, test tube method it was noticed that arrowroot starch heated from 65° to 79° did not entirely lose its anisotropic condition at the higher temperature, but when the sample was plunged into water heated to 75° the starch lost its polarizing properties immediately. Navy bean starch when tested by the water-bath method did not completely gelatinize at 83°, but when suddenly exposed to a temperature of 76° gelatinized at once. In other words, it appears that if starch is subjected to a gradual rise of temperature in the presence of water, that the gelatinizing point may become indefinite and not so uniform as when the starch is heated for a few minutes within three to five degrees of the true point of gelatinization.

We have selected as the true gelatinizing temperature the complete disappearance of optical activity. E. T. Reichert\* has recorded the temperature of the tube and the temperature of the water-bath, when anisotropy disappeared in practically all of the granules, then selected the mean as the temperature of gelatinization. Necessarily the number of starch granules on a slide would vary, consequently we deemed it better to wait for a total disappearance. The results are given in Table III.

\*Carnegie Institution. Differentiation and Specificity of Starches, p. 298 (1913).

TABLE III

## Gelatinizing Temperature of Starches from the Grain Sorghums

KINDS OF STARCH	THERMO SLIDE READINGS	AVERAGE
White kaoliang	78.0° C. 78.0° C. 78.1° C.	78.0° C.
Brown kaoliang	76.2° C. 76.4° C. 76.4° C.	76.3° C.
Feterita	75.4° C. 75.6° C. 75.6° C.	75.5° C.
Orange cane	72.2° C. 72.4° C. 72.6° C.	72.4° C.
Red kafir	74.8° C. 75.0° C. 75.0° C.	75.0° C.
White kafir	72.0° C. 72.1° C. 72.4° C.	72.2° C.
Pink kafir	64.6° C. 65.0° C. 64.6° C.	64.7° C.
White milo	74.0° C. 73.8° C. 74.4° C.	74.1° C.

KINDS OF STARCH	THERMO SLIDE READINGS	AVERAGE
Yellow milo	76.8° C. 76.8° C. 77.0° C.	76.9° C.
Barso	72.8° C. 72.8° C. 73.2° C.	72.9° C.
Sudan grass	72.4° C. 72.6° C. 72.4° C.	72.5° C.

The proposed method does not require so much time as the water-bath method, and the gelatinizing point can be determined with more accuracy. When the determination is made by the water-bath method the readings must be made on separate portions, and many minutes are required for a single determination. By means of the preliminary test the approximate gelatinizing point may be determined and then the temperature fixed by one or two more tests on the thermo-slide. A series of tests were made by both methods, the results of which are shown in Table IV.

TABLE IV  
Gelatinizing Temperatures of Some Common Starches as Determined By the Thermo-Slide and Water-Bath Methods

KIND OF STARCH	TEMPERATURES ARE DEGREES CENTIGRADE			
	THERMO SLIDE METHOD	AVERAGE	WATER-BATH METHOD	AVERAGE
Arrowroot	74.4 74.8 74.6	74.6	74 to 75	74.5
Corn	70.6 71.2 71.0	70.9	71 to 72	71.5
Navy bean	76.0 75.1 75.4	75.6	75 to 76	75.5
Sweet potato	82.6 82.6 82.4	82.5	82 to 83	82.5
Irish potato	67.6 67.8 67.8	67.7	67 to 68	67.5
Wheat	65.5 65.0 65.1	65.2	65 to 66	65.5

The advantages of the thermo-slide method over the water-bath method are numerous. When working with the latter one is compelled to withdraw portions of the starch from the tube and make a slide at each degree, or fraction of a degree, whereas the thermo-

slide permits constant observation of the starch. The water-bath method requires more time for each test and the gelatinizing point cannot be determined with the same accuracy as by the proposed method. Moreover, the total time necessary is very much shorter, as numerous determinations have been made by the thermo-slide method which required less than five minutes to complete each test.

### **CHARACTERISTIC REACTIONS**

In this section the preparation of the special reagents and the methods of using them are first described. The details of each observation follow, and are reported in the same order for the tests on the individual starches.

#### **Microscopic Form of Starch Granules**

In the study of the form of the starch granules of the grain sorghums, the starch under investigation was placed on a slide, moistened with .005% iodine solution and examined with the oil immersion lens.

#### **Chloral-Hydrate Iodine Reaction of the Starches**

The reagent used in this reaction was a saturated solution of chloral-hydrate saturated with iodine. A small amount of the starch was placed on the slide, moistened with a drop or two of the reagent and observed through the microscope. The polarizing microscope was used to determine the degree of disintegration of the cell substances.

#### **Chromic Acid Reaction**

The test solution contained 25% chromium trioxid. A small amount of starch was placed on a slide, a drop of the chromic acid solution added, covered with a cover glass, and the action noted under the microscope.

#### **Ferric Chloride Reaction**

A one-half saturated solution of ferric chloride was made by saturating 25 cc. and diluting with an equal volume of water. The reaction of this reagent on the starch was studied by moistening a small amount of the starch with the solution and observing the behavior under the microscope.

#### **Gentian Violet Reaction**

A 0.5% solution of gentian violet was made and kept in a brown glass bottle in the dark. Each day portions of this solution were diluted ten times, giving a solution containing 0.05% gentian violet. Five cc. of this were placed in a test tube and a small amount of starch added. Slides were made at intervals of one, five, ten and thirty minutes after the staining was commenced. The tints were designated as light, medium and heavy violet.

#### **Pyrogallic Acid Reaction**

The solution used in this reaction was made as follows: 25 cc. distilled water were saturated with pyrogallic acid, diluted to 100 cc. and 4 g. oxalic acid added.

The starch was placed on a slide, a drop or two of the pyrogallic acid solution added, covered with a cover glass, and examined under the microscope.

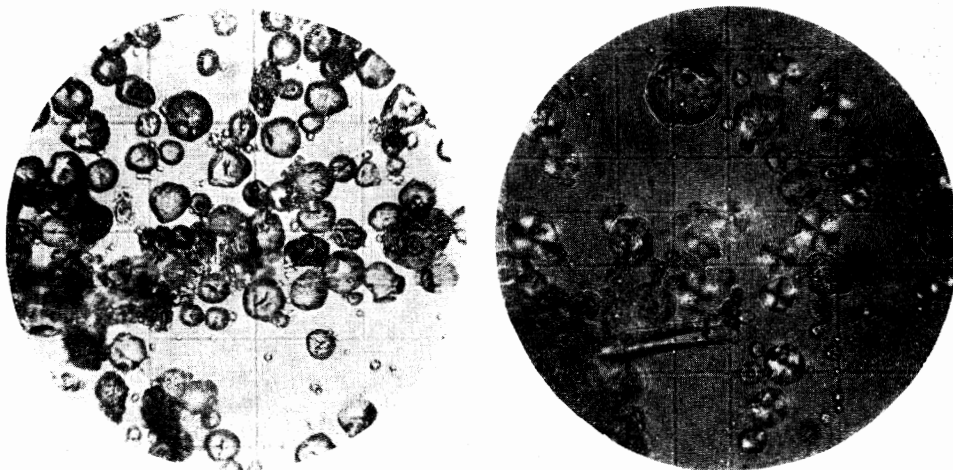
### Safranine Reaction

A 0.5% solution of safranine was made and kept in a brown glass bottle in a dark place. Each day portions of this solution were diluted ten times, giving a solution containing 0.05%. This was the solution used for the color reactions. Five cc. of the 0.05% solution was placed in a test tube and a small amount of starch added. Slides were made at intervals of one, five, ten and thirty minutes after the staining was commenced, and examined under the low power of the microscope. The stains were designated as light, medium and dark red.

### WHITE KAFIR

**Microscopic Form.**—Hilum in center. Round. Some seen in clusters of three or four grains. Often show pressure facets. No striae or lamellae visible. Often see crevices from hilum which are not regular in shape, but generally consist of three separate channels running toward the edge, making about 120° angles.

The illustrations are arranged under each starch with the photograph by polarized light to the right. The original showed a magnification of 300, but this has been reduced to 240, in each case, by the engraver.



Size.—4 to 23 microns. Average 17 microns.  
Gelatining temperature, 72.2° C.

### Chloral-Hydrate Iodine Reaction

The action was slow in starting and did not attack the hila as readily as in other starches. The grains were stained a light violet-blue. The hila in most of them became distinct in five minutes and were in the shape of inverted cones with rough edges. Fissures around the hila had disappeared somewhat, but had not widened into the hilal cavity. Only a few of the grains had disintegrated after fifteen minutes; occasionally one was seen that had broken up into several pieces. In a few the reaction had started at the edges; these showed a dark blue portion enclosing a lighter portion. After standing all night some were still unaffected and these had taken on a deeper violet color. The reacted grains still retained a distinct blue coloration, but they had faded somewhat. A few of these contained unaffected granules which were violet in color. Where the grains swelled very much they appeared to have ruptured on the top, leaving the interior exposed, which was of a lighter blue than the outer edge.

In some grains the action had taken place from the center toward one side more than the other, leaving a part with the violet coloration intact, while the remainder had a pale blue color.

#### **Chromic Acid Reaction**

The reaction began in about one-half minute and proceeded with fair rapidity. Striae were to be seen on almost all of the grains, but these gave place in a short time to a granular structure which persisted for three or four minutes. In some of the grains there was a round hole at the top, where the hilum had been, which developed after the hilum had been closed. Through this opening a fine granular structure was apparent. It appeared that a part of the outer covering had been dissolved more readily here than elsewhere. The action on this starch was very much slower than on any of the others. Many of the grains presented the appearance of hollow spheres. The external portion seemed to be very resistant to this reagent. Action was complete within fourteen minutes.

#### **Ferric Chloride Reaction**

The hilum was stained a dark brown. Within three minutes a number of grains had reacted; fissures developed around the hila which widened and came together as the grains swelled. No striae developed. As the grains reached their maximum size they became more transparent and their outline less distinct. The radial fissures persisted in the enlarged grains as a shallow, wide hollow, running lengthwise. After thirty minutes the majority had reacted, the grains swelling to about four to eight times their normal size, but retained their normal shape. After standing over night the grains dissolved leaving only a cluster of gas bubbles to mark the place where they had been.

#### **Gentian Violet Reaction**

The grains took on a light stain in one minute which did not change on longer staining.

#### **Pyrogallic Acid Reaction**

The action started on some in one minute and was completed in eleven minutes. Some of the grains developed a much coarser granular structure than others, while on many a fine granular structure was seen through the opening at the top. On swelling a rough surface was formed and fine striae appeared on many. The swollen grains were about three to six times the normal size. After forty minutes the granular formation had disappeared, but none of the grains had gone into solution. Examined again in two hours the grains presented rough outlines with very few gas bubbles and no granular structure.

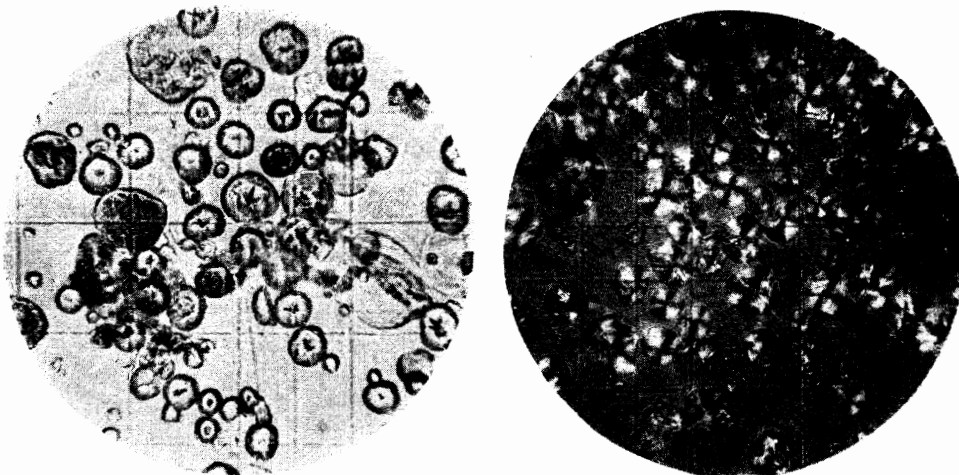
#### **Safranine Reaction**

The grains stained very lightly in one minute; deepened slightly in ten minutes, giving a final tint of light red.



**RED KAFIR**

**Microscopic Form.**—Shape, round; hilum in center. Three-pronged fissures on most of the grains. A number show pressure facets. No lamellae or striae to be seen.



**Size.**—6 to 33 microns. Average 18.  
Gelatinizing temperature, 75.0°.

**Chloral-Hydrate Iodine Reaction**

The reaction began in three minutes, and in twenty-two minutes about one-half, and in thirty-six minutes most of the grains had lost their polarizing properties. However, after two hours many of them had not reacted, and even after standing all night some had resisted the reagent. The reaction was not very rapid after the first effect on the hila; these became enlarged and very prominent and took on the shape of an inverted cone and a brownish-blue color, which deepened as the reaction proceeded. The cavities at the hila did not have smooth edges, but appeared to be covered with small fissures running down into the point of the cone.

The starch grains stained a light blue almost immediately, which became more violet and much deeper as the reaction proceeded. In some grains marked striae were seen running from the hila to the edge and gradually disappeared as the reaction proceeded. Examined after standing all night they had taken on a deeper violet stain, the hollow at the hila being a very dark brownish-violet. Some of the grains had disintegrated entirely and others had developed fissures, leaving lobes of unattached granules. Many that had been acted upon showed small granules of unattached starch.

**Chromic Acid Reaction**

The action started immediately and appeared to commence at the center of the starch grain. Striae appeared running from the center to the edge, the grains first swelling then spreading, rapidly going into solution. The granules became filled with numerous minute bubbles which soon disappeared. After all the grains had dissolved there still remained many small bubbles in the liquid. The reaction was complete in three minutes.

**Ferric Chloride Reaction**

The hilum was dark and very prominent. The action began on a few almost immediately, causing the central cavity to enlarge. After

eight minutes about one-third of the grains had reacted; the hila cavities had disappeared in the swollen grains which became elongated in many instances and the surfaces appeared to become rough. After one-half hour, approximately half of the granules had swollen to over five times their normal size, but it was two hours and forty minutes before all of the grains showed indications of swelling. Their outlines were not very regular and the surfaces in many places were roughened by fissures and ridges. No lamellae, striae or granular structures were seen, but there were a few gas bubbles.

#### Gentian Violet Reaction

In one minute the grains had taken on a light stain, which did not change on longer standing.

#### Pyrogallic Acid Reaction

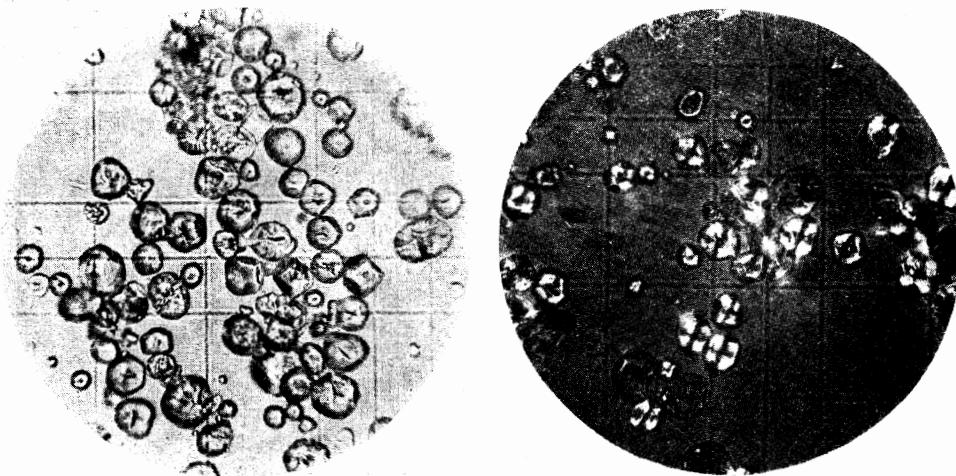
The action started in five minutes, proceeding very slowly and regularly. In approximately ten minutes half of the grains had reacted; three-quarters in fifteen minutes, while all had reacted in one and one-half hours. The grains swelled to about two or three times their normal size. Striae developed around the hila; these widened until the hilal vicinity became a cavity. The interior of the grain, as seen through this cavity, presented a striated, granular appearance. A rough, angular area developed in the vicinity of the hila, while small gas bubbles appeared in some of the grains. In five hours the grains had become very granular in structure and very large, but still had many fissures and crevices on their surfaces. None of the grains had gone into solution, but a number of small bubbles were seen in them.

#### Safranine Reaction

The grains had taken on very little color in one minute, which increased slightly in intensity for ten minutes. There was no apparent change after this; the final color being light red.

#### PINK KAFIR

**Microscopic Form.**—Round hilum in center. There were fissures of the straight and three-pronged type on most of the grains. Some show pressure facets. No striae or lamellae visible.



**Size.**—6 to 20 microns. Average 16.  
Gelatinizing temperature, 64.7°.

### **Chloral-Hydrate Iodine Reaction**

Action started almost immediately at the hila, which swelled and covered about one-fourth to one-half the surface of the grain. After twenty-five minutes many had reacted and taken on a deep blue color, while others were colored a light blue with the hila a little darker. These seemed to become more prominent and the fissures had widened somewhat and formed a part of the hilal cavities. These, when focused, refracted the light like gas bubbles. Occasionally a grain was seen where the reaction had commenced on the side; in these the starch substance seemed to have flowed out and attained a lighter colored stain than the other granules. Examined again in twenty-five minutes, a dark portion appeared surrounding a lighter one in the center of some, which was very irregular in shape. After standing all night the reacted grains had lost their deep blue stain and had taken on a light violet. Very few of them contained unaffected granules. Some had been divided into two or three lobes by the reagent acting along the fissures across the grain. The cavities around the hila were very prominent and dark colored, but had not enlarged since the first twenty-five minutes. The grains had swollen from five to ten times their normal size, and had a light area in the center surrounded by a darker one. Occasionally a grain was seen that had a dark circle covering about one-half the area in the center, and the outer edge was lighter in color. Most of the grains retained their original shape on swelling.

### **Chromic Acid Reaction**

The reaction on this starch was extremely rapid. By the time the cover glass could be put on and the microscope focused, practically all of the grains had disintegrated. They swelled immediately to about four times their normal size, closing the hilal cavity, from which a gas bubble emerged which contracted and generally disappeared. No striae appeared on any of these grains. The action was complete in one minute.

### **Ferric Chloride Reaction**

The grains had begun to show the action of the reagent within one minute. The hilal cavity enlarged, but was closed as the entire grain swelled. The reaction of this starch was very rapid; after twelve minutes half of the grains had reacted, and after three hours all of the grains were about ten times their normal size, but apparently no solution of the grains had taken place. The action was uniform.

The outline was not very definite and regular; ridges and shallow fissures occurred over the surface. No striae, lamellae or granular structures were seen and but a trace of gas bubbles.

### **Gentian Violet Reaction**

The grains took on a light stain in one minute which deepened in color for thirty minutes giving a final color of medium violet.

### **Pyrogallic Acid Reaction**

The action was very rapid as half of the grains had been affected in two minutes. In three minutes all the grains had swollen and the reaction seemed to be over. They all had a rough, angular appearance, but none had striae and very few had any granular structure. In most cases the opening at the top where the hilum had been, covered almost the entire grain. Examined again in four minutes no change

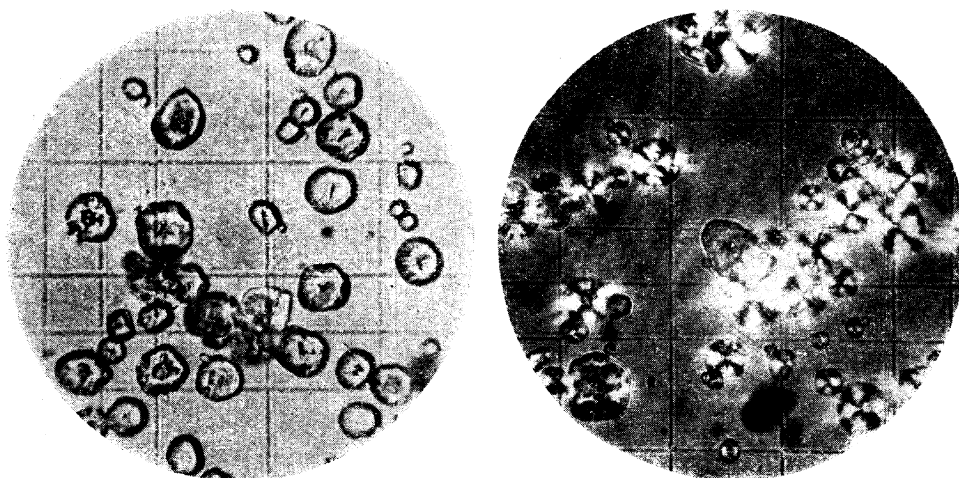
had taken place. The same was true after two hours, except that they may have been a little larger. The surface was very rough; formation of gas bubbles quite evident, but no granular structure.

#### Safranin Reaction

The grains took on a light stain which increased slightly in depth for five minutes. The final tint was light red.

#### WHITE MILO

**Microscopic Form.**—Grains round. Hilum in center. Striae present on occasional grains. No lamellae visible. Fissures run from hilum toward edge of grain. They consist principally of a single fissure running across the grain and are seen on but approximately half of the granules. Pressure facets seen only occasionally. Never seen in clusters.



**Size.**—6 to 29 microns. Average 20 microns.  
Gelatinizing temperature, 74.1°.

#### Chloral-Hydrate Iodine Reaction

The action commenced almost immediately and seemed to start at the hila, which took on a very light stain. The hilal cavity in these grains were not very regular; some were like inverted cones, but most of them were just ragged holes. After ten minutes very few grains were stained a dark blue, but the fissures around the hila had disappeared and fine striae were seen in a number of grains. After standing all night there were many grains still unaffected. These all had very prominent hila, and were stained a dark brownish violet, while the remainder of the grain was violet. Some of the grains still contained unaffected granules, while others seemed to have gone into solution to a certain extent.

#### Chromic Acid Reaction

Reaction was very similar to that of yellow milo, and was complete within four minutes.

#### Ferric Chloride Reaction

The hila were dark brown and very prominent. The grains began to swell in four minutes, and as they enlarged the hilum closed, leaving generally a shallow fissure. Sometimes radial fissures developed

which widened and consolidated, forming a large opening. The grains were from five to ten times the normal size. The reagent acted on all of the starch within twenty-five minutes. Only a few striae and a slight granular structure appeared.

The grains passed into solution when left in the reagent over night, leaving clusters of gas bubbles.

#### Gentian Violet Reaction

The grains stained very lightly during the first minute, but continued to deepen slightly for thirty minutes, giving a final color of light violet.

#### Pyrogallic Acid Reaction

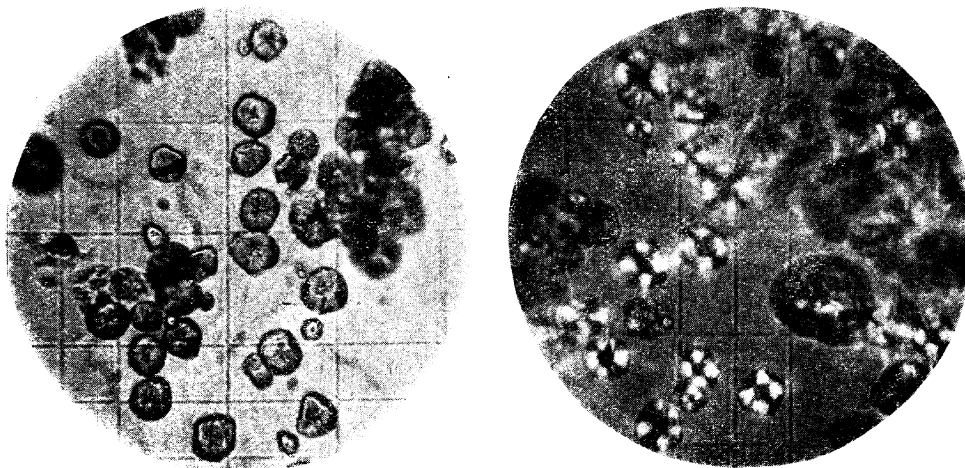
The action began on some in one minute; half of them had reacted in six minutes, while all had reacted in twenty minutes. Fissures appeared to radiate from the hilum in some and striae in others. They seemed to widen and run together, forming an opening in the top. A marked granular structure was seen in the interior of many grains. The swollen grains were from two to four times the normal size. Examined again in two hours they did not have any granular structure, although they were very large, with rough edges, and the openings in the top were very conspicuous. Few gas bubbles were noted.

#### Safranine Reaction

The grains took on a light stain in one minute, which increased in intensity for thirty minutes, giving a final tint of medium red.

#### YELLOW MILO

**Microscopic Form.**—Hilum in center. Round. Striae seen on occasional grains. No lamellae. Fissures on only a few, and these are principally single, straight ones. Some grains are quite irregular and show considerable number of pressure facets.



**Size.**—6 to 26 microns. Average 17.  
Gelatinizing temperature—76.9°.

#### Chloral-Hydrate Iodine Reaction

The action was fairly rapid, as it began in three minutes, and one-half had reacted in thirty-five minutes, but some were still unaf-

ected after standing all night. The hila took on a deep color, almost immediately, and appeared in most of the grains as large, well defined spots or craters. About one-fourth of the grains had taken on a deep blue color in fifteen minutes, and fine striae appeared in a few. The hila enlarged much faster than the rest of the grain, having fissures running from the top to the bottom of the cavity; they also had rough sides and indicated that the starch in the immediate vicinity went into solution. The hila covered from one-fifth to one-half of the surface. Some of the grains had commenced to react at one end, and in this case that part was dark blue, the rest being violet. Very few grains ruptured or lost their rounded appearance, and the swollen ones were somewhat more rounded than the normal ones. After standing all night many had not reacted, but were stained dark violet. In some the action proceeded in one direction from the center and left the other side unaffected, or simply divided into several segments, while some of the reacted grains still contained granules of unaffected starch. Where the grains had reacted there was a lighter rugged area in the center, extending to the outer edge in some cases.

#### **Chromic Acid Reaction**

The reaction of this starch was practically identical with brown kaoliang starch.

#### **Ferric Chloride Reaction**

Some of the grains had begun to react within three minutes, the hila taking on a dark brown color. Within three hours the grains had all reacted and swollen to five to ten times their normal size, large openings being seen in some. There were no lamellae, striae or gas bubbles present, and no granular structure developed.

#### **Gentian Violet Reaction**

The grains took on a light stain in one minute and were not changed by longer staining.

#### **Pyrogallic Acid Reaction**

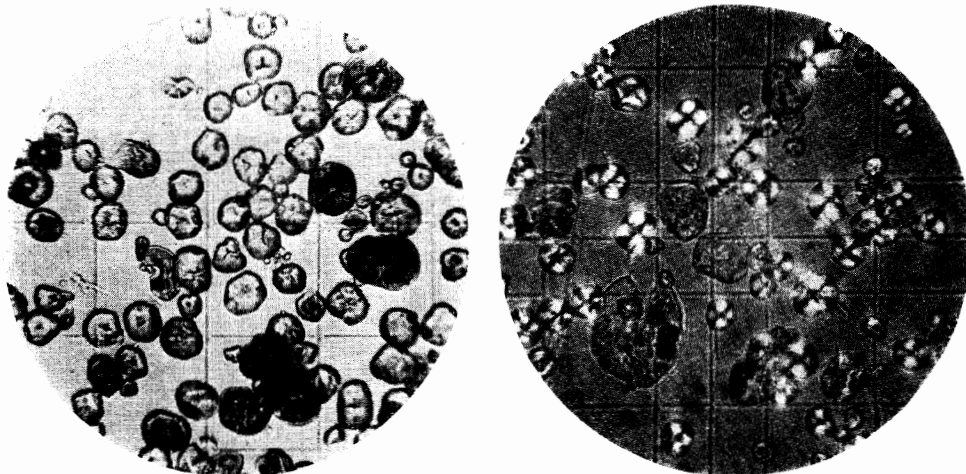
The action started in one minute on some of the grains, but most of them were unaffected. In ten minutes about three-fourths of the grains had begun to react, and in thirty minutes almost all of them had been affected. Fine striae appeared on some grains, which seemed to run together and form an opening in the top. The hilum appeared as a light spot. The interior of the grains had a marked granular structure with some rather large granules scattered throughout the smaller ones. Gas bubbles developed both on the outside and inside of the grains. The grains increased about three to six times their normal size. When examined after three hours they were very large and had a rough, angular surface; gas bubbles were also noticed at this time.

#### **Safranine Reaction**

The grains took on a very light tint in one minute, which increased in intensity for thirty minutes, giving a final tint of about medium red.

## BROWN KAOLIANG

**Microscopic Form.**—Shape round, hilum in center. No striae or lamellae. Very few grains show fissures. Pressure facets not very marked.



**Size.**—3 to 23 microns. Average 16.  
Gelatinizing temperature, 76.3°.

**Chloral Hydrate Iodine Reaction**

The action proceeded rather slowly as only one-half of the grains had reacted to the chloral hydrate in thirty minutes, while many were not affected even after standing all night. Nearly all of the grains were stained a light violet-blue at first, which gradually deepened. The hila appeared as round, brownish spots, and faint striae were seen in a few grains. After twenty minutes quite a number had reacted, showing deep ruptures, and in some cases large pieces had been separated almost entirely from the remainder of the grain. A few had reacted only at one end, and in these cases one part of the grain was stained a deep blue, the other a violet. The outer edge of the grains appeared to be slightly darker than the interior, and the fissures around the hila gradually disappeared. It seems as if these closed up and did not give rise to the enlarged wells seen in the hilal vicinity. When the grains broke they usually opened only on one side and developed a lighter, rugged area in the center, which extended to the outer edge in many instances. Examined after thirty-five minutes, the hila had developed into inverted cones with rough sides, covering from one-fifth to one-half the area of the grains. After standing all night the grains were much darker in color and the hila were very dark in all. Some of the reacted grains still showed unaffected portions, others had divided into two or three parts by the reagent acting in channels across the grains, leaving the lobes with their normal polarizing properties.

**Chromic Acid Reaction**

The action on this starch was not so rapid as on red kafir and orange cane. The swelling did not cause the fissures around the hilum to close, but they appeared to dissolve. Striae developed as the swelling diminished; then the granulated structure came into view, persisting much longer than it did with kafir starch. The outer part was the last to go into solution. The action was complete within four minutes.

#### Ferric Chloride Reaction

The hila were not prominent in this starch; action began in four minutes. The outlines of the grains were fairly regular, and they were about the same shape as the normal grains. Neither striae, lamellae, gas bubbles nor granular structures were seen. The smaller grains were about five to ten times the size of the normal grains. The action was complete in two hours and fifteen minutes.

#### Gentian Violet Reaction

In one minute the grains had taken on a light stain which did not darken but very slightly on longer treatment.

#### Pyrogallic Acid Reaction

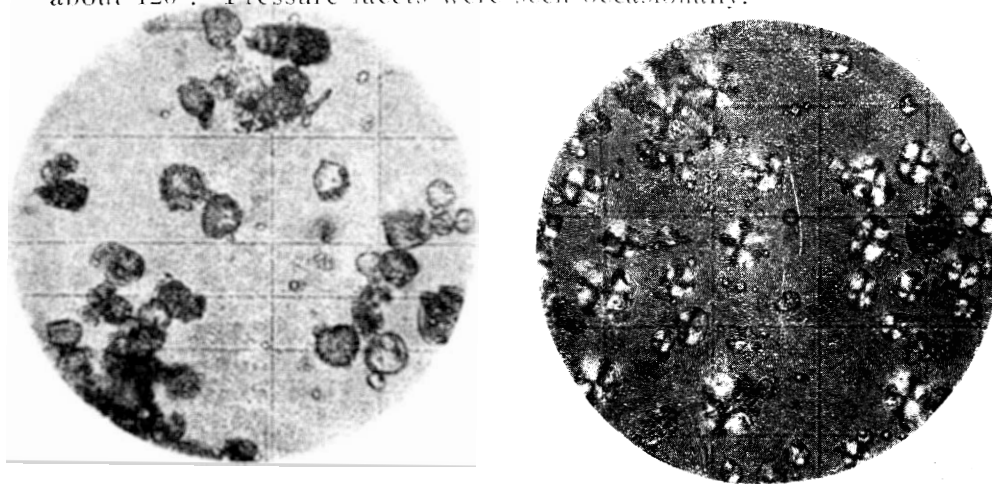
The action started in four minutes and proceeded very slowly. About half of the grains had reacted in fifteen minutes, while most of them had reacted in thirty-five minutes. The surface around the hila developed a rugged, angular appearance with fissures running in all directions. Many had the opening at the top, while some showed striae covering this part of the grain. Most of the grains seemed to be broken up into numerous small rods running from the inside to the outer edge and leaving a hollow space in the center. This may account for the granular appearance of the center. As the action progressed small gas bubbles appeared in many grains, and rather large ones in a few. Examined again after three hours they still presented a rough, granular appearance and had gas bubbles scattered throughout them.

#### Safranin Reaction

The grains took on a very light stain in one minute, which increased in intensity for thirty minutes, giving a final tint of about medium red.

### WHITE KAOLIANG

**Microscopic Form.**—Grains round. Hilum in center. Striae seen occasionally. No lamellae. Fissures from hilum to edge are seen in one-third of the grains. About one-half of the fissures are single, straight channels, others run in three directions, making angles of about 120°. Pressure facets were seen occasionally.



**Size.**—4 to 23 microns. Average 20.  
Gelatinizing temperature 78.0°



### **Chloral Hydrate Iodine Reaction**

The action started on the hila almost immediately, and they were stainedd much darker than the remainder of the grains. The crevices around the hila gradually disappeared, some widening out to form the cavity. The reaction was very slow after the first effect in the immediate vicinity of the hila. After standing all night there were still quite a number of unaffected grains which had not deepened much in color and were much lighter than the corresponding grains of the other starches studied. The reacted grains had retained most of their blue color, and some contained unaffected granules. Some were affected only by being broken up into many small granules; others had a dark center and a lighter portion surrounding it.

### **Chromic Acid Reaction**

The reaction started immediately with rapid swelling of the grains. Striae developed, but soon disappeared, and a granular structure was seen. Action was complete in four minutes.

### **Ferric Chloride Reaction**

Within two minutes some of the grains had begun to react. The hila were very prominent as dark brown spots, and these closed as the grains swelled. Fissures radiated from the hila; these widened and ran together, forming an opening in the top of the grains. These grains did not appear to have the form of hollow spheres. After six minutes about one-third of the grains had reacted, and within twenty-five minutes all had shown some action of the reagent. After standing all night the grains had dissolved with the evolution of gas.

### **Gentian Violet Reaction**

The grains took on a light stain in one minute and did not change in subsequent staining.

### **Pyrogallic Acid Reaction**

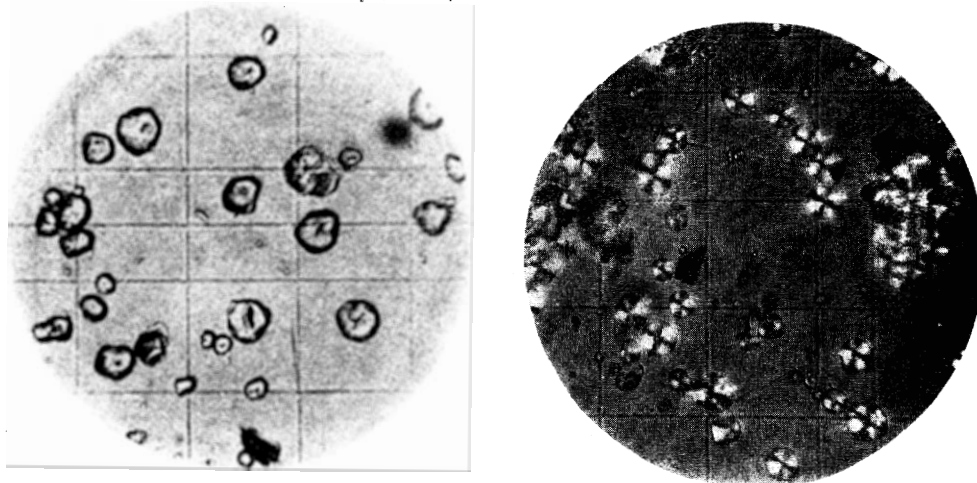
The action started on most grains in two minutes and was complete in ten minutes. The grains swelled from two to four times their original size and developed a rough, angular surface. An occasional grain had a granular surface, but most of them did not have this structure. Fissures developed, radiating from the hila, which widened and came together, forming a large opening on top of the grain. Gas bubbles were noted in only a few. Examined again in one and one-half hours, the grains had a marked granular structure, the surface was irregular and many gas bubbles were seen.

### **Sefranine Reaction**

The grains took on a light stain in one minute, which increased in intensity for thirty minutes, giving a final color of medium red.

## FETERITA

**Microscopic Form.**—Shape, round; hilum in center. Striae on occasional grains. No lamellae visible. Fissures on most grains. Pressure facets not very conspicuous.



**Size.**—6 to 26 microns. Average 18.  
Gelatinizing temperature 75.5°.

**Chloral Hydrate Iodine Reaction**

The action seemed to start immediately at the hilum, but not in the rest of the grain. When once started it proceeded rather slowly, as only one-fifth of the grains had swelled much after standing all night. The grains were stained a very light blue, but the hila darkened and deepened, and the action always proceeded from the hila to the edges. A number had reacted in fifteen minutes, taking on the deep blue color. Occasionally a grain was seen to break into several pieces; the hila of these appeared like an inverted cone with rough sides. Fine striae appeared on some grains. After standing all night all the hila were large and dark, but otherwise most of the grains were normal. The unaffected grains were stained darker, whereas some that had been a deep blue when first examined had almost entirely lost their color, being a very faint blue and almost invisible. Some of them had been divided into two or three pieces by the reagent acting along a channel across the grain.

**Chromic Acid Reaction**

The first action was the swelling of the grain, which began in about one-half minute. Where there was a central cavity alone it closed, but if there were crevices in addition the cavity and crevices combined and enlarged. Striae appeared all over those grains having crevices around the hila, but the top of the grain seemed to disappear at once if no crevices were present. Those cells showing striae usually developed granular structures, apparently in the interior of the starch grain only. This disappeared leaving the shell of the grain to dissolve last. The action was complete in three minutes.

**Ferric Chloride Reaction**

The action began within three minutes. The hilum was very prominent as a dark-brown spot or fissure, but as the grain swelled this was obliterated, leaving a ridge in its place.

All of the grains had reacted within three hours. These were about five to ten times the size of the normal grains, with a comparatively smooth surface. A rather wide, shallow fissure was seen in a good many grains; probably the last trace of the deep, narrow fissure seen at the hilum of the normal grains. No striae, granular structure or gas bubbles developed.

#### Gentian Violet Reaction

In one minute the grains had stained a light violet and did not change.

#### Pyrogallic Acid Reaction

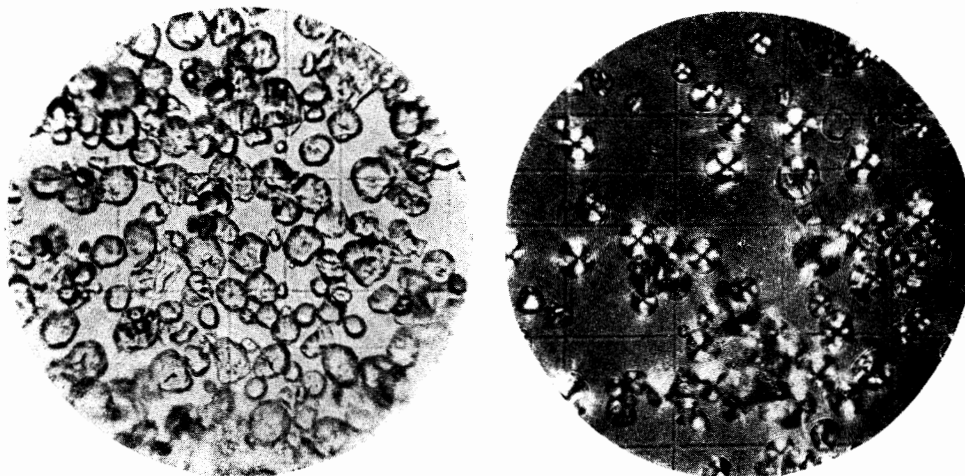
The action started in one minute and proceeded fairly rapidly. In seven minutes three-fourths of the grains had reacted, while all had reacted in twenty minutes. The granules swelled and assumed the form of a hollow sphere with openings at the hila. Some developed a rough, angular surface, while others were covered with fine striae and showed a fine, granular structure in the hollow of the grains. Very few had become round on swelling. There were not many gas bubbles formed in this starch. Examined again in two and one-half hours, the grains were not very granular; some, however, had a coarse structure with large granules. No solution of the grains was evident.

#### Safranine Reaction

The grains took a light stain in one minute, which increased in intensity for ten minutes, giving a final tint of about medium red.

#### DARSO

**Microscopic Form.**—The grains occur singly, are round or oblong, with no sharp edges or corners, and very few pressure facets. The hilum is in the center of the grains, with a fissure running through it in some instances. Lamellae or striae do not occur.



**Size.**—6 to 23 microns. Average 18.  
Gelatinizing temperature, 72.9°.

#### **Chloral Hydrate Iodine Reaction**

The hilal cavity was stained immediately, while the rest of the grain was not affected. The hila had principally the shape of inverted cones and were stained a deep brownish color, while the rest of the grains stained a very pale blue. On some grains the action started on the edge and proceeded toward the center. After one-half hour the hila had enlarged and the grains had taken on a deeper violet color; the cavities at the hila were irregular in shape, and striae were observed on some of the grains; the reagent seemed to follow these. In two and one-half hours only one-fourth of the grains had reacted, and after standing all night many were still unaffected by the chloral hydrate. The unaffected grains had taken on a somewhat deeper stain, being now a violet-blue, while the reacted grains had lost most of their color. It seems as if the grains had either completely reacted or not at all.

#### **Chromic Acid Reaction**

All of the grains swelled in about one minute. Striae developed and the granular structure was present. The crest around the hilum seemed to be the first portion of the outer part of the grain to dissolve, and this gave a view into the interior of the grain. Action was complete in three minutes.

#### **Ferric Chloride Reaction**

The action began within three minutes. The hilal cavity was large at first and of a dark brown color. As the grains swelled these diminished in size and finally disappeared leaving only a shallow fissure. About one-half of the grains reacted within twenty minutes, striae developing in some, together with a fine granular structure. At the end of an hour all of the grains had reacted. After standing all night the grains had dissolved with the evolution of considerable gas.

#### **Gentian Violet Reaction**

The grains were a light violet at the end of one minute, and did not change on further treatment.

#### **Pyrogallic Acid Reaction**

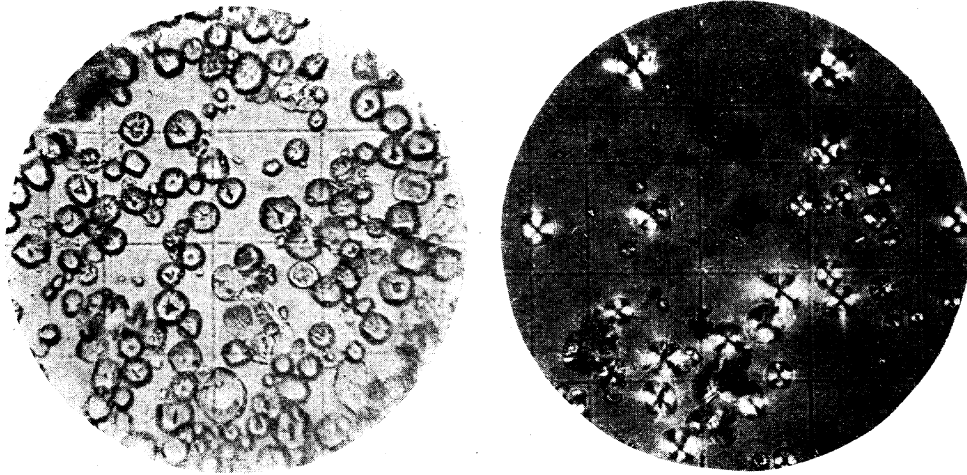
The action started on a few grains in one minute, but was not very rapid. In ten minutes most of them had commenced to react, though all had not reacted before three hours had elapsed. The entire grain swelled; some were smooth in general outline and others were rough. The fissures around the hilum widened, but never disappeared entirely. Some had developed striae, but very few had any granular structure. In thirty minutes one-half of the grains had swollen from two to four times their normal size, and a granular structure was noted in a few of the largest. On the top of nearly all there was a shallow fissure extending nearly across the grain, while in many there was a hollow space where the hilum should have been. No striae, lamellae, granular structure or gas bubbles were noted after three hours, neither was there any apparent solution of the starch granules.

#### **Safranine Reaction**

The grains took on a light stain in one minute, which increased very slightly in intensity for thirty minutes, giving a final color of light red.

## SUDAN

**Microscopic Form.**—The grains are round with pressure facets on a few. The hilum is in the center and generally has a small fissure running through it. In some these fissures are three-pronged. The grains do not occur in clusters, and lamellae or striae are not visible.



**Size.**—4 to 23 microns. Average 14.  
Gelatinizing temperature, 72.5°.

**Chloral Hydrate Iodine Reaction**

Part of the grains were reacted upon immediately and stained a deep blue color, but most of them were stained only a very pale blue. After thirty minutes approximately 50% had reacted. The pits at the hila were stained at once and were very prominent. All of the grains had taken on a deeper blue color, while the hilal cavities were irregular at the edges, and stained a dark brownish violet. In many the action seemed to start from the center and proceeded in all directions, while in some it proceeded in only one direction; in others it started at the side and extended over the entire grain. Examined again in an hour the grains had enlarged to three or four times their original size, and usually had a rugged edged, lighter colored central portion surrounded by a darker part. After standing all night some had lost most of their color, while others had taken on a slightly deeper stain.

**Chromic Acid Reaction**

As the grains swelled the hila dissolved and striae developed on the surface. As these disappeared the granular structure became evident. Very few gas bubbles were observed. The action of the reagent on this starch was about the same as on darso, and was complete in three minutes.

**Ferric Chloride Reaction**

The action began within twelve minutes. The hila, which were very prominent and of a dark brown color, disappeared as the grain swelled. In some cases fissures developed from the hilum; these increased in size and came together forming a large hollow in the top of the grain. About 25% of the grains had reacted within fifteen minutes. Striae and developed on some of the grains, and a coarse, granular structure was seen in a few. All of the grains had

reacted within an hour. After standing all night the grains had completely dissolved with the evolution of a considerable quantity of gas.

#### Gentian Violet Reaction

The grains stained a light violet in one minute, which deepened in color for ten minutes, giving a final color of medium violet.

#### Pyrogallic Acid Reaction

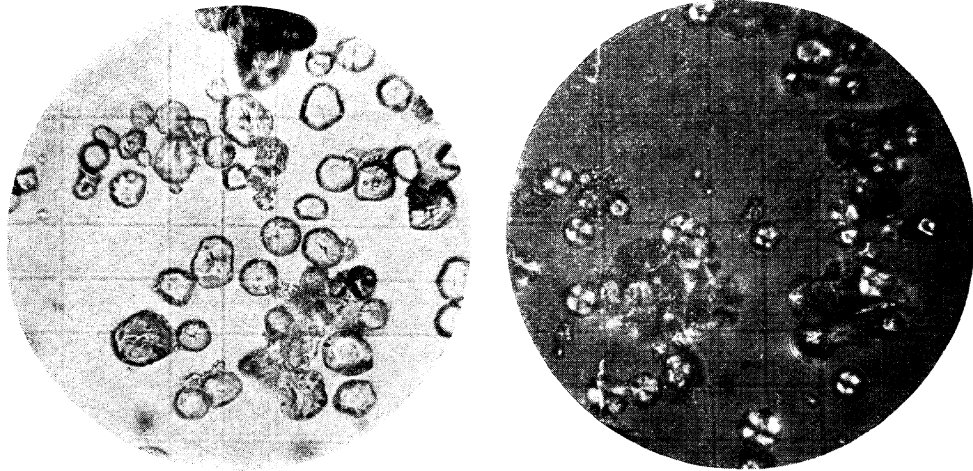
The action commenced on some in three minutes and had started on most of the grains in ten minutes. In twenty-five minutes half of the grains had swollen to a large size, and in an hour and ten minutes all had swollen to two to four times their normal size. Some had developed large openings around the hila, which were very irregular in outline and not very definite. None had developed striae, and there was no granular structure visible in any at this time. In three hours all of the grains had reacted. Some of them were rounded, but most were irregular in shape. The majority of these had shallow fissures or small ridges along their surface on the longitudinal axis. There was no evidence of striae, granular structures, lamellae or gas bubbles.

#### Safranin Reaction

In one minute the grains had taken on no stain at all, but the final light red color developed within five minutes.

### ORANGE CANE

**Microscopic Form.**—Shape round, hilum in center. Fissures in about one-third to one-fourth of the grains. Fissures mostly irregular—neither three-pronged nor straight. No striae or lamellae visible.



**Size.**—6 to 29 microns. Average 16.  
Gelatinizing temperature, 72.4°.

#### Chloral Hydrate Iodine Reaction

The action was not very rapid at first, as only one-half had been affected in twenty minutes. In one-half hour most of the grains had reacted, but the action was not complete before an hour. Most of the grains were stained a light violet-blue; a few stained a very dark blue, and the color gradually deepened as the action proceeded, taking on a more brownish appearance. The hila began to enlarge, deepen and darken; the fissures from the hila to the edge gradually disap-

peared. Fine striae appeared running from the hilum to the edge of the grain. Some of the hila looked like inverted cones, with crevices running down the sides. After the reaction had proceeded for some time the hila developed into pits and the grains showed a large hollow space in the center stained lighter than the surrounding portions. Examined in one-half hour, the grains had taken on a deep blue color and had increased from two to four times their normal size. Some of the reacted grains showed signs of lamellae. After standing all night many of the grains had not reacted to chloral hydrate, although they had all taken on a deeper violet color. Some had been disintegrated entirely, while others had broken into several pieces; these pieces retained their normal polarizing properties. A few of the grains that had completely reacted had a dark blue spot in the center, a lighter area around this, and then another dark area. The dark area seemed to be the outside covering of the grain.

#### **Chromic Acid Reaction**

The action began immediately. The grains swelled at once, closing the central cavity and the fissures around it; as this proceeded a bubble was seen to emerge from the cavity and float on top of the grain. When the action began, fine striae appeared all over the granule, which disappeared as the reaction advanced, then the grains seemed to become filled with fine granules which disappeared almost immediately and the nucleus went into solution. The last part to disappear was the outside of the granule. After all of the starch granules were dissolved the liquid contained a large number of small bubbles. The action was complete at the end of three minutes.

#### **Ferric Chloride Reaction**

The cavity in the center was dark brown and enlarged slightly upon the application of the reagent. About one-half of the grains had reacted within five minutes and had rough outlines.

The fissures around the hilum closed when the grain reached a very large size without development of the striae, bubbles or granular structure. Within half an hour all of the grains had reacted and reached from four to six times their normal size. The hila had disappeared with the exception of a shallow fissure which extended along the longitudinal axis of the grain. No striae, lamellae, granular structures or gas bubbles were seen. The grains had rough surfaces and irregular outlines. The action was complete in three and one-half hours.

#### **Gentian Violet Reaction**

In one minute the grains had taken on a light stain which deepened slightly for five minutes, giving a final stain of light violet.

#### **Pyrogallic Acid Reaction**

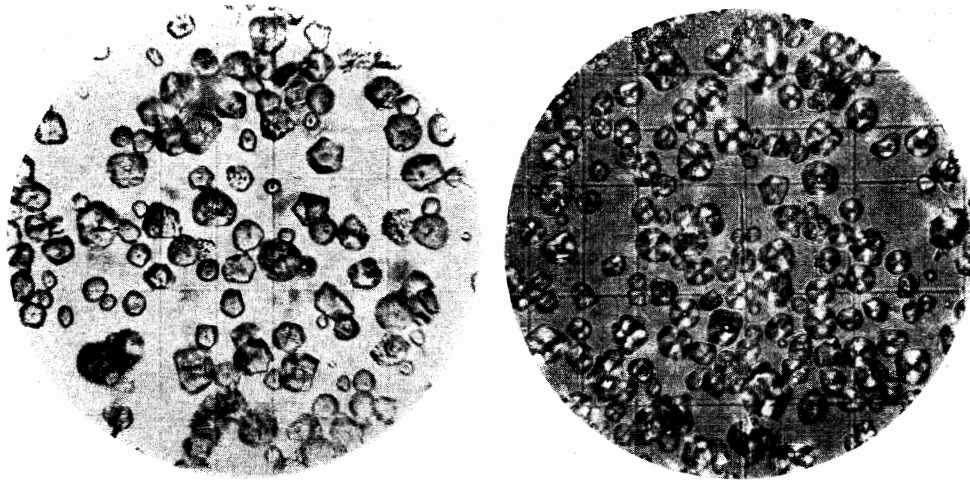
The action started in one or two minutes; in eight minutes three-fourths of the grains were affected, while in twenty-five minutes all of them had begun to react. Upon swelling, the fissures around the hila were not closed, but broadened out and converged at the center, forming a large opening; none of the grains became rounded, but developed irregular, angular surfaces. Most of them showed a hollow space around the hilal vicinity surrounded by a fringe, while gas bubbles formed in the cavity. Examined again after three hours, the grains presented a granular structure and were all very large, but had not lost their rough appearance. There was no evidence of solution, though gas bubbles were scattered throughout the grains.

**Safranin Reaction**

In one minute the grains were only faintly stained. They increased in intensity of color for ten minutes. After this there was no change; the final color was light red.

**CORN**

**Microscopic Form.**—The grains are angular with the hilum in the center. Pressure facets are visible on most of the grains and striae on a few. Small fissures, often running in three directions from the center, occur in many of the grains. There are no lamellae, and the grains do not occur in clusters.



**Size.**—6 to 23 microns. Average 14.  
Gelatinizing temperature, 70.9°.

**Chloral Hydrate Iodine Reaction**

The action was very slow as very few had been affected after ten minutes and only one-sixth after one hour, while many had not reacted to chloral hydrate even after standing all night. The grains did not stain very dark, but the hila were affected immediately and stained a brownish color and enlarged somewhat. The hila cavities had smooth edges, and the shape of inverted cones. After one hour the swollen grains had increased from two to four times in size, had stained a deeper blue, and still retained their polyhedral form. No striae or lamellae appeared at this time. After standing all night most of the grains had taken on a darker blue color, but the grains which had reacted to chloral hydrate had lost most of their color and in some small, unaffected granules having a blue stain, appeared.

**Chromic Acid Reaction**

The action commenced immediately. The grains swelled to about two or three times their normal size, but retained their rough-edged character. No gas bubbles were seen. Striae appeared on most of the granules. These disappeared shortly and the granular structure of the grains developed, but this did not last long. The last part to dissolve was the outer covering. Action was complete within four minutes.



#### Ferric Chloride Reaction

The action started within five minutes. After twenty minutes about one-half of the grains had reacted. The hilal cavity disappeared as the swelling proceeded, while a wide, shallow fissure was seen on top of most of the grains. On an occasional grain striae developed, while on others radial fissures formed around the hilum. These fissures widened and came together forming a rather large opening in the top of the grain. Most of the grains retained their rough character and rounded shape when reacted upon. After one-half hour no granular structures or lamellae were noticed and there was no evolution of gas. The grains increased five to ten times their normal size.

#### Gentian Violet Reaction

The grains took a good stain in one minute which deepened in color for ten minutes, giving a final color of medium violet.

#### Pyrogallic Acid Reaction

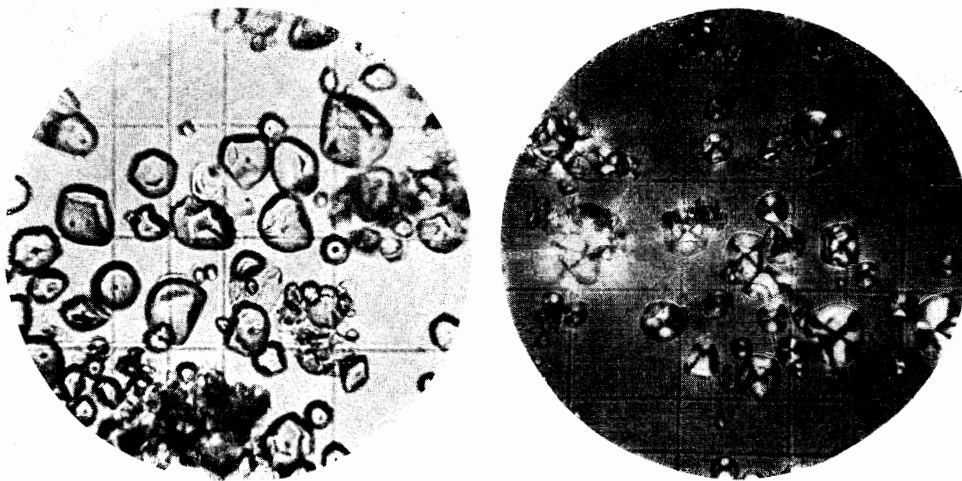
The action was slow in starting as only a few had reacted in four minutes, but when once started proceeded rapidly. In eight minutes half had reacted and they had practically all been affected in sixteen minutes. The action was complete in one-half hour. At first, fine striae extended rapidly from the hilum for about one-half the distance across the grain; these enlarging until a large opening with finer-fringed edges had developed. The surface was rough and angular, very much like the normal grain. No granular structures were noted. Examined again in two and one-half hours, no striae, granular structure or lamellae were noted, but many of them had a large fissure extending over most of the surface of the grain.

#### Safranine Reaction

In one minute the grains had taken on a light stain. This did not change.

### SWEET POTATO

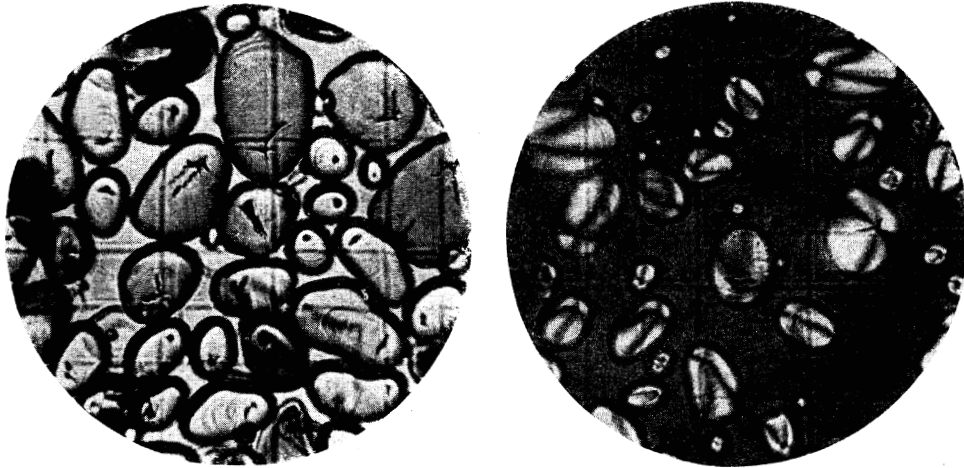
**Microscopic Form.**—The grains are irregular in shape with the hilum in the center in some, but occasionally toward one end. They are often seen in clusters of two to four, and occasionally a double grain. A few grains have striae and fissures around the hilum, but none have lamellae. Pressure facets are present on nearly all.



Size.—6 to 40 microns. Average 19.

**IRISH POTATO**

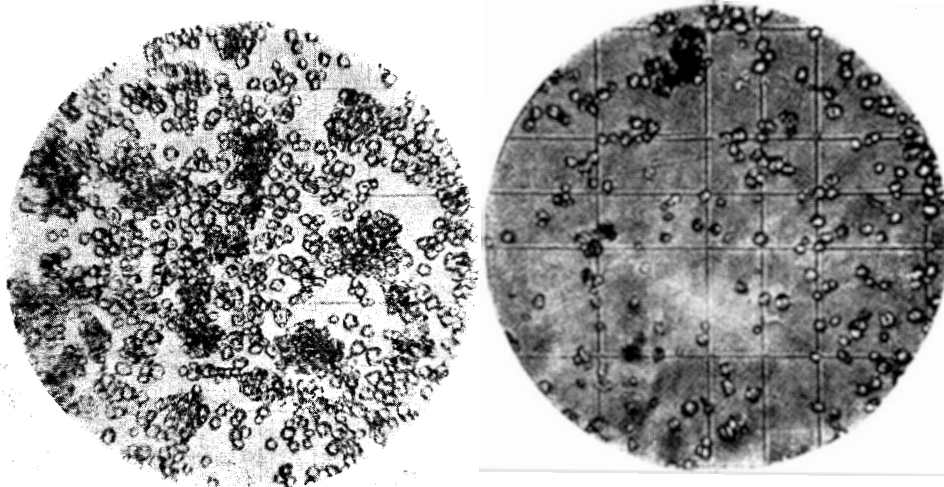
**Microscopic Form.**—The hilum is at one end and generally has fissures running across and along the grain. No striae are present, but lamellae are very large at the distal end, becoming smaller toward the proximal end. The grains are very regular in shape and show no signs of pressure facets.



**Size.**—10 to 110 microns. Average 40.

**RICE**

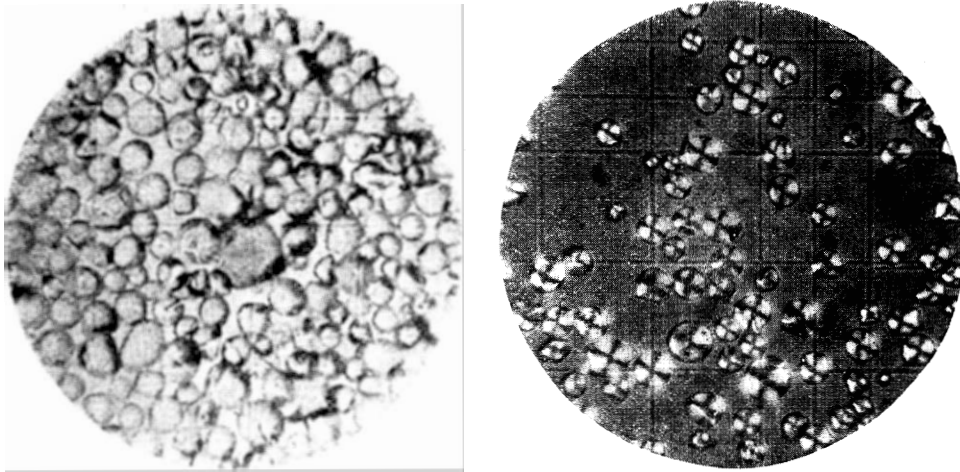
**Microscopic Form.**—The grains are uniformly small and angular, with no hilum, striae or lamellae. All have pressure facets and many occur in short chains and small clusters.



**Size.**—2 to 6 microns. Average 4.

ARROWROOT

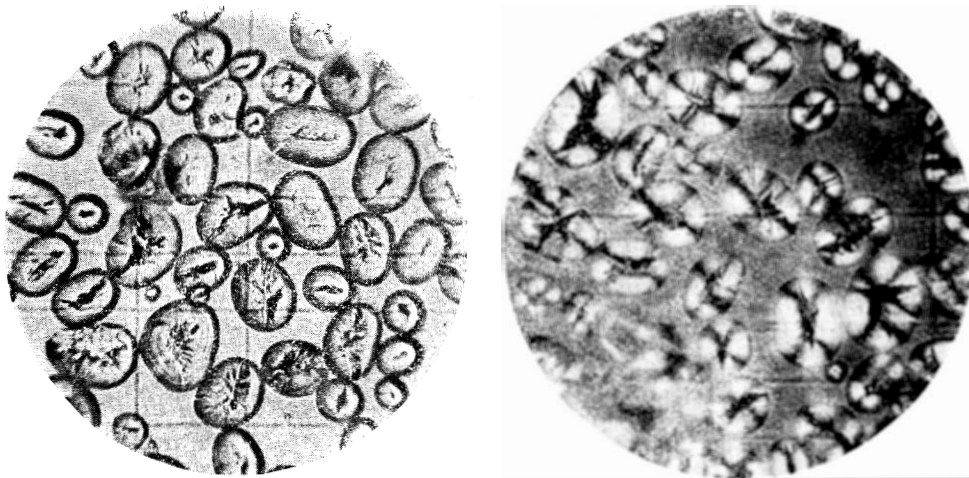
**Microscopic Form.**—The grains are round and regular, with occasionally one with a flat side. The hilum is in the center and is seen as a small, shallow depression from a few of which shallow fissures radiate. There are no lamellae or striae visible.



Size.—7 to 21 microns. Average 17.

NAVY BEAN

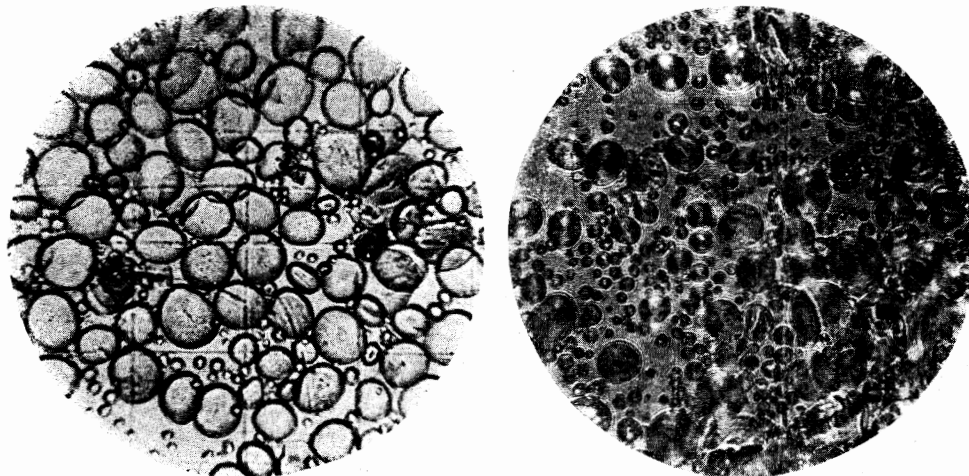
**Microscopic Form.**—The grains are oblong with the hilum in the center and a deep, wide fissure running almost the entire length of the grains. In many cells smaller fissures come off the side of the larger ones. The grains occur singly with no striae, lamellae or pressure facets.



Size.—13 to 43 microns. Average 30.

## WHEAT

**Microscopic Form.**—The grains are round, with neither lamellae, striae, hilum nor facets. They are either large or very small, very few being of medium size.



Size.—3 to 39 microns. Average 23.

## INTENSITY OF THE IODINE REACTION

A 0.5% solution of iodine in the presence of potassium iodide in water was prepared as a stock solution from which solutions varying from 0.1% to 0.01% were prepared. Standard slides were made by staining Irish potato starch with the different iodine solutions. This starch stains very easily, and so good slides of different intensity of color were readily obtained. Small amounts of the starches under examination were placed on the slide and a drop of 0.1% iodine solution added, then the depth of color compared with the standard slides. Irish potato starch stains a pure blue with dilute iodine, but deep violet with stronger solutions.

The number opposite each starch indicates the strength of the iodine solution used with potato starch to produce a tint of the same depth as that obtained with 0.1% iodine solution on the starches examined.

TABLE V

KIND OF STARCH	IODINE REQUIRED FOR SAME TINT AS POTATO STARCH
Red kafir .....	.02 percent
Orange cane .....	.02 percent
Brown kaoliang .....	.02 percent
Yellow milo .....	.01 percent
Feterita .....	.01 percent
Pink kafir .....	.03 percent
White kafir .....	.01 percent
White milo .....	.02 percent
White kaoliang .....	.02 percent
Darso .....	.02 percent
Sudan grass .....	.01 percent
Rice .....	.03 percent
Arrowroot .....	.03 percent
Navy bean .....	.01 percent
Corn .....	.02 percent
Sweet potato .....	.01 percent
Wheat .....	.02 percent

All of the starches produce a good color with the .01% iodine solution, but do not appear to respond to the test so well as potato starch.

## SUMMARY

The grains of the grain sorghums contain starch in amounts varying from 55% to 64%. Feterita, the milos and kafir, containing about 64% starch, seem to be especially suitable as raw materials for the manufacture of high grade starch by the commercial processes, and if they are used will require practically no change in the machinery now in common use for manufacturing starch from corn.

The starches of the grain sorghums show a definite shape when examined microscopically, and exhibit the distinguishing crosses peculiar to starches when viewed by polarized light.

The grains are similar in shape to those of cornstarch, but somewhat larger in size. The measurements are summarized in Table VI.

TABLE VI

## Size of Starch Granules of the Grain Sorghums Compared with Other Starches

KIND OF STARCH	DIAMETER MICRONS	AVERAGE
White kafir .....	4- 23	17
Red kafir .....	6- 33	18
Pink kafir .....	6- 20	16
White milo .....	6- 29	20
Yellow milo .....	6- 26	17
Brown kaoliang .....	3- 23	16
White kaoliang .....	4- 23	20
Feterita .....	6- 26	18
Darso .....	6- 23	18
Sudan grass .....	4- 23	14
Orange cane .....	6- 29	16
Navy bean .....	13- 43	30
Rice .....	2- 6	4
Corn .....	6- 23	14
Irish potato .....	10-110	40
Sweet potato .....	6- 40	19
Wheat .....	3- 29	23
Arrowroot .....	7- 31	17

The gelatinizing temperature of the starches extracted from the grain sorghums varies from 64.7°, pink kafir, to 78°, white kaoliang. The gelatinizing temperature of most of them is close to 74°, and consequently slightly higher than cornstarch. The points of gelatinization have been determined in this work by a new method and with original apparatus.

The comparative staining values of these starches, with the addition of a few common starches, are summarized in Table VII:

TABLE VII  
Comparative Staining Values

KIND OF STARCH	GENTIAN VIOLET		SAFRANINE	
	DEPTH OF STAIN	MINUTES REQUIRED TO STAIN	DEPTH OF STAIN	MINUTES REQUIRED TO STAIN
White kafir .....	Light	1	Light	10
Red kafir .....	Light	1	Light	10
Pink kafir .....	Medium	30	Light	5
White milo .....	Light	30	Medium	30
Yellow milo .....	Light	1	Medium	30
Brown kaoliang .....	Light	1	Medium	30
White kaoliang .....	Light	1	Medium	30
Feterita .....	Light	1	Medium	10
Darso .....	Light	1	Light	30
Sudan grass .....	Medium	10	Light	5
Orange cane .....	Light	5	Light	10
Navy bean .....	Light	10	Light	1
Rice .....	Light	30	Light	5
Corn .....	Medium	10	Light	1
Irish potato .....	Heavy	10	Medium	1
Sweet potato .....	Medium	30	Light	10
Wheat .....	Light	5	Light	10
Arrowroot .....	Medium	30	Light	5

There are no striking differences in the appearance of the various sorghum starches, but on the whole they resemble cornstarch when subjected to the action of staining and swelling reagents.

The fact that there are but slight differences in these starches confirm the very close botanical resemblances of the plants.



